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HEATING

# A COMPREHENSIVE GUIDE TO HEAT TREATMENT

A collection of articles by Dan Herring,
**The Heat Treat Doctor™**

# A SPECIAL MESSAGE FROM DAN HERRING,
# THE HEAT TREAT DOCTOR™

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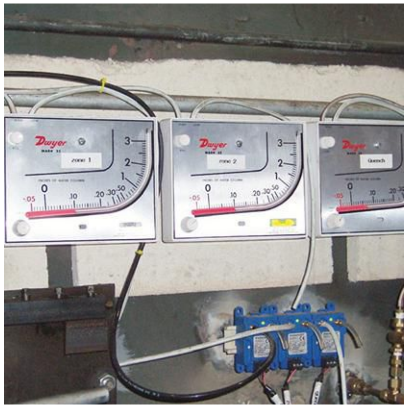

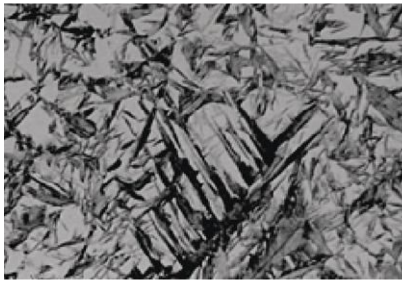

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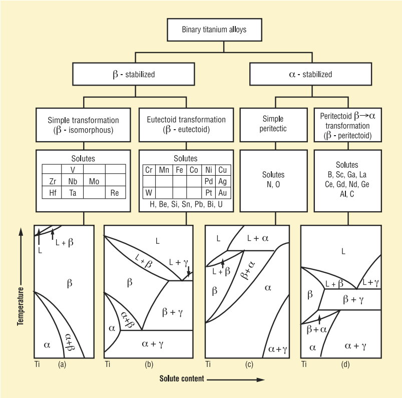

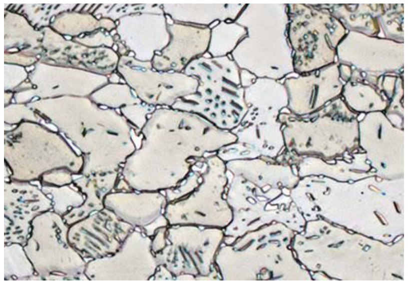

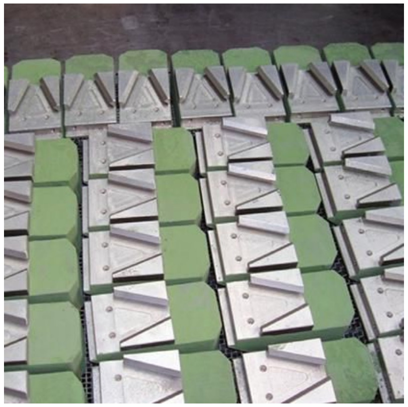

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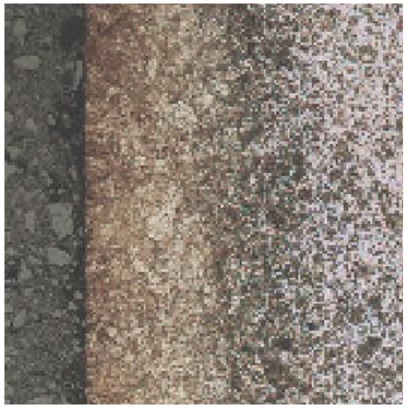

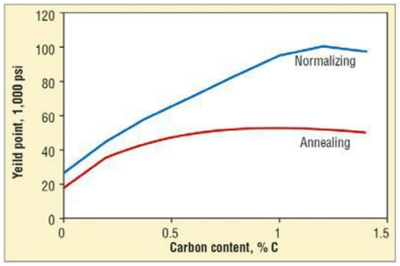

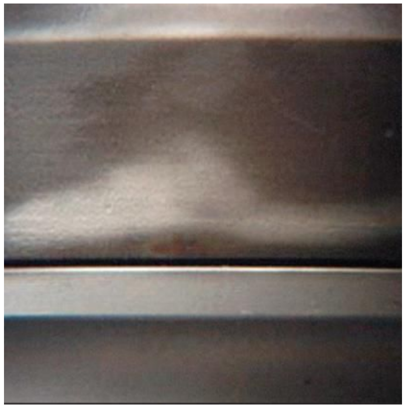

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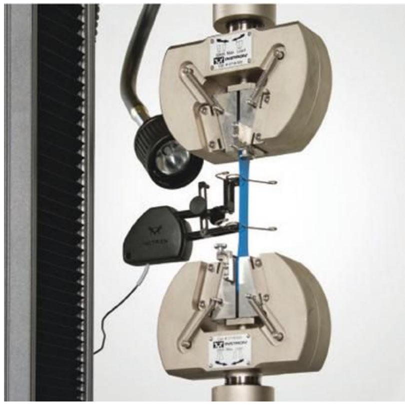

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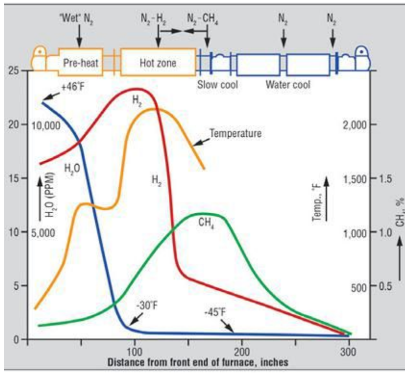

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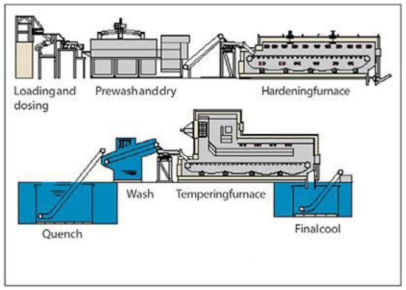

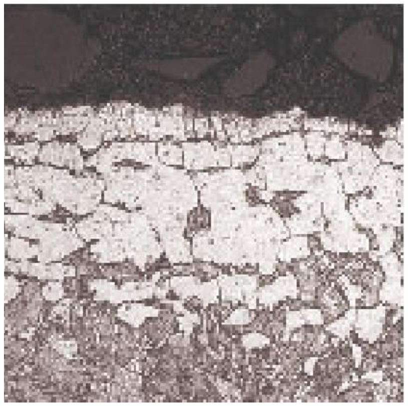

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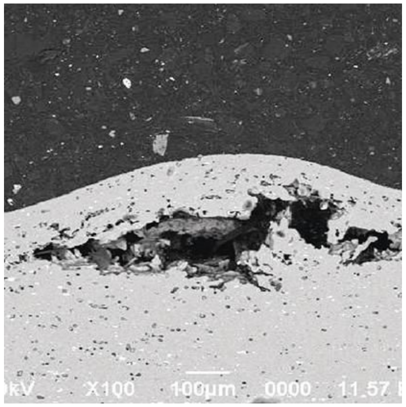
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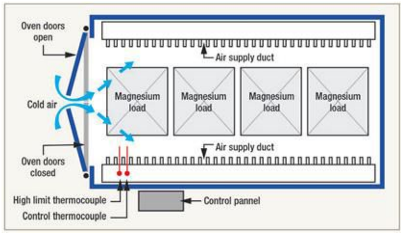
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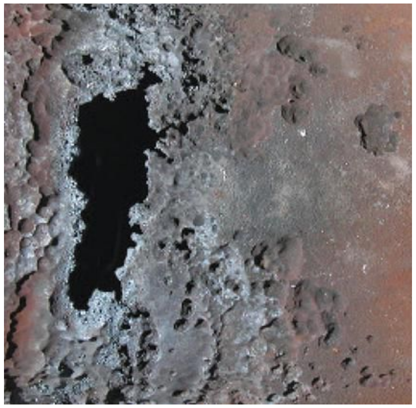

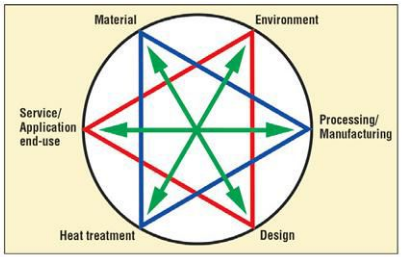
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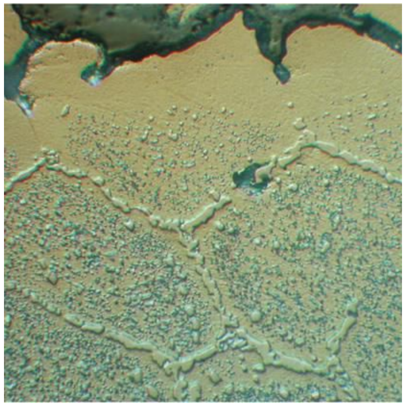

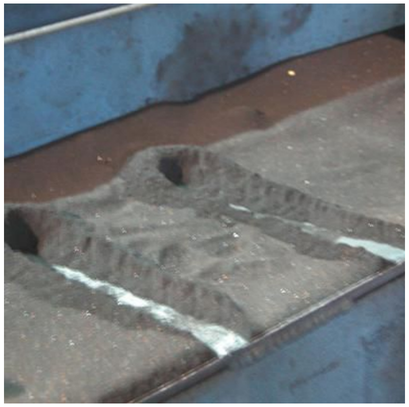

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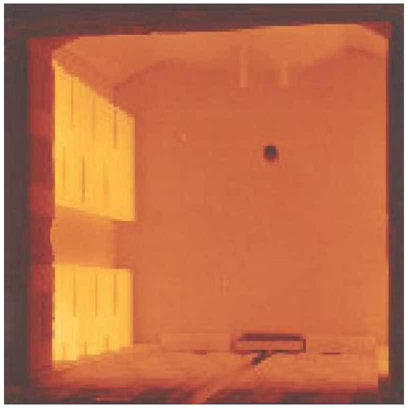

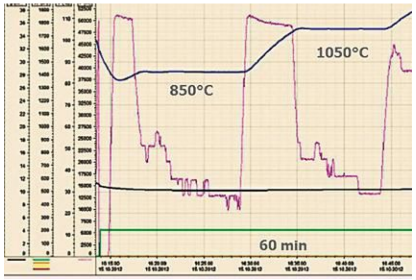

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# What is Heat Treating, and Why Do We Do It?

What began as a simple labor of love years ago, namely to share what we've learned with the heat-treat industry, has reached an impressive milestone. This is the 100th column for "The Heat Treat Doctor"! So, how do we celebrate? By asking ourselves the most fundamental of questions: What is heat treating, and why do we do it? Let's learn more.

Heat treating is a core manufacturing competency and can best be defined as "the controlled application of time, temperature and atmosphere to produce a predictable change in the internal structure (i.e. microstructure) of a material." Thus, metallurgists are responsible to predict the microstructural changes that will occur in a component, while heat treaters are responsible for controlling the process and equipment variables so that the desired outcome will be achieved.

Heat treating allows us to vary the properties (mechanical, physical, metallurgical) of a given material to optimize its design performance. We heat treat, therefore, quite literally because we must. It is the most cost-effective way to achieve the desired outcome. One of the greatest challenges we face as heat treaters is that the same flexibility that allows us to manipulate the end result of a heat treatment, tests our ability to control and repeat our processes to achieve our customer's desired product performance time after time.

Heat treating is a vital part of manufacturing. Thus, it is critical for any organization that relies on this technology to understand the relationship that exists between the variables that influence product response to heat treatment; namely material choice, properties, part design, manufacturing practices and heat-treating methods. This relationship is best illustrated by the Model of Material Science (Fig. 1).

The model is intended to help us understand the inter-relationship between each technology link. Just as a chain is only as strong as its weakest link, so too is the success of a heat-treatment operation dependent on proper execution of each phase of the model. The model explains how to achieve results using both the older philosophy (discovery-based methodology) and the newer approach (scientific-based) methodology.

The scientific- or engineering-based methodology (down arrow) starts by considering the needs of the end user, that is the performance

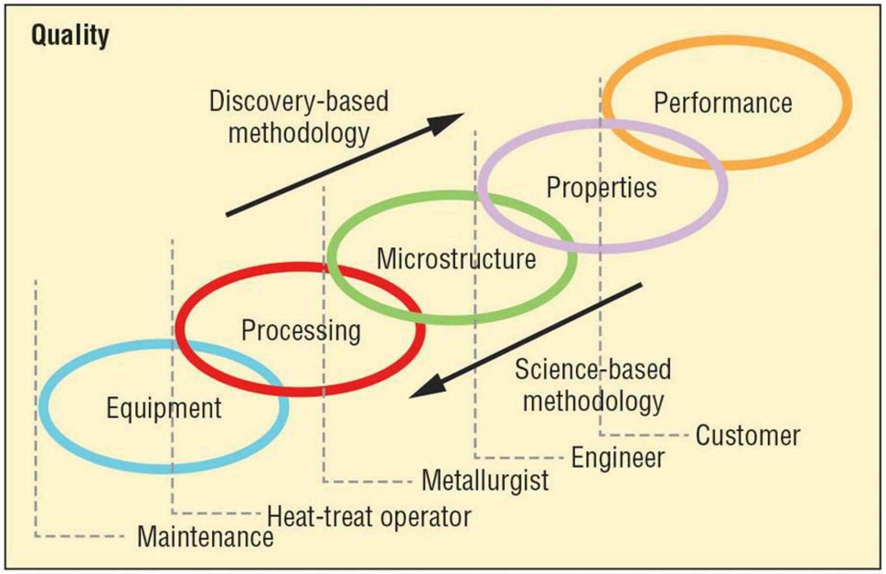

Fig. 1. Model of Material Science

Fig. 2. The Apache Helicopter (Photography courtesy of The Boeing Company)

demands of a specific product, which, in turn, requires the design engineer to select a material capable of achieving certain mechanical, physical and metallurgical properties. These properties can only be developed in the selected material by producing the correct microstructure that in turn dictates a particular heat-treatment process or series of processes be run in a specific piece of equipment.

The discovery or trial-and-error methodology (up arrow) begins with material selection. It involves using a specific piece of equipment to run a heat-treatment process or series of processes, thus producing a specific microstructure in the material, which, in turn, determines the mechanical, physical and metallurgical properties that the material will achieve and ultimately defines the end-use performance capability of the product.

While both methods can be employed with success, most heat-treatment operations today focus on the engineering methodology to more accurately predict and control the material (and hence product) response to heat treatment. Quality assurance and control is achieved by monitoring and checking each step along the way.

A good example of using this methodology is the range of products and services used in the most demanding service applications (Fig. 2). The Apache helicopter, for example, is a unique fighting platform, one that places the highest demand on each subsystem including the transmission, engines and weapons platform. The heat-treating challenges are obvious to ensure that each flight system is optimized. This type of challenge underscores the need to optimize our processes and to ensure that our equipment is under total control.

Many metalworking operations (e.g., grinding, stamping, rolling, forming, machining and plating) shape, size or produce finishes on metals, but only heat treating can significantly change the ultimate condition (physical, mechanical, metallurgical) of these shapes. Virtually all materials can have their properties enhanced by heat treatment.

Many heat-treating operations fall into two basic categories: softening (e.g., annealing, normalizing) and hardening (e.g., through hardening, case hardening). Softening removes stresses, refines grain structure and puts a material in a workable condition for subsequent operations. Hardening often improves surface hardness and wear resistance, increases toughness and improves resistance to impact so that the final product is a useful engineering material.

**Table 1.** Heat-treating equipment supplied by type of process[a]

| Type of Process | Equipment supplied | Type of process | Equipment supplied |
|---|---|---|---|
| Age hardening | 3% | Nitriding | 3% |
| Annealing | 8% | Nitrocarburizing | <1% |
| Austempering | <1% | Normalizing | 4% |
| Bake-out | <1% | Other | 9% |
| Brazing | 7% | Oxidizing | <1% |
| Carbonitriding | 2% | Physical vapor deposition | <1% |
| Carburizing | 8% | Plasma processes – other | <1% |
| Chemical vapor deposition | <1% | Quenching | <1% |
| Co-Firing | <1% | Reduction | <1% |
| Cryogenics | < 1% | Shrink fitting | <1% |
| Decarburization | <1% | Sintering | 8% |
| Deep freezing | <1% | Soldering | <1% |
| Glass-to-metal sealing | <1% | Solution treating | 3% |
| Hardening | 21% | Steam treating | <1% |
| Homogenizing | <1% | Stress relief | 5% |
| Metallizing | <1% | Tempering | 9% |

[a]Source: The HERRING GROUP, Inc.

**Table 2.** Type of heat-treating equipment supplied by number of units[a]

| Type of equipment | Number of units |
|---|---|
| Applied energy[1] | 12% |
| Atmosphere furnaces[2] | 62% |
| Miscellaneous | 4% |
| Ovens | 14% |
| Vacuum furnaces[3] | 8% |

[a]Source: The HERRING GROUP, Inc. Notes: 1. Flame, induction and laser. 2. Batch (48%) and continuous (52%) types; a) Batch styles include: box; mechanized box (e.g., integral quench); pit; bell, tip-up, car-bottom; and gantry designs; b) Continuous styles include: cast-link conveyor, mesh-belt conveyors; humpback, pusher; rotary drum; rotary hearth; shaker hearth; walking-beam, vibratory tube and custom designs. 3. Batch (98%) and continuous (2%) types. a) Batch styles include: horizontal, top-loader, bottom-loader designs. b) Continuous styles include: car-bottom, monorail and pusher designs.

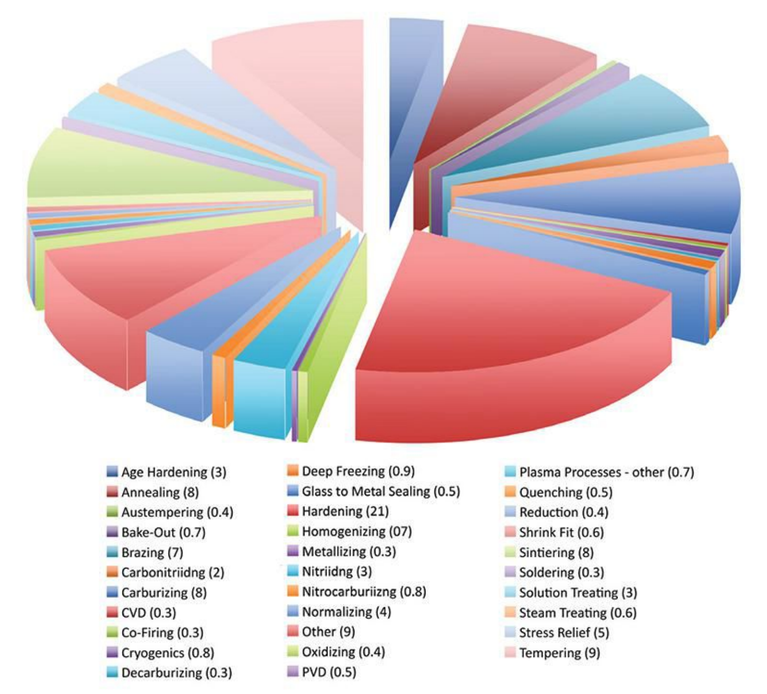

Fig. 3. Heat-treat market (by type of process)

## Heat-Treat Market Size

Heat treating in North America is conservatively estimated to be a $20-22.5 billion industry servicing more than 18,000 manufacturers. It can be further divided between captive shops (approximately 88-92%) and commercial (approximately 8-12%) shops. Further subdivisions are possible by process (Fig. 3, Table 1) and equipment (Fig. 4, Table 2).

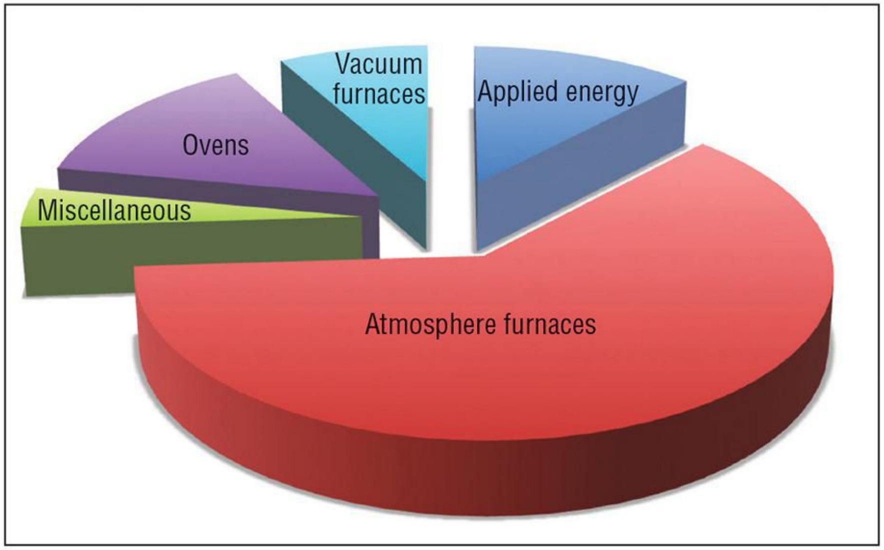

Fig. 4. Heat-treat market (by type of equipment)

## Summing Up

For the heat-treatment industry to survive it must remain the most cost-effective solution to our customer's needs. It is important, therefore, that we understand what makes it such a vital part of the success of today's products and anticipate how it must evolve to stay the choice for tomorrow's innovations. Threats abound, but so too do opportunities for growth and expansion in traditional and non-traditional markets. Heat treating has broken the stereotype of being a low-tech industry. Striving for continuous improvement and relying on sound scientific and engineering principles, with a dash of everyday common sense sprinkled in the mix, will ensure continued success. Rest assured, The Heat Treat Doctor and his column will be there to help!

## References

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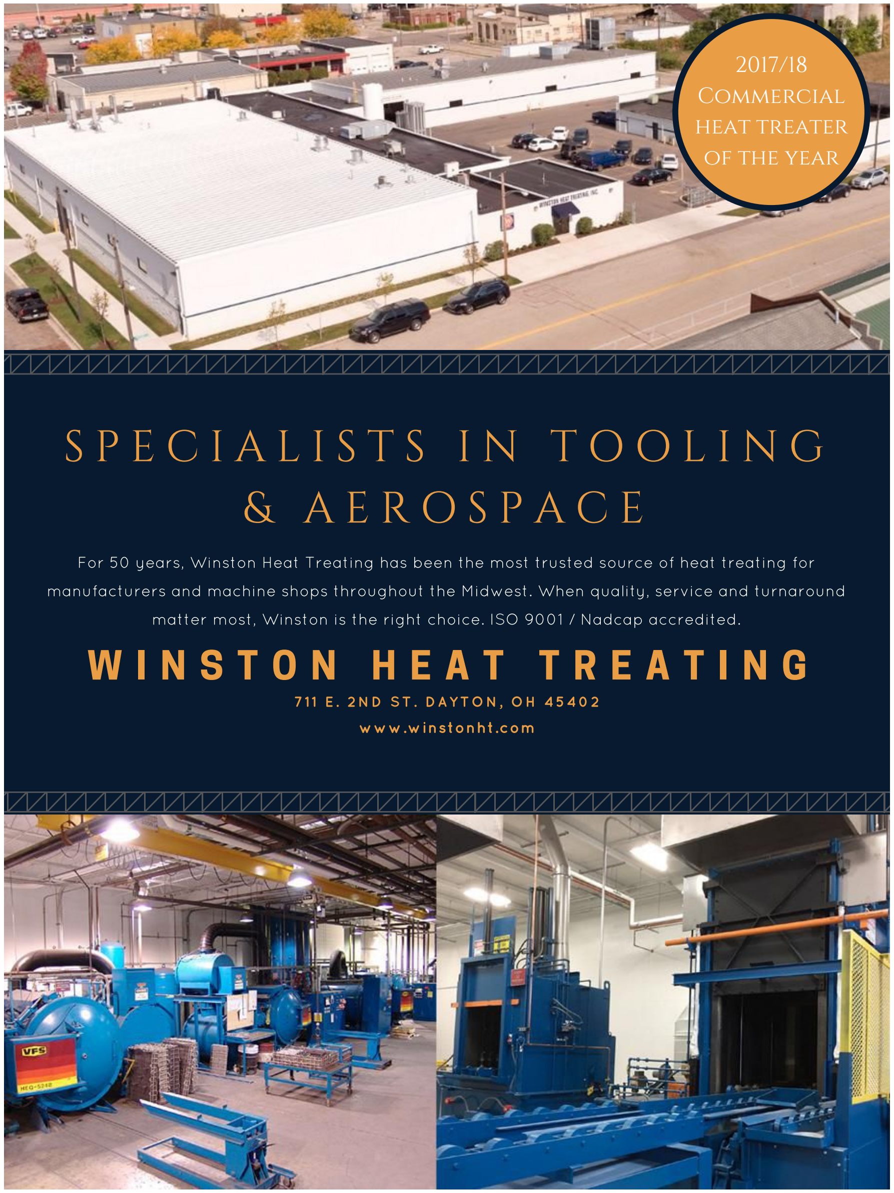

# The Relevance of Metallurgy in Engineering and Manufacturing

We live in a material world. Today, it is the role of materials science (Fig. 1) to study, develop, design and operate processes that transform raw materials into useful engineering products intended to improve the quality of our lives. It is often said that material science is the foundation upon which today's technology is based, and real-world applications would not be possible without the material scientist. With such a broad-ranging scope, the question is, if we really need metallurgical engineers, why are we graduating so few of them? Part of the answer lies in the role metallurgists play in product design and manufacturing. Let's learn more.

The industrial revolution thrust metals to the forefront of technology, and they have stayed there ever since, becoming the very foundation of our modern society. One cannot envision a life where transportation and communications systems, buildings and infrastructure, industrial machines and tools, and safety/convenience devices are not part of our daily lives. Today, other materials have emerged as complements for (or threats to) metal's dominance. Composites are one such example.

Metallurgy is the part of materials science and materials engineering that studies the physical and chemical behavior of metallic elements, their intermetallic compounds and their alloys. This definition is all-encompassing and includes the study of processes run in furnaces and ovens, the forging and rolling of metals, foundry operations, electrolytic refining, creation and use of metal powders, welding, heat treatment and much more.

Metallurgy is also the technology of metals: the way in which science is applied to the production of metals (including heat treatment), and the engineering of metal components for use in consumer products and manufactured goods. The production of component parts made from metals is traditionally divided into several major categories:

- Mineral processing, which involves gathering mineral products from the Earth's crust.
- Extractive metallurgy, which is the study and application of the processes used in the separation and concentration of raw materials. Techniques include chemical processing to convert minerals from inorganic compounds to useful metals and other materials.
- Physical metallurgy, which links the structure

of materials (primarily metals) with their properties. Concepts such as alloy design and microstructural engineering help link processing and thermodynamics to the structure and properties of metals. Through these efforts, goods and services are produced.

## What is Metallurgical Engineering?

Metals and mineral products surround us everywhere – at home, on our way to and from work and in our offices or factories. They form the backbone of modern aircraft, automobiles, trains, ships, and endless recreational vehicles; buildings; implantable devices; cutlery and cookware; coins and jewelry; firearms; and musical instruments. The uses are endless. While threats abound from alternative material choices, metals continue to be at the forefront and are the only choice for many industrial applications.

Developing new materials, new processes to make them, and testing new theories and models to understand them are the focal points for today's metallurgist. We have the means to measure properties at the macro, micro, nano and atomic scales, giving us unprecedented access to fuel new developments. The strong dependence of our society on metals gives the profession of metallurgical engineering its sustained importance in the modern world.

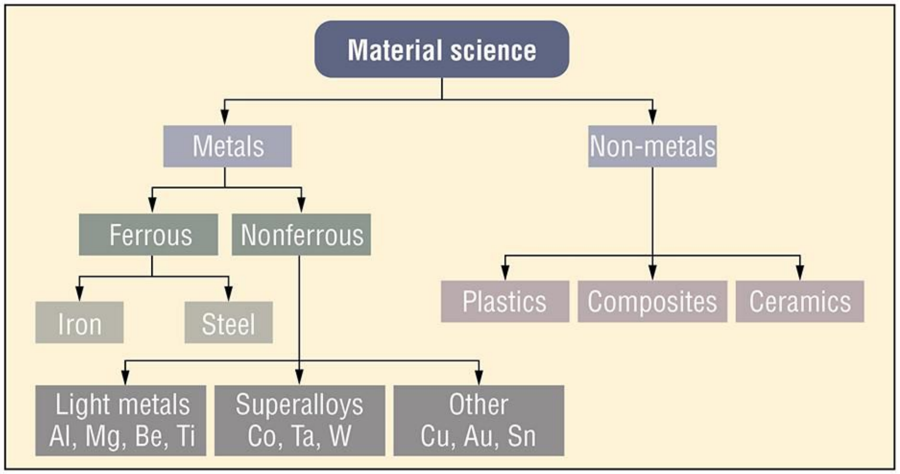

Fig. 1. The material science disciplines

It is believed by most that our economic and technical progress into the 21st century will depend in large part on further advances in metal and mineral technology. For example, advancements in energy technologies, such as the widespread use of nuclear fusion, will only be possible by material developments not yet in existence. The future is indeed bright for today's material scientists and those engineers who chose metallurgy as their career choice.

## Why are there so few metallurgists?

The demand for careers in metallurgy is not at the forefront of our educational system due in large part to the inability of the metallurgical community to communicate to management our role in engineering and manufacturing. While metallurgists should be involved in all aspects of modern engineering, this is seldom the case. The reason for this is often centered around a misunderstanding of what we do, which is made more difficult by how we begin the answer to every question with “it depends.” In many cases, this leads management’s belief that other engineering disciplines can replace our skill set. The failure of management to understand what we do is often a failure to understand the engineering life cycle and the interrelationship of engineering disciplines to each other.

## Engineering Life Cycle

In the design of any engineered component, it is necessary to fully understand and address two key questions that the metallurgist is best qualified to answer, namely:

1. **What must the component endure during service (i.e., what are the product requirements)?**
   Questions such as the following must be addressed: What are the rigors of the application, and what is the design life? Must the component part provide premier service, or is there an adequate design life involved (i.e., will other factors end its service long before its useful life is expended)? What loading, lubricants, temperature and contaminants are involved? What other service/performance aspects specific to a particular product must also be factored into the selection process?
2. **How will the component part be made (i.e., what are the process requirements)?**
   Questions such as the following must be addressed: How will its basic form be generated, and how will it be heat treated – if at all? Will it be important to introduce particular mechanical properties? If so, how – by heat treatment or mechanical means? Is geometry or surface finishes important? Will special coatings be used? Is dimensional control (stability or stability at temperature) an issue? What other processing aspects, specific to the particular product, must be considered?

Obviously, product/process engineering, performance engineering and metallurgical engineering are not separate entities (Fig. 2). They are highly interdependent, and all these disciplines must be considered. However, one must also recognize that today’s cost demands often require compromises in material and manufacturing selection to meet logistical, supply-chain and inventory requirements. Fortunately, that does not mean that selection needs to be minimized. If done correctly, the needs of all parties can usually be met with excellent success

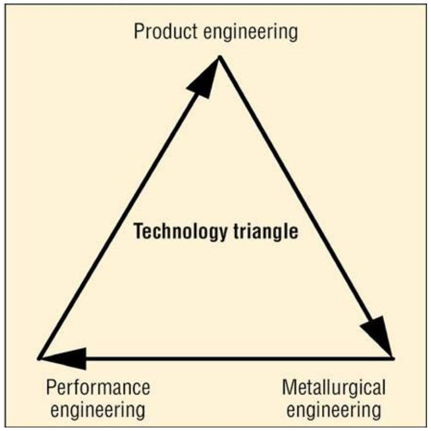

Fig. 2. Technology triangle

while maintaining realistic economic, manufacturing and performance goals.

The role of the metallurgist is especially important during the engineering stage of product development. A metallurgist's participation enhances both the design and the capability of a manufacturing process to achieve the desired outcome. During this phase, there is a point at which manufacturing commences. In order to make this decision, input is required from the so-called technology triangle. The role of the metallurgist or metallurgical engineering group is to provide critical input in the following areas:

- Materials selection
- Manufacturing strategy
- Process development
- Equipment selection
- Controls development
- Variability assessment
- Testing criteria

Metallurgists and metallurgical engineers are also responsible for interfacing with manufacturing to meet production demands in an environmentally responsible way by designing processes and products that minimize waste, maximize energy efficiency, increase performance and facilitate recycling. Metallurgists have seldom been viewed as part of the manufacturing mainstream, however, which is another part of the problem. Gone are the days when every manufacturing plant had a chief metallurgist and multiple metallurgists on staff.

## In Summary

It is often said that mechanical, electrical or computer-related problems can always be solved – if one dedicates enough time and money to the task. However, solving a metallurgical problem is not a function of money. Its solution may be impossible to achieve, forcing one to revisit the very design of the product and its end-use. It is for this reason that the metallurgist exists and is the person who must be involved in every product design. As metallurgists, it is our responsibility to make sure that educators and executives understand the role we play.

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# The Influence of Process- and Equipment-Induced Variability on Product Quality

Like the words to your favorite song playing over and over in your mind, the heat treater's repeating lyric is producing the best quality parts by controlling both equipment- and process-induced variability. Once this lesson is learned, good things happen. Let's learn more.

A problem that recently crossed the Doctor's desk serves to illustrate this concept. A cast link conveyor-belt furnace running various-size bolts from multiple materials (Table 1) was shut down due to a serious quality issue: soft parts across the product line. Over time, a series of minor problems had been reported. These related to the operation of the equipment, none of which had been addressed due to the demands of production.

1. Belt overloading – questionable accuracy and functionality of the weigh scale
2. Sensor positioning – failure to halt loading when overloading or belt stoppage occurred
3. Loader functionality – failure to consistently position parts uniformly
4. Gearbox drive failures – shear-pin breakage
5. Belt tracking – drive roller adjustment
6. Part hang-up during transfer to quench
7. Quench slot and quench chute alloy warpage and refractory wear
8. Controls issues – erratic temperature
9. Noise and vibration in the area of the fans
10. Operational practices – intermittent versus continuous use
11. Maintenance and training of operators, supervisors

When the equipment ran in the past, it produced consistently good work. The last complete reline of the furnace had occurred over 10 years ago. The furnace cast link belt – a major cost item – was approximately three years old and had two length reductions. Past experience suggests that a third belt reduction is possible prior to belt replacement. This translates to approximately one year of remaining service life left for the belt.

## Equipment Inspection

A thorough inspection of the entire furnace system was conducted. The overall condition of the furnace was rated as "good," but a number of problems were revealed.

**Table 1. Bolt materials**

| Steel | C | Mn | P (max.) | S (max.) | Si | Cr | Ni | Mo | B |
|---|---|---|---|---|---|---|---|---|---|
| 1022 | 0.18/0.23 | 0.70/1.00 | 0.040 | 0.050 | | | | | |
| 1035 | 0.32/0.38 | 0.60/0.90 | 0.040 | 0.050 | | | | | |
| 1038 | 0.35/0.42 | 0.60/0.90 | 0.040 | 0.050 | | | | | |
| 1042 | 0.40/0.47 | 0.60/0.90 | 0.040 | 0.050 | | | | | |
| 1045 | 0.43/0.50 | 0.60/0.90 | 0.040 | 0.050 | | | | | |
| 1050 | 0.48/0.55 | 0.60/0.90 | 0.040 | 0.050 | | | | | |
| 4037 | 0.35/0.40 | 0.70/0.90 | 0.040 | 0.040 | 0.20/0.35 | | | 0.20/0.30 | |
| 4140 | 0.38/0.43 | 0.75/1.00 | 0.040 | 0.040 | 0.20/0.35 | 0.80/1.10 | | 0.15/0.25 | |
| 5141 | 0.39/0.44 | 0.70/0.90 | 0.040 | 0.040 | 0.20/0.35 | 0.70/0.90 | | | |
| 5115 | 0.13/0.18 | 0.70/0.90 | 0.040 | 0.040 | 0.20/0.35 | 0.70/0.90 | | | |
| 8637 | 0.35/0.40 | 0.75/1.00 | 0.040 | 0.040 | 0.20/0.35 | 0.40/0.60 | 0.40/0.70 | 0.15/0.25 | |
| 10B21 | 0.18/0.23 | 0.60/0.90 | 0.040 | 0.050 | | | | | 0.0005/0.0030 |
| 10B30 | 0.28/0.34 | 0.60/0.90 | 0.040 | 0.050 | | | | | 0.0005/0.0030 |
| 15B30 | 0.28/0.34 | 0.70/1.20 | 0.040 | 0.050 | | | | | 0.0005/0.0030 |
| 15B35 | 0.33/0.39 | 0.70/1.20 | 0.040 | 0.050 | | | | | 0.0005/0.0030 |
| 50B35 | 0.33/0.40 | 0.75/1.00 | 0.040 | 0.040 | 0.20/0.35 | 0.40/0.60 | | | 0.0005/0.0030 |

**Weigh Scale, Vibratory Loader and Pan Dump System – Overall rating: "F"**

1. The scale failed to "zero."
2. The scale was not weighing part dumps consistently.
3. The scale (overall weight) rating was lower than the actual weight being dumped per charge.

**Refractory Insulation – Overall rating: "C"**

1. The majority of the brickwork inside the furnace was stable and undamaged.
2. No significant cracking of the refractory walls or arch were evident (in undamaged areas).
3. Areas of missing refractory were found.
    - a. One course of arch brick spanning a distance of approximately 66% of the arch width was missing in the immediate vicinity of circulating fan #2.
    - b. The missing refractory separated at the mortar joint.
    - c. Fan #2 was recently replaced reportedly due to "noise" and "shaking." Excessive fan vibration was found to be the root cause of this problem.
4. The area at the rear of the furnace (near the quench chute) had damaged and missing refractory on both sidewalls.
5. Areas of damaged refractory were found.
    - a. Damage to the refractory was extensive in the areas around the radiant tubes. Tube installation techniques were suspected. Training of maintenance personnel on the techniques involved is needed.
    - b. The tube support ends had an extensive amount of refractory missing.
    - c. The burner end had fiber blanket "plugs," some of which needed repair.
    - d. The areas observed will experience excessive heat loss and temperature differentials. However, the fact that these are located near a heat source will tend to mask these effects.
6. The area at the rear of the furnace underneath the cast link belt showed evidence of refractory movement (i.e., bulging) outward.
7. Some damage was evident at the interface between the sidewalls and the arch. Bulging was also observed.
8. The arch area at the rear of the furnace had a number of insulating bricks that had moved (i.e., bulged) outward.

## Special Notes:

1. A section of brick from the furnace sidewall was examined for carbon penetration. Nothing abnormal was observed for a furnace performing the carburizing process.

2. Prior investigations of the furnace interior revealed parts getting stuck in the area of the refractory slot and alloy quench chute.
3. A large number of parts had fallen from the belt and were observed throughout the length of the furnace under the belt. These must be removed prior to returning the furnace to service.

## Alloy Components

1. Cast link belt – Overall rating: "A"
    a. Specific observations:
    (i) No evidence of abnormal wear, distortion or other damage.
    (ii) Belt edges (sides) were in excellent shape.
2. Radiant tubes – Overall rating: "A"
    a. Minor evidence of warpage and sagging.
3. Thermocouple protection tubes – Overall rating: "B+"
    a. Warpage
    (i) Slight amount of sagging observed on thermocouple tubes.
4. Fans – Overall rating: "C-"
    a. Fan #1 showed evidence of metal dusting (i.e., reduction in alloy thickness) in the blade area. Replacement was recommended.
    b. Fan #2 showed no evidence of damage to either the fan blades or fan shaft.
    c. Fan #3 had several holes in the fan blades, which appear to be casting defects. Replacement was recommended.

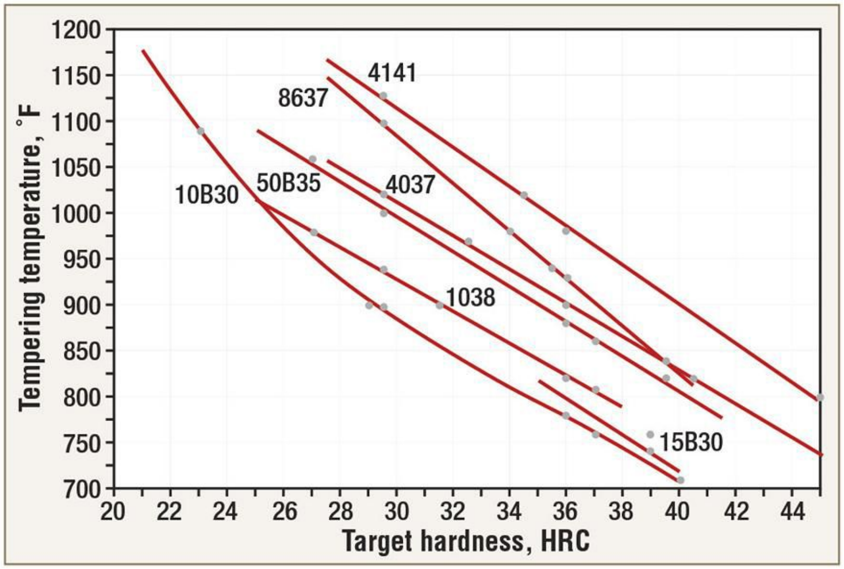

Fig. 1. Acceptable product hardness (product size-dependent) based on austenitizing at 870˚C (1600˚F) and quenching into 70˚C (160˚F) oil

5. Drive and idler rollers – Overall rating: none
    a. The front roller could not be inspected.
    b. No significant warpage or damage of the rear roll was observed (limited inspection was possible).
6. Quench chute – Overall rating: "B"
    a. Some warpage of the chute was evident, but replacement was not warranted.
7. $O_2$ probe protection sleeve – Overall rating: "A"

**Special Note:** Failure of the plant air system was identified as the root cause of damage to the quench chute area (which was air cooled). An alternative method of cooling (nitrogen, water) was recommended if this issue continued to be a problem.

## Root Cause

The soft-product issue was a direct result of the overloading of the cast link belt. The root cause was the cumulative effect of all the minor contributory factors, however, which introduced equipment-induced variability into the process. This resulted in improper heat treatment of the product being run. The parts were being overloaded and either were not reaching the proper temperature, were not at temperature for an adequate amount of time prior to quenching or were hanging up and losing temperature in the quench chute area. Together, these factors overwhelmed the process and combined to produce unacceptable metallurgical and mechanical properties in the final product. Once corrected, acceptable product (Fig. 1) was easily obtained.

## Final Thoughts

This is a classic example where a series of minor equipment and ancillary-item problems resulted in a major quality issue. The old adage that there is never enough time to do it right but always enough time to do it over again is unacceptable. Take the time to correct problems as they arise, even if they interrupt production. This will help avoid major downtime and expense in the future.

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# Industry 4.0 and the 7 Key Considerations in Heat Treating

As anyone who reads this column knows, The Doctor likes to define heat treating as "the controlled application of time, temperature and atmosphere to produce a predictable change in the internal structure (i.e., microstructure) of a material."

The reason for this is that, by its very nature, heat treating allows us to vary the properties (mechanical, physical, metallurgical) of a given material. We can make the parts we heat treat stronger, tougher or more corrosion-resistant. In other words, we can optimize their design performance. But one can, if they are not in control, inadvertently do just the opposite. By now, most of us have heard the buzz about Industry 4.0 and are perhaps wondering what it is, how it will impact us and why we should spend our precious resources implementing it in the heat-treat industry. Let's learn more.

## 7 Key Considerations

In our day-to-day working lives, most (if not all) of us are faced with meeting production demands around tight manufacturing deadlines in an ever-changing materials climate. So for us, there is an alternate and equally important definition of heat treating: "Getting the job done right, the first time and every time." To accomplish this goal, we must satisfy what has been referred to as the seven key considerations in heat treating[1] to ensure our success and survival, namely:

1. To know, metallurgically, what you want to accomplish
2. To be able to predict the outcome of a heat-treatment operation
3. To have repeatability built into the process
4. To get the most out of the equipment we have and to use state-of-the art heat-treating equipment whenever possible
5. To be aware of how changes to our manufacturing operations influence the outcome of a given heat-treatment process or series of processes
6. To not compromise on quality
7. To know our costs and predict our profitability

## Industry 4.0 and the Internet of Things[2]

Industry 4.0 is intended to help us accomplish all of the above. A catchy name coined from the software industry, it is intended to represent the focus on factory automation and data exchange in a manufacturing environment. Recognizing that human

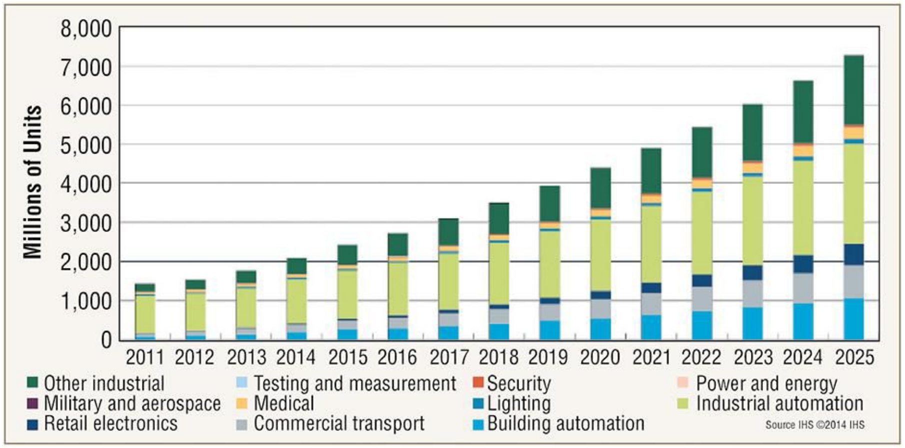

Fig. 1. Industrial devices connecting to the Internet[3]

beings create many of the issues that prevent true process control and repeatability, there is a need to help them with what they do.

Industry 4.0 tools include things such as a "cyber" interface with physical systems (i.e., where a physical device is monitored or controlled by computer systems integrated with the Internet), the Internet of Things (IoT), cloud-based computing and even cognitive computing (i.e., the simulation of human thought by computer modeling). These systems are starting to dominate all industries (Fig. 1).

Industry 4.0 creates what today we refer to as a "smart factory." Industry 4.0 is one in which computers and automation are linked with robotics and connected remotely to computer systems equipped with machine-learning algorithms that can learn and control the systems we run with very little input from human operators. This is why in the realm of heat treatment the emphasis is being placed on in-line single-piece product flow (Fig. 2).

While the term IoT is rather new, the notion of attaching various devices (e.g., sensors) to equipment for measurement and data collection has been with us for many years. But these devices were not always compact or reliable. By way of example, a residual-gas analyzer attached to a vacuum furnace was unheard of a dozen years ago but is a practical reality today.[4]

While various applications and solutions using these devices may have been slow to implement up to now, data collection and reporting done through a myriad of devices and platforms is the next big thing. It is being rapidly implemented into the heat-treat industry, as evidenced by programs such as Ipsen's PdMetrics and SECO/Vacuum Technologies' SECO/Predictive maintenance tool package, which avoid physical collection of information by a technician.

In addition, the reliability of devices being attached to a heat-treat furnace has improved dramatically. Today's powerful computers and cloud tools mean that the analysis process is almost instantaneous and allows us to gain insight from the data we collect, which heretofore was a time-

Fig. 2. Integration of heat treating into true single-piece-flow manufacturing (courtesy of SECO/Vacuum Technologies, LLC)

consuming and labor-intense process.

## Predictive and Preventive Maintenance[5]

A heat-treat maintenance technician who installs and utilizes smart sensors will discover that they help determine the condition of any part of the system or the entire furnace, predict breakdowns (based on mean time between failure) and pinpoint the most cost-effective time and method for maintenance without loss of productivity. That is, Industry 4.0 tools try to predict failure before it occurs using continuous observations and health checks.

A technique of mitigating equipment risk is by performing regularly scheduled maintenance. The schedule is predetermined by forecasting the overall use and normal wear and tear within a given time period. In short, predictive maintenance follows a predict-and-fix model, whereas preventive maintenance follows a repair-and-replace model.

## The Factory of the Future

Quite some time ago now,[6] in an Orwellian-style dream The Doctor presented a vision of the factory of the future. It is worth repeating here given its eerie similarity to where we might be headed.

"The dream. The factory floor is shrouded in darkness and strangely silent. The hum of machinery – the music of manufacturing – and the sound of workers scurrying to and fro are missing. The Factory of the Future welcomes you! An order arrives. In a dazzling display, the lights come on, the factory springs to life, and the shop floor becomes a whirlwind of activity as metal is shaped, heat treated and shipped. The factory floor once again becomes dark and still. The cycle continues. Gone are the heat treaters, having been integrated into the automation sequence. The 'metallurgist on a chip' makes all the right decisions. Is this a vision of our future or a practical necessity to compete? Perhaps this view is a bit extreme. But what does the future hold? The Heat Treat Doctor dusts off his crystal ball …"

We now fast forward to a factory visit by The Doctor only a month or so removed from the time this article will appear in print. The executive running the company shared, "My goal is to arrive here in the morning with a cup of coffee in hand and be the only employee in the plant." His implementation of automation, robotics, vision systems, material handling, in-line testing capability and data acquisition/statistical process control was impressive. The reason – the challenge facing U.S. automotive manufacturers to compete in today's global economy.

## Conclusion

Modernization of the heat-treat shop is critical to both our success and survival as an industry. It will not only be expected from us by our customers but soon be demanded by them as well. As such, it is time to embrace these technological changes in all that we do!

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# ARP-1962: The Need for In-Plant Training and Education Programs

A number of companies and organizations offer a variety of training programs to ensure that heat-treat operators, supervisors, maintenance personnel, quality people, engineers, managers and just about everyone who is involved in heat treating knows and understands the subject. Companies want their employees to know as much as possible about what their company does, what products they produce and how these products are manufactured. The heat-treat process is no exception.

Most training programs are, or should be, designed to meet the requirements of a formal structured program such as SAE-ARP-1962 (Training and Approval of Heat-Treating Personnel). This document calls out both classroom instruction and OJT (on-the-job training) for heat treaters. However, a key element to the success of any program is the inclusion of a "hands-on" training portion by someone skilled in the type of processes and equipment being run. Let's learn more.

## How We Learn

It is important to recognize that learning continues over one's lifetime and is not just a one-time event. Different people learn in different ways: through books, in formal (classroom) settings, by word-of-mouth, by reading trade journals (such as Industrial Heating!), by watching others, from the Internet, by participating in training programs, by trial and error and by being shown how (and, hopefully, why) something works. This last method is what hands-on training is all about. This type of training has been found to be highly effective in shortening the learning curve and breaking bad habits. In addition, the recipients gain practical experience on the furnace systems relevant to them.

Fig. 1. Training methods

## The Value of Training

The payback for training is tremendous including such benefits as:

- Immediate reinforcement of newly acquired knowledge
- Linking of theory and practice in a natural way
- Deeper understanding of the intricacies of heat treating
- A more satisfied and responsible heat-treat operator
- Individuals who see their company investing in them for the future
- A more confident management team due to the investment in personnel training
- Confident employees more willing to take on new responsibilities and offer positive suggestions for continuous improvement
- Customers who gain confidence in the company's ability to provide a quality product or a solid professional service
- The satisfaction and, more importantly, the motivation gained by knowing that things are done correctly

## The Need for Continual Reinforcement

It is essential that BOTH initial and (annual) reinforcement training be done. Too often companies train individuals and feel their obligation is complete. In education circles it is well known that a student must be exposed to the same subject matter (in slightly different forms) a minimum of four to six times before they gain a thorough understanding of the subject. An instructor knows that they must present (teach) a subject at least three times themselves before he or she fully understands it! So how can a student only be exposed once? New hires must also go through both basic and advanced training in order to be successful.

## Why In-House Training?

Valid and compelling arguments can be made for online training (or so-called distance learning), home-study courses and off-site training in a formal classroom

Fig. 2. Typical classroom training session, Ipsen-U (Courtesy of Ipsen USA)

## A Training Course Outline for Vacuum Heat Treatment

**Vacuum heat treating can provide an example of a basic in-house training program that combines classroom instruction with training "on the shop floor." Subjects include (in broad terms): vacuum basics, vacuum principles and vacuum applications. Here's a typical course outline:**

A. Introduction to Heat Treating
   1. What is heating treating, and why do we do it?
   2. Heat-treating processes and equipment

B. Fundamentals of Vacuum
   1. What is vacuum?
   2. Common vacuum units
   3. Pump-down effects (the theory of gases)
   4. Vapor pressure, partial pressure

C. Principles of Vacuum and Vacuum Furnaces
   1. Vacuum pumping systems (the molecule movers)
   2. Vacuum measurement (the molecule counters)
   3. Vacuum valves

D. Vacuum Fundamentals
   1. Vapor pressure, partial pressure and related topics

E. Introduction to Vacuum Furnace Equipment
   1. Types of vacuum furnaces
   2. Hot-zone construction – Materials of construction
   3. Heating elements – Types and repair methods
   4. Control strategies and instrumentation choices
   5. Water systems

F. Vacuum Heat-Treating Applications
   1. Introduction to vacuum processing
   2. The importance of cleaning
   3. Annealing and partial annealing (stress relief)
   4. Normalizing
   5. Fundamentals of brazing
   6. Heat treatment of stainless steels
   7. Heat treatment of tool steels
   8. Low-pressure (vacuum) carburizing and carbonitriding
   9. Sintering

G. Quenching in Vacuum
   1. Factors influencing part distortion and quenching
   2. Fundamentals of gas pressure quenching
   3. Fundamentals of oil quenching

H. Troubleshooting
   1. Maintenance practices, procedures and tips
   2. Maintenance of vacuum subsystems and components
   3. Troubleshooting instrumentation and controls
   4. The why, when and how of leak checking a vacuum furnace (leak rates, leak detection and leak repair)
   5. Setting up a PLANNED preventative-maintenance program
   6. Work preparation (loading)
   7. Baskets, fixtures and racks – Design, alloy selection, maintenance
   8. Diffusion bonding, eutectic melting, outgassing and related topics

I. Testing and Quality Assurance
   1. Types of mechanical testing: Understanding the basics
   2. Principles of hardness testing (Rockwell and Rockwell superficial)
   3. Practical considerations for successful hardness testing
   4. Hardness and hardenability
   5. Failure analysis – Nondestructive testing methods

J. Basic Skill Reinforcement
   1. Temperature scales
   2. Temperature measurement methods (What is a thermocouple?)
   3. Systems of units
   4. Material types and designations
   5. Surface oxidation

K. Miscellaneous
   1. Industry standards and specifications (Nadcap, CQI-9, AMS, ASTM, etc.)

L. Safety
   1. Safety in the heat-treat shop
   2. Gas safety
   3. Confined-entry spaces

M. Management Overview
   1. Management responsibilities
   2. Lean, green and agile manufacturing and lean heat treatment
   3. The challenges ahead

environment. Each of these methods is necessary but should not take the place of in-house education that includes a hands-on element. Home-field advantage should not be underestimated. Individuals to be trained need to see that what they have learned can help them right away, on a day-in and day-out basis. Too often more formalized training materials sit on shelves, sadly never to be looked at again.

In most organizations, OJT is the "default training method" used in lieu of more formalized training. Traditional OJT has been found to be somewhat erratic in that it can produce inconsistent or unequal results from individual to individual. In the writer's experience, one year of OJT is equivalent to approximately 20 hours of classroom training.

OJT is limited by the skill (and experience) of the instructor and in some cases has been found to take up to twice as long as anticipated. It has been reported[2] that traditional OJT training could be replaced by a structured training approach with the result being training time reduced by some 72%, problem-solving ability increased by 130% and wasted time and effort reduced by 76%.

## Why the "Hands-On" Approach?

Training based on a "read it, see it, do it" philosophy combines sound scientific and engineering principles with practical real-world experience and must include plenty of practical examples. Individuals who follow this approach not only learn the subject but also understand how to apply it in their everyday jobs. See you on the shop floor!

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# Conducting Kaizen Events in the Heat-Treat Shop

The Doctor is notorious for showing up and participating in Kaizen events at his customers' plants. These events are the most dynamic learning tool available to the heat-treat industry and involve everyone within an organization, from management, engineering, quality and metallurgy to supervision, maintenance, heat-treat operators and helpers. It is a learning tool of incredible effectiveness. Let's learn more.

## Why a Kaizen event?

The goal of a Kaizen event is to hold a focused, short-duration, task-oriented activity in which everyone involved is "hands on." Nowhere is this more effective than in the heat-treat shop, where the success of the overall manufacturing operation is so dependent on the health of the equipment and processes being run. One piece of equipment or process is normally selected, with the intent of either optimizing its performance or streamlining its functionality so that setups, work flow and processes run efficiently.

Kaizen events are usually three to five days of intense activity where the only job of those participating is to accomplish the task at hand. The following activities are typical:

- Defining the problem/goal (Why are we doing this?)
- Documenting the current state (Where are we?)
- Brainstorming and developing a future state (Where would we like to be?)
- Implementation (How do we accomplish the goal?)
- Training (How do we get better at what we do?)
- Developing a follow-up plan (How do we sustain our success moving forward?)
- Presenting results (What have we learned?)
- Celebrating successes (What's next for us to learn or do?)

Kaizen events are one of the best ways of accomplishing lean manufacturing, improving the overall operation and promoting morale amongst employees. It is critically important, however, to understand the types of problems for which Kaizen events should and should not be used. With proper

**Table 1. Kaizen event action item list**

| Subsystem | Reason | Notes | Action items/team recommendation |
|---|---|---|---|
| Oil heaters | Productivity | Cleaned and tested | Change to vertical orientation to avoid draining tank for replacement |
| Oil pump | Quality | Functionality | Installed pressure gauges on inlet and outlet |
| Oil temperature | Quality | Connected T/C to data recorder | Added temperature recorder |
| Heat exchanger | Safety | Potential water leakage into oil from water-to-oil heat exchanger | Replace with air-to-oil heat exchanger |
| Heat exchanger | Quality | Cleaned and tested | Check for flow restrictions |
| Circulation baffles | Quality | Inspect for damage | Straightened as per manufacturer's recommendations |
| Oil agitator | Quality | Check clearance (1 inch) | Repaired draft tube |
| Stand pipe | Safety | Clogged | Moved standpipe location, piped outlet to a drum for spill containment |
| Vestibule cooling | Quality | Check for flow in each area of the oil-cooled jacket | Need method for operator to visually check for flow |
| Oil level site gauge | Safety | Better indicator needed | Redesign site gauge |
| Oil level min-max | Safety |  | Welded min-max lines on dip stick |
| Front and back baffle plates | Productivity | Oil circulation restriction points | Install grating and add holes for better oil circulation |
| Flame curtain | Safety | Operator can adjust height of flame curtain | Guideline for adjustment needed. Do we want to remove ability of operator to adjust this? |
| Front door | Safety | Elbow to deflect flame up the hood | Thread the hole for addition of elbow |
| Nitrogen purge | Safety | Added nitrogen purge to vestibule | Installed coupling in vestibule and piped nitrogen (including flowmeter) to this location. |

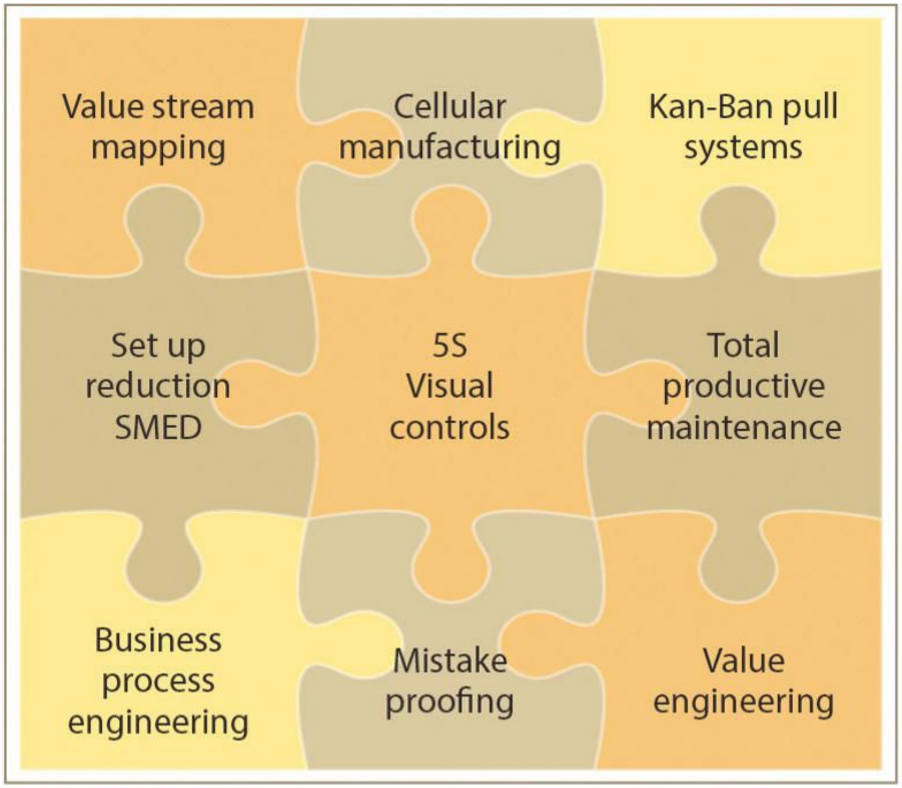


Fig. 1. Considerations in a continuous-improvement plan

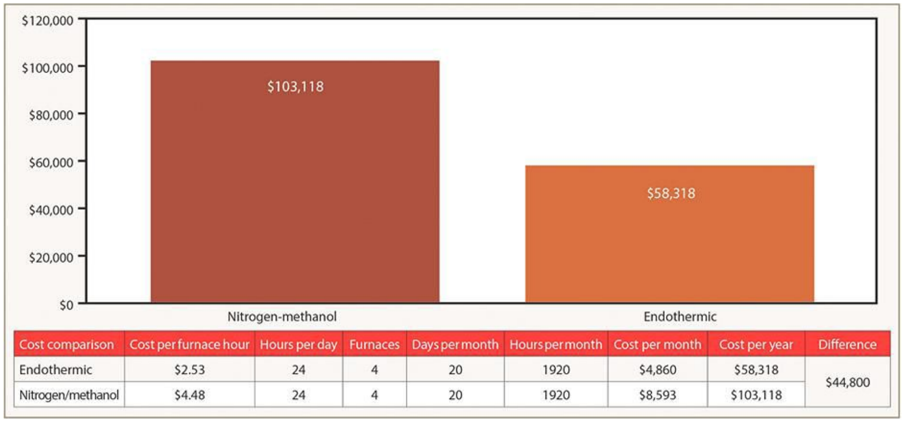
planning, Kaizen events can bring breakthrough improvement to an organization.

## Blueprint for Success

A Kaizen event was performed on one of five integral-quench furnaces in a captive heat-treat shop. The intent was to do a thorough inspection, cleaning, repair and modification (as necessary) of the furnace as a model to implement changes on the other units. The oldest furnace in the line was selected. The goal was to establish a continuous-improvement plan (Fig. 1), including a preventive-maintenance schedule, critical spares management, predictive tool use, TPM and 5S, OEE[2] and review safety/lockout-tagout-tryout procedures, and conduct training.

The work involved looking into the history of the furnace, major disassembly and cleaning, which included draining the quench tank and shoveling out the sludge accumulation from the bottom of the tank, as well as evaluating all of the components on the equipment. A number of items were uncovered (Table 1), including an analysis of the current nitrogen/methanol system versus a potentially new endothermic-gas atmosphere generator (Fig. 2). This convinced management to purchase a gas generator.

Another example of a Kaizen event involved looking at TPM (total productive maintenance) in a single-chamber vacuum furnace. The drivers were that cycle times were being extended, minor cycle interruptions occurred too often, temporary repairs were being done, major breakdowns were occurring at a higher-than-normal frequency, the quality results weren’t as repeatable as expected, and no one had quantified these losses. The work focused on avoiding “the seven wastes,” namely



| Cost comparison | Cost per furnace hour | Hours per day | Furnaces | Days per month | Hours per month | Cost per month | Cost per year | Difference |
|---|---|---|---|---|---|---|---|---|
| Endothermic | $2.53 | 24 | 4 | 20 | 1920 | $4,860 | $58,318 | $44,800 |
| Nitrogen/methanol | $4.48 | 24 | 4 | 20 | 1920 | $8,593 | $103,118 | |

Fig. 2. Comparison analysis of nitrogen/methanol and endothermic

transportation, inventory, motion, waiting, over-processing, over-production and defects (remember Tim Wood).

The goal of the exercise was to achieve:

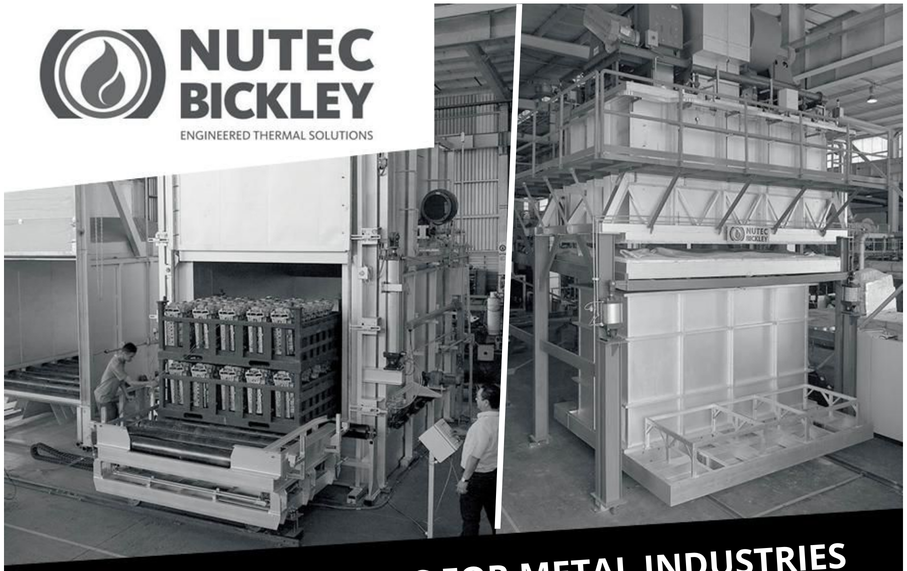
- Safety improvements in equipment operation and working environment
- Reliability of equipment for material conversion, customer satisfaction and lean manufacturing/reduction in WIP inventory
- Preservation of precious capital assets
- Prerequisite to smaller lot sizes and inventory reduction

To accomplish the work effort, the vacuum furnace was disassembled (hot zone removed and vacuum pumps dismantled), cleaned and reassembled. One of the major issues that was uncovered included the design of the front door seal, which was contributing to part discoloration and hot-zone (graphite insulation) deterioration. It was discovered that the door-seal design had been discontinued by the manufacturer, and a retrofit was available. The extent and impact of part contamination (detrimental to the pumping system) was another major finding, and better, more efficient cleaning and more frequent pump maintenance was implemented.

A pleasant surprise during the work was the condition of the internal heat exchanger, shell and internal cooling fan, which confirmed the proper operation of the water system and robustness of the design to avoid contamination even though dirty work was being introduced.

## Heat-Treat Training

Training at a Kaizen event is often spontaneous and done “as needed, where needed” typically on the shop floor in one-on-one situations and then summarized when the group gathers to discuss the progress being made. Informal lunch-and-learn sessions are extremely effective with topics either planned or arising from the work being performed. Reference the web version of this column for



detailed outlines of atmosphere and vacuum heat-treating training programs.

## Summary

Kaizen events in the heat-treat shop bring greater understanding of the challenges that heat treaters face in their day-to-day activities and create a can-do attitude when it comes to problems. Actively participating gives management a greater appreciation for and understanding of our needs and what they can do to help. It is a win-win situation for all involved. Schedule one today!

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# A Primer on Thermodynamics and Kinetics for the Heat Treater

The heat treater might be surprised to learn that in order to understand how various heat-treatment processes create a particular microstructure and its corresponding mechanical properties, it is necessary to have a fundamental understanding of both thermodynamics and kinetics (in this case involving solid-state phase transformations as a function of temperature and composition).[1] Furthermore, when one wants to understand the fundamentals of processes such as gas carburizing or nitriding, we must also employ these principles. Let's learn more.

Thermodynamics is that branch of science concerned with the relations between heat and all other forms of energy (e.g., mechanical, electrical or chemical). By contrast, kinetics is the study of reaction rates – the forces acting on various mechanisms (e.g., chemical, physical).

## Thermodynamics is Related to Stability[2]

Thermodynamics is all about how stable materials are in one state as opposed to another rather than about things moving or changing. In other words, thermodynamics has nothing to do with time. One must also be careful not to confuse thermodynamic quantities (e.g., Gibbs free energy of a chemical reaction) with kinetic ones (e.g., activation energy of a chemical reaction).

Thermodynamics can tell you only that a reaction should proceed to completion because the chemical products (i.e., what is formed in a chemical reaction) are more stable and thus have a lower free energy than the reactants (the substance that undergoes change in a chemical reaction). Another, albeit more scientific, way of saying this is that the reaction has a negative free energy.

The change in free energy ($\Delta G$) is negative. Therefore, the reaction is spontaneous. The formula $\Delta G = - RT\ln K$ says all this at once, where R is the gas constant, T is absolute temperature in degree Kelvin and K is the equilibrium constant. Still another way of saying this is that the reaction has a large equilibrium constant, signifying that if nature could ever attain equilibrium, there would be many more products present than there are reactants; the products are at a lower free energy, or more stable, than the reactants.

Because of this, the reactants want to be converted into the products. For example, graphite and

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Table 1. Summary of the differences between K and k[2]

| Equilibrium constant, K | Rate constant, k |
|---|---|
| Thermodynamic, not kinetic | Kinetic, not thermodynamic |
| K is dimensionless | Rate constant changes with temperature and in the presence of a catalyst (e.g., the catalyst in an endothermic gas generator). The catalyst changes the activation energy for the rate-determining step. |
| K is independent of the reaction mechanism. It is written from the products and reactants in the final, overall reaction equation. | The bottleneck (slowest step) determines the reaction rate (and corresponding rate law). One must know the reaction mechanism. Only reactants appear in the rate law. |
| The equilibrium constant takes into account essentially constant concentrations (solids, pure liquids). | Steady-state approximation can be used since intermediary products do not influence the rate law. |
| The value of K depends on the stoichiometric coefficients of the equation to which it is referenced. | |
| Equilibrium may take a long time to achieve. | |
| For exothermic reactions, heat is a product. For endothermic reactions, heat is required (Le Chatelier). | |

diamond are both forms of carbon, but graphite has a lower free energy. Therefore, diamond wants to convert into graphite. Another example is that your skin wants to dissolve in the soap when it is washed. In this case, the products of the dissolution reaction (namely, your skin dissolved in the soap) are more stable than the reactants (your undissolved skin and the soap separately).[2]

The above examples were chosen specifically because even though the reaction should proceed (since the products are more stable than the reactant), it does not. Your hands are safe when you wash them as is the diamond on a woman's finger. Even though the reaction is thermodynamically favorable, it is slow. It is far too difficult to get the diamond to break all of its bonds and re-form them to a different, more-stable graphite configuration. Even though the reaction should go thermodynamically, it does not because it is kinetically unfavorable.

## Kinetics is Related to Reactivity[2]

Kinetics is about how quickly or slowly species react. Kinetics can tell you how fast the reaction will go but, as seen above, doesn't tell you anything about the final state of things once it gets there. Reactions occur at many different rates. For example, compare a geological change with that of combustion in a burner.

The rate constant, k, measures how fast a chemical reaction reaches equilibrium assuming the reactants were supplied with enough activation energy to enable the reaction to proceed in the forward direction – reactants to products. This requirement for input of energy symbolizes the fact that the reactants are unreactive under certain conditions. The reaction must have some form of energy input before it can proceed; otherwise, the reactants cannot cross the activation-energy threshold and convert to products. The reaction is activated by energy supplied to the reactants by different energy sources. The rate of reaction, the rate constant and the kinetic energy required for activation of reaction indicate how fast the reaction reaches equilibrium.

One can compare the equilibrium constant (K) of a reaction with the rate constant (k), which tells you the rate of an elementary step in the reaction mechanism (Table 1).

## Heat-Treat Example[3]

Kinetics of mass transfer at the gas-steel interface for carbon diffusion in steel is an example of research in recent years that was dependent on a fundamental understanding of thermodynamics and kinetics. It has allowed us to develop a fundamental understanding of the mass transfer during the gas-carburizing process and included modeling of the thermodynamics of the carburizing atmosphere with various enriching gases. The models accurately predict: the atmosphere gas composition during the enriching stage of carburizing; the kinetics of carbon transfer at the gas-steel surfaces; and the carbon diffusion coefficient in steel for various process conditions and steel alloying.

From a thermodynamic standpoint, the generation of the carburizing atmosphere in gas-carburizing atmosphere reactions is a rather complex process involving the interaction of numerous gases. It has been estimated that nearly 180 chemical reactions occur simultaneously in the carburizing atmosphere, among which only the following three reactions are important and determine the rate of carbon transfer from the carburizing atmosphere to the steel surface:

(1) $2CO \rightarrow C_{(\gamma\text{-Fe})} + CO_2$

(2) $CH_4 \rightarrow C_{(\gamma\text{-Fe})} + 2H_2$

(3) $CO + H_2 = C_{(\gamma\text{-Fe})} + H_2O$

While carburizing most rapidly proceeds by CO-molecule decomposition, the by-products of the carburizing reactions ($CO_2$ and $H_2O$) act as decarburizing agents. The presence of $CO_2$, even in small quantities, requires a high CO concentration to balance this decarburizing action. Therefore, these decarburizing species must be reduced for the process to proceed further. Generally, the maximum amount of $CO_2$ that is tolerated at a particular carburizing temperature without causing decarburization can be determined based on thermodynamic calculations.

Since carburizing with endothermic gas only is practically inefficient and requires large flow rates, the endothermic carrier gas is enriched by blending with an additional hydrocarbon gas. The purpose of the enriching gas is to react with $CO_2$ and $H_2O$, thus

reducing their concentration and producing more CO and $H_2$ as reaction products by:

(4) $CH_4 + CO_2 = 2CO + 2H_2$

(5) $CH_4 + H_2O = CO + 3H_2$

Although enriching reactions 4 and 5 are slow and do not approach equilibrium, the effectiveness of the carburizing process is determined by the atmosphere carbon potential and controlled by the ratio of $CO/CO_2$ and H2/H2O components in the heterogeneous water-gas reaction:

(6) $CO + H_2O = CO_2 + H_2$

## Summary

It is a mistake for all of us not to have a basic understanding of the role of thermodynamics and kinetics in heat treatment. They are two words that belong in every heat-treater's lexicon.

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# Heat Transfer

Most, if not all, of us know that heating (and cooling) of parts happens by three mechanisms: radiation, convection and conduction. While simple examples illustrate the basic concepts, the underlying science, while a bit more complex, is something all heat treaters should know. Let's learn more.

## The Science Revealed

### Radiation

Radiation is a method of heat transfer that does not rely upon direct contact between the heat source and the object being heated. For example, we feel heat from the sun (Fig. 1) even though we are not touching it. Radiation is also line-of-sight heating. If an object (such as a component part buried inside a dense load) is not directly in the path of the radiant energy, it will only heat when the surrounding objects (parts, in this example) are heated and re-radiate their heat energy to the buried object. This is why part loading/spacing in high-temperature atmosphere furnaces and in vacuum furnaces is so important.

Everyday experience has taught us that black or dull surfaces are better at absorbing radiant heat energy than shiny, reflective surfaces. What we don't often appreciate is that the radiant energy (the amount of thermal energy being radiated per second per unit area) is proportional to the fourth power of (absolute) temperature (Eq. 1). Thus, we must be extremely careful when we heat something to ensure that we do not overheat it by radiation. This is why, for example, aerospace specifications do not allow direct exposure of radiant heating sources to the parts (e.g., during the solution heat treating of aluminum), which is performed close to the melting point of the material. Heat loss by radiation can occur in any situation, but it becomes more important as the temperature increases. This is why insulation in high-temperature furnaces must be carefully designed.

The rate of radiant heat transfer is given in equation 1.

$$Q=A\bullet K\bullet F_e\bullet F_s\bullet(T^4_{radiator} - T^4_{receiver}) \qquad (1)$$

Q is the heat transferred in watts (Joule/second), A is the surface area in $m^2$, K is a constant (Stefan-Boltzmann), $F_e$ and $F_s$ are the emissivity of the emitter and the surface respectively, and T is the temperature of the radiator and receiver (in ˚K).

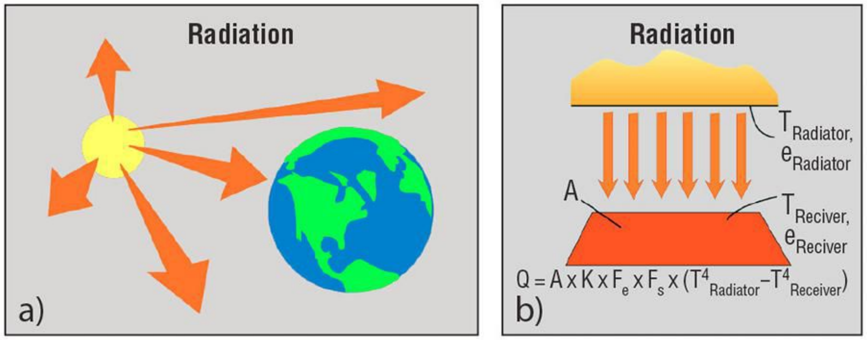

Fig. 1. Example of radiation[1] (a) The sun heating the Earth, (b) The science of radiant heat transfer

## Convection

In simplest terms, convection occurs when a (solid) object comes in contact with a liquid or gas at a different temperature. It will always involve the liquid or gas in some state of motion. A hair dryer (Fig. 2) is a simple example in which heated air exits the dryer at a speed such that it transfers heat energy to the object it strikes (wet hands, wet hair, etc.).

The rate of convective heat transfer is given in equation 2.

$$Q=h_c \bullet A \bullet (T_{fluid} - T_{object}) \qquad (2)$$

Q is the heat transferred in watts (Joule/second), $h_c$ is the heat-transfer coefficient in W/m$^2$-˚K, A is the surface area in m$^2$, T is the temperature in ˚K, and t is the time in seconds.

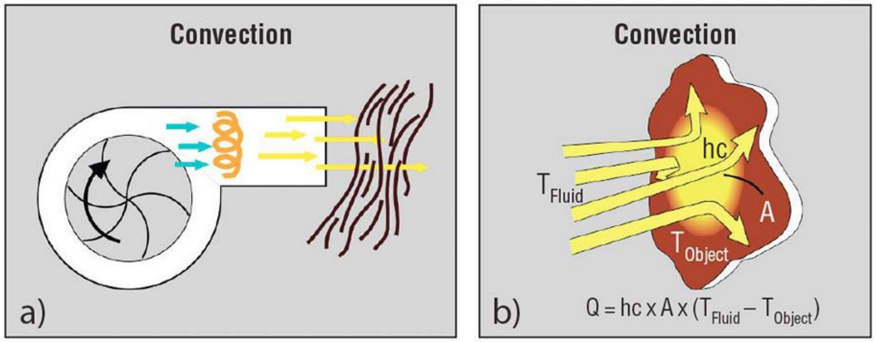

Fig. 2. Convection example[1] (a) Hair dryer, (b) Science of convective heat transfer

## Conduction

Conduction occurs when two objects at different temperatures are in contact with each other. Heat flows from the warmer object to the cooler object until they are both at the same temperature. A classic example of heat transferred between two bodies is when a metal spoon is left in a hot cup of coffee (Fig. 3). Solids are better conductor than liquids, and liquids are better conductor than gases.

The rate of conductive heat transfer is given in equation 3.

$$Q=k \bullet \frac{A \bullet (T_{hotter} - T_{colder})}{L} \qquad (3)$$

Q is the heat transferred in watts (Joule/second), k is the thermal conductivity of the material in W/m-˚K, A is the surface area in m$^2$, $T_{hotter} - T_{cooler}$ is the temperature difference across the material in °K, and L is the thickness of the material in meters.

While both conduction and convection require physical contact to transfer heat energy, by contrast, radiation does not require contact between the heat source and the object being heated. Thus, the heat transfer to an object at a specific temperature by radiation and convection occurs differently (Fig. 4).

## Summary

In the real world, it is important for us to keep in mind how heat energy is being transferred to the parts we are processing so that we can avoid issues such as overheating, underheating, distortion and surface/subsurface melting. Here are a few more important takeaways:

- Parts heated by radiation (a $T^4$ relationship) will receive heat energy far more rapidly than parts heated by convection (a $T^1$ relationship).
- Heat transfer by radiation, while (thermally) inefficient at temperatures under about 540°C (1000°F), becomes highly efficient as the temperature increases. This is one reason why radiant furnaces have a difficult time holding temperature uniformity at low temperatures.
- Heat transfer by convection is (thermally) efficient at low temperatures – up to approximately 650°C (1200°F) – but far less efficient as the temperature increases.
- Heat transfer by conduction is responsible for bringing heat to the inside of a part or extracting heat from the interior (core) of a part. Time is an

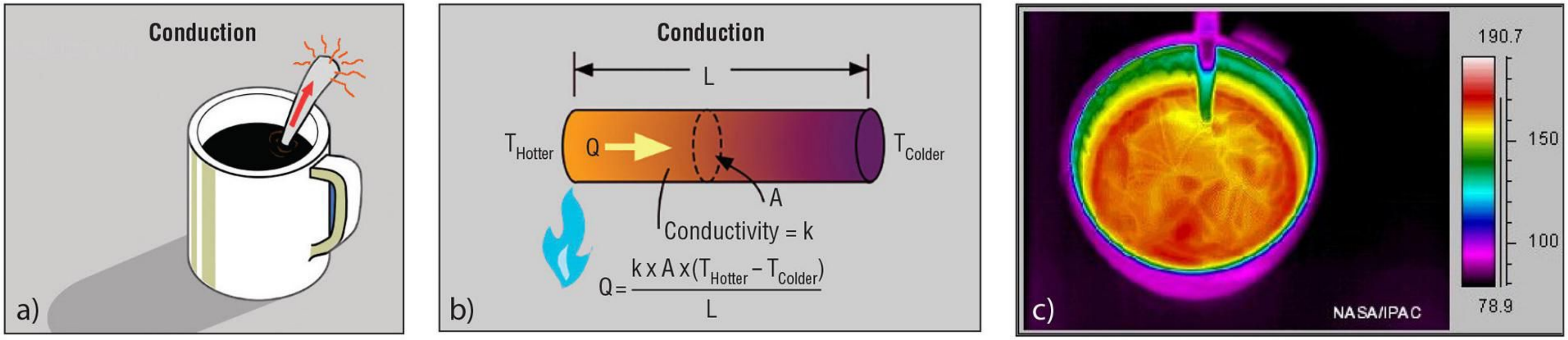

Fig. 3. Conduction example[1,2] (a) Spoon in hot coffee cup, (b) The science of conductive heat transfer, (c) A thermal infrared image of a coffee cup filled with a hot liquid. Notice the rings of color showing heat traveling from the hot liquid through the metal cup and the metal spoon.

important factor in heating or cooling to ensure the center of the part is at temperature.

- Only a small portion of the heat energy delivered to a furnace or oven is available to heat the load. The balance is lost due to a variety of reasons (e.g., flue losses, wall losses, opening losses, heat storage, etc.).

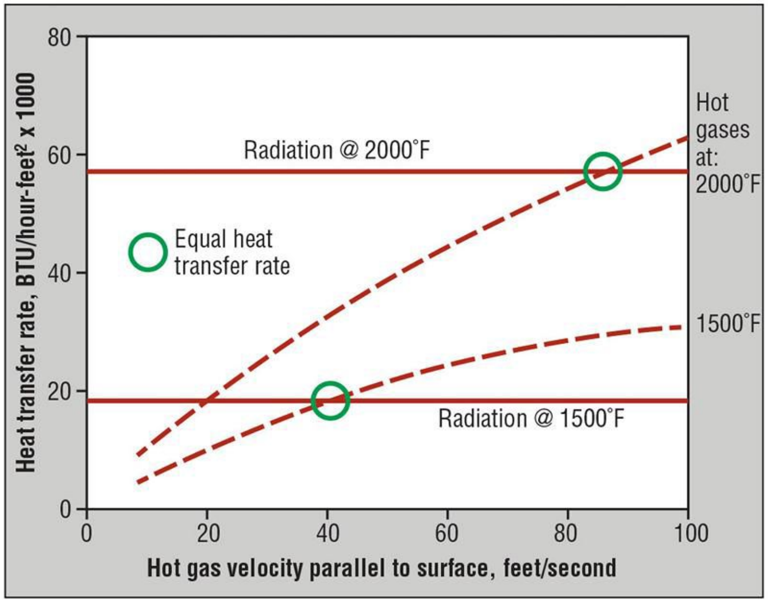

Fig. 4. Heat transfer to a 540°C (1000°F) object by radiation and (hot gas) convection[1]

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# Heating

Ever wonder what influences the heating (and cooling) of a component part or workload during heat treatment? Ever ask yourself why we care so very much about the influence of temperature and time, ramp rates and when to begin soaking a load? The time has come to answer these questions. Let's learn more.

Under ideal heating conditions, temperature is uniformly applied to the entire surface of each part within a workload; in the same manner and at the same time; under the same atmosphere conditions; and in equipment perfectly suited to the nature of the process. In the real world this never happens.

The manner in which heat is applied to the surface of a workpiece is highly dependent on loading. The design/method of operating the furnace plays a significant role as does the influence of process variables such as time and rate of heating (or cooling), which are as equally important as temperature.

One of our goals in heating is to reach the process temperature, which is determined by the material, the properties (physical, mechanical, metallurgical) we are trying to achieve, and the very nature of the process we are running. We must also make a distinction between the temperature of the chamber (or bath) in which the part or workload is placed and the temperature of the part itself, which is affected by the exposure time and rate of heat absorption. Another factor to take into account is the (required and actual) temperature uniformity of the chamber in which the heating occurs, which is why so much emphasis is placed on checking and rechecking this variable.

By comparison, time is a variable factor influenced by the method of heat transfer to and from the surface of the part. This includes such items as loading and exposure (i.e. the relationship of one part to another and to the heat source); the difference between the part temperature and the chamber temperature (which is why in some instances heat heads are not allowed); emissivity; heat-transfer methods (radiation, convection, conduction); the geometry/shape and mass of the part (which is why we care about the ruling or maximum part cross section, thin/thick sections and radical change of section); and a variety of other factors (Fig. 1). This is why we often hear the phrase "provided the part can absorb the heat energy at the rate delivered." All of the above factors affect the rate at which heating or cooling should take place, regardless of how fast heat could be transferred.

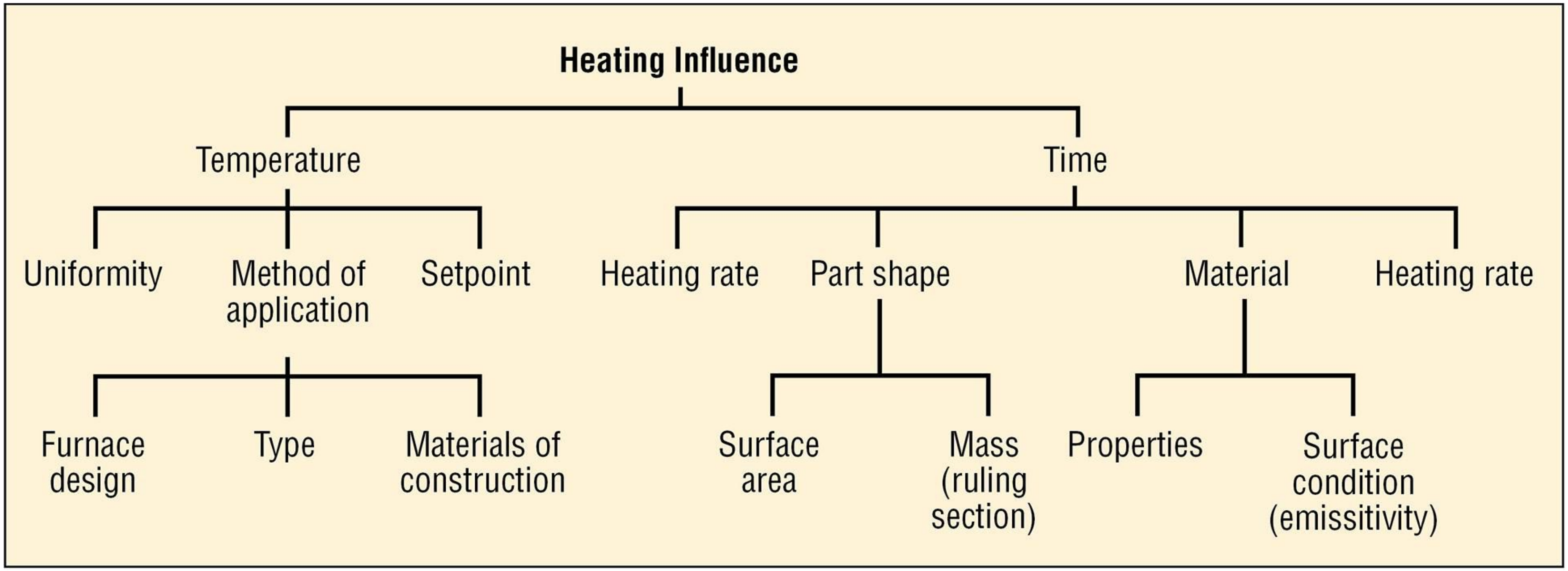

Fig. 1. Temperature-time factors[1]

## The Influence of Loading

Of all the variables affecting heating, loading is the one most often taken for granted. Non-uniformity of results, even from the same heat of steel, can be introduced if different parts of the workload are unequally exposed to heat (or cooling). Hence, it is not merely time and temperature but a time-temperature-mass-surface relationship that must be factored into the heating time.

Component parts come in all shapes and sizes. To meet this demand, standard and custom furnaces have been designed to accommodate the many workload configurations. Loading arrangements fall into two general classes: weight-limited and volume-limited. In either case, when loading parts in furnace baskets or onto racks, the goal is often to maximize loading efficiency. As heat treaters, however, we must also be concerned with proper part spacing (i.e. how parts are situated within the load for optimal heat transfer, atmosphere circulation, temperature uniformity and heat extraction during quenching so as to minimize dimensional variation).

How parts are loaded is very much a function of the

**Table 1. Critical temperature examples**[2]

| SAE No. | Approximate critical temp.on heating, °C (°F) | | Approximate critical temp. on cooling, °C (°F) | | Quench temp., °C (°F) | Martensite transformation, °C (°F) | |
|---|---|---|---|---|---|---|---|
| | $Ac_1$ | $Ac_3$ | $Ar_3$ | $Ar_1$ | | $M_s$ | $M_f$ |
| 1050 | 725 (1340) | 770 (1420) | 755 (1390) | 690 (1270) | - | - | - |
| 4140 | 750 (1380) | 795 (1460) | 745 (1370) | 695 (1280) | 815 (1500) | 340 (640) | 220 (425) |
| 52100 | 725 (1340) | 770 (1415) | 715 (1320) | 690 (1270) | 850 (1560) | 175 (345) | - |
| 8630 | 730 (1350) | 805 (1480) | 725 (1340) | 655 (1210) | 870 (1600) | 365 (690) | 280 (540) |

style of furnace being used. Final spacing is dictated by concerns for heating, soaking, atmosphere flow, the volume and type of quench media (brine, water, polymer, oil, salt, air), and gross load weight. A number of "rules of thumb" used in the industry can help, but determining the proper spacing around parts is critical and is often best done by trial and error. In general, the gap around a part should be no less than 25% of its envelope diameter and no greater than 75%. Again, one must factor in both process and equipment variables, including the type and construction of the quench tank, nature of the process and part mass to name a few. Despite the almost limitless choices, some common-sense rules apply.[6]

Other factors include: using batch or continuous

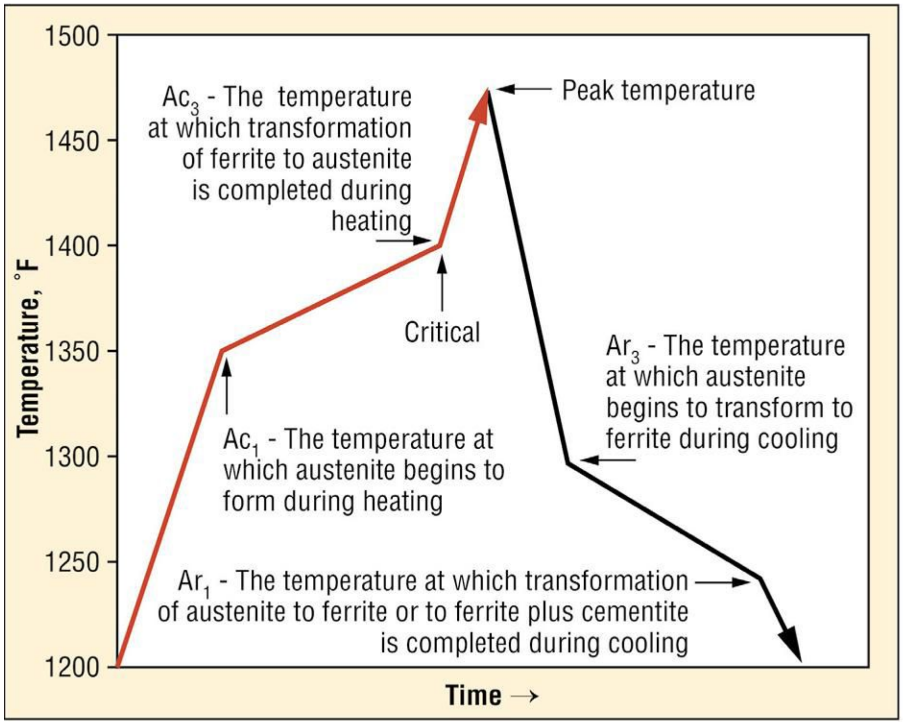

Fig. 2. Transformation temperatures for 01 tool steel[3]

processes, having skilled (i.e. trained) operators, avoiding human error, the manner in which heat is applied, control of process and equipment variables, and understanding/properly adjusting automatic controls (e.g., instrumentation, sensors, valves, etc.).

## Critical Temperatures

In heat treatment, we attempt to influence (dare I say control?) the microstructure of a material in part by the method of heating and/or cooling. This includes the microstructure, distribution of microconstituents, grain size, mechanical properties and the mechanisms by which they are brought about. The critical temperature is precisely that temperature where a phase change occurs in a metal during heating or cooling (i.e. the temperature at which we reach a critical point on the phase-transformation diagram for that material).

For example, on the iron-iron carbide phase diagram, critical temperatures (critical points) are denoted by the letter "A" (for "arrest") followed by either the letter "c" (for the French word "chauffage," meaning heating) or "r" (an abbreviation for the French word "refroidissement," meaning cooling). These signs, Ac or Ar, are followed by subscripts 1, 2 or 3, which indicate the particular point being referenced. Thus, Ac$_1$ refers to the temperature at which austenite begins to form during heating, while Ar$_1$ refers to the temperature at which transformation of austenite to ferrite (or to ferrite plus cementite) is complete during cooling. Similarly, Ac$_3$ refers to the temperature at which the transformation of ferrite to austenite is completed during heating, and Ar$_3$ refers to the temperature at which austenite begins to transform to ferrite during cooling.

Varying the rate of heating will have an effect on the rate of transformation and dissolution of microstructural constituents. In general, the temperature of transformation increases as the rate of heating increases.

In the case of O1 tool steel (Fig. 2), austenite begins to form on heating to approximately 730˚C (1350˚F). As we continue to heat, we see that the rise of temperature slows (as heat is absorbed during this transformation) until we reach approximately 760˚C (1400˚F), where the transformation to austenite is complete and the temperature rise resumes its original rate. After soaking for a predetermined time at the peak temperature, which is of our choosing, the steel is withdrawn from heat and begins cooling. Austenite begins transforming back into ferrite at about 700˚C (1295˚F), and the rate of cooling slows (as heat is being released during transformation). When the transformation is complete around 670˚C (1240˚F), the original cooling rate resumes.

## Some Effects of Alloying Elements[5]

In steel heat treatment, alloying elements produce a variety of mechanical properties and microstructures. One group of elements (e.g., Ni, Co, Mn, Pt, Pd, Rh and Ir) lowers the $Ac_3$ temperature and broadens the austenite-phase region. Another group lowers the $Ac_3$ temperature. In this group, elements such as N, C, Cu, Zn, Au and Re first broaden then cause the austenite-phase region to vanish. Other elements elevate the $Ac_3$ temperature, narrow and then close the austenite-phase region (e.g., Cr, Mo, W, Si, Ti, Al and Be). Elements such as Zr, Ta, Nb and Ce are responsible for producing other phases before the austenite field closes.

## Final Thoughts

To understand heating of a material, one must carefully consider all of the factors that can influence such a seemingly simple task. Hopefully, we now have a greater appreciation for this important step in the process.

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# Cooling Transformation Characteristics of Large Parts

Did you ever wonder if the test coupon run with the load is truly representative of the results obtained on the parts themselves? The answer we tell ourselves is yes, it is. However, at least a shadow of doubt creeps in as the part size becomes larger and the test coupon size remains the same. Conducting tests on actual parts, while often both time-consuming and expensive, is extremely rewarding. Let's learn more.

The cooling transformation characteristics of large SAE/AISI 4320 mill pinions were investigated by instrumenting the teeth of the pinion with thermocouples (Fig. 1) so that the quench rate and resultant metallurgical properties could be investigated and compared to that of the standard 2.5-inch (63-mm) test coupon run with every load. The pinion weighed approximately 18.5 tons (37,000 pounds) and was some 18 feet (5.5 meters) long. The largest diameter was 35 inches (890 mm) with a 4-inch (100-mm) gun-drilled hole through to the core.

The pinions are designed to meet AGMA 6014/AGMA 321 specification requirements. They are carburized in an atmosphere pit-style furnace for 80 hours at 1750-1775˚F (955-970˚C) in an endothermic gas atmosphere with natural gas additions holding a carburizing potential of 1.1% for the first 75% of the

Fig. 1. Test pinion with thermocouples in place

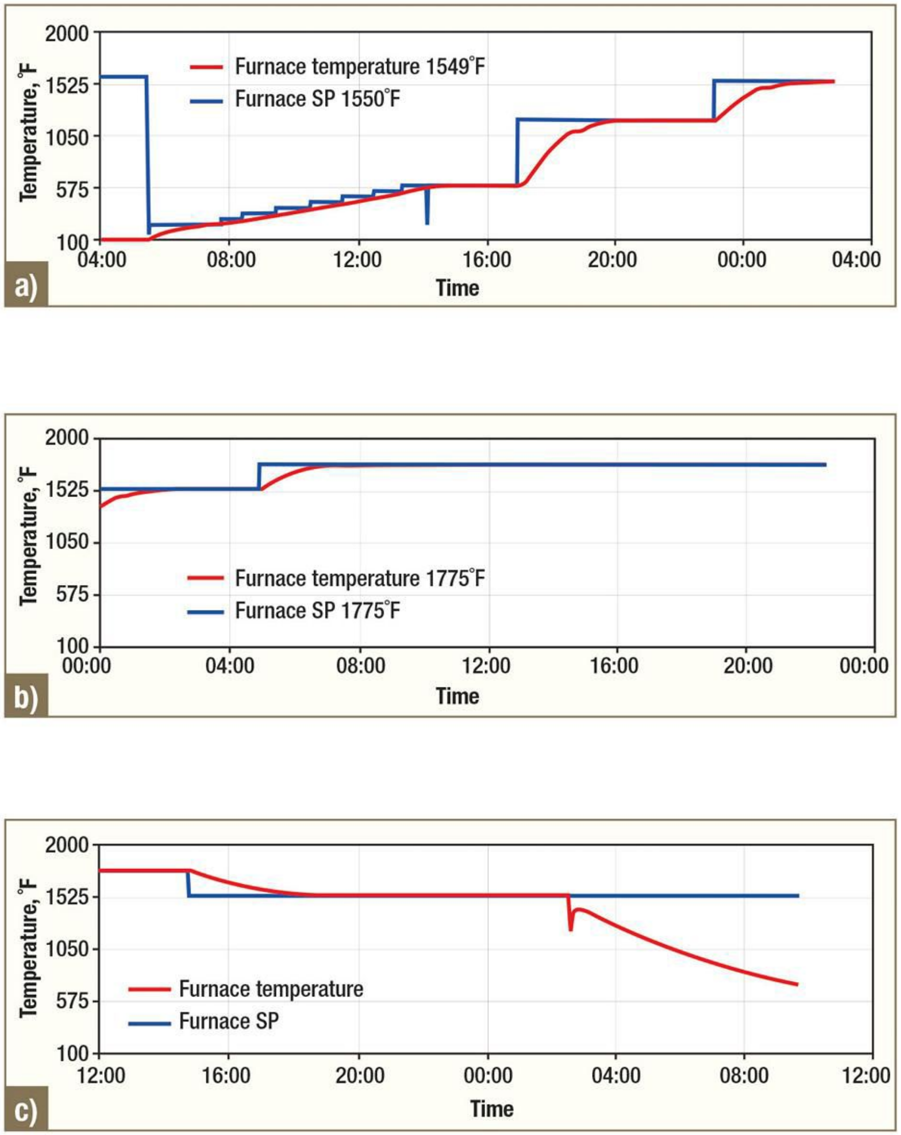

Fig. 2. Heating profiles: (a) preheat; (b) heat to carburizing temperature; (c) drop temperature for hardening

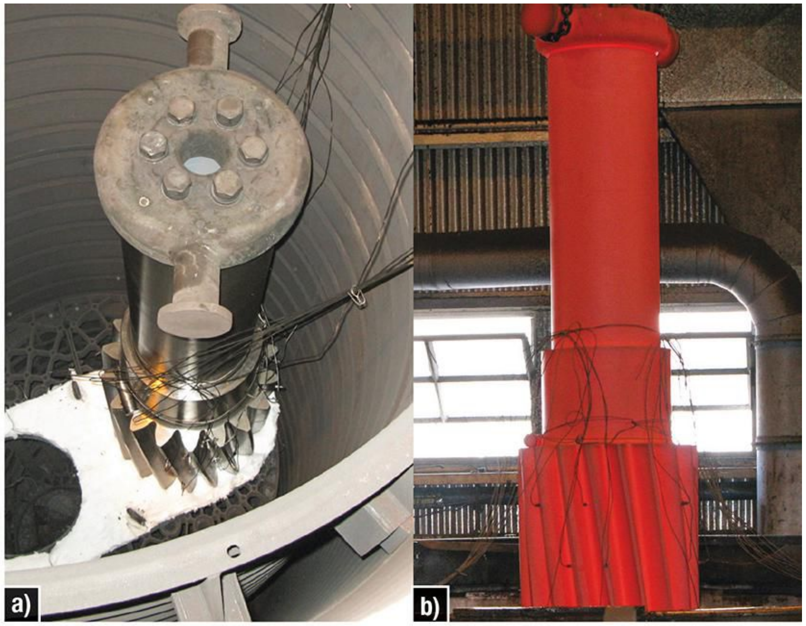

Fig. 3. Test part: (a) in position in furnace for heating test; (b) during transfer to the quench

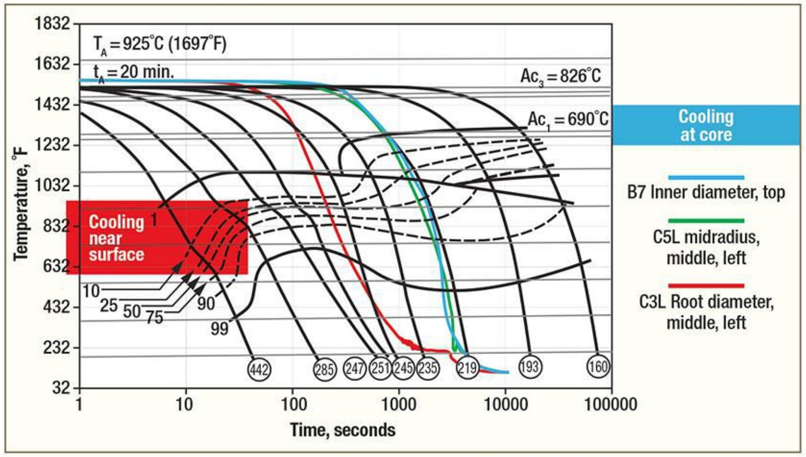

Fig. 4. Test data superimposed on continuous-cooling diagram for SAE 4320

cycle, followed by a 0.95% carbon potential for the balance of the cycle time. The targeted effective case depth is 0.190-0.240 inch (4.83-6.10 mm).

The part is then cooled in the furnace to 1550˚F (845˚C), stabilized and quenched into well-agitated polymer (UCON E), followed by tempering. The polymer concentration is held in the range of 19%. Resultant surface hardness (of the part) is in the range of 58-59 HRC, while the test coupon measures 62 HRC with a surface carbon of 0.80±0.20%.

In production, a load of two pinions is charged into a hot furnace, and the heating and drop temperature are performed in steps (Fig. 2). This same type of procedure was used for this test with the exception that the furnace was brought up from ambient temperature with a single pinion in place (Fig. 3).

## Summary

Test data of this type is rarely published, but it is invaluable. The test provided actual cooling transformation characteristics of the part (Fig. 4) as well as valuable data on the speed and uniformity of cooling (Table 1). The surface hardness (62 HRC) and microstructure of the test coupon were deemed acceptable as a quality-control measure once the test data was collected (i.e., microstructure reviewed and the tensile and yield strength, applied torsion stress and applied bending stress determined on the actual part). The materials and heat treatment were found to meet or exceed case and core strength requirements.

**Table 1. Selected cooling data (pinion teeth)**

| Elapsed time[a] (seconds) | Inner diameter B7 | Mid-radius C5L | Root diameter C3L |
|---|---|---|---|
| | °F (°C) | °F (°C) | °F (°C) |
| 0:00 | 1550 (843.3) | 1548 (842.2) | 1548 (842.2) |
| 1:30 | 1549 (842.8) | 1550 (843.3) | 1427 (842.8) |
| 2:00 | 1548 (842.2) | 1551 (843.9) | 1267 (686.1) |
| 2:30 | 1548 (842.2) | 1551 (843.9) | 1193 (645.0) |
| 3:00 | 1549 (842.8) | 1551 (843.9) | 1040 (560.0) |
| 5:00 | 1550 (843.3) | 1542 (838.9) | 736 (391.1) |
| 10:00 | 1535 (835.0) | 1452 (788.9) | 446 (230.0) |
| 18:00 | 1349 (731.7) | 1241 (671.7) | 277 (136.1) |
| 30:00 | 1077 (580.6) | 948 (508.9) | 233 (111.7) |
| 45:00 | 784 (417.8) | 654 (345.6) | 224 (106.7) |
| 60:00 | 312 (155.6) | 231 (110.6) | 165 (73.9) |

Notes: [a] After immersion into the quench tank.
[b] Data is typical for the 14 thermocouple positions.

# An Overview of Fundamental Material Properties

When we create products for the natural world, engineers and heat treaters tend to focus on making components that are stiff, strong and resistant to applied forces. Nature, on the other hand, chooses a different path, opting in general for objects that are highly flexible. To resolve the apparent conflict between the two requires an understanding of material science. Let's learn more.

## Materials Science

Materials science can be thought of as the science and technology of materials (Fig. 1). The central theory behind materials science involves relating the microstructure of a material to its macromolecular physical and chemical properties. By understanding and then changing the microstructure, materials scientists tailor the properties of a material to create custom or even brand-new materials with specific properties for specific uses.

## Stress and Strain

When we think about a solid object, we often assume it is perfectly rigid. While we know it is made up of atoms, we don't think of it as moving or stretching. All solid objects have a measureable stretch, however, especially if one thinks about elastic deformation, which is the ability of an object to return to its original form once a force has been applied to it.

Of critical importance is the force per unit area over which it acts, not the force applied to an object. We call this stress. If we wish to quantify how much an object will stretch, we divide the length that the object stretched by the original length and call this strain (aka fractional elongation).

## Stress–Strain Curves

Using a stress-strain curve (Fig. 2) for a given material, we can determine how much force we can exert on an object before it breaks (i.e., the maximum stress it can withstand). We call this the strength (aka ultimate tensile strength) of a material. On the other hand, if we are interested in knowing how far we can stretch a material, we talk about the maximum strain it can withstand. In the world of nature, we call this the extensibility of a

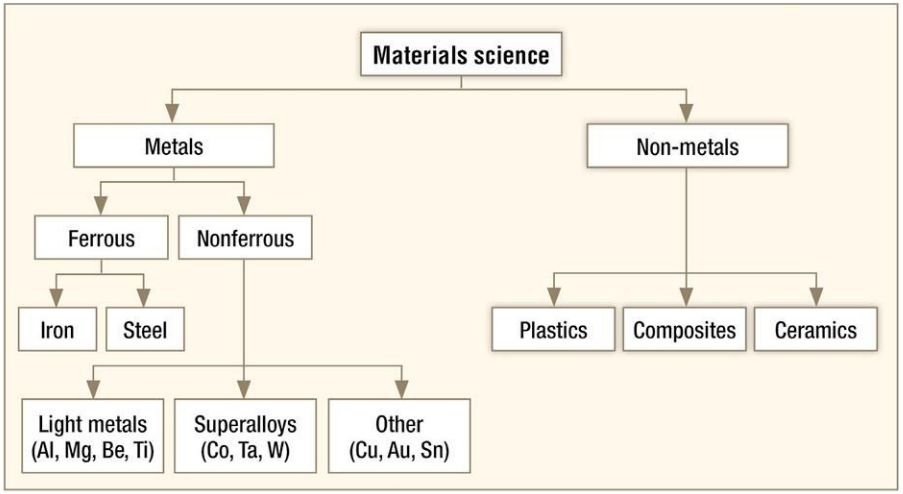

Fig. 1. Materials science

material.

When we exert force on a material and it returns back to its original shape and size, we say that the material is elastic or exhibits elastic deformation. In nature, we call this the extensibility of use. If the force we apply is too great, the material deforms permanently, and we say that it has exhibited plastic deformation. The yield strength (aka proof strength) is that point at which we have exceeded the elastic limit; the material has exceeded its ability to return to its original shape. Furthermore, when one talks about the elastic limit it is common to talk about the 0.2% offset yield strength – the amount of stress that will result in a plastic strain of 0.2%.

We are often interested in the ratio of yield strength to tensile strength in a material as an indication of the effectiveness of a given hardening operation for steels. In general, the greater the percentage of martensite achieved, the higher the ratio will be. Conversely, the greater the percentage of non-martensitic transformation products resulting from the hardening operation, the lower the ratio will be. Use of this ratio is a function of the steel type, the hardening and tempering recipes and the equipment selected to do the job. It is best considered in conjunction with empirical (historical) data and is a useful warning signal if the ratio is in variance with what would normally be expected. Hardness and microstructure checks should then be instituted to determine if some aspect of the desired metallurgy is not being achieved.

Finally, the “classic” appearance of stress-strain curves for steels is much different for other materials. For example, the tendons in our bodies have a stress-strain curve that is concave downward with a great deal of area underneath it, whereas blood vessel arterial ways are concave upward with a relatively small area underneath. We know that tendons (or sinew) are a tough band of fibrous connective tissue usually connecting muscle to bone and must be capable of withstanding a significant amount of applied tension. By contrast, arteries must be extremely flexible – being essentially elastic tubes with muscular walls that expand and contract to pump blood through the body.[2]

## Other Material Properties

In the world of engineering, we talk about Young's Modulus of Elasticity (i.e., the slope of the curve usually in the elastic region). A material with a high value is stiffer than a material with a low value. Since high-stiffness materials are subject to smaller elastic deformations for a given amount of load, it follows that a high value is desired for a material such as a beam that spans long distances. In nature, we often refer to Young's Modulus as the stiffness of a material (i.e., resistance to elastic deformation or the stress required to produce a given amount of elastic strain), or a measure of how much pull (stress) it takes to produce a given stretch (strain).

The energy per unit volume required to exert a force per unit area on a material is a measure of the material's toughness. Toughness is a fundamental material property measuring the ability of a material to absorb energy and withstand shock up to fracture (i.e., the ability to absorb energy in the plastic range). In other words, toughness is the amount of energy per unit volume that a material can absorb before rupturing and is represented by the area under the (tensile) stress-strain curve. In service, this loading often occurs in the form of (sudden) impact. In nature, toughness is often referred to as the work of extension.

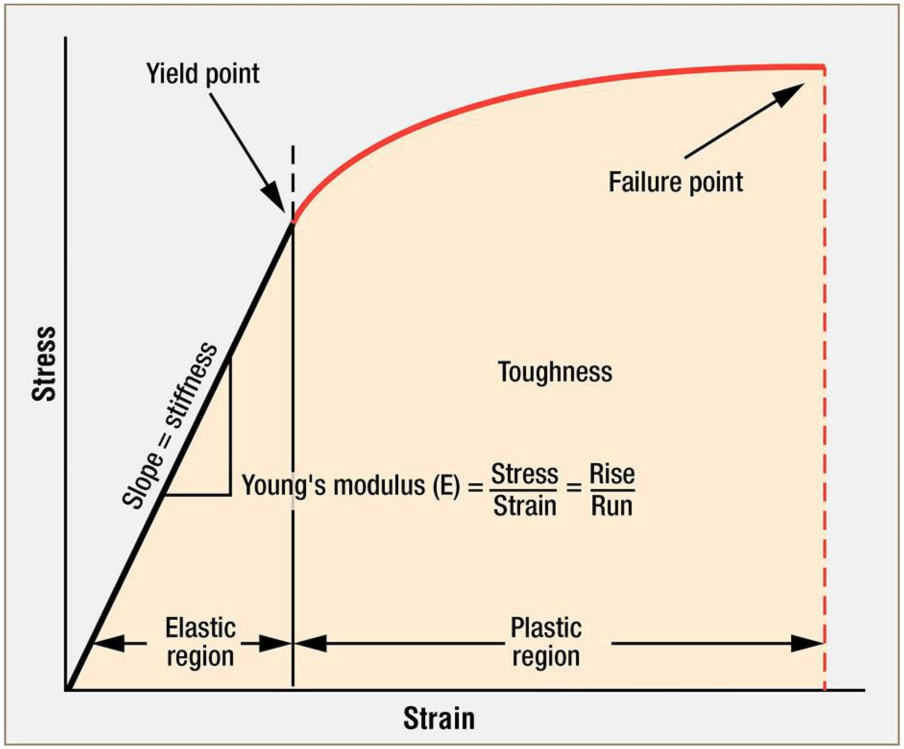

Fig. 2. Stress-strain curve[1] (adapted by the author)

Resilience is the ability of a material to absorb energy when it is deformed elastically and release that energy upon unloading. The modulus of resilience is defined as the maximum energy that can be absorbed per unit volume without creating a permanent distortion. A rubber band is a good example, where the work done as you stretch it comes back out of it when released. On the stress-strain curve this represents both the work performed during release divided by the work done in the stretch.[2]

Flexibility (bending without breaking) of a material comes to the fore in applications involving impact or resistance to external forces (e.g., wind or waves). It is important to remember that while strength is often of overriding concern, flexibility cannot be overlooked. However, flexibility can do more than improve toughness and impact resistance. It provides a means for a component part to adapt when loaded in a particular manner. Think about how the elements (wind, rain) cause a flag to tatter and tear, but a leaf better resists those same elements.

Other noteworthy mechanical properties of interest are hardness, brittleness, plasticity, malleability, ductility, (compressive and tensile) strength, durability and stability.

## Summary

Heat-treat engineers and heat treaters alike must take into consideration all of these fundamental material properties when designing a product and planning how to optimize its heat-treatment response. Common sense and nature itself play a role in helping us visualize these tasks.

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# Material Certification Sheets: What They Are and How They Help

When trying to determine a material's response to heat treatment, it is important to understand the form, prior treatments, chemical composition, grain size, hardenability and perhaps even the mechanical properties from which the parts were manufactured. The certification sheet for the material in question supplies such information. Sadly, they are seldom consulted until after a problem has occurred. It's time to learn what these documents are, why they are so useful and how to interpret them. Let's learn more.

## Form and Prior (Mill) Treatment

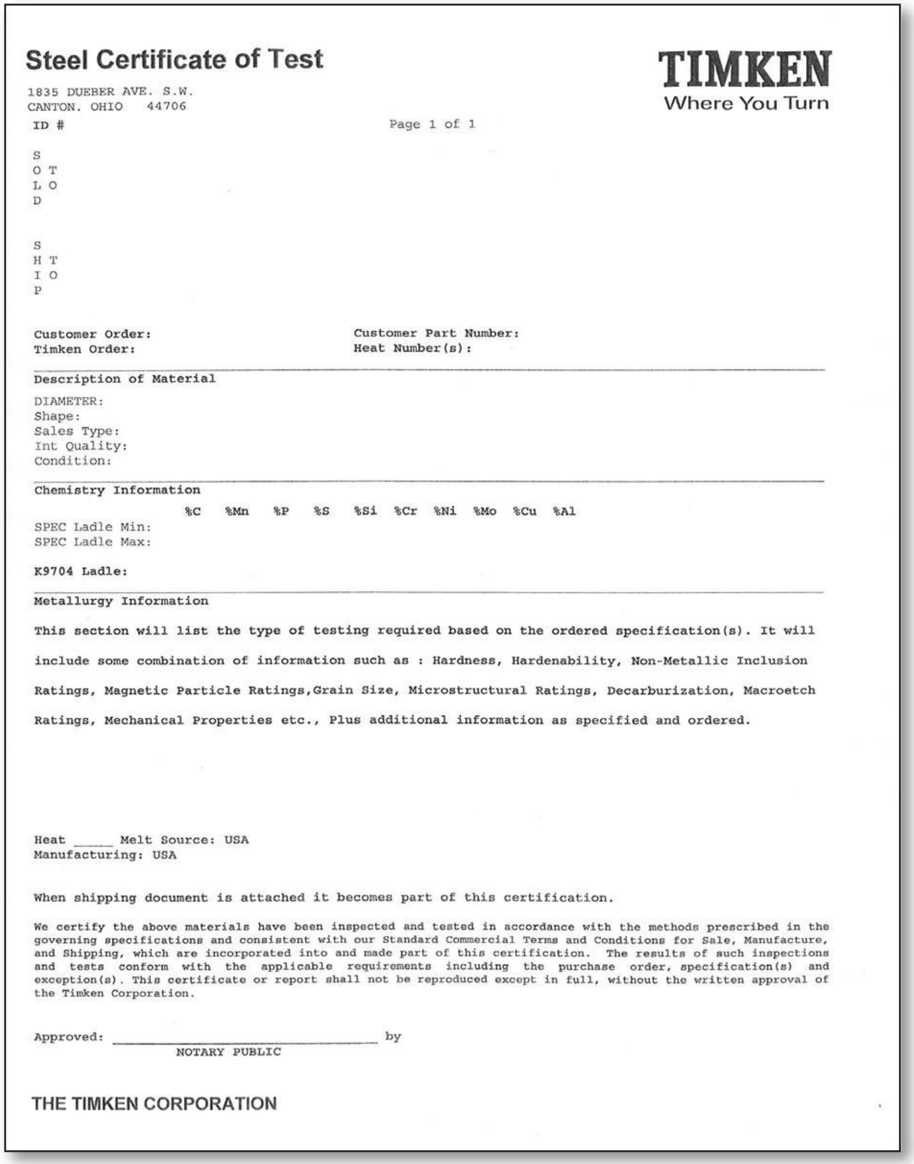
Knowing the form, size and origin of the raw material can help the metallurgist or heat treater anticipate how the material will behave during manufacturing and change during heat treatment. For example, the material may be hot or cold rolled and be supplied from bar stock, tubing, wire, strip or plate. The material may be wrought, cast, forged or made from powder-metallurgy methods. The material certification sheets also tell you the source and prior mill treatment. A forging may have been normalized at the mill or may need this type of treatment before manufacturing component parts. Bar stock may be annealed to a lamellar or spheroidized structure for machining or wire partially annealed after being drawn.

## Steelmaking Process and Applicable Standards

There can be subtle but meaningful differences in products made via Basic Oxygen Furnaces (BOF), primarily hot-metal-based steelmaking, and those made via Electric Arc Furnaces (EAF), primarily scrap-based practices. The material may be aluminum killed (fine grain) or silicon killed (coarse grain) and/or treated by various other elemental additions (e.g., calcium, tellurium). ASTM, AMS, AISI, SAE or other similar U.S. or international standards may be called out and should be consulted prior to heat treatment.

## Chemistry

Different chemical elements influence the response of a material to heat treatment (Table 1). In general, the greater the amount (e.g., weight percentage) of the alloying element(s), the more pronounced the effect would be. For example, greater strength is achieved by adding carbon (C), manganese (Mn) or nickel (Ni). Corrosion resistance can be enhanced by



**Steel Certificate of Test**

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CANTON. OHIO 44706
ID #

Page 1 of 1

S
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**Customer Order:**
**Timken Order:**

**Customer Part Number:**
**Heat Number(s):**

**Description of Material**

DIAMETER:
Shape:
Sales Type:
Int Quality:
Condition:

**Chemistry Information**

| | %C | %Mn | %P | %S | %Si | %Cr | %Ni | %Mo | %Cu | %Al |
|---|---|---|---|---|---|---|---|---|---|---|
| SPEC Ladle Min: | | | | | | | | | | |
| SPEC Ladle Max: | | | | | | | | | | |
| **K9704 Ladle:** | | | | | | | | | | |

**Metallurgy Information**

This section will list the type of testing required based on the ordered specification(s). It will include some combination of information such as : Hardness, Hardenability, Non-Metallic Inclusion Ratings, Magnetic Particle Ratings,Grain Size, Microstructural Ratings, Decarburization, Macroetch Ratings, Mechanical Properties etc., Plus additional information as specified and ordered.

Heat _____ Melt Source: USA
Manufacturing: USA

When shipping document is attached it becomes part of this certification.

We certify the above materials have been inspected and tested in accordance with the methods prescribed in the governing specifications and consistent with our Standard Commercial Terms and Conditions for Sale, Manufacture, and Shipping, which are incorporated into and made part of this certification. The results of such inspections and tests conform with the applicable requirements including the purchase order, specification(s) and exception(s). This certificate or report shall not be reproduced except in full, without the written approval of the Timken Corporation.

Approved: ______________________________ by
NOTARY PUBLIC

THE TIMKEN CORPORATION

Fig. 1. A typical material certification sheet[1]

adding chromium (Cr) or copper (Cu). Machinability is improved by adding lead (Pb), sulfur (S) or selenium (Se). High-temperature properties are retained by adding tungsten (W) or molybdenum (Mo). Most of the alloying elements, either singularly or in combination, service more than one of these purposes.

## Grain Size

Grain size can have a significant effect on heat treatment. Steels with ASTM grain size 1-4 are considered coarse grain while 5-8 are considered fine grain (Table 2). Large (coarse) grain size is generally associated with greater hardenability but lower hardness (strength) and ductility. In heat-treated steels, the grain size after heat treatment (typically but not always martensite) is not readily measured. Instead, we measure the size of the prior austenite grains since it can be correlated to the properties of the heat-treated steels. Special etching procedures may well be needed to reveal these prior grain boundaries.

Many of the important mechanical properties of steel, including yield strength and hardness, the ductile-brittle transition temperature and susceptibility to environmental embrittlement, can be improved by refining the grain size. The improvement can often be quantified using the Hall-Petch relationship. The quantitative improvement in properties varies with $d^{-1/2}$, where d is the grain size. There are special techniques to further reduce the grain size. The most common is the use of multiple quenches. This involves repeating the austenizing and quenching process several times.

## Hardness and Hardenability

Material certification sheets usually provide a report on the hardness and hardenability of the material after mill processing and, when specified, supply information on the hardenability of the material (by providing Jominy and/or Ideal Diameter (DI) values) and in some cases on Carbon Equivalence (CE). Hardness is a measure of how hard or strong the material is, while hardenability may be thought of as the property that determines the depth and distribution of hardness when steel is austenitized and quenched.

## Cleanliness

Steel cleanliness is one measure of steel quality. The content of elements such as phosphorus, sulfur, total oxygen, nitrogen and hydrogen are usually, but not always, minimized. Likewise, the amount, morphology and size distribution of various species of nonmetallic inclusions should generally be minimized (Table 3). It is well known that the individual or combined effect of carbon, phosphorus, sulfur,

**Table 1.** Common alloying elements and their effects on steel[1, 2]

| Element | Effect on steel | General remarks |
|---|---|---|
| Aluminum | Deoxidation | Added for grain-size control (grain refinement). A strong nitride former. |
| Boron | Hardenability | Significantly enhances core hardenability; caution must be used with respect to case hardenability in carburized parts. |
| Carbon | Hardness, strength, wear | Has the greatest influence on solid-solution strength and hardenability. Forms a carbide or carbonitride with Fe and also with Cr, Mo, V, W and Cb. |
| Chromium | Corrosion resistance, hardenability | Improves high-temperature oxidation. A carbide former (better than manganese) that improves abrasion resistance. |
| Cobalt | Hardness, wear | Improves hot hardness. Weak carbide former. Decreases hardenability. |
| Columbium (Niobium) | Formation of carbonitrides in microalloyed steels; elimination of chromium-carbide precipitation in stainless steels | Excellent microalloying agent. Controls grain-size (grain refinement). Strong carbide and nitride former. |
| Copper | Corrosion resistance, strength | Has dual effects (can be both beneficial or detrimental) and must be carefully controlled during steelmaking. Enhances atmospheric corrosion resistance and imparts in precipitation-hardening stainless steels. Promotes hot shortness and weldability. |
| Lead | Machinability | Negative environmental effects must be taken into consideration prior to usage. |
| Manganese | Strength, hardenability, improved response to heat treatment | Increases solid-solution strength. Counteracts brittleness caused by sulfur by forming manganese sulfide (MnS). |
| Molybdenum | High-temperature strength, hardenability | Increases solid-solution strength. Strong carbide former (stronger than chromium). Improves high-temperature properties such as creep strength. Counteracts temper embrittlement. |
| Nickel | Toughness, strength | Toughens steels, especially at low temperature. Increases solid solution strength and hardenability. |
| Nitrogen | Strength | Increases the number of nitrides (in microalloyed steels) and controls grain size. |
| Phosphorous | Strength | Promotes temper embrittlement. Increases strength in low-carbon steels. |
| Silicon | Deoxidizer (for coarse-grain steels), hardenability, temper resistance | Ferrite strengthener. Improves hardenability. Promotes grain-boundary oxidation in carburized steels. |
| Sulfur | Machinability | Impurity element in most steels. Added to enhance machining. |
| Tellurium | Machinability | Significant toxicity issues during steelmaking. |
| Titanium | Microalloying | Significant microalloying addition. Very strong carbide and nitride former. Ties up nitrogen in steels (especially important in boron steels). |
| Tungsten | Hardness | Very hard carbides. Strong wear resistance. Improves abrasive resistance. Increases solid-solution strength and hardenability. |
| Vanadium | Fine grain, toughness, impact resistance | Significant microalloying addition. Grain-size control (grain refinement). Carbide and strong nitride former. Minimizes loss of strength during tempering. |

**Table 2. Austenite grains in steel (McQuaid Ehn)[3]**

| ASTM grain size | Number of grains per $in^2$ at 100X | Diameter (in microns) |
|---|---|---|
| 1 | ≤ 1.5 | 254 |
| 2 | 1.5-3 | 180 |
| 3 | 3-6 | 127 |
| 4 | 6-12 | 89.8 |
| 5 | 12-24 | 63.5 |
| 6 | 24-48 | 44.9 |
| 7 | 48-96 | 31.8 |
| 8 | ≤ 96 | 22.5 |

nitrogen, hydrogen and total oxygen in steel can have a remarkable influence on steel properties, such as tensile strength, formability, toughness, weldability, cracking resistance, corrosion resistance and fatigue resistance.

## Mechanical Properties

When requested or specified, the common mechanical properties shown on material certification sheets include some combination of strength, ductility and/or toughness. Examples include tensile and yield strength, percent elongation, reduction in area and Charpy values. If the customer requests special testing, these results are reported as well. In other instances, the customer may request specific mechanical testing, which will also be reported on the material certification sheet. The heat treater should pay particular attention to all mechanical-property data when designing his recipes and choosing his equipment and processing parameters.

**Table 3. Influence of typical impurities on mechanical properties[4]**

| Element | Form | Mechanical properties influenced |
|---|---|---|
| Sulfur, oxygen | Sulfide and oxide inclusions | • Ductility, Charpy impact value, anisotropy<br>• Formability (elongation, reduction of area and bendability)<br>• Cold forgeability, drawability<br>• Low-temperature toughness<br>• Fatigue strength |
| Carbon, nitrogen | Solid solution | Solid solubility (enhanced), hardenability |
| | Settled dislocation | Strain aging (enhanced), ductility and toughness (lowered) |
| | Pearlite and cementite | Dispersion (enhanced), ductility and toughness (lowered) |
| | Carbide and nitride precipitates | • Precipitation, grain refining (enhanced), toughness (enhanced)<br>• Embrittlement by intergranular precipitation |
| Phosphorous | Solid solution | • Solid solubility (enhanced), hardenability (enhanced) |
| | | • Temper brittleness |
| | | • Separation, secondary work embrittlement |

## In Conclusion

Material certification sheets are an invaluable tool for metallurgists and heat treaters and should be consulted before any heat treatment is performed on every load of parts. In this way, our recipes and cycles can be optimized to take into account the particular circumstances surrounding how the steel was specified and produced.

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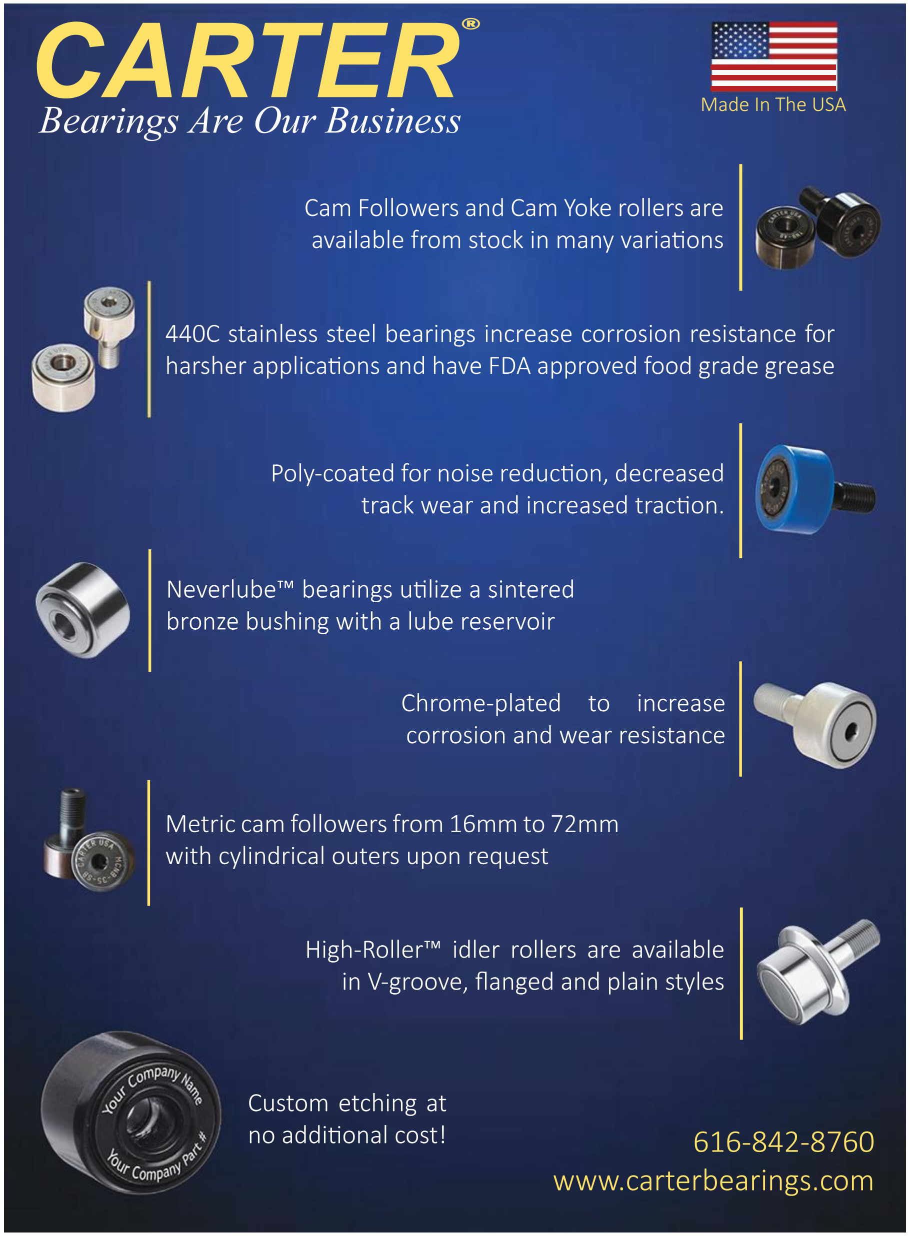

# The Influence of Manganese in Steel

They say that everyone and everything gets 15 minutes of fame somewhere, sometime, somehow. Well, for manganese, its time is now. The effect of a particular alloying element on both the steelmaking process and the steel's response to heat treatment depends on the individual element and on its (complex) interactions with other elements, either individually or collectively. Manganese is considered, next to carbon, the most important elemental addition to steel. Let's learn more.

Strictly from a heat treater's perspective, the purpose of adding alloying elements to steel is to enhance the material's response to heat treatment, which in turn results in improvement of the mechanical and physical properties of the steel. Alloying additions can be made for one or more of the following reasons:

- To increase hardenability
- To help reduce part distortion
- To produce a finer grain size
- To improve tensile strength without appreciably lowering ductility
- To avoid quench cracking
- To gain toughness
- To achieve better wear resistance
- To improve hot hardness
- To achieve better corrosion resistance

Beside the role manganese plays in deoxidation of steel and modification of sulfides present, it is a major alloying element, has complex interactions with carbon and is used to control inclusions. Manganese is beneficial to surface quality in all carbon ranges with the exception of rimmed steels (<0.15%C) and is particularly beneficial in high-sulfur steels. Manganese contributes to strength and hardness but to a lesser degree than carbon. The increase depends on the carbon content – higher-carbon steels being affected more by manganese. Higher-manganese steels decrease ductility and weldability (but to a lesser extent than carbon). Manganese also increases the rate of carbon penetration during carburizing.
The effects of manganese can be summarized as:[2]

1. Lowers the temperature at which austenite

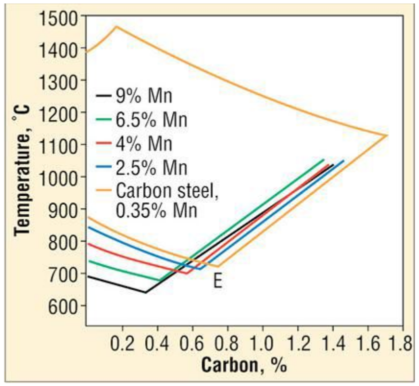

Fig. 1. Effect of manganese on the shape of the austenite field[2]

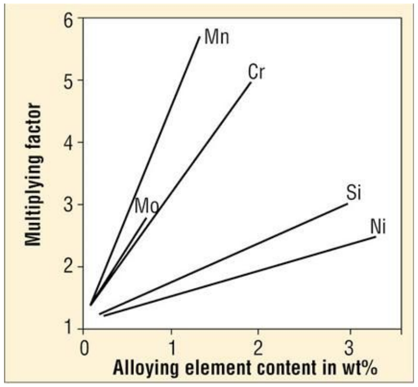

Fig. 2. Effect of alloying elements on hardenability – Grossmann multiplying factors[2]

begins to decompose
2. Extends the metastable austenitic region and delays the commencement of all the austenite decomposition reactions
3. Favors the formation of lower bainite and suppresses the upper bainite reaction on isothermal transformation
4. Is the most effective alloying addition for lowering the martensite-start ($M_S$) temperature
5. Favors the formation of ε-martensite
6. Has little effect on the strength of martensite and on the volume change from austenite to martensite
7. Has little or no solution-hardening effect in austenite and between 30–40 $MN/m^2$ per wt. % in ferrite (by lowering the stacking-fault energy of austenite, manganese increases the work-hardening rate)
8. By lowering the $M_S$ temperature, manganese prevents the deleterious effects of autotempering
9. Lowers the transformation temperature, causing substantial grain refinement
10. In general, lowers the tough-to-brittle impact transition temperature (due to its grain-refinement action)
11. Increases the propensity for weld cracking due to the effect on hardenability. The severity of its influence depends to a great extent on the type of steel and the welding techniques.
12. Does not increase the susceptibility of the steel to delayed fracture due to hydrogen absorption
13. Improves the fatigue limit
14. Reduces the number of cycles to failure under high strain conditions
15. Forms five carbides ($Mn_{23}C_6$, $Mn_{15}C_4$, $Mn_3C$, $Mn_5C_2$ and $Mn_7C_3$), the dominant one being $Mn_3C$, which forms a continuous range of solid solutions with $Fe_3C$, thus reducing the solubility of carbon in α-iron
16. Prevents the formation of an embrittling grain-boundary cementite
17. Suppresses the yield extension in deep-drawing

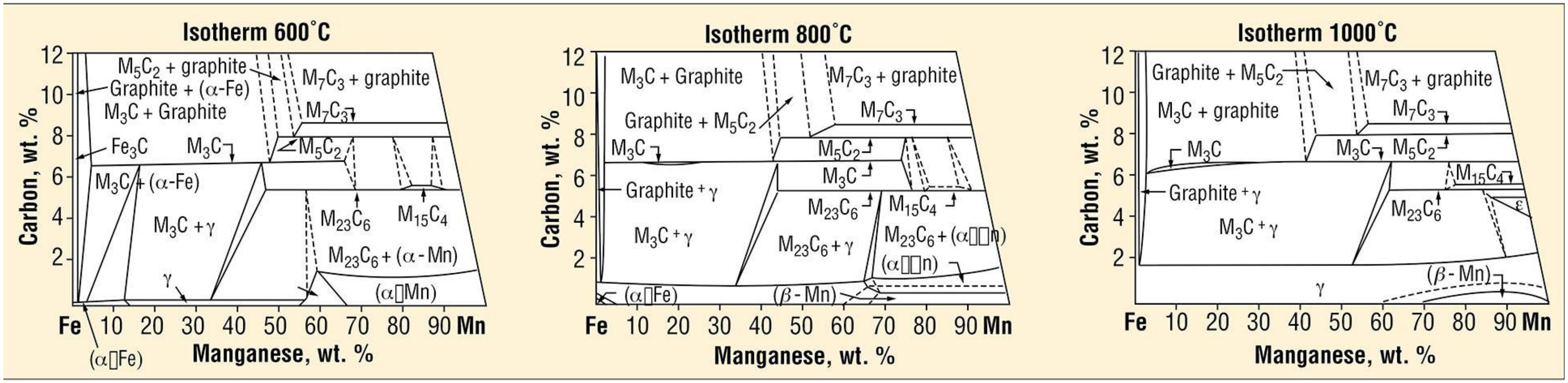

Fig. 1. Isothermal sections from the iron-manganese-carbon ternary diagram 600˚C (1110˚F), 800˚C (1475˚F) and 1000˚C (1825°F)

steels by virtue of its grain-refinement effect
18. Suppresses strain aging
19. In combination with nitrogen, has a solid-solution hardening effect and improves high-temperature properties
20. Extends the range of use of low-carbon steels
21. Has a strong influence on the pearlite morphology of high-carbon steels
22. Extends the range of use of high-carbon steels through its grain-refining and pearlite-refining actions
23. Raises strength values in bainitic steels by reducing grain size and increasing dispersion hardening
24. Allows bainitic steels to be produced by air hardening
25. Increases hardenability
26. Slows down the temper reactions in martensite
27. Assists interphase precipitation
28. Improves austemper and martemper properties
29. Increases temper embrittlement unless the carbon content is very low and trace element impurities are minimal
30. In spring steels, promotes ductility and fracture toughness without undue loss in tensile strength
31. Removes the risk of hot shortness and hot cracking when the ratio of manganese to sulfur is greater than 20:1 by forming a higher melting-point eutectic with sulfur than iron sulphide
32. Has a major influence on the anisotropy of toughness in wrought steels due to the ability to deform manganese sulfides during hot working

33. Forms three manganese sulfide morphologies (Type I, II and III) dependent upon the state of oxidation of the steel
34. Enhances free-cutting steels
35. Increases the stability of austenite
36. Has similar atomic size as iron (Mn = 3.58Å, Fe = 3.44 Å)
37. Lowers the stacking-fault energy of austenite (in contrast to alloying element additions such as chromium or nickel)
38. Allows lower solution temperatures for precipitation-hardening treatments in highly alloyed austenite due to increased carbon solubility
39. Forms intermetallic compounds suitable for precipitation-hardened austenitic steels
40. Plays a major role in controlling the precipitation process that occurs during isothermal transformation to austenite
41. Increases the rate of carbon penetration during carburizing
42. Contributes, in combination with nitrogen, to the performance of work-hardenable austenitic stainless steels
43. Improves hot corrosion resistance in sulfurous atmospheres
44. Enhances wear-resistance in carbon-containing austenitic steels where the manganese content is between 12-14%
45. Improves response of low-alloy steels to thermomechanical treatments
46. Strengthens certain steels by maraging by producing an austenitic structure using manganese-containing compounds
47. Enhances the performance of TRIP steels
48. Promotes ferro-elastic behavior in appropriate steels
49. Less tendency to segregate within the ingot
50. In general, improves surface quality.

## Summing Up

Manganese is the type of addition that is easily overlooked but can play a significant role in helping steels achieve their mechanical and metallurgical properties (e.g., hardness when austempering of medium-to-high-carbon steels).

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# Tramp Elements and Their Influence on Steel and Steel Heat Treatment

As heat treaters, we must be aware of what tramp elements are and how they can affect our heat-treatment operations. While certification sheets and chemical analyses performed on incoming raw material do a fine job of identifying the major chemical constituents and may indicate conformance to a particular steel standard, it is those pesky tramp elements that cause The Doctor sleepless nights. Let's learn more.

Today, steel is a globally sourced commodity, and scrap-based steel production in electric arc furnaces is a popular steelmaking practice. The use of scrap has and will continue to grow, which means the presence of possibly more tramp elements in the future.

In simplest terms, any element that is present in the steel's composition but not a deliberate addition is considered a tramp element. They cannot be removed by simple metallurgical processes. Tramp elements (e.g., Cu, Ni, Sn, As, Cr, Mo, Pb and others) are highly dependent on the steel grade and enter the steel through four main sources,[1] namely:

- Iron ore or pig iron
- Impurities in ferroalloy additions
- Refractories
- Scrap steel

An important distinction must also be made between tramp elements that have an effect due to their presence in solid solution (e.g., Mo, Cr, Ni, Cu) and those that have an effect due to their segregation at interfaces such as surfaces and grain boundaries (e.g., Sn, As, Sb). In addition, elements with an atom size smaller than the solvent atoms (e.g., C, N, B) may also segregate at grain boundaries, competing with tramp elements and often protecting the interfaces from possible detrimental effects due to residual enrichment.[1]

**Table 1. Tramp-element effects on mill processing conditions[1]**

| Condition or property affected | Tramp element(s) responsible |
|---|---|
| Hot shortness[a] | Cu, Ni, As, Sn |
| Grain-boundary embrittlement[b] | Sb, Sn, As |
| Segregation | Sn |
| Recrystallization annealing | Mo, Cr, Sn |
| Hot ductility | Zn, Sn |
| Hardenability[c] | All |
| Weldability[d] | Mo, Cr, Cu, Ni |

Notes: a) Affects surface condition of hot-rolled and pickled strip • b) During batch or continuous annealing of low-carbon steels • c) Austenite to ferrite transformation • d) High-strength steel grades

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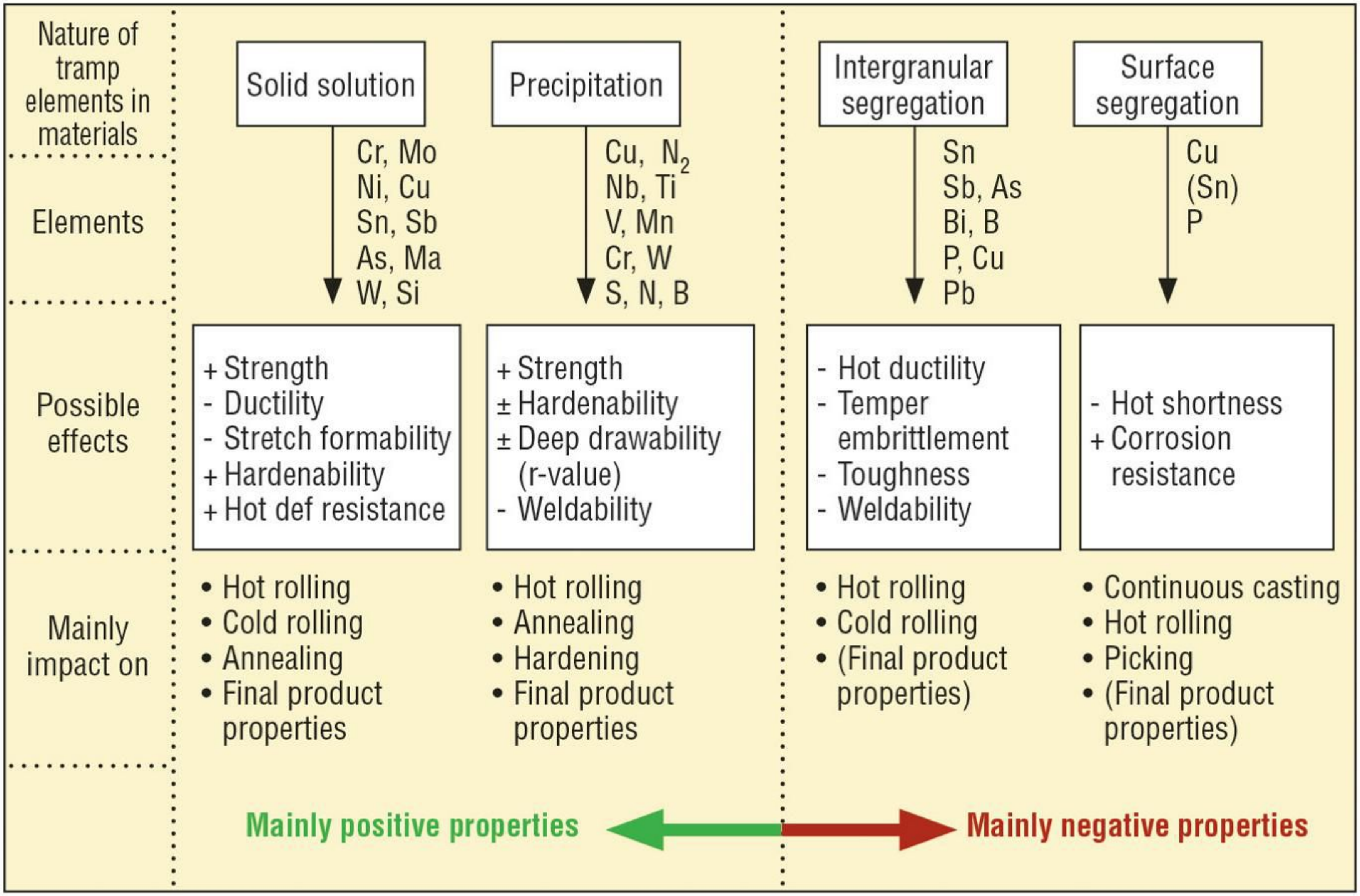

| Nature of tramp elements in materials | Solid solution | Precipitation | Intergranular segregation | Surface segregation |
|---|---|---|---|---|
| Elements | Cr, Mo<br>Ni, Cu<br>Sn, Sb<br>As, Ma<br>W, Si | Cu, $N_2$<br>Nb, Ti<br>V, Mn<br>Cr, W<br>S, N, B | Sn<br>Sb, As<br>Bi, B<br>P, Cu<br>Pb | Cu<br>(Sn)<br>P |
| Possible effects | + Strength<br>- Ductility<br>- Stretch formability<br>+ Hardenability<br>+ Hot def resistance | + Strength<br>± Hardenability<br>± Deep drawability (r-value)<br>- Weldability | - Hot ductility<br>- Temper embrittlement<br>- Toughness<br>- Weldability | - Hot shortness<br>+ Corrosion resistance |
| Mainly impact on | • Hot rolling<br>• Cold rolling<br>• Annealing<br>• Final product properties | • Hot rolling<br>• Annealing<br>• Hardening<br>• Final product properties | • Hot rolling<br>• Cold rolling<br>• (Final product properties) | • Continuous casting<br>• Hot rolling<br>• Picking<br>• (Final product properties) |

Fig. 1. Overview of the nature of trace-element effects on downstream processes and final properties[3]

**Table 2. Effects of increases in residual elements on various properties of steels[2]**

| Property | Cu | Ni | Cr | Mo | Sn | Sb |
|---|---|---|---|---|---|---|
| Hardenability | + | + | + | + | +, 0 | +, 0 |
| Strength/ hardness | + | + | + / - | + | + | + |
| Ductility | + | + / - | + / - | - | - |  |
| Impact resistance | + | + | 0 | - | 0, - |  |
| Temper embrittlement |  |  |  |  | + | + |

## Annealing and Mill Effects[1,2]

In general, tramp elements contribute to an increase in strength with a resultant loss of ductility. Thus, casting, forming, drawing and annealing operations performed at the mill can be affected (Table 1). For example, molybdenum and chromium present in extra-low-carbon steels increase resistance to hot deformation, requiring higher rolling loads. The presence of tin and arsenic will adversely affect recrystallization kinetics during continuous annealing of certain cold-rolled steel grades and require an increase in annealing temperature.

Copper, one of the most recognizable tramp elements, is responsible for surface defects related to scaling and cracking (hot shortness). It is a good indicator of the amount of scrap used in the steelmaking process, often present at levels of 0.20% or greater. Nickel, if present in about the same percentage, offsets the Cu effect, but Sn and As amplify it. The presence of even minute amounts of tin (0.05% added to a steel containing 0.22% copper) increases the tendency toward cracking.

## Quench and Temper Effects[2]

Downstream processing and final properties are also affected by tramp elements (Fig. 1, Table 2). Grain-boundary embrittlement (Table 3), which can occur in even low-alloy structural steels, is one such property and is a function of heat treatment and composition, with major alloying elements and residual (tramp) elements playing an important role. Most embrittlement occurs in steels in the quenched-and-tempered condition with a martensitic structure. This susceptibility is reduced somewhat in bainitic, pearlitic or ferritic structures.

Grain-boundary embrittlement can manifest itself as temper embrittlement, stress-corrosion cracking, hydrogen embrittlement, creep rupture, stress-relief cracking and fatigue-crack growth. Very small total amounts (<200 ppm) of residuals can cause embrittlement, but concentrations are often a function of heat-treatment time and temperature. The types of embrittlement elements responsible (Table 3) can be categorized as:

- Class 1: Elements that are the primary cause of embrittlement
- Class 2: Elements that enhance embrittlement
- Class 3: Elements that improve grain-boundary cohesion
- Class 4: Elements that inhibit grain-boundary em brittlement

For example, Class-2 elements are necessary for temper embrittlement to occur. Recall that temper embrittlement (TE) is a result of cooling slowly through the temperature range of 350-575°C (660-1070°F) or holding in the range for relatively long times. Certain elements (e.g., Mo, La, Ce) have been found to reduce temper-embrittlement effects but may affect other properties, such as fatigue (especially the rare-earth elements).

**Table 3. Grain-boundary embrittlement elements (by class)[2]**

| Class 1 | Class 2 | Class 3 | Class 4 |
|---|---|---|---|
| Bismuth<br>Tin<br>Antimony<br>Selenium<br>Sulfur<br>Tellurium<br>Arsenic<br>Germanium<br>Phosphorous<br>Silicon | Nickel[a]<br>Chromium[a]<br>Manganese[a] | Carbon<br>Nitrogen<br>Boron[b]<br>Tungsten<br>Beryllium | Molybdenum<br>Zirconium<br>Vanadium<br>Titanium<br>Tungsten<br>Lanthanum<br>Cerium |

Notes: a). At concentrations >1%, these elements raise the grain-boundary segregation ratio to a level high enough for temper embrittlement to occur due to the presence of elements such as Sn, Sb and P. • b).Boron, while improving grain-boundary cohesion, may cause other problems such as increased hardenability in heat-affected zones (causing concerns over cold cracking).

Finally, segregation (micro or macro) can increase the level of both alloying elements and trace elements (especially P, S, Sn and As) and has been found to adversely affect certain properties. Toughness and the heat-affected zone in weldments are typical examples. Casting practices are extremely important to help negate segregation effects.

## Carburizing Effects[4]

At elevated temperatures, some of the impurities present in low-alloy steels tend to segregate in areas such as grain boundaries and at the near surface. The case depths from gas carburizing, for example, can be negatively impacted since the carbon transfer is impeded.

In one experiment,[4] a 16MnCr5 steel (SAE 5115) was gas carburized at 930°C (1700°F) in an atmosphere of CO-H2O-H2-He. A carbon potential of 1.3% was achieved after 1,000 minutes for the standard alloy (0.0025% Sb). Increasing the antimony content to 0.017% (170 ppm) extended this time to 2,000 minutes and increasing it to 0.058% (580 ppm) extended the time to 6,000 minutes.

The following sequence was found for the retarding effect on gas carburizing of the these tramp elements: Sb > Sn > P > Cu > (Pb).

In some instances, such as high-temperature carburizing at 1040°C (1900°F), it is advantageous to deliberately add certain trace elements (e.g., Al, Nb, Ti, N) to pin the grain boundaries and help prevent excessive grain growth.

## Final Thoughts

Tramp elements often come to our attention when solving heat-treatment problems, where it is critical to know the full chemistry of the material under investigation (and not just the chemistry as reported on the mill certification sheets). Often, the prior heat-treatment history is also highly relevant.

Finally, it is important to understand how each tramp element affects the steel, either singularly or in combination with other elements. Going forward, by knowing the tramp elements present, we can better design our heat-treatment recipes and avoid costly failures. And The Doctor will sleep better at night.

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# Segregation and Banding in Carbon and Alloy Steel

No one likes surprises in manufacturing, and this is especially true during heat treatment. To avoid them, our attention is often focused on the type of material being supplied to us and the process/equipment variables we must control. What we don't often consider is the condition of the incoming raw material. In this regard, the result of alloy segregation and banding after heat treatment can cause considerable angst between the heat treater and their customer. Let's learn more.

Many people believe steels are classified only by their chemistry. In point of fact, the steelmaking process used; the casting process employed (ingot or continuous casting); the size/shape of semi-finished (blooms, billets or slabs) or finished products (plate, sheet or bars); and the properties they are required to have for the end-use application play a major role.

## Segregation

Steels are not chemically homogeneous; they do not have uniform chemistry throughout the entire cross-sectional area of their manufactured shapes. When steel is cast, the first material to solidify is the outer area adjacent to the mold walls (this surface zone is referred to as the chill zone). This results in a thin layer of equiaxed crystals with the same composition as the liquid metal. These crystals continue to grow inward as columnar grains in a dendritic shape parallel to the thermal gradient. Finally, solidification ends as the liquid temperature drops and neighboring grains impinge upon each other in the central zone of the as-cast shape (Fig. 1).

The solidification process causes macroscopic as well as microscopic partitioning of chemical elements as the liquid metal cools. Macroscopically, segregation occurs at the centerline of continuously cast products

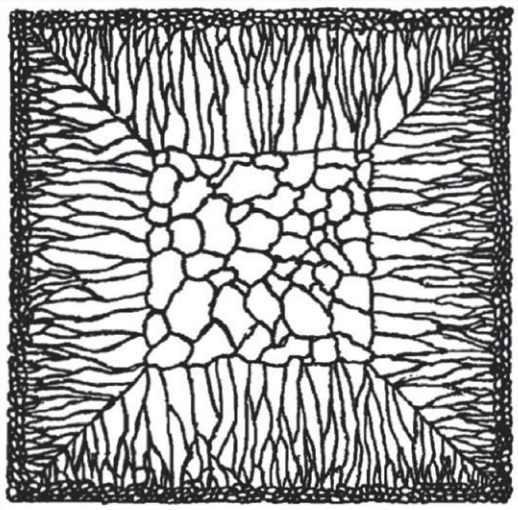

Fig. 1. Schematic diagram of zones of crystal morphologies in an as-solidified section of steel. Illustrated are the outer chill zone, the columnar zone and the interior equiaxed zone. Macro and microsegregation are not illustrated in this view.[1]

Fig. 2. Banded 4340 microstructure (50x, Vilella's Reagent)

and at the centerline, top and bottom of ingots. Microscopically, segregation occurs between dendrites throughout the solidified section. Subsequent mechanical hot working (e.g., rolling) creates longitudinal bands of varying elemental chemistry.

Microsegregation is the difference in composition between the center of the dendritic stem (i.e. the dendritic core) and the region between the dendrite arms. The first liquid to solidify (in the center of the dendrite) will be alloy-rich if the alloy addition raises the melting temperature, whereas the alloy (i.e. solute-rich) area will be concentrated at the interdendritic regions if the alloy addition lowers the melting temperature.

For the heat treater, alloy segregation can produce differences in the hardenability of the steel. The difference in hardenability between alloy-rich and alloy-lean regions can manifest itself by creating harder and softer areas of martensite and mixed transformation products such as bainite.

## Banding

Banding is caused by segregation of the alloying elements during solidification. Subsequent hot-working operations result in segregation aligned in the direction of working, which results in the banded appearance delineated in the microstructure. The distribution of microsegregation (in wrought steels) depends on how much working has been done to shape the part. The diffusion rates of the alloying elements in steel control the homogenization of the casting. For example, chromium and molybdenum homogenize readily, while nickel homogenizes very slowly. The alternating bands of varying alloy chemistry result in different microstructures, orientated parallel to the rolling direction of the material (Fig. 2).

Additional mechanical and/or thermal treatments to remove or reduce microsegregation add to the cost of manufacture and oftentimes are not considered economically feasible. For example, a high degree of homogenization can result from soaking the segregated steel at an elevated temperature (often above 1200°C or 2200°F) for a very long time (up to 100 hours or more in some cases).

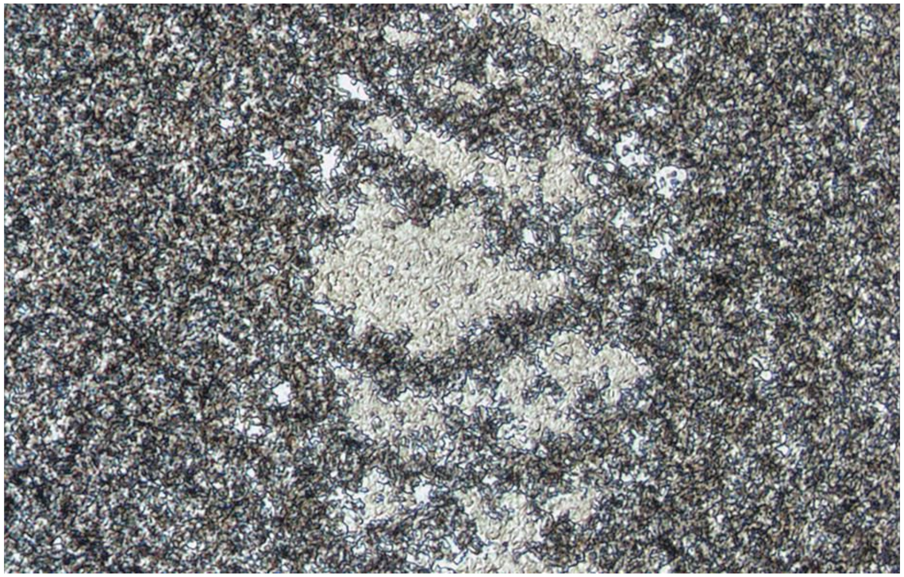
Fig. 3. 4340 clutch plate. A region of reformed (untempered) martensite (white) within the banded area of the heat-affected zone. This area exhibited hardness values significantly higher than those of the surrounding area. (1250x, Vilella's Reagent)

The primary cause of banding is due to the segregation of substitutional alloying elements (e.g., manganese, chromium, molybdenum) during (dendritic) solidification. Cooling rate, austenitic grain size and austenitizing temperature also influence the severity of microstructural banding.

Banding occurs in all steels. While reheating of as-cast products and hot rolling tend to reduce chemical segregation, other factors (related to solid-state phase transformations and residual solidification) result in a greater or lower degree of banding in the microstructure of all finished steel products.

## Effect of Banding on Heat Treatment

In general, hardness and microstructure will be heavily influenced by segregation and banding. Alloy-rich areas tend to transform to martensite or bainite, while alloy-lean areas show increasing amounts of pearlite and ferrite (due to slower cooling rates). For case-hardened parts, the hardness of the case will be impacted, particularly if higher concentrations of retained austenite or bainite are formed in the primarily martensite structure. In certain applications, retained austenite converts to

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untempered martensite in service (Fig. 3), resulting in hardness and property variation that can cause dimensional change or even component failure.

Tensile, yield and fatigue strength tend to be relatively unaffected by the presence or absence of microsegregation, although ductility and toughness properties are negatively impacted.

As an example, spotty hardness and poor mechanical properties can result from segregated or banded microstructures having significant amounts of coarse pearlite, coarse ferrite or ferrite clusters. In induction hardening, higher temperatures and longer heating times are required to fully austenitize these structures and may result in grain growth, formation of coarse martensite on quenching, surface oxidation/decarburization and increased chance of distortion. Thus, having a prior microstructure consisting of fine pearlite or a quench-and-tempered structure is the most highly desired for induction hardening.

## Summary

Segregation and banding may or may not be detrimental to the final product, but the effects of these conditions should either be evaluated before the raw material is heat treated or negated (to the extent possible) by appropriate thermal treatments (e.g., annealing, normalizing).

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# Influence of Alloying Elements on Austenite

Many of our heat-treatment processes require us to choose the proper austenitizing temperature so that subsequent transformation will produce the desired microstructure and properties. This choice is influenced by a number of factors, including the presence of various alloying elements and their effect on the composition, grain size and transformation of the austenite. Let's learn more.

## Classification of Alloying Elements

The ability of alloying elements to promote the formation of a particular phase or to stabilize that phase is well known.[1-4] Alloying elements (the most influential of which are shown in bold) are broadly classified as:

- Austenite formers (e.g., C, Ni, Mn, N, Cu, Co, Zn, Au)
- Austenite stabilizers (e.g., Ni, Mn, Co, Pt)
- Ferrite formers (e.g., Cr, Si, Mo, W, Al, B, Ta, Ti, V, Zr, Nb)
- Ferrite stabilizers (e.g., Si, Mo, Cr, Al, Be, P, Ti, Mo, V)
- Carbide formers (e.g., Cr, W, Mo, V, Ti, Nb, Ta, Zr)
- Carbide stabilizers (e.g., Cr, Mn, Si, W, Mo, V, Hf, Ti, Ta, Zr)
- Nitride formers (e.g., Al, Cr, Ti, Mo, V, Nb, Ta, Zr)

It should also be noted that the influence of carbide stabilizers is dependent on the presence of other alloying elements in steel. Austenite stabilizers lower the eutectoid temperature, thereby expanding the austenite phase field (i.e., temperature range over which austenite is stable). By contrast, ferrite formers raise the eutectoid temperature, thereby shrinking the austenite phase field.

In addition, alloying elements affect such heat-treatment issues as the temperature of martensite formation, formation of pearlite and bainite during isothermal transformation and resistance to tempering.

## Alloying Effects

As additions to steel, certain alloying elements are known to have very specific effects (Table 1). These same elements influence the size of the austenite

**Table 1. Alloying-element effects[4]**

| Alloying element | Effect(s) |
|---|---|
| Co | Improvement in hot hardness |
| Cr | Formation of wear-resistance carbides; corrosion resistance (in large amounts); oxidation resistance on heating; increased depth of hardening |
| Cu | Precipitation-hardening effect (over 0.80%); atmospheric corrosion resistance |
| Mn | Increased depth of hardening |
| Mo | Reduces tendency toward temper embrittlement; high-temperature strength (especially when other carbide formers are present); hot hardness |
| Nb | Fixation of carbon |
| Ni | Improves toughness without brittleness; improves low-temperature impact resistance |
| P | Atmospheric corrosion resistance |
| Si | Graphitization effects |
| Ti | Fixation of carbon |
| V | Grain-size control; improvement in toughness |
| W | High-temperature strength; improvement in hot hardness |

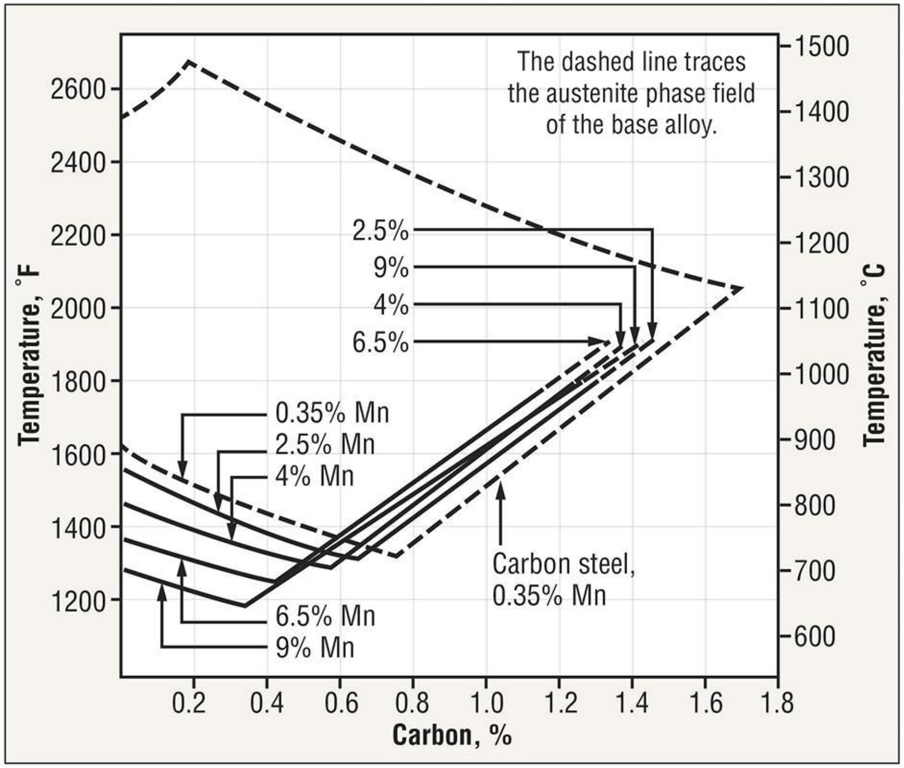

Fig. 1. Effect of manganese and carbon on the austenite phase field as a function of temperature[2]

phase field (Figs. 1-5; note: Figs. 3-5 are only online). In these figures, the field shown is for pure austenite formed at equilibrium. In using them, it is important to note that compositions lying to the right of the triangles will be largely austenite, with increasing amounts of carbide (or possibly graphite in the case of high-silicon steels). Below the areas of pure austenite (i.e., at lower temperature), a three-phase zone exists in which ferrite, austenite and carbide will be present even at equilibrium. To the left of the austenite field, austenite with more or

**Table 2. Alloying-element influence during heat treatment[4]**

| Effect | Influence of alloying element | |
|---|---|---|
| Carbide formation | Very strong: Ti, Nb, V | Strong: Cr, Mo, W |
| | Fairly strong: Mn | None: P, Ni, Cu, Si |
| Decrease eutectoid content | Strong influence: Mn, Cr, V | No influence: Si, Cu, Al |
| | Weak influence: Ni, Mo, W | |
| Improve depth of hardening | Strong: Mo, W, Mn, Cr | Medium: Si |
| | Weak: Cu, Ni | Very weak: V |
| Retard grain growth of austenite | Very strong: Al | Fairly strong: V, Ti, Zr |
| | Medium: Mo | Very weak: Mn |
| | Weak: Cr, Si | No influence: Ni |
| Stabilize martensite on tempering | Strong influence: Mo, W, V (fair) | |
| | Weak influence: Cr (weak), Mn (very weak) | |
| Strengthen ferrite | Very strong: P | Strong: Si, Mn |
| | Fairly strong: Ni, Cu, Cr, Mo, W | Weak: V |

less ferrite is found.[2]

Alloying elements also confer certain desirable characteristics for heat treatment (Table 2). For example, all alloying elements (with the possible exception of Co) lower the martensite start ($M_s$) temperature, with carbon having the greatest influence. In those steels over 0.5% C, the martensite finish ($M_f$) temperature is typically below room temperature, implying that retained austenite will form on hardening. Similarly, these same alloying elements delay the formation of ferrite and cementite (i.e., the nose of the TTT diagram

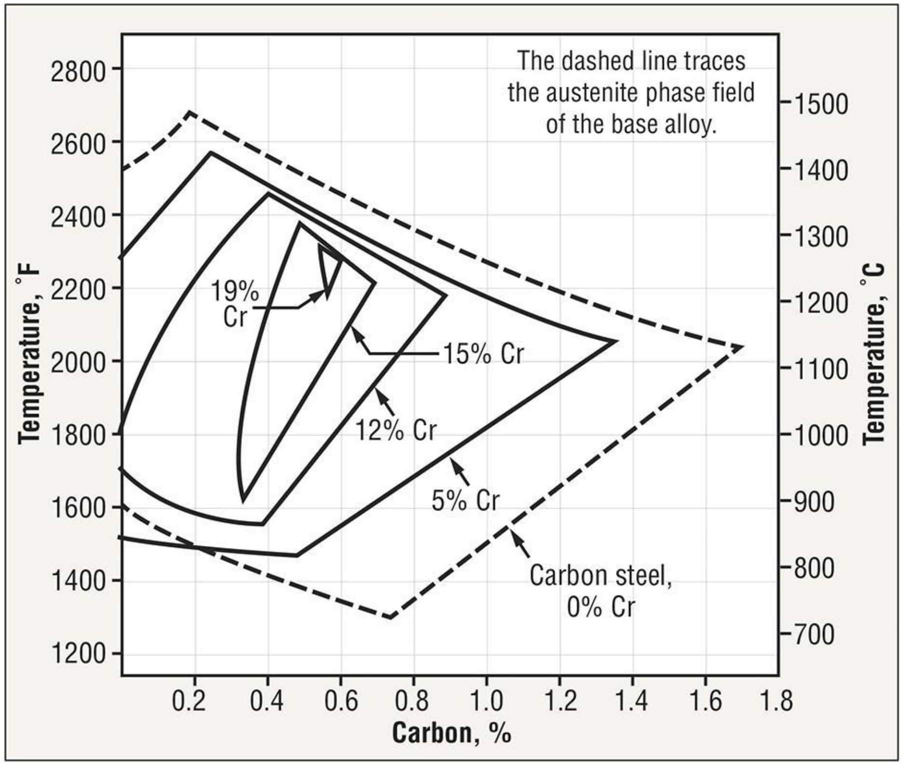

Fig. 2. Effect of chromium and carbon on the austenite phase field as a function of temperature[2]

is shifted to the right), and some elements affect the bainite transformation more than the pearlite transformation. In addition, the pearlite-bainite transformation is delayed slightly at carbon contents over 1% (helpful, for example, in carburizing and hardening of tool steels).

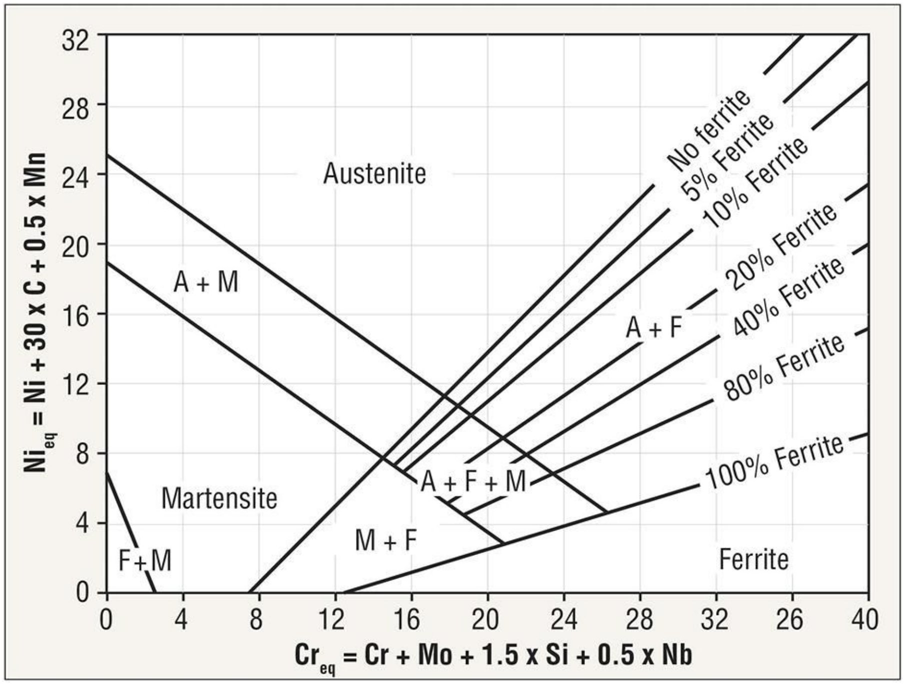

Fig. 6. Schaeffler diagram (stainless steel grades are illustrated)[6]

## Schaeffler-Delong Diagrams

The Schaeffler diagram (Fig. 6) and the Schaeffler-Delong diagram (Fig. 7) can be used to predict the influence of microstructural transformation by three or more alloying elements.[5] In these diagrams, the austenite formers are arranged along the y-axis and the ferrite formers along the x-axis. The original Schaeffler diagram involved only Ni and Cr, but the Delong version includes other elements and gives them coefficients that reduce them to their Ni or

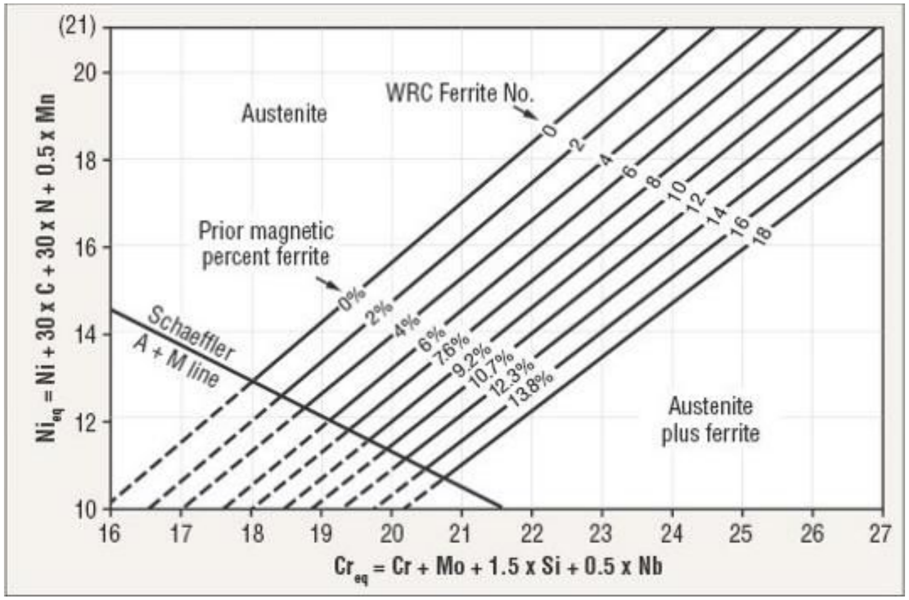

Fig. 7. Delong diagram for stainless steel
Note: The Schaeffler austenite-martensite boundary is included for reference.

Cr equivalents. A "nickel equivalent" is calculated for the austenite-stabilizing elements (Equation 1) and a "chromium equivalent" for the ferrite-stabilizing elements (Equation 2). These diagrams are particularly useful for predicting ferrite levels in austenitic stainless steel welds.

The nickel and chromium equivalents can be determined as follows:

1. Ni (equivalent) = Ni + (30 x C) + (0.5 x Mn) + (30 x N)
2. Cr (equivalent) = Cr + Mo + (1.5 x Si) + (0.5 x Nb)

## Conclusion

In order to select the right austenitizing temperature or make minor adjustments to published data, the heat treater needs to understand the influence of alloying elements on the austenite phase field. The austenitizing temperature is influenced not only by the presence of alloying elements and their relative amounts but also by how they interact with one another. Selecting an improper austenitizing temperature (too high or too low for the given steel) can result in an undesirable microstructure and failure to achieve desired properties.

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# Martensite

“Martensite is our friend,” so sayeth the heat treater, but what is martensite, really? And why is a tempered martensitic structure the single-minded goal of every heat treater when hardening steel? Let’s learn more.

## Martensite Formation

In order to form martensite we need to heat steel into the austenite field (above $Ac_3$) and quench rapidly enough from the austenite phase to avoid pearlite formation. The rate must be fast enough to avoid the nose of the Time-Temperature-Transformation (TTT) curve – the so-called critical cooling rate for the given steel. The formation of martensite involves the structural rearrangement (by shear displacement) of the atoms from face-centered cubic (FCC) austenite into a body-centered tetragonal (BCT) martensitic structure. This change is accompanied by a large increase in volume and results in a highly stressed condition. This is why martensite has a higher hardness than austenite for the exact same chemistry.

The martensite transformation, while not instantaneous, is significantly faster than diffusion-controlled processes such as ferrite and pearlite

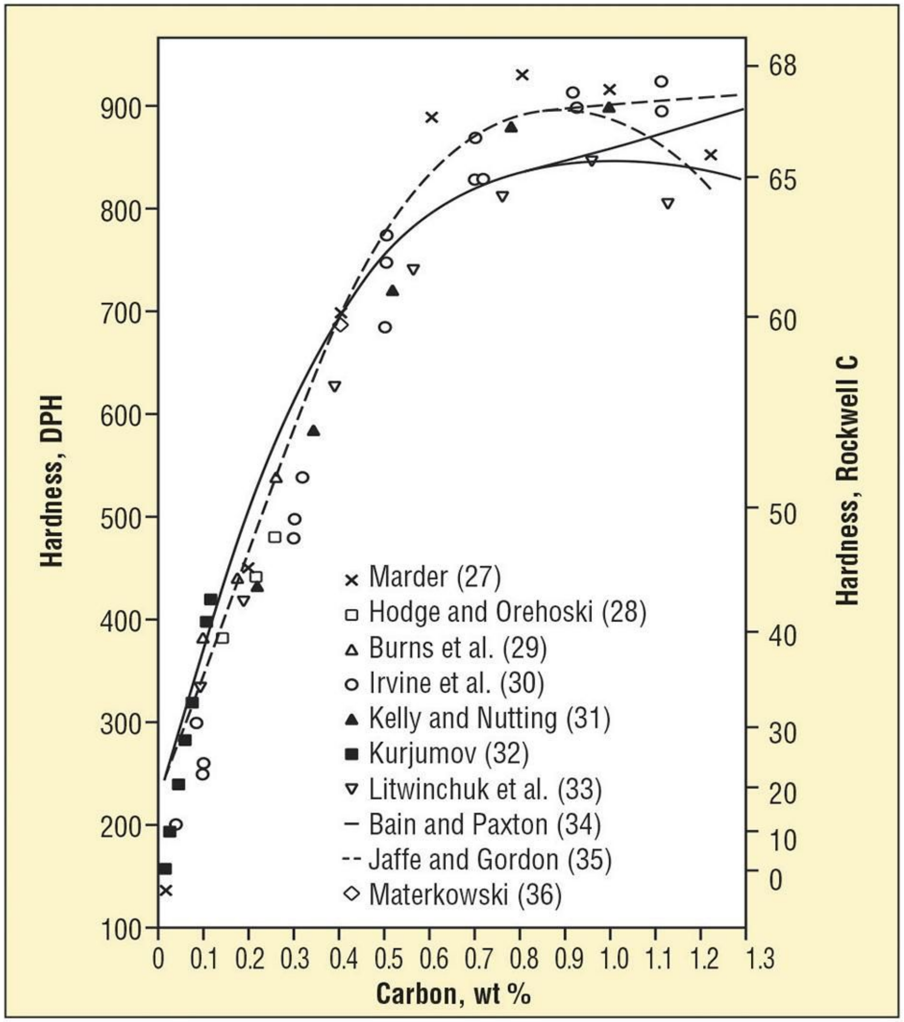

Fig. 1. As-quenched hardness vs. carbon content[1]

formation that have different chemical compositions than the austenite from which they came. Thus, martensite is a meta-stable, strain-induced state that steel finds itself in. The resultant steel hardness is (primarily) a function of its carbon content (Fig. 1).

## Martensite Morphology

Morphology is a term used by metallurgists to describe the study of the shape, size, texture and phase distribution of physical objects. Martensite can be observed in the microstructure of steel in two distinctly different forms – lath or plate – depending on the carbon content of the steel (Fig. 2). In general, lath martensite is associated with high toughness and ductility but low strength, while plate

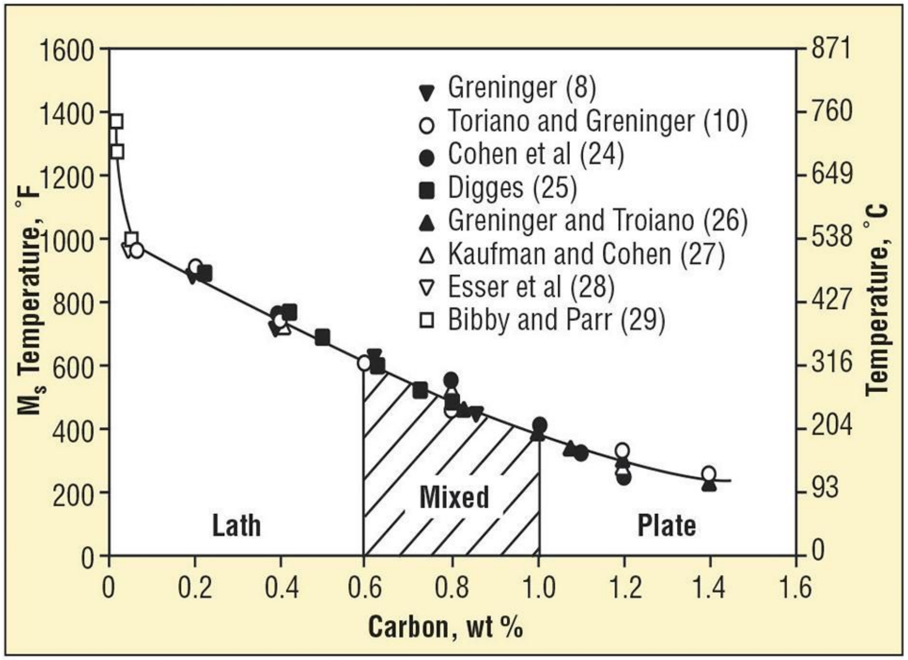

Fig. 2. Formation of lath and plate martensite[1]

Fig. 3. Lath martensite example (carburized 8620)[4]

Fig. 4. Plate martensite example (carburized 8620)[4]

Fig. 5. Coarse martensite (carburized SAE 9310)

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martensite structures are much higher in strength but tend to be more brittle and non-ductile.[2]

For alloys containing less than approximately 0.60 wt.% carbon, lath martensite appears as long, thin plates often grouped in packets (Fig. 3). Plate (or lenticular) martensite is found in alloys containing greater than approximately 0.60 wt.% carbon. The microstructure is needle-like or plate-like in appearance across the complete austenite grain (Fig. 4). With carbon contents between 0.60 and 1.00 wt.% carbon, the martensite present is a mixture of lath and plate types.

**Table 1. Crystal structures formed in martensite or quasi-martensite transformations[6]**

| Alloy system | Crystal structure change[a] |
|---|---|
| Co, Fe-Mn, Fe-Cr-Ni | FCC to HCP |
| Fe-Ni | FCC to BCC |
| Fe-C, Fe-Ni-C, Fe-Cr-C, Fe-Mn-C | FCC to BCT |
| In-Ti, Mn-Cu | FCC to BCT |
| Li, Zr, Ti, Ti-Mo, Ti-Mn | BCC to HCP |
| Cu-Zn, Cu-Sn | BCC to FCT |
| Cu-Al | BCC to HCP (distorted) |
| Au-Cd | BCC to Orthorhombic |
| $ZrO_2$ | Tetragonal to Monoclinic |

Notes: [a]FCC = face-centered cubic; BCC = body-centered cubic; HCP = hexagonal close packed; BCT = body-centered tetragonal; FCT = face-centered tetragonal;

As the carbon content increases, so-called high-carbon martensite twins begin to replace dislocations within the plates. This transformation is accompanied by the volumetric expansion mentioned earlier, creating (residual) stress in addition to the strains due to interstitial solute atoms. At high carbon levels these stresses can become so severe that the material cracks during transformation when a growing plate impinges on an existing plate.[3] Thus, coarse martensite (Fig. 5) and plate martensite are less desirable structures in most applications.

## $M_s$ and $M_f$ Temperatures

The martensite transformation begins at the martensite start ($M_s$) temperature and ends at the martensite finish ($M_f$) temperature and is influenced by carbon content. Increasing the carbon content of the austenite depresses the $M_s$ and $M_f$ temperatures, which leads to difficulties in converting all of the austenite to martensite. $M_s$ and $M_f$ temperatures are also important in welding, as they influence the residual stress state.[5] $M_s$ and $M_f$ temperatures can be calculated, and if you need to know them for a particular steel, one source for this data is at www.thomas-sourmail.org/martensite.html, which lists over 1,000 different steel types.

## Tempered Martensite

All steels containing martensite should be tempered. As heat treaters, we need to know that martensite in steel produces a hard, brittle microstructure that must be tempered to provide the delicate balance necessary between strength and toughness needed to produce a useful engineering material. When martensite is tempered, it partially decomposes into ferrite and cementite. Tempered martensite is not as hard as just-quenched martensite, but it is much tougher and is finer-grained as well.

## Final Thoughts about Martensite

The heat treater might be interested to know that martensite formation is not restricted just to steels because other alloy systems produce crystallographic changes of a similar nature (Table 1). Learning more about martensite is an essential part of what we need to do as heat treaters since it is one of the defining characteristics of our industry.

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# A Discussion of Retained Austenite

What is retained austenite and how does it affect the properties of a component? How much, if any, retained austenite should be present in a particular component microstructure? Is the presence of retained austenite in a microstructure a good thing or a concern? These are questions that metallurgists have spent countless hours debating. What do we as heat treaters need to know about retained austenite and how is retained austenite viewed by various industries? Let's learn more.

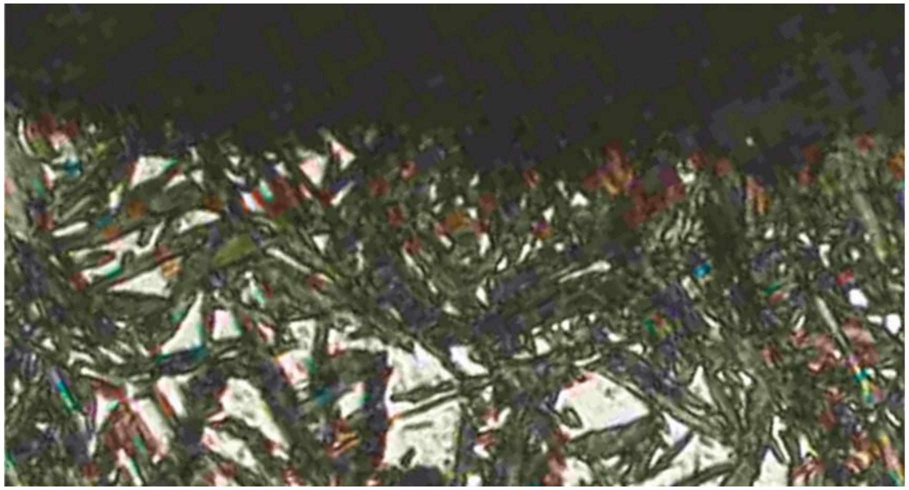

Fig. 1. RA present in a case carburized component [1]. Photomicrograph courtesy of Alan Stone, Aston Metallurgical Services (www.astonmet.com). Etchant: 2% nital. 1,000x

## What is retained austenite?

Austenite that does not transform to martensite upon quenching is called retained austenite (RA). Thus, retained austenite occurs when steel is not quenched to the $M_f$, or martensite finish, temperature; that is, low enough to form 100% martensite. Because the $M_f$ is below room temperature in alloys containing more than 0.30% carbon, significant amounts of untransformed, or retained austenite, may be present, intermingled with martensite at room temperature (Fig. 1). Retained austenite is a specific crystalline form of iron and steel. The dark-colored needles shown are tempered martensite crystals and the light-colored areas are retained austenite crystals. The amount of retained austenite is a function of the carbon content, alloy content (especially nickel and manganese), quenchant temperature and subsequent thermal and/or mechanical treatments.

Depending on the steel chemistry and specific heat treatment, the retained austenite level in the case can vary from over 50% of the structure to nearly zero. While large amounts of retained austenite (>15%) can be detected and estimated by optical microscopy, specialized equipment and techniques, such as x-ray diffraction methods, are required to accurately measure the amount of retained austenite to as low as 0.5%.

## Why is retained austenite problematic?

The very characteristics that give retained austenite many of its unique properties are those responsible for significant problems in most applications. We know that austenite is the normal phase of steel at high temperatures, but not at room temperature. Because retained austenite exists outside of its normal temperature range, it is metastable. This means that when given the opportunity, it will change or transform from austenite into martensite. In addition, a volume change (increase) accompanies this transformation and induces a great deal of internal stress in a component, often manifesting itself as cracks.

## How does RA behave?

Martensite is hard, strong and brittle while austenite is soft and tough. In some instances, when combined, the mixture of austenite and martensite creates a composite material that has some of the benefits of each, while compensating for the shortcomings of both.

For any given application, mechanical properties are affected by a high percentage of retained austenite content. For example, retained austenite affects the following properties of bearing steels:

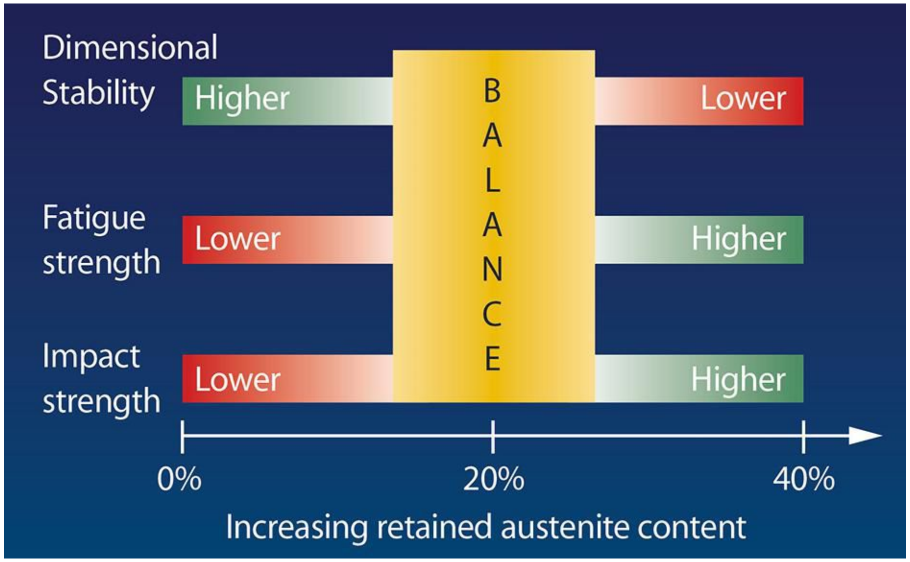

Fig. 2. Balancing properties and RA content [2]

- Dimensional stability: Retained austenite will transform to martensite if the temperature drops significantly below the lowest temperature to which it was quenched, or if the room temperature austenite is subjected to high levels of mechanical stress. Martensite, a body centered tetragonal crystal structure, has a larger volume than the face centered cubic austenite that it replaces. Where transformation occurs, there will be a localized 4-5% increase in the volume of the microstructure at room temperature and a resulting dimensional change in the geometry of the component. If great enough, this dimensional change could lead to growth and in severe instances, crack initiation.

- Fatigue: Low retained austenite content and fine austenitic grain sizes, which create a microstructure of finely dispersed retained austenite and tempered martensite, prevent nucleation of fatigue cracks, or retard fatigue crack initiation until very high stress levels are reached. In contrast, low-stress applications that fracture at low cycles are related to high retained austenite levels and coarse austenite grain sizes. For example, one type of fatigue strength of interest to many users is rolling contact fatigue. Two aspects of retained austenite can improve rolling contact fatigue life. First, the inherent ductility of retained austenite helps to delay crack growth by blunting the tips of cracks as they form. Second, as retained austenite transforms during service, compressive residual stresses increase in the case. These compressive stresses delay crack growth by functioning like a vise and clamping cracks closed. These benefits are not present in a part with insufficient retained austenite.
- Impact: Impact strength is the measure of the ability of a steel to resist fracture when subjected to a sharp blow. Austenite is not only very tough, but also it has higher impact strength than martensite. The steel's impact strength increases with increasing austenite content. Higher impact strength can provide extra protection against cracking, which, in turn, helps prevent such problems as spalling.

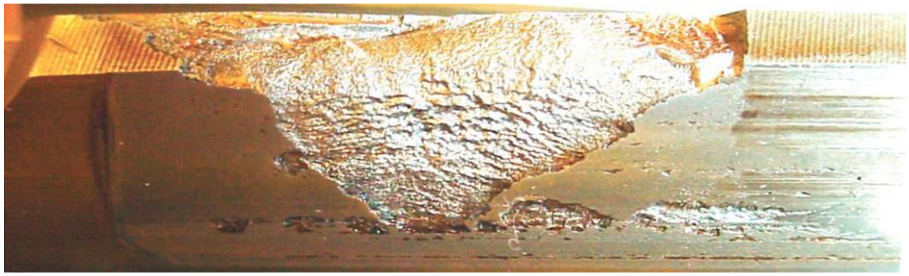
Fig. 3. (above) Gear tooth failure due to spalling (macropitting)

It is important to recognize that a balance must be created between the mechanical properties of a component and the optimum percentage of retained austenite for a given application (Fig. 2).

## How some industries view retained austenite

Retained austenite is highly undesirable in components for the tool and die industry. RA is recognized as a major cause for premature failure. The low hardness of RA is also incompatible with most applications that require the maximum attainable hardness to resist wear.

The bearing and gear industries have a more favorable view toward having some retained austenite (5 to 30% determined by optical metallography, usually by comparison to known standards). While some of the same mechanisms that affect tooling applications also affect gears, there are some major differences. Gears are typically made of case-hardened steel, which has high impact strength. While most tools fail by wear or fracture, many gear failures are the result of spalling in the tooth area. Spalling is progressive macropitting that occurs when pits coalesce and form irregular craters, which cover a significant area of the tooth surface (Fig. 3). Spalling occurs when the surface of a metal component is subjected to repeated cyclic loads. A crack forms and grows until a small portion of the surface breaks loose, damaging the surface and adding debris to the system. The gear industry balances the amount of retained austenite in a gear tooth to delay the onset of spalling by suppressing crack growth.

## How is the percentage of RA reduced?

Tempering is one method used to transform retained austenite. A key is to hold for an adequate amount of time at temperature. Multiple tempers are often performed to ensure the maximum amount of retained austenite has been transformed. Other popular methods include cold treatment at -120ºF (-85ºC) or cryogenic treatment to -320ºF (-195ºC). It is well documented that as the temperature is lowered the degree of transformation increases.

## Conclusion

By controlling the level of retained austenite, its beneficial effects can be realized without suffering from its negative influences, such as excessive dimensional growth. Many industries have found a "sweet spot" exists for retained austenite content to achieve a balance of fatigue/impact strength and dimensional stability.

To obtain the optimum level of retained austenite requires a delicate balance of controls and must take into account such items as material chemistry and heat treatment process variables. These variables include steel chemistry, carbon content, austenitizing temperature, quenching rate and tempering temperature.

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# Alloy Carbides

The subject of carbide formation in steels has intrigued The Doctor ever since he first peered into a microscope and observed them (Fig. 1). It is also a very important subject for the heat treater to better understand. Carbides can be our friend or our enemy. Let's learn more.

## Carbide Formation

In simplest terms, alloying elements in steels can be divided into two groups: those that do not form carbides (e.g., Al, Co, Cu, N, Ni and Si) and those that do (e.g., Mn, Cr, W, Mo, V, Zr, Nb, Ta and Ti). This latter group is arranged in accordance with their affinity for carbon.

The periodic table of elements (Fig. 2) tells us these carbide formers fall to the left of iron. Unstable carbides, those that will dissociate on heating, can be found at the far left end of each row, while the elements closest to iron form extremely stable carbides that dissociate at temperatures much higher than the critical temperatures for steel.[1]

Carbide formation in steels is typically limited to a few carbide types (Table 1). Here M represents the carbon-forming elements in steel. The types, combinations and amount of alloying elements present complicate carbide formation. For example, in a Cr-Mn steel, the carbide $(Cr,Mn,Fe)_{23}C$ forms in lieu of a $Cr_{23}C$ carbide.[1]

The stability of the carbides is highly dependent on the presence of other alloying elements in the steel. For example, while manganese is a (very) weak carbide former, it is a relatively potent carbide stabilizer. Remember, all carbide formers are also nitride formers.

Some alloying elements (e.g., Ni, Co, Al) cause graphitization of cementite (iron carbide) and for this reason are not typically added to high-carbon steels in any appreciable amount unless to counteract a strong carbide former.

In addition to forming carbides, alloying elements influence ferrite and pearlite interaction, martensite and bainite transformation, retained austenite and quenching (by influencing the alloy transformation diagram).

Table 1. Carbide formation in steels[1]

| Group-I carbides | Examples | Group-II carbides | Examples |
|---|---|---|---|
| $M_3C$ | $Fe_3C$, $Mn_3C$ | MC | WC, VC, TiC, NbC, ZrC |
| $M_{23}C_6$ | $Cr_{23}C_6$ | $M_2C$ | $Mo_2C$, $W_2C$, $Ta_2C$ |
| $M_7C_3$ | $Cr_7C_3$ | | |
| $M_6C$ | $Fe_3Mo_3C$, $Fe_3W_3C$ | | |

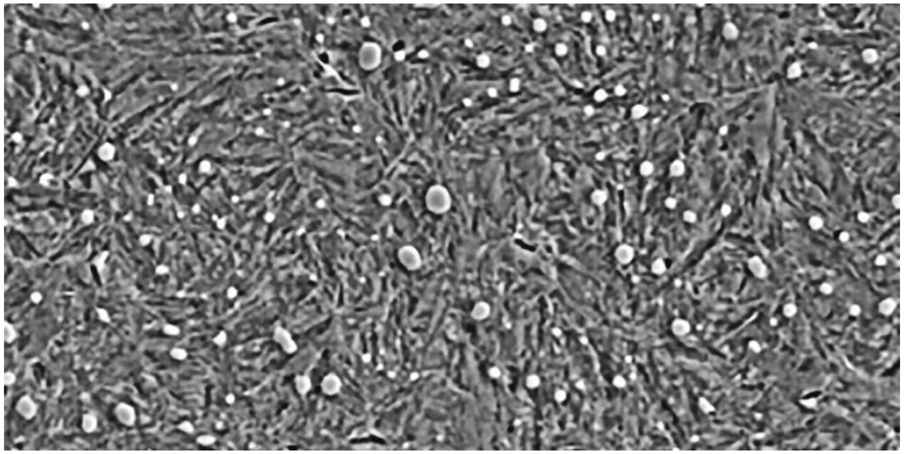


Fig. 1. Finely dispersed alloy carbides in a matrix of tempered martensite. Vacuum-hardened and high-pressure gas-quenched 52100 steel (3,900X).

## Carburized Steels[3]

In general, finely dispersed carbides are not considered to be detrimental to carburized alloy steels. Small spheroidal carbides or incidental carbides observed in many high-carbon martensite structures are considered routine. However, grain-boundary carbides, massive carbides that occur on edges and corners, network carbides and carbide necklaces are deemed detrimental to mechanical properties and should be avoided.

The number of carbides present in low-temperature-tempered carburized alloy steels is typically less than 10%. While carbides are harder than the surrounding matrix (martensite/austenite), they do not have an appreciable effect on Rockwell (macro) hardness at this percentage. Carbides are known to enhance wear resistance. It has also been reported that contact loading of certain types of gears (at very high contact pressures away from the fatigue limit) may be enhanced, but grain-boundary and network carbides are known to be detrimental to bending fatigue and should be avoided.

Steps many heat treaters and design engineers have found useful in minimizing carbide formation during carburizing include:

- Use of fine-grained steels (to reduce the amount of carbon deposited at the grain boundaries) or elements that pin the grain boundaries to avoid excessive grain growth.
- Limiting of the furnace-atmosphere carbon potential. For example, running at a carbon potential in excess of the limit of saturation of carbon in austenite (Acm) can create carbides during carburizing. Processes using these techniques must have ways to avoid carbide formation.
- Use of a constant carbon potential (e.g., 0.80%) throughout the carburizing cycle (as opposed to a boost/diffuse cycle).
- Avoidance of excessively long, slow cooling (i.e., drop temperature) steps in the carburizing process (to avoid network carbides).
- Subcritical annealing after carburizing and slow cooling prior to reheat and quench.

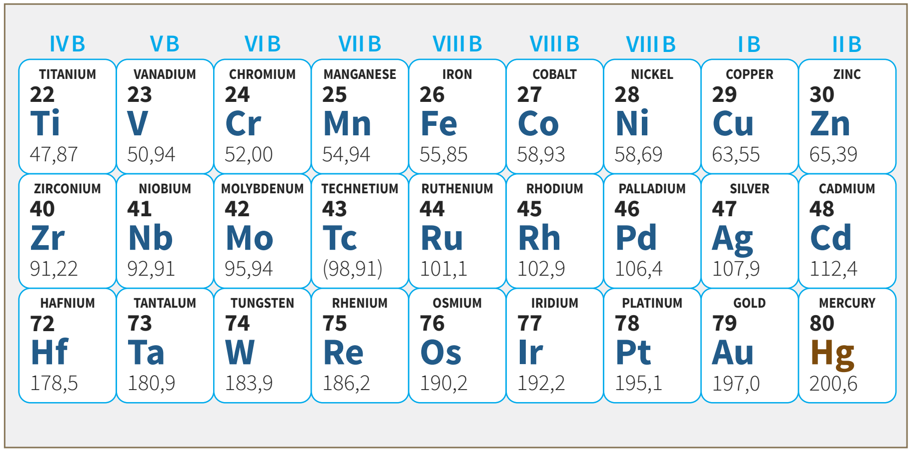
- Avoidance of geometry effects such as sharp corners or radii. Edge round where possible.
- Limiting the reheat cycle temperature to avoid grain growth, excessive distortion and retained austenite.

## Tempering[6]

Tempering of alloy steels differs from that of low-carbon steels in that the presence of retained austenite and strong carbide-forming elements results in the precipitation of finely dispersed alloy carbides (often referred to as temper carbides) at temperatures greater than about 500˚C (930˚F). These markedly contribute to the hardness of alloy steels. The hardness of martensite in alloy steels initially decreases as the tempering temperature is increased, but then carbon supersaturation is relieved by the precipitation of carbides (iron carbides at low temperature, alloy carbides at higher tempering temperatures where substitutional alloying allows diffusion to take place) and reaches a maximum between 500-700˚C (930-1300˚F).

## Tool Steels[7]

Alloying to create large amounts of carbides is a major difference between low-alloy steels and tool steels. Tool-steel carbides have been discussed previously,[8] but the focus here is on the relative hardness of the various carbides. Tool steels contain carbon, anywhere from about 0.5% to over 2%. Tool steel with 0.5% carbon will harden into the 60 HRC range during heat treatment. Therefore, any excess carbon will combine with other elements to form carbide particles. These carbide particles are extremely small and constitute from less than 5% to over 20% of the total volume of the microstructure. The actual hardness of individual carbide particles depends on their chemical composition, but, in general, chromium carbides are 65-70 HRC, molybdenum and tungsten carbides are in the range of 75 HRC, and vanadium carbides are in the range of 80-85 HRC.



| IVB | VB | VIB | VIIB | VIIIB | VIIIB | VIIIB | IB | IIB |
|---|---|---|---|---|---|---|---|---|
| TITANIUM<br>22<br>**Ti**<br>47,87 | VANADIUM<br>23<br>**V**<br>50,94 | CHROMIUM<br>24<br>**Cr**<br>52,00 | MANGANESE<br>25<br>**Mn**<br>54,94 | IRON<br>26<br>**Fe**<br>55,85 | COBALT<br>27<br>**Co**<br>58,93 | NICKEL<br>28<br>**Ni**<br>58,69 | COPPER<br>29<br>**Cu**<br>63,55 | ZINC<br>30<br>**Zn**<br>65,39 |
| ZIRCONIUM<br>40<br>**Zr**<br>91,22 | NIOBIUM<br>41<br>**Nb**<br>92,91 | MOLYBDENUM<br>42<br>**Mo**<br>95,94 | TECHNETIUM<br>43<br>**Tc**<br>(98,91) | RUTHENIUM<br>44<br>**Ru**<br>101,1 | RHODIUM<br>45<br>**Rh**<br>102,9 | PALLADIUM<br>46<br>**Pd**<br>106,4 | SILVER<br>47<br>**Ag**<br>107,9 | CADMIUM<br>48<br>**Cd**<br>112,4 |
| HAFNIUM<br>72<br>**Hf**<br>178,5 | TANTALUM<br>73<br>**Ta**<br>180,9 | TUNGSTEN<br>74<br>**W**<br>183,9 | RHENIUM<br>75<br>**Re**<br>186,2 | OSMIUM<br>76<br>**Os**<br>190,2 | IRIDIUM<br>77<br>**Ir**<br>192,2 | PLATINUM<br>78<br>**Pt**<br>195,1 | GOLD<br>79<br>**Au**<br>197,0 | MERCURY<br>80<br>**Hg**<br>200,6 |

Fig. 2. Partial periodic table of the elements[2]

The amount and type of carbide present in a particular grade of steel is largely responsible for differences in wear resistance (for the same relative hardness, tool steels with greater amounts of carbides or carbides of a higher hardness will show better resistance to wear). Large volume fractions of carbides optimize hardness and wear resistance, but there is a trade-off with hot forming, heat treatment and machinability. Very high amounts of carbide particles present in a material (such as in tungsten carbide) can lead to problems in grinding and lower toughness.

## Summary

Alloy carbides play a significant role in both the microstructure and resultant mechanical properties of alloy steels. For this reason the heat treater must pay particular attention to them, know when and why they are being formed, and if they will be helpful or harmful to the end-use application of the product.

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# NMTPs (Non-Martensitic Transformation Products)

The Doctor's first exposure to the subject of non-martensitic transformation products (NMTPs) in steel was many years ago as a young metallurgist who was asked to comment on the microstructure observed in the root of a carburized gear tooth. A "poor quench" was the simple explanation offered for what was observed. NMTPs require a bit more study today, but they are still undesirable and a subject the heat treater should better understand and strive to avoid. Let's learn more.

## Martensitic Transformation

Most of us know that a martensitic structure is the desired outcome of our steel heat-treatment process, but we often fail to fully appreciate the underlying transformation mechanism. We know our quench must be rapid – less than a second or two for a carburized 8620 steel – to avoid the nose of the curve and prevent the formation of NMTPs. Did you ever wonder, however, how a part weighing just a few pounds or a load of parts weighing several thousand pounds can reach these desired quench rates? Or how we can achieve that delicate microstructural balance between the higher-carbon surface of a carburized part and the carbon-gradient-related sub-case and low-carbon core? Both the martensite start ($M_s$) and martensite finish ($M_f$) temperatures are a function of carbon and alloy content. A higher carbon level, for example, reduces the $M_s$ point. The secret is in the martensite transformation process itself.

To begin with, martensitic transformations occur extremely fast (approaching the speed of sound) and have been reported to reach speeds as high as 1,100 m/s (3,280 feet/second) in steel.[1] Furthermore, martensitic transformations are often referred to as diffusionless, meaning they do not rely on diffusion mechanisms (i.e. the long-range diffusion of atoms) for the nucleation and growth of new phases. In these diffusionless transformations, atoms move only short distances (on the order of interatomic spacing) in order to create the new phase. Pearlite formation is an example of nucleation and growth by diffusion.

Since a martensitic transformation involves the coordinated movement of atoms, it is considered an ordered phase change. This change in

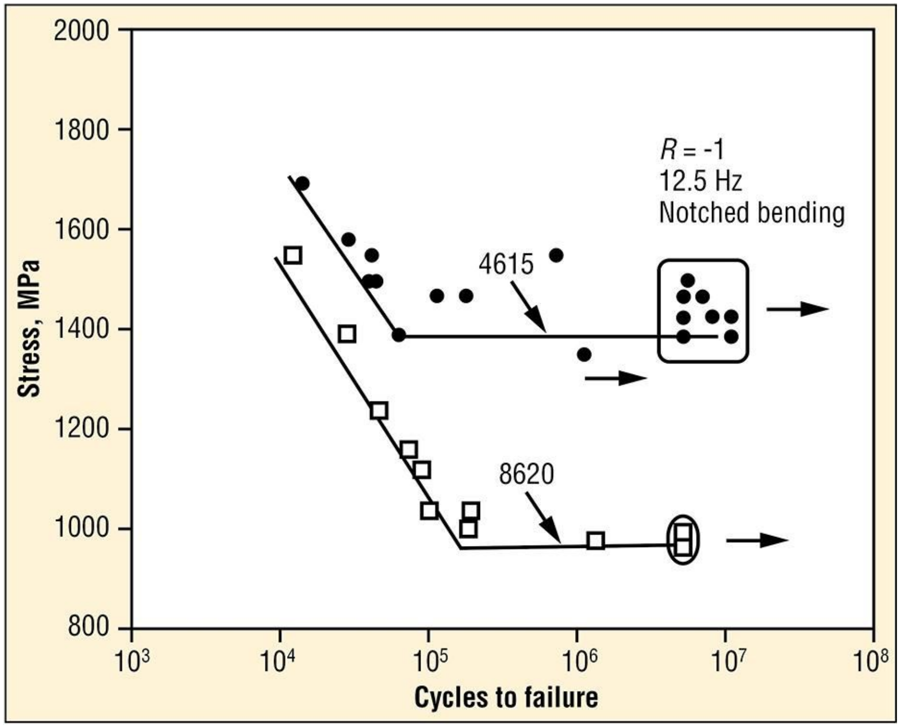

Fig. 1. Residual stress as a function of depth below the surface of direct-quenched 8620 and 4615 steel samples with and without the presence of NMTPs[6]

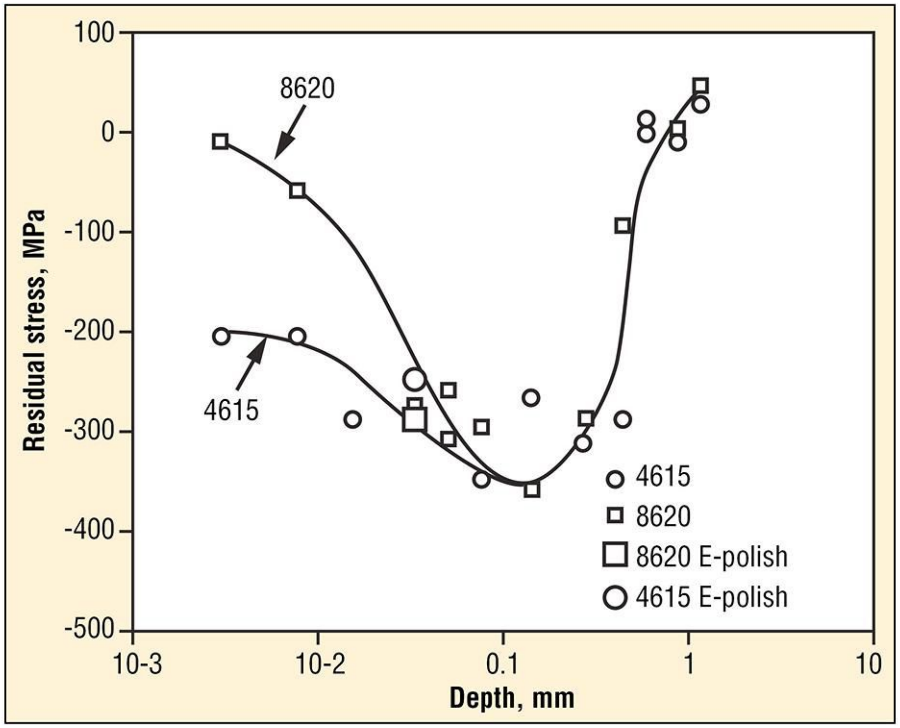

Fig. 2. Influence of NMTPs on the surface of 8620 steel samples when compared to the absence of NMTPs on the surface of 4615 steel samples[6]

crystallographic structure from body-centered cubic (BCC) austenite to body-centered tetragonal (BCT) martensite means that there is a strain associated with the transformation. This is why a heat-treated part that has transformed to martensite is often referred to as being in a strain-induced state. The preferred sites for carbon as an interstitial alloying element are the reason why a tetragonal structure forms. The Bain model or Nishiyama-Wassermann path model can be used to describe this transformation.

Martensite is not represented on (equilibrium) phase diagrams because it differs from equilibrium in two significant ways. Martensite grows without diffusion, so it retains the same chemical composition as austenite, and the shape of the deformation causes strains that must be accounted

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for before transformation can happen. Also, the hardness of martensite is a function of the carbon content (since the interstitial carbon atoms hinder dislocation movement and promote solid-solution hardening).

Martensite formation rarely goes to completion because of the strain associated with the product that leads back to stresses in the parent phase. On cooling of metastable austenite to the $M_s$ temperature, for example, approximately 1% martensite forms, is time independent and is approximately 99% complete at the $M_f$ temperature. It's a so-called athermal transformation that is a function of the temperature to which the part is quenched and not the time.

Finally, martensitic transformations are not limited to steels and occur in nonferrous alloys, pure metals, ceramics, polymers, inorganic compounds and even solidified gases. The mechanism of solid-state transformation is similar.

## Non-Martensitic Transformation

You may be asking yourself, why do we want to avoid NMTPs? The carburizing and subsequent quench hardening of low-alloy steel gears is an example. Surface microstructures typically contain martensite, retained austenite and in some cases NMTPs (e.g., bainite, ferrite, pearlite and mixed microstructures). The NMTPs are caused by a reduction of near-surface case hardenability due, among other reasons, to a loss of alloying elements (e.g., Cr, Mn, Si) to oxidation.[4] Not only is the surface hardness reduced, the residual surface stresses may become less compressive or even tensile (Fig. 1).

For example, 8620 with NMTPs on the surface have been shown to have inferior bending-fatigue properties when compared to more highly alloyed steels such as 4615 (with higher nickel and molybdenum) and a lower sensitivity to surface oxidation by virtue of reduced manganese and chromium contents, which do not form NMTPs (Fig. 2). If the hardenability of the steel is sufficient to prevent the formation of NMTPs, surface oxidation has a far lower effect on bending fatigue performance.

Looking at the bigger picture, we must constantly strive to find ways to improve fatigue performance and lengthen service life. To this end, we want to decrease the surface cyclic tensile stress and/or increase the surface yield stress, thereby increasing the resistance to fatigue-crack nucleation. To achieve this, surface-modification processes based on heat treatment (e.g., carburizing, carbonitriding, laser hardening and induction hardening), non-uniform plastic deformation (e.g., peening and deep rolling) or selected surface-alloy modification (e.g., ion implantation and chemical or physical vapor deposition) can be used singly or in combination with one another. With respect to heat treatment, processes must be carefully controlled and matched to the particular alloy(s) involved to ensure that undesirable features (e.g., incorrect microstructure, microcracks, etc.) are not introduced during manufacture.[5]

## Summing Up

The presence of NMTPs in the microstructure should act as an early warning system for the heat treater. The presence of NMTPs signals that potential problems may exist in the loading arrangement, the quenching process or the furnace atmosphere and may be being strongly influenced by the part geometry or choice of material. Companies wishing to avoid issues with NMTPs should specify microstructure in addition to mechanical properties and hardness values so as to minimize any performance-related surprises.

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# Tool-Steel Carbides

Today, tool-steel heat treatment is based on a simple premise. To obtain the optimum performance from any given grade, every step of the heat-treating process must be precisely controlled — from stress relief and preheating to austenitizing temperature and time, quenching, deep freeze and tempering. Process and/or equipment variability cannot be tolerated, which is why vacuum processing (Fig. 1) is a popular choice for tool-steel heat treatment.[1]

The selection of any tool steel depends on a combination of factors including component design, application end-use and performance expectation. For any given application, the goal of heat treating is to develop an optimized microstructure (Fig. 2) to help achieve the proper balance of desired properties (Table 1), namely hot (red) hardness, wear resistance, deep hardening and/or toughness.

## Carbide Influence[3]

As we know, tool steels are alloyed with different types of carbide-forming elements including vanadium, tungsten, molybdenum, chromium and, in some alloys, cobalt. The addition of alloying elements serves two basic purposes: to improve hardenability and to provide harder and thermally stable carbides.

Carbides contribute to strengthening of tool steels in two different ways:

1. Carbides provide resistance to (abrasive) wear given that alloy carbides are significantly harder than the matrix material.
2. Carbides contribute to higher yield strengths by impeding the mobility of matrix dislocations.

Carbide-forming elements are, in general, substitutionally dissolved in the crystal lattice (i.e. ferrite or austenite). In other words, when atoms are relatively similar in size, the atom-exchange method is by direct exchange (i.e. substitution). Thus, these elements tend to cause local lattice distortions (since their atomic radii are different from that of iron). However, the total strain is minimized so that less total lattice distortion results. Hence, there is a driving force for diffusion of alloying elements to these sites.[4]

Diffusion of substitutionally dissolved elements is temperature-dependent. At temperatures above approximately 500˚C (930˚F), which is lower than the typical tempering temperatures for most tool


Fig. 1. Typical tool-steel hardening-furnace installation

steels, the diffusion of alloying elements becomes significant, and they will start to develop carbides.[5] The effect of precipitation of alloy carbides is evident, especially in high-speed steels, where precipitation of fine and ultrafine alloy carbides starting at approximately 550˚C (1020˚F) is responsible for the secondary-hardening effect, giving these steels excellent red hardness.[6]

Even in alloys that contain a relatively large amount of strong carbide-forming elements, however, MC-type carbides do not form below approximately 300˚C (570˚F).[5]

It is also noteworthy that alloy carbide grows at the expense of cementite ($Fe_3C \rightarrow$ alloy carbide) either in-situ by nucleation at cementite/ferrite interfaces followed by growth or from dissolution of cementite by separate nucleation and growth at favorable lattice locations.

Finally, caution should be observed because carbides, although beneficial in tool steels for a number of reasons (e.g., strength and wear resistance), are also likely crack-initiation sites in instances where high application stress is involved.

## Carbide Characteristics

### Tungsten and Molybdenum Carbides

Tungsten (W) promotes the formation of $M_6C$-type carbides[7] where the metal (M) is tungsten, iron (Fe), molybdenum (Mo) or a combination. These carbides dissolve in the austenite matrix at temperatures ranging from approximately 1150˚C (2100˚F) to the solidus temperature. However, in practice they do not dissolve completely since time at austenitizing temperature is typically only a matter of a few minutes.

Molybdenum (Mo) promotes formation of $M_2C$-type carbides.[7] These carbides become unstable at elevated temperatures, and they transform to $M_6C$-type carbides by reaction with iron at approximately 750˚C (1380˚F).[7,8]

**Table 1. Effect of tool-steel alloying elements on properties[2]**

| Tool-steel property characteristic | Alloying element (by potency)[a] |
|---|---|
| Hot (red) hardness | W, Mo, Co (with W or Mo), V, Cr, Mn |
| Wear resistance | V, W, Mo, Cr, Mn |
| Deep hardening[b] | Mn, Mo, CR, Si, Ni, V |
| Toughness[c] | V, W, Mo, Mn, Cr |

Notes: a) Distortion is best managed by additions of Mo (with Cr), Cr and Mn. b) Tool must be austenitized at a high enough temperature to dissolve the vanadium carbide. c) Toughness is achieved via grain refinement.

**Table 2. Stages of tempering[13]**

| Stages[a] | Temperature range | Characteristic |
|---|---|---|
| One | 210-480°C<br>(100-250°F) | The lowering of the carbon content of the martensite to approximately 0.25%, and the formation of transition carbides. |
| Two | 390-570°C<br>(200-300°F) | The transformation of retained austenite to ferrite and cementite ($Fe_3C$). |
| Three | 480-660°C<br>(250-350°F) | The replacement of transition carbides and low-temperature martensite by cementite and ferrite. |

Notes: The precipitation of finely dispersed alloy carbides responsible for secondary hardening (in highly alloyed steels) is sometimes referred to as the fourth stage of tempering.

The addition of both tungsten and molybdenum typically results in grain refinement.[9] The fraction of Mo and W bounded as carbides ties up carbon and improves hardenability (by raising $M_s$ and $M_f$ temperatures). The fraction of tungsten and molybdenum in solid solution slows down pearlite and bainite nucleation and growth.

### Vanadium Carbides

Vanadium (V) forms very hard and thermally stable MC-type carbides (typically $M_2C$) as isolated particles. These carbides improve resistance to abrasive wear and provide excellent cutting performance.[7, 9, 10]

Vanadium carbides have highly limited solubility in the lattice matrix. Therefore, addition of vanadium will not delay the rate of diffusional decomposition of austenite. The $M_s$ and $M_f$ temperatures are raised by binding carbon (i.e. forming carbides), thereby improving the hardenability. Adding vanadium also results in grain refinement of the matrix.[9]

### Chromium Carbides

In tool steels, chromium will form carbides – typically type $Cr_{23}C_6$ and in some instances $Cr_7C_3$ – during annealing, depending on the chromium content. These carbides dissolve during austenization at temperatures exceeding approximately 900˚C (1650˚F) and are totally dissolved at approximately 1100˚C (2012˚F).[10]

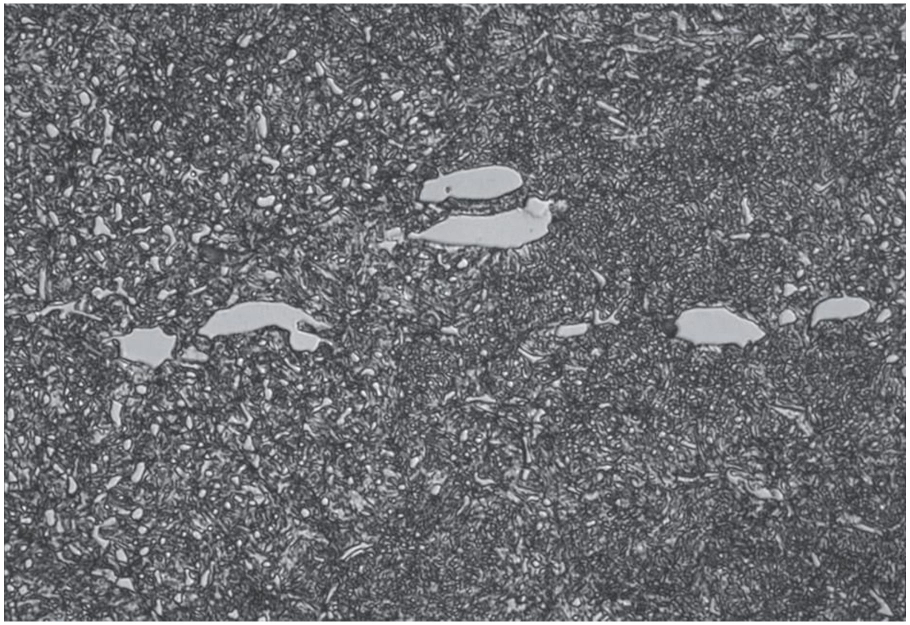

Fig. 2. A2 tool-steel microstructure – 1250X, 2% Nital (Courtesy of Aston Metallurgical Services Company, Inc.)

### Cobalt Carbides

Cobalt raises the temperature at which martensite transformation begins and is complete. Cobalt is the only alloying element in high-speed steels, for example, that can appreciably increase the thermal stability up to approximately 650˚C (1200˚F) and secondary hardness up to 67-70 HRC[8] at the expense of toughness and wear resistance.[9]

The addition of cobalt also causes the solidus temperature to rise. It is possible to dissolve a larger percentage of cobalt carbides during austenization, which enhances hardenability. The high austenization temperature results in a relatively large amount of retained austenite after quenching, but this effect is somewhat compensated for by a lower stability of the austenite owing to the presence of cobalt.[10,11]

## Tempering[12]

During the heating portion of the tempering cycle, plain-, medium- and high-carbon steels experience three principal transformation stages (Table 2): namely precipitation of eta carbides, transformation of retained austenite into lower bainite and precipitation of cementite. In tool steels containing secondary alloying elements (e.g., V, Mo, W), a fourth transformation is said to occur, namely precipitation of MC and MC-type alloy carbides that nucleate independently.[15-18]

During this first transformation stage, metastable carbide ($Fe_{2,4}C$) having a hexagonal crystal structure precipitates from the supersaturated martensite.[19,20]

Precipitation of these highly dispersed carbides is reported to enhance strengthening in these steels. However, low-carbon martensite causes only a modest drop in strength and hardness of steel tempered in that temperature range.[16,18]

During the second stage, a non-homogeneous mixture of supersaturated ferrite and cementite (lower bainite) forms. It should be noted that this transformation occurs only in steels containing more than 0.3% C because the amount of the austenite remaining in the steel after quenching strongly depends on carbon content.[15,16,18]

Cementite is formed during the third stage, resulting in further lowering of the carbon content of the matrix and dissolution of metastable carbides, which allows recovery to occur.[18,19]

Above approximately 400˚C (750˚F), diffusion of alloying elements (e.g., V, Mo, W) begins, the cementite gradually dissolves and nucleation of MC and M2C carbides is possible. This leads to an increase of hardness of tempered steel and is referred to as secondary hardening.

## Concluding Thoughts

Understanding the role of carbides and carbide dissolution in tool steels is important to ensure optimal metallurgical characteristics are achieved so that optimum tool performance results.

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# Lightweight Materials for the Transportation Industry

Several newsworthy items crossed The Doctor's desk late last year: the announcement that Alcoa officially opened the world's largest aluminum-lithium plant in Lafayette, Ind., (IHDaily NewsBrief, Oct. 15); the release of The Aluminum Quick Reference App (ASM International, Oct. 16); and word that Alcoa has signed a $1 billion contract with Boeing to supply multiple aluminum components over the next several years (Oct. 20).

These announcements peaked The Doctor's interest in learning more about the battle being waged between aluminum and alternative materials in the transportation industry. To understand who will win and why, one must look at the drivers this industry faces to stay technologically relevant as well as the lightweight material options, properties and costs. Let's learn more.

## Automotive

Fuel consumption, greenhouse gas (GHG) emissions and global dimming/warming are the principle drivers for change in the automotive industry. For every 100 kg of weight reduction in an automobile, a fuel savings of 0.3-0.5 L per 100 kilometers is possible with a reduction in $CO_2$ emissions of 0.8-1.1 kg per 100 kilometers.

The interest in moving to aluminum and other lightweight alloys in automobiles is fueled in large part by government regulations/mandates, cost (performance versus cost in dollars saved per kilogram), safety (i.e., How light should we go?) and performance (acceleration, braking, handling, noise, vibration and harshness). Other options

Fig. 1. Corvette Z06 showcasing the use of lightweight alloys

**Table 1. Aerospace aluminum alloys**

| Alloy series | Alloy | Properties | Applications |
|---|---|---|---|
| 1xxx | Pure | Low strength, excellent thermal/electrical conductivity and corrosion resistance, high reflectivity | Fuel filters, electrical conductors, radiator tubing, lighting reflectors, decorative components |
| 2xxx | Cu | High strength, relatively low corrosion resistance, good elevated-temperature strength | Aircraft skin, fittings, wheels, ballistic armor, forged and machined components |
| 3xxx | Mn | Medium strength, good formability, good corrosion resistance | Storage tanks, heat exchangers, pressure vessels |
| 4xxx | Si | High castability, high machinability, high fluidity, low ductility | Large housing castings |
| 5xxx | Mg | Medium strength, good formability, excellent marine corrosion resistance | Interior trim, pressure vessels, armor plate, marine and cryogenic components |
| 6xxx | Mg, Si | Medium-to-high strength, good corrosion resistance, easily extruded | Piping, marine screw stock, door and window frames, exterior trim |
| 7xxx | Zn | Very high strength, prone to stress corrosion, poor corrosion resistance | Aircraft construction, armor plate |
| 8xxx | Li | Very high strength, low density | Aircraft and aerospace structures, foil, heat-exchanger fin stock |

are conventional low-carbon steel, with a cost advantage while targeting weight reduction; and HSLA steels and AHSS (advanced high-strength steels, or so-called “lightweight steel”), where weight reduction is achieved by thinner/lighter material made possible by higher strength.

Aluminum advantages are in the total amount of aluminum used per vehicle, life-cycle/recycling advantages, space frame versus monocoque (i.e., skin or “egg shell”), loading and forming/joining (e.g., the absence of spot welds, use of rivets, etc.).

An example is the Corvette Z06 (Fig. 1), which is ranked as *Car & Driver’s* number-one performance-ranked coupe and convertible. It can achieve 650 horsepower in a choice of two transmissions: a 7-speed manual or an 8-speed paddle-shift automatic. The driver-centric Z06 includes technologies like a Driver Mode Selector, which allows for customization of vehicle performance dynamics with the turn of a knob. On or off the track it is available with an industry-exclusive Performance Data Recorder, which records high-definition video with telemetry overlays on playback.

Another example is the number-one selling truck in the U.S., the Ford F150, which has undergone a dramatic redesign taking advantage of the best of steel and lightweight alloys. Aluminum alloy 6463 is used in the body and bed to reduce the total vehicle weight by 320 kg (705 pounds).

BMW has introduced an Al-Mg die-cast engine block, which is 15% lighter than a comparable all-Al alloy engine. Both BMW and GM have an all Mg block (Fig. 2) under development that is 25% lighter than an all-Al engine.

## Aerospace

Aluminum has a long history in aviation (Table 1). Fuel consumption, GHG emissions sustainability and global dimming/warming are also the principle change drivers in the aerospace industry. Aluminum's dominance is being challenged today by GFRPs (graphite-fiber reinforced plastics), carbon/carbon composites and aluminum-lithium alloys.

For example, the Orion spacecraft (Fig. 3) has olive-green aluminum-lithium metal panels designed to be covered by an advanced version of the thermal protection tiles that were earlier used on the space shuttle. Design challenges such as pressure testing cracks during weld strength testing have yet to be fully addressed.

Fig. 2. All-magnesium, six-cylinder engine block developed by the DOE with GM's R&D team

Fig. 3. Astronaut compartment on the Orion spacecraft

Alloying with lithium reduces structural mass by three effects:

- Displacement: A lithium atom is lighter than an aluminum atom. Each lithium atom then displaces one aluminum atom from the crystal lattice while maintaining the lattice structure. For every 1% (by weight) of lithium added, the density of the resulting alloy is reduced by 3% and increases in stiffness by 5%. This effect works up to the solubility limit of lithium in aluminum, which is 4.2%.
- Strain hardening: Introducing another type of atom into the crystal strains the lattice, which helps block dislocations. The resulting material is stronger, which allows less of it to be used.

- Precipitation hardening: When properly aged, lithium forms a metastable $Al_3Li$ phase ($\delta$') with a coherent crystal structure. Precipitates strengthen the metal by impeding dislocation motion during deformation.

Finally, all-aluminum combat ships have been introduced and are designed to be fast, maneuverable and modular – or, as they like to say in the Navy, lethal, flexible, survivable and affordable. Aluminum in the 5xxx series (e.g., 5086, 5083, 5456, 5454) and 6xxx series alloys (e.g., 6005, 6061, 6082) are in use. The properties and attributes of aluminum that make it an attractive choice are its high strength-to-weight ratio; density (one third of steel); corrosion resistance (although pitting and crevice-corrosion problems have arisen); weldability; ease of forming, bending and machining; availability and diversity of semi-finished products; high thermal and electrical conductivity; nonmagnetic properties; and recyclability.

## Conclusion

What stands out in this discussion is that the materials are changing and with them our role as heat treaters. Moving forward, changes in the alloys, product form, function and specifications will influence (and perhaps dictate) the way in which we will perform heat treatment.

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# Understanding Aluminum Heat Treatment

The heat treatment of aluminum and aluminum alloys are precision processes, which must be carried out in furnaces and ovens properly designed to provide very precise thermal conditions and equipped with adequate control instruments to ensure required uniformity and repeatability of temperature-time cycles. Heat-treat process details must be carefully established, controlled and documented for each type of product to ensure that required properties are achieved. Let's learn more.

## Types of Heat Treatment

Heat treatments applied to aluminum and its alloys are:

- Preheating or homogenizing to reduce chemical segregation of cast structures and to improve material workability
- Annealing to soften strain-hardened (work-hardened) and heat-treated alloy components, to relieve stresses and to stabilize properties and dimensions
- Solution heat treatment to improve mechanical properties by putting alloying elements into solution
- Precipitation (age-hardening) heat treatment to provide hardening by precipitation of constituents from solid solution

Representative drop bottom oven used for solution treating/quenching aluminum. (Courtesy of Wisconsin Oven Corp., East Troy, Wis.)

Loading aluminum castings into aging oven. (Courtesy of Wisconsin Oven Corp., East Troy, Wis.)

## Homogenization (ingot preheating treatment)

The initial thermal operation applied to castings and ingots (prior to hot working) is referred to as homogenization, which has one or more purposes depending on the alloy, product and fabricating process involved. One of the principal objectives is improved workability since the microstructure of most alloys in the as-cast condition is quite heterogeneous. This is true for alloys that form solid solutions under equilibrium conditions and even for relatively dilute alloys.

## Annealing

Annealing can be used for both heat treatable and non-heat treatable alloys to increase ductility with a slight reduction in strength. There are several types of annealing treatments dependent to a large part on the alloy type, initial structure and temper condition. In annealing, it is important to ensure that the proper temperature is reached in all locations of the load. The maximum annealing temperature is also important in achieving good results.

Full annealing (temper designation “O”) produces the softest, most ductile and most versatile condition. It is performed for both heat treatable and non-heat treatable aluminum alloys. The rate of softening is strongly temperature dependent; that is, the time required can vary from a few hours at low temperature to a few seconds at high temperature.

Other forms of annealing include stress relief annealing to remove the effects of strain hardening in cold worked alloys, partial annealing (or recovery annealing) done on non-heat treatable wrought alloys to obtain intermediate mechanical properties, and recrystallization characterized by the gradual formation and appearance of a microscopically resolvable grain structure.

## Precipitation Hardening

Heat-treatable aluminum alloys contain amounts of soluble alloying elements that exceed the equilibrium solid solubility limit at room (and moderately higher) temperature. The amount present may be less or more than the maximum that is soluble at the eutectic temperature.

In most precipitation hardenable systems, a complex sequence of time-dependent and temperature-dependent changes is involved. The relative rates at which solution and precipitation reactions occur with different solutes depend upon the respective diffusion rates, in addition to solubility and alloy contents.

## Solution Heat Treating

The purpose of solution heat treatment is the dissolution of the maximum amount of soluble elements in the alloy into solid solution. The process consists of heating and holding the alloy at a temperature sufficiently high and for a long enough period of time to achieve a nearly homogenous solid solution in which all phases have dissolved.

Care must be taken to avoid overheating or under-heating. In the case of overheating, eutectic melting can occur with a corresponding degradation of properties such as tensile strength, ductility and fracture toughness. If under-heated, solution is incomplete, and strength values lower than normal can be expected. In certain cases, extreme property loss can occur.

In general, a temperature variation of ±10°F (±6°C) from control set point is allowable, but certain alloys require even tighter tolerances. The time at temperature is a function of the section thickness of the material and may vary from several minutes to many hours. The time required to heat a load to the treatment temperature also increases with section thickness and loading arrangement and, thus, the total cycle time must take these factors into consideration.

## Quenching

Rapid, uninterrupted quenching in water or polymer is, in most instances, required to avoid precipitation detrimental to mechanical properties and corrosion resistance. The solid solution formed by solution heat treatment must be cooled rapidly enough to produce a supersaturated solution at room temperature, which provides the optimal condition for subsequent age (precipitation) hardening.

Quenching is in many ways the most critical step in the sequence of heat treating operations. The objective of quenching is to preserve as nearly intact as possible the solid solution formed at the solution heat treating temperature by rapidly cooling to some lower temperature, usually near room temperature.

Water and polymer quenchants are the most widely used quenching media. In immersion quenching, cooling rates can be reduced by increasing temperature. Conditions that increase the stability of a vapor film around the part decrease the cooling rate.

## Aging (age hardening)

Age hardening is achieved either at room temperature (natural aging) or with a precipitation heat treatment (artificial aging) cycle. The same general rules used in solution heat treatment (temperature uniformity, time at temperature) apply for precipitation hardening.

Aging at room temperature (natural aging). Most of the heat-treatable alloys exhibit age hardening at room temperature after quenching, the rate and extent varying from one alloy to another. Since the alloys are softer and more ductile immediately

**Process matrix**

| Process | Solution heat treating | Quenching | Aging (age hardening) |
|---|---|---|---|
| Goal | Take into solid solution the maximum practical amount of soluble hardening elements (e.g., Cu, Si, Mg) in the alloy | Preserve as much as possible the solid solution formed at elevated (solution-treating) temperatures | Cause precipitation to occur (the degree of stable equilibrium for a given grade is a function of time and temperature) |
| Purpose | It is necessary to produce a solid solution to take advantage of the precipitation hardening reaction (aging) | Cool rapidly enough to produce a supersaturated solution | Create a change in which the structure recovers from an unstable or "meta" stable condition (produced by quenching or cold working) |

after quenching than after aging, straightening and forming operations are normally performed in the as-quenched condition. The process window for forming after quenching can be enlarged by keeping the alloy refrigerated prior to forming.

Aging at elevated temperature (artificial aging). The effects of precipitation on mechanical properties are greatly accelerated (and usually accentuated) by reheating the quenched material to a temperature range of about 212 to 424°F (100 to 200°C). A characteristic feature of elevated-temperature aging effects on tensile properties is that the increase in yield strength is more pronounced than the increase in tensile strength. Also, ductility, as measured by percentage elongation, decreases. Thus, an alloy in the T6 temper has higher strength but lower ductility than the same alloy in the T4 temper.

In certain alloys, precipitation heat treating can occur without prior solution heat treatment, because some alloys are relatively insensitive to cooling rate during quenching, thus they can be either air cooled or water quenched. In either condition, these alloys will respond strongly to precipitation heat treatment.

In general, the principles and procedures for heat treating wrought and cast aluminum alloys are similar. However, for cast alloys, soak times and quenching media are often different due to the section sizes involved. Typically, soak times are longer and quenchants such as boiling water are used to reduce quenching stresses in complex shapes.

# Aluminum Heat Treatment Revisited: Common Concerns and How to Resolve Them

When heat treating aluminum and aluminum alloys, it is important to understand the challenges we face and why absolute control of process and equipment variability is so very critical. To aid the heat treater in this regard, The Doctor has gathered together in one place the most commonly reported process problems along with some recommendations on how to resolve them. Let's learn more.

## Heat-Treat-Related Issues

The most commonly reported problems with heat treatment of aluminum include concerns over:

- Improper racking of parts – This results in distortion due in large part to the inability of the quenchant to extract the heat at a sufficient rate to achieve the desired mechanical properties. Racking may also create thermally induced distortion (since the creep strength of aluminum is poor). Proper racking (Fig. 1) avoids these issues.
- Excessive heating/ramp rates – These contribute to thermal distortion and should be avoided. Proper racking of parts helps uniform heating.
- Higher-than-anticipated residual-stress levels – Heat treatment not only affects mechanical properties but also directly influences residual-stress levels. Some of the various causes are differential cooling during quenching between surfaces and interior regions (including post-solidification cooling of castings), ramp rates and changes in temperature at intermediate steps. Residual stress is a function of (large) differential cooling rates, section thickness, abrupt changes in section size and material strength. It is important to understand that stresses induced by quenching are many times more pronounced than stresses from other types of processes (including casting).
- Variation in time/temperature/quench parameters – These ultimately result in changes to mechanical and/or physical properties, both part-to-part and load-to-load. These include slow part transfer times, improper (slow) quench, overheating, underheating or changes in time-temperature parameters during precipitation hardening. For example, larger

Fig. 1. Proper part racking

particles or precipitates result from longer times and higher temperatures.

- Overheating – The concern is incipient or eutectic melting. For example, solution heat treatment involves temperatures close to the melting point of many aluminum alloys (especially the 2xxx series, often only a few hundred degrees below their melting point). Proper temperature is needed to promote dissolution in the solid state of the alloying elements present.

- Underheating – This results in loss of mechanical properties due to inadequate supersaturation. When the aging temperature is low and/or the aging time is too short, solute atom gathering zones (GP zones) are not easily formed, which leads to low strength after aging.
- Inadequate quenching resulting in distortion – The problem/challenge is the movement of part(s) into the quench, especially when manual quenching is critical. Parts must enter smoothly. (The heat-treat shop term is that you want to avoid "slapping" the quench with the part.) Uniform heat transfer across the part avoids differential cooling and differential thermal strain. Horizontal changes in heat transfer are often more insidious than vertical ones. Keeping the quenchant at the proper temperature, controlling its rate of rise, ensuring uniform flow and optimizing the process for the chosen quenchant (e.g., air, water, polymer) are critical. For example, the cooling rate of polymers can be varied to suit a specific application by changing concentration, temperature and the degree of agitation, which affects both uniformity of heat transfer and quench rate during the nucleate boiling stage. Maintenance of the quenchant is also important. Parts of complex shapes such as forgings, castings, impact extrusions and components produced from sheet metal may be quenched at slower quenching rates to improve distortion characteristics.
- Surface blistering/high-temperature oxidation – These conditions are best described in The Heat Treat Doctor column "High Temperature Oxidation – A Case Study," Industrial Heating, February 2016.
- Overaging – This may result in loss of mechanical properties. When the aging temperature is high and/or the aging time is long, the critical nucleus size of the phase precipitated from the supersaturated solid solution can be increased, resulting in low strength properties after aging.
- Underaging – This may also result in loss of mechanical properties.
- Improper natural aging – Times may vary from around five days for the 2xxx series alloys to around 30 days for other alloys. The 6xxx and 7xxx series alloys are considerably less stable at room temperature and continue to exhibit changes in mechanical properties for many years. With some alloys, natural aging may be suppressed or delayed for several days by refrigeration at -18°C (-1˚F) or lower. It is common practice to complete forming, straightening and coining before aging changes the material properties appreciably. For example,

conventional practice allows for refrigeration of 2014-T4 rivets to maintain good driving characteristics.
- Improper artificial aging – Artificial aging (aka precipitation heat treatment) is a lengthy, low-temperature process. Temperature control is critical along with maintaining a minimum temperature uniformity of ±6˚C (±10˚F). The targeted uniformity should be ±4˚C (±7˚F).
- Inadequate time at temperature – The consequence is failure to achieve mechanical properties. Too short a time will result in inadequate supersaturation; too long a time often manifests itself in part distortion.
- Inadequate temperature uniformity – This may result in an inability to achieve or may alter mechanical properties. The typical process temperature uniformity requirement is ±6˚C (±10˚F), with ±3˚C (±5˚F) preferred for most aerospace applications.
- Improper cold working after solution treating – This often results from a lack of understanding of the response of the alloy in question. For example, cold working of 2xxx series alloys in the as-quenched condition greatly increases its response to later precipitation treatment.
- Inadequate cooling rate if annealing solution-heat-treated product – A maximum cooling rate of 20˚C (40˚F) per hour must be maintained until the temperature is reduced to 290˚C (555˚F). The cooling rate is unimportant below this temperature.

## Casting-Related Issues

It should be mentioned in passing that there are a number of ingot defects in the mill state that can influence subsequent heat treatment and mechanical properties. Some of these include:
- Porosity/midline porosity – due to lack of mass feeding, hydrogen segregation or surface oxide layers (often due to air pockets)
- Inclusions – casting impurities (due to grain refiners or air pockets) in the form of carbides, borides and oxides to name a few
- Macro or microsegregation – inhomogeneous solute composition and hard intermetallic and second-phase particles. Proper homogenization helps negate this concern.
- Deformation/shrinkage – due to stress/strain induced by cooling conditions
- Hot tears – due primarily to feeding issues
- Issues related to rolling (sheet or plate) or stretching (extrusion, bar and plate) to produce higher mechanical properties. If the higher properties

are required, however, reheat treatment should be avoided.

Other related issues can be found in the online version.

## Summary

The solution to most aluminum heat-treatment-related problems is to understand what can go wrong; establish good practices and procedures; be consistent (and repeatable) in the execution of these procedures; monitor the process in as close to real-time as possible; and maintain furnace records and time-temperature charts to confirm that the intended practices were indeed performed.

Finally, be sure that the testing methods used to confirm that the parts are good are robust and adequate to ensure proper performance in the field. Heat treaters have heard this all before, but nowhere is it more critical than in the heat treatment of aluminum and aluminum alloys.

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# Temper Designations for Aluminum Alloys: What They Are and Why We Need to Know

The properties of aluminum alloys depend on a combination of chemical composition and response to thermal and/or mechanical treatments (i.e. cold work), so it is important to have a designation system that identifies the exact state or condition of the material at any point in its manufacture. Temper designations for aluminum alloys do just that but are often the subject of angst among heat treaters. It's time to clear up any confusion. Let's learn more.

In order to better understand temper designations, it is important to recall that there are two categories of aluminum: heat-treatable and non-heat-treatable alloys. For example, the 1xxx, 3xxx and 5xxx series wrought aluminum alloys cannot be hardened by heat treatment. These non-heat-treatable alloys produce optimum mechanical properties through strain hardening, that is, through the application of cold working.

By contrast, the 2xxx, 6xxx and 7xxx series wrought aluminum alloys are heat treatable, while the 4xxx series consist of both heat-treatable and non-heat-treatable alloys. For cast alloys, the 2xx.x, 3xx.x, 4xx.x and 7xx.x series cast alloys are heat treatable (note: mechanical-hardening methods are not generally applied to castings). Recall that in previous

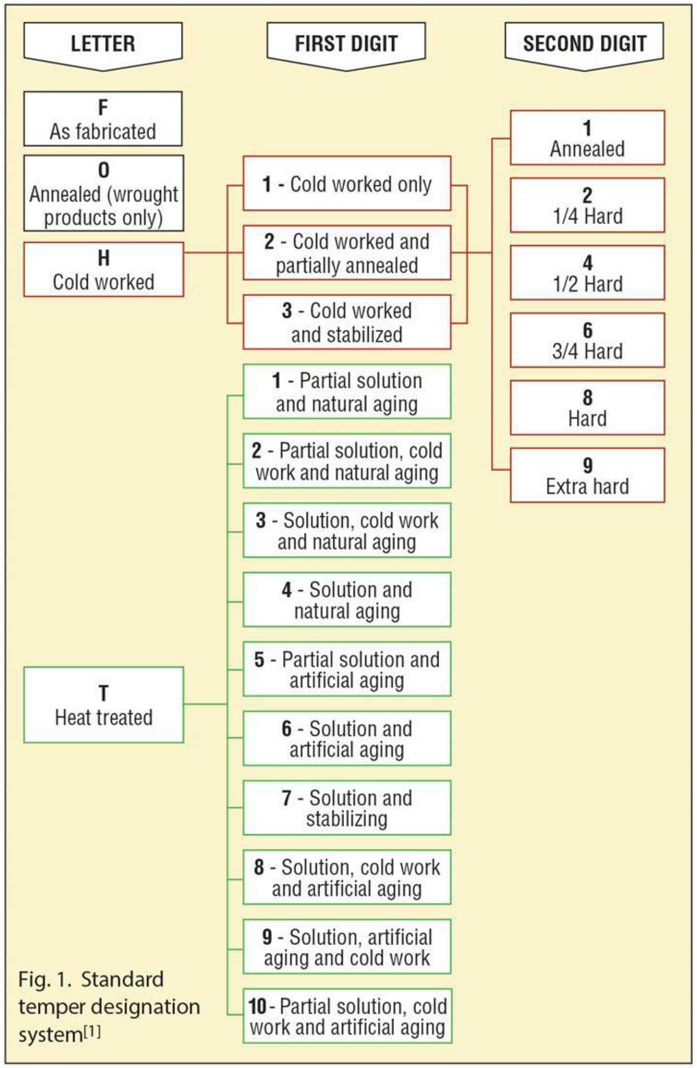

Fig. 1. Standard temper designation system[1]

Heat Treat Doctor columns we have talked about how to achieve optimum mechanical properties through heat treatment (c.f. "Understanding Aluminum Heat Treatment," Industrial Heating, February 2006 and "Heating Treating of Aluminum Castings," Industrial Heating, February 2010).

The temper designation system is used for all forms of wrought and cast aluminum and aluminum alloys except ingots and is simply an extension of their alloy numbering system, which consists of a series of letters and numbers (Fig. 1) that follow the alloy designation number being separated by a hyphen (e.g., 6061-T6). Should some other variation of the same sequence of basic operations be applied to the same alloy, which results in different characteristics, then additional digits are added to the designation (see below).

## Basic Temper Designations

Table 1 contains a list of the most common temper designations.

## Common Subdivisions

Table 2 contains a list of some of the most common subdivisions of the basic tempers shown in Table 1. The online version of this article (www.industrialheating.com) contains a more complete listing.

**Table 1. Basic temper designations**[3]

| Letter Code | Meaning | Description |
|---|---|---|
| F | As fabricated | Applies to the products of shaping processes in which no special control over thermal conditions or strain hardening is employed. For wrought products, there are no mechanical-property limits. |
| H | Strain hardened | Applies to wrought products that only have their strength increased by strain hardening (with or without supplementary thermal treatments to produce some reduction in strength). Two or more digits always follow the H. |
| O | Annealed | Applies to wrought products that are annealed to obtain the lowest strength and to cast products that are annealed to improve ductility and dimensional stability. A digit other than zero may follow the O. |
| T | Thermally treated | Applies to products that are thermally treated, with or without supplementary strain hardening, to produce stable tempers other than F, O or H. One or more digits always follow the T. |
| W | Solution heat treated | An unstable temper condition applicable only to alloys that spontaneously age at room temperature after solution heat treatment. This designation is specific only when the period of natural aging is indicated after the letter code (for example, W 1/2 hour) |

**Table 2. Common subdivisions – T temper[3]**

| Letter Code [a], [b] | Meaning | Description |
|---|---|---|
| T1 | Cooled from an elevated-temperature shaping process and naturally aged to a substantially stable condition | Applies to products that are not cold worked after cooling from an elevated-temperature shaping process or in which the effect of cold work in flattening or straightening may not be recognized in mechanical-property limits. |
| T2 | Cooled from an elevated-temperature shaping process, cold worked and naturally aged to a substantially stable condition | Applies to products that are cold worked to improve strength after cooling from an elevated-temperature shaping process or in which the effect of cold work in flattening or straightening is recognized in mechanical-property limits. |
| T3 | Solution heat treated, cold worked and naturally aged to a substantially stable condition | Applies to products that are cold worked to improve strength after solution heat treatment or in which the effect of cold work in flattening or straightening is recognized in mechanical-property limits. |
| T4 | Solution heat treated and naturally aged to a substantially stable condition | Applies to products that are not cold worked after solution heat treatment or in which the effect of cold work in flattening or straightening may not be recognized in mechanical-property limits. |
| T5 | Cooled from an elevated-temperature shaping process and artificially aged | Applies to products that are not cold worked after cooling from an elevated-temperature shaping process or in which the effect of cold work in flattening or straightening may not be recognized in mechanical-property limits. |
| T6 | Solution heat treated and artificially aged | Applies to products that are not cold worked after solution heat treatment or in which the effect of cold work in flattening or straightening may not be recognized in mechanical-property limits. |
| T7 | Solution heat treated and overaged/stabilized | Applies to wrought products that are artificially aged after solution heat treatment to carry them beyond a point of maximum strength to provide control of some significant characteristic. Applies to cast products that are artificially aged after solution heat treatment to provide dimensional and strength stability. |
| T8 | Solution heat treated, cold worked and artificially aged | Applies to products that are cold worked to improve strength or in which the effect of cold work in flattening or straightening is recognized in mechanical-property limits. |
| T9 | Solution heat treated, artificially aged and cold worked | Applies to products that are cold worked to improve strength. |
| T10 | Cooled from an elevated-temperature shaping process, cold worked and artificially aged | Applies to products that are cold worked to improve strength or in which the effect of cold work in flattening or straightening is recognized in mechanical-property limits. |

**Notes:** [a] Numerals 1 through 10 following the T indicate specific sequences of so-called basic treatments. [b] Additional digits, the first of which shall not be zero, may be added to designations T1 through T10 to indicate a variation in treatment, which significantly alters the product characteristics that are or would be obtained using the basic treatment.

**Table 3. Common subdivisions – O temper[3]**

| Letter Code | Meaning | Description |
|---|---|---|
| O1 | Thermally treated at approximately same time and temperature required for solution heat treatment and slow cooled to room temperature. | Applies to products that are to be machined or formed prior to solution heat treatment by the user. Mechanical-property limits are not applicable. |

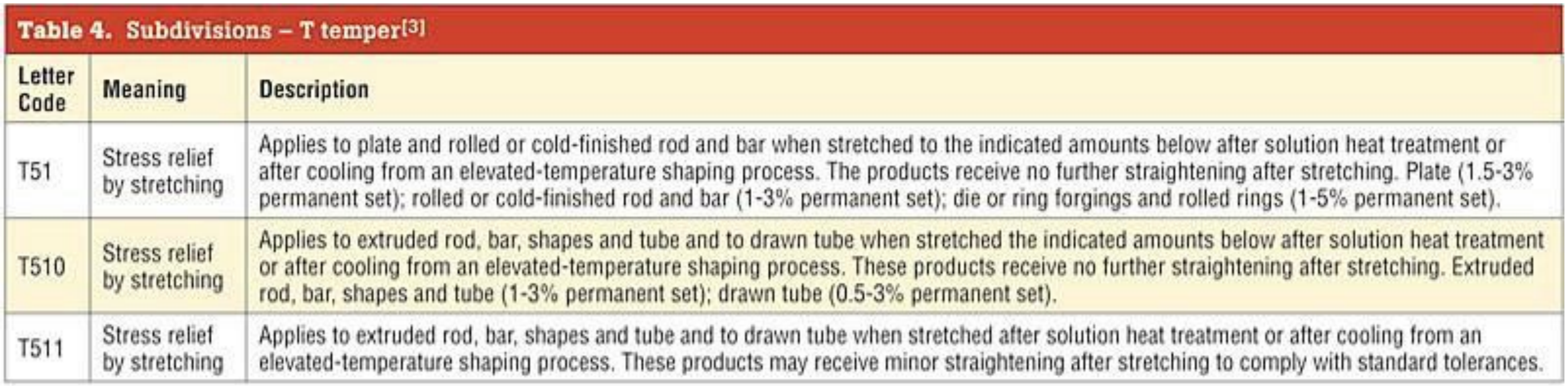

**Table 4. Subdivisions – T temper[3]**

| Letter Code | Meaning | Description |
|---|---|---|
| T51 | Stress relief by stretching | Applies to plate and rolled or cold-finished rod and bar when stretched to the indicated amounts below after solution heat treatment or after cooling from an elevated-temperature shaping process. The products receive no further straightening after stretching. Plate (1.5-3% permanent set); rolled or cold-finished rod and bar (1-3% permanent set); die or ring forgings and rolled rings (1-5% permanent set). |
| T510 | Stress relief by stretching | Applies to extruded rod, bar, shapes and tube and to drawn tube when stretched the indicated amounts below after solution heat treatment or after cooling from an elevated-temperature shaping process. These products receive no further straightening after stretching. Extruded rod, bar, shapes and tube (1-3% permanent set); drawn tube (0.5-3% permanent set). |
| T511 | Stress relief by stretching | Applies to extruded rod, bar, shapes and tube and to drawn tube when stretched after solution heat treatment or after cooling from an elevated-temperature shaping process. These products may receive minor straightening after stretching to comply with standard tolerances. |

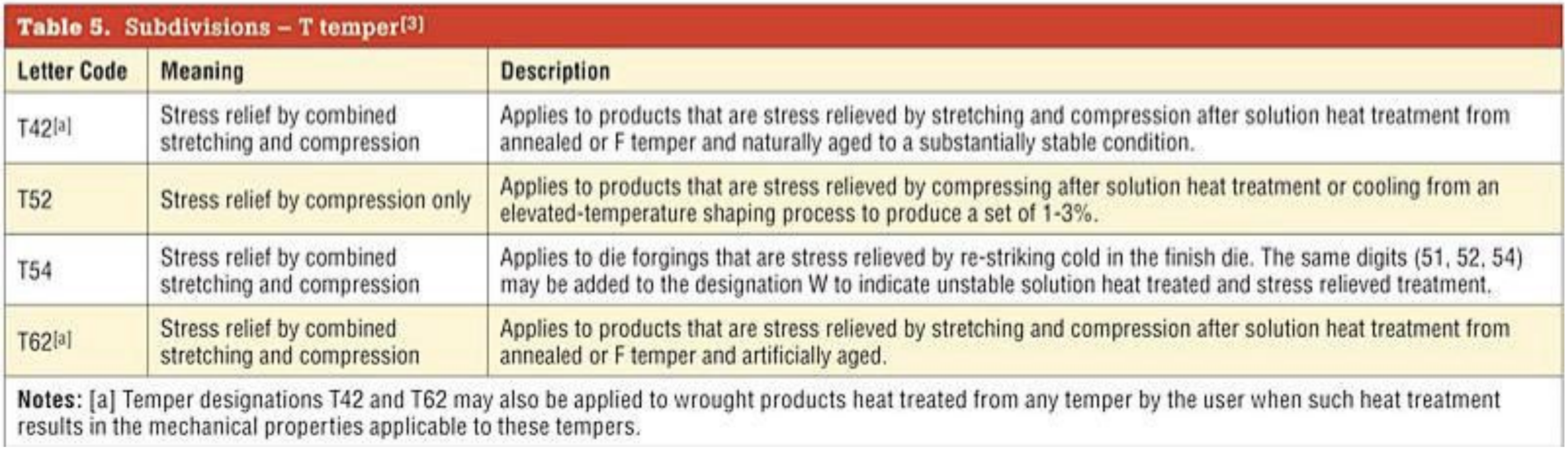

**Table 5. Subdivisions – T temper[3]**

| Letter Code | Meaning | Description |
|---|---|---|
| T42[a] | Stress relief by combined stretching and compression | Applies to products that are stress relieved by stretching and compression after solution heat treatment from annealed or F temper and naturally aged to a substantially stable condition. |
| T52 | Stress relief by compression only | Applies to products that are stress relieved by compressing after solution heat treatment or cooling from an elevated-temperature shaping process to produce a set of 1-3%. |
| T54 | Stress relief by combined stretching and compression | Applies to die forgings that are stress relieved by re-striking cold in the finish die. The same digits (51, 52, 54) may be added to the designation W to indicate unstable solution heat treated and stress relieved treatment. |
| T62[a] | Stress relief by combined stretching and compression | Applies to products that are stress relieved by stretching and compression after solution heat treatment from annealed or F temper and artificially aged. |

**Notes:** [a] Temper designations T42 and T62 may also be applied to wrought products heat treated from any temper by the user when such heat treatment results in the mechanical properties applicable to these tempers.

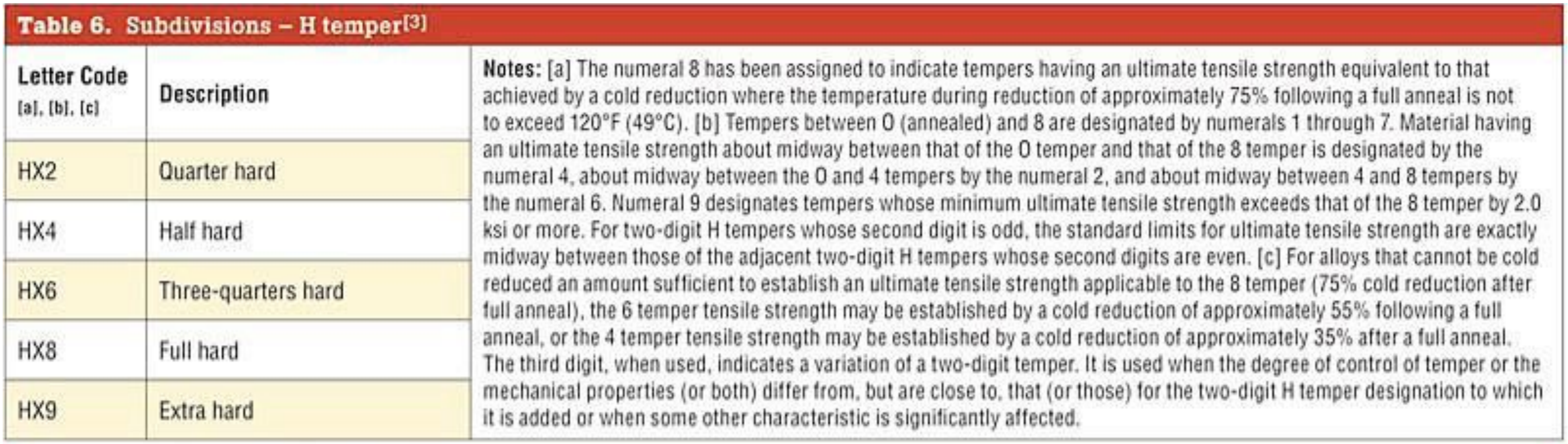

**Table 6. Subdivisions – H temper[3]**

| Letter Code [a], [b], [c] | Description |
|---|---|
| HX2 | Quarter hard |
| HX4 | Half hard |
| HX6 | Three-quarters hard |
| HX8 | Full hard |
| HX9 | Extra hard |

**Notes:** [a] The numeral 8 has been assigned to indicate tempers having an ultimate tensile strength equivalent to that achieved by a cold reduction where the temperature during reduction of approximately 75% following a full anneal is not to exceed 120°F (49°C). [b] Tempers between O (annealed) and 8 are designated by numerals 1 through 7. Material having an ultimate tensile strength about midway between that of the O temper and that of the 8 temper is designated by the numeral 4, about midway between the O and 4 tempers by the numeral 2, and about midway between 4 and 8 tempers by the numeral 6. Numeral 9 designates tempers whose minimum ultimate tensile strength exceeds that of the 8 temper by 2.0 ksi or more. For two-digit H tempers whose second digit is odd, the standard limits for ultimate tensile strength are exactly midway between those of the adjacent two-digit H tempers whose second digits are even. [c] For alloys that cannot be cold reduced an amount sufficient to establish an ultimate tensile strength applicable to the 8 temper (75% cold reduction after full anneal), the 6 temper tensile strength may be established by a cold reduction of approximately 55% following a full anneal, or the 4 temper tensile strength may be established by a cold reduction of approximately 35% after a full anneal. The third digit, when used, indicates a variation of a two-digit temper. It is used when the degree of control of temper or the mechanical properties (or both) differ from, but are close to, that (or those) for the two-digit H temper designation to which it is added or when some other characteristic is significantly affected.

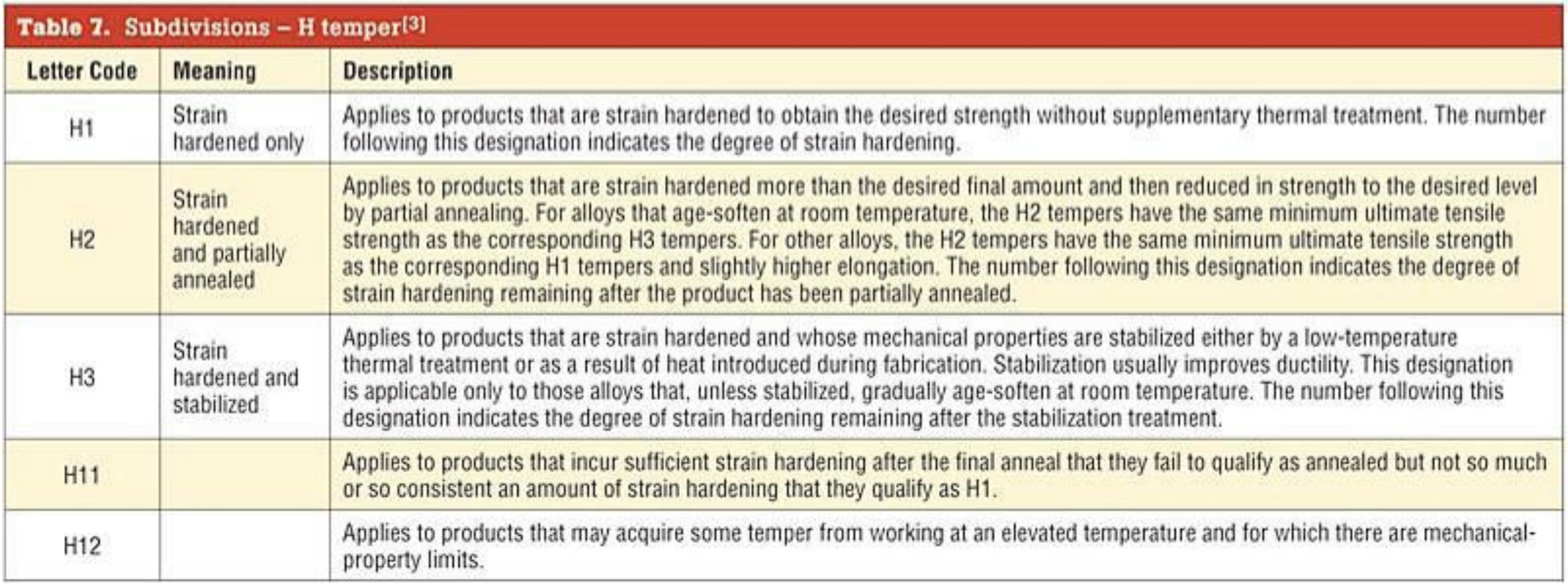

**Table 7. Subdivisions – H temper[3]**

| Letter Code | Meaning | Description |
|---|---|---|
| H1 | Strain hardened only | Applies to products that are strain hardened to obtain the desired strength without supplementary thermal treatment. The number following this designation indicates the degree of strain hardening. |
| H2 | Strain hardened and partially annealed | Applies to products that are strain hardened more than the desired final amount and then reduced in strength to the desired level by partial annealing. For alloys that age-soften at room temperature, the H2 tempers have the same minimum ultimate tensile strength as the corresponding H3 tempers. For other alloys, the H2 tempers have the same minimum ultimate tensile strength as the corresponding H1 tempers and slightly higher elongation. The number following this designation indicates the degree of strain hardening remaining after the product has been partially annealed. |
| H3 | Strain hardened and stabilized | Applies to products that are strain hardened and whose mechanical properties are stabilized either by a low-temperature thermal treatment or as a result of heat introduced during fabrication. Stabilization usually improves ductility. This designation is applicable only to those alloys that, unless stabilized, gradually age-soften at room temperature. The number following this designation indicates the degree of strain hardening remaining after the stabilization treatment. |
| H11 | | Applies to products that incur sufficient strain hardening after the final anneal that they fail to qualify as annealed but not so much or so consistent an amount of strain hardening that they qualify as H1. |
| H12 | | Applies to products that may acquire some temper from working at an elevated temperature and for which there are mechanical-property limits. |

## Temper Variations (O)

A digit following the O, when used, indicates a product in the annealed condition has special characteristics (Table 3). It should be noted that the O temper is not part of the strain-hardened (H) series. Variations of O tempers do not apply to products that are strain hardened after annealing and in which the effect of strain hardening is recognized in the mechanical properties or other characteristics.

## Unregistered Tempers

The letter P has been assigned to denote H, T and O temper variations that are negotiated between manufacturer and purchaser. The letter P immediately follows the temper designation that most nearly applies.

## Stress-Relieved Tempers

The following specific additional digits have been assigned for stress-relieved tempers of wrought products.

### Stress Relief by Stretching

Table 4 contains this information.

### Stress Relief by Compression or Combination of Stretching and Compression

See Table 5 for this data.

## Strain-Hardened Tempers

The digit following the designations H1, H2 and H3 indicates the degree of strain hardening (Table 6). Table 7 provides additional strain-hardened temper designations.

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# Tempus Fugit – Optimizing Process Time for Aluminum Alloys

The aluminum heat-treatment industry is an exciting place to work these days, as demonstrated by ongoing research activities into how mechanisms triggered by heat treatment can lead to shorter cycle times for solution heat-treating and aging processes.[1] The hope is that this type of work will result in specification changes reflecting these advanced techniques in the not-too-distant future. Let's learn more.

Heat-treatable aluminum alloys are ones that contain elements designed to increase their strength by thermal processing. These elements exhibit increasing solid solubility with increasing temperature. In addition, the alloying elements are present in concentrations that exceed their equilibrium solid solubility at room (and moderately higher) temperature.

A typical heat-treat sequence (Table 1) involves solution heat treating (solutionizing) followed by rapid cooling (quenching) and age hardening (aging, precipitation hardening) to obtain high strength and other important mechanical properties. The relative rates at which solution and precipitation reactions occur depends to a large extent on the respective diffusion rates of the different solutes in addition to their solubility and alloy contents. Bulk diffusion coefficients for a number of the commercially important alloying elements in aluminum have been determined by experimental methods.

## Aging Fundamentals[3]

Aging must take place below both the solvus temperature (the temperature at which the alloying elements will dissolve and disperse) and the Guinier-Preston (GP) zone solvus line (Fig. 1) in order for uniform precipitation to occur.

In most precipitation-hardenable systems, a complex sequence of time-dependent and temperature-dependent changes is involved. Three types of interfaces are common during nucleation and growth of the precipitates: coherent, semi-coherent and incoherent. The resultant precipitates can occur in a variety of shapes, generally determined by a minimal energy state, particle size, matrix misfit and interfacial energy. Examples include needles, spheres, cubes and

**Table 1. Process description**[2]

| Process | Solution heat treating | Quenching | Aging (age hardening) |
|---|---|---|---|
| Goal | To take into solid solution the maximum practical amount of soluble hardening elements (e.g., Cu, Si, Mg) in the alloy. | To preserve as nearly intact as possible the solid solution formed at elevated (solution-treat) temperatures. | To cause precipitation to occur (the degree of stable equilibrium for a given grade is a function of time and temperature). |
| Purpose | To take advantage of the precipitation-hardening reaction (aging), it is first necessary to produce a solid solution. | To cool rapidly enough to produce a super-saturated solution. | To create a change in which the structure recovers from an unstable or "meta" stable condition (produced by quenching or cold working). |

disks. Precipitation normally occurs in the following sequence: solid solution, solute clustering, transition precipitates and equilibrium precipitates. Since strength is related to the volume fraction of the various precipitates (as well as their particle size), the number and type of alloying elements are important.

One of the concerns during precipitation hardening is the formation of precipitate-free zones (PFZ) that may form near grain boundaries (Fig. 2). PFZs can adversely affect ductility, fracture toughness and fatigue-crack-initiation resistance as well as increase susceptibility to stress corrosion cracking. Lowering the aging temperature, which effectively decreases diffusion rates and the solute super saturation, can minimize PFZs. Consequently, age-hardened alloys can be subjected to a double-aging treatment: a low-temperature age followed by a higher-temperature age. The low-temperature age increases the number of precipitates and minimizes the formation of PFZs, while the higher-temperature age accelerates the growth of precipitates.

## Double Aging

Double aging can also result in shorter cycle times in certain alloy systems, offering potential savings in both time and energy. An example of research in this field is the study of 7075 aluminum, a major aerospace structural alloy. In particular, the effects of double aging (DA) and thermomechanical double aging (TMDA) on accelerating the kinetics of precipitation have been documented in literature,[4] and the benefits have been briefly discussed.[5] It has been reported that by using the appropriate heat-treatment parameters, the time to peak hardness can be reduced from 48 hours to two hours with higher ductility and only a 6% decrease in yield and tensile strength.[5]

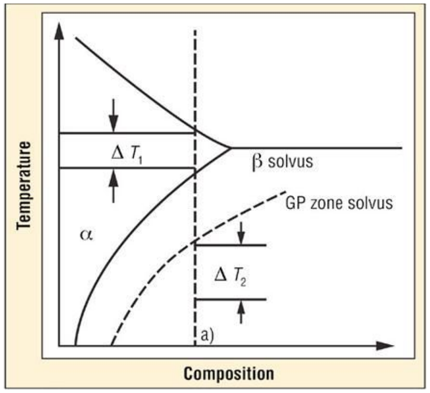

Fig. 1. Phase diagram showing the beta-solvus and GP-zone solvus for a hypothetical alloy system. For composition (a), the solution heat-treatment and aging temperatures are shown ($\Delta T_1$ and $\Delta T_2$ respectively).[3]

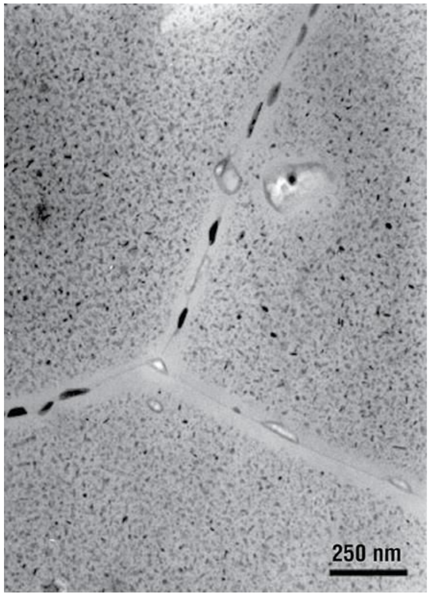

Fig. 2. Example of precipitation-free zones near grain boundaries[4]

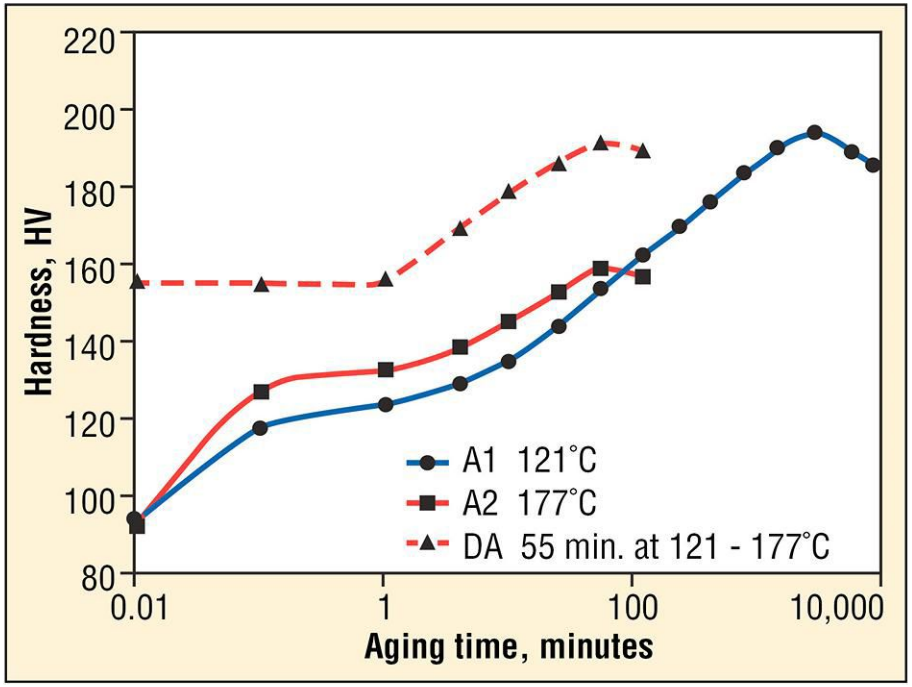

Fig. 3. Effect of single and double aging on hardness of 7075 aluminum[5]

Perhaps the most important consideration for maximum hardness in the DA treatment is controlling the time of the first age (Fig. 3). Increasing the time in the first age at 121°C (250°F), the peak hardness is achieved after the second age at 177°C (350°F), provided the time is optimized (in this case 55 minutes).

With respect to the second age (Fig. 4), using the same conditions as in the first aging treatment, namely 121°C (250°F) and 55 minutes, the peak hardness is similar for several second-age temperatures, namely 149°C (300°F) and 177°C (350°F). However, the time to reach peak hardness decreases with an increase in temperature.

## In Conclusion

While space considerations prevent us from summarizing the work being done on other wrought and cast alloy systems, such as two-step and high-temperature solution treatments and rapid heat treatment of aluminum high-pressure die castings,

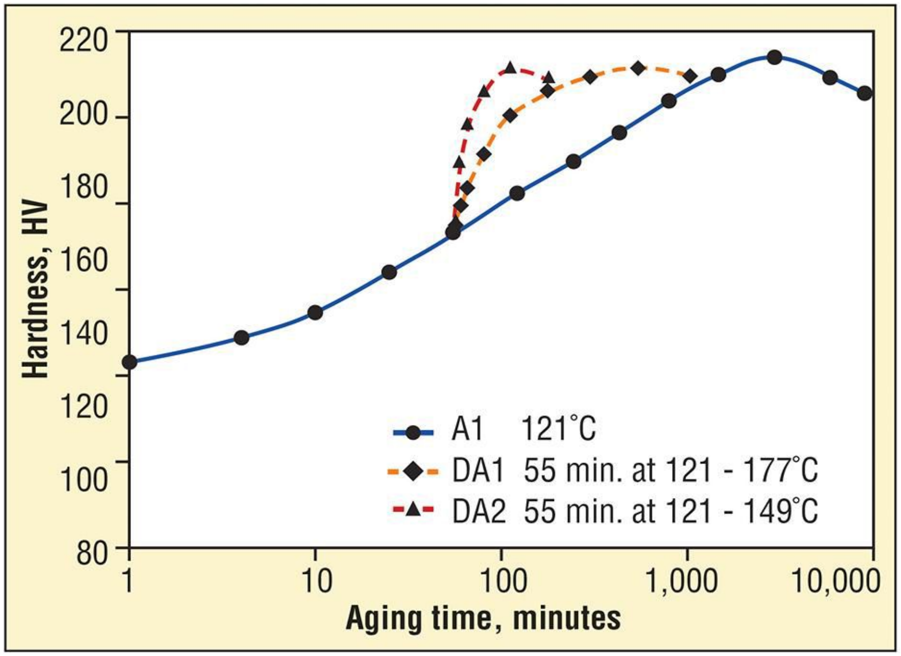

Fig. 4. Effect of second-aging temperature on double-aging treatment of 7075 aluminum[5]

progress continues in an effort to optimize cycle times.

Supporting research of this nature is critical to the heat-treatment industry as we strive to better serve our customers, increase productivity and save energy. The use of appropriate double aging and/or thermomechanical double aging is but one example that shows there can be a significant return on our investment from better understanding and then accelerating the kinetics of precipitation

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# Innovations in Aluminum Heat Treatment

The heat treater is always looking to improve his processing techniques, shorten cycle times and lower costs without sacrificing production quality. Recent innovations in aluminum heat treating suggest this is possible. Let's learn more.

## Double Aging (DA)

Double aging (Fig. 1) is a technique used for reducing total aging time for various aluminum alloys. The effects of double aging and thermomechanical double aging (i.e. a combination of work hardening and heat treatment) on enhanced mechanical properties of a 7075 aluminum alloy have recently been reported.[1] Results illustrate the effects of accelerating the kinetics of precipitation on hardness and strength (via tensile testing). By applying the appropriate heat-treatment cycle, the time to peak aging can be reduced by a factor of up to 36 without a substantial decrease in hardness or tensile properties.

As heat treaters we know age-hardenable aluminum alloys can be hardened or strengthened by natural or artificial aging. The science tells us that a supersaturated solid solution is formed after the solution heat treatment. Aging at temperatures well below the solutionizing temperature produces Guinier-Preston (GP) zones and metastable coherent precipitates that harden the alloy up to the peak condition. However, stable and incoherent precipitates start forming in the overaged condition. The type of precipitation varies with alloy composition.

For example, the most important factor influencing the DA treatment in an Al-Zn-Mg alloy such as 7075 is the time of first aging. Test results on 7075 show positive effects of the DA treatment, with the ductility being higher with slightly less than a 6% decrease in yield and tensile strength. Double aging this alloy to peak hardness results in a significantly reduced processing time – from 48 hours to 2 hours – and such processing can lead to reduced energy usage and reduced production costs resulting from the accelerated kinetics of precipitation.

Again, there is an optimum time, which varies with both the composition of the alloy and temperature of first aging treatment. A higher second aging temperature accelerates the precipitation in 7075, but it cannot be so high as to cause significant reduction in volume fraction of precipitates or dissolution of GP-zones.

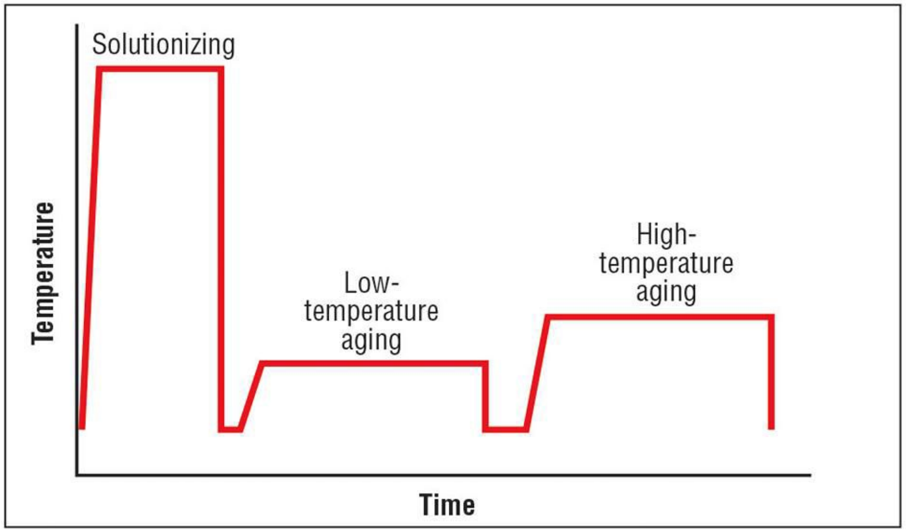

Fig. 1. Double-aging heat-treatment process cycle

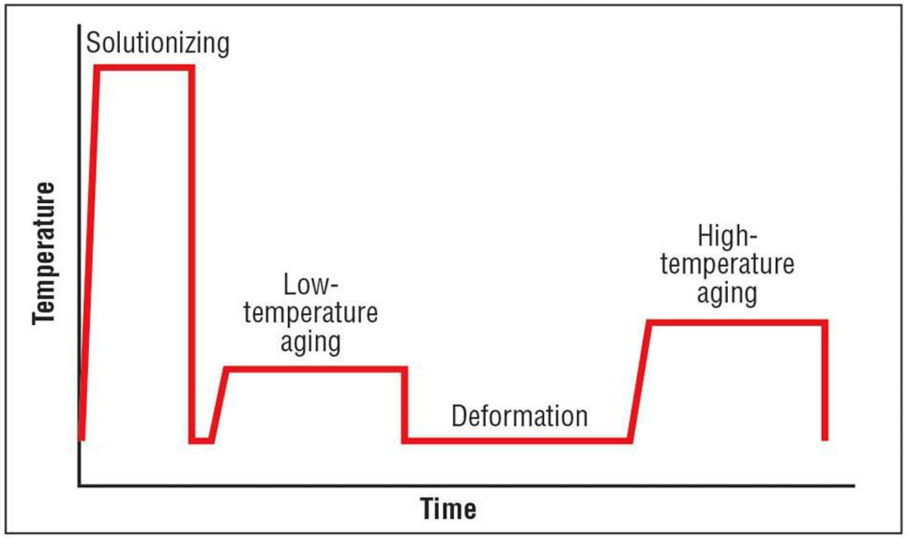

Fig. 2. Double aging and thermomechanical heat-treatment process cycle

Thermomechanical double aging (Fig. 2) causes further acceleration of precipitation, reducing the total heat-treatment time to only 80 minutes. This results in an increase in hardness and strength in 7075 alloy beyond the conventional aging procedure, but the ductility is reduced relative to conventional single age or the double-aging heat treatments.

## Rapid Heat Treatment (RHT)[2,3]

When cast components for structural applications such as alloy wheels are manufactured using Al-Si-Mg-based casting alloys (typically A356 and A357), T6 heat treatment is in most cases an essential step in the manufacturing process. The T6 heat treatment provides two beneficial effects: an improved ductility and fracture toughness through spheroidization of the eutectic silicon particles in the microstructure and a higher alloy yield strength (YS) through the formation of a large number of fine precipitates that strengthen the soft aluminum matrix. The first benefit is realized through the solution treatment, normally at a temperature around 1005°F (540°C), while the second benefit is achieved through the combination of solution treatment, quenching and artificial aging in the temperature range of 285-340°F (140-170°C).

In the casting industry, it is often specified that a cast component should be solution treated for six hours at 1005°F (540°C). While the benefit of T6

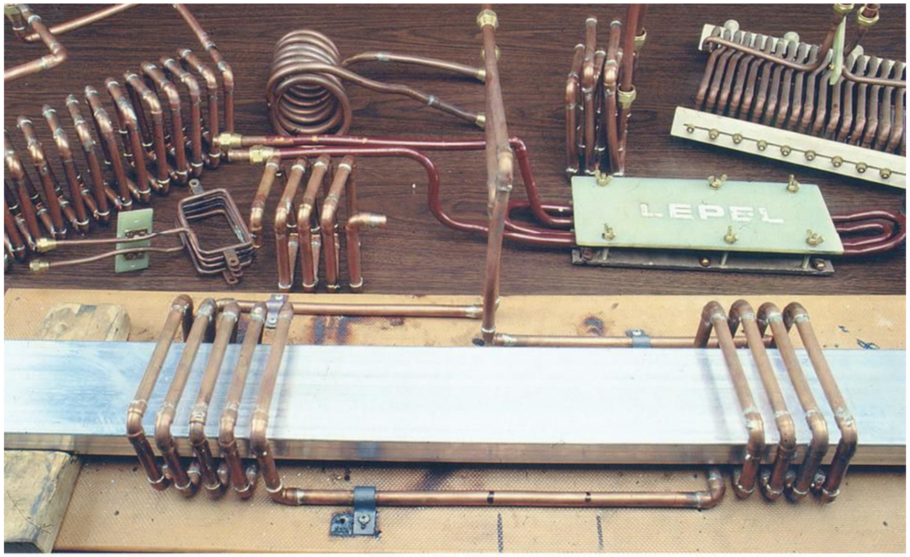

Fig. 3. Typical induction coils used in the RHT process

heat treatment is accepted, the additional cost and production time associated with such a treatment is also substantial. Positive results have been reported for permanent mold-cast test bars of a modified A356 alloy where a solution treatment of 50 minutes at 1005°F (540°C) is sufficient to attain more than 90% of the maximum YS, more than 95% of the ultimate tensile strength (UTS) and nearly 90% of the maximum elongation for a given aging condition.

Other studies have investigated the effect of even shorter (0-30 minute) solution-treatment time at 1005°F (540°C) or 1020°F (550°C) on the microstructure and properties of fully modified Al-7wt.%Si-0.3wt.%Mg alloys. In addition, it has recently been demonstrated that common high-pressure diecasting (HPDC) alloys, such as those based on the Al-Si-Cu and Al-Si-Mg-(Cu) systems, may be successfully heat treated without causing surface blistering or dimensional instability. The potential exists therefore to develop and evaluate secondary HPDC alloys designed specifically for rapid heat treatment while still displaying high castability.

Significant improvements in the tensile properties for HPDC alloys may be achieved by a truncated solution treatment of 15 minutes at 915-940°F (490-505°C), quenching and then artificial aging at temperatures in the range of 300-425°F (150-220°C).

## Retrogression Heat Treatment (RHT)[4]

Retrogression heat treatment (Fig. 3) is used for improving formability of aluminum extrusions. It is defined as a means of rapidly heat treating 6xxx and 7xxx aluminum alloy extrusions in various tempers, either locally or throughout their length, in order to return to or approach their highly ductile state in their solutionized and freshly quenched state.

For these extrusions in some tempers – such as T4, T5, T6, and even T7, T8, and T9 – RHT involves heating into the solvus temperature range and quenching. Unlike conventional solutionizing heat treatments that involve long times, RHT can be

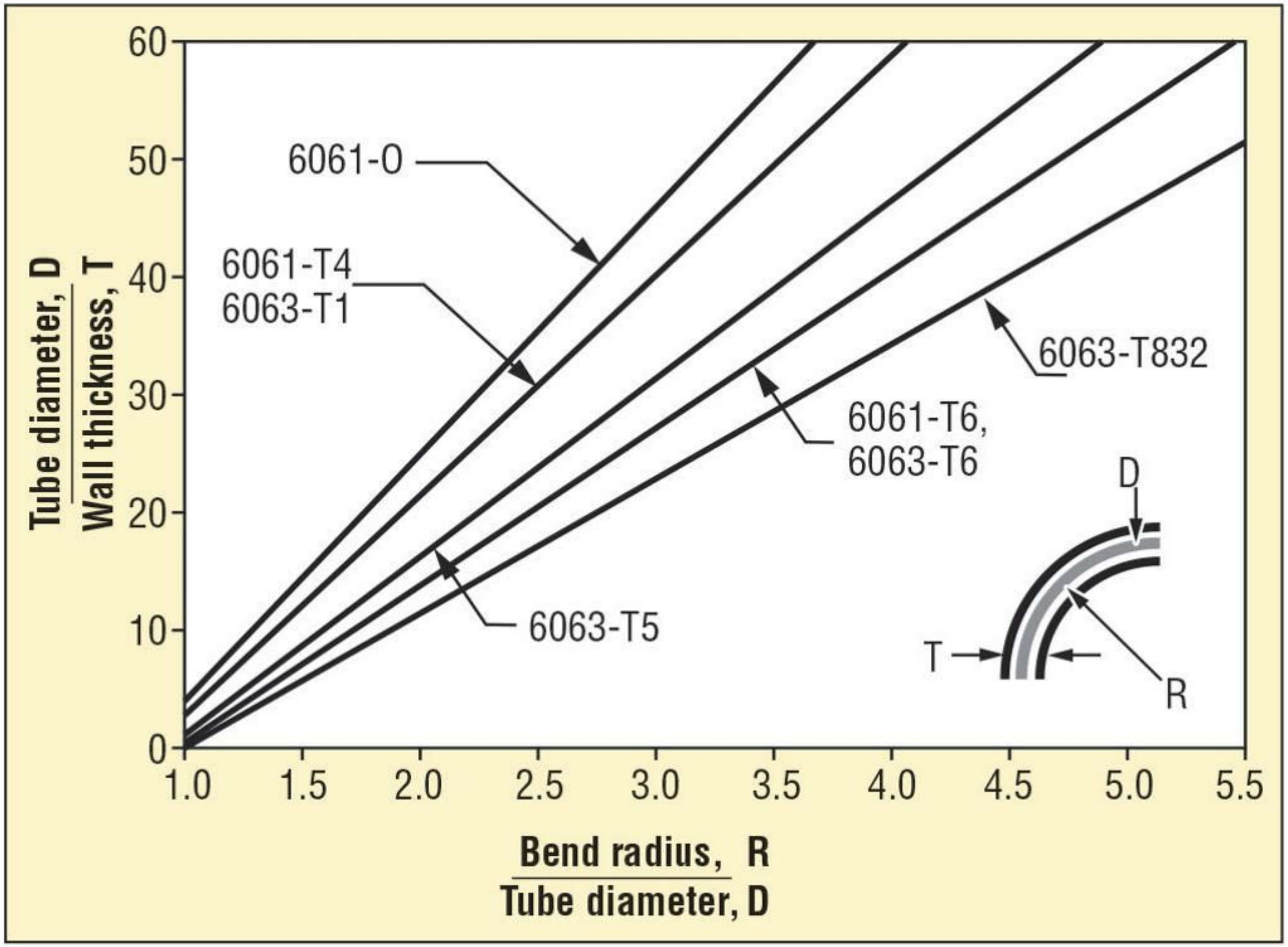

Fig. 4. Bending relationship Note: In the above figure, if the tube diameter to wall thickness ratio lies above the respective lines for the given tempers, the tube generally cracks during bending.

conducted within a matter of seconds and synchronized with a given forming operation. Several commercial applications of the RHT process have already been demonstrated, including forming of aluminum extrusions (Fig. 4) and the forming of heat-treated automotive sheet.

The localized heating and quenching in RHT is applied to materials in high-strength tempers only in regions that will subsequently experience heavy deformation during forming, thus maintaining high strength elsewhere. Although the method of heating in RHT can vary, induction heating has been found to be most reliable and consistent in applying the RHT process to aluminum alloys.

## Summing Up

While most heat-treatment processes for aluminum alloys are dictated by specifications (e.g., AMS 2770 or AMS 2771) it is important to understand that ongoing research is looking into ways of reducing cycle times and minimizing costs. Stay tuned.

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# Heat Treatment of Titanium Alloys

“I know the Greek alphabet well,” exclaimed the heat treater. “Alpha, beta, alpha-beta, gamma and so on.” “Oh, you must process titanium alloys,” surmised the scholar. How did he know? Let’s learn more.

Titanium has many attributes that are useful in today’s modern society. It is a relatively lightweight, corrosion-resistant structural material that can be strengthened dramatically through alloying and, in some cases, by heat treatment. Among its many advantages for aerospace, military and commercial: good strength-to-weight ratio, low density, low coefficient of thermal expansion, good corrosion resistance, good oxidation resistance at intermediate temperatures, good toughness and (relatively) low heat-treatment temperatures.

## Alloy Classification

Titanium alloys are typically classified as pure titanium, alpha, beta and alpha-beta alloys. There are also so-called near-alpha-phase and near-beta-phase (i.e. metastable beta) alloys. Under equilibrium conditions, pure titanium and alpha phase have hexagonal-closed-packed (HCP) structures up to 882˚C (1620˚F). Above this temperature, they transform to beta (β) phase having a body-centered-cubic (BCC) structure up to

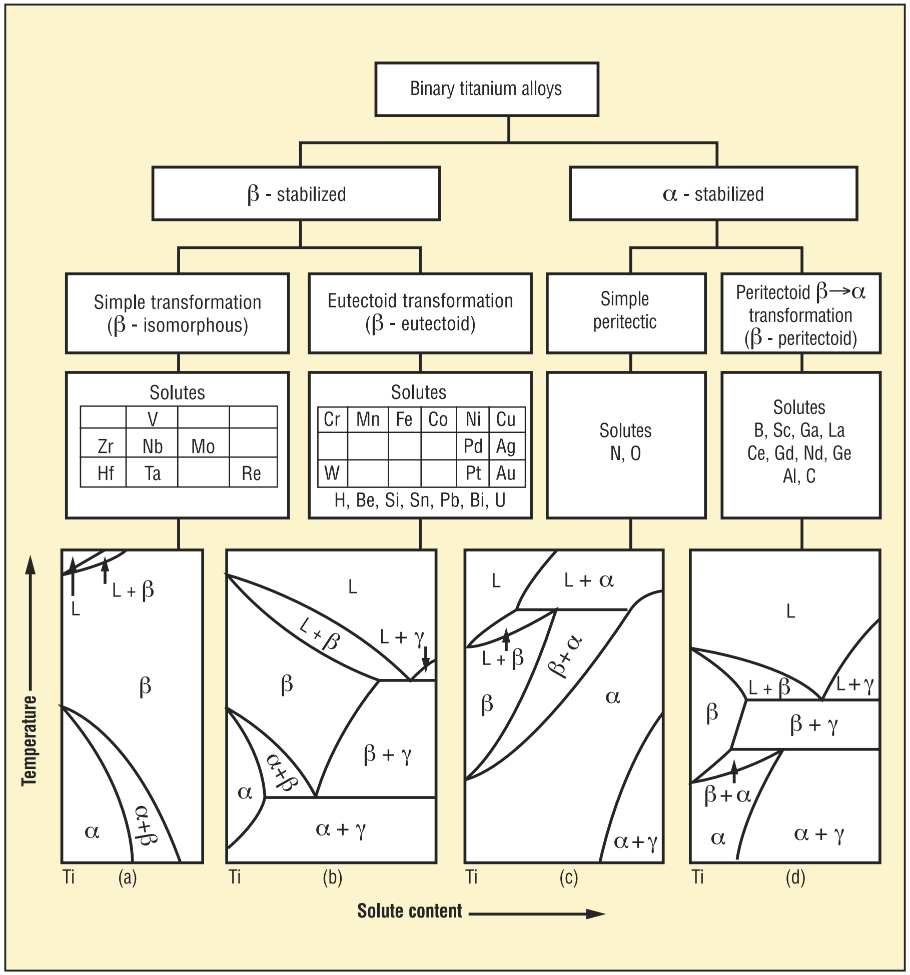

Fig. 1. Classification of binary titanium alloys, alloying elements and their effects on the phase diagram[1,3]

the alloy's melting point. Near-alpha alloys typically have a small amount of beta-phase (1-2%) stabilizing elements. In near-beta alloys, significant additions of beta stabilizer suppress the Ms temperature to below ambient, and the beta phase is retained at room temperature by rapid cooling or quenching from the alpha-beta phase. The inherent properties of all these structures are quite different.

Alloying elements (Fig. 1) generally stabilize one or the other of these phases. The alpha phase is stabilized by aluminum, gallium, germanium, boron, carbon, oxygen and nitrogen, while the beta phase is stabilized by molybdenum, vanadium, tantalum, niobium, iron and hydrogen.

The boundary between the alpha and beta phase and the two-phase alpha-beta region (Fig. 2) is called the alpha transus and beta transus respectively. The beta-stabilized system has Ms and Mf temperatures associated with it.

The titanium grades most commonly used have compositional specifications determined by ASTM E120 (Standard Test Methods for Chemical Analysis of Titanium and Titanium Alloys) for the commercially important alloys. Military specifications are found under MIL-T-9046 and MIL-T-9047, and aerospace material specifications for bar, sheet, tubing and wire are under AMS specification numbers 4900-4980. In addition, large aircraft companies have their own set of alloy specifications. Because titanium alloys are used in a variety of applications, several different material and quality standards are specified. Among these are ASTM, ASME, AMS, the U.S. military and a number of proprietary sources.

Titanium alloys may be divided into two principal categories: corrosion resistant and structural.[3-9] The corrosion-resistant alloys are generally based on a single-phase (alpha) with dilute additions of solid-solution strengthening and alpha-stabilizing elements such as oxygen (interstitial), palladium, ruthenium and aluminum (substitutional). These alloys are used in the chemical, energy, paper and food-processing industries to produce highly corrosion-resistant tubing, heat exchangers, valve housings and containers. The single-phase alpha alloys provide excellent corrosion resistance, good weldability, and easy processing and fabrication but a relatively low strength.

The beta phase is stabilized by additions such as molybdenum, vanadium, niobium, iron (substitutional) and hydrogen (interstitial). A dispersion of alpha in the beta matrix along with solid-solution strengthening of both the alpha and beta phases lead to higher-strength alloys referred to as structural alloys. The structural alloys can be divided into four categories: the near-alpha alloys, the alpha-beta alloys, the beta alloys and the titanium aluminide (ordered) intermetallics (based on $Ti_xAl$ where x = 1 or 3). With titanium alloys used in structural applications, optimization of mechanical properties is very important. Therefore, processing and microstructure control are critical.

## Heat Treatments

Not all heat treatments are applicable to titanium alloys because of the differences in composition and microstructure. Alpha alloys generally are not heat treatable, having medium strength, good notch toughness and good creep resistance.

The response of titanium alloys to heat treatment depends on their composition and the effect of heat treatment on the alpha-beta phase balance. Strength of annealed alloys increases gradually and linearly with increasing alloy contents.

Alloys of the beta type respond to heat treatment, are characterized by higher density than pure titanium and are more easily fabricated. The purpose of alloying to promote the beta phase is either to form an all-beta-phase alloy having commercially useful qualities, to form alloys that have duplex alpha and beta structure to enhance heat-treatment response (i.e. changing the alpha and beta volume ratio) or to use beta eutectoid elements for intermetallic hardening.

Quenching from the beta-phase field gives a martensitic transformation with improved strength (depending on composition). Rapid quenching of titanium with relatively few alloying elements from the beta-phase field gives maximum strength at $M_f$. For highly alloyed titanium, rapid quenching from beta-phase field gives lowest strength, but the maximum strength is obtained after aging. The most important beta alloying element is vanadium.[1]

Beta and alpha-beta alloys are heat treated to enhance specific properties. The general classifications for these heat treatments include:

- Stress relief – to reduce residual stress due to fabrication (e.g., forming, machining, welding) or heat treatment
- Process annealing – to optimize microstructure, manufacturability, dimensional stability and service life
- Solution treat and age – for mechanical-property development (e.g., strength, ductility, fracture toughness, creep resistance, fatigue strength)

Heat treatments specific to alpha-beta alloys[1] include stress relief; mill or full anneal; recrystallization annealing (RA); duplex annealing (DA); beta annealing (βA); solution treat and age (STA); solution treat and overage (STOA); beta solution treat and age (βT STA); beta solution treat and overage (βT STOA); and beta

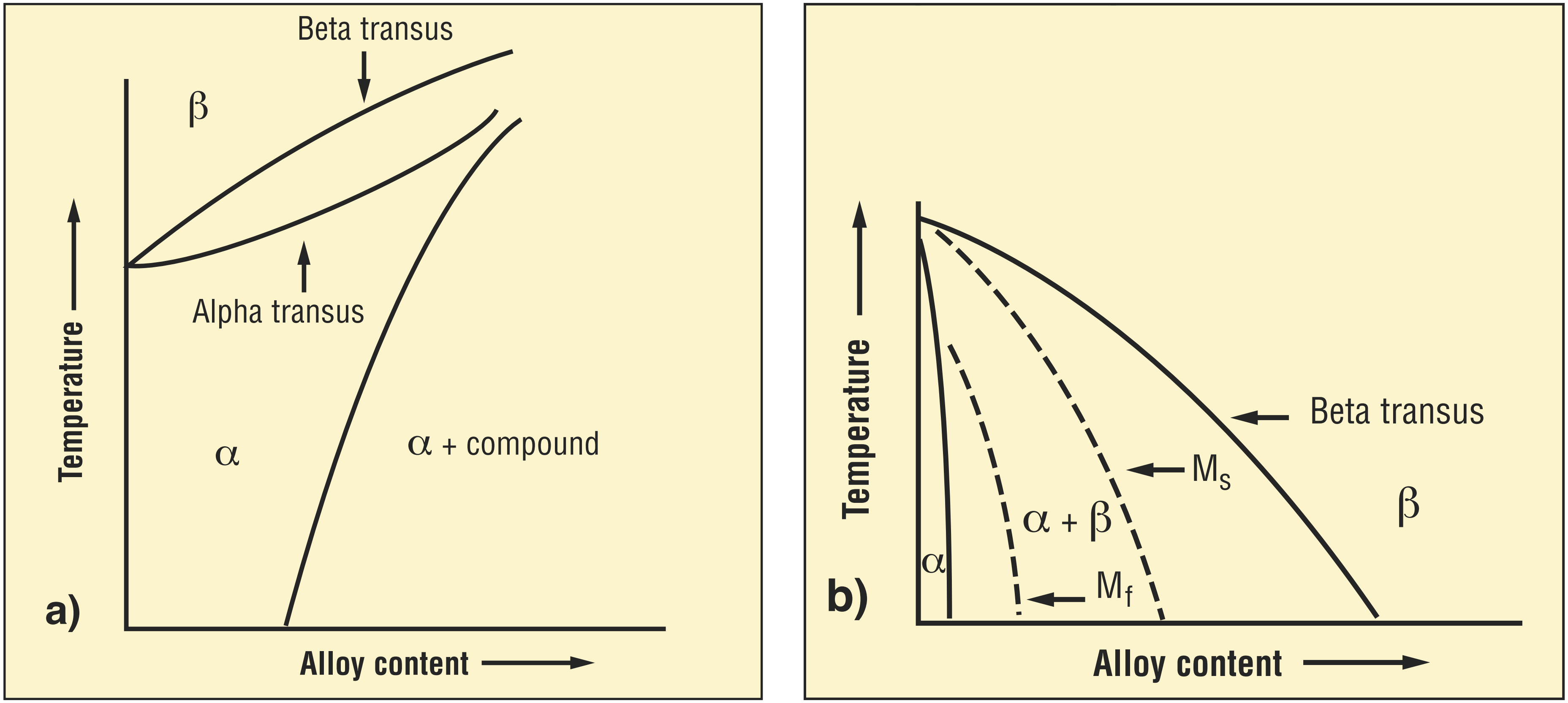

Fig. 2. Simplified phase diagram (a) alpha-stabilized system and (b) beta-stabilized system[1,2]

plus alpha-beta solution treat and age (TRIPLEX STA).

Heat treatments applicable to metastable beta titanium alloys[1] include solution treating, sub-transus solution treating, supra-transus solution treating, direct aging, solution treat plus single age, solution treat plus duplex age and pre-age solution treat and age (PASTA).

In some applications, titanium alloys have been case hardened (nitrided and carburized) to enhance certain surface properties. Laser surface alloying and ion implantation are possible but not commonly used.

## In Conclusion

One of the biggest challenges faced by the heat treater is to understand and comply with the large number of specifications applicable to a particular titanium alloy grade. By way of example, some of the common AMS heat-treat specifications for Ti-6Al-4V alloys (including legacy specifications) include: AMS 2801, AMS 4901, AMS 4904, AMS 4905, AMS 4911, AMS 4928, AMS 4931, AMS 4935, AMS 4943, AMS 4945, AMS 4965, AMS 4967, AMS 4975, AMS 4979, AMS 4984, AMS-H-81200 and AMS-T-9046 to name a few.

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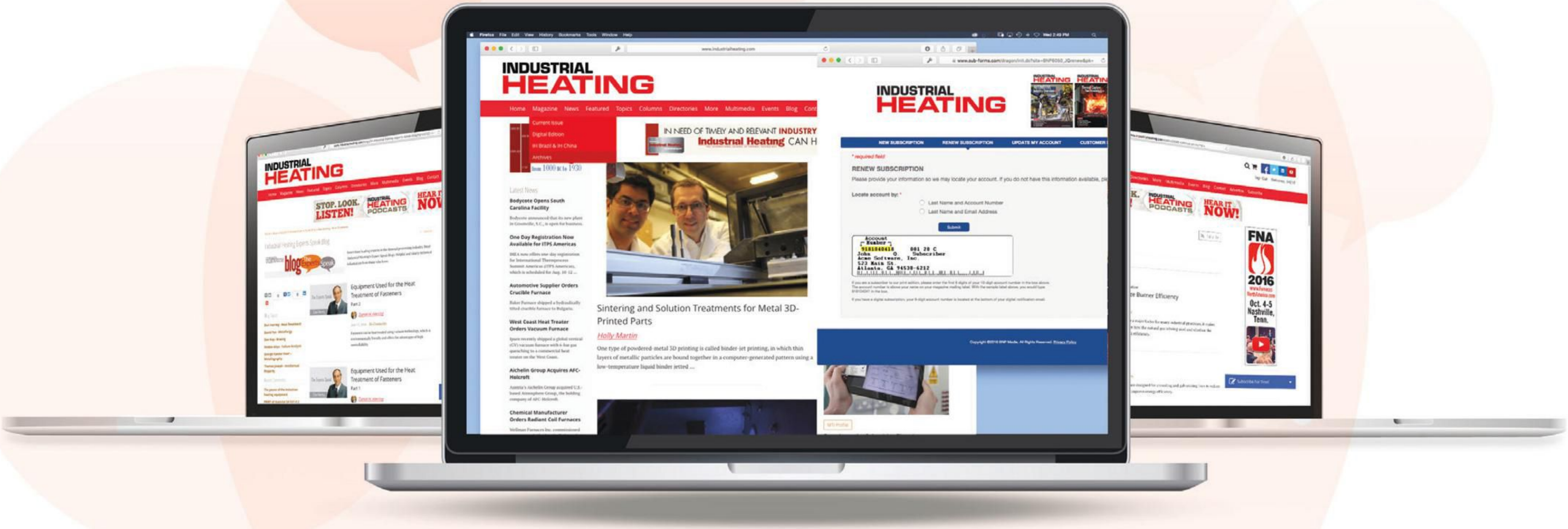
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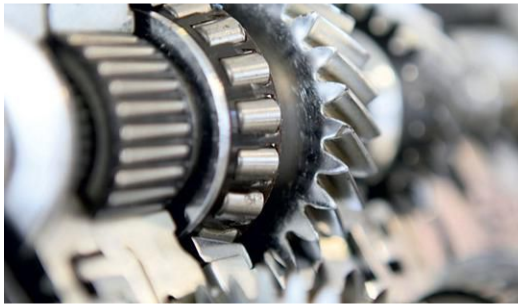


# Internal Furnace Pressure

Do you know what the internal pressure is, or should be, inside your atmosphere furnace? Or why this is an important value to be aware of when it comes to safety or process control? Everyone needs to know the answers to these questions, but few do. Let's learn more.

Most atmosphere furnaces, whether they be batch or continuous, operate at internal pressures in the range of 0.01-0.10 inches of water column (2.5-25 Pa), with the upper value being a good targeted operating pressure for most furnaces. A very tight furnace system might reach as high as 0.20-0.30 inches of water column (50-75 Pa), but this is far less common in the heat-treat industry. To put this pressure range in perspective, an internal pressure of 0.10 inches of water column (2.5 Pa) is about equivalent to the pressure exerted on a flat table by 10 one-dollar bills stacked one atop another.

Common misconceptions are that increasing the flow alone will automatically increase the pressure or that simply adding more weights to an exhaust flapper will, in and of itself, force the furnace pressure higher. Pressure will always be lower in poorly sealed batch furnaces or in continuous furnaces since they are open on both ends.

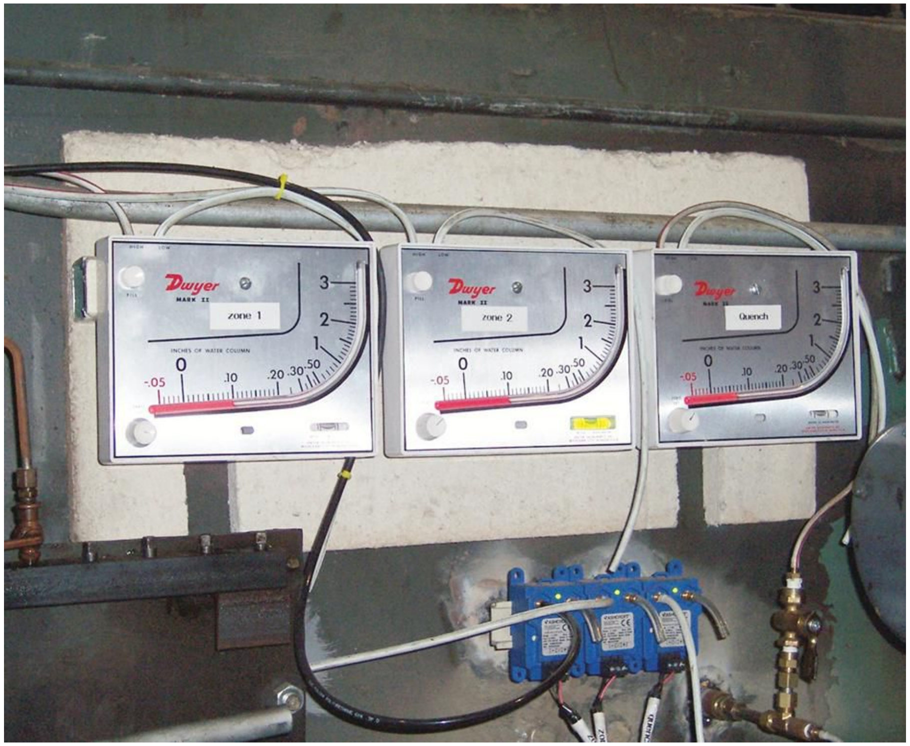


Fig. 1. Pressure-monitoring system for a two-chamber integral-quench furnace with oil quench

## Measuring Furnace Pressure

Use of a simple manometer (Fig. 1) is a fast, easy and inexpensive way to monitor furnace pressure. Digital displays, recorders and remote signal transmission simplify the task of accurate measurement and control. It is always valuable to monitor changes in pressure during normal operation (Fig. 2) and to determine the "normal" length of time a furnace is in a negative-pressure situation (e.g., after a load is discharged to the quench tank in an integral-quench furnace). Unusually long periods of negative pressure or changes in pressure are a good way to determine if furnace troubleshooting or maintenance is necessary.

## Eliminating Furnace Leaks

We all know, or should know, that a furnace atmosphere must be present in sufficient volume and pressure so as to exclude air from entering the furnace atmosphere. Both parameters are important for safe operation, especially during door openings, load transfers and when the furnace is operating below 1400°F (760°C). In addition, if one is trying to precisely control the active furnace atmosphere to reach a specific carbon potential or dew point inside the furnace, changes in pressure can upset the delicate balance of the atmosphere.

Typically, leaks present below the furnace hearth will draw air in and those above the hearth will leak atmosphere out. Finding leaks above the hearth can often simply be done using a small torch to light off the combustible gas (observing all appropriate

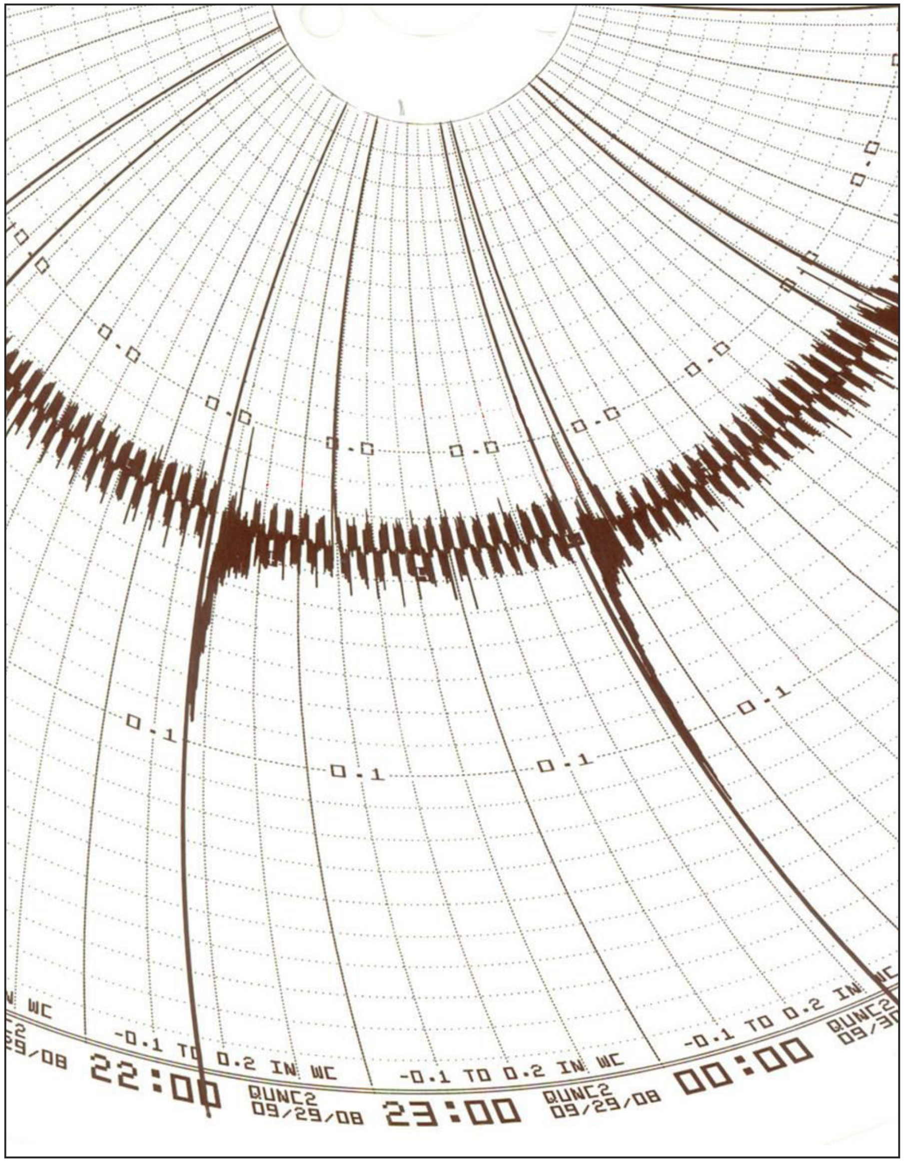


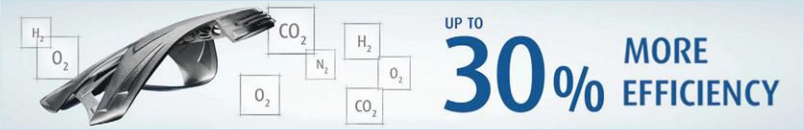
Fig. 2. Pressure chart for a two-chamber integral-quench furnace

safety precautions!), but leaks below the hearth line are much more difficult to find. Techniques such as over-pressurizing the furnace (to force atmosphere out of these lower areas) or smoke-bomb testing are commonly used to locate them. Keep in mind a furnace leak that affects the atmosphere is typically considered a large leak, with a total area in the neighborhood of 0.75 in$^2$ (480 mm$^2$) or greater.

## Leaky Radiant Tubes

Pressure changes that occur within a furnace are often due to leaky radiant tubes. If a tube ruptures, the combustion products will leak out and mix with the furnace atmosphere, creating a fluctuation in the internal atmosphere pressure in the furnace. If you suspect that you have a leaky tube, here are a few suggestions on how to find it from those who work on these problems every day. Remember that the burner, recuperator (if present) and all associated piping are very hot, and proper personal protective equipment must be used to avoid injury.

It is important to recognize that leaks in radiant tubes generally begin as a pinhole or small crack and gradually increase in size. Symptoms include an erratic (oscillating) carbon potential (i.e. oxygen-probe millivolt values) or a furnace dew point that slowly creeps up higher and higher. In either case,



the atmosphere control system usually calls for more enriching-gas additions to hold the setpoint value. As the leak worsens, still more enriching gas is needed, until the point when you may not be able to hold the desired carbon-potential setpoint. Parts may even show evidence of decarburization despite large amounts of enriching gas in use.

Pinhole leaks are difficult to find with conventional techniques because any carbon dioxide ($CO_2$) produced from combustion will react with endothermic gas present. Larger leaks are generally easier to spot but have been known to open and close with changing temperature, making the task far more difficult. In all cases, a recording system will assist in more quickly detecting upward or downward trends.

One method for leak testing is as follows:

1. Lower the furnace temperature to around 1500°F (815°C).
2. Stop the flow of all enrichment gases (e.g., hydrocarbon gas, air, ammonia).
3. Record the millivolt output from the oxygen probe as the combustion system cycles on and off at setpoint. It is always a good idea to record the combustion-system output percentages as well.

Note: The millivolt signal from the oxygen probe should be essentially steady state (i.e. not oscillating wildly).

4. Raise the furnace temperature to 1700°F (925°C) and record the millivolt signal as the furnace heats to its new setpoint with the combustion system on high fire.

Note: Under normal circumstances, as you raise temperature, the millivolt signal on the oxygen probe will steadily increase. If you have a leaky tube, you either won't see an increase or there will be only a minimal increase in millivolt values.

5. When you reach the new higher setpoint, and as the combustion system cycles on and off, record the carbon-potential millivolt values and the combustion-system output percentages.
6. Turn the temperature setpoint back to 1500°F (815°C) so that the combustion-system output is zero and record the probe millivolt values as the temperature drops.

Note: The millivolt signal should steadily decrease. If it does not or if there is only a minimal decrease, a leak will be present.

7. Repeat Steps 1-6 again to confirm results.

Note: If the furnace is equipped with a plunge-cool option (where air can be blown through the tubes), heat back to a setpoint of 1700°F (925°C) and activate the plunge-cool feature. If the millivolt values fall rapidly, there is a leaky tube.

8. If you suspect a leaky tube, you may be able

sealed and one or more still leaking, the $CO_2$ content will not rise as fast during a burner firing cycle. Analyzing the data will help locate the leaky tube.

Note: Testing tubes for leaks with pressurized air outside of the furnace is not recommended by a number of OEM manufacturers since the tubes are not designed as pressure vessels and will not normally withstand pressures greater than 5 psig (35 kPa).

4. A similar test with an oxygen meter is to shut off the gas to a burner and allow the tube to purge. When the oxygen level reaches approximately 18-20%, shut off the air. Watch the oxygen meter, and if the level drops, this tube is suspect.

Recuperators can also be leak tested following the manufacturer's recommendations. This often involves pressure testing. If the pressure is lost immediately, the recuperator is leaking. A small leak is often permissible. However, that recuperator should be marked for future testing or replacement.

## Summary

As far as this writer is concerned, there is no excuse not to have internal-pressure-monitoring capability on every heat-treating furnace running protective atmosphere. The amount of information that can be gained far outweighs the cost of adding these types of sensors to our furnaces.

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# Hydrogen for the Heat-Treat Shop

Hydrogen and hydrogen-bearing atmospheres are arguably the most versatile furnace atmospheres employed in the heat-treatment industry. How hydrogen is produced and the various supply options available are important questions that need to be better understood. Let's learn more.

Hydrogen is said to be the highest-volume gas consumed in the heat-treating industry.[1] Its usefulness is due in large part to its unique chemical and physical properties (Table 1). Applications using hydrogen often rely upon its reducing and/or protective atmosphere characteristics. For steel and stainless steels, examples include bright annealing, brazing, metal injection molding, sintering and welding. CVD coatings and crystal growing also utilize hydrogen gas. In general, processing of stainless steels requires a higher purity level than carbon steel, in which the atmosphere is often either blended (e.g., nitrogen/hydrogen) or diluted (e.g., endothermic/nitrogen).

Table 1. Physical properties of common gases (@ 25°C, 1 bar)[2]

| Gas type and property | Argon | Helium | Hydrogen | Nitrogen |
|---|---|---|---|---|
| Density @ 15°C, kg/m$^3$ | 1.6687 | 0.167 | 0.0841 | 1.170 |
| Density ratio to air | 1.3797 | 0.138 | 0.0695 | 0.967 |
| Molar mass, kg/kmole | 39.948 | 4.0026 | 2.0158 | 28.0 |
| Specific heat capacity (Cp), kJ/kg-K | 0.5024 | 5.1931 | 14.3 | 1.041 |
| Thermal Conductivity (λ), W/m-K | 0.0177 | 0.1500 | 0.1869 | 0.0259 |
| Dynamic viscosity (η), N-s/m$^2$ | 0.0000226 | 0.00001968 | 0.00000892 | 0.00001774 |

## Production Methods

A number of production methods exist for generating hydrogen. These include steam reforming (i.e., collection and extraction of hydrogen from fossil fuels), electrolysis and thermolysis (both of which extract hydrogen from water molecules). Supply modes often depend on consumption (Fig. 1).

Traditionally, hydrogen has been purchased through large-scale reformers at or near the site of use or from industrial-gas suppliers via truck delivery or dedicated pipeline to the site. Steam reforming and similar thermal-cracking techniques have typically been best matched to very large-volume, steady-state processes, such as hydrogen supply for chemical plants and refineries.

On-site hydrogen-generation systems (Fig. 2) are becoming a popular choice for the heat-treatment industry and include methods such as water electrolysis, which matches typical industry-required flow-rate ranges and usage patterns. Hydrogen purity is typically

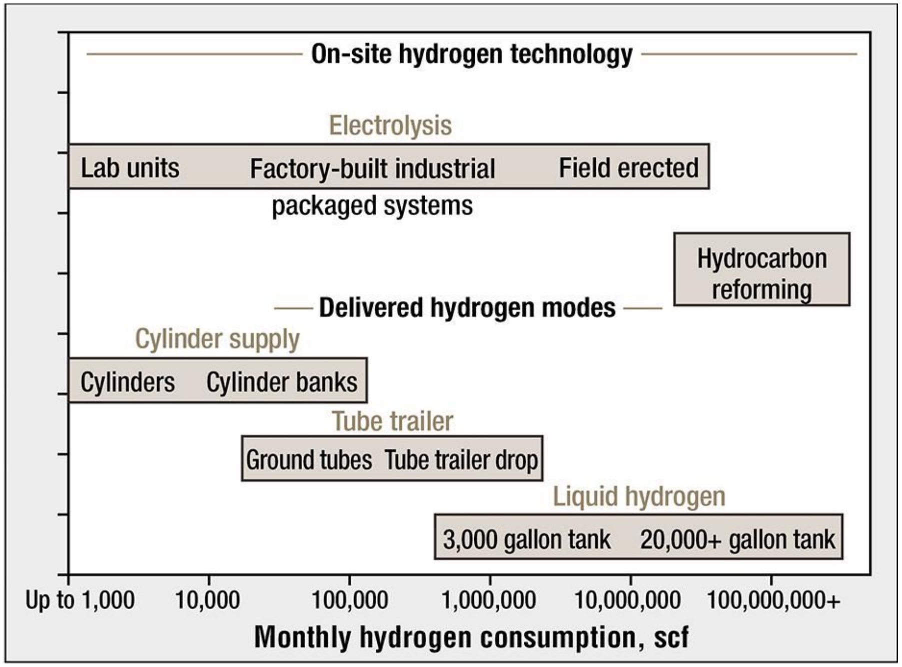

Fig. 1. Typical U.S. hydrogen supply modes[1]

99.9995+% (or better).

With respect to electrolysis, three cell technologies are in use: solid oxide electrolysis cells (SOECs), alkaline electrolytic cells (AECs) operating at high electrolyte (KOH or $K_2CO_3$) concentrations and polymer electrolyte membrane cells (PEMs). SOECs operate at high temperature (typically at or near 800°C), while AECs operate in the range of 200°C. By contrast, PEMs typically operate below 100°C.

Fig. 2. Typical on-site hydrogen generator (courtesy of Proton Onsite)

Both approaches have advantages and drawbacks. Large-scale on-site generation provides a reliable supply of hydrogen. Few heat-treating applications require hydrogen in such a large volume to justify the high capital expense of a conventional reforming generator, however, which is the technology most widely used in on-site applications. For most heat treaters, therefore, the option is to rely on delivered hydrogen. The downside of this approach is typically higher total cost of hydrogen, delivery surcharges and the potential for supply disruptions.[4]

## On-Site Hydrogen Generation

On-site hydrogen-generation systems employing water electrolysis are a good fit for the needs of the heat-treatment industry because they match the flow-rate range

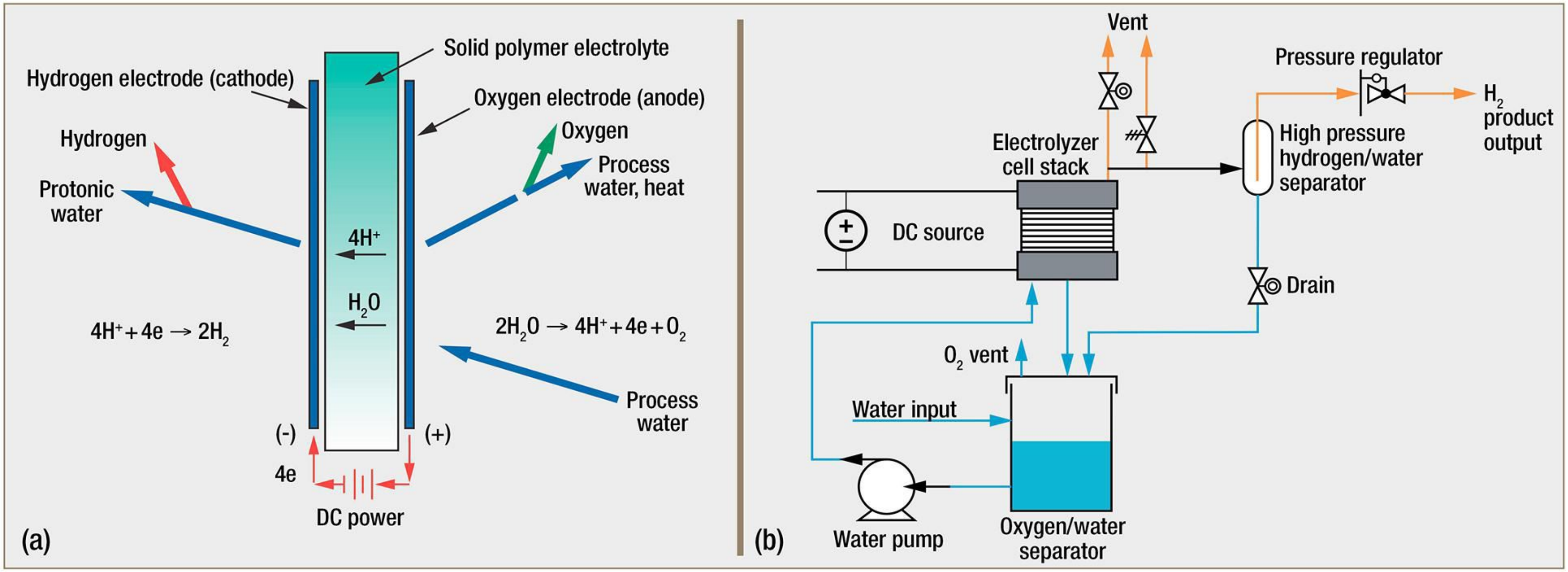

Fig. 3. PEM electrolysis method for producing hydrogen; (a) electrolysis process; (b) electrolysis system

and usage patterns well. Systems employing polymer electrolyte membrane (PEM) water electrolysis technology (Fig. 3) provide the purest hydrogen, at various pressure levels without a compressor, at variable flow rates and within a compact space envelope. The hydrogen produced is very pure at 99.9995+% or better. While hydrogen purity is extremely valuable to some customers, it is less important to others and is primarily based on what the hydrogen is being used for. The most important parameters about hydrogen purity are:

- What are the trace impurities? In particular, heat treaters need to know about nitrogen and carbon compounds.
- How dry is the gas? Often, water content is not always specified. One wants the driest hydrogen possible for the best results on the broadest range of metallurgical applications. If humidification is required for certain processes, the heat treater should add moisture through use of a saturator (bubbler).

The technology illustrated here typically employs a platinum catalyst in combination with a membrane separator to split deionized water into its constituent parts (hydrogen and oxygen) and the hydrogen sent off as a process gas. When a DC voltage is applied to the electrolyzer, water molecules at the anode are oxidized to oxygen and protons (H+ ions) while electrons are released. The protons pass through the membrane to the cathode, where they combine with electrons from the other side of the circuit and are reduced to hydrogen gas. Once generated, the hydrogen is dried and supplied to the desired process application. It is produced at the same rate that it is used, requiring no storage of hydrogen.

## Hydrogen Supply Choices

Which is best; a delivered product or on-site generation? In North America, hydrogen is widely available in a variety of supply options and delivery methods, so the choice is up to the individual user. A broad consensus of those using on-site technology points to the following advantages:

- Safety and compliance – often cited as less hazardous than storage sources
- Site utilization – ease of compliance to NFPA safety guidelines, especially those related to hydrogen storage
- Materials flexibility – versatility to process a wider variety of alloys
- Atmosphere cost predictability – cost of on-site-generated hydrogen becomes a fixed cost of operation

## Summary

The need for hydrogen in the heat-treatment industry continues to grow, and on-site hydrogen production capability is an intriguing alternative that should be explored by heat treaters.

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# Flowmeter Basics

Everyone knows what a flowmeter is, and, yet, few of us really understand them the way we should. The sad reality is that once flowmeters are installed and operating we tend to take them for granted. This can often lead to serious flow errors and potential process or safety issues that compound themselves over time. Let's learn more.

## What is a flowmeter, really?

A flowmeter is a device used for measuring the flow of gases or liquids. There are actually two different ways to measure flow – by volumetric means (Fig. 2a) and by mass-flow techniques (Fig. 2b). As heat treaters, we are probably more familiar with the volumetric-flow measurement of gases. The principle involves the displacement of the gas volume over time. Atmosphere furnaces, gas generators and combustion systems typically use these types of devices. Mass flow involves measuring the weight of a gas, and these are commonly found on vacuum furnaces that meter in gases for partial-pressure operation.

## What types of flowmeters are there?

As heat treaters, we typically focus on the measurement of gases. Anyone who has seen a nitrogen/methanol system, however, is aware that liquid methanol must be metered into the furnace. Flowmeters are also used for measuring liquid flow. In addition to flowscopes and mass flowmeters, other common types include: orifices, rotameters, positive-displacement meters, electromagnetic meters, ultrasonic (Doppler-effect)

Fig. 1. Typical flow control panel (multiple-zone continuous furnace)

devices, turbine meters, wedge flow devices, impact meters and turbine meters.

## What are the features and advantages of the most common flowmeter types?

Variable-area flowmeters offer:

- Mechanical flow measurement with only a single moving part, ensuring measurement reliability
- Application versatility and availability of a variety of construction materials, inlet and outlet sizes and types
- Easy installation with generally no straight pipe requirements
- Low pressure drops
- Linear scales, allowing easy flow measurement interpretation
- Electronic output availability, preserving the benefits of mechanical flow measurement

Tapered-tube rotameters offer:

- Low instrument cost (when glass or plastic metering tube is used)
- Accuracy at very low flow rates

Slotted-cylinder flowmeters offer:

- A flow range of 25:1 since flow-measurement accuracy is determined by the precision of the slot manufacturing operation

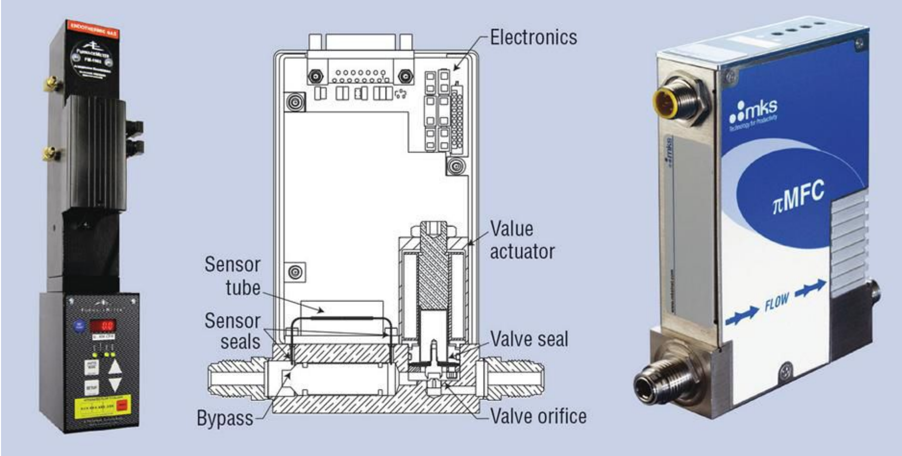
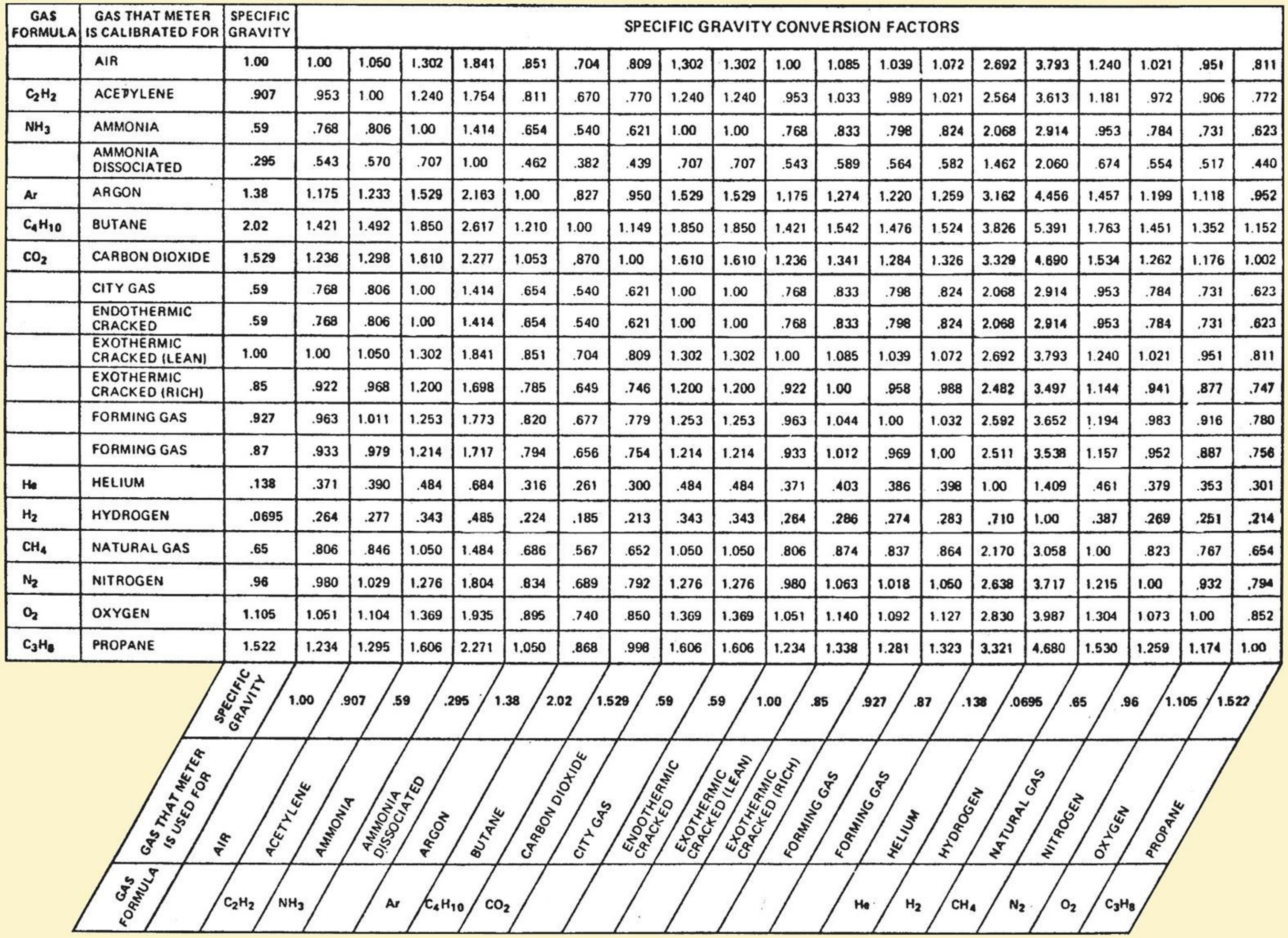


Fig. 2. Flowmeter types (a) Endothermic gas FurnaceMeter (Courtesy of Atmosphere Engineering Company); (b) Mass-flow controller (Courtesy of MKS Instruments)

- Instrument specifications can be changed by field replacement of the slotted tube and float, without having to repipe the flowmeter body
- Ability to handle high flows and pressures
- Improved immunity to the effects of pulsating flows, with no minimum back-pressure

Limitations common to both tapered-tube and slotted-cylinder variable-area flowmeters are that vertical mounting is required and that they contain moving parts.

The user should also be aware that the accuracy of mass flowmeters and mass-flow controllers is determined by two factors: flow calibration and repeatability. Proper instrument calibration ensures



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|---|---|---|---|---|---|---|---|---|---|---|---|---|---|---|---|---|---|---|---|---|---|
| | AIR | 1.00 | 1.00 | 1.050 | 1.302 | 1.841 | .851 | .704 | .809 | 1.302 | 1.302 | 1.00 | 1.085 | 1.039 | 1.072 | 2.692 | 3.793 | 1.240 | 1.021 | .951 | .811 |
| $C_2H_2$ | ACETYLENE | .907 | .953 | 1.00 | 1.240 | 1.754 | .811 | .670 | .770 | 1.240 | 1.240 | .953 | 1.033 | .989 | 1.021 | 2.564 | 3.613 | 1.181 | .972 | .906 | .772 |
| $NH_3$ | AMMONIA | .59 | .768 | .806 | 1.00 | 1.414 | .654 | .540 | .621 | 1.00 | 1.00 | .768 | .833 | .798 | .824 | 2.068 | 2.914 | .953 | .784 | .731 | .623 |
| | AMMONIA DISSOCIATED | .295 | .543 | .570 | .707 | 1.00 | .462 | .382 | .439 | .707 | .707 | .543 | .589 | .564 | .582 | 1.462 | 2.060 | .674 | .554 | .517 | .440 |
| Ar | ARGON | 1.38 | 1.175 | 1.233 | 1.529 | 2.163 | 1.00 | .827 | .950 | 1.529 | 1.529 | 1.175 | 1.274 | 1.220 | 1.259 | 3.162 | 4.456 | 1.457 | 1.199 | 1.118 | .952 |
| $C_4H_{10}$ | BUTANE | 2.02 | 1.421 | 1.492 | 1.850 | 2.617 | 1.210 | 1.00 | 1.149 | 1.850 | 1.850 | 1.421 | 1.542 | 1.476 | 1.524 | 3.826 | 5.391 | 1.763 | 1.451 | 1.352 | 1.152 |
| $CO_2$ | CARBON DIOXIDE | 1.529 | 1.236 | 1.298 | 1.610 | 2.277 | 1.053 | .870 | 1.00 | 1.610 | 1.610 | 1.236 | 1.341 | 1.284 | 1.326 | 3.329 | 4.690 | 1.534 | 1.262 | 1.176 | 1.002 |
| | CITY GAS | .59 | .768 | .806 | 1.00 | 1.414 | .654 | .540 | .621 | 1.00 | 1.00 | .768 | .833 | .798 | .824 | 2.068 | 2.914 | .953 | .784 | .731 | .623 |
| | ENDOTHERMIC CRACKED | .59 | .768 | .806 | 1.00 | 1.414 | .654 | .540 | .621 | 1.00 | 1.00 | .768 | .833 | .798 | .824 | 2.068 | 2.914 | .953 | .784 | .731 | .623 |
| | EXOTHERMIC CRACKED (LEAN) | 1.00 | 1.00 | 1.050 | 1.302 | 1.841 | .851 | .704 | .809 | 1.302 | 1.302 | 1.00 | 1.085 | 1.039 | 1.072 | 2.692 | 3.793 | 1.240 | 1.021 | .951 | .811 |
| | EXOTHERMIC CRACKED (RICH) | .85 | .922 | .968 | 1.200 | 1.698 | .785 | .649 | .746 | 1.200 | 1.200 | .922 | 1.00 | .958 | .988 | 2.482 | 3.497 | 1.144 | .941 | .877 | .747 |
| | FORMING GAS | .927 | .963 | 1.011 | 1.253 | 1.773 | .820 | .677 | .779 | 1.253 | 1.253 | .963 | 1.044 | 1.00 | 1.032 | 2.592 | 3.652 | 1.194 | .983 | .916 | .780 |
| | FORMING GAS | .87 | .933 | .979 | 1.214 | 1.717 | .794 | .656 | .754 | 1.214 | 1.214 | .933 | 1.012 | .969 | 1.00 | 2.511 | 3.538 | 1.157 | .952 | .887 | .756 |
| He | HELIUM | .138 | .371 | .390 | .484 | .684 | .316 | .261 | .300 | .484 | .484 | .371 | .403 | .386 | .398 | 1.00 | 1.409 | .461 | .379 | .353 | .301 |
| $H_2$ | HYDROGEN | .0695 | .264 | .277 | .343 | .485 | .224 | .185 | .213 | .343 | .343 | .264 | .286 | .274 | .283 | .710 | 1.00 | .387 | .269 | .251 | .214 |
| $CH_4$ | NATURAL GAS | .65 | .806 | .846 | 1.050 | 1.484 | .686 | .567 | .652 | 1.050 | 1.050 | .806 | .874 | .837 | .864 | 2.170 | 3.058 | 1.00 | .823 | .767 | .654 |
| $N_2$ | NITROGEN | .96 | .980 | 1.029 | 1.276 | 1.804 | .834 | .689 | .792 | 1.276 | 1.276 | .980 | 1.063 | 1.018 | 1.060 | 2.638 | 3.717 | 1.215 | 1.00 | .932 | .794 |
| $O_2$ | OXYGEN | 1.105 | 1.051 | 1.104 | 1.369 | 1.935 | .895 | .740 | .850 | 1.369 | 1.369 | 1.051 | 1.140 | 1.092 | 1.127 | 2.830 | 3.987 | 1.304 | 1.073 | 1.00 | .852 |
| $C_3H_8$ | PROPANE | 1.522 | 1.234 | 1.295 | 1.606 | 2.271 | 1.050 | .868 | .998 | 1.606 | 1.606 | 1.234 | 1.338 | 1.281 | 1.323 | 3.321 | 4.680 | 1.530 | 1.259 | 1.174 | 1.00 |
| | | SPECIFIC GRAVITY | 1.00 | .907 | .59 | .295 | 1.38 | 2.02 | 1.529 | .59 | .59 | 1.00 | .85 | .927 | .87 | .138 | .0695 | .65 | .96 | 1.105 | 1.522 |
| | | GAS THAT METER IS USED FOR | AIR | ACETYLENE | AMMONIA | AMMONIA DISSOCIATED | ARGON | BUTANE | CARBON DIOXIDE | CITY GAS | ENDOTHERMIC CRACKED | EXOTHERMIC CRACKED (LEAN) | EXOTHERMIC CRACKED (RICH) | FORMING GAS | FORMING GAS | HELIUM | HYDROGEN | NATURAL GAS | NITROGEN | OXYGEN | PROPANE |
| | | GAS FORMULA | | $C_2H_2$ | $NH_3$ | | Ar | $C_4H_{10}$ | $CO_2$ | | | | | | | He | $H_2$ | $CH_4$ | $N_2$ | $O_2$ | $C_3H_8$ |

Table 1. Specific-gravity conversion factors (Courtesy of Waukee Engineering Company)

## Table 2. Temperature conversion factors

| Temperature, ˚F (˚C) | Scale multiplier |
|---|---|
| 50 (10.0) | 1.02 |
| 60 (15.6) | 1.01 |
| 70* (21.0) | 1.00 |
| 80 (26.7) | .99 |
| 90 (32.2) | .98 |
| 100 (37.8) | .97 |
| 110 (43.3) | .965 |
| 120 (48.9) | .96 |
| 130 (54.4) | .95 |
| 140 (60.0) | .94 |
| 150 (65.6) | .93 |

* Flowmeter calibrated for 70˚F (21˚C)

starting-point accuracy. Repeatability is the measure of continuous performance-to-specification over the lifetime of the device. Most mass flowmeters and mass-flow controllers have an accuracy of ±1% of full scale and a repeatability of ±0.25% of full scale.

## Is it easier to control a gas or a liquid?

Interestingly, liquids are easier to measure and control because of their small compressibility. For most volumetric-flow applications, the incoming pressure in liquid systems does not need to be closely controlled. By their very nature, liquids can be captured easily and measured to a high degree of accuracy. Gases on the other hand, because of their compressibility, require more complex sensing and control methods.

## What is the accuracy range of a gas flowmeter?

When measuring gas flows in heat-treating applications there is an important distinction between the operating range of a flowmeter and the design range when purchasing a new meter. Plan to operate a flowmeter in a range not below 25% and not above 90% of the flowscope's scale capacity. In other words, if your flowmeter is rated for 0-2,000 cubic feet per hour (cfh), you can be assured of accuracy when the flow is between 500 cfh and 1,800 cfh. Flows outside these limits are not considered accurate.

A good "rule of thumb" for sizing a flowmeter is to purchase your meter "in the middle third." The flowmeter should be chosen so that the actual flow will be no less than 1/3 and no higher than 2/3 of the scale you select. This gives you the ability in actual operation to compensate for unexpected changes in flow requirements that may occur. Often over the life of a heat-treating furnace, process requirements and operating conditions change – sometimes dramatically – and you want your gas measurement to remain accurate.

## Should I have my flowmeters recalibrated?

If a change of operating conditions is permanent, such as the desire to constantly operate at a different pressure, then recalibration of the flow measurement device is strongly recommended. As a rule, flowmeters used in heat-treating applications are designed for a maximum temperature of 150˚F (66˚C) and an operating pressure up to 50 psig (345 kPa). However, application-specific flowmeters have maximum operating pressures outside these ranges.

## What affects my gas measurements?

If knowing the proper flowrate is important to you, be aware that a change in temperature, pressure or specific gravity of the gas from that for which the meter was calibrated will cause a serious error in the

**Table 3. Pressure conversion factors**

| Pressure, psi (kPa) | Scale multiplier |
|---|---|
| 0.5* (3.4) | 1.00 |
| 1 (6.9) | 1.03 |
| 2 (13.8) | 1.06 |
| 3 (20.7) | 1.10 |
| 4 (27.6) | 1.13 |
| 5 (34.5) | 1.16 |
| 10 (69.0) | 1.30 |
| 15 (103.4) | 1.42 |
| 20 (137.9) | 1.53 |
| 30 (206.9) | 1.75 |
| 40 (275.8) | 1.93 |
| 50 (344.8) | 2.10 |

*Flowmeters are typically calibrated for 0.5 psi (3.4 kPa)

indicated scale reading. (These topics are covered in detail in the online portion of this column.) It is quite common in a heat-treat shop to find flowmeters operating at different pressures and temperatures than they have been calibrated for.

## Do I need to maintain my flow devices?

All flowmeters eventually require maintenance. It is a sad truth that some units require more maintenance than others, so this factor should be considered when a unit is selected. However, in most heat-treating operations the equipment manufacturer has already made that choice for you, so understanding what maintenance is required and when it should be performed is of paramount importance.

Flowmeters have moving parts and require internal inspection, especially if the fluid is dirty or viscous. For example, in furnaces using endothermic gas, the flowmeters often become contaminated with soot (carbon) and must be cleaned by CAREFULLY disassembling the flowmeter and cleaning all internal moving parts as well as replacing the dirty fluid in the flowmeter tube. Caution: This involves isolating the flowmeter, or performing maintenance when the unit is shut down, and must be done in a safe manner as many of the gases involved are asphyxiants as well as being flammable, toxic and possibly life-threatening.

Remember also that electromagnetic flowmeters and all flow measurement devices that use secondary instruments such as pressure sensors to actuate a control valve or send a signal to a remote source must be periodically inspected, calibrated, repaired and/or replaced. Improper location of the flowmeter itself, the secondary sensor or readout devices can result in measurement errors and hidden costs.

### Do I really need to learn about my flowmeters to be in control, stay in control, operate safely and keep the cost of my operation as low as possible?

Simply stated, yes. Hopefully, this discussion has helped reinforce this idea. Now go out today and check your flow devices!

### What do I need to consider if I want to use a flowmeter for one gas, but it is calibrated for another?

One of the most common problems seen in heat-treat shops is that operating personnel and supervisors are unaware of the consequences of a flowmeter that has been calibrated for use with one gas while having another gas flowing through it. When switching gases, the change in specific gravity of the two gases is the principle factor that must be taken into account. Specific gravity is the ratio of the density of the gas under consideration to the density of dry air (at standard temperature and pressure, 77˚F (25˚C) and 14.7 psi).

Table 1 provides a quick conversion chart. To calculate the actual flow rate of gas being metered, multiply the indicated scale reading of the flowmeter by the factor shown in the table.

Equation (1) allows us to calculate the actual flow when a change in specific gravity occurs.

$$Fa = Fi \times \sqrt{\frac{SG_1}{SG_2}} \qquad (1)$$

Where:
Fa = actual flow
Fi = flow indicated scale reading
SG2 = specific gravity of gas to be used in the flowmeter
SG1 = nameplate (calibrated) specific gravity

### How do I compensate for changes in pressure and temperature?

A change in temperature and/or pressure of the gas from for which the meter was calibrated will cause a serious error in the indicated scale reading. However, there is an easy way to calculate the effect of these changes.

### Temperature Compensation

Equation (2) allows us to calculate the actual flow when a change in temperature occurs.

$$Fa = Fi \times \sqrt{\frac{T_1}{T_2}} \qquad (2)$$

Where:
Fa = actual flow
Fi = flow indicated scale reading
T1 = nameplate (calibrated) temperature + 460 (English system) or nameplate (calibrated) temperature + 273 (metric system)
T2 = new temperature + 460 (English system) or new temperature + 273 (metric system)

A summary for some common values is shown in Table 2.

## References

1. "North American Combustion Handbook, 2nd Edition," North American Manufacturing Company, Cleveland, OH.
2. Braziunas, Vytas and Daniel H. Herring, "A Flowmeter Primer," Heat Treating Progress, March/April 2004.
3. Mr. Eric Jossart, Atmosphere Engineering Company, private correspondence.

# Simulation Software (Part One: Atmosphere Carburizing)

One of the Doctor's many New Year's resolutions is to convince every heat treater to purchase and use simulation software for recipe creation and to aid in process control and troubleshooting. The time has come – the technology is proven and robust. Today, we will talk about simulators and controls for atmosphere carburizing. In our March 2018 column (since February's focus is on nonferrous heat treatment), we will talk about low-pressure carburizing simulators. Then in a follow-up column, we will introduce you to nitriding/ferritic nitrocarburizing simulators. Let's learn more.

## Why Simulators?

We all know how important precise furnace atmosphere control is to successful heat treating. Meeting or exceeding customer specifications and producing both predictable and repeatable metallurgical and mechanical property results are the goal of every heat-treat operation. Nowhere is this more critical than in carburizing and other case-hardening processes where we deliberately change the composition of the furnace atmosphere at various points in the cycle. In addition, changes to material composition, part loading and/or process parameters (time, temperature, carbon potential) influence the final results.

In gas carburizing, the addition of carbon at the part surface followed by diffusion allows us to achieve both the desired case depth and hardness. Measurement and control of furnace atmospheres are often performed continuously using in-situ (oxygen probe) devices or via extractive (dew point or infrared) methods. A combination of techniques is often used.

Simulation and modeling software allow us to predict and determine in real time the carbon profile. Taking this one step further, one can use this type of software to control the process by utilizing the atmosphere inputs and varying the time to achieve the desired case depth and carbon profile at any given temperature. Prediction of hardness profiles is just an algorithm away.

## Historical Overview

Years ago, the only effective way of trying to control the carburizing process was to establish a relationship between the volume of enriching gas to that of the carrier gas. This led to rules of thumb such as adding natural gas at 10% of the endothermic gas flow for

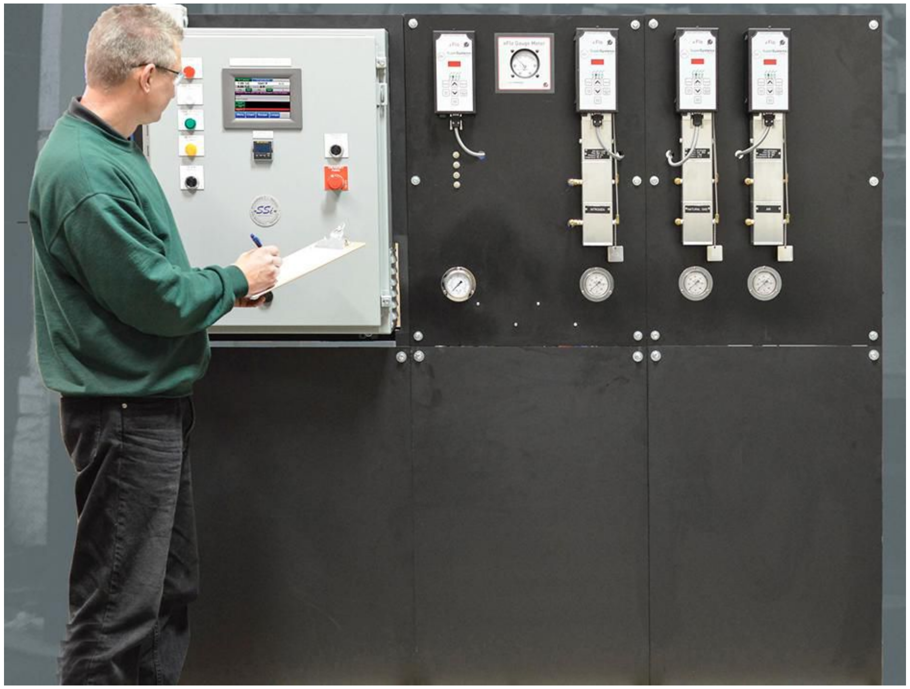


Fig. 1. Typical vacuum furnace control system (courtesy of Super Systems, Inc.)

carburizing or adding ammonia additions at 3-5% of the endothermic gas flow when carbonitriding. This was followed by techniques involving measurement of the carbon potential using either dew point or periodic shim-stock measurements.

These methods were common up until the early 1970s, when the first oxygen (carbon) probes were introduced. Once the zirconia carbon-sensor technology was proven robust, the technology swept the industry. Over the years, this measurement method has been refined, first by introducing a sensor correction factor and then using three-gas (CO, CO2 and CH4) infrared analysis in conjunction with the oxygen probe to input a more accurate carbon setpoint. It then evolved further with the introduction of continuous, nondispersive infrared analyzers that measure continuously rather than periodically, allowing for automatic operation.

Today, coil tests are being used to simplify shim-stock verification methods. Finally, true representation of atmosphere was achieved using metallurgical evaluation of parts in combination with the aforementioned methods. From all these results, proper correction to the carbon calculation can be determined, yielding more accuracy on the in-situ control parameter from the oxygen probe.

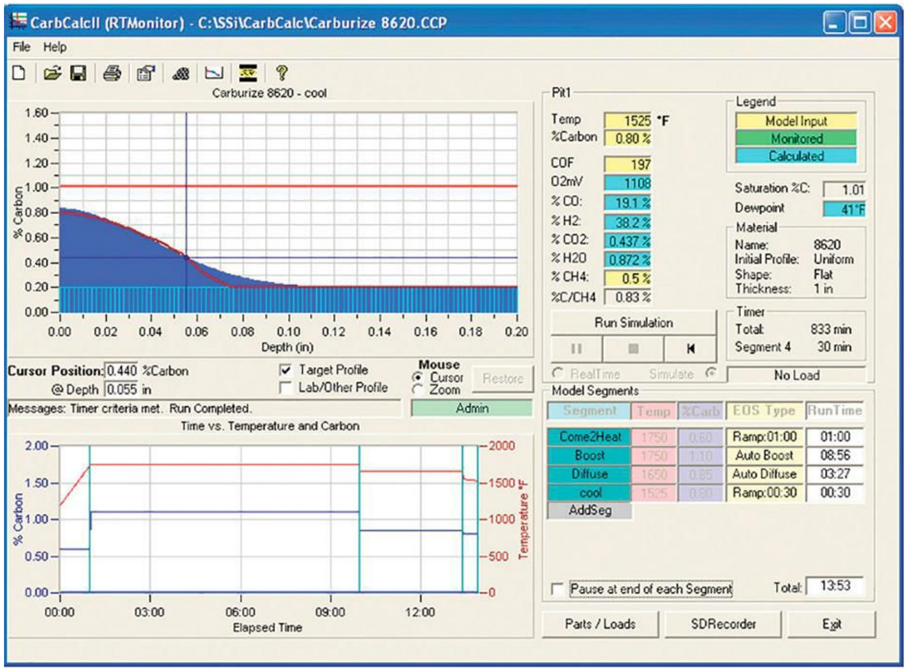
While modeling/simulation of the carburizing process began in earnest in the 1970s, it wasn't until the advent of more powerful, smaller computers and their industrialization that programs emerged to allow complex calculations in real time. However, these tools only became of interest to the heat-treat industry over the past two decades as demands for higher quality and tighter control became paramount.

One of the lessons learned was that calculating the carbon potential for gas carburizing requires assumptions with regard to material chemistry, base atmosphere composition, temperature and changes to the atmosphere over time. The composition of the base atmosphere and the way in which the gas is measured were found to have significant effects on the accuracy of the calculated carbon potential.

Today, many control methods are time-based with changes to carbon and temperature setpoints. Due to the variable nature of the process, however, this method does not provide the necessary accuracy of control or repeatability of results. In addition, it is common to be conservative ("err on the safe side") when providing a time for segments of the process. Simulators and their associated control systems (Fig. 1) optimize the carburizing process.

## How to Optimize Results

The best way in which to utilize a predictive model in real-time control is to ensure that the variables accurately represent the conditions to which the parts are exposed; to build confidence by running data-logged values through the modeling software; and to compare those values to metallurgical results. Using this three-step process of building, verifying and controlling allows for accuracy in method and confidence in results. "What-if" analysis is another important aspect of these programs, allowing different inputs (e.g., time, temperature and boost-diffuse steps) to be explored.



Cursor Position: 0.440 %Carbon
@ Depth 0.055 in

☑ Target Profile
☐ Lab/Other Profile

○ Cursor
● Zoom

Messages: Timer criteria met. Run Completed.

Temp 1525 °F
%Carbon 0.80 %
COF 197
O2mV 1108
% CO: 19.1 %
% H2: 38.2 %
% CO2: 0.437 %
% H2O 0.872 %
% CH4: 0.5 %
%C/CH4 0.83 %

Saturation %C: 1.01
Dewpoint 41°F

Material
Name: 8620
Initial Profile: Uniform
Shape: Flat
Thickness: 1 in

Timer
Total: 833 min
Segment 4 30 min

No Load

Model Segments

| Segment | Temp | %Carb | EOS Type | RunTime |
|---|---|---|---|---|
| Come2Heat | 1750 | 0.60 | Ramp:01:00 | 01:00 |
| Boost | 1750 | 1.10 | Auto Boost | 08:56 |
| Diffuse | 1650 | 0.85 | Auto Diffuse | 03:27 |
| cool | 1525 | 0.80 | Ramp:00:30 | 00:30 |
| AddSeg | | | | |

☐ Pause at end of each Segment
Total: 13:53

Fig. 2. CarbCalc II simulator screen shot (courtesy of Super Systems, Inc.)

## Atmosphere Carburizing Simulator Example

One simulation program on the market is CarbCalcII (Super Systems Inc.). This control software package is capable of not only predicting the carbon profile developed during atmosphere carburizing, it can be used for real-time monitoring, real-time control, process replay modes and what-if simulation. It includes the capability to predict the carbon-transfer rate between the furnace atmosphere and steel during boost-diffuse carburizing or steady-state (constant carbon-potential) carburizing and during temperature drops and stabilization at hardening temperature. The ease of use, recipe development and ability to "tweak" the cycle are key advantages of the software.

Multiple screens and operating modes (Fig. 2) allow the user to input key process variables (including material chemistry and atmosphere composition), create multiple segment recipes, view the impact on results and view the carbon profile (actual versus desired). For example, the software comes with an alloy database that includes the most common steels used in heat treating. Within the standard materials is the ability to change an element's chemistry (e.g., the ability to input the actual heat carbon content). It also allows the user to create custom chemistries to support new or foreign alloys that are becoming more common today.

In real-time and replay modes, the output allows users to display the carbon profile using data retrieved from the control instrumentation while process control runs in the background. Carbon profiles can be altered, and the program will modify the required time at temperature based on the actual carbon profile received from the control instrumentation. In addition, there is a load-tracking system to enable historical information on previously processed loads to be quickly retrieved and profiles re-created or modified.

## Summary

Atmosphere carburizing simulators and similar predictive control tools help heat treaters do their jobs more efficiently, with better control, less operator intervention and at lower cost. In addition, they simplify record keeping and provide the type of documentation demanded by today's manufacturing community. If you or your heat treater atmosphere carburize, make a promise to yourself and your company to insist that simulators are part of the process. Simply stated, it will improve quality and keep our industry competitive.

## References

1. Herring, Daniel H., Atmosphere Heat Treatment, Volume II, BNP Media, 2015
2. Oakes, Jim, "Understanding Atmosphere in Carburizing Applications Using Simulation and Real-Time Carbon Diffusion," Conference Proceedings, Thermal Process Modeling and Computer Simulation Conference, ASM International, 2014
3. "Carbon Diffusion Model for Atmosphere Furnaces," CarbCalc II Operations Manual, Super Systems, Inc., 2017

# What is Vacuum?

When we talk in terms of heat treating in vacuum, most people think we do so in a space entirely devoid of matter. In reality, this isn't true. In practical terms then, a vacuum is a space with a highly reduced gas density. Just how many gas molecules are still present and how they react inside the vacuum furnace is something we should better understand. Let's learn more.

## Common Vacuum Units

Pressure in vacuum applications is commonly measured in microns or Torr here in the U.S. or millibar (mbar) elsewhere. A Torr is 1/760th of atmospheric pressure. Atmospheric pressure, or the pressure exerted by the weight of air on a column of mercury, at standard temperature (0ºC) and pressure (sea level) is 29.9 inches or 760 Torr ($10^{13}$ mbar).

One of the mystifying things about vacuum and vacuum furnaces, especially in the heat-treating industry in the U.S., is the confusing way in which vacuum units are used. Devices installed on furnaces often measure in different units, which force us to think in terms of microns, Torr, inches of mercury, millibar, bar, microbar, Pascal, kilopascals, inches of water column and atmospheres (psia or psig)! This is extremely confusing, especially to those who are not familiar with vacuum terminology. To avoid confusion, try to stay with one unit of measure, converting everything to this common base. To help, the conversions between common vacuum units are shown in Table 1.

## A Little Vacuum Theory

Most of us know that a gas is a collection of molecules and that most gases are mixtures of many different kinds of molecules. Gas molecules are in constant motion. The higher the temperature, the faster these molecules move, and as one might expect, the motion of gas molecules dramatically slows down as a gas is cooled. With an increase in temperature, there is an increase in kinetic energy (or energy of motion). Molecular collisions occur between molecules. If enclosed, these molecular collisions also occur against the walls of their container, resulting in a pressure rise. In other words, pressure is simply the force per unit area that a gas exerts on the walls of its container.

Avogadro (1811) determined that a mole of any

**Table 1.** Common Vacuum Units

| Millibar (mbar) | Torr (mm Hg) | Microns | Inch of Hg Absolute | Atmo-spheres | psia | Millibar (mbar) | Torr (mm Hg) | Microns | Pascal | Microbars |
|---|---|---|---|---|---|---|---|---|---|---|
| 1013.25 | 760.00 | 760000 | 29.9 | 1 | 14.7 | 0.27 | 0.20 | 200 | 27.0 | 270.0 |
| 982.05 | 736.60 | 736600 | 29 | 0.97 | 14.2 | 0.13 | 0.10 | 100 | 13.0 | 130.0 |
| 948.19 | 711.20 | 711200 | 28 | 0.94 | 13.8 | 0.12 | 0.09 | 90 | 12.0 | 120.0 |
| 914.32 | 685.80 | 685800 | 27 | 0.90 | 13.3 | 0.11 | 0.08 | 80 | 11.0 | 110.0 |
| 880.46 | 660.40 | 660400 | 26 | 0.87 | 12.8 | 0.09 | 0.07 | 70 | 9.0 | 90.0 |
| 846.60 | 635.00 | 635000 | 25 | 0.84 | 12.3 | 0.08 | 0.06 | 60 | 8.00 | 80.0 |
| 812.73 | 609.60 | 609600 | 24 | 0.80 | 11.8 | 0.07 | 0.05 | 50 | 7.0 | 70.0 |
| 778.87 | 584.20 | 584200 | 23 | 0.77 | 11.3 | 0.05 | 0.04 | 40 | 5.0 | 50.0 |
| 745.00 | 558.80 | 558800 | 22 | 0.74 | 10.8 | 0.04 | 0.03 | 30 | 4.0 | 40.0 |
| 711.14 | 533.40 | 533400 | 21 | 0.70 | 10.3 | 0.03 | 0.02 | 20 | 3.0 | 30.0 |
| 677.28 | 508.00 | 508000 | 20 | 0.67 | 9.8 | 0.013 | 0.01 | 10 | 1.3 | 13.0 |
| 643.41 | 482.60 | 482600 | 19 | 0.64 | 9.3 | 0.012 | 0.009 | 9 | 1.2 | 12.0 |
| 609.55 | 457.20 | 457200 | 18 | 0.60 | 8.8 | 0.011 | 0.008 | 8 | 1.1 | 11.0 |
| 575.69 | 431.80 | 431800 | 17 | 0.57 | 8.3 | 0.009 | 0.007 | 7 | 0.9 | 9.0 |
| 541.82 | 406.40 | 406400 | 16 | 0.53 | 7.8 | 0.008 | 0.006 | 6 | 0.8 | 8.0 |
| 507.96 | 381.00 | 381000 | 15 | 0.50 | 7.4 | 0.007 | 0.005 | 5 | 0.7 | 7.0 |
| 474.09 | 355.60 | 355600 | 14 | 0.47 | 6.9 | 0.005 | 0.004 | 4 | 0.5 | 5.0 |
| 440.23 | 330.20 | 330200 | 13 | 0.43 | 6.4 | 0.004 | 0.003 | 3 | 0.4 | 4.0 |
| 406.37 | 304.80 | 304800 | 12 | 0.40 | 5.9 | 0.003 | 0.0020 | 2 | 0.3 | 3.0 |
| 372.50 | 279.40 | 279400 | 11 | 0.37 | 5.4 | 0.0013 | 0.0010 | 1 | 0.13 | 1.3 |
| 338.64 | 254.00 | 254000 | 10 | 0.33 | 4.9 | 0.0012 | 0.0009 | 0.9 | 0.12 | 1.2 |
| 304.78 | 228.60 | 228600 | 9 | 0.30 | 4.4 | 0.0011 | 0.0008 | 0.8 | 0.11 | 1.1 |
| 270.91 | 203.20 | 203200 | 8 | 0.27 | 3.9 | 0.0009 | 0.0007 | 0.7 | 0.09 | 0.9 |
| 237.04 | 177.80 | 177800 | 7 | 0.23 | 3.4 | 0.0008 | 0.0006 | 0.6 | 0.08 | 0.8 |
| 203.18 | 152.40 | 152400 | 6 | 0.20 | 2.9 | 0.0007 | 0.0005 | 0.5 | 0.07 | 0.7 |
| 169.32 | 127.00 | 127000 | 5 | 0.17 | 2.4 | 0.0005 | 0.0004 | 0.4 | 0.05 | 0.5 |
| 135.46 | 101.60 | 101600 | 4 | 0.13 | 2.0 | 0.0004 | 0.0003 | 0.3 | 0.04 | 0.4 |
| 101.59 | 76.20 | 76200 | 3 | 0.10 | 1.5 | 0.0003 | 0.0002 | 0.2 | - | |
| 67.73 | 50.80 | 50800 | 2 | 0.066 | 0.98 | 0.00013 | 0.00010 | 0.1 | - | |
| 33.87 | 25.40 | 25400 | 1 | 0.033 | 0.49 | 0.00012 | 0.00009 | 0.09 | - | |
| 1.33 | 1.00 | 1000 | 0.039 | 0.0013 | 0.019 | 0.00011 | 0.00008 | 0.08 | - | |
| 1.20 | 0.90 | 900 | 0.035 | 0.0011 | 0.017 | 0.00009 | 0.00007 | 0.07 | - | |
| 1.07 | 0.80 | 800 | 0.031 | 0.0010 | 0.015 | 0.0000800 | 0.00006 | 0.06 | - | |
| 0.93 | 0.70 | 700 | 0.028 | 0.0009 | 0.013 | 0.0000667 | 0.00005 | 0.05 | - | |
| 0.80 | 0.60 | 600 | 0.024 | 0.0008 | 0.012 | 0.0000533 | 0.00004 | 0.04 | - | |
| 0.67 | 0.50 | 500 | 0.020 | 0.0006 | 0.010 | 0.0000400 | 0.00003 | 0.03 | - | |
| 0.53 | 0.40 | 400 | 0.016 | 0.0005 | 0.008 | 0.0000267 | 0.00002 | 0.02 | - | |
| 0.40 | 0.30 | 300 | 0.012 | 0.0004 | 0.006 | 0.0000133 | 0.00001 | 0.01 | - | |

**Table 2. Classification of Vacuum**

| Quality of vacuum | Pressure range, Torr (mbar) |
|---|---|
| Rough | 760 to 1 (1013 to 1.33) |
| Fine | 1 to 1 x $10^{-3}$ (1.33 to 1.33 x $10^{-3}$) |
| High | 1 x $10^{-3}$ to 1 x $10^{-7}$ (1.33 x $10^{-3}$ to 1.33 x $10^{-7}$) |
| Ultra-High | < 1 x $10^{-7}$ (< 1.33 x $10^{-7}$) |

gas occupies a volume of 22.4 liters and contains 6.02 x $10^{23}$ molecules at standard temperature and pressure. To create a vacuum in any closed vessel, therefore, some of the gas molecules must be removed.

Now for a surprise and an important concept. At atmospheric pressure, one cubic centimeter of air contains approximately 2.69 x $10^{19}$ molecules all moving around in a random motion. As you might expect, this results in a fantastic number of collisions. In other words, the mean free path between molecules (or the average distance a molecule can travel before colliding with another molecule) is only about 2.6 x $10^{-6}$ inches. So if we pump a one-cubic-centimeter volume down to a micron (1 x $10^{-3}$ Torr), which is a vacuum level commonly used in heat treating, we still have about 3.54 x $10^{13}$ molecules, or well over half of them remaining! You might be wondering how this can be an acceptable condition for heat treating, especially when about 20% of those remaining molecules are oxygen? The answer is that the mean free path increases dramatically, reducing the probability of molecular collision with the surface of the workpiece.

If we continue to pump down to say a

thousandth of a micron (1 x $10^{-6}$ Torr), the level of a good diffusion-pumped vacuum furnace, the number of molecules per cubic centimeter is still 3.54 x $10^{10}$, but the mean free path increases to over 30 miles (48 km). The path of the molecules is ultimately limited by the walls of the vessel and not by the collisions between molecules. Flow as we know it doesn't exist. This is the reason for the relatively large opening and piping used for diffusion pumping systems. If the opening to the diffusion pump weren't that large, any molecules that migrated into the pump suction stream would rebound and move to other parts of the chamber. By comparison, at 200 miles above the earth, the vacuum is 1 x $10^{-8}$ Torr. At 400 miles it is 1 x $10^{-10}$ Torr, and in deep (or outer) space it is 1 x $10^{-16}$ Torr.

## What is a Good Vacuum?

Simply stated, the more molecules that are removed, the better the vacuum. The quality of a vacuum is described by the degree of reduction in gas density, i.e., gas pressure. One distinguishes four different vacuum levels or qualities as shown in Table 2. The heat treatment of steel is carried out in three of these - namely rough, fine and high. The majority of applications are processed in the fine vacuum range.

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# Vacuum Tools for Heat Treat Productivity


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# Gas Ballasting of Vacuum Pumps (Part 1)

The Doctor was called in because the patient was feeling poorly and was unable to go to work. On closer examination a milky, gassy, dark brown substance was found to be the culprit. Why, do you ask, is your vacuum furnace not producing acceptable parts? Perhaps part of the answer lies in the fact that your vacuum-pump oil is in need of gas ballasting. Let's learn more.

Gas ballasting is essential for oil-sealed pumps such as rotary-vane (i.e., mechanical) pumps, but it is often poorly understood or neglected as part of the daily maintenance routine on a vacuum furnace. As a matter of good practice, it should be done for about 20-30 minutes each day and is normally performed before processing parts (not once the furnace is running a load!).

Today, some vacuum pumps are provided with an automatic gas ballast feature, but even these features should never be taken for granted. Put simply, gas ballast is a means of allowing a vacuum pump to handle gases containing condensable vapors or moisture without contaminating the pump oil.

In order to properly discuss the use of gas ballast, it is helpful to review the purpose of the pump oil.

## Vacuum-Pump Oil – A Review

The oil in rotary-vane pumps serves several critical purposes. These pumps are designed with very tight physical clearances between the moving parts (the vanes or rotors) and the stationary parts (the pump housing or "stator"). A thin layer of oil is required in the gaps between these parts and acts as a seal, without which the pump could not generate a vacuum. In addition, the oil lubricates the sliding and rotating surfaces in what can be a very harsh environment of low pressure, high surface velocity and high

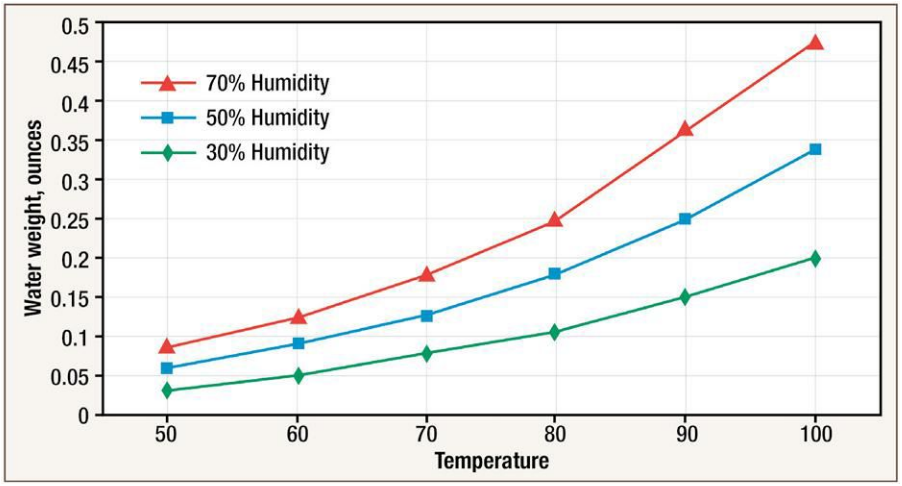

Fig. 1. Water vapor in humid air[4]

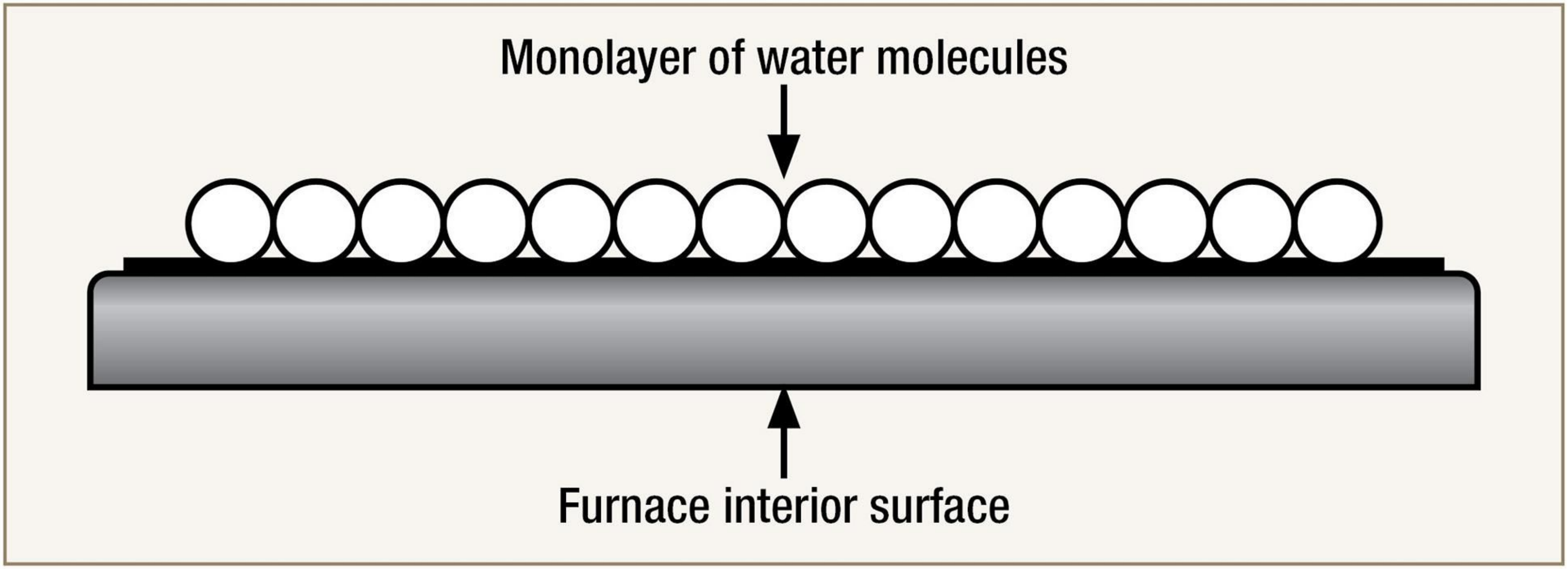

Fig. 2. Water molecules collect on interior furnace surfaces[5]

temperature.

The oil also cools the pump by removing heat from the areas where it is generated and allowing it to be dissipated in the oil reservoir. For these reasons, the viscosity, vapor pressure and other qualities of the oil greatly impact pump performance. It is also critical for the oil to remain uncontaminated. In the world of vacuum, however, this is easier said than done.

If the oil does become contaminated with moisture or other impurities, it cannot seal, lubricate or cool the pump. The result is a failure to achieve the required vacuum level in a reasonable period of time. If the pump continues to operate with contaminated oil, it can overheat and potentially seize up, causing downtime and requiring an expensive repair or rebuild.

## How does contamination of pump oil occur?

One of the chief causes of pump-oil contamination is moisture. When a vacuum chamber or furnace is opened and closed during the unloading and reloading

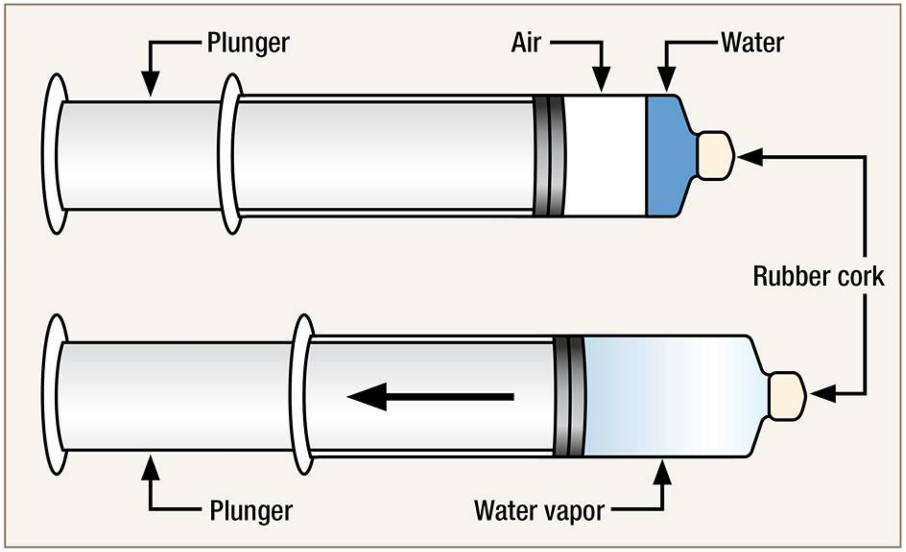

Fig. 3. Experiment to demonstrate vapor pressure[6] (adapted by the author)

sequence, moisture in the form of high-humidity air (Fig. 1) enters the chamber from the factory. In addition, moisture in the form of water may enter the furnace with the workload. This water vapor will attach to the interior chamber surfaces in the form of a monolayer, or single layer, of molecules (Fig. 2).

In the case of a vacuum furnace, the porous high-surface-area insulation can absorb a substantial amount of moisture in a very short time. The next time the chamber is put under vacuum, this water layer will form ice crystals on pump-down then evaporate into vapor as the furnace is heated and will be carried into the pump along with the pumped gas. Gas ballasting eliminates the water that winds up in the pump oil.

Water vapor is not the only contaminant that may be contained in the pumped gas. Solvent vapors from cleaning compounds or molecules from lubricants or greases found on the component parts placed into the vacuum furnace may also be released from the process, be carried in the pumped gas stream and enter the pump. Without gas ballast they will also mix with the pump oil and contaminate it, which destroys pumping efficiency.

## Vapor Pressure

The reason the moisture or solvent vapor mixes with the pump oil is related to the vapor pressure of the moisture or solvent. Vapor pressure is a property of all liquids that causes them to boil at any pressure below it and condense at any pressure above it. One can think of vapor pressure as the pressure required to maintain a liquid in liquid form. If the pressure in the environment drops below this threshold, boiling will occur.

The vapor pressure is also a function of temperature such that water, for example, will boil at room temperature at pressures below 24 mbar (18 Torr), which is the vapor pressure of water at room temperature.

Vapor pressure and its effect on boiling can be illustrated by a simple experiment using a syringe and a small amount of water (Fig. 3). First, pull back the plunger to draw a little water into the syringe. Then cap the open end of the syringe using a rubber cork so it is sealed from the environment. Next, quickly pull back on the plunger, reducing the pressure inside the syringe. The water will boil as soon as the pressure in the syringe drops below the vapor pressure of the water, even though it is at room temperature. Finally, move the plunger back in, and the water vapor will condense back into liquid water as soon as the pressure inside the syringe rises back above the vapor pressure of the water.

The same sequence followed in the syringe experiment occurs in a vacuum process. As a vacuum is drawn on a chamber, any residual water (or other volatile liquid) in the chamber boils into a vapor as soon as the chamber pressure drops below its vapor pressure. This water vapor, which is now held just barely above its vapor pressure, is drawn into the pump. After reaching the compression side of the pump, it is compressed above its vapor pressure, at which point it immediately condenses back into a liquid in the same way the water in the syringe condenses back into a liquid when the plunger is moved back in. This liquid in the pump then forms droplets that mix with the oil, causing the contamination. The gas ballast averts this by causing the outlet valve to open before the vapor condenses, and the vapor is discharged together with the ballast.

## Summary

Now that you better understand how vacuum-pump oil is contaminated, next time we will discuss how gas ballasting is performed and address its advantages and limitations.

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# Gas Ballasting of Vacuum Pumps (Part 2)

Before the holidays we talked about how vacuum-pump oil becomes contaminated. It is now time to talk about how to overcome this problem using gas ballasting – how it is performed, its advantages and limitations. Let's learn more.

## What is gas ballast?

Gas ballast is the introduction of a non-condensable gas (e.g., air or nitrogen) into a rotary-vane pump, during the compression stage to intentionally impact the efficiency of the pump thereby heating the oil inside and helping to drive out water and other condensed liquids present in the pump oil. In addition to rotary-vane pumps, it is also used in scroll pumps and piston pumps, to name a few. Wolfgang Gaede developed the gas ballast principle in 1935, which was first applied to rotary-vane pumps.

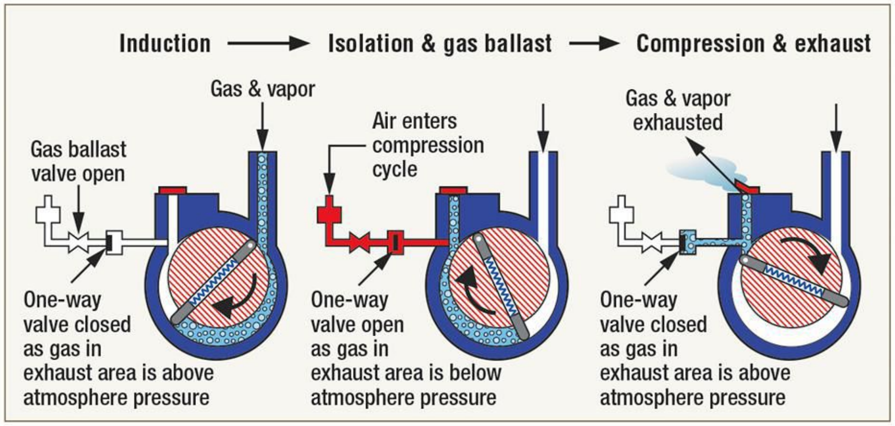

Fig. 1. The gas ballast sequence in a rotary-vane pump (courtesy American Vacuum Society)

The ballast gas is drawn into the pump chamber through a one-way valve (aka gas ballast valve) located on the pump. It is often said that the ballast gas is injected, but in actuality gas is being pulled into the pump by the rotating pump rotor, which produces reduced pressure inside the pump.

## How does gas ballast work?

Gas ballast prevents condensation of vapors inside the pump by diluting the pumped gas with the air being drawn in through the ballast valve. Once the air is drawn in and mixes with the pumped gas, the water vapor being carried in the pumped gas makes up a smaller percentage of the gas, which now includes the ballast gas (air) as well. The water vapor is then too diluted to condense into a liquid. Another way to say this is that the ballast gas dilutes the vapor in the pumped gas so that the partial pressure of the water never reaches its saturated value during compression.

The ballast is introduced at the beginning of the compression cycle. After injection starts and the ballast gas is drawn into the pump, the pump rotor continues to rotate, which increases the pressure generated in the pump. This forces the one-way ballast valve closed but not until sufficient dilution has occurred. As the rotor continues to turn, the pump discharge valve is forced open and discharges the mixture of pumped gas, ballast gas and water vapor (Fig. 1).

Although the point in the pump cycle at which the ballast gas is injected is on the compression side of the pump, it is still below atmospheric pressure prior to the ballast valve opening, which forces the ballast valve to open and allow the ballast gas to be drawn into the pump. Although the pump is compressing the pumped gas at this point, it is not compressing it above atmospheric pressure. If it were, the ballast valve would not open. This can be confusing because common terminology describing gas ballast refers to the ballast gas being injected on the compression side of the pump, when it could be more intuitively described as being pulled into the second stage of the pump.

The ballast valve is a one-way valve that closes as soon as the pump pressure reaches atmospheric pressure, which is the exact point at which the pump outlet valve opens. This forces the pumped gas to immediately start exiting the pump after the ballast is introduced and before condensation of the pumped moisture or other vapor can occur inside the pump.

Fig. 2. Gas ballast selector switch (courtesy Edwards Vacuum)

In addition to diluting the condensable water and other vapors, the gas ballast raises the temperature of the pumped gas by roughly 20°C (36°F), which also helps reduce condensation of vapors. Gas ballast is also used to decontaminate pump oil that has already been contaminated with condensed vapor, which can take several hours for severely contaminated pump oil. To prevent contamination during normal operation, pump manufacturers recommend running the pump with the inlet valve closed and the gas ballast on for

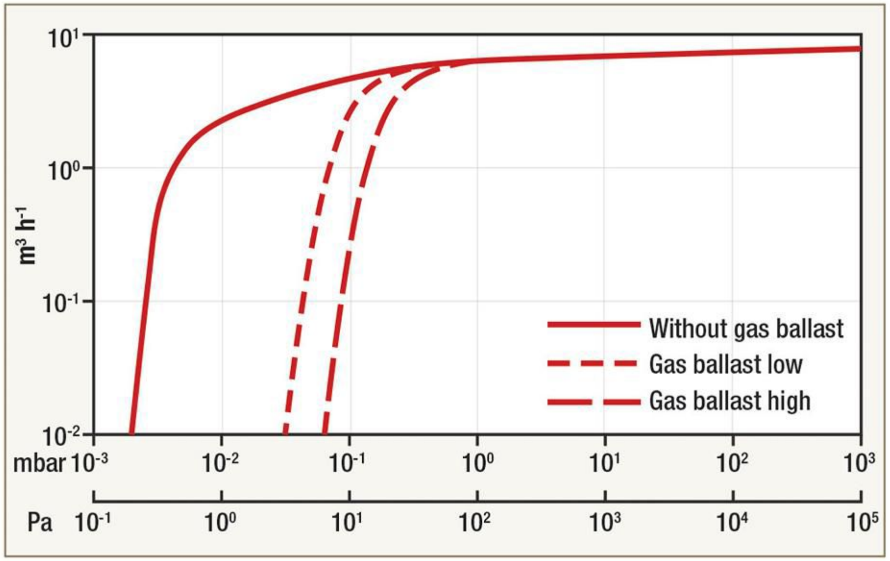

Fig. 3. Effect of gas ballast on pump performance (courtesy Edwards Vacuum)

20-30 minutes after every cycle. This removes residual condensed vapors from the oil after each operating cycle, preventing them from accumulating and causing contamination. However, this recommendation is not often followed in practice given production demands on the equipment.

Although air is the most common ballast gas due to its low cost, it is not used when the moisture, oxygen or hydrogen contained in the air would react with the process gases. Nitrogen is preferred in these cases. Although more expensive, it is more inert than air.

The amount of ballast gas is selectable on many of today's pumps, with a low flow and a high flow available (Fig. 2) or via a rotating knob to allow adjustment on a continuum as the knob is turned in or out. The negative effect of ballast on ultimate vacuum and oil loss is less in the low-flow mode than during high flow.

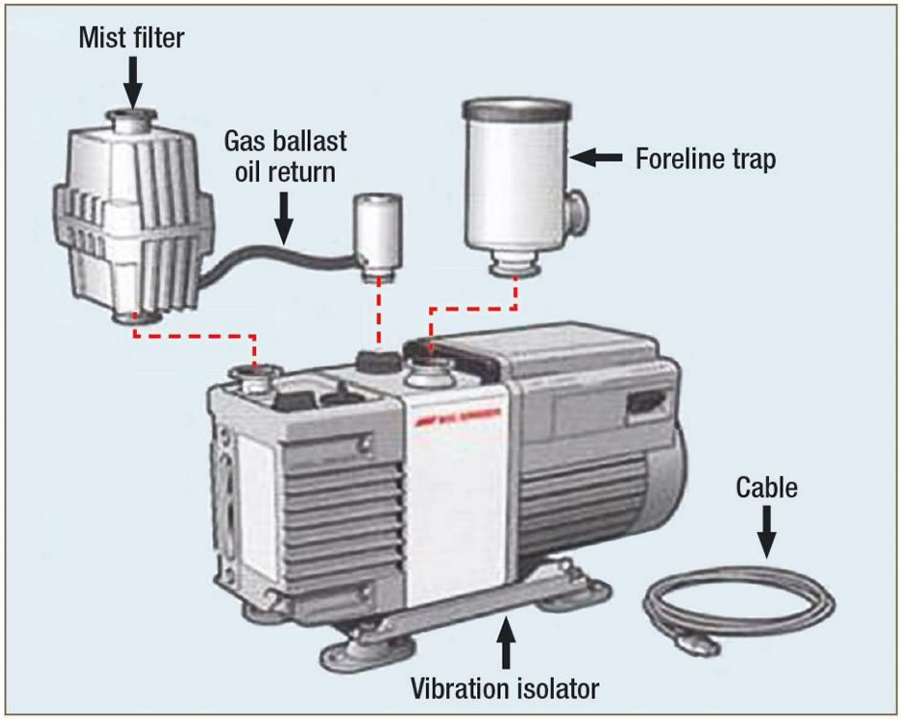

Fig. 4. Oil-mist filter and oil return line on a rotary-vane pump (courtesy Edwards Vacuum)

## Limitations

Although an essential tool for many applications, gas ballasting has several drawbacks that must be considered. The biggest is that it reduces the ultimate vacuum of the pump (Fig. 3) since the ballast gas negatively impacts the effectiveness of the pump by decreasing the pressure difference across the pump rotor. For this reason, the gas ballast valve should be closed in normal furnace operation. If it is not, the result can be failure to achieve the desired vacuum level for the process being run. Most pump manufacturers offer an automatic gas ballast feature, which avoids this problem but subjects the pump to ballasting even if no contamination exists.

Gas ballast also increases the rate of oil discharged from the pump. Although this is far more desirable than having it accumulate in the pump oil, measures must be taken to collect this oil to prevent its discharge from the plant. It is typically removed using either an oil-mist eliminator or coalescing filter (Fig. 4). When the discharged oil volume is high enough, it can be directed back into the pump for reuse via an oil return line.

## Summary

Gas ballasting is an integral part of the successful operation of any vacuum furnace and should be considered a routine part of daily operation. Many heat treaters ballast their vacuum pumps for 20-30 minutes at the beginning of each day while planning their schedules and preparing loads to be run. Make it part of your routine. You will be glad you did.

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# The Use of Residual-Gas Analyzers as a Vacuum Analysis Tool

Residual-gas analyzers are finding their way out into the heat-treat shop, primarily for process control and contamination monitoring in vacuum systems. Any tool that can help us better understand what is happening inside our vacuum furnaces is a welcome addition. Let's learn more.

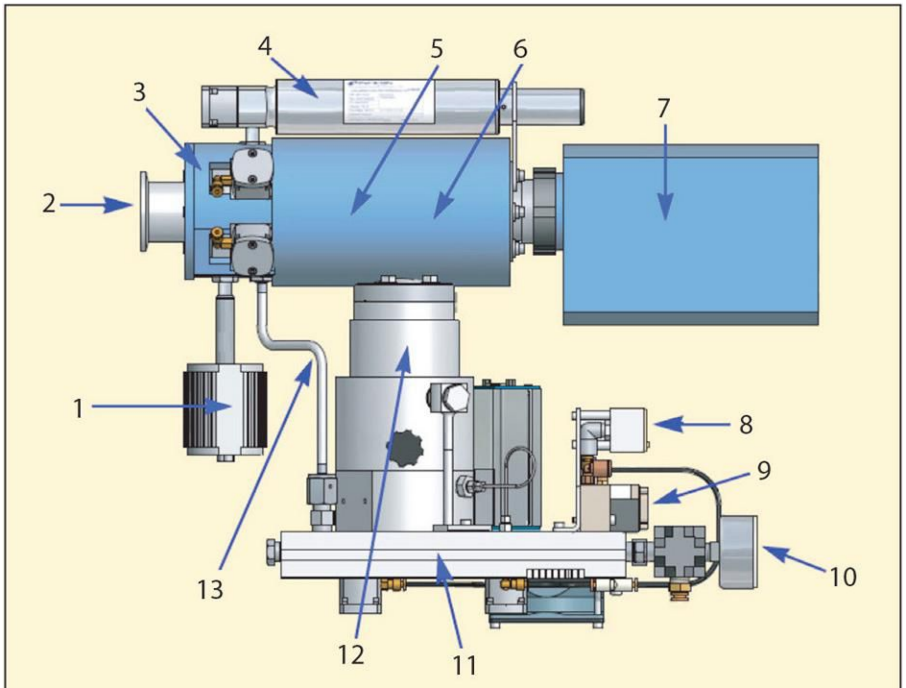
## What is an RGA?

A residual-gas analyzer, or RGA for short (Fig. 1), is a compact mass spectrometer. Originally designed for use only in the laboratory, the technology is robust enough to operate out on the shop floor. These devices are often mounted in-situ on a vacuum furnace (Fig. 2). A typical RGA analysis can reveal how much of a particular gas species is present either in the vacuum vessel or in the pump manifold (Fig. 3).

In many cases an RGA is used to monitor the quality of the vacuum and can easily detect minute traces of impurities, which possess sub-ppm detectability in the absence of background interferences in the low-pressure gas environment (down to $10^{-14}$ torr levels). Applications include finding both small and large leaks in a vacuum system, distinguishing leaks from outgassing, quantifying the process background and determining the effectiveness of gas-line purging.

Fig. 1. Residual-gas analyzers (courtesy of Infi con)
(a) Compact monitoring system (CPM) – differentially pumped RGA

An RGA can act as a helium leak detector (helium having an atomic mass unit of 4) but cannot measure the leak in a quantitative way (i.e., it is difficult to determine the speed of the



1. Process pressure gauge
2. Process connection (CF40, KF40 or KF25)
3. HexBlock™ inlet
4. Optional calibration reference
5. CPM sensor (inside manifold)
6. Sensor manifold and heater
7. CPM electronics module
8. Pressure switch
9. Valve solenoids
10. Nitrogen regulator
11. Integrated foreline block
12. UHV turbo molecular pump
13. Bypass connection

Fig. 1. (b) Typical RGA component parts

leak). With a vacuum system pumped down to lower than $10^{-5}$ torr, an RGA can check the integrity of the vacuum seals and the quality of the vacuum so as to detect air leaks, virtual leaks and other contaminants at low levels before a process is initiated.

Table 1. Mass values

| Mass number (major peak) | Constituent | Mass number (major peak) | Constituent |
|---|---|---|---|
| 43 | Acetone | 36, 37 | Hydrogenchloride |
| 28 | Air | 2 | Hydrogen |
| 45 | Alcohol,isopropyl | 4 | Helium |
| 31 | Alcohol, methyl | 39-43 | Hydrocarbons |
| 17 | Ammonia | 16 | Methane |
| 40 | Argon | 14, 28 | Nitrogen |
| 44 | Carbon dioxide | 32 | Oxygen |
| 28 | Carbonmonoxide | 29 | Propane |
| 35, 37 | Chlorine | 17, 18 | Water |

## Differentiating Leaks from Outgassing

The basic RGA scans from atomic mass units 2 to 50 (Table 1) and can often identify excessive outgassing (i.e., high peaks for water at mass 17 and 18), differentiating it from a leak in which we find high peaks at 14 and 28 (both nitrogen) and sometimes 32 (oxygen). Other indicators of an air leak are argon (40) and carbon dioxide (44). The oxygen peak is often missing or very low, even if there is an air leak.

Fig. 2. In-situ mounting of an RGA unit (courtesy of Vac Aero International)

## Fingerprinting

An RGA scan of a vacuum system prior to running a process can provide valuable insights into the condition of the vacuum environment. When problems occur (and they invariably do), having a baseline for comparative purposes is extremely helpful.

## How an RGA Works

The internals of an RGA (Fig.4) consist of an ion source, mass spectrometer and a measurement section. The residual gas is ionized when it collides with the electrons discharged from the high-temperature filament, and the ions that result accelerate and converge at the mass spectrometer. Direct and alternating current voltages in the mass spectrometer are applied to a series of cylindrical electrodes (quadropoles), which allow the ions to be separated by mass. The separated ions are detected as electric current by a Faraday cup with the ion current being proportional to the mass (partial pressure) of the residual gas.

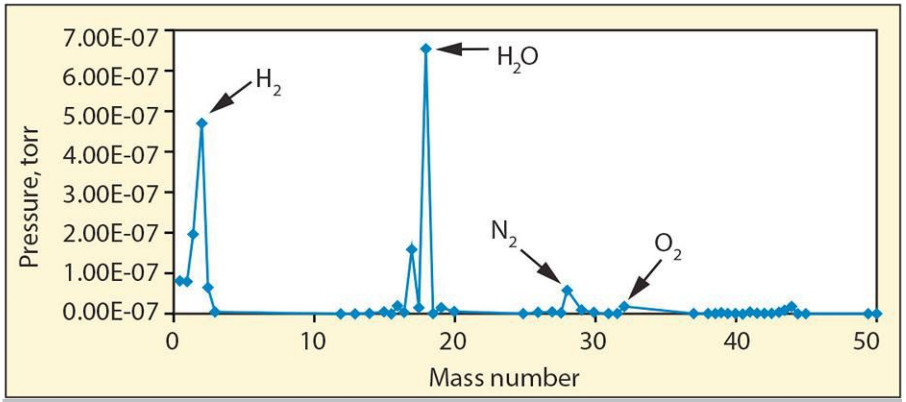


Fig. 3. Typical RGA analysis of a vacuum vessel (courtesy of Solar Atmospheres Inc.)

The operation of the RGA is quite simple, although the mathematics of the quadrupole mass analyzer section is complex. First, an "ionizer" converts

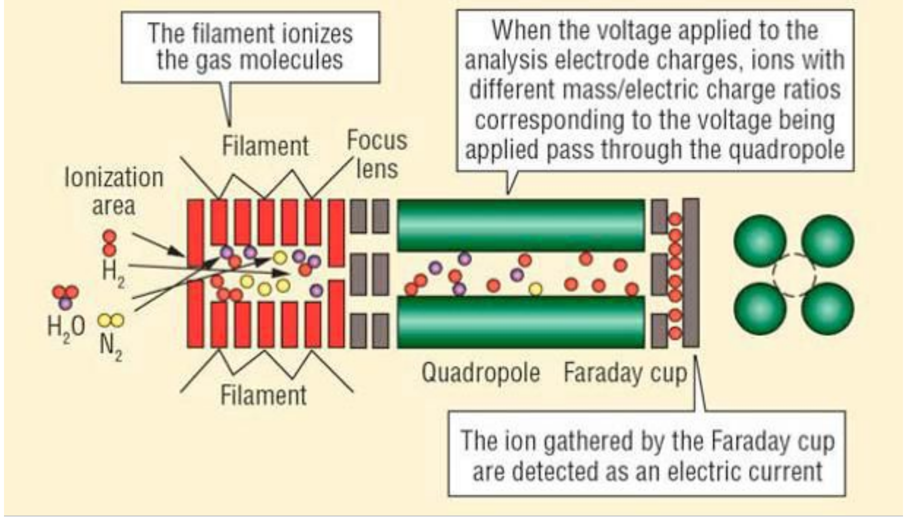


Fig. 4. Cross section of an RGA

many neutral gas molecules into positive ions in a well-controlled region at a specified electric potential. These ions are next accelerated by a series of electrostatic “lenses” and formed into a beam that has about 20 eV of energy. The ion beam is subsequently passed into the quadrupole mass analyzer region. This region acts as a filter. It will very nicely pass through ions with a user-chosen mass-to-charge ratio (M/e), but all other ions get pushed aside into walls, where they neutralize and become undetectable. The ions that are passed through this filter are detected as current either at a Faraday cup or using a secondary electron multiplier (also known as a “channeltron”). The channeltron gives a large amplification of the signal from ions and is consequently used to enhance the sensitivity of the RGA. By choosing a mass-to-charge ratio and making a measurement of the signal obtained, one can immediately find out the number of those molecules present in the ionizer region of the RGA. By sweeping through a whole range of M/e ratios, one can find a whole range of molecules that are present and begin to understand the full range of chemical components in the gas.

The actual analyzer is located in the vacuum and consists of the following principle components:

- An ion source ionizes neutral gas particles, which are then sorted in the mass filter on the basis of their mass-to-charge ratio (m/e ratio).
- The ion current is measured using a Faraday detector or a secondary electron multiplier (SEM) after the ions have left the separating system. The measured current is a parameter of the partial pressure of the respective gas molecules or a parameter of fractals that may possibly have been generated in the ion source.
- A data-analysis system processes the ion currents measured with the aid of the detector and presents these currents in various forms.

Today, data-analysis software programs are capable of supporting the user in interpreting mass spectra.

## Maintenance and Repair

Despite our best efforts, an RGA will need to be repaired. The heat-treat environment coupled with possible exposure to atmospheric pressure, pump oils or other contaminants are a fact of life. Filament replacement, ion-source cleaning and filter cleaning are examples of what can be done in the field. However, disassembly of certain components (such as the mass filter assembly) will result in costly and time-consuming factory repairs. An RGA is still a delicate instrument and should be treated as one.

## Final Thoughts

Residual gas analyzers can be a very effective tool to analyze system gas loads resulting from real leaks, virtual leaks or chamber-wall outgassing. RGAs have a number of advantages over traditional, dedicated gas leak detectors, including the ability to differentiate between different gas species and comparable sensitivity levels, the ability to detect internal or “virtual” leaks, and the ability to detect and analyze outgassing problems.

## Online exclusive:

For more discussion of how an RGA works and to reference figure 4, see our full online version at www.industrialheating.com/RGA.

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# Diffusion Bonding: The Process (Part 1)

One of the many vacuum processes to take note of today is that of diffusion bonding (aka diffusion welding or thermo-compression bonding). This technology, although highly specialized, continues to gain in popularity with design engineers and is finding more applications throughout manufacturing. Let's learn more.

## Processes

There are many types of diffusion bonds requiring a variety of thermal profiles (Fig. 1). These can be categorized as follows.

### Solid-State Diffusion Bonding

A solid-state bond can be defined as a method in which two or more mating parts are joined together without the use of an interface layer (i.e., without the application of a material between the parts applied by plating, sputtering, ion implanting or brazing or in the form of a foil). The resulting interface must be joined at or below the melting point of either parent material or any resulting eutectic that may form (i.e., $T_{bond} < T_{melting}$ point).

Typically, one sees a 50% or greater increase in grain size. The total part strain must be in the order of 2-4% to achieve a high-quality hermetic seal. The strain requirement means that considerable load (i.e., unit-normal force) must be applied to the bonding surfaces in order to achieve proper results.

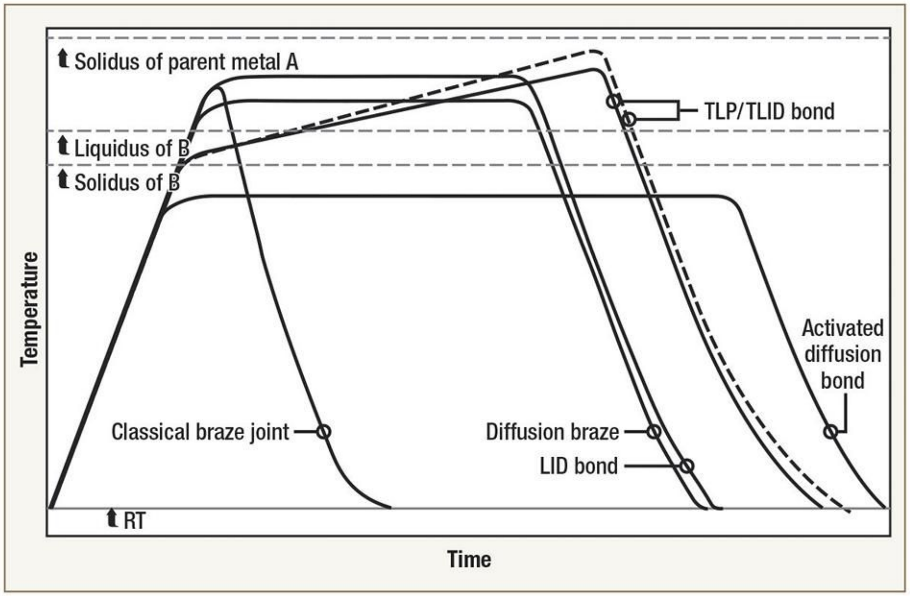

Fig. 1. Types of diffusion bonds and their thermal heating profiles

### Activated Diffusion Bonding

In the activated diffusion-bonding method, the surfaces to be bonded are coated with a solid material, typically having a smaller atomic diameter and higher vapor pressure than the parent material. The resulting bond is enhanced by the presence and diffusion/mass transport rate of this secondary material. The process is run at or below the melting point of either parent material or any resulting eutectic that may form ($T_{bond} < T_{second\ material\ solubility}$).

Activated diffusion bonding is routinely performed on materials that suffer strength disadvantages due to grain growth or structures that may not be heavily strained. Parts having poor surface finish, materials that are degraded by grain growth (e.g., nickel- and cobalt-based superalloys) or bonds requiring ultra-low hermetic-seal values are typically bonded using this method. This is particularly true when the structure contains very tiny embedded passages that could become plugged or blocked by a bonding method relying on liquid generated during the bonding operation.

### Liquid Interface Diffusion (LID) Bonding

In this bonding method, one purposefully places a secondary material at the bond interface with the intention that it will melt and produce a liquid during the bonding operation (similar to conventional brazing), but it should be emphasized that there are significant differences between these two processes.

Specifically, the starting interface in the LID bond is typically very thin – on the order of 3.81 microns (0.00015 inches) thick. By comparison, a typical braze joint is usually at least 38.1 microns (0.0015 inches) thick. Because the starting thickness is so thin, it is also normal that the second (or "B") material is nearly completely diffused out into the lattice of the parent material during LID bonding. The liquid flows outward into the surface asperities, helping to minimize the voids that may otherwise be caused from parts with poor surface finish or minor defects (such as burrs).

In a typical braze joint (Fig. 2), the microstructure typically shows a defined layer of the braze-alloy chemistry and has fillets at the outer interfaces at each layer. The resulting strength of the diffusion-brazed joint and the LID bond joint can often vary by substantial amounts. This is due to the relative amount of actual parent-to-parent metal surface being bonded. In addition, the added LID bonding layer may be selected to promote the formation of a eutectic with the parent metal so that the bonding temperature can be lowered to just above the eutectic of the resulting system ($T_{bond} > T_{eutectic}$ or $T_{liquidus\ of\ the\ second\ material}$).

The resulting diffusion rate occurs at three to five orders of magnitude faster than solid-state diffusion bonding. Therefore, LID bonding may be done more

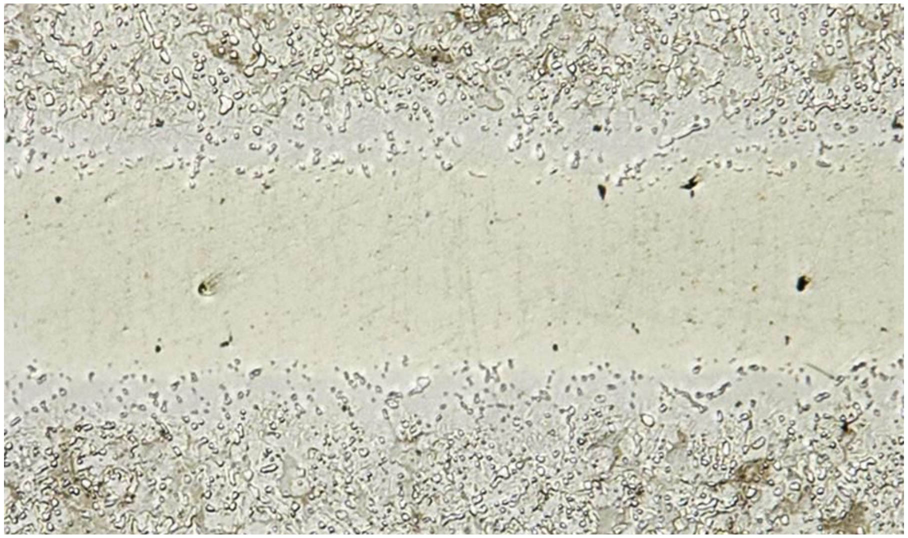

Fig. 2. 420 stainless steel LID-bonded microstructure. Metglas® MBF 20 foil 0.38 mm (0.0015-inch) thick. Processed at 1080˚C (1980˚F) for one hour at temperature using a ramp rate above 970˚C (1776˚F) – the alloy solidus – of 18˚C/h (30˚F/hour), loaded at 2,760 kPa (400 psi) unit-normal stress all at 10-5 Torr (1,000X).

quickly and at lower strain energy than solid-state or activated diffusion-bonding methods. LID bonding is used for large parts with large internal-passage geometries – over 3.175 mm (0.125 inches). LID bonding is also used in instances where the risk of plugging of the passages is a concern or where part size is such that there may not be enough mechanical force available from the differential-expansion tooling or hydraulic system/rams to achieve a good bond.

An LID bond joint can be quite thin and produces excellent diffusion interfaces with the parent material. This type of bond can be used directly adjacent to passages that are quite small, such as 0.16 mm x 0.16 mm (0.0625 inch x 0.0625 inch), with very little risk of plugging. The actual amount of liquid that is generated can be controlled using slow ramp rates –18˚C/hour (30˚F/hour) for example. Even at pressures up to 2,760 kPa (400 psi) unit-normal stress and very little parent-metal strain (less than 0.1%) occurs, and only a small percentage of the alloy is displaced.

## Transient Liquid Phase (TLP or TLID) Diffusion Bonding

TLP bonding involves surfaces that may be similar in quality and chemistry to the thin-layer LID bonding method, but the process prevents liquid from actually forming to any significant amount (i.e., retards the actual melting of the braze alloy by controlling the in-situ eutectic formed). As a result, the risk of plugging a small passage can be substantially reduced while otherwise still allowing the successful bonding of parts that may have a poor surface finish.

Slow ramp/heating rates can be continued above the solidus temperature of the second material all the way up to temperatures well above the liquidus of the interface system provided that the ultimate temperature never exceeds the incipient melting point of the parent material.

## Summary

Vacuum diffusion bonding has made great strides in recent years and is a technology that design engineers and heat treaters need to know more about. Past limitations such as dirty materials, uneven contact surfaces and poor equipment design (yielding uneven pressure) have been overcome, and the result is a robust process worthy of consideration.

**For the rest of this discussion (Part 2), please see Chapter 6.3.**

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# The Annealing Process Revealed Part One: Basic Principles

One of the most fundamental processes that must be performed on steel is annealing. While a relatively simple heat treatment to perform, there are a number of factors that must be carefully considered and controlled. In this article, we will discuss the basic principles behind the annealing process. Let's learn more.

## Purpose of Annealing

Annealing serves many purposes. For example, steel wire is annealed to improve its ductility and to relieve internal stress created by drawing, cold forming or uneven cooling after hot rolling. Annealing will also help to refine the grain size.

### Effect of Annealing on Microstructure

On heating, low-carbon (<0.030%) steels form ultra-fine particles of austenite as they reach, then exceed, the lower critical temperature (Ac$_1$). As the temperature rises, excess ferrite continues to dissolve, finally disappearing at the upper critical point (Ac$_3$). As the temperature continues to climb, the grain size increases.

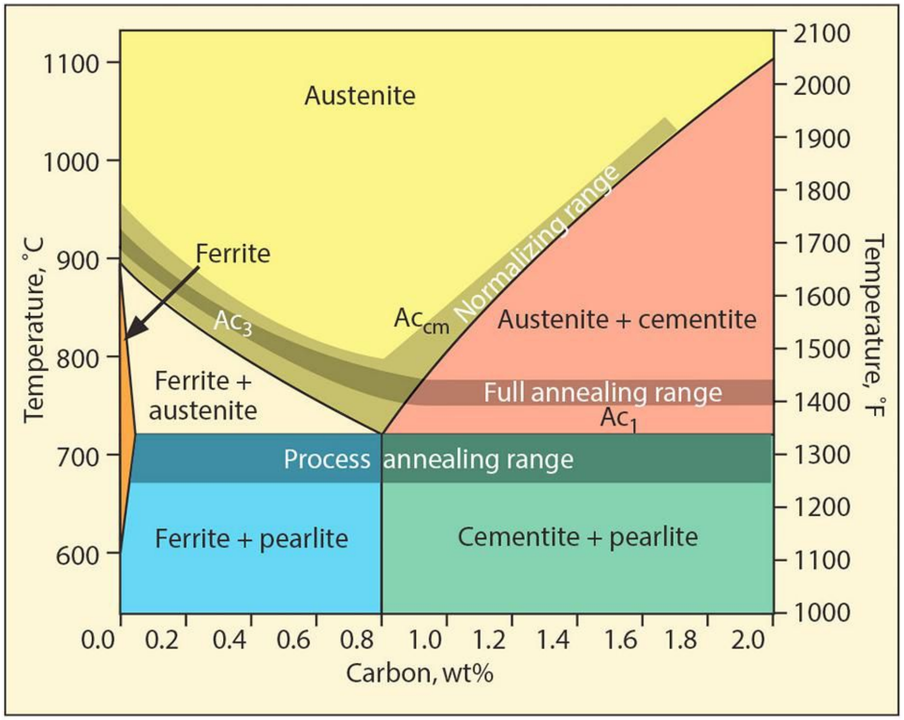


Fig. 1. Full annealing and process annealing ranges

The properties obtained as a result of annealing depend on the amount of carbon present, the coarseness of the ferrite and pearlite, and their relative distribution throughout the matrix. These factors are influenced by:

a. The size of the austenite grains; the smaller

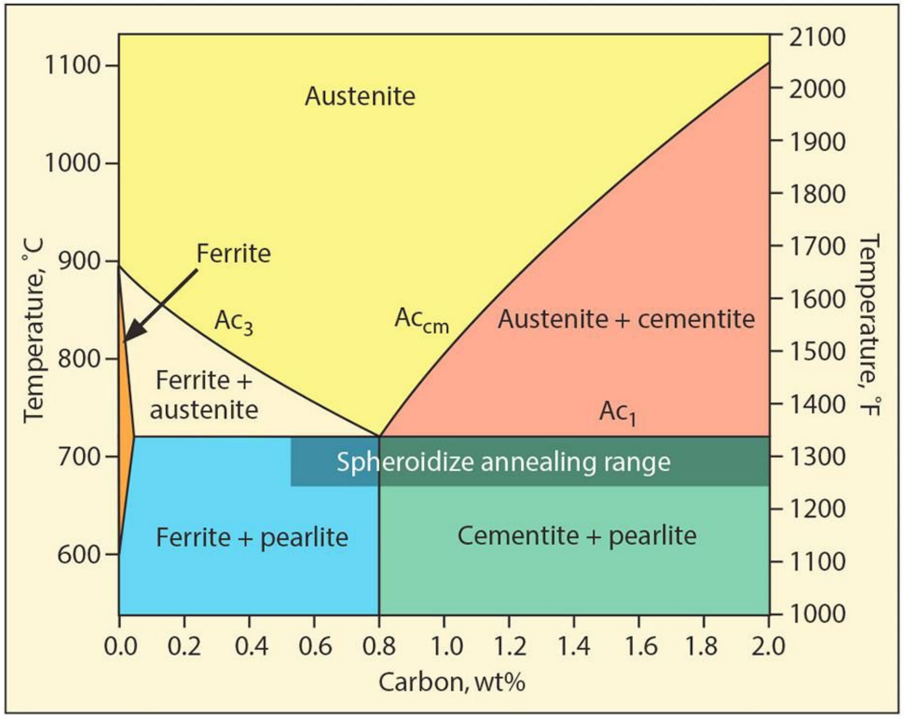


Fig. 2. Spheroidize annealing range

their
    size, the better the distribution of the ferrite and
    pearlite
b. The rate of cooling through the critical range
c. Time at temperature, which is necessary for
carbon
    to uniformly distribute in austenite

On slow cooling through the critical range, ferrite formation begins at the austenite grain boundaries. Large, rounded ferrite particles are formed, evenly distributed among the (relatively) coarse pearlite. With a higher rate of cooling, a network structure of small ferrite grains is produced with fine pearlite distributed in the center of these grains.

Since annealing cycles are performed in and around the critical temperatures, it is important to remember that, as far as transformation is concerned, the cooling practice is critical. The rate with which the steel passes through this range will determine the microstructure, hardness and other properties of the transformed product. A very slow rate of cooling will result in the softer microstructure, which is spheroidic. A faster rate results in lamellar pearlite of varying degrees of coarseness and hardness. If the rate of cooling is too rapid, formation of the soft products of transformation will be suppressed and the harder constituents – bainite and martensite – formed. These latter microstructures are undesirable in the annealed structure.

## Types of Annealing

### Full Anneal

Full annealing involves heating to a temperature at least 50˚F (28°C) above $Ac_3$. At this temperature, the steel is completely austenitic and the method and rate of cooling the austenite are very important for proper microstructure and other related properties. In full annealing, it is essential to know the critical temperatures on heating and cooling, the Isothermal Transformation (IT) diagram and the Continuous Cooling Transformation (CCT) diagram

The rate at which the steel cools from the annealing temperature to the upper cooling critical temperature is unimportant as long as the proper cooling rate is observed passing through the critical cooling range. Because of the difficulty of maintaining uniform temperature in a typical commercial annealing load, common practice is to cool from the annealing temperature, to and through the transformation range at a single uniform rate. Similarly, the rate of cooling after transformation is unimportant because it has no effect on the microstructure and hardness. However, it is common practice to maintain the uniform rate on cooling well past the lower cooling critical to ensure a proper anneal in case the critical temperatures were incorrect.

IT diagrams predict the microstructure after transformation, the temperature at which this transformation will take place and the time required so their use allows closer control of the end product. It is only necessary to cool the steel to the temperature where the desired microstructure will form, hold until transformation is complete and then cool in any convenient manner. The only precaution required is to cool the steel to the desired transformation temperature at a rate that will avoid underheating. CCT diagrams may also be useful to determine the rate of continuous cool that will result in a given product.

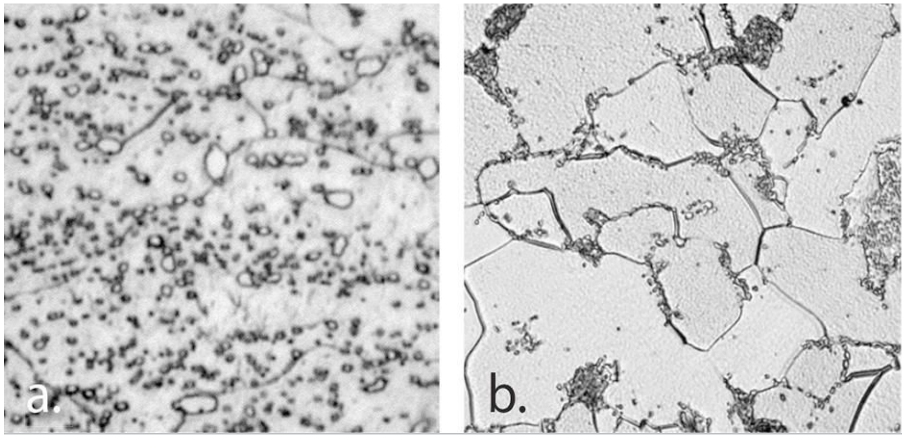


Fig. 3. Differences in annealed microstructures[2] (a) Spheroidize anneal (b) Process anneal (Courtesy of Aston Metallurgical Services Co., Inc.)

### Spheroidize Anneal

Spheroidize annealing is beneficial when subsequent machining and/or hardening is required (since the microstructure consists of rounded cementite particles in a ferrite matrix). The spheroidized condition is the true equilibrium state of the steel and is its softest condition. The spheroidized microstructure also possesses good cold-forming characteristics. Generally, the larger the spheroids and the more distance between them, the greater the ability of the steel to be cold formed.

The simplest method of spheroidizing is to employ a subcritical anneal (see below). A more common commercial method consists of heating to a temperature of 50˚F (13-26˚C) below $Ac_1$, hold at this temperature, then increase the temperature setpoint between $Ac_1$ and $Ac_3$ and hold again. Following the second soak period, the temperature is decreased slowly. Another common method is to heat to a temperature of 50˚F (5-26˚C) below the $Ac_3$, holding at one temperature and then increase it to slightly above $Ac_3$ followed by slow (controlled) cooling.

It is essential in any of these practices that nuclei be present to ensure formation of spheroids. The nuclei may be undissolved cementite, carbon concentration gradients (inhomogeneous austenite) or, in some instances, nonmetallic inclusions. If excessively long annealing times are employed at relatively high temperatures, however, a very coarse and abnormal agglomeration of the cementite particles will result. This condition is extremely undesirable from the standpoint of machinability. Furthermore, cementite particles of this size are extremely difficult to dissolve in austenite and result in poor response in subsequent hardening operations.

The importance of prior condition in spheroidize annealing applies to all steels regardless of carbon content, and the presence of coarse pearlite is undesirable because of resistance to spheroidization.

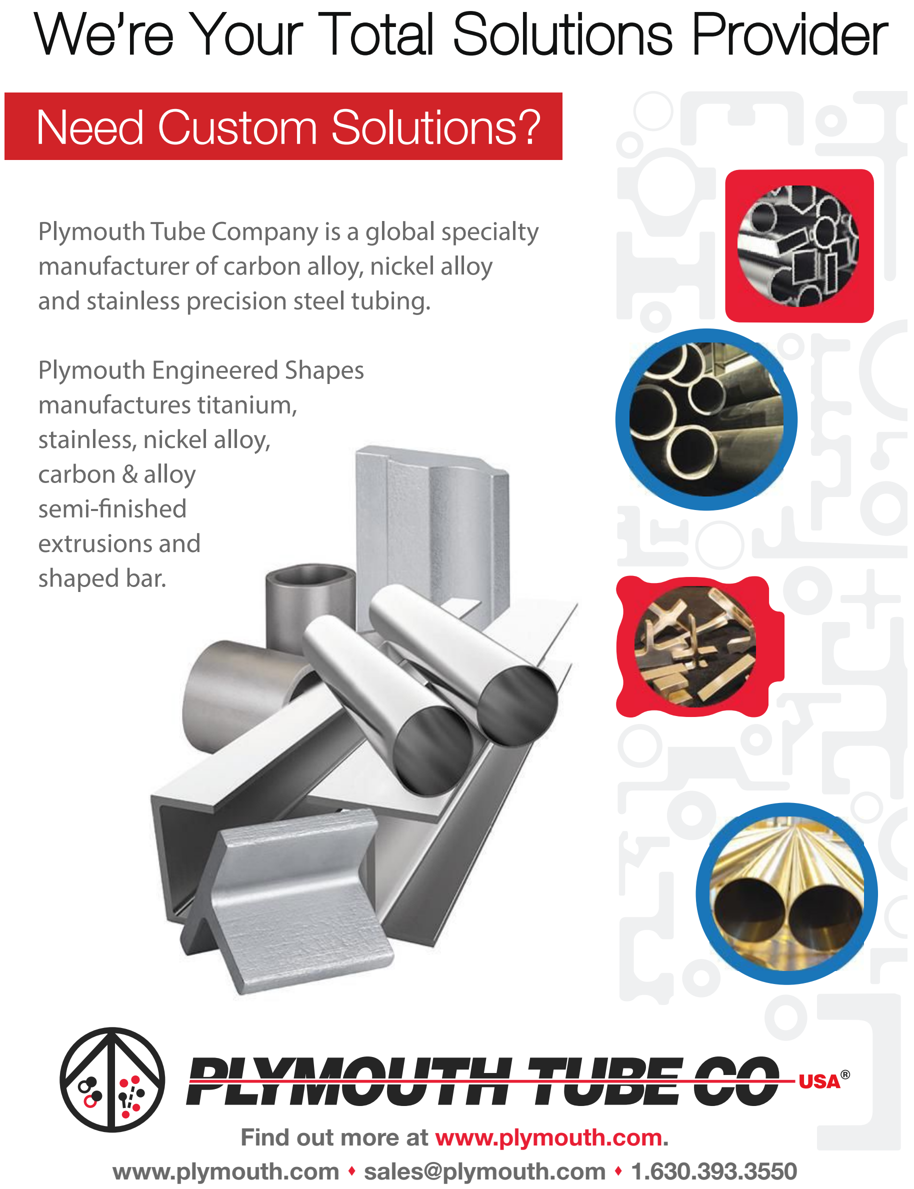
### Process (Subcritical) Annealing

Subcritical annealing consists of annealing cold-worked steel to a temperature below the lower critical temperature ($Ac_1$) or transformation range and cooling by a convenient means. This method can be the same as recrystallization or process annealing.

In this procedure, the steel is heated to a temperature about 25˚F (13˚C) below the $Ac_1$ and held there for a prolonged period of time. As a result, the existing cementite particles coalesce (ball up) and form spheroids. For this type of annealing, a fine prior microstructure such as martensite, bainite or fine pearlite is desirable. Coarse cementite in the prior structure should be avoided because the large cementite particles do not coalesce as readily as the finer ones. Because of the comparatively long times required to spheroidize at the subcritical temperature, this procedure is seldom used in commercial practice.

The recrystallization temperature of pure iron is in the region of 930˚F (500˚C). Consequently, a higher temperature brings about rapid recrystallization.

In subcritical annealing, a degree of caution must be exercised in selecting the annealing temperature since the rate of heating also has an effect. If Ac$_1$ has been incorrectly determined austenite will form, resulting in unwanted retained austenite, untempered martensite or both. When subcritical (process) annealing large loads, it may be expedient to use a furnace temperature that is somewhat higher than Ac$_1$ to speed up the process. Such a method, however, demands close control to prevent any of the charge from becoming austenitized. Prolonged annealing induces greater ductility (at the expense of strength) and may cause serious embrittlement problems.



# The Annealing Process Revealed Part Two: Furnace Atmosphere Considerations

Annealing is a relatively simple heat treatment to perform, but there are a number of factors that must be carefully considered and controlled. One of the most important is the furnace atmosphere that surrounds the parts as they heat and cool. Let's learn more.

The purpose of a furnace atmosphere varies with the desired end result of the heat-treating process. In general, the atmospheres used in the heat-treating industry have one of two common purposes:

- To protect the material being processed from chemical reactions that could occur on their surfaces (e.g., oxidation), that is, to be passive (chemically inert) to the metal surface.
- To allow the surface of the material being processed to change (e.g., adding carbon, nitrogen or both), that is, to be reactive (chemically active) to the metal surface.

When annealing wire, we most often want the atmosphere to protect the product in question rather than be reactive with it. Air atmospheres or those using products of combustion from direct-fired burners are not capable of preventing steel parts from oxidation or decarburization. It is for this reason that various protective atmospheres (generated, pure or mixed) are used (Table 1). The type of gas or gas mixtures used depends on the metal being treated, the treatment temperature, part contamination and the surface requirements of the product being annealed. Atmosphere control is often an important consideration as well.

Purging is another critical first step in the annealing process and should take place when the workload is cold (prior to heating). Nitrogen or lean (non-combustible) exothermic gas are common choices, and purging is considered complete when less than 1% oxygen is present as measured by use of an oxygen analyzer. The "rule of thumb" of five volume changes in an hour is often used to establish the required flowrate.[1]

## Types of Annealing Atmospheres

Characteristics of the most common annealing furnace atmospheres can be summarized as follows:

Table 1. Typical atmosphere types and composition[2]

| Atmosphere | Type | % $H_2$ | %$N_2$ | % CO | % $CO_2$ | Typical Dew Point °C (°F) |
|---|---|---|---|---|---|---|
| Endothermic | Generated | 37.5 - 40 | 40 | 20 | 1 – 1.5 | -1 to 10 (+30 to +50) |
| Exothermic (Rich) | Generated | 12 – 14 | 71 - 76 | 8 - 10 | 4 - 6 | +38 to -40 (+100 to -40) |
| Dissociated Ammonia | Generated | 75 | 25 | 0 | 0 | -40 to -75 (-40 to -50) |
| Hydrogen | Pure | 100 | 0 | 0 | 0 | -70 to -85 (-95 to -120) |
| Nitrogen-DA | Diluted | 90 | 10 | 0 | 0 | -45 (-50) or better |
| Nitrogen-Hydrocarbon | Blended | 90 – 98 | 2 – 5 | 1 – 5 | 0.50 | -60 (-80) or better |
| Nitrogen-$H_2$ | Mixed | 3 – 5 | 95 – 97 | 0 | 0 | -51 (-60) |
| Nitrogen-Methanol | Blended | 35 - 40 | 38 - 45 | 20 | 1 | -60 (-80) or better |

Table 2.
Typical dew-point levels[2]

| Dew Point, °F (°C) | Water Vapor (ppm) |
|---|---|
| +46 (+8) | 10,590 |
| +25 (-4) | 4,320 |
| 0 (-18) | 1,240 |
| -40 (-40) | 127 |
| -90 (-68) | 3.4 |

Note: 1% moisture = 127 ppm water vapor = +40°F dew point

## Nitrogen and Nitrogen-Hydrocarbon Blends

Pure (100%) nitrogen atmospheres are seldom used in annealing because they are not air (oxygen) excluding. Leaks or other sources of air infiltration cannot be compensated for. However, nitrogen is often blended with small percentages of hydrocarbon gas (methane, propane, propylene) or combined with methanol ($CH_3OH$) to create a suitable atmosphere provided it is properly controlled (Table 1). The nitrogen-hydrocarbon flows are often dictated by a predetermined recipe (Fig. 1).

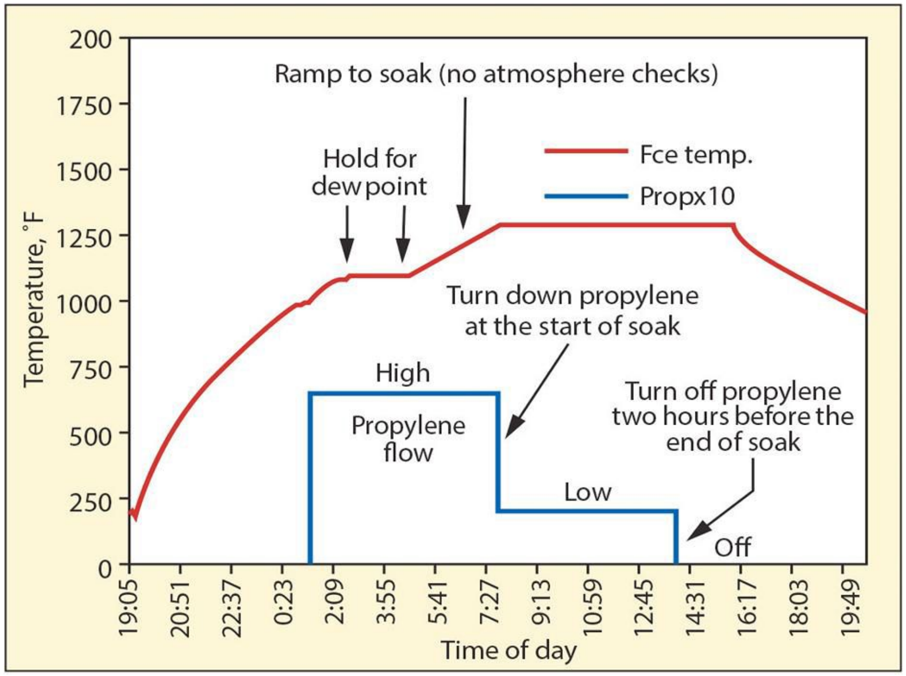


Fig. 1. Typical annealing cycle for steel wire (nitrogen-hydrocarbon mixture)[3]

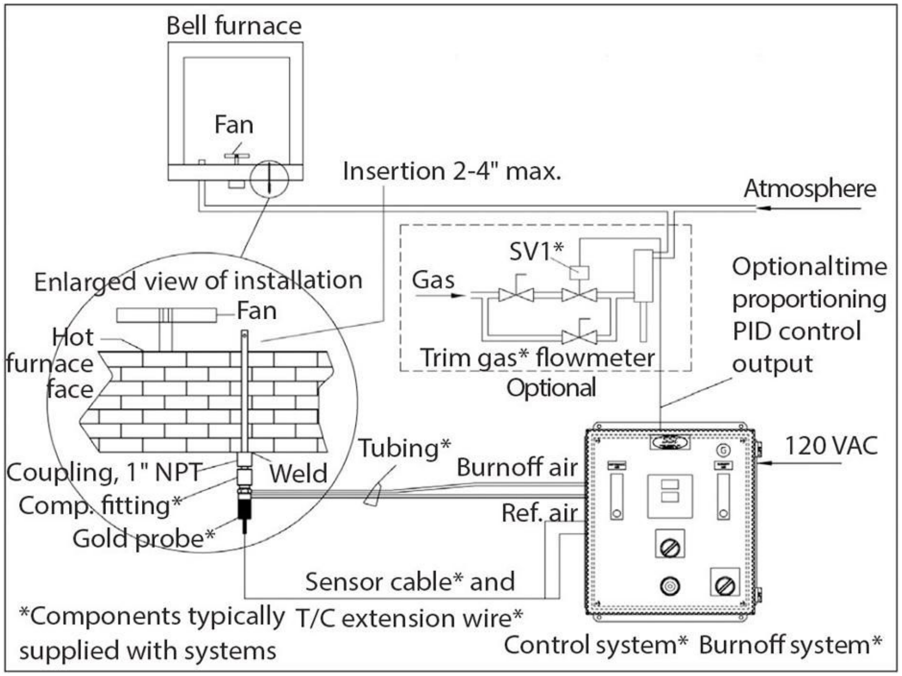


Fig. 2. Typical bell annealing furnace sampling system (Schematic courtesy of Super Systems, Inc.)[4]

## Hydrogen and Hydrogen-Nitrogen Blends

Annealing under a controlled atmosphere of 100% hydrogen prevents oxidation and product discoloration. Hydrogen is a highly reducing gas, so hydrogen annealing promotes “surface cleaning” of oxidized parts by reducing the oxides present on the wire. It is commonly referred to as “bright annealing.” Hydrogen may decarburize steel parts, so appropriate cautions must be taken. From an economic perspective, an atmosphere of 100% hydrogen is the most expensive. Lower cost nitrogen-hydrogen blends can also be used effectively.

## Dissociated Ammonia

Dissociated ammonia is produced by the cracking (dissociation) of ammonia into a gas having a composition of 75% hydrogen, 25% nitrogen. It has many of the same benefits as hydrogen but can be supplied at lower cost.

## Exothermic Gas

Rich exothermic gas is generated by partially combusting an air-gas mixture of (approximately) 6.5 parts of air to 1 part natural gas. Other hydrocarbon fuels can be used as well. Exothermic gas is the most widely used protective atmosphere for annealing, especially for annealing of low-carbon steel. However, it will decarburize medium-carbon and high-carbon steels because of the carbon dioxide ($CO_2$) and water vapor. When decarburization concerns exist, coolers are used to reduce the dew point from typically +38°C (+100°F) to +4.5°C (+40°F). In some cases, refrigerant dryers are used to further reduce the dew point from typically +4.5°C (+40°F) to -40°C (-40°F).

Purified rich exothermic gas, in which $CO_2$ levels are lowered to less than 0.1% $CO_2$ (typically 800 ppm or less), is used for short-cycle annealing and process annealing of medium- and high-carbon steels of the straight-carbon and certain alloy types. For long-cycle annealing, however, the high carbon monoxide (CO) content results in soot deposits on the work and other surface effects as a result of the relatively long soak times in which the work is in the critical low-temperature range of 480-700°C (900-1300°F), where these adverse gas reactions can occur. In short-cycle annealing, these effects are minimal, and in some instances, high CO gas is desirable because of its high carbon potential. Purified lean exothermic gas is sometimes used for long-cycle annealing of medium- and high-carbon steels of the straight-carbon and alloy types.

## Endothermic Gas

Endothermic gas (also called Endo or Rx™ gas) is produced when a mixture of air and fuel is introduced into an externally heated retort at 2.5:1 to 3.5:1 air-to-gas ratios. The retort contains an active catalyst, which is needed for cracking the mixture. Leaving the retort, the gas is cooled rapidly to avoid carbon reformation (in the form of soot) before it is sent into the furnace. An endothermic gas atmosphere requires carbon control for precise and repeatable carbon levels.

## Vacuum

Simply stated, vacuum is the absence of an atmosphere and represents the most ideal condition under which annealing can take place. Vacuum annealing, which can also be done in a partial-pressure atmosphere of an inert (e.g., nitrogen or argon) or reactive gas (e.g., hydrogen), is sometimes referred to as “bright annealing” due to the surface finishes produced. Because vacuum annealing is also the most costly, time consuming and least conducive to mass production, it is usually restricted to only certain materials such as titanium or tantalum.

## Control Schemes

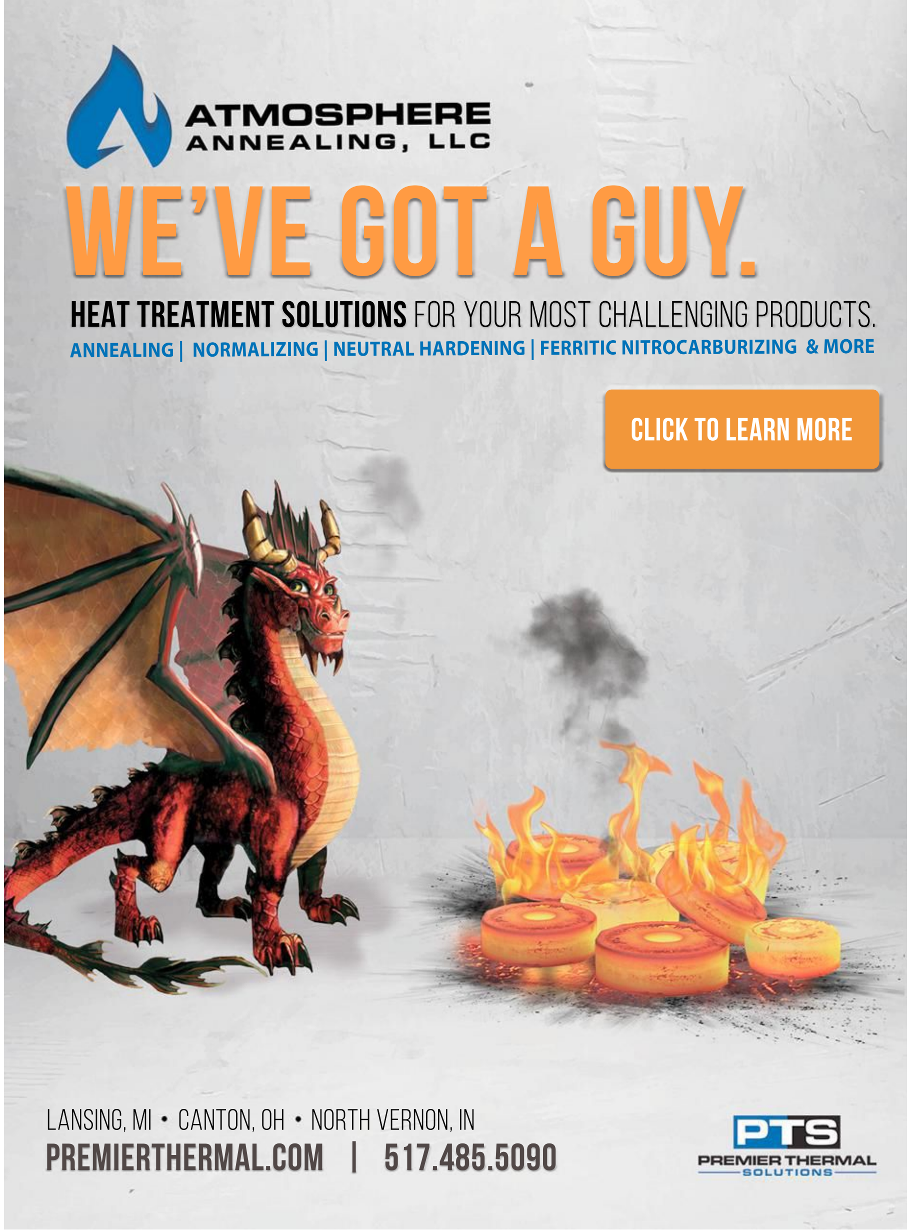
The composition of the furnace atmosphere is constantly changing, so we must use measurement and control devices (Fig. 2) to ensure good metallurgical quality control. This is accomplished by making sure that several of the following control methods are monitoring and/or controlling the process:

- Dew-point analysis
- Infrared analyzer (single or multiple gas analyzers)
- Oxygen probes

The trend today is to use multiple measurement tools to obtain the most accurate snapshot of the atmosphere in real time.

### Oxygen Measurement

The measurement of oxygen is important to judge both the effectiveness of the purging cycle (<1% $O_2$) at the beginning of the run as well as the effectiveness of the atmosphere during cooling (ppm range). Oxygen analyzers and oxygen probes are used for this purpose.



## Gas Analysis Methods

Dew point is defined as the temperature that water vapor starts to condense. In simplest terms then, a dew-point analyzer measures the amount of water vapor present in the furnace atmosphere (Table 2). This information can then be used to determine the carbon potential of the atmosphere. It is considered an indirect measurement technique since it involves pulling a gas sample from the furnace into the instrument.

Dew point, infrared (3-gas analyzers) and oxygen probes provide measurements commonly used to monitor the furnace atmosphere. The latter is an in-situ measurement device (it does not rely on a sample being drawn from the furnace). It is important to recognize that contaminants present on the wire or rod will tend to volatilize off during the initial heating phase. These contaminants must be removed by flushing (i.e. volume changeover) of the atmosphere, and the atmosphere must be stable prior to the onset of further heating or soaking at temperature.

For example, a properly controlled nitrogen/propylene atmosphere can achieve repeatable results on subcritical annealing of ferrous wire and wire rod.[5] The balance of process parameters and cost include:

- Accurate and repeatable measurement/inference of decarburizing agents that occur at trace levels ($O_2$, $H_2O$ and $CO_2$)
- Integration of the temperature control with the atmosphere control
- Control of the ramp from 1100°F (590°C) to the final soak temperature, typically 1300°F (705°C), in order to prevent decarburization
- Avoidance of carbon deposition (soot) by shutting off the propylene during soak after all major reactions are complete
- Lowering nitrogen flows to absolute minimum levels
- Using additional higher flows of propylene to speed up cycle time (leading to cost savings in fuel, electricity and nitrogen)
- Controlling depth of partial decarburization to meet specific manufacturing requirements of wire users
- Reducing/eliminating the air-burnoff segment to introduce less oxidation and speed up the ramp time on subsequent cycle

# The Annealing Process Revealed Part Three: Annealing of Steel Coils

Annealing can be performed in either batch or continuous furnaces. Box, pit and car-bottom furnaces are examples of batch units, while mesh-belt and roller-hearth styles are examples of continuous equipment. For annealing of steel coils, however, the most common type of furnace used is a bell-type furnace (Figs. 1-3). Let's learn more.

## Annealing Equipment

Fig. 1. Typical workload (bell annealer)[6]

Bell annealing (Fig. 4) heats batches of metal that are placed on a base assembly, enclosed by an inner cover and covered by the heating bell (furnace). An overhead crane is used to load the base and move the heating bell that is suspended from the crane. The base assembly normally includes a fan (optional) to provide a source of convection to enhance the heat transfer to the charge. The inner cover contains the desired atmosphere and protects the charge from the heating source. Direct fired, tangentially fired, radiant tube and electric elements are common heating methods. After annealing, cooling is performed by removing the heating bell but leaving the inner cover in place to maintain the protective atmosphere. If a bright finish is desired, the metal must be cooled to near ambient temperature before opening the workload to air. A gas-to-water heat exchanger or forced-cooler system is often used to shorten the cooling times, especially at low temperatures. The forced cooler replaces the heating bell at the end of the heating cycle and circulates air or sprays a fine water mist to accelerate the cooling of the outside of the inner cover.

Fig. 2. Retort being lowered into position[6]

Fig. 3. Heating bell being lowered in place[6]

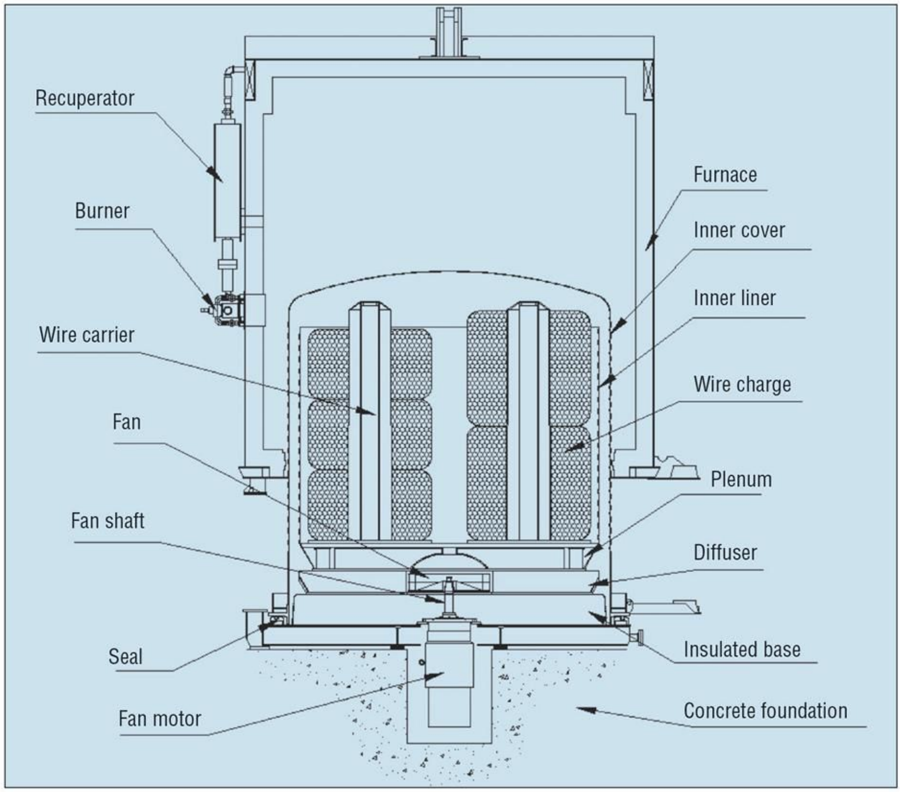


Fig. 4. Sectional view of a typical bell annealing furnace for wire products[7]

## Annealing Problems

### Microstructural Issues

In heat treating, changes happen to the internal structure (or microstructure) of the material and are not readily apparent to the naked eye. This is one of the reasons why process- and equipment-induced variables must be tightly controlled. For example, for many cold-heading components, process anneals are used to try to obtain a spheroidized microstructure (as opposed to a more lamellar structure). If lamellar, the more open (i.e. further apart) the lamellar structure, the softer it is and the more easily it is worked.

A typical low-carbon steel microstructure (Fig. 5) consists of ferrite (white areas) and pearlite (dark areas). Pearlite is a combination of ferrite and iron carbide ($Fe_3C$).

Properly distributed carbides are acceptable in most wire-drawing operations. However, some process annealing cycles (Fig. 6) produce agglomerated carbides (white globular regions) outlining the grain boundaries (necklacing). The result is that the wire

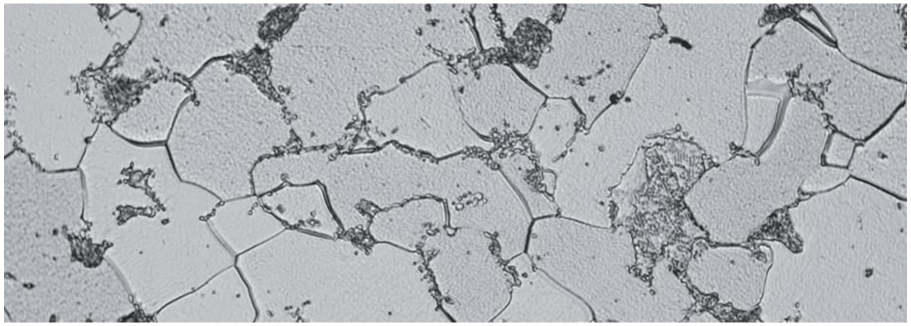


Fig. 5. Process-annealed microstructure – low-carbon steel[8] (Photomicrograph courtesy of Aston Metallurgical Services Co., Inc.)

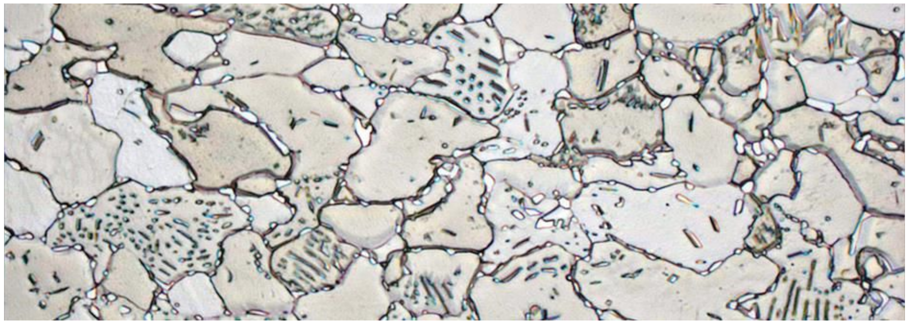


Fig. 6. Process-annealed microstructure – low-carbon alloy steel[8] (Photomicrograph courtesy of Aston Metallurgical Services Co., Inc.)

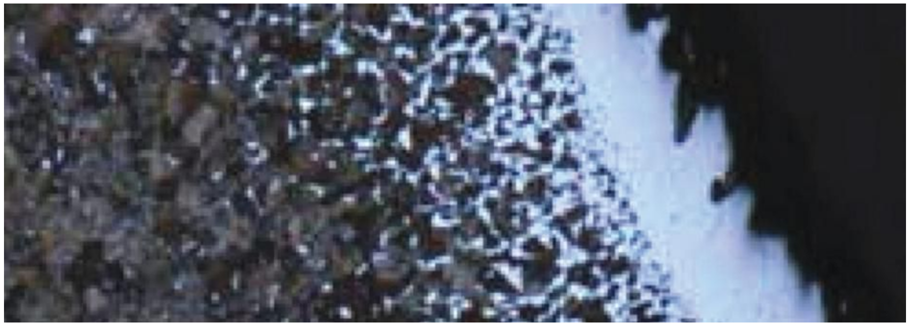


Fig. 7. Decarburized surface microstructure

will be more difficult to draw down to a smaller size without internal tears or breakage.

The presence of non-metallic inclusions, as well as the matrix microstructure, is also important considerations. The carbon content and cleanliness of the steel influence the volume fractions of ferrite, pearlite and carbide – what form the carbide is in (fine vs. coarse) as well as the morphology of those particles – and their size, shape and distribution. For example, fine wire drawers (e.g., tire wire, valve spring wire) are adamant about excluding nondeformable oxides, sulfides, carbides and nitrides for forming and fatigue reasons. Equally undesirable is carbon segregation, especially along the centerline of the wire, because it creates islands of martensite.

## Decarburization

By definition, decarburization is a result of (usually unintentionally) removing carbon from the surface of steel when it is heated and held at temperature or when in contact with certain types of furnace atmosphere (Fig. 7). The result is a change in mechanical properties at the surface, most notably lower surface hardness and loss of fatigue resistance. Decarburization can be total (100% ferrite or “free” ferrite) or partial, and it can vary in depth from a few ten thousandths to several thousandths of an inch.

In most cases, it is no more than 2% of the total thickness of the material. Carbon restoration can be used to reverse the effects of decarburization by driving carbon back into the surface of the rod or wire.

### Soot

A carbon-bearing furnace atmosphere can become out of control and cause sooting (carbon deposits) to occur on the surface of the wire (Eq. 1). This Boudouard (or gas producer) reaction may occur in a CO-rich furnace atmosphere in the critical range of 500-300˚C (932-572˚F). Of equal concern is carbon pickup, which in some cases results in surface cracking.

$$(1)\quad 2CO \rightarrow CO_2 + C$$

## Summing Up

The choice of furnace and control of the furnace atmosphere is critical to the success of the annealing process. Each step in the process must be well understood in order to achieve the proper microstructure, grain size and mechanical properties in the annealed wire.

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# The Importance of Normalizing

"Why bother normalizing?" is a question frequently asked of The Doctor. Why indeed? Let's learn more.

## Why Normalize?

Normalizing imparts both hardness and strength to iron and steel components. In addition, normalizing helps reduce internal stresses (Fig. 1) induced by such operations as forging, casting, machining, forming or welding. Normalizing also improves microstructural homogeneity and response to heat treatment (e.g. annealing or hardening) and enhances stability by imparting a "thermal memory" for subsequent lower-temperature processes. Parts that require maximum toughness and those subjected to impact are often normalized. When large cross sections are normalized, they are also tempered to further reduce stress and more closely control mechanical properties.

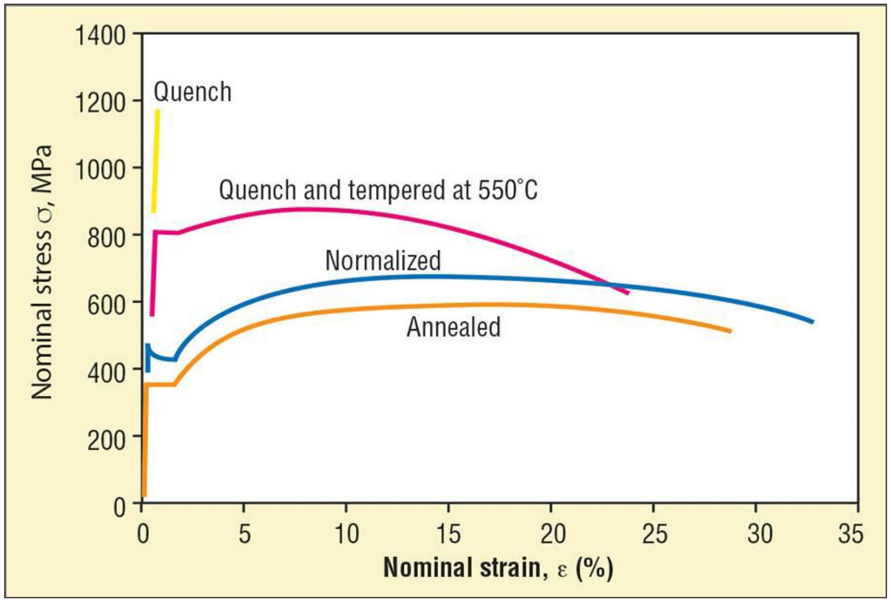

Fig. 1. Stresses induced by heat treatment (SAE 1045)[1]

Normalizing is typically performed in order to:

- Improve machinability
- Improve dimensional stability
- Modify and/or refine the grain structure
- Produce a homogeneous microstructure
- Reduce banding
- Improve ductility
- Provide a more consistent response when hardening or case hardening

By way of example, many gear blanks are normalized prior to machining so that during subsequent hardening or case hardening dimensional changes such as growth, shrinkage or warpage will be better controlled.

Soak periods for normalizing are typically one hour per inch of cross-sectional area but not less than two hours at temperature. It is important to remember that the mass of the part or the workload can have a significant influence on the cooling rate and thus on the resulting microstructure. Thin pieces cool faster and are harder after normalizing than thicker ones. By contrast, after furnace cooling in an annealing process, the hardness of the thin and thicker sections are about the same.

Low-carbon steels typically do not require normalizing. If these steels are normalized, however, no harmful effects result. Castings with relatively uniform wall thickness and section sizes are usually annealed rather than normalized. Other castings, especially those having a complex shape or interconnected thick and thin sections and are thus prone to high levels of residual stresses, benefit from normalizing. The microstructure obtained by normalizing depends on the composition of the castings (which dictates its hardenability) and the cooling rate.

## How it Works

The normalizing of steel (Fig. 2) is carried out by heating approximately 100°F (38°F) above the upper critical temperature ($Ac_3$ or $Ac_{cm}$) followed by cooling in air to room temperature, or at no greater than one-bar pressure using nitrogen if the process is being run in a vacuum furnace.

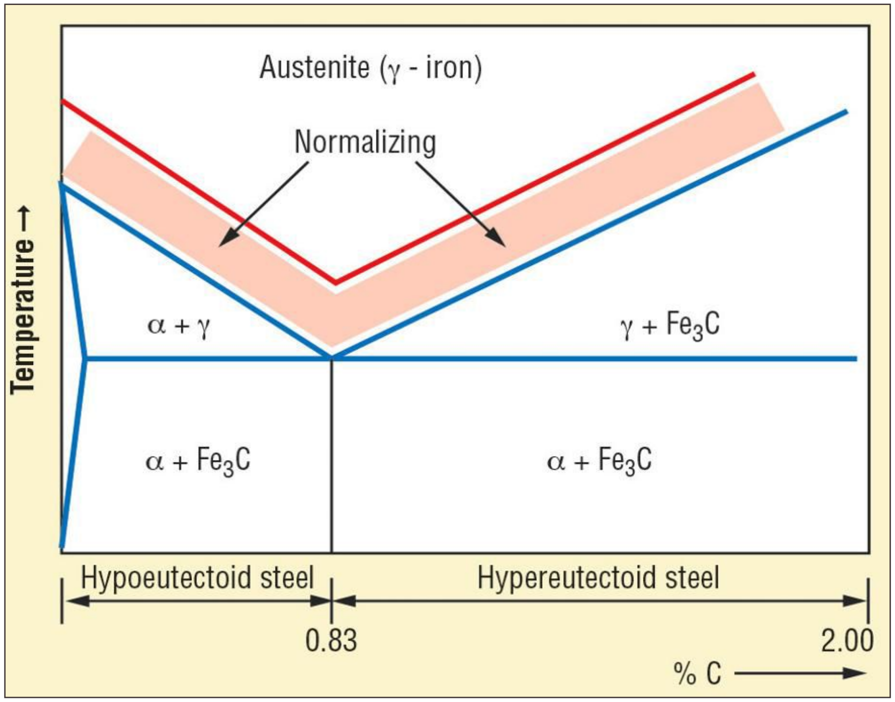

Fig. 2. The normalizing process[2]

Normalizing is often considered from both a thermal and a microstructural standpoint. In the thermal sense, normalizing is austenitizing followed by a relatively slow cool. In the microstructural sense, the areas of the microstructure that contain about 0.80% carbon are pearlitic, while areas of low carbon are ferritic.

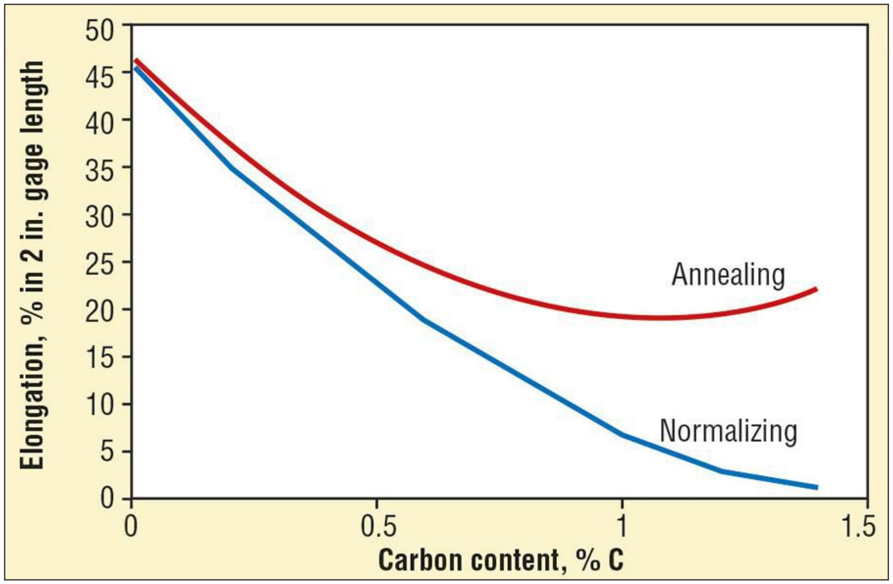

Fig. 3. Normalizing and ductility[2]

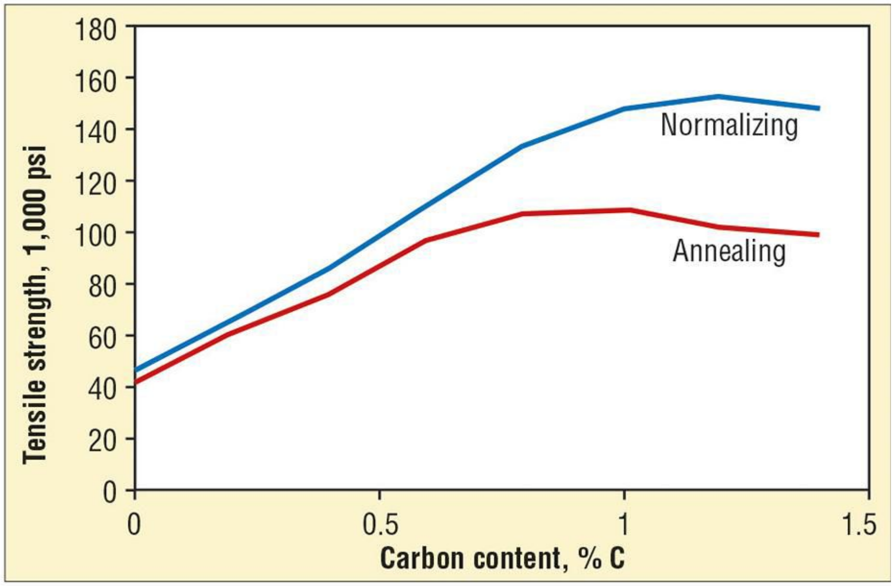

Fig. 4. Normalizing and tensile strength[1]

## Normalizing vs. Annealing

Normalizing differs from annealing in that the metal is heated to a higher temperature and then removed from the furnace for air cooling rather than furnace cooling. For many manufacturing engineers there is often a great deal of confusion as to when to specify normalizing and when to call out annealing. There is a logical reason for this because, in many instances, the procedure for normalizing and that of annealing are one and the same. For example, very-low-carbon steel can be almost fully annealed by heating above the transformation range and cooling in air.

In normalizing, the cooling rate is slower than that of a quench-and-temper operation but faster than that used in annealing. As a result of this intermediate cooling rate, the parts will possess a hardness and strength somewhat greater than if annealed but somewhat less than if quenched and tempered. The slower cooling rate means normalized sections will not be as highly stressed as quenched sections. Thus, normalizing is a treatment where a moderate increase in strength is achieved without undue increase in stress.

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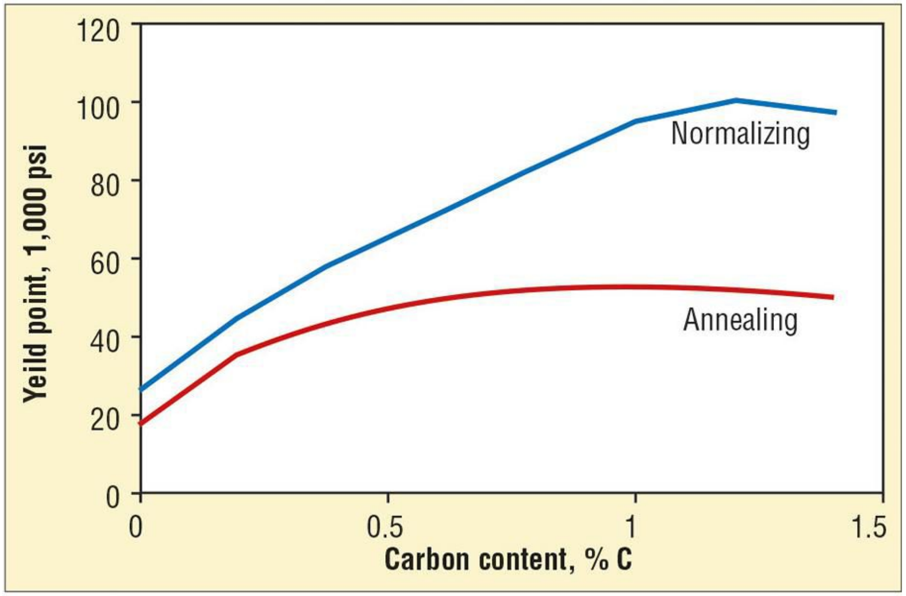


Fig. 5. Normalizing and yield strength[1]

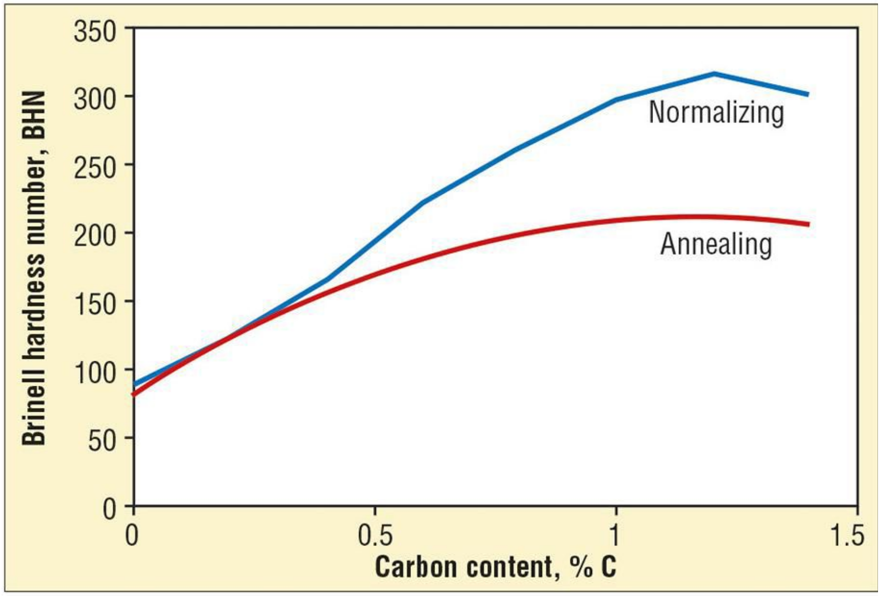


Fig. 6. Normalizing and hardness[1]

## Effect of Normalizing on Physical Properties

Annealing and normalizing do not present a significant difference on the ductility of low-carbon steels. As the carbon content increases, however, annealing levels off, with respect to a property such as elongation, around 20%. On the other hand, the ductility of the normalized high-carbon steels continues to drop to the 1–2% level (Fig. 3).

The tensile strength (Fig. 4) and the yield point (Fig. 5) of the normalized steels are higher than the annealed steels. Normalizing and annealing do not show a significant difference on the tensile strength and yield point of the low-carbon steels. Normalized high-carbon steels, however, exhibit much higher tensile strength and yield point than those that are annealed.

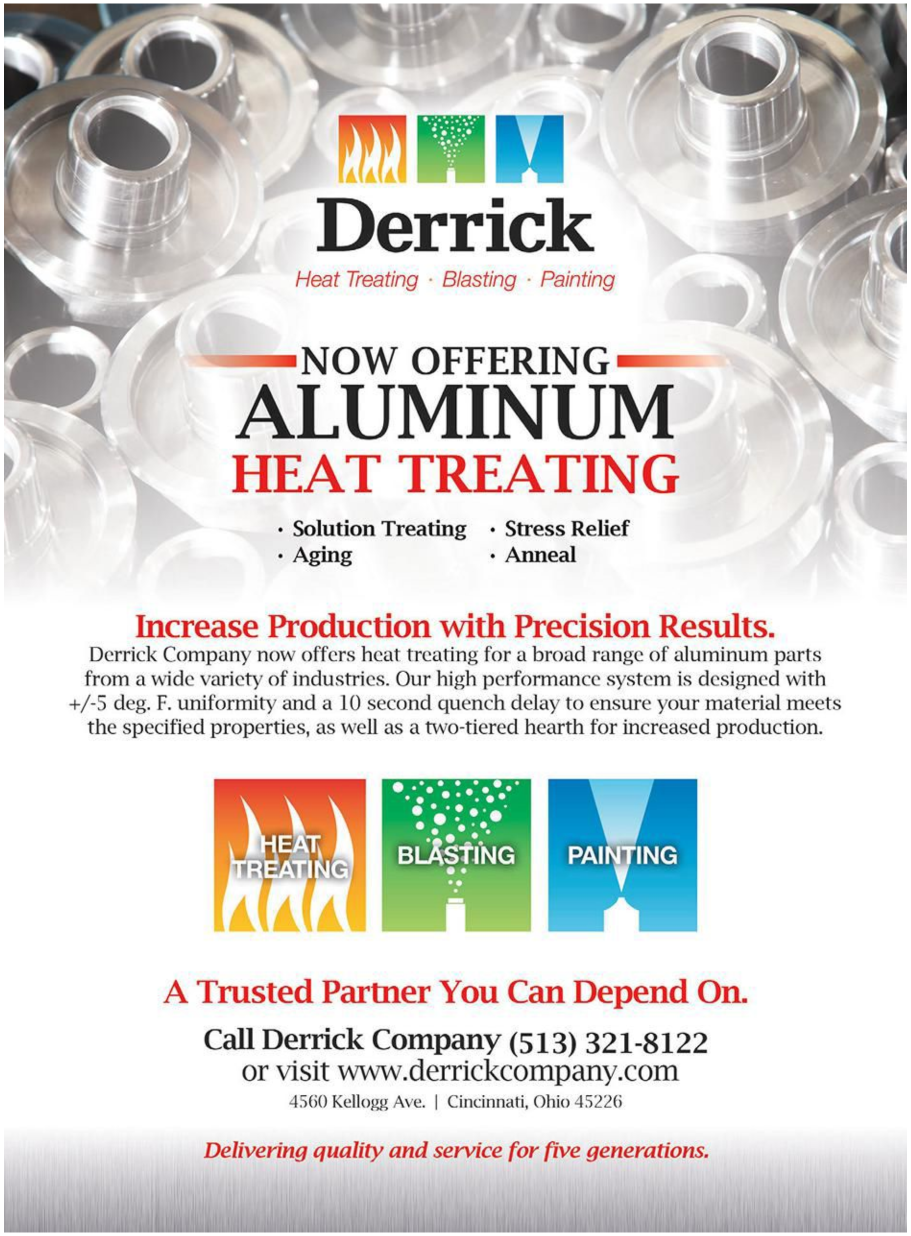
Low- and medium-carbon steels produce similar hardness levels when normalized or annealed. However, when high-carbon steels are normalized, they maintain higher levels of hardness than those that are annealed (Fig. 6).

## In Conclusion

Normalizing is a process that improves part quality and plays an important role in controlling dimensional variation in hardening and case hardening. Normalizing should be done whenever dimensional stability is important or when manufacturing operations are expected to impart significant amounts of stress into the material. Normalizing helps avoid many heat-treating problems.

## References

1. Rapid Product Deployment Research Centre (http: rpdrc.ic.polyu.edu.hk)
2. School of Engineering Technologies, Farmingdale State College (www.le.farmingdale.edu)



# Atmosphere Gas Carburizing – Case Studies, Lessons Learned (Part 1)

One of the most fundamental processes that must be performed on steel is annealing. We begin with an interesting tale of what happens if we naively follow along. Just ask the four young Oysters who become enthralled with the seemingly idle chatter of the Walrus and the Carpenter, ending up as the main course at dinner.

"The time has come," the Walrus said,
"To talk of many things:
Of shoes – and ships – and sealing wax –
Of cabbages – and kings –
And why the sea is boiling hot –
And whether pigs have wings."
– The Walrus and the Carpenter, Louis
Carroll, Through the Looking-Glass
and What Alice Found There, 1872.

Atmosphere gas carburizing is a process so familiar to most heat treaters it is too often taken for granted. We trust our oxygen-probe readings to keep us safe, and we expect the outcome of the process to never change. But occasionally we get in trouble, and when we do, valuable lessons emerge. Let's learn more.

Table 1. Part surface area to load-size relationship for typical integral-quench furnaces

| Load Size (width × length × height) mm (inches) | Maximum Part Surface Area $m^2$ ($ft^2$) |
|---|---|
| 600 x 900 x 600 (24 × 36 × 24) | 16.7–23.2 (180–250) |
| 760 x 1200 x 760 (30 × 48 × 30) | 27.9–37.2 (300–400) |
| 900 x 1200 x 900 (36 × 48 × 36) | 37.2–46.4 (400–500) |
| 900 x 1800 x 900 (36 × 72 × 36) | 58.0–67.4 (625–725) |

Table 2. Part spacing requirements for typical batch loading

| Part Diameter | | Horizontal Spacing (inside) | | Vertical Spacing (inside) | |
|---|---|---|---|---|---|
| mm | inches | mm | inches | mm | inches |
| ≤ 25 | ≤ 1 | 6–19 | 0.25–0.75 | 13–19 | 0.5–0.75 |
| 25–50 | 1–2 | 13–38 | 0.5–1.5 | 19–25 | 0.75–1 |
| 50–75 | 2–3 | 19–57 | 0.75–2.25 | 25–38 | 1–1.5 |
| 75–100 | 3–4 | 57–75 | 2.25–3 | 38–50 | 1.5–2 |
| ≥ 100 | ≥ 4 | ≥ 75 | ≥ 3 | ≥ 50 | ≥ 2 |

We will start by looking at various external and internal factors that can affect the carburizing process, uncover issues related to process and/or equipment variability, discover where the pitfalls might lie and talk about what we can do to avoid them.

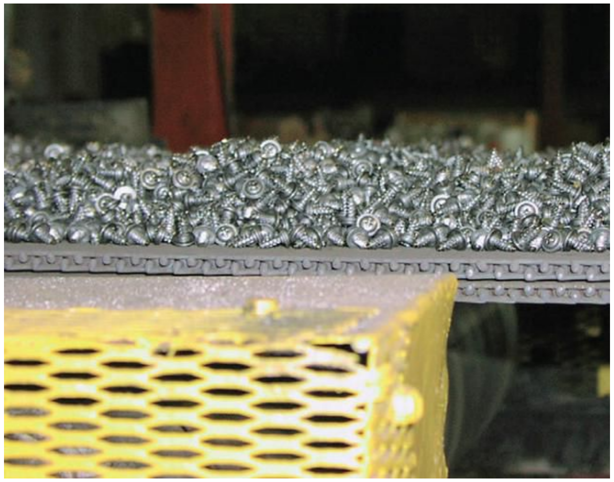


Fig. 1. Example of random loading of fasteners on a mesh belt prior to carbonitriding

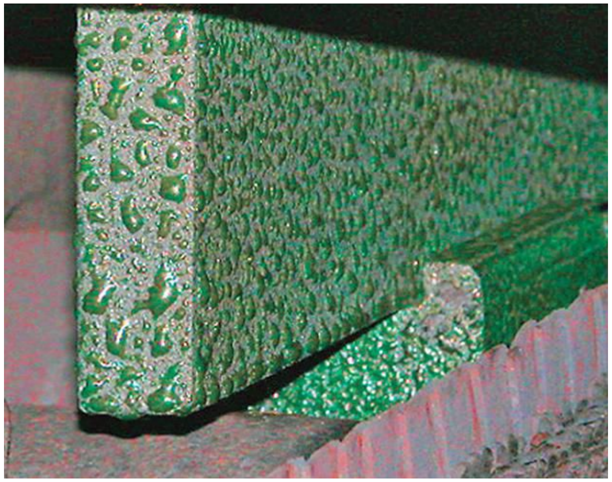


Fig. 2. Internal furnace contamination – sodium deposits in the form of a glassy coating

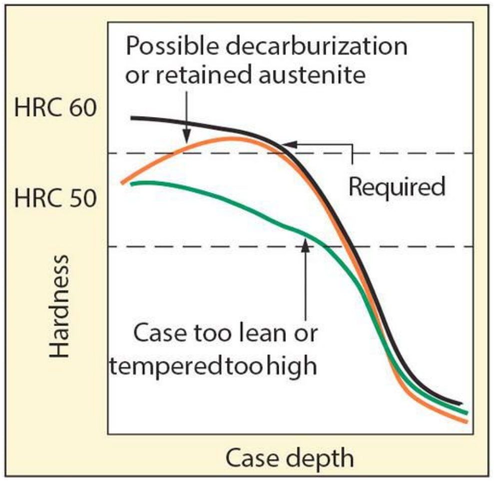


Fig. 3. Low case hardness[3]

## Part Loading

Many times, variation in case depth and other carburizing problems can be traced back to how parts are loaded in baskets and fixtures. Loading arrangements generally fall into one of two broad categories: weight-limited or volume-limited. In either case, when loading parts in furnace baskets or onto racks, our first instinct is to maximize loading efficiency. However, as heat treaters must also be concerned with proper part spacing (i.e. positioning parts within the load for optimal heat transfer), atmosphere circulation, temperature uniformity and heat extraction during quenching (to minimize distortion). And while trial and error is often the most prudent path, we must also take into consideration:

- Furnace-induced factors (often a function of the style of furnace in use). Being aware of the process limitations induced by a given design is an invaluable aid when things go wrong.
- Part geometry and orientation factors. We need to ask ourselves questions such as, "How much space should be left between parts?" and "Is random loading (Fig. 1) or nesting possible or even prudent?"

For example, bearing races of various diameters – a typical volume-limited load configuration –

are often nested inside one another, producing an “optically dense” workload that is difficult to uniformly heat in many cases. In this instance, the cycle must be adjusted to allow enough time for the interior parts to reach temperature. Here, the furnace fan (type, speed, rotational direction, location) plays a significant role in the heating process. Fasteners are another example of where random loading in either continuous or batch-type (Table 1) units is most often used to handle the sheer volume of parts to be run. In this case, atmosphere penetration throughout the load, cleanliness of the parts entering the furnace and allowing adequate time at temperature are considerations that must be factored into the process. If parts are not bulk loaded, a good rule of thumb is that the gap around a part should be no less than 25% and no greater than 75% of the parts’ envelope diameter (Table 2).

## Part Cleaning

Although atmosphere gas carburizing demands only a moderate level of cleanliness (compared to many other processes or industries), contamination, such as cutting oils and residues left on parts, can cause significant problems both in our equipment (Fig. 2) and on the parts themselves. Carburizing and carbonitriding tend to be far more forgiving with respect to the amount of contamination (e.g., oils, water, cleaning residues, etc.) that can be tolerated without interfering with case development and the quality of the resultant microstructure. Still, it is important to remember that cleaning must be done to at least a level appropriate for the intended application

Fig. 4. Damaged gear teeth due to lack of adequate carburization

## Carburizing Process Problems and Their Solutions

### Inadequate Case Depth

Not achieving the desired case depth (Fig. 3) can be due to a number of factors, some of which are carburizing at too low a carbon potential (i.e. too lean a furnace atmosphere), partial or complete decarburization of the part surface from air infiltration due to a leaky furnace, processing at the wrong temperature perhaps due to malfunctioning or improperly located thermocouples, retained austenite in the case region or a "slack" quench.

Steps that can be taken to correct these maladies include increasing the carburizing potential of the furnace atmosphere (particularly if boost/diffuse carburizing is being performed), changing the carburizing process (e.g., carburizing and slow cooling followed by a subcritical anneal prior to reheat and quench), subzero treatments and selecting the proper tempering temperature.

## Shallow Case or No Case Depth

Producing shallow case depth or areas where there is no case development points to incomplete surface preparation prior to carburizing, the presence of surface contaminants or possibly the misapplication of selective carburization methods (i.e. stop-off paints or poorly adhering copper plate). Another area of concern is how the parts are being received from upstream operations. Dirty dunnage and suspect transport methods may add a level of contamination (e.g., rust) that is unacceptable to the carburizing process.

Solutions to these problems include controlling the cleaning process, cleaning the parts washer as well as replacing its solution on a frequent basis, and handling parts with clean gloves.

## Coming Up

In part 2, we will discuss problems associated with retained austenite, decarburizing/de-alloying, intergranular oxidation, case leakage, case cracking/separation, case crushing, untempered/tempered martensite effects and other issues.

## References

1. Herring, D. H., "How to Load Parts in Furnace Baskets," Heat Treating Progress, November/December 2003.
2. Herring, D. H., "It's Time to Clean Up Our Act!," Industrial Heating, January 2008.
3. Weires, Dale J., Gear Metallurgy, Effective Heat Treating and Hardening of Gears Seminar, SME Short Course, 2007.
4. Mr. Darwin Behlke, Twin Disc, Inc., private correspondence.

# Atmosphere Gas Carburizing – Case Studies, Lessons Learned (Part 2)

We continue the discussion started last month on atmosphere carburizing, namely the problems we encounter in the heat-treat shop and the solutions that must be implemented to achieve a successful outcome. Let's learn more.

## Carbides and Carbide Necklaces

The formation of grain-boundary (i.e. massive) carbides and carbide necklaces (Fig. 1) has been the subject of a great deal of study but one that is directly related to process variables that are out of control. These include too high a carbon potential of the atmosphere during the boost portion of the cycle, insufficient diffusion time, too short a soak time at temperature and hardening from too low a temperature, to name a few.

Fortunately, the formation of carbides can be minimized by steps such as controlling the carbon potential, adding more diffusion time to the recipe and changing the hardening temperature (or time). This is one of the reasons metallurgists are so concerned about verifying the oxygen (carbon) probe readings by use of a three-gas analyzer to determine the actual CO value, performing shim-stock testing to determine actual surface carbon and taking dew-point measurements to compare with historical information.

## Retained Austenite

Austenite that does not transform to martensite upon quenching is called retained austenite (RA). RA occurs when steel is not quenched to its Mf (martensite finish) temperature (i.e. low enough to form 100% martensite). Since the Mf drops below room temperature in alloys containing more than 0.30% carbon, significant amounts of untransformed (retained) austenite may be present, intermingled with martensite at room temperature (Fig. 2).

Causes for high percentages of RA include a carbon potential that is too high and direct quenching from carburizing temperature. Leaning out the carbon potential, slow cooling followed by a sub-critical anneal (optional), and reheating and quench from a lower hardening temperature are solutions as well as introducing a subzero treatment, typically in the range of -62˚ to -100˚C

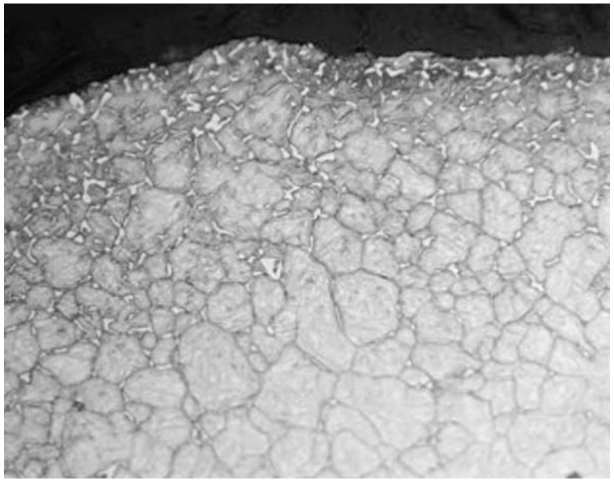


Fig. 1. Bearing race corner exhibiting retained austenite (white areas)

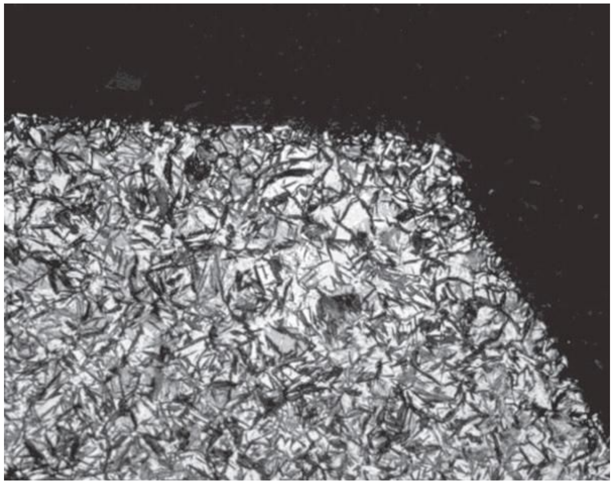


Fig. 2. Bearing race corner exhibiting retained austenite (white areas)

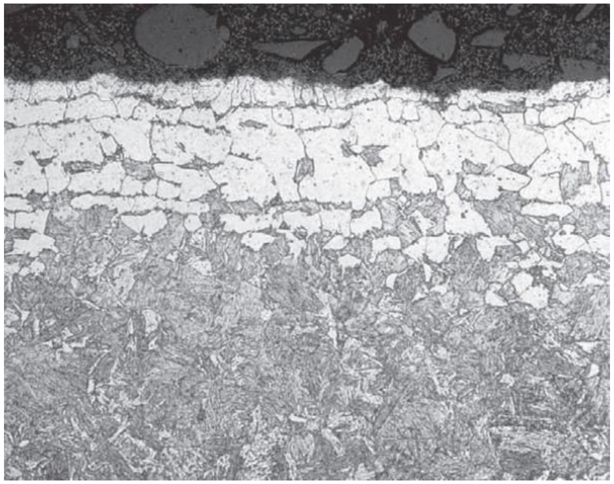


Fig. 3. Total decarburization on a steel part surface

(-80 to -150˚F).

RA is problematic because it is metastable. Stress, elevated temperature or time will cause RA to transform into untempered martensite. In addition, a volume change (increase) accompanies this transformation and induces a great deal of internal stress in a component, increasing the likelihood of cracking.

## Decarburization and Dealloying

If a steel part is exposed to elevated temperatures in the presence of air (Fig. 3), carbon will be depleted from the surface of the part (i.e. decarburization) and/or alloying elements such as manganese and chromium will be oxidized at the surface (i.e. dealloying). These effects generally occur when air leaks are present in the equipment, an improper carbon potential (too low) is used during the hardening process for the alloy in question, when preheating in air prior to loading into a protective atmosphere furnace is done above 370˚C (700˚F), or when parts are hardened without adequate atmosphere protection.

Proper furnace maintenance, including checking radiant tubes for pinhole leaks and periodic pressure testing, combined with proper atmosphere control typically eliminate equipment variables related to this problem. Copper plating or selective stop-off paints (if used) must be adherent and properly applied.

## Intergranular Oxidation

Intergranular oxidation (IGO) and inter-granular attack (IGA) are commonly associated with oxygen present during the carburizing portion of the cycle. In atmosphere carburizing, some IGO/IGA is unavoidable, typically 0.013 mm (0.0005 inches) or less, but can negatively affect mechanical properties such as bending fatigue life.

Corrective action involves improved atmosphere control, being sure that the furnace is leak-free and/or switching to an alternative carburizing method such as low-pressure "vacuum" carburizing. Post-heat-treatment solutions often involve grinding of the surface to remove this effect.

## Low Case Hardness

Low hardness in the carburized case (Fig. 4) is often caused by carburizing with a carbon potential that is too lean, higher than normal amounts of RA, partial decarburization, a "slack" quench or over tempering. The surface-hardness drop can typically be corrected by using one of the following methods: increasing carburizing boost time (e.g., higher carbon potential in the atmosphere); carburizing, slow cooling, sub-critical annealing (optional), reheating and quenching from a lower hardening temperature; introducing a subzero treatment; and/or selecting the correct tempering temperature.

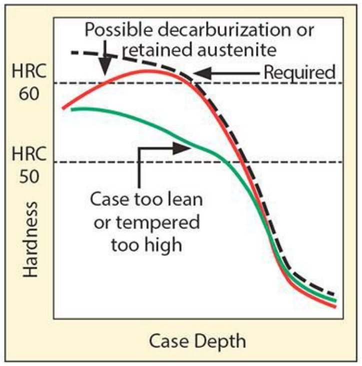


Fig. 4. Low case hardness

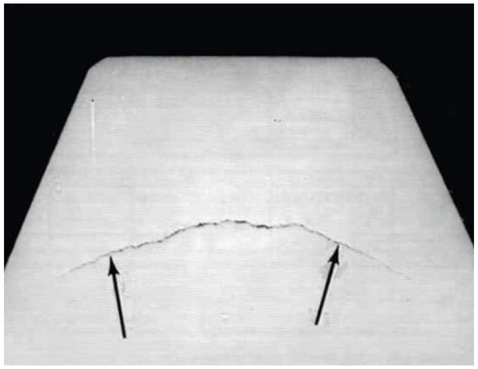


Fig. 5. Case/core separation in a gear tooth

## Selected Carburization and Case Leakage

During carburizing, a variety of stop-off paints and/or copper-plating methods (i.e. masking techniques) may be used to selectively carburize certain component areas. If these techniques prove faulty, the carburizing atmosphere can "leak" under the protective layer. Causes include surface contamination or improper surface preparation (i.e. oils, greases, dirt remaining on the surface) leading to blisters or irregularities; inadequate drying time; attempting to paint in too high a relative humidity atmosphere; improper copper-plating methods (e.g., adherence issues such as flaky surfaces, too thin a layer of copper); and overly aggressive blasting after plating.

Selecting the proper stop-off technique and material for the job, preparing surfaces properly, allowing adequate drying time, performing a low-temperature bake at 150˚C (300˚F), using "controlled" cleaning (after and prior to carburizing) and baking of parts after copper plating will ensure a proper outcome. When post-nital-etch checking of gears, for example, suspect areas appear as irregular, dark-gray indications in an area that should be light gray in appearance.

## Case Cracking/Case Separation/Case Crushing

Occasionally, cracks (Fig. 5) are found to occur within the case (typically originating in the subsurface). This phenomenon is known as case/core separation (or case cracking/case separation) and often leads to case crushing (Fig. 6) – the inability of the case to support the applied load. In gears, this is not to be confused with pitting, a form of surface fatigue failure of a gear tooth. Microcracking near massive carbides is also reported to cause case cracking.

Case/core separation is often due to improper part geometry (e.g., thin and thick sections on the same component) and/or carburizing to a case depth that is too deep. Eliminating high carbon concentrations at edges and in corners, allowing adequate stock allowance (for possible post-heat-treat material removal) and selecting the proper carburized case depth are all ways to help eliminate this phenomenon.

## Tempering Effects

The question is often asked of a carburized part, should the tempering temperature be selected to achieve the targeted hardness in the case, the core or both? As it turns out, the case is much more sensitive to the tempering temperature selected than the core.

Tempering temperature, time at temperature and, in some instances, cooling rate after tempering are important factors to consider. The goal is to produce a tempered-martensite structure in the carburized-case region while maintaining proper surface hardness.

## Other Issues

For the most part, the problems with atmosphere carburizing are well known as are their solutions. It is "the enemy we know," which is somehow a comforting thought. Control of process- and equipment-induced variables combined with a robust quality-assurance program will avoid the problems discussed here as well as others that might arise.

So, there you have it. Enough information about carburizing problems/solutions to avoid the pitfalls of taking the process for granted and assuming nothing can go wrong. Remember, the old oyster in the oyster bed remained where he was and didn't wander off with the Walrus and the Carpenter. Experience kept him off the dinner table.

## References

1. Herring, D. H., Fundamentals of Carburizing and Carbonitriding, Practical Learning Series, ASM International, 2001.
2. Weires, Dale J., Gear Metallurgy, Effective Heat Treating and Hardening of Gears Seminar, SME Short Course, 2007.
3. Mr. Darwin Behlke, Twin Disc Inc., private correspondence.

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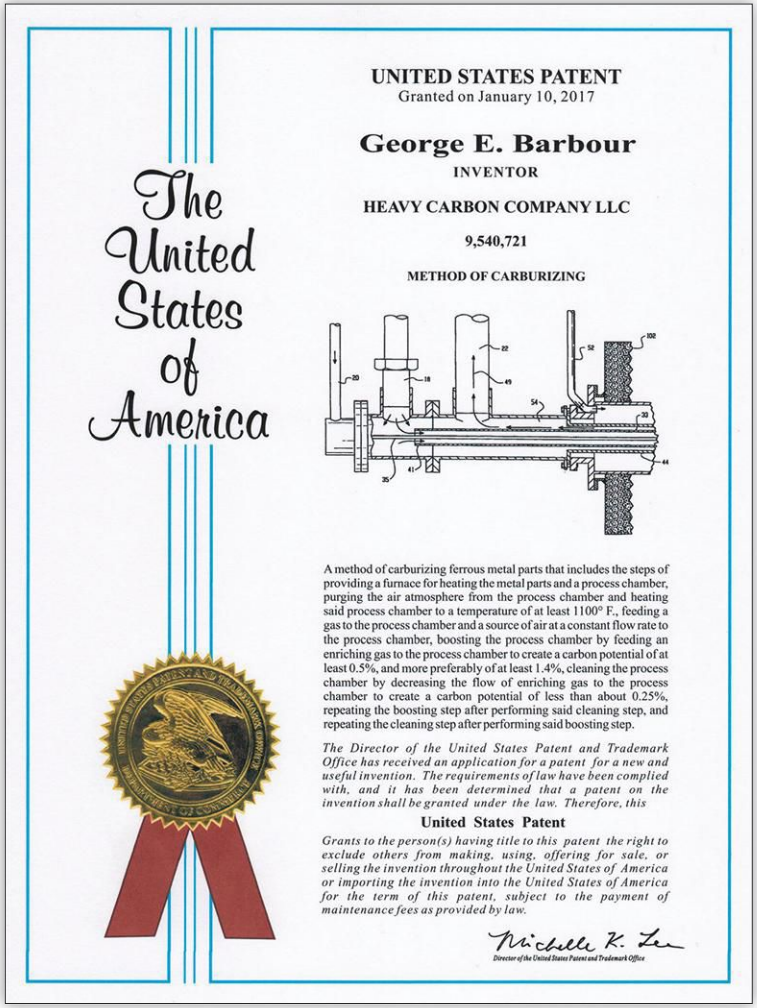
# Method for Accelerating Atmosphere Carburizing

Invention is defined as the act of creating something new or original, often in the form of a process or device. It is commonly thought to be 1% inspiration and 99% perspiration. Such is the case (pardon the pun) for one individual that The Doctor met nearly 40 years ago, who was absolutely convinced he had found a better method of atmosphere carburizing. His success is both a testament to the human spirit and a triumph of unwavering belief in an idea (combined with a great deal of persistence and hard work). The time has come to report on what appears to be a revolutionary breakthrough in atmosphere carburizing. Let's learn more.

Heavy Carbon Company LLC (Pittsford, Mich.) and its founder/inventor, George Barbour, have been granted a patent (Fig. 1) for a technology and device that allows the user to carburize faster – over the limit of saturation of carbon in austenite – without forming excessive carbides or sooting up the furnace. In addition, the system can be easily retrofitted to any atmosphere-carburizing furnace.

In two separate and in-depth case studies, one of which will be reported in detail in an upcoming article in *Industrial Heating*, The Doctor has witnessed the invention at work. Testing was done at



The United States of America

**UNITED STATES PATENT**
Granted on January 10, 2017

**George E. Barbour**
INVENTOR

**HEAVY CARBON COMPANY LLC**

**9,540,721**

**METHOD OF CARBURIZING**

A method of carburizing ferrous metal parts that includes the steps of providing a furnace for heating the metal parts and a process chamber, purging the air atmosphere from the process chamber and heating said process chamber to a temperature of at least 1100° F., feeding a gas to the process chamber and a source of air at a constant flow rate to the process chamber, boosting the process chamber by feeding an enriching gas to the process chamber to create a carbon potential of at least 0.5%, and more preferably of at least 1.4%, cleaning the process chamber by decreasing the flow of enriching gas to the process chamber to create a carbon potential of less than about 0.25%, repeating the boosting step after performing said cleaning step, and repeating the cleaning step after performing said boosting step.

*The Director of the United States Patent and Trademark Office has received an application for a patent for a new and useful invention. The requirements of law have been complied with, and it has been determined that a patent on the invention shall be granted under the law. Therefore, this*

**United States Patent**

*Grants to the person(s) having title to this patent the right to exclude others from making, using, offering for sale, or selling the invention throughout the United States of America or importing the invention into the United States of America for the term of this patent, subject to the payment of maintenance fees as provided by law.*

Michelle K. Lee
*Director of the United States Patent and Trademark Office*

Fig. 1. The patent

Euclid Heat Treating Company (Euclid, Ohio), a commercial heat treater running a typical batch IQ furnace, and then compared with in-house processing using conventional endothermic-gas carburizing. The metallurgical analysis results of these side-by-side comparison tests along with the tangible benefits to end users are impressive.

Oerlikon Fairfield (Lafayette, Ind.) is one company that has explored this technology and found it meets their expectations. The Endocarb system, as it is called, allows for rapid yet precise variation of the carbon potential in the furnace at critical times, thereby shortening the overall carburizing time. This system differs from conventional carburizing in several key ways:

1. The device is the source of endothermic gas and can be mounted on the carburizer, simplifying gas piping and avoiding transmission issues. The close proximity of the carrier gas also minimizes the risk of leaks and decreases the amount of upkeep necessary to maintain the system. The system can easily be set up so that the furnace runs at reduced endothermic-gas flowrates.
2. At 925°C (1700°F) the process can be set to run at a carbon potential of 1.5%, which is significantly higher than conventional values often in the range of 1.05-1.10% but certainly no higher than 1.2%.
3. The increased carbon potential decreases the run time for the load. For a larger 320-kg (700-pound) bull gear (Fig. 2), the carburizing time to achieve a 2.3-mm (0.090-inch) effective case was decreased by nearly 18%.
4. While conventional carburizing maintains a constant carbon potential during the boost phase, the Endocarb system uses “carbon cycling” (i.e., the process starts at a much higher potential, then is rapidly lowered well below the initial setpoint). This cycling continues throughout the run. In addition, at certain points, air is pumped into the furnace and a “controlled burnout” performed. As a result, atmosphere is rejuvenated and the furnace itself remains clean and soot-free.

## Trial Plan

In order to determine the quality and potential cost savings of this technology, tests comparing it against conventional carburizing were deemed absolutely necessary. Oerlikon Fairfield decided to conduct such an investigation to determine whether the time savings claimed could be achieved without sacrificing metallurgical quality or the performance characteristics of their gears.

Fig. 2. Carburized bull gear

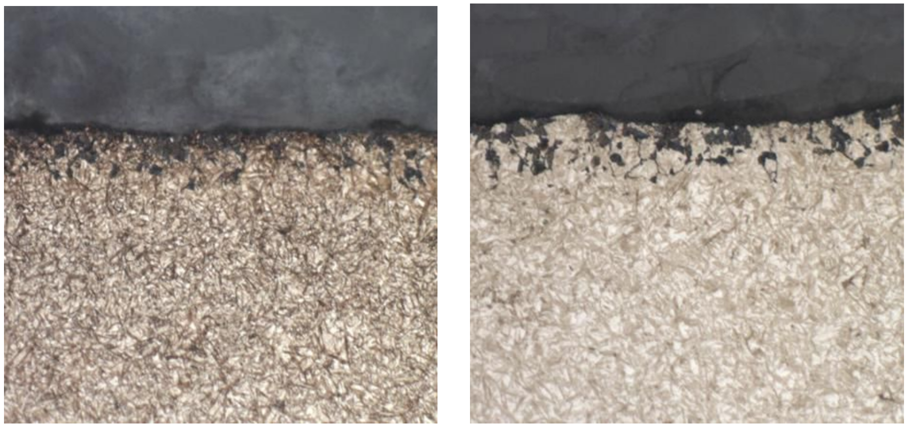


Fig. 3. Typical microstructure of bull-gear tooth #2 flank (midpoint) showing a matrix of tempered martensite and retained austenite (3% Nital, 500X)

Two identical gear sets were chosen for the trials involving six different materials (8620, 4320, 8822, 4820, 3310 and 4817) and eight different part numbers. Metallurgical results were compared to both internal and customer specifications and included checks of surface and core hardness (flank and root), effective case depth (tip, flank, root), case and core microstructure, carbide morphology, surface carbon content, retained-austenite percentages, NMTP percentages, decarburization and IGO/IGA.

For testing purposes, the austenitizing and carburizing temperatures were held constant for both trials. The parts were furnace-cooled after carburizing from both processes, then reheated and quenched in the same furnace to eliminate any difference related to the quenching process.

## A Glimpse at the Test Results

Several of the important benchmarks were consistency of surface hardness (Table 1) and effective case depth (50 HRC). Samples were evaluated independently by all parties involved and the writer.

The bottom line is that the parts in both trials met all specifications and few, if any, significant differences were observed, indicating that this new technology performed in a virtually identical way to the existing technology.

Microstructural evaluation (Fig. 3) confirmed that high-quality parts were produced in both tests, indicating that the Endocarb system had a distinct advantage based on shorter cycle time and a more cost-effective process.

It should be noted that the technology has been investigated by other interested companies. Although the results of these trials have not been made public, similar results in terms of quality and cycle-time savings have been observed.

## Summary

As the readers of this column know, we do not endorse companies or their products per se, but rather focus on heat-treating processes, methods and equipment innovations that advance the state-of-the-art. This is one such instance. Look for the full article in the April 2018 issue of *Industrial Heating*, which will provide a more in-depth analysis of the technology, summarize all test results and present a business case for implementing the system. Trust, me, you will like what you see.

**Table 1. Surface hardness comparison**

| Surface hardness (HRC, tooth midpoint) | Bull gear A | Bull gear B | Bull gear C | Diametrical pitch (inches) |
|---|---|---|---|---|
| Standard process[a] | 61.5 | 63.8 | 63.8 | 2.7922 |
| Standard process[b] | 62.7 | 63.1 | 63.1 | 2.7922 |
| New technology[a] | 63.7 | 62.6 | 61.6 | 2.7922 |
| New technology[b] | 63.5 | 62.7 | 63.3 | 2.7922 |

Notes: a. Oerlikon Fairfield testing; b. Third-party testing

# Secrets to Controlling the Carburizing Process

A wise man once said that anything worth doing is worth doing right. In the case of carburizing, this is especially true since, from both an engineering and a heat-treating perspective, we often take the process for granted – a dangerous precedent that can get us in big trouble. It's time to review the basics. Let's learn more.

Carburizing, whether performed in atmosphere or vacuum, is of critical importance to the performance of a given product. For example, in the case of gears, the method of heat treating influences properties (Fig. 1) while carbon content and its distribution in the carburized case affects such engineering properties as:

- Strength (static and dynamic)
- Toughness
- Pitting resistance
- Case-crushing strength (to determine minimum case depth)
- Wear resistance
- Sensitivity to cracking
- Grinding burns
- Operating life

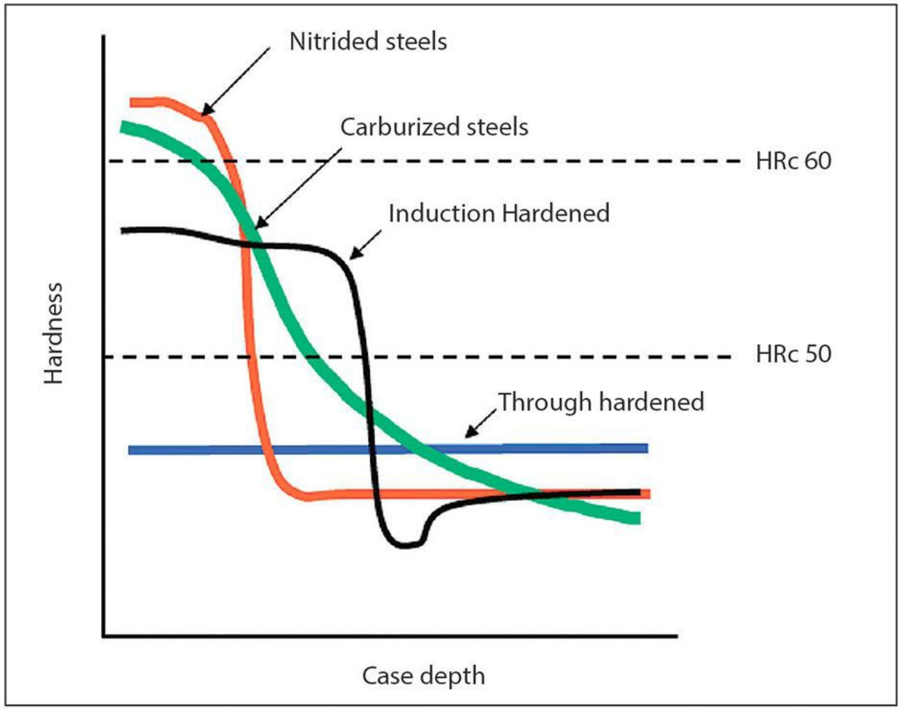


Fig. 1. Influence of heat-treating process on the gear-tooth hardness profile[1]

While the effective case depth (ECD) of a gear is often measured as 50 HRC, the depth of high hardness – generally considered >58 HRC – is a major contributor to the improvement of the properties listed above. Other considerations in the

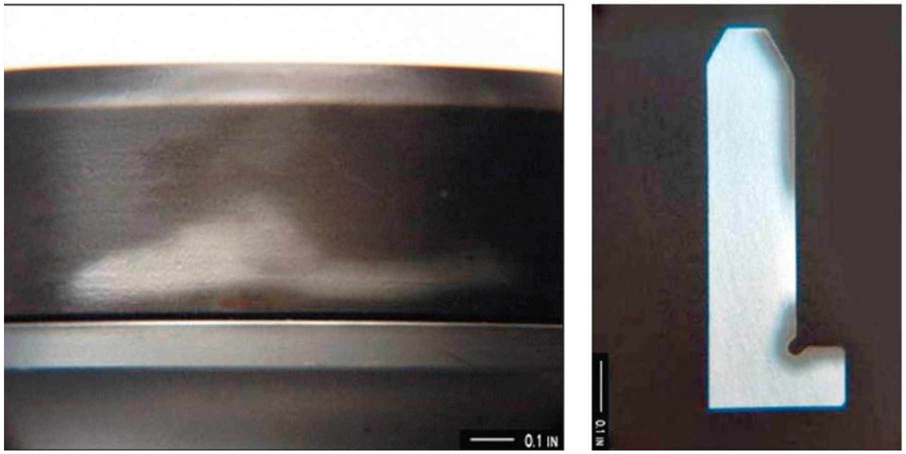


Fig. 2. Example of improper carburization – soft spots. (a) Nital etched bearing journal revealing irregular white patch. (b) Bearing-journal cross-section revealing area of no carburization.

production of a quality gear include microstructure (tip, active flank, root), core hardness at the center of the tooth, hardness at the tooth flank and root, effective case depth (root fillet), and case-depth variation (flank-to-root).

## The Importance of Specifications

In most organizations, the engineering department selects the material and specifies the properties required to satisfy a given customer's product performance demands. They often write the heat-treat specifications as well. The interpretation of these specifications, however, is often left to the heat treater, who must be aware of his equipment limitations, operating condition and current maintenance state.

Well-written specifications serve a number of important purposes.

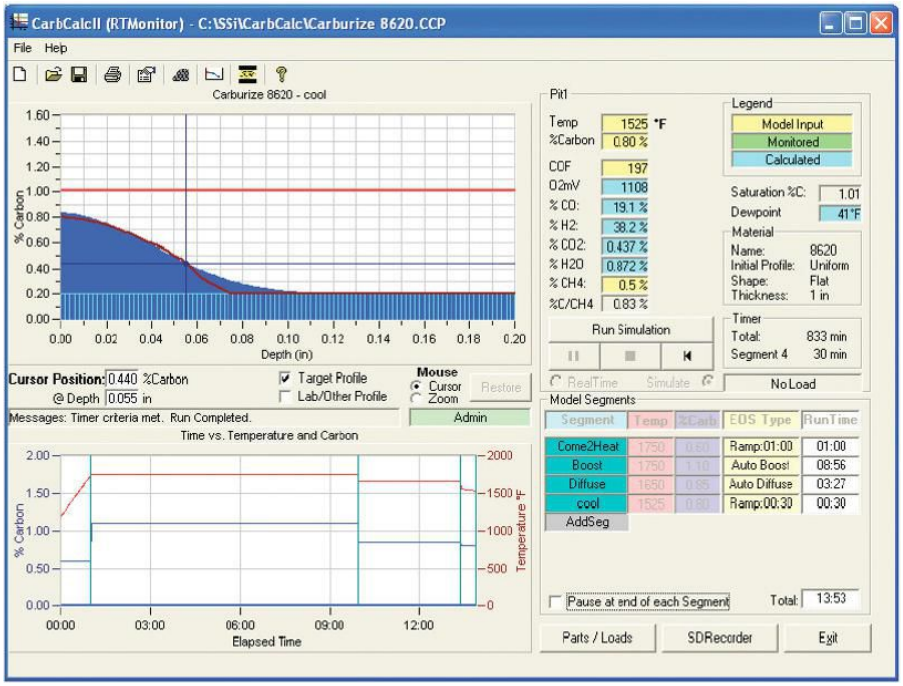
- To provide a way to convey the desired properties (e.g., mechanical, physical and metallurgical) for a specific material and part number
- Serving as a set of instructions, often in the form of a recipe for the heat treater to follow
- To provide a mechanism to capture changes to the manufacturing process (including heat treatment) so that innovations or experience-based procedures are not lost
- Serving to help the engineer choose the correct heat-treatment method and communicate it with less drawing clutter.

## Elements of a Good Specification

Too often specifications do not reflect what is actually being done. We forget to fully document the entire heat-treatment process, including the anticipated distortion state after heat treat and what must be done to compensate for it (e.g., stock allowances, straightening procedures, post-machining methods). In addition, a feedback step MUST be included so that changes can be captured into the document. Working together, the engineer and heat treater should call out the following as a minimum:

- Scope
- Application (including allowable tolerances)
- Drawing reference
- Pre-processing steps
- Pre-cleaning
- Stress relief (if necessary)
- Loading (configuration, fixturing, net and gross loading)
- Carburizing (temperature, time, carbon potential)
- Hardening (temperature, time)
- Tempering (number, temperatures, times)
- Deep freezing (if required)
- Inspection methods (e.g., hardness, NDT, etc.)
- Post-cleaning
- Straightening (if required)
- Post-processing steps
- Metallurgical checks
- Rework allowances
- Records and reports
- Manufacturing feedback (e.g., heat treating)

The application should detail the desired microstructure (e.g., martensite percentage, allowable non-martensitic transformation products, carbide type and distribution, allowable



Cursor Position: 0.440 %Carbon
@ Depth 0.055 in

☑ Target Profile
☐ Lab/Other Profile

Mouse
● Cursor
○ Zoom

Messages: Timer criteria met. Run Completed.

Pit1

Temp 1525 °F
%Carbon 0.80 %
COF 197
O2mV 1108
% CO: 19.1 %
% H2: 38.2 %
% CO2: 0.437 %
% H2O 0.872 %
% CH4: 0.5 %
%C/CH4 0.83 %

Saturation %C: 1.01
Dewpoint 41 °F

Material
Name: 8620
Initial Profile: Uniform
Shape: Flat
Thickness: 1 in

Timer
Total: 833 min
Segment 4 30 min

No Load

Model Segments

| Segment | Temp | %Carb | EOS Type | RunTime |
|---|---|---|---|---|
| Come2Heat | 1750 | 0.60 | Ramp:01:00 | 01:00 |
| Boost | 1750 | 1.10 | Auto Boost | 08:56 |
| Diffuse | 1650 | 0.85 | Auto Diffuse | 03:27 |
| cool | 1525 | 0.80 | Ramp:00:30 | 00:30 |
| AddSeg | | | | |

☐ Pause at end of each Segment
Total: 13:53

Fig. 3. Example of carburizing simulation software (Courtesy of Super Systems, Inc.)

retained-austenite percentage, etc.) as well as required testing (e.g., case depth, surface and core hardness, Charpy values, etc.). Important properties such as rolling-contact-fatigue values and maximum bending stress for gearing should also be identified.

Testing methods and locations should be clearly identified (Fig. 2) and the type of test samples specified. Sampling methods, anticipated values (e.g., oxygen-probe millivoltage, three gas-analyzer readings, dew-point values) and confirmation methods such as shim stock, turn bars, spectrographic-analysis coupons and fracture bars should be fully detailed. The failure to provide complete information means that the heat treater must try to anticipate these types of requirements, which may unintentionally introduce variables into the process.

Communication between engineering and the heat treater or between engineer and supplier is critical to success. The excuse "he should know what to do" just isn't part of an acceptable quality plan today.

## Material Selection

In A number of important factors need to be considered when choosing a material for carburizing, not the least of which is the steelmaking process, chemistry, trace elements (and their percentages), inclusions (type and distribution), material form, hardenability (Jominy, DI), grain size and mill treatments. It is especially important to know the source of the raw material (e.g., foreign or domestic source, mill heat or mixed lot). These elements can be divided into the following general classes:

- Design aspects
- Environment
- Metallurgical requirements
- Manufacturing needs

For example, manufacturing must be concerned about material form, cost, availability and quantity, heat treatment, inspection, equipment availability and condition, familiarity with the material in question, and machinability to name a few.

## How to Achieve Total Control

Today, whether atmosphere or vacuum carburizing, a number of simulation packages (Fig. 3) can be employed to ensure repeatability and documentation of results. These systems not only provide for real-time monitoring and control of results, but they allow "what if" scenarios to aid in the establishment and refinement of process cycles.

Despite these control measures, more and more companies are requiring the sacrifice of one or more parts in a load for complete metallurgical work-up and mechanical testing. This procedure – once almost exclusively restricted to aerospace – has become commonplace in many automotive and commercial heat-treat shops. One caution, however, is to select parts from representative areas within the load, not just those that are convenient to the operator. Predetermining where the part is to be taken from in the load assures that results truly represent the heat treatment that has taken place.

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# Interpreting Carburized Case Depths (Part 1: Hardness Testing Methods)

The Internet is a wonderful place, full of invaluable information available, literally, at our fingertips. Did you ever wonder, however, how much of the information you're viewing is truly accurate? An intriguing question raised by a reader about hardness testing of carburized case depths is one such example. Let's learn more.

## The Question

As part of suggested guidelines for designers, it was recommended that hardness on drawings be specified as Rockwell "C" with a minimum spread of 5 points or as a minimum hardness value, again in Rockwell C. The statement was then made that if total case depth is less than 0.76 mm (0.030 inch), an accurate reading cannot be obtained on "C" scale because a 150-kg load will drive the penetrator through the case and into core material, giving a false reading. Various Rockwell scales were recommended (Table 1) for use with minimum total case depths. The question was, "How accurate is this data?"

## The Answer

There is indeed a minimum case depth that will allow its accurate determination by indentation

**Table 1.** Rockwell scales as a function of total case depth

| Total case depth, mm (inches) | Internet source recommended Rockwell scale |
|---|---|
| 0.76 (0.030) | "C" |
| 0.61 (0.024) | "A" |
| 0.53 (0.021) | "45N" |
| 0.46 (0.018) | "30N" |
| 0.38 (0.015) | "15N" |
| < 0.38 (0.015) | File check |

**Table 2.** Rockwell scales as a function of total case depth[1]

| Effective case depth,[a] mm (inches) | Estimated total case depth[b] | SAE J423 recommended Rockwell scale |
|---|---|---|
| 0.53 (0.021) | 0.79 (0.031) | C |
| 0.46 (0.018) | 0.68 (0.027) | D |
| 0.38 (0.015) | 0.56 (0.022) | A |
| 0.30 (0.012) | 0.46 (0.018) | 45N |
| 0.25 (0.010) | 0.38 (0.015) | 30N |
| 0.18 (0.007) | 0.25 (0.010) | 15N |
| <0.18 (0.007) | <0.25 (0.010) | Microhardness |

Notes: Effective case depth defined as 50 HRC as determined by microhardness testing. Total case depth estimated using the formula (3/2) x effective case depth

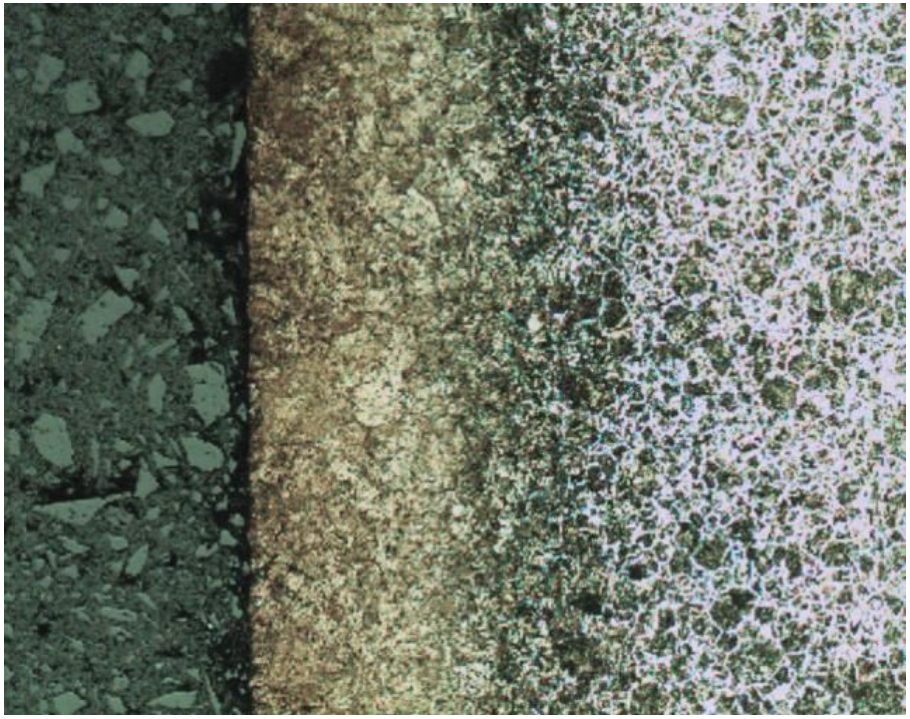

Figure 1. Total case-depth determination by the visual method[4]

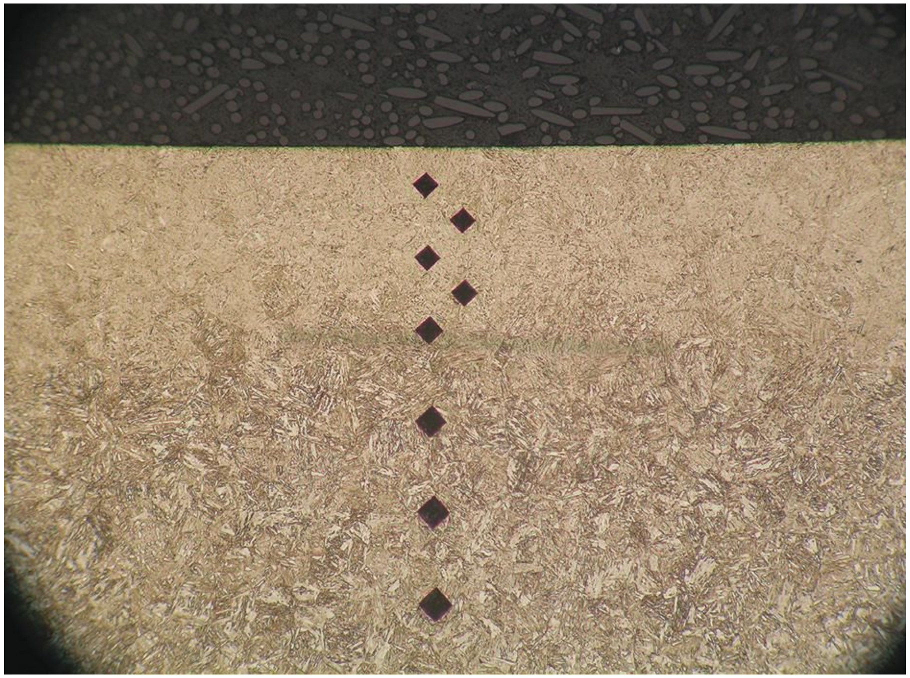

Figure 2. Example of effective case-depth determination by microhardness testing (courtesy of Solar Atmospheres, Inc.)

surface hardness measurements using standard and superficial hardness tests (Table 2). Note that the values are listed for effective case depths in this table, which are different than the total case depth information in Table 1.

## Understanding Hardness Testing of Case-Hardened Parts

In general, there are four controlling factors in the selection of the proper scale for hardness testing, namely type of material (chemistry and hardenability), thickness of the specimen, width of the area to be tested and scale limitations. It is this last factor that is often open to interpretation.

In the case of Rockwell C, typical applications (per ASTM E18) include "deep case-hardened steel." In the case of Rockwell A, its use includes "shallow case-hardened steel." These statements, while interesting, do not in and of themselves tell us if the data in Table 1 is correct.

The regular or superficial Rockwell scales are established such that an infinitely hard material will read 100 on the diamond penetrator scales (or 130 on the ball penetrator scales). Thus, one regular Rockwell number represents a penetration of 0.002 mm (0.000080 inch). Therefore, a reading of 60 HRC indicates penetration (from minor to major load) of 100 – 60 = 40 x 0.002 = 0.08 mm (0.0032 inch). By contrast, in Rockwell superficial testing, one superficial Rockwell number represents a penetration of 0.001 mm (0.000040 inch). Therefore, a reading of 90 HR15N indicates penetration from minor to major load of (100 – 90) x 0.001 = 0.01 mm (0.0004 inch).[2] However, Rockwell superficial testing, due to the lighter applied load, has a greater margin for error, which is why Rockwell C or Rockwell A testing is preferred.

## Understanding Case Depth[3]

Here are the most commonly accepted definitions for case depth in terms of carbon content.

- **Total case depth:** The depth at which the carbon content of the steel is 0.04% above the core carbon content of the steel. In other words, total case depth is the point at which differences in the chemical or physical properties of the case and core no longer can be distinguished (Fig. 1). Many metallurgists determine the total case depth by (nital) etching a steel sample and measuring the zone affected by the etchant.
- **Effective case depth:** The depth at which the carbon content of low-alloy steel is 0.40%, or approximately 0.30% for medium-alloy and high-alloy steels.

Since it is often time consuming to measure the carbon concentration as a function of depth in a carburized component to determine effective case depth, other techniques (such as microhardness) are commonly used. In certain situations involving very deep case depths, hardness is measured directly on the A or C scales. In these instances, the following definitions are used.

- **Effective case depth (U.S.):** The distance from the surface (in inches) to a point within the case where the hardness is 50 HRC (542 Knoop or 513 HV). This measurement is done on a cross section by microhardness techniques with a Knoop or Vickers indenter (Fig. 2). These values are then converted to Rockwell C hardness if a 500 gram-force load is used per ASTM E384 (latest revision). Interpolation is required to arrive at the proper case-depth value. The effective case depth depends on the carbon gradient and the case hardenability, but

50 HRC is typically equated to a carbon content of 0.40% in low-alloy steels and 0.30% in medium- and high-alloy steels.

- **Effective case depth (international):** The distance from the surface (in millimeters) to a point within the case where the hardness is 550 HV (approximately 52.5 HRC). This measurement is done by a Vickers microhardness method with a pre-defined load of 1 kg and is not converted to Rockwell C. The effective case depth still depends on the carbon gradient and the case hardenability. For highly alloyed case-hardening steels, the carbon content for 550 HV is typically in the range of 0.25-0.30%. For medium-alloy grades, it is approximately 0.30-0.35% C, and for low-alloy steels, it is approximately 0.35-0.40% C.

## Case-Depth Callouts on Prints

Case depth on engineering drawings should be stated either as total case depth, effective case depth or finished effective case depth after a defined amount of material is removed from the surface. It is important to note that if neither total nor effective case depth is specified, one must not assume that total case depth is intended (although at one time many years ago this was a commonly held belief).

A "rule of thumb" to determine total case depth from the effective case depth is to multiply by 3/2, or to determine effective case given the total case depth is to multiply the total case depth by 2/3. The steel grade, chemistry and hardenability of material (as well as case depth) can affect this percentage.

The following are several recommendations for writing clear specifications that unambiguously define the desired case.[3]

- Use wording and sketches, as needed, to define test locations and methods. However, do not use verbiage to define a hardened case.
- Specify the test procedure at each location and for each characteristic. State the maximum and minimum specification tolerance.
- Specify the condition of the part at the time of testing (as-hardened, after temper, after final grind).
- Show the draft print and specification to your test lab and part producer and check their interpretations of the specification against your requirements.

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# Comparing and Contrasting Carbonitriding and Nitrocarburizing

The terminology of heat treating is sometimes challenging. Heat treaters can be inconsistent at times, using one word when they really mean another. You have heard the terms carbonitriding and nitrocarburizing and know they are two different case-hardening processes, but what are the real differences between them? Let's learn more.

Part of our confusion stems from the fact that years ago carbonitriding was known by other names – "dry cyaniding," "gas cyaniding," "nicarbing" and (yes) "nitrocarburizing."

## The Carbonitriding Process

Carbonitriding is a modified carburizing process, not a form of nitriding. This modification consists of introducing ammonia into the carburizing atmosphere in order to add nitrogen into the carburized case as it is being produced (Fig. 1).

Carbonitriding is typically done at a lower temperature than carburizing, from as low as 700-900°C (1300-1650°F), and for a shorter time than carburizing. Since nitrogen inhibits the diffusion of carbon, a combination of factors result in shallower case depths than is typical for carburized parts, typically between 0.075 mm (0.003 inch) and 0.75 mm (0.030 inch).

It is important to note that a common contributor to non-uniform case depth during carbonitriding is to introduce ammonia additions before the load is stabilized at temperature (this is a common mistake in furnaces that begin gas additions upon setpoint recovery rather than introducing a time delay for the load to reach temperature). It is important to remember as well that when the ammonia addition is halted, desorption of nitrogen will begin to occur.

The temperature range in which carbonitriding is performed is necessarily lower since the thermal decomposition of ammonia is extremely rapid, which limits nitrogen availability at higher austenitizing temperatures. A more brittle structure is formed at lower temperatures, and operating furnaces below 760°C (1400°F) can be a safety concern.

The nitrogen in carbonitrided steel enhances hardenability and makes it possible to form martensite in plain-carbon and low-alloy steels that initially have low hardenability. Examples of these steels include SAE grades 1018, 12L14 and 1117. The nitrides formed contribute to the high surface hardness. Like

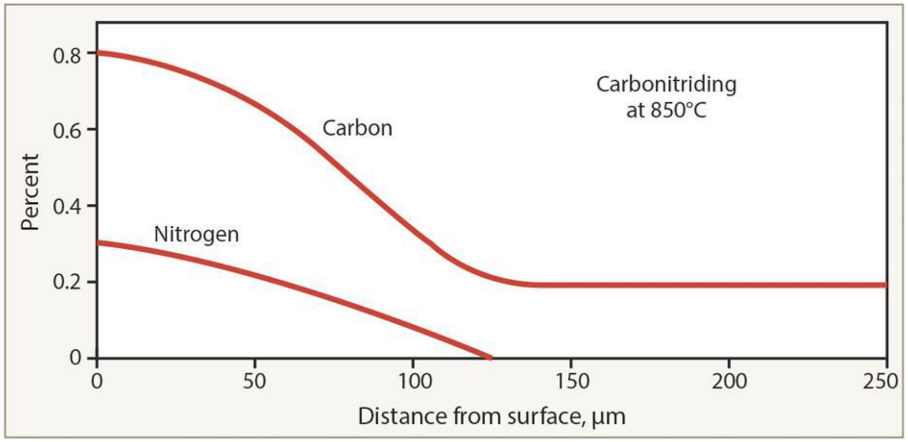

Fig. 1. Carbon and nitrogen profiles developed during carbonitriding

carbon, manganese or nickel, nitrogen is an austenite stabilizer, so retained austenite is a concern after quenching. Controlling the ammonia percentage will reduce the amount of retained austenite and should be done if hardness or wear resistance is reduced. Another consequence of high nitrogen percentages is the formation of voids or porosity. In general, it is recommended that the nitrogen content at the surface be limited to 0.40% maximum.

A common variation of the carbonitriding process is to introduce ammonia during the latter portion of the cycle, typically in the last 0.5-1 hour before the load is quenched. Any loss of hardenability that might occur due to internal (or intergranular) oxidation is partially compensated for by nitrogen absorption.

Several other points are worth mentioning. The presence of nitrogen in the carbonitrided case increases the resistance to softening on tempering (similar to some alloying elements), and the higher the nitrogen content, the higher the material's resistance to softening. Higher tempering temperatures – up to 230°C (440°F) – are often used on carbonitrided parts. The resistance to tempering manifests itself in wear properties. Carbonitrided gears, for example, exhibit better wear resistance than many carburized gears. Shallow case depth in thin-section parts of unalloyed steel, such as die-cutting punches, can be used without tempering (but this is never recommended).

## The Nitrocarburizing Process

Today, "ferritic nitrocarburizing" is commonly referred to simply as "nitrocarburizing" (and hence the confusion with the older name for carbonitriding).

### Ferritic Nitrocarburizing (FNC)

Nitrocarburizing is a modification of the nitriding process, not a form of carburizing. This modification consists of the simultaneous introduction of nitrogen and carbon into the steel in its ferritic condition; that is, below the temperature at which austenite begins to form during heating (Fig. 2).

Nitriding is typically performed using ammonia with or without dilution of the atmosphere with dissociated ammonia or nitrogen/hydrogen in the temperature range of 500-580°C (925-1075°F), although 565°C (1050°F) is traditionally considered the upper limit. By comparison, nitrocarburizing is typically performed in the temperature range of 550-600°C (1025-1110°F) in atmospheres of 50% endothermic gas + 50% ammonia or 60% nitrogen + 35% ammonia + 5% carbon dioxide. Other atmospheres that vary the composition, such as 40% endothermic gas + 50% ammonia + 10% air, are also used. The presence of oxygen in the atmosphere activates the kinetics of nitrogen transfer. The thickness of the "white" or "compound" layer is a function of gas composition and gas volume (flow). Nitrocarburizing is often followed by an oxidizing treatment to enhance both corrosion resistance and surface appearance.

A complex sequence is involved in the formation of a nitrocarburized case. It is important that a very thin layer of single-phase epsilon ( ) carbonitride is normally formed between 450°C (840°F) and 590°C (1095°F). This compound layer has an underlying diffusion zone containing iron (and alloy) nitrides and absorbed nitrogen associated with it. The white layer has excellent wear and anti-scuffing properties and is produced with minimum distortion. The diffusion zone, provided it is substantial enough, improves fatigue properties such as endurance limit, especially in carbon and low-alloy steels. Some of the increased hardness of the case is due to a diffusion zone beneath the compound layer, especially in the more highly alloyed steels with strong nitride formers.

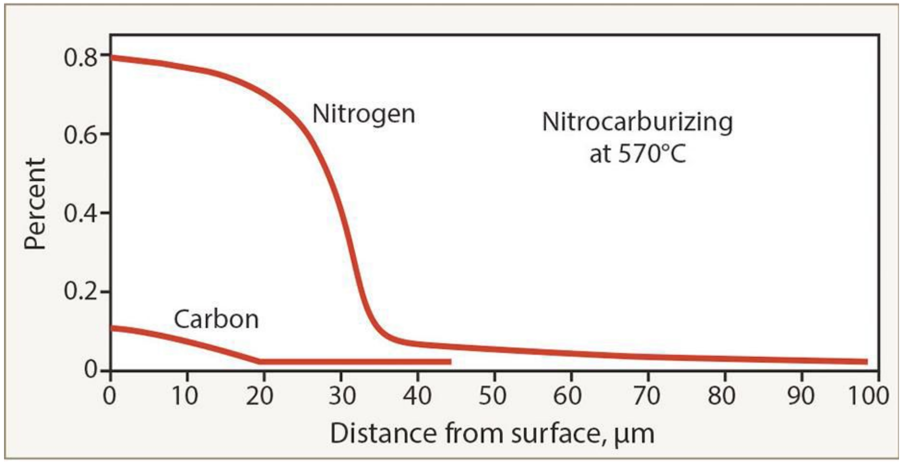

Fig. 2. Nitrogen and carbon profiles developed during ferritic nitrocarburizing

It is not uncommon to observe porosity of the compound layer due to the presence of a carburizing reaction at the steel surface, which influences the nitriding kinetics and therefore the degree and type of porosity at the surface of the epsilon ( ) layer. Three different types of layers can be produced: no porosity, sponge porosity or columnar porosity. Some applications require deep nonporous epsilon layers. Others applications where, for example, optimum corrosion resistance is needed benefit from the presence of sponge porosity. Still others benefit from columnar porosity, where oil retention can enhance wear resistance.

### Austenitic Nitrocarburizing (ANC)

A lower-temperature variant of carbonitriding is austenitic nitrocarburizing. This process takes place in the temperature range of 675-775°C (1250-1425°F). It can be controlled to produce a surface compound layer of epsilon (ε) carbonitride with a subsurface of bainite and/or martensite produced on quenching, resulting in a good support structure for the hard surface. The microstructure is particularly useful in intermediate stress-point contact-resistance applications (e.g., helical gears).

## Summary

Understanding these processes better allows each heat-treatment method to be used to its best advantage.

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# Masking Techniques (Part 1)

Certain applications require that we protect part surfaces from oxidation or limit the areas on a component part where a case-hardening process is to be performed. To accomplish these tasks, various masking techniques are employed, including copper plating, stop-off paints, physical masking and leaving excess stock allowance. It is important to understand where, why and how each method is used. Let's learn more.

One should not assume that any masking method has been properly applied or will work well for a given application without testing and taking into consideration such factors as material, part geometry, cleaning, application, drying, heat-treatment process, removal method, inspection and end-use performance.

## Plating

Copper, properly applied, is generally considered the best method of part protection. It is also the most costly. Copper plating can be used in both atmosphere and vacuum carburizing. AMS 2418 Rev H (2011) details the process requirements. A nickel strike (nickel flash) is often used as an under layer for improved copper adhesion, especially when processing highly alloyed steels (e.g., Pyrowear 675, M50Nil, Vasco X2). At high hardening temperatures, however, nickel has been observed to diffuse into the base metal, which has the same appearance under the microscope as a decarburized surface layer.

Platinum and palladium plating are also reported to work well for specialized materials (e.g., titanium) but are extremely expensive and should only be considered if the project cost warrants such expense.

## Paints

Stop-off paints are widely used throughout the heat-treat industry either as an alternative to copper plating or for touch-up on plated surfaces that have been damaged.

### Carburizing and Carbonitriding

The most commonly used stop-off paints for carburizing and carbonitriding are solvent- or water-based coatings with copper or boron as the main ingredient. Paints containing copper are not particularly suited for carbonitriding because of the possible chemical reaction with ammonia in the furnace atmosphere. The biggest advantage of boron-based paints is that the residues that remain

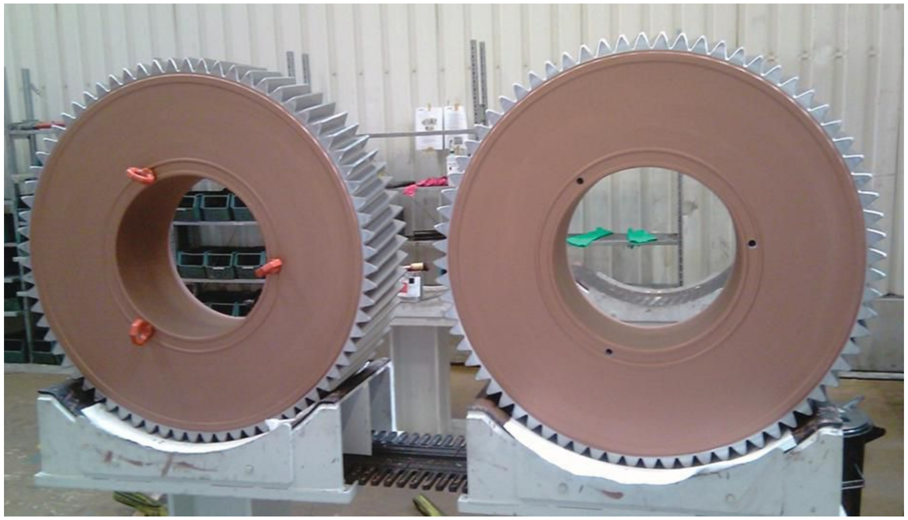

Fig. 1. Wind-energy gears ready for deep-case carburizing
(Photograph courtesy of The Duffy Company and Nüssle GmbH & Co. KG.)

are soluble in hot water and alkaline solutions. To a certain degree, they "rinse off" during quenching. These paints are the best choice for applications where mechanical cleaning is not a viable option.

Boron-based paints go into a semi-liquid state at temperature when heated. If the coating is too thick, the mass of the paint will cause it to run onto uncoated areas. It is for this reason that the paint thickness should be limited to 0.2-1.0 mm (0.008-0.040 inch). Only one coat is required as long as this coating is uniform. Thicker is not better. Two coats are only recommended for geometries prone to carburization from two sides, such as threads.

Solvent-based paints containing boron oxide are still in common use today and are considered by many to offer the best protection in this class of coatings.

### Deep-Case Carburizing

For total case depths over 2 mm (0.080 inch), silicate-based paints are recommended over boron-based paints (Fig. 1). Silicate-based stop-off paints are normally applied in two or three layers depending on the required case depth. After heat treatment, the glass-like residue is not water or solvent soluble but must be removed by blasting. An advantage of these paints is that they will not run even if the coating thickness is excessive. Unlike water-based boron paints, they are not subject to the problem of glazing of the furnace interior.

### Vacuum Carburizing

In general, stop-off paints used for vacuum carburizing are similar (but not identical) to those used in atmosphere carburizing, especially where acetylene is used as a hydrocarbon-gas choice. Boron paints are used for applications requiring the paint to be washed off after heat treatment. If mechanical removal (shot blasting) of the stop-off paint is acceptable, silicate-based copper-oxide paint should be the first choice.

### Gas Nitriding and Nitrocarburizing

Stop-off for gas nitriding and nitrocarburizing (Fig. 2) contain fine tin powder dispersed in a lacquer, consisting of a solvent and synthetic binder or water and synthetic emulsion. The stop-off effect is based on a layer of molten tin dispersed onto the part surface, which acts as a gas-tight barrier that prevents the diffusion of nitrogen. It must be noted that preheating coated parts in air must be limited to 380˚C (715˚F) maximum. Exceeding this temperature limitation will prove detrimental to the uniformity of the tin plating. After processing, powdery residues that can be easily removed by wiping or brushing remain. It must be noted that there is a microscopic layer of tin left on the part surface. In the event this is problematic, blasting or machining can remove it.

### Plasma (Ion) Nitriding

For ion nitriding, the most commonly used stop-off technology is mechanical masking (shielding). If the geometry of the part does not lend itself to this type of protection, stop-off paints are available either based on copper (electrically conductive) or ceramic ingredients (nonconductive). The residues of these paints are powdery and are most often removed by wiping or brushing.

Fig. 2. Tool-steel parts ready for gas nitriding
(Photograph courtesy of The Duffy Company and Nüssle GmbH & Co. KG.)

### Scale Prevention

Stop-off paints are available to prevent scale and oxidation in furnaces that are running processes such as annealing, normalizing and stress relief in air or products of combustion. These paints are used for annealing, stress relieving, normalizing and hardening. In these applications, the entire part surface is coated to prevent scale from forming when heated up to 850˚C (1560˚F). There are also lacquers and ceramic-based coatings available for applications up to 1200˚C (2200˚F). These coatings create a glass-like barrier to prevent the scale from forming. Upon cooling, the coating will begin to spall off due to differential expansion of the coating and the part surface. Mechanical removal after processing is usually still required.

## Application Methods

Brushing, although labor intensive, is perhaps the most common application method for stop-off paints. Using a flat, clean brush with soft bristles, the paint should be applied in an even, thin layer of uniform thickness. When applying the paint to the part, resist the temptation to put excessive pressure on the brush, and let the paint flow off the brush in a uniform manner. If the paint rolls off the part surface and back to the brush, there is oil or some contaminants on the surface that must be removed. If solvent-based stop-off is used, storing brushes between coats must be done in a container with the same solvent as the paint to ensure there is no adverse chemical reaction or paint contamination.

Dipping (part immersion) is the simplest way to coat large numbers of parts. If the area to be coated is at the end of the part, semiautomatic or continuous coating can be achieved with minimal investment in equipment.

Automatic dispensing and spraying can also be used if the part geometry and area to be masked are conducive to this method. Robotics and automated handling systems can be employed for high-volume applications (such as in the automotive industry). If spraying is used, it must be carefully controlled to prevent overspray and misting into the air.

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# Masking Techniques (Part 2)

We continue our discussion from last month on the where, why and how masking methods are used to protect part surfaces from oxidation or limit the areas on a component part where a case-hardening process is to be performed. Let's learn more.

## Lessons Learned

For those of us who have used (and abused) stop-off paints, valuable lessons have emerged, the first of which is that each manufacturer's product type seems to work somewhat differently. Each paint is manufactured according to proprietary formulas and, as such, their performance and protection in a given application will vary. Therefore, it is mandatory to precisely follow the advice given by the manufacturer in their technical literature and to discuss your application needs with them.

Another lesson is that no product is perfect. Just because a product works in one application, say for medium case-carburizing protection, it does not ensure that it will work for deep-case carburizing. Often, a variety of stop-off paint products are necessary. Manufacturers are also constantly striving to improve their products (and introducing new ones), making it somewhat confusing to know which product is best for a given application. Having good support from the manufacturer is critical.

There is a general "rule of thumb" that no more than 30% of the total area of the parts within a furnace load be coated with stop-off paint. Exceeding this amount can create an imbalance in the furnace atmosphere. As the parts are heated, water vapor or gases generated by thermally cracking the paint binders are released. Over time, the furnace atmosphere will recover, but the cycle disruption can influence the case-hardening process.

Product shelf life is limited (1-3 years) and dictated by the manufacturer. It is a good idea to mark the container using a simple use-by date. The paints should also be stored indoors at room temperature, and older product should be used first. Water-based paints must not be allowed to freeze.

Ideally, parts and paint should both be at room temperature when the paint is applied to clean, dry surfaces. (Caution: A clean, dry surface is essential to proper adhesion.) After thoroughly stirring the paint, the coating should be applied as uniformly as

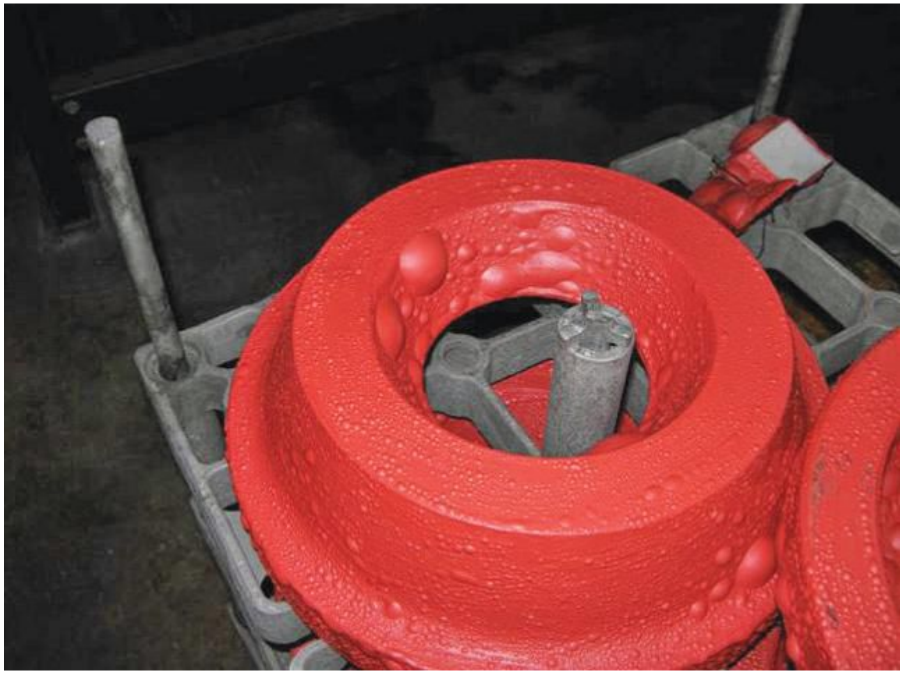

Fig. 1. Example of blistered part surface

possible in a way that the surfaces to be protected are covered completely and the part surface is completely hidden from view. Some stop-off paints are diluted before use. This should not be done arbitrarily but by following manufacturer's instructions, and if this is done, care must be taken to avoid a situation where the paint runs onto unintended surfaces during application. Multiple (2-3) thin coats are often applied, but excessive coating thickness must be avoided. A common misconception is that the thicker the coating, the better the protection. This is not the case.

Required drying time is dependent on factors such as paint composition, viscosity, coating thickness, ambient temperature and part temperature. Drying time is significantly influenced by the relative humidity in the room. Typical drying times vary from a few hours to several shifts. Solvent-based paints will dry relatively fast due to the accelerated evaporation of the solvent. Drying time for aqueous-based paint can be shortened by placing them in an air oven or furnace at a maximum temperature of 180˚C (350˚F). Exceeding this temperature could cause the paint to flow onto uncoated areas as well as compromise the effectiveness of the paint. One of the consequences of inadequate drying time is blistering or peeling (Fig. 1), which may burst and throw stop-off onto uncoated areas and cause spotty case hardening.

Recognize that many stop-off paints are hygroscopic, meaning they will absorb moisture as they sit around. It is recommended that painted parts be heat treated within 24 hours of the last coat. In high-humidity environments, it is recommended to store painted parts in an oven running no higher than 80°C (175°F) until they are used.

The painted areas of the part must be racked to ensure these areas do not come into contact with uncoated areas of other parts in the furnace load. It is also recommended that you do not use different types of stop-off paints in the same furnace load. This situation could compromise the effectiveness of one or both of the products, especially if a solvent-based boron paint and a water- or silicate-based paint are in the same load. Water- and silicate-based paints give off water vapor during the heat-up cycle, which can attack the solvent-based paint and cause it to run onto uncoated areas.

After heat treatment, it is recommended that the residue of the paint be removed promptly. The residue of some stop-off paints can react with humidity and cause a corrosive attack of the part surface. Many users are overly concerned with ease of removal and select stop-off paints based on this criterion alone. This decision must be carefully weighed against the protection afforded by the product.

In general, stop-off paints are effective for only one heat-treat operation (e.g., hardening followed by quenching). There is one exception to this rule, namely certain silicate-based paints that will prevent carbon penetration not only after carburizing and slow cool cycle but through reheat and subsequent oil or gas quenching. The parts can be cooled further in air to room temperature. These same parts can be reheated in atmosphere and oil quenched for the hardening cycle.

## Common Problems

Most of the problems encountered when using stop-off paints are well known and preventable. These include:

**Adherence:** Improper paint adherence is almost always traced back to surface cleanliness, insufficient drying time or poor application techniques.

**Blistering/Peeling:** Insufficient drying time is another leading cause of stop-off paint failures. All stop-off paints utilize a thinning agent, which must be fully evaporated prior to introduction into the furnace. Insufficient drying will cause the thinning agent to outgas, lifting areas of the paint off the part surface (blister) during heat-up.

**Incomplete Coverage and Edge Effect:** This most often occurs because of miscommunication as to which surfaces need protection or which areas need coverage. Good communication often negates these concerns. Unwanted case at the edges of the painted surface is also a concern overcome by extending the edges of the painted surfaces (if allowable).

**Product Misapplication:** One of the most catastrophic problems with stop-off paints is the choice of the wrong product for the job.

**Furnace Damage:** Damage to the furnace interior has also been traced back to the use of certain stop-off paints. Common problems include deposits on the tips of oxygen probes (skewing carbon-potential readings) and deposits on the refractory surfaces, causing "glazing" (Fig. 2). For example, boron oxide is known to combine with the water vapor present in endothermic-gas atmospheres to form boric acid. The boric acid reacts with the silicon in the refractory, causing a eutectic. This phenomenon normally happens over a long time period, and deposits may occur in areas of temperature transition (inner doors, etc.).

Both solvent- and water-based boron stop-off paints break down during the heat-treat cycle. For a water-based stop-off, chemically combined water will evolve as low as 190˚C (375˚F). Resins and binders in solvent systems evolve around 360˚C (680˚F). It is a time-temperature relationship.

## Excess Stock Allowance

This is the most-costly and least-preferred masking method and involves considerable post heat-treat time and expense (post machining). For certain areas of parts where the geometry is highly distortion-prone, however, adding extra stock provides selective carburization while minimizing distortion.

## Final Thoughts

Effective masking techniques are those that work. While experience is the greatest teacher, understanding what level of protection is needed and how best to achieve it before running component parts is critical. Million-dollar mistakes have occurred because of poor plating adherence, failure to properly clean part surfaces and using the wrong product for a given application. Get the facts and learn from others' mistakes so as to avoid your own.

Fig. 2. Glaze-coated refractory installation

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# Gas Nitriding: Something Old and Something New

The Doctor has always been fascinated with anything involving case hardening. Gas nitriding is no exception; a seemingly old and well-established technology that changes so little that it begs the question, "What's new?" Well, as it turns out, quite a bit. Let's learn more.

## Historical Overview[1-6]

Gas nitriding of iron and steel is a thermochemical process dating back well over 100 years. Dr. Adolph Machlet (U.S.) of the American Gas Company of Elizabeth, N.J., in 1913 patented the first gas nitriding process utilizing an atmosphere of ammonia. He also patented, in 1914, a process for nitrocarburizing using an ammonia and hydrocarbon gas mixture. This was followed in 1924 by a patented process developed by Dr. Adolph Fry (Germany) of Krupps Steel Company, who also employed an atmosphere of pure ammonia. Fry's investigative work on the nitriding process took place at roughly the same time as Machlet's but was interrupted by World War I. Fry's work also led to the development and patenting of steels specifically engineered for nitriding – Nitralloy® steels containing aluminum and chromium.

These single-stage nitriding processes were carried out at maximum nitrogen potential and resulted in brittle cases with high porosity as well as networks of iron carbonitrides at the grain boundaries. For most component parts, this less-than-optimal microstructure required removal of the "white layer" produced, typically by grinding.

Starting in 1922, work was under way at the U.S. Bureau of Standards on nitriding specimens with the goal of understanding the relationship between iron and nitrogen. This work was supported by C. B. Sawyer and Fry, who were both actively working on this subject around 1923 and offered versions of an iron-nitrogen phase diagram. These efforts culminated in the establishment of an updated iron-nitrogen phase equilibrium diagram in 1929.

In the 1930s, E. Lehrer (Germany) introduced, for the iron-nitrogen (Fe-N) system, a diagram relating temperature and nitrogen potential. This allowed one to accurately determine the phase boundaries in the iron-nitrogen (temperature-composition) system. The Lehrer diagram shows the solubility of nitrogen in ferrite as a function of the nitriding potential. To this end, thin iron foils were homogeneously nitrided in flowing

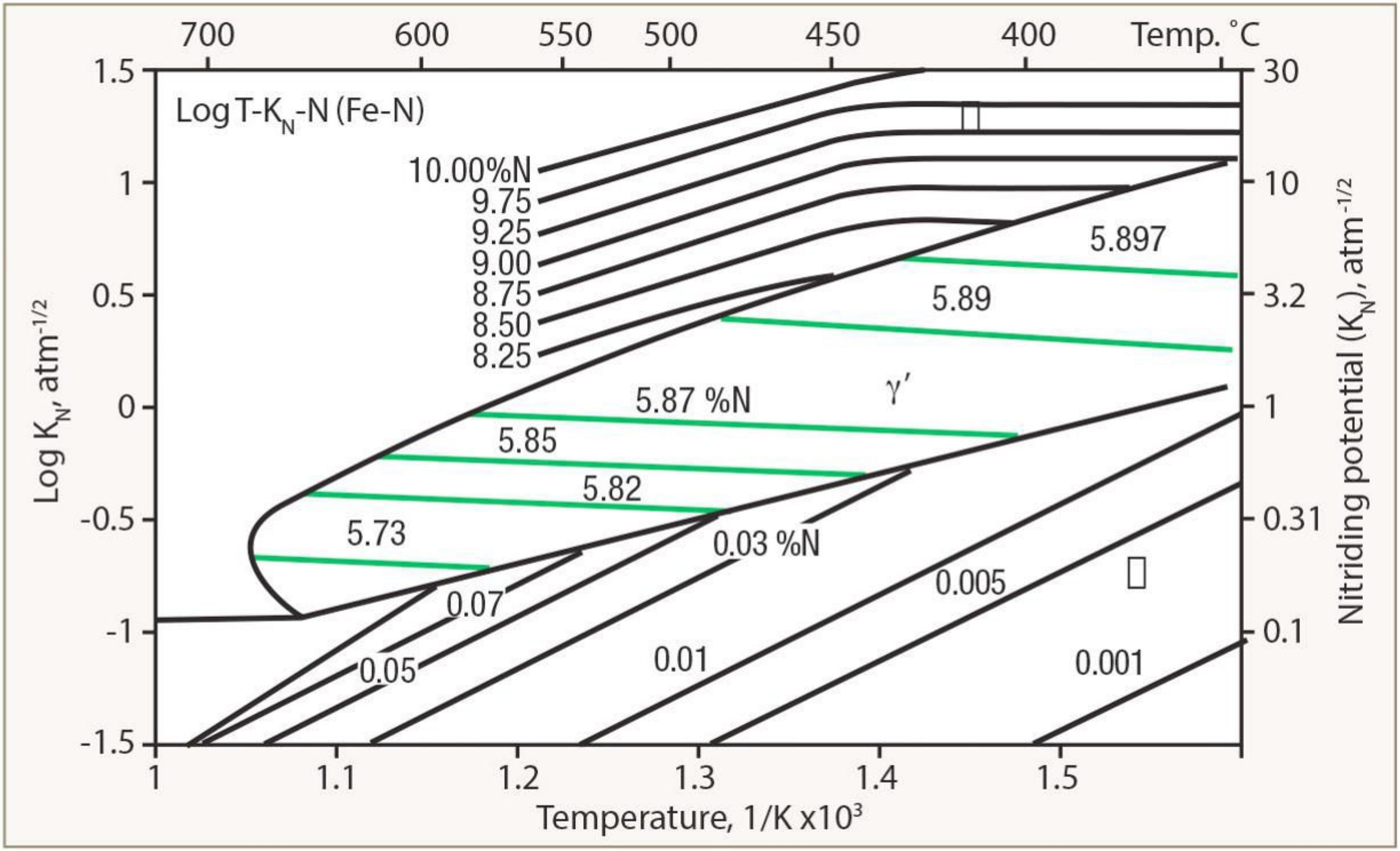

**Fig. 1. Modified Lehrer diagram (L. Małdziński) – nitriding potential vs. temperature for a given phase**

gas mixtures comprised of ammonia and hydrogen. This allowed the nitriding process and resultant case microstructure to be better understood.

Work was then undertaken to reduce the nitriding potential and the problematic white layer and culminated in 1943 with a process patented by Carl Floe (U.S.) using a two-stage nitriding process in an atmosphere of ammonia and dissociated ammonia. The Floe (pronounced "flow") process created a nitrogen-rich layer (in the first stage), then diffused this layer (in the second stage) to produce a more optimal case microstructure and reduce the need for post-heat-treatment grinding. This process helped more accurately develop a targeted case depth and microstructure.

## Modern Developments

Single-stage and two-stage gas nitriding, as originally developed, is still in use today. By the 1990s, however, Leszek Małdziński (Poland) modified the Lehrer diagram to incorporate iso-concentration curves after analysis of the results of experiments also involving thin iron foils (Fig. 1).

In simplest terms, these curves represent constant nitrogen concentration in iron (both on the surface and at the interphase boundary) as a function of temperature and nitriding potential. This overcame a shortcoming in the Lehrer diagram, which predicts the phase structure of the nitrided layer but does not provide information about the concentration of nitrogen (in α, γ′ and ε phases) as a function of temperature and nitriding potential. This is the basis for understanding the kinetics of nitrided layer growth.

Thus, not only can we determine the nitriding potential at any given temperature, but we can also predict the microstructural phases that will

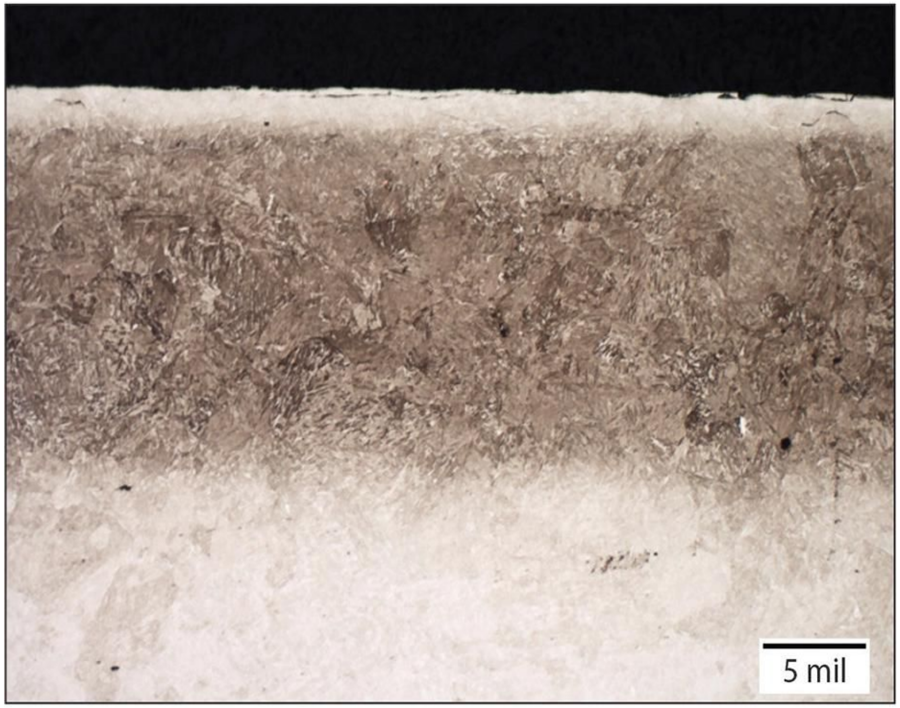

Fig. 2. Nitralloy 135M component with a uniform nitrided case approximately 0.40 mm ( 0.016 inch) in depth with evidence of a shallow 0.0055 mm (0.00022 inch) white layer along the surface.

be produced, paving the way for practical process simulators. Hence, modern gas nitriding was born, and all-new equipment, processes and controls in use today are based on this diagram.

## Putting Theory into Practice

Nitriding is a gaseous diffusion process in which nitrogen is absorbed into the surface of the steel (Fig. 2). The process begins at the surface of the part with the formation of a compound (aka white) layer, which depends to a large extent on the composition of the steel (and, in particular, the carbon content).

The phases formed within this surface layer are the epsilon ($\varepsilon$) phase and the gamma-prime ($\gamma'$) phase. Each has attributes that enhance the mechanical properties of the case and can be varied in percentage as a function of temperature and gas chemistry. The thickness of the compound layer is a function of material (plain-carbon steel forming a thicker compound layer than alloy steels), temperature, time and the process-gas composition.

Underneath the compound layer is the diffusion layer, where the diffusion of nitrogen away from the surface results in the formation of nitrides and, in some instances, carbonitrides. In general, the compound zone will have high hardness, good wear resistance, improved corrosion and other tribological properties. The diffusion zone improves fatigue resistance and other mechanical properties.

The parameter that defines the rate of dissociation of ammonia is the nitriding potential (KN). In practice, the nitriding potential is selected so as to produce the required case depth and microstructure. The higher the nitriding potential, the higher the nitrogen concentration will be both at the surface and in a particular phase ($\alpha$, $\gamma'$ or $\varepsilon$). Thus, in order to maintain a given nitriding potential, one must increase the rate of the flow of ammonia. Clearly, the higher the nitriding potential needed, the greater the flowrate of ammonia. The same is true of temperature; as it increases so too does the ammonia flowrate.

Today, advanced nitriding processes control the ammonia flowrate into the furnace retort at a given temperature and, as a result, are capable of producing single ($\alpha$), dual ($\gamma'+\alpha$) or three-phase ($\varepsilon+\gamma'+\alpha$) compound layers.

## Lessons Learned[8]

Some practical lessons have been offered to the industry and may be documented as follows:

- Increasing the temperature will increase the case depth and increase the white layer, provided an atmosphere allowing for formation of a white layer is not controlled.

- Set the nitriding potential to match the desired phase on the parts' surface. Carbon will shift the boundary to the epsilon phase to lower nitriding potentials; increasing amounts of nitride-building elements will shift the boundary to higher nitriding potentials.
- Nitride-building elements have a high impact on the nitrogen flux needed to saturate the structure. Therefore, diluting the nitriding atmosphere with nitrogen or treating the part at low pressures will stop a proper nitriding of high-alloy steels earlier compared to low-alloyed or carbon steels.
- Increasing the furnace pressure will increase the growth of the white layer, but this effect will slow down by increasing the nitride-building alloying elements.

## Summary

Gas nitriding has returned to its roots, requiring only ammonia as a process gas in systems specifically designed to vary or stop the gas flow to achieve the desired metallurgical microstructure and hardness. Flowrates are minimized and controlled by process simulators and simple sensors. Emissions from these systems are extremely low as well, so no longer can we tell if nitriding is being done in the heat-treat shop by the odor of ammonia in the air.

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# The Role of Furnace Atmosphere in Sintering (Part 1)

Powder metallurgy is a fascinating science in which customizable powder blends are used to develop unique material properties. It is particularly appealing to manufacturing due to its economics. As such, this technology continues to experience strong growth in an ever-expanding family of component parts. Both sintering and the heat treatment of sintered components require careful understanding and control of the furnace atmosphere because its role must change from one point in the process to another. Let's learn more.

## Sintering

In simplest terms, sintering (aka solid-state sintering) is the diffusion bonding of adjacent powder particle surfaces. It can be argued that sintering is not a heat-treatment operation per se but a thermal process applied to so-called "green" compacts in order to impart structural integrity and improve mechanical properties, the foremost of which is strength. Sintering causes the following changes to occur within the part:

- Particle bonding (resulting in a decrease of pore volume and an increase in density)
- Grain growth and an increase in the number and strength of interparticle bonds
- Pore morphology (size, shape)
- Alloying and homogenization
- Dimensional (reduced surface area)
- Reduction of lattice defects

The sintering process is governed by a number of material and process variables that produce a change in the part microstructure, thereby influencing its mechanical properties. These include:

- Temperature and time
- Powder particle morphology (i.e., size, shape and distribution of powder particles)
- Composition of the powder
- Density
- Sintering parameters (time-temperature-atmosphere)

## Sintering Atmospheres

The selection of a sintering atmosphere is most often dictated by the choice of material, desired properties, part design (e.g., density, mass, geometry), production demands and product end-

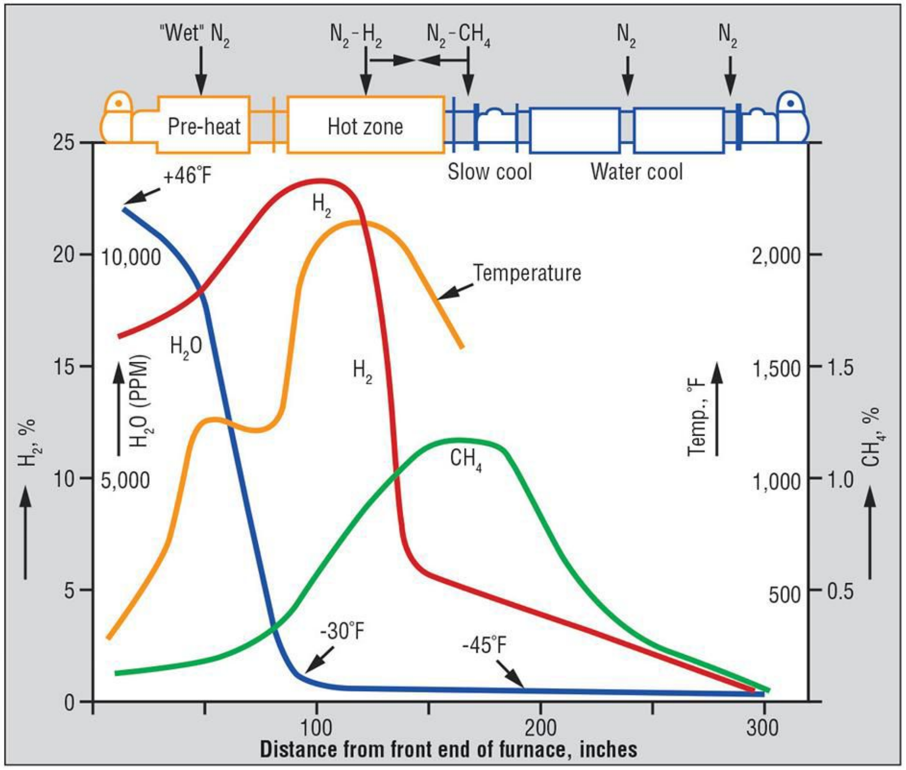
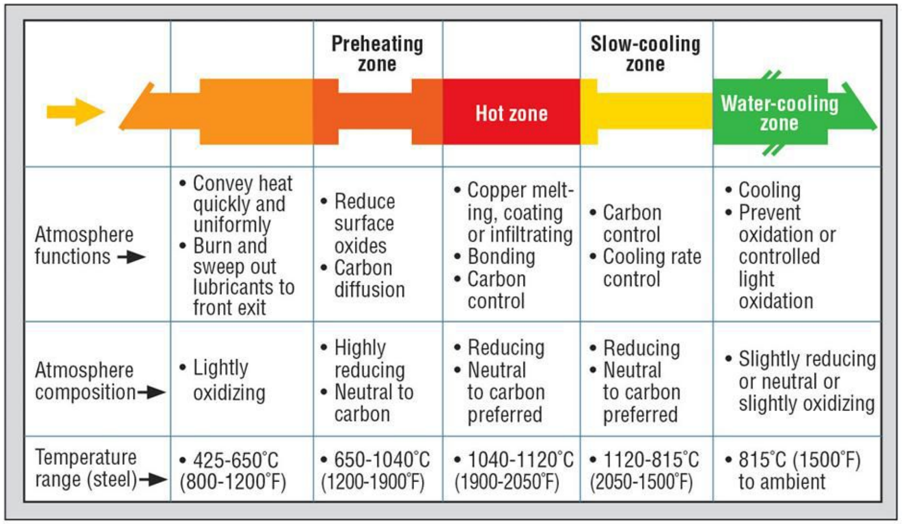


Fig. 1. Role of the furnace atmosphere as a function of position in the sintering furnace[7]

use service application. Other factors – such as furnace design, sintering parameters (e.g., time, temperature, lubricant, loading) and part dimensional tolerances – also play an important role. The actual choice of atmosphere (vacuum, pure gases, mixtures, blended gases, generated gases) further depends on ease of control, cost and possibly

facilities-related issues. The choice of a sintering atmosphere must always be taken into consideration due to its influence on final properties.

The purpose of a sintering atmosphere is multifaceted – to aid in lubricant removal from the green compact, reduce residual surface oxides (in order to promote bonding between adjacent powder particles) and protect the compacts from oxidation during the sintering process. For iron-based alloys, the furnace atmosphere may also be called upon to prevent decarburization through hydrocarbon-gas enrichment.



| | | Preheating zone | Hot zone | Slow-cooling zone | Water-cooling zone |
|---|---|---|---|---|---|
| Atmosphere functions → | • Convey heat quickly and uniformly<br>• Burn and sweep out lubricants to front exit | • Reduce surface oxides<br>• Carbon diffusion | • Copper melting, coating or infiltrating<br>• Bonding<br>• Carbon control | • Carbon control<br>• Cooling rate control | • Cooling<br>• Prevent oxidation or controlled light oxidation |
| Atmosphere composition→ | • Lightly oxidizing | • Highly reducing<br>• Neutral to carbon | • Reducing<br>• Neutral to carbon preferred | • Reducing<br>• Neutral to carbon preferred | • Slightly reducing or neutral or slightly oxidizing |
| Temperature range (steel)→ | • 425-650°C (800-1200°F) | • 650-1040°C (1200-1900°F) | • 1040-1120°C (1900-2050°F) | • 1120-815°C (2050-1500°F) | • 815°C (1500°F) to ambient |

Fig. 2. Role of a sintering furnace atmosphere by location inside the furnace

The delubrication operation (aka delube, burn-off, debinding) required by most PM parts can be handled either as an independent function in a separate furnace or as an integral part of a continuous sintering furnace. There are several factors to consider with each approach.

Many stainless steel and some metal injection molded (MIM) components (depending on the feedstock) are delubed or debound in a standalone batch furnace due to both the nature of the lubricants used, the ability to control the time/temperature/atmosphere profile and to avoid furnace contamination (especially in vacuum and pusher furnaces). Cost of operation of an independent furnace and part handling after delubrication become important issues in many cases.

In a continuous furnace with an integral preheat/delube section, muffle designs predominate in order to contain the effluent and provide precise time temperature/atmosphere control. The atmosphere inside the delubrication chamber must be oxidizing, which is achieved by sending a portion of the gas through water to become highly saturated, or by use of air additions. Meanwhile, the atmosphere in the high-heat section of the furnace must be highly reducing (Fig. 1).

Designing equipment such that gas enters in the proper location and/or is distributed in selected locations within the chamber (via inlet tubes with holes oriented at variable angles opposite the direction of travel) is critical. Exothermic gas and/or nitrogen are typical delube atmospheres. With nitrogen, the use of air additions or the saturation of the atmosphere (by passing it through heated water) aids greatly in lubricant removal.

Atmosphere requirements in a sintering furnace vary considerably depending on the type of furnace (e.g., mesh-belt conveyor, pusher, walking-beam), style of furnace (batch or continuous) and if delubrication (aka delube, dewax) is required to be performed.

The basic atmosphere requirements for a continuous mesh-belt furnace (Fig. 2) are as follows:

- In the delubrication zone, where temperatures are typically in the range of 250-700˚C (480-1300˚F), a high dew-point atmosphere in the range +4.5˚C to +20˚C (+40˚F to +70˚F) is generated, often by mixing dry and wet gas or by air additions both intended to aid in lubricant removal.
- In the sintering zone, where temperatures vary by the material being sintered, a low dew-point atmosphere in the range of -29˚C to -40˚C (-20˚F to -40˚F) aids in oxide reduction to promote bonding of the powder-metal

particles together.

- In the post-cool (aka carbon restoration) zone where temperatures are often in the 800-900˚C (1475-1650˚F) range, or in some instances at the end of the sintering zone, (optional) carbon control of certain materials prevents surface decarburization.
- In the cooling zone, sufficient gas flow is needed to prevent oxidation. O2 levels in commercial practice often run in the 10-50 ppm range (maximum). The goal is to attain the lowest practical level of oxygen.

The most common sintering atmospheres are mixtures of nitrogen/hydrogen or dissociated ammonia diluted by nitrogen additions. Hydrocarbons are used if carbon restoration is required. H2/N2 ratios vary from as low as 5-7% to as high as 20-30% hydrogen. Stainless steels and some tool steels are often processed in 100% hydrogen as are MIM parts. They are commonly run in either pusher furnaces or vacuum furnaces operating with a partial pressure of hydrogen.

It is also important to recognize that changes to the furnace-atmosphere composition occur while reacting to the metal powder (e.g., reduction of oxides can enrich the atmosphere with water vapor); decarburization enriches the atmosphere with carbon monoxide; and certain types of atmospheres (e.g., endothermic gas) can vary from carburizing to decarburizing as a function of temperature and produce unwanted carbon in the form of soot.

## Summary

The choice of furnace atmosphere depends in large part on the metallurgy of the materials being sintered in combination with cost, productivity and the properties produced.

Part 2 will discuss the role of furnace atmospheres in sinter hardening and post-heat-treatment sintering.

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# Steam Treating

Steam has been involved in some way, shape or form with a number of memorable events in the Doctor's life – whether doing calisthenics in a Turkish sauna at 17 or being whipped with wet eucalyptus branches in a Moscow steam bath some 20 years later (stories best left for another time and place). For many of us, however, staring down at a dinner plate of steamed broccoli or cauliflower is as up close and personal with steam as it gets. Yet steam is one of the simplest and most basic of heat-treating atmospheres. Let's learn more.

Steam treating (a.k.a. bluing or blackening) is a time-temperature-atmosphere-dependent process where the performance and quality of the surface and subsurface layers depends to a great extent on surface cleanliness (prior to steam treatment) and the overall integrity (i.e. gas tightness) of the equipment. As an atmosphere, steam can be used for scale-free tempering and stress relief of wrought or powder-metal (P/M) parts of ferrous or nonferrous materials. While not necessary for all components, steam treatment benefits include: reducing the susceptibility to rusting on steel parts – that is, avoidance of the formation of undesirable $Fe_2O_3$ (hematite), sealing porosity, providing a base material for additional (powder or paint) coatings, extending shelf life, improving mechanical properties (e.g., apparent hardness, compressive strength, wear characteristics), and as a decorative coating producing a blue-gray to blue-black surface appearance.

## How it Works

Steam treating is performed on ferrous parts through a deliberate addition of steam ($H_2O$) into a tightly sealed heat-treating furnace in the temperature range of 315-540˚C (600-1000˚F) so as to ensure that only $Fe_3O_4$ (magnetite) is formed on the surface (Eq. 1).
The following oxidizing reaction takes place at the surface of the parts:

$$3Fe + 4H_2O\ (g) \leftrightarrow Fe_3O_4 + 4H_2\ (g) \qquad (1)$$

A typical cycle begins by thoroughly cleaning the individual parts of oils and other contaminants. Heating then usually takes place in air (or another oxidizing but non-carburizing furnace atmosphere). Ferrous parts are typically heated in the 315-375˚C (600-700˚F) range before steam introduction, while for nonferrous parts this value is around 150˚C (300˚F). Purging of

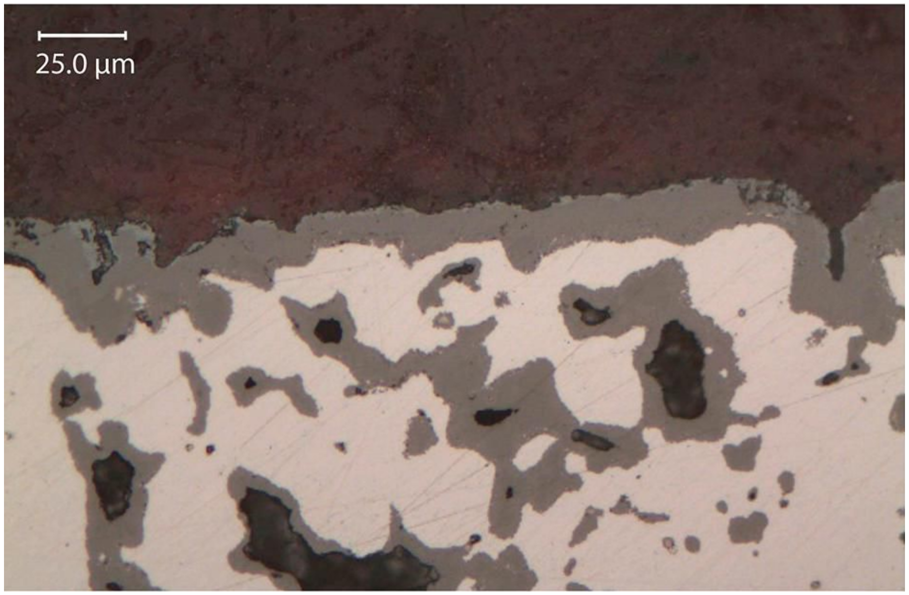

Fig. 1. 4200 steel (0.60%Mo, 0.45%Ni, 0.28%Mn) with 2% Cu and 1% graphite admixed; steam treated for one hour at 575°C (1067°F)

the furnace with steam then takes place to an oxygen level less than 1% (10,000 ppm), typically around 0.1% (1,000 ppm). This step must be complete before the temperature exceeds 425˚C (800˚F) for ferrous parts. A dew point in the furnace of +15˚C (+60˚F) or higher is typical. After soaking, parts are cooled in steam to an intermediate temperature before removal or rapid quenching finishes the cycle. Steam treatment is reportedly most effective on parts with a maximum carbon content of 0.5-0.8%.

## Steam Treatment of P/M Parts

Many P/M parts are processed in furnaces exposed to superheated (e.g., 160˚C/320˚F steam) at a temperature around 550˚C (1020˚F). The oxide layer formed is typically 5-7µm (0.00020-0.00028 inch) thick on the surface with interconnected subsurface porosity (Fig. 1). $Fe_3O_4$ is stable and tenacious, forming a bluish or bluish-black surface layer that does not easily break down.

The $Fe_3O_4$ oxide has a hardness of approximately 50 HRC and is highly corrosion-resistant. The thickness of the coating grows with the square root of treating time and can vary from just over 1 µm (0.00005 inch) to just over 7 µm (0.00030 inch). Applications such as sealing require that the part be in the dry steam at 540˚C (1000˚F) for about 60 minutes. For applications where corrosion resistance or hardness is important, a retention time of only about 30 minutes may be required to achieve the desired results.

## Steam Treatment of Motor Laminations

The performance of steel in an electro-magnetic circuit is measured by variations in eddy current, hysteresis losses and changes in (magnetic) permeability. The presence of carbon and to a lesser extent sulfur, oxygen and nitrogen in steel increases eddy current and hysteresis losses while lowering permeability. In-process

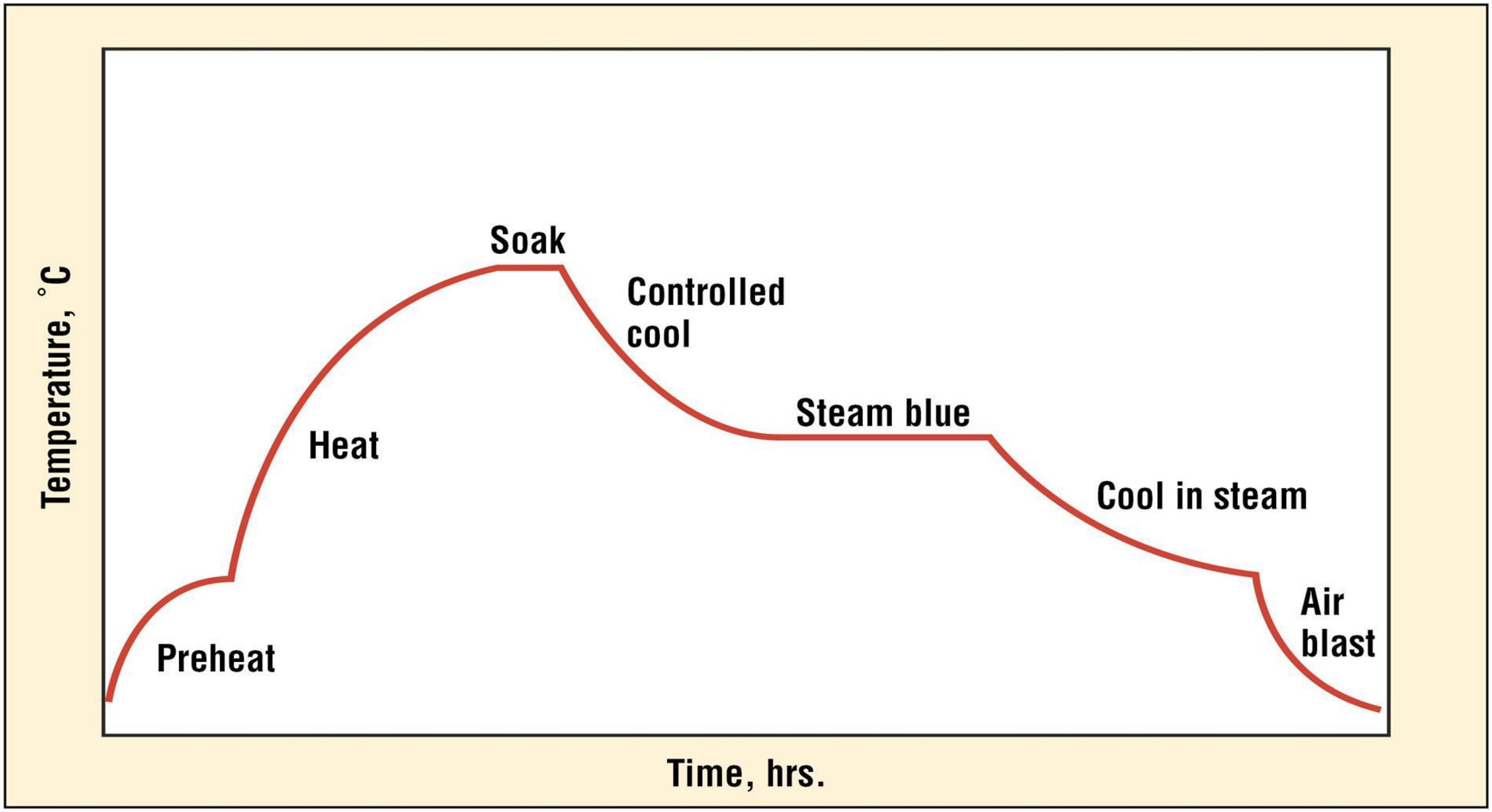

Fig. 2. Combination annealing and bluing cycle for motor laminations[1]

annealing, in addition to removing strains induced from cold working, reduces carbon levels (from steels with up to 0.08%) to typically less than 0.01%C. Eddy current losses vary with lamination thickness and lamination coating. For most 60 Hz applications, 0.60 mm (0.024 inch) is reported to balance optimum stamping qualities

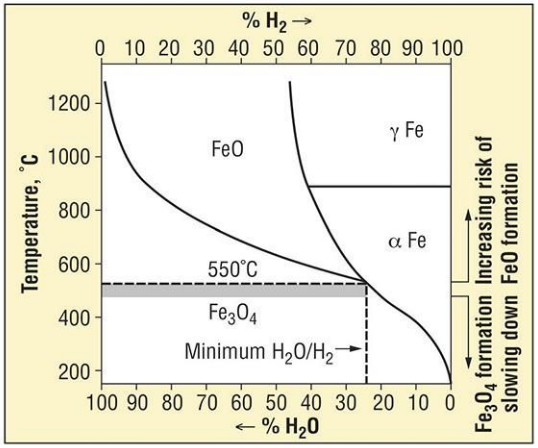

Fig. 3. Acceptable and unacceptable steam-treatment conditions for ferrous materials[3]

with acceptable eddy current losses.

To reduce eddy current losses between laminations, electrical steels are normally coated to increase inter-laminar resistance. These coatings may be organic, inorganic or an oxide ($Fe_3O_4$) applied by exposing the lamination to either a high dew-point exothermic atmosphere or a super-heated steam atmosphere (Fig. 2) for a period of up to one hour at a temperature of around 510˚C (950˚F). This coating generally increases resistance between laminations and provides rust and corrosion protection.

## Steam Treatment of High-Speed Steels

The presence of a tenacious oxide coating on some high-speed steel cutting tools is reported to help prevent chip buildup on cutting edges while enhancing grinding, drilling, cutting and endurance (feed rates). However, steam treating is not recommended in cutting applications for very soft or nonferrous materials.

## Steam Treatment of Nonferrous Materials

For nonferrous materials, loads are purged at 150˚C (300˚F), heated to the required soak temperature, and cooled under a steam atmosphere back down to 150˚C (300˚F) after the final soak and before air or water quenching. The treatment performs both a stress relief and anneal for bronze, brass, copper and silver alloys. Post treatments such as bright dip, buffing or pickling are reportedly reduced.

## Potential Problems

The most commonly reported problems with steam treating could be categorized as follows:

### Layer Reversal

The hydrogen ($H_2$) gas created by the steam-treating process is constantly diluting the steam ($H_2O$). If the hydrogen concentration rises too high, the reaction is reversed and the oxide layer reduces. In order to prevent this, three measures are important: (1) maintain a sufficiently high turbulence in the steam; (2) create good circulation paths throughout the load; and (3) bleed controlled amounts of air (or oxygen) into the furnace chamber to keep the hydrogen concentration down to an acceptable level.

### Flaky Surfaces

In most instances, neither the steam temperature nor the part temperature should exceed 550˚C (1020˚F) because, above this temperature, the reaction (Eq. 1) is more and more superseded by the reaction:

$$Fe + H_2O\ (g) \rightarrow FeO + H_2 \qquad (2)$$

which forms a gray, flaky and loosely adhering layer of FeO (wüstite) on the surface of the parts and provides no corrosion protection at all.

### Discolored Surfaces

Pinkish or inconsistent discoloration of the part surface may indicate the presence of undesirable $Fe_2O_3$, suggesting that the entire load was not above 315˚C (600˚F) prior to the introduction of the steam. Black spots on parts suggest improper cleaning and residues left on parts prior to processing, while white spots often indicate water contaminants or chemicals.

Reddish discoloration suggests that the steam was not dry – that is, liquid water was present and reacted with the iron on the surface of the iron part to form $Fe_2O_3$. A brown or brownish-black discoloration suggests air was in contact with the ferrous component while in the presence of the steam.

## In Conclusion

Steam treating is a versatile tool in the heat-treater's arsenal and one that should not be forgotten when its benefits are needed. Typical steam-treating applications include automotive, hydraulics, agriculture, marine, home appliances, lawn and garden, and off-road construction components of wrought and powder metal.

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# Types of Burners and Combustion Systems

The heat treater often has choices for burners and combustion systems when purchasing new equipment or rebuilding older furnaces and ovens. While natural gas prices are highly competitive today, a key consideration is to match the heating and temperature uniformity needs of the applications with the best and most cost-effective systems. Let's learn more.

## Introduction

Air for combustion can be supplied to the burner in several ways. Primary air is a term used to describe air supplied and mixed with fuel prior to ignition. This is usually controlled through orifices and valves where all combustion air is mixed with the fuel and is ready to ignite as soon as it reaches the burner nozzle. The term "pre-mix" is used for burners and combustion systems where the air and gas are mixed prior to the burner nozzle. When they are mixed at or within the burner nozzle, it is called a "nozzle-mix" system.

### Pre-Mix Burners

There are various pre-mix systems, but all must proportion the fuel and air separately to create an air/gas mixture that will effectively burn at the burner nozzle. The air and gas volume flow must be controlled and mixed prior to being piped to the burner nozzle. A mixing device blends them to a thoroughly combustible mixture.

### Nozzle-Mix Burners

Air and gas are mixed at the burner nozzle in nozzle-mix burners. Some nozzle-mix burners keep the air and gas separated until the point of ignition, whereas others mix some of the primary air prior to the point of ignition. Sealed-nozzle mixed burner systems depend entirely on primary air. By contrast, secondary air is supplied to the flame after it is ignited and is brought in at the burner. An example of both types is in atmospheric burners, which use about 70% primary air and 30% secondary air.

### Direct-Fired, Indirect-Fired Combustion Systems

Combustion systems can be divided into two general categories: direct-fired and indirect-fired systems. In direct-fired applications, the products of combustion are exposed to the work, whereas indirect systems fire into radiant tubes or find the work protected from the

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flame by such items as retorts and muffles. In most direct-fired combustion systems (e.g., box furnaces), secondary air is pulled into the furnace through leaky doors, other openings and negative furnace pressure.

Recuperative systems also provide many benefits. There are two types of air preheaters: recuperators (Fig. 1) and regenerators. Recuperators are gas-to-gas heat exchangers placed on the furnace stack. Internal tubes or plates transfer heat from the outgoing exhaust gas to the incoming combustion air while keeping the two streams from mixing. Recuperators are available in a wide variety of styles, flow capacities and temperature ranges. Regenerators include two or more separate heat-storage sections. Flue gases and combustion air take turns flowing through each regenerator, alternately heating the storage medium and then withdrawing heat from it. Cost justification is based on a payback analysis (Equation 1).

(1) Typical payback period = (Cost of combustion air preheating system obtained from the supplier or contractor) ÷ (Reduction in fuel usage, Million BTU/hour × number of operating hours per year × cost of fuel per Million BTU)

## Sizing a Combustion System

Heat input must balance with the heat demand to avoid over-temperature or under-temperature conditions, which could negatively affect the heat-treatment process. Using burners with heat output too great will cause them to cycle more. It is much preferred to have burner output more closely match the heat required in order to reduce cycling and smooth out temperature swings in the furnace, which are created during cycling. Temperature control within a cycle is a very important process-control parameter.

## Plunge Cooling

One of the benefits of utilizing indirect heating via radiant tubes in furnaces is the possibility of rapid temperature changes during any portion of the cycle (e.g., dropping from carburizing to hardening temperature prior to quenching). This feature can be accomplished by turning off the fuel to the combustion system and allowing cool combustion air to flow through the tubes. The furnace must be equipped with this feature to ensure burners properly relight after the cooling segment.

## Controls and Safety

Anyone who operates or maintains burners or combustion systems must understand the basic function of the various components that work together to make their system work effectively and safely. Each

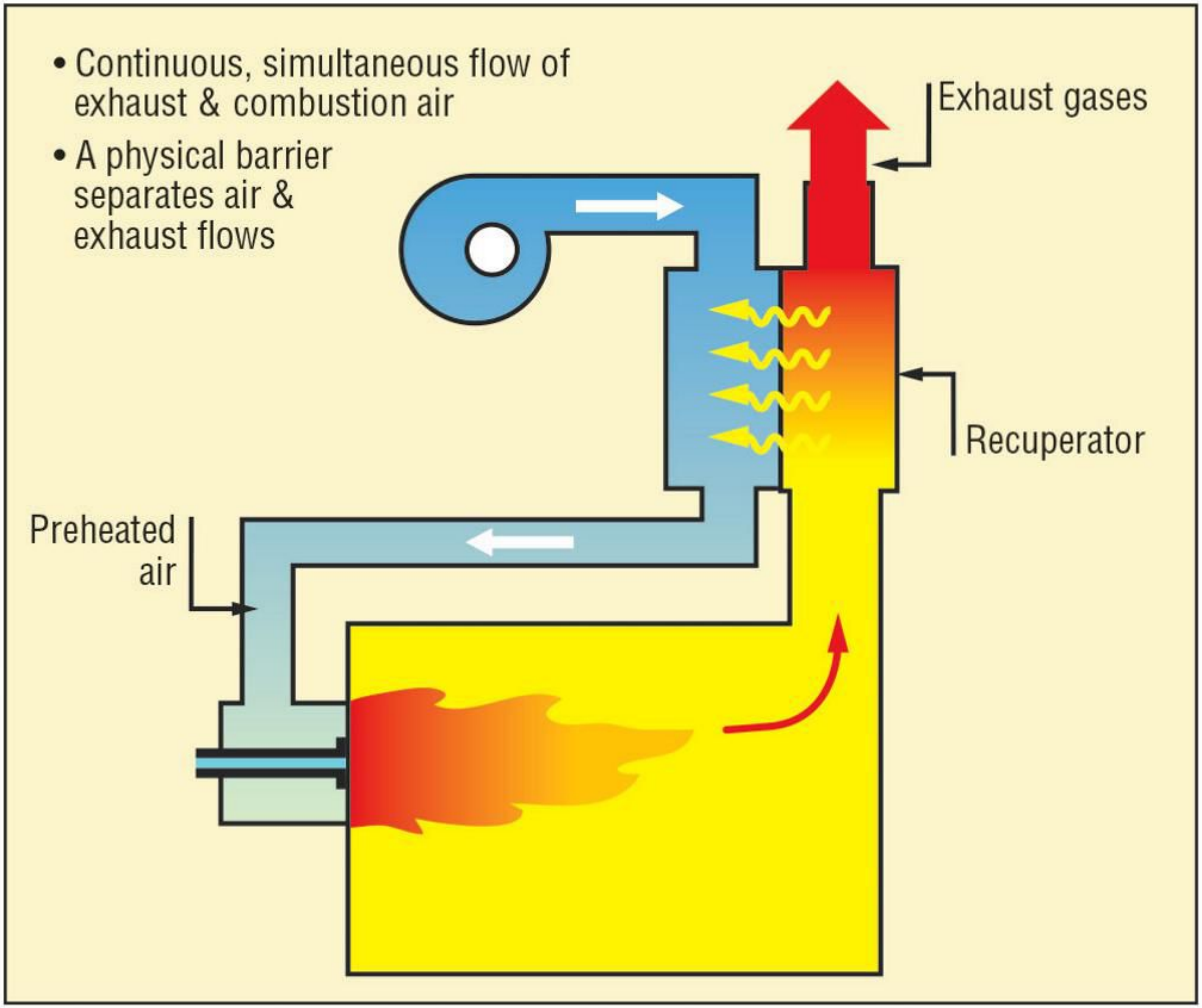

Fig. 1. Recuperative burner systems

component has an important function. Some are for operation and control, while others are strictly safety devices that automatically shut the system down to prevent damage and personal injury.

When looking at a combustion system for the first time, people are often a bit overwhelmed with the amount of valves, controls and other components. This initial fear is normal because no one should operate a combustion system without first understanding all of the components involved, their function and their safe operation. Having a healthy respect also provides good motivation to learn what each device is doing and how they are controlled for safe start-up, operation and shutdown.

## System Monitoring for Safe Operation

Combustion systems have layers of safety checks and devices designed to protect operators and equipment from harm. In the event that a safety device is not working properly, other devices in the system are designed to back them up. An example of this is the fact that two main gas shutoff valves are used in series. If there is a leak or failure of one, the other is there to shut off the gas. Another example involves normally closed valves (i.e. valves held open when power is applied). These valves will automatically shut in the event of an electric power outage.

## Safety Devices

The first line of defense is the flame monitor, which must be used in all combustion systems. The absence of flame at the burner will trigger the system to shut off the main gas valves.

In pre-mix combustion systems, a second line of defense is a device called a flame arrester. If the combustion were to burn back into the pre-mix supply line, the flame will stop at the flame arrester. A flame arrester is defined in NFPA 86 as “a device that prevents the transmission of a flame through a flammable gas/air mixture by quenching the flame on the surface of an array of small passages through which the flame must pass.”

A flame arrester installed in a pre-mixed system that includes a way to both extinguish the flame and shut off the fuel supply is called an automatic fire check. An automatic fire check utilizes a spring-loaded shutoff valve that is held in place by two bimetallic rods during normal operation. If a flashback occurs, the heat from the flame will cause the bimetallic rods to bend and release the spring-loaded shutoff valve. Fire checks should be inspected a minimum of twice a year to ensure they are functioning properly.

The combustion safety system continually monitors and controls the system. There are different requirements depending on the stage of operation. The different stages are categorized as follows:

- Pre-ignition – A timed pre-ignition purge cycle
- Trial-for-ignition period – A fixed period of time (15 second or less) for pilot and main burners to ignite
- Operation – Safety sequencing has logic systems. Activation of any safety interlock will result in a safety shutdown. Safety interlock devices can be hardwired in series, use relays or be connected to a PLC system. PLC system requirements are spelled out in detail for safety standards and must include at least one manual emergency switch.
- Restart – Occurs when flame is not detected. In some instances, the standard allows for an automatic trial to re-ignite the burners.
- Failure – Occurs when one of the interlocks fails to operate within its design parameters
- Shutdown – Occurs when the system ceases to function either due to failure or by an action of the operator. There is often a post-purge cycle initiated at this time.

A number of safety checks must be made prior to ignition. Monitoring devices send electronic signals back to the combustion control system to ensure that certain conditions are all met prior to ignition. The monitors provide continual feedback signals during combustion to ensure that all of the conditions are met to allow the system to continue operation. The control system will use this information to determine whether to allow the process to continue, try to relight the system or shut it down. Shutdown begins with the closing of the gas valves, followed by a purge time in which the air must continue to run in order to ensure that any uncombusted fuel has been evacuated or vented.

## Summary

Understanding the basics of combustion will provide tangible benefits to the heat treater, including faster heat-up times and load recovery (due to higher flame temperatures and greater heat transfer); greater efficiency (more available heat); reduced pollution (minimum exhaust volumes, reduced fuel use); and cost savings (more cost competitive vs. alternative energy sources).

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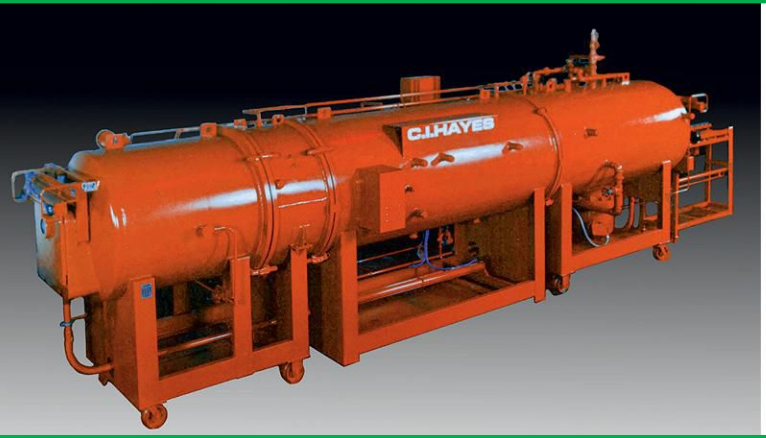

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# Heat-Resistant Materials: Alloy Systems

Adapting to change is a basic human trait, but in the world of heat treating, we often need a gentle push to help move us forward. In the immortal words of Bob Dylan, "The Times They Are a Changin'," and when it comes to heat-resistant alloys, we must adapt to compete. Let's learn more.

The family of heat-resistant alloys represented by cast HT and its close cousins (e.g., HK, HU) as well as their wrought equivalents (e.g., 330) are to the heat treater what motherhood and apple pie is to our society as a whole – sacred cows! We've used these alloys for so long that we often don't look to see if something better has arrived.

Yet newer alloys and alloy systems are available that offer unique advantages with surprisingly competitive paybacks. While time (and space) prohibits us from addressing this subject in great depth, one example of a more advanced alloy system will suffice to make our point.

**Table 1.** Total creep strength for indicated strain rate

| Temperature, °C (°F) | Creep strength, MPa (psi) | Creep strength, MPa (psi) |
|---|---|---|
| | **0.0001%** | **0.00001%** |
| 650 (1200) | 184.8 (26,800) | 120.0 (17,405) |
| 705 (1300) | 127.5 (18,500) | 80.0 (11,550) |
| 760 (1400) | 65.0 (9,427) | 37.0 (5,366) |
| 815 (1500) | 29.0 (4,210) | 13.3 (1,925) |
| 870 (1600) | 16.5 (2,390) | 8.9 (1,291) |
| 930 (1700) | 10.3 (1,490) | 6.3 (915) |
| 980 (1800) | 6.8 (980) | 3.9 (555) |
| 1040 (1900) | 4.0 (585) | 2.2 (312) |
| 1095 (2000) | 2.3 (330) | 1.03 (152) |
| 1150 (2100) | 1.0 (145) | 0.40 (58) |

## 602 CA® Alloy[1]

602 CA® is a wrought material developed by VDM and distributed in the U.S. under the designation RA 602 CA®. Inspired by aerospace coatings applied to turbine blades, this product is a nickel-based alloy that employs a high chromium content along with aluminum and yttrium additions that produce a tightly adherent oxide, allowing the alloy to operate at temperatures in excess of 1230°C (2250°F). The alloy is extremely resistant to grain growth at high temperatures and is resistant to carburization. The

**Table 2.** Rupture strength to break at indicated time[4]

| Temperature, °C (°F) | Rupture strength, MPa (psi) | Rupture strength, MPa (psi) |
|---|---|---|
| | 1% in 10,000 hours | 1% in 100,000 hours |
| 650 (1200) | 215.0 (31,180) | 165.0 (23,931) |
| 705 (1300) | 149.6 (21,700) | 94.0 (13,750) |
| 760 (1400) | 78.0 (11,310) | 40.0 (5,802) |
| 815 (1500) | 33.8 (4,900) | 17.1 (2,450) |
| 870 (1600) | 22.5 (3,260) | 12.2 (1,769) |
| 930 (1700) | 15.4 (2,230) | 8.0 (1,160) |
| 980 (1800) | 10.1 (1,460) | 5.1 (735) |
| 1040 (1900) | 6.8 (980) | 3.3 (479) |
| 1095 (2000) | 4.5 (655) | 2.1 (311) |
| 1150 (2100) | 3.0 (435) | 1.4 (203) |

aluminum in the alloy allows for the formation of a continuous, homogenous and self-repairing alumina subscale, while the yttrium improves the adhesion and spalling resistance of the chromium- and aluminum-oxide scales.[2]

## Creep and Rupture Properties

This alloy also has excellent creep-rupture properties. Creep and rupture strength (Tables 1-2) are important benchmarks in determining the life expectancy of high-temperature alloys.[3]

**Table 3.** Weight change (mg/m$^2$h) for cyclic carburization data in $CH_4/H_2$ atmosphere, Ac = 0.80%.[4]

| Temperature, °C (°F) | 850 (1562) | 1000 (1832) | 1150 (2102) |
|---|---|---|---|
| 310 | 130 | 305 | ---- |
| 800AT | 143 | 339 | 813 |
| 600 | 50 | 190 | 626 |
| 601 | 64 | 170 | 508 |
| RA 602 CA | 13 | 70 | 175 |

## Carburization Resistance

The tenacious oxide layer present on this alloy is also responsible for its excellent carburization resistance (Table 3) in a heat-treat atmosphere at a 0.80% carbon potential. The data shows that this alloy is significantly more resistant to carburization than typical austenitic alloys and Inconel. Carburization leads to embrittlement/cracking, and alloys that are more resistant will retain their ductility longer.

## Grain Growth

A common concern involving components exposed to extremely high temperatures for long periods of time is (brittle) fracture. At temperatures exceeding the annealing temperature of a heat-resistant alloy, grain

**Table 4.** Effect of time on ASTM grain size for various alloys at 1120°C (2050°F) for 990 hrs.[2]

| Time (hour) | 0 | 2 | 24 | 72 | 184 | 344 | 510 | 670 | 830 | 990 |
|---|---|---|---|---|---|---|---|---|---|---|
| | ASTM Grain Size | | | | | | | | | |
| RA 602 CA | 7 | 7 | 7 | 7 | 6.5 | 6.5 | 6.5 | 6.5 | 6.5 | 6.5 |
| 601 | 5 | 5 | 1.5 | 1 | 1 | 0 | 0 | 00 | 00 | 00 |
| 601 GC | 5.5 | 5.5 | 5 | 5 | 3.5 | 3.5 | 3 | 3 | 3 | 2.5 |
| RA330 | 7 | 3.5 | 3.5 | 3 | 3 | 2.5 | 2 | 2 | 2 | 1.5 |
| RA333 | 4 | 4 | 4 | 3 | 2.5 | 2 | 2 | 2 | 2 | 1 |
| 600 | 8 | 4 | 4 | 0 | 0 | 0 | 00 | 00 | 00 | 00 |

growth can be expected (Table 4) and leads to loss of ductility. RA 602 CA shows no appreciable grain growth.

## Metal Dusting (aka Catastrophic Carburization)

Metal dusting is a form of carburization at relatively low temperatures that leads to rapid, catastrophic corrosion of heat-resistant alloys. For years, RA333® has been an excellent choice to negate these effects, but availability in some forms in recent years has limited its use. RA 602 CA has also proven superior in resisting the effects of metal dusting due to its chemistry additions.

## Weldability/Formability

RA 602 CA is weldable by GTAW, GMAW, SMAW and PAW. Proper selection of shielding gases is critical. Shielding gases (or electrode coatings in the case of SMAW) are dependent on the welding process. Welding guidelines are available from the supplier, including welding instructions for dissimilar metals.

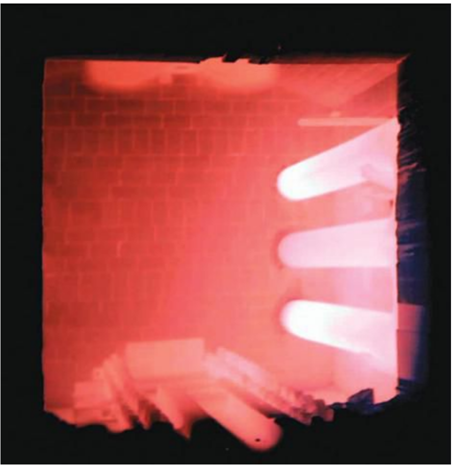

Fig. 1. RA 602 CA® radiant tubes after one year of service

This alloy may be hot worked in the 900-1200°C (1650-2190°F) range and immediately quenched in water, but it should not be formed between 595-815°C (1100-1500°F). Heating must be done in a tightly temperature-controlled furnace and a neutral to slightly oxidizing atmosphere. Fluctuating between an oxidizing and reducing atmosphere must be avoided. Natural gas should not contain more than 0.5% sulfur or 0.1% by weight of fuel oil. Never use a torch to heat the material because this will often lead to cracking.

The high carbon content (0.15-0.25%) in the material causes rapid work hardening. Components made from this alloy may be bent 120 degrees around a radius equal to three times the material thickness (3T) for material up to 0.4-inch thick. As with all nickel alloys, the shear drag (burr) must be removed to prevent crack initiation.

### Typical Heat-Treat Applications

RA 602 CA is used for many applications, including radiant tubes, furnace rolls, muffles, retorts, atmosphere and vacuum furnace fixtures, and grids to name a few.

One steel mill has reported using this alloy for slab reheat furnace rolls at temperatures of 1250°C (2280°F) for over two years. The key to success is the high creep strength imparted by the carbon content and the constant rolling motion that prevents stresses from the slab weight from being concentrated on any particular point.

Radiant tubes (Fig. 1) fabricated from this alloy are an alternative to cast tubes with, in general, a significant weight reduction. For example, an 8 mm (5/16 inches) thick cast tube can be made from 3-mm (11-gauge) sheet. When firing continuously in the working zones of continuous or high-production batch furnaces, significant energy savings can be achieved from the lower mass, which in turn allows more of the heat generated to be used for heating material instead of keeping equilibrium in the tube. It also allows for lower firing temperatures because there is a smaller gradient across the tube due to the section thickness. Exact savings will be reliant on furnace condition, insulation integrity and operating conditions but should be 10% or more depending on the thickness reduction.

Lightweight rod-mesh baskets and liners are being fabricated from this alloy to take advantage of its higher creep strength, resistance to grain growth and retention of ductility. The result is improved basket life and fewer issues when straightening of baskets is required. It has been reported that high-pressure gas quenching of high-speed tool steels hardened at temperatures in excess of 1065°C (1950°F) allows for a weight reduction of up to 10%.

Low-pressure vacuum carburizing is another example of where alloy 602 CA is making inroads. The aluminum content allows for retention of the alloy's oxide layer, while more traditional alloys (such as Inconel 600, 601 and RA330) lose their protective chromium and/or silicon oxides.[5]

## Summing Up

Alloy 602 CA is one example of a family of new high-temperature alloys available from a multitude of suppliers that are increasingly being used in the heat-treat industry. There are many other applications for these alloys throughout the thermal-processing industry as both cost-effective alternatives to more traditional wrought alloys or as substitutes for cast alloys in high-temperature applications. As a heat treater, it is worth the time and effort to investigate and use this new generation of materials.

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# The Role of Furnace Atmosphere in Sintering (Part 2)

The heat treatment of powder-metal (PM) parts presents a number of unique challenges, influenced primarily by material and density considerations. Common post-sintering processes include sinter hardening and conventional post-sintering heat-treatment operations. Let's learn more.

## Sinter Hardening

The process of sinter hardening is an alternative to conventional post-sintering hardening or case hardening and quenching, which takes place in a separate heat-treatment furnace. Sinter hardening is conducted inside the sintering furnace itself and involves rapid cooling after sintering to transform the microstructure to martensite. Cooling speeds vary by alloy, but rates in the range of 1-2°C/sec. (2-4°F/sec.) from 760-205°C (1400-400°F) are typical.[1] Carbon control of the furnace atmosphere is particularly important to achieving desired mechanical properties.

The chemistry and alloying method must be such that sufficient hardenability exists in the material to allow proper transformation of the microstructure for a given cooling rate. Typical sintering furnaces are equipped with water-jacketed cooling sections to cool parts using free convection. For some small parts, this cooling rate is sometimes (but not often) sufficient to achieve sinter hardening. For more massive parts, increased velocity and control of the rate of temperature loss are necessary to achieve the desired cooling rate (Fig. 1).


Fig. 1. Cooling-chamber detail on a sinter-hardening furnace (courtesy of Abbott Furnace Company)

There continues to be considerable discussion among experts as to the importance of the flow direction of the furnace atmosphere. In many designs, atmosphere flows in a top-down manner, and the potential exists to develop a hardness gradient in the part, especially if it has significant mass. For this reason, all equipment may not perform the same, and some parts might be better suited for conventional heat treating.

Sinter hardening ferrous alloys has been widely used for a number of years for the production of press and sintered powder-metal parts with ultimate tensile strength (UTS) around 1,200 MPa and hardness values of 40 HRC at a sintered density of 7.2 g/cm$^3$. The new generation of sinter-hardened materials raises these limits even further.

Advantages of sinter hardening include:

- The need for a secondary quench-hardening treatment is eliminated.
- The reduced distortion of parts due to the less-severe quench leads to better dimensional control.
- Sinter-hardened parts do not need an oil-removal step prior to finishing operations such as plating.
- The tempering of sinter-hardened parts in air is more straightforward than for quench-hardened parts.
- Parts that have been quenched in an oil bath retain a considerable amount of oil in their pores. If tempering at a temperature above 200°C (400°F) is required, the oil-quenched parts must first be tempered below 200°C (400°F) to burn off the entrapped oil prior to tempering at the higher temperature.

## Conventional Heat Treatment

As stated earlier, the heat treatment of PM parts involves taking into account the density of the part being processed. Porosity (i.e., void space) means that PM parts are difficult to heat and cool.

Various consolidation and/or sintering techniques can be performed on a “green” part to achieve high density, including metal injection molding, hot isostatic pressing, P/M forging and liquid-phase sintering. However, conventional “press and sinter” is the most widely used method and results in a typical density range of 6.8-7.2 g/cm$^3$. By comparison, wrought carbon steel (0.40%) has a density of 7.84 g/cm$^3$.

In many cases, hardening and case hardening followed by quenching allow us to achieve the desired final part strength and hardness. Other popular processes include annealing, stress relief, tempering and steam treating.[1] Approximately 60% of current PM steels have their physical and mechanical

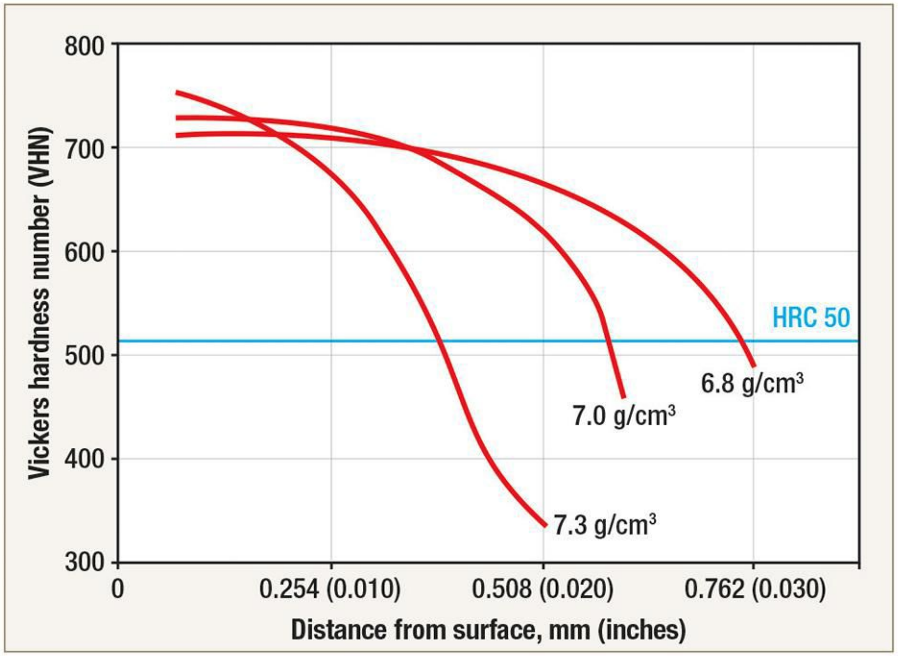

Fig. 2. Effect of density on vacuum carburized case depth at 925˚C (1700˚F)

properties enhanced after sintering by some form of secondary heat-treatment operations.

Ultimate tensile strength, hardness, wear, corrosion resistance and compressive strength can, in general, be improved by heat treating while properties such as impact resistance and ductility may be adversely affected. With this in mind, the selection of material chemistry and part process parameters are critical considerations for the successful application of heat-treatment techniques to PM parts.

The PM factor is a term used to describe a multitude of variables that influence the heat treatment of ferrous powder-metal parts. The most critical of these parameters include:

- Part density
- Material composition
- Quenching or cooling method
- Process factors
- Equipment-induced variables

Factors such as the type of base iron or steel powder, as well as the amount and type of alloy additions and sintering parameters, are unique to the PM industry. When planning or executing a secondary heat-treatment operation, the most important variables to consider are density, microstructure, carbon and alloy content, process cycle and furnace atmosphere. In carburizing, for example, carbon is absorbed more rapidly than in conventional wrought materials (due to part density), and case depths develop much more rapidly (Fig. 2).

The quench media and the hardenability of the material have significant influence on as-quenched properties. Oil quenching, though less severe than water or brine, is preferred due to improved distortion control and minimized cracking. Control of oil temperature ensures load-to-load consistency, and the use of a "fast" (9-11 second) oil is preferred because of improved heat-transfer characteristics.

Since PM parts can absorb up to 3% oil (by weight), subsequent cleaning operations can be difficult. Incomplete cleaning leads to the creation of a great deal of "smoke" during tempering and potential safety concerns with respect to breathing these fumes. There is also a concern about fire due to the presence of large amounts of oil in the tempering furnace and/or ventilation ducts.

Quenching in water, brine or polymer as an alternative to oil can improve the rate of heat transfer. In many cases, however, this accelerates part corrosion due to residual fluid trapped near the surface. For this same reason, salt quenching can also create problems.

Temperature is one of the process variables that must be taken into consideration in secondary heat-treatment operations. In applications such as hardening and case hardening, temperature must be high enough to fully austenitize the material so that it will quench to a martensitic structure. Oil quenching, for example, may require a higher austenitizing temperature to achieve a structure similar to water or brine. It is also important to note that some secondary operations, such as tempering or steam treating, do not raise the parts to austenitizing temperature. However, the uniform distribution and dissipation of heat is a major factor in the consistency of the end product.

Time is another process variable that influences secondary heat treatment. Soak times up to 50% longer than wrought materials are typical. This is due to lower thermal conductivity of the porous P/M material.

Finally, the choice of atmosphere for hardening and sinter hardening of PM components is necessitated by the resultant metallurgy of the sintered material in combination with cost, productivity and the properties produced. Choices such as endothermic gas or nitrogen/methanol are popular for hardening and quenching, while hydrogen/nitrogen or dissociated ammonia/nitrogen atmospheres enriched with hydrocarbon gases are often used in sinter hardening.

## Summary

Ultimately, the desired mechanical properties dictate the type of post-sintering heat treatment and atmosphere selection. With powder metallurgy increasing in popularity, heat treaters must be able to adapt their equipment to the needs of the PM industry.

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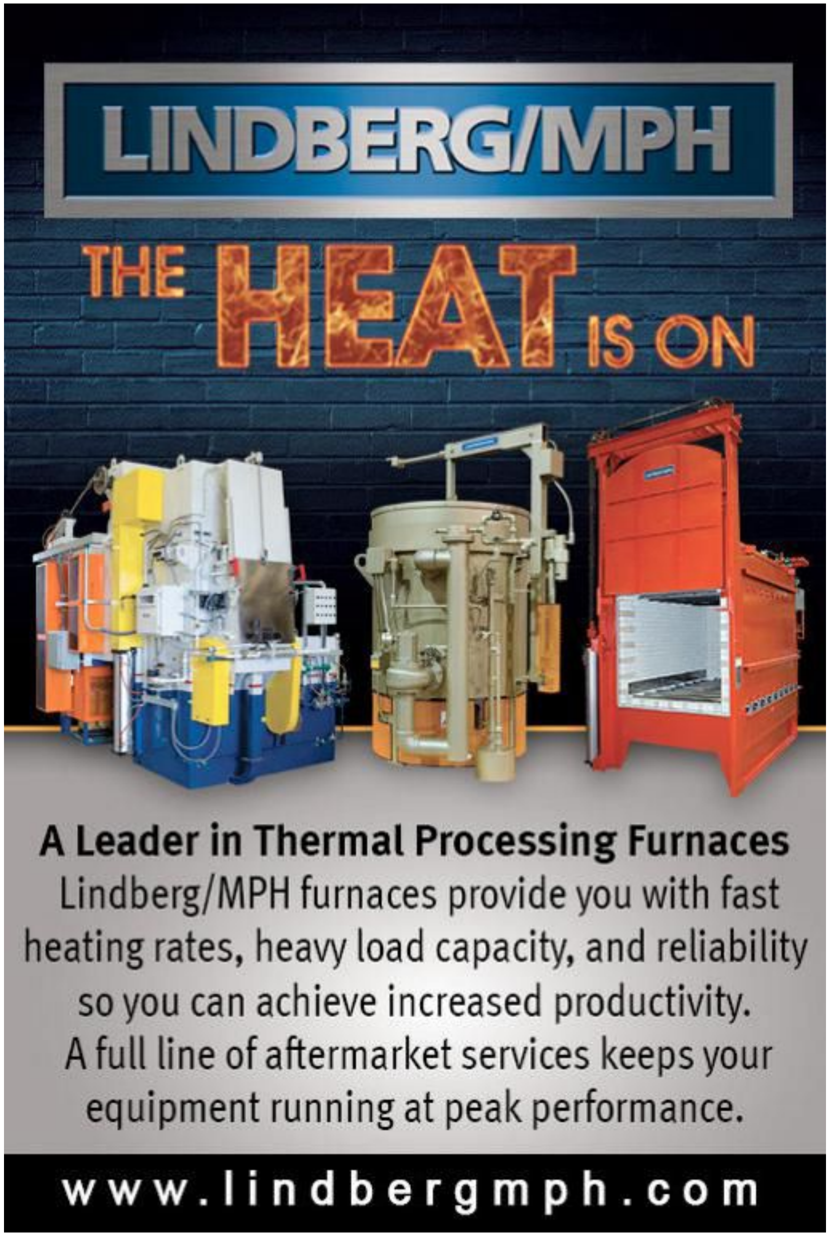

# Cold Traps

In vacuum applications, cold traps are added to vacuum pumping systems either to remove unwanted contaminants (e.g., water, solvents, acidic or alkaline compounds) from the gas stream or to prevent pump backstreaming. These conditions can cause loss of efficiency, poor product quality or damage to the vacuum pumping system. Let's learn more.

In simplest terms, cold traps work by sublimating a gas molecule, which means that the molecule is transformed directly from the gas phase to the solid (crystalline) phase, bypassing the liquid phase. The gas crystallizes out on the cold metal surface often appearing as "frost" in the trap.

Cold traps that are large enough and cold enough to collect the condensable vapors emanating from the chamber should be chosen. Cold traps and cold caps are also used to prevent oil vapors from backstreaming (i.e. oil migration from the pumps into the chamber). In such cases, a baffle or a section of pipe containing a number of cooled vanes will be attached to the inlet of an existing pumping system. By cooling the baffle, either with a cryogenic liquid such as nitrogen or by use of an electrically driven Peltier element (a thermoelectric heat-pump device in which one side is cooled while the opposite side heats up when a voltage is placed across the device, transferring heat from one side of the device to the other), oil vapor molecules that strike the baffle vanes will condense and be removed from the pumped cavity.

Cold traps are also recommended in systems where a large amount of outgassing or contaminants may be present. Examples include systems where "dirty" parts are run, brazing applications where filler-metal vaporization is present and freeze drying where a large amount of liquid must be removed from the vacuum environment.

## Types

Vacuum pumps will often perform better and last longer when used in conjunction with an appropriate inlet trap or filter. Cold traps work well for condensable gases such as water, solvents or oils but are often called upon to handle other forms of contamination such as solids (e.g., carbon in the form of soot).

Several types are in common use.

- Mechanical refrigeration traps: These traps are basically small refrigerators and vary in size depending on the amount of gas to be processed.

Fig. 1. Contaminated diffusion pump

They can achieve trap surface temperatures between -40 to -70˚C (-40 to -94˚F). Automatic defrost cycles and single- and two-stage cascade styles are available. Mechanical refrigeration is considered the most expensive type of cold trap but also the one needing the least attention. They are limited in size, generally to about 0.20 $m^3$/min (7 ft3/min), due to cost considerations.

- Dry-ice (foreline) traps: Dry ice and alcohol are used to produce a slurry placed in a trap well, which allows the surface of the vessel to reach -75˚C (-103˚F). This is low enough to condense most volatile materials. The trapping surface of the center well is visible during operation through the top view ring. Defrost and cleanup is made easy by lifting out the trapping well after venting.

  A typical vessel consists of an electropolished type-304 stainless steel with an outer wall in the vicinity of 1.65 mm (0.065 inch) thick with welded-in ports. Some designs use an acrylic plastic cover over the cold wall. These systems are typically lower in cost than many other cold-trap types.
- Liquid-nitrogen traps: Liquid-nitrogen cold traps prevent contaminants from the chamber to make their way into the pumps, where they will either contaminate the pump (Fig. 1) or cause breakdown of the pump fluid. In either case, severe loss of efficiency and poor vacuum levels result. In the example shown, the diffusion pump was only capable of reaching 4 x $10^{-3}$ Torr rather than the typical 1 x $10^{-5}$ Torr range achieved with a clean pump. In addition, these types of cold traps prevent backstreaming of the pump.

  The nitrogen traps are typically small, efficient and maintenance-free but must be filled and defrosted either manually or automatically. Handling of liquid nitrogen is very easy but can be dangerous if safe handling procedures are not followed (see below).

Cold traps should be used in all high-vacuum systems to prevent backstreaming of the vapor from the diffusion pump into the system. Although most pumping systems have very low backstreaming tendencies, the ultimate vacuum, which can be achieved by a given pumping system, is generally in the 10-6 Torr range. When cold traps are provided just above the pump throat, oil vapors passing this point are condensed and returned to the pump. The pump will then be able to reach a lower pressure than would otherwise be possible.

When comparing clean, outgassed and tight vacuum systems with and without cold traps (Fig. 2), it can be noted that without a cold trap the ultimate pressure in the vessel being pumped is in the neighborhood of $10^{-6}$ to $10^{-7}$ Torr. By placing a cold trap between the pump inlet and the vessel, the ultimate pressure will reach $10^{-9}$ Torr. It should be noted that the ultimate pressure attainable with a given pump depends to an extent on the type of pump fluid used. This gain in ultimate pressure, however, is accompanied by a lowering of the pumping speed – in this case by about 40%. One solution to this problem is to make the trap diameter larger than the pump throat.

When liquid nitrogen is used, it is necessary to keep the reservoirs in the cold trap at a reasonably constant level to ensure constant cooling of the baffles. This can be done manually, but an automatic filling device is preferred for large systems.

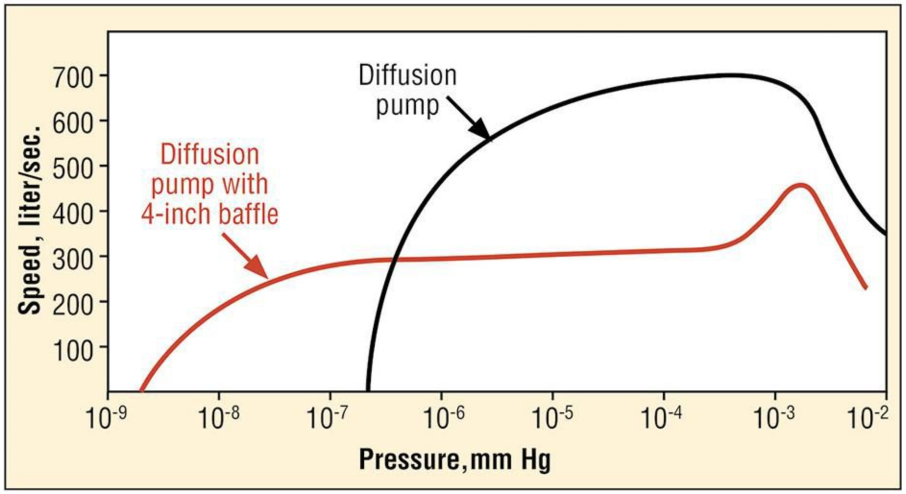

Fig. 2. Typical pumping characteristics with and without a cold trap[3]

## Safety Considerations

Cold traps should be checked frequently to make sure they do not become plugged with frozen material. After completion of a vacuum cycle in which a cold trap has been used, the system should be vented in a safe and environmentally acceptable way. Otherwise, pressure could build up, creating a possible explosion and sucking pump oil into the system. Cold traps under continuous use should be cooled electrically and monitored by low-temperature probes.

Appropriate training and personal protective equipment, including gloves and a face shield, should be used to avoid contact with the skin when using cold baths. Dry gloves should be used when handling dry ice. Lowering of the head into a dry-ice chest is to be avoided because carbon dioxide is heavier than air and asphyxiation can result. The preferred liquids for dry-ice cooling baths are isopropyl alcohol or glycols, and the dry ice should be added slowly to the liquid portion of the cooling bath to avoid foaming. The common practice of using a combination of acetone and dry ice as a coolant is not recommended. Dry ice and liquefied gases used in refrigerant baths should always be open to the atmosphere. They should never be used in closed systems, where they may develop uncontrolled and dangerously high pressures.

Extreme caution should be exercised in using liquid nitrogen as a coolant for a cold trap. If such a system is opened while the cooling bath is still in contact with the trap, oxygen may condense from the atmosphere. The oxygen could then combine with any organic material in the trap to create a highly explosive mixture. Thus, under no circumstances should a vacuum line with a cold trap be opened to air while the liquid-nitrogen source (e.g., Dewar) is in place until the trap has been removed because there is a potential for the formation of liquid oxygen in nitrogen-cooled vessels. Also, if the system is closed after even a brief exposure to the atmosphere, some oxygen (or argon) may have already condensed. Then, when the liquid-nitrogen bath is removed or when it evaporates, the condensed gases will vaporize, producing a pressure buildup and the potential for explosion.

## In Conclusion

Cold traps are a welcome addition to the pumping-system arsenal, especially where low vacuum levels or large contaminant loads are present. They should be considered by heat treaters concerned with improving product quality and wanting to reduce the frequency of pump maintenance.

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# Diffusion Bonding: The Equipment (Part 2)

The Doctor has always been intrigued by the innovation and design capability of engineers. Nowhere is this better illustrated than in equipment used for diffusion-bonding applications. These designs demonstrate ingenuity as well as the ability to adapt conventional vacuum units into a technology with proven performance. Let's learn more.

Multiple stacked component layers (Fig. 1) are typical in vacuum hot-press applications (Fig. 2), which utilize diffusion bonding. Since many metals are easily oxidized, the process is commonly run in specialized vacuum furnaces operating in the range of $10^{-4}$ to $10^{-6}$ Torr or in a hydrogen partial-pressure atmosphere. Hot-press furnace systems operate at temperatures from 400-1230°C (760-2250°F) with up to 30 tons (60,000 pounds) of compacting force or higher. Specialized systems are capable of extending the maximum operating temperature, pressure and vacuum levels.

What we know from our discussion about the process (*Industrial Heating*, April 2017) is that diffusion bonding can unintentionally occur anytime parts are held in intimate contact with one another at a sufficiently high temperature such that interatomic diffusion occurs between the surfaces. In vacuum hot-press diffusion bonding, however, this process is intentional and aided by the application of both temperature and pressure.

In general, vacuum levels can be reduced near to, or just below, the boiling point of any of the major alloy constituents present in the materials being processed. This can be predicted from Ellingham diagrams (*Industrial Heating*, April 2011). The mass transport

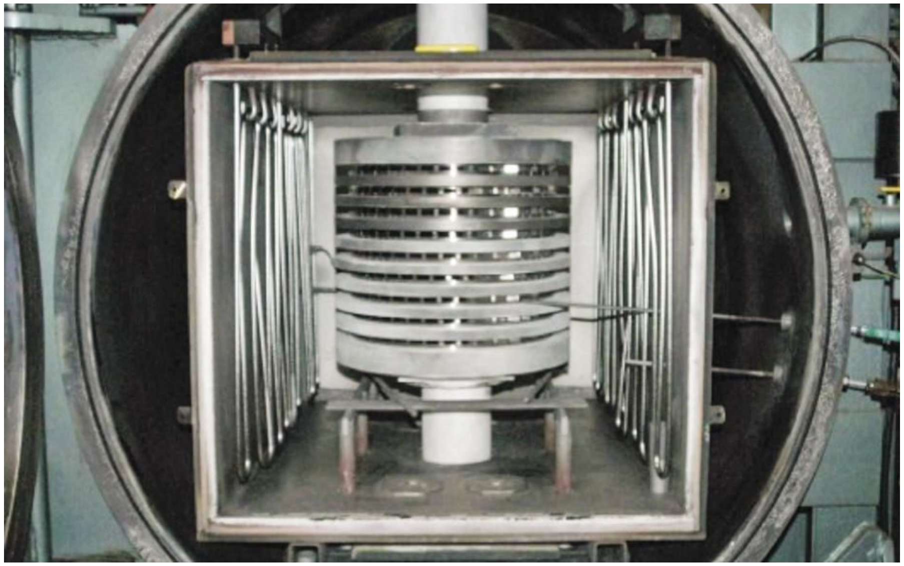


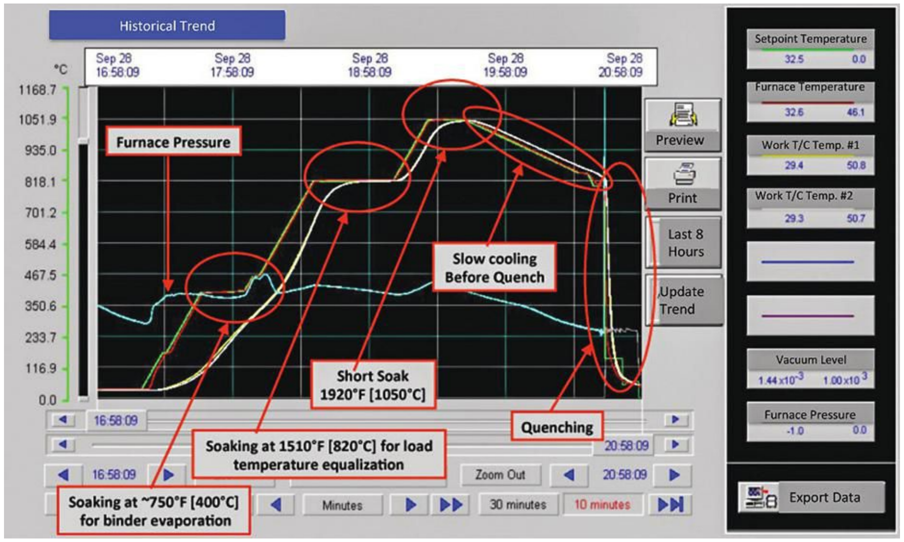
Fig. 1. Typical load in a vacuum hot press (VHP) consisting of nine turbine-engine stator nozzles being simultaneously bonded and flattened (courtesy of Refrac Systems)

rate for the highest vapor-pressure alloy constituent is used to assist in determining bonding rates and controlling the quality of the bond.

Many diffusion-bonding operations are conducted using simple dead weights placed on the top of the workload, which is then placed inside the furnace on a base plate. This ensures that all surfaces of the bond interface are in intimate contact. For thicker parts or stacks where residual stress may be present from prior manufacturing operations, however, a natural stress relief occurs upon heating and often results in uncontrolled distortion. In these instances additional force is required to maintain flatness, making the use of dead-weight loads impractical.

Over the years, the need for larger stacks has resulted in the routine use of tooling methods that make use of either a differential expansion mismatch or some type of active-loading system, such as pressure bellows or pneumatic/hydraulic ram systems that can be used to apply the necessary amount of force.

With bolted differential expansion or so-called caged-tooling designs, the parts almost always suffer from the fact that the maximum load is applied before reaching the final bond temperature. This can result in part distortion (due to strain). Since the parts are also under significant stress during heat-up, surface degradation and bending/bowing (often referred to


Furnace Pressure

Soaking at ~750°F [400°C] for binder evaporation

Soaking at 1510°F [820°C] for load temperature equalization

Short Soak 1920°F [1050°C]

Slow cooling Before Quench

Quenching

Setpoint Temperature 32.5 0.0

Furnace Temperature 32.6 46.1

Work T/C Temp. #1 29.4 50.8

Work T/C Temp. #2 29.3 50.7

Vacuum Level 1.44x10-3 1.00x10 3

Furnace Pressure -1.0 0.0

Fig. 2. Typical vacuum hot-press cycle (courtesy of SECO/Vacuum Technologies)

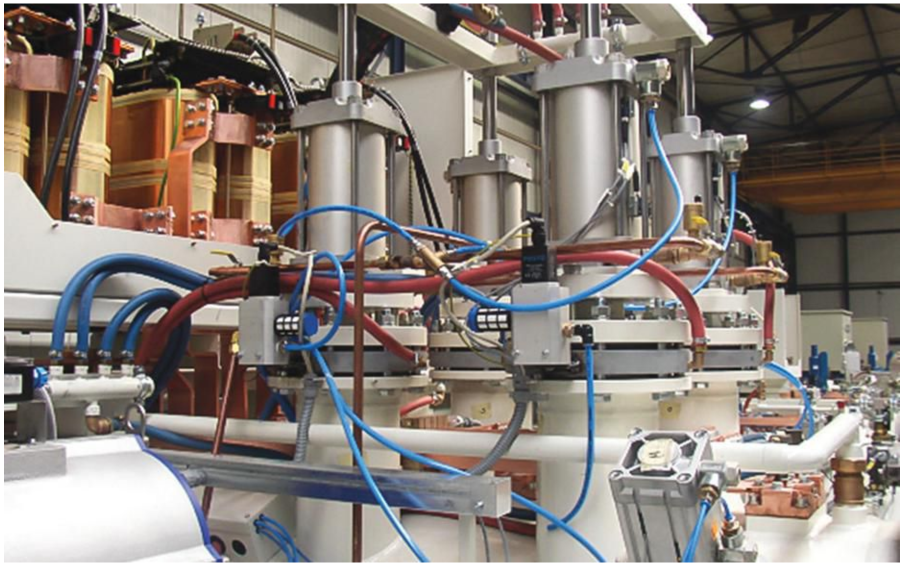


Fig. 3. Exterior view of furnace platens (courtesy of SECO/Vacuum Technologies)

as “potato chipping”) may result. This is due to differential expansion (shear stress working against the outwardly growing surface layers) during heating and cooling. One indication that this is happening is galling tracks on the part surfaces.

The need for better control of vacuum level and applied load stress has led to the development of more advanced vacuum hot-press systems. These are conventional vacuum furnaces with the addition of external rams (Fig. 3) and a hot platen inside the furnace. The arrangement is similar to an arbor press. Flat platens distribute the load uniformly onto all part surfaces. The advantages of uniform force are significant from both a mechanical and metallurgical perspective.

A typical small hot press (Fig. 4) would have features such as:

- 300 x 300 mm (11.8 x 11.8 inches) refractory-metal pressing plates
- 150 kN (33,720 pound-force) pressing force
- 350°C (2460°F) maximum temperature
- Mid-10-6 Torr ($10^{-6}$ mbar) ultimate vacuum

Recent advances in the diffusion-bonding industry have led to the development of vacuum hot-press systems that include gas-quench cooling capabilities with internal heat exchangers so that the parts may be diffusion bonded and then quenched to optimize the post-bond heat treatment (solution annealing, aging or hardening) and resulting material properties. Typical features of these specialized systems might include:

- 2,275-kg (5,000-pound) load capacity
- 914 x 1,220 mm (36 x 48 inches) pressing platen footprint
- 890 kN (200,000 pound-force) pressing force
- 1455°C (2650°F) maximum temperature
- Mid-$10^{-6}$ Torr ($1x10^{-6}$ mbar) ultimate vacuum
- Partial-pressure control for hydrogen
- 2-bar gas-quench cooling

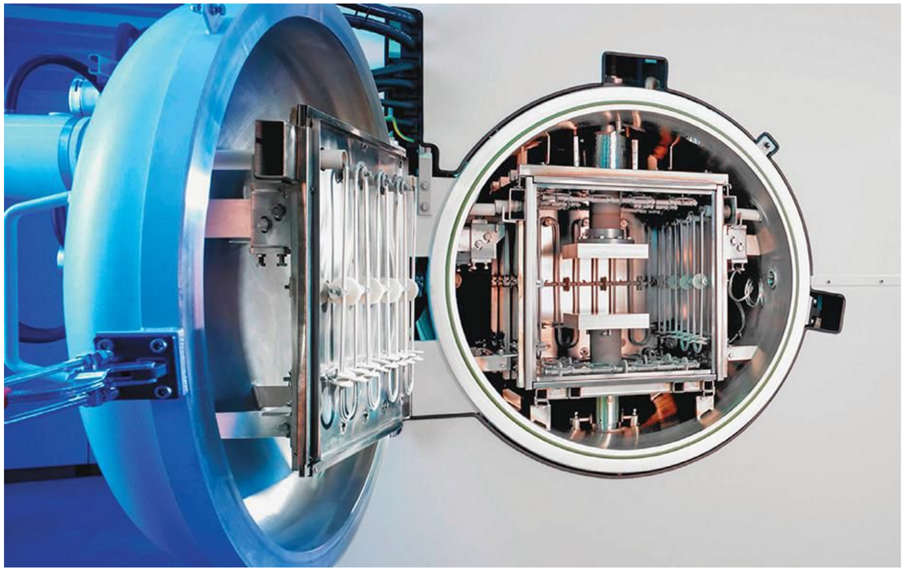


Fig. 4. Interior view of a typical hot-press vacuum furnace (courtesy of PVA Industrial Vacuum Systems GmbH)

## Summary

Many different tooling and bond joint-preparation methods can be used in vacuum diffusion bonding. Ultimately, the end user must make the decision on the best method(s) to employ to minimize part production cost. This decision is often driven by such factors as part geometry, allowable strain, resultant microstructure or simply the total number of parts that must be produced.

Vacuum diffusion bonding has made great strides in recent years and overcome many of the problems that plagued the technology in the past (e.g., dirty materials, uneven contact surfaces, equipment design limitations and equipment design flaws). As such, it has become a robust technology and one worthy of consideration by the design engineer.

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# The Navy C-Ring Test – A Practical Tool for the Heat Treater

Heat treaters are forever curious about how their furnaces are performing; in particular the uniformity of properties that will be achieved throughout the load. We often look to sophisticated tools for answers, but there is a simple, yet highly effective, method for quantifying our furnace's performance – the Navy C-ring test. Let's learn more.

Originally designed to study dimensional changes that occur in the heat treatment of hardened and/or case-hardened components, the power of the Navy C-ring test is that it can be adapted and used while running production loads to determine both the overall performance capability (i.e., condition) of the furnace and the heat-treatment process being conducted in it. It can also be used to compare in-house heat treatment with that of outside commercial services.

This test can be structured in such a way as to aid in the evaluation of atmosphere or vacuum furnaces. Processes such as normalizing, hardening and case hardening can be examined and the performance of oil or high-pressure-gas quenching methods studied. The test can be extended from its original purpose to reveal the following types of information (as a function of position within the workload) in both the "as-quenched" and "as-tempered" condition:

- Hardness uniformity (surface, core)
- Dimensional change (distortion)
- Quench system (oil type, agitation, temperature) effectiveness/uniformity
- Carburizing uniformity (effective and total case depth plus case variation by position)
- Microstructural uniformity (including retained austenite levels)
- Material hardenability
- Material surface stress state
- Sensitivity to cracking (as a function of various quench conditions or media)

One is not limited to a particular material grade. As such, C-rings are made from both ferrous (e.g., steel, stainless steel, tool steel) and nonferrous (e.g., aluminum, titanium) materials. For steel parts, SAE 1010, 4140, 4340, 8620 and 9310 are typical examples. It is important that the C-rings are made of the same material (and ideally from the same heat) as

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the production parts.

## What is a Navy C-ring?

Essentially, the Navy C-ring is a short cylinder with an eccentric hole and open in one extreme (Fig. 1). The original design (specified to conform to U.S. Navy Department Specifications for Tool Steels, No. 47S5c, July 1, 1921)[3] has a thickness of 25 mm (1 inch). Modifications of this design are common to mirror the size and thickness of the actual parts being processed. It is important to run C-rings of the same physical dimensions within a given load.

## How to Conduct the Test

All parts should be measured before and after heat treatment using a coordinate measuring system (CMM) to precisely determine geometrical dimensions. This is critical for subsequent statistical analysis of the data. The Navy C-ring samples can then be positioned in a workload in either a vertical (if an optional hole is drilled in the specimen for hanging) or horizontal orientation. Typically, a minimum of nine rings is used, positioned in the corners and center of the load (like a temperature uniformity survey) along with production parts. The rings can also be positioned in individual baskets stacked to make up a load.

Testing of samples in both the as-quenched and as-tempered condition should be done for surface and core hardness, microstructure, case depth (via microhardness), retained austenite and (if desired) residual stress via X-ray diffraction (XRD) to obtain a complete data set.

## Original Test Focus

Historically, the main focus of the Navy C-ring test has been to evaluate dimensional changes. In simplest terms, distortion of an engineered component can be defined as a change in its shape or volume during either manufacturing (including heat treatment) or in service.

Distortion during quenching is the result of differential volume changes due to heat extraction and/or phase transformations. These dimensional changes can dramatically influence manufacturing productivity due to necessity for post-heat-treatment machining operations. Furthermore, when distortion is severe, the potential for crack formation becomes a paramount concern.

The major factors that influence distortion[1] are cooling rate, hardening treatment (e.g., carburizing, ferritic nitrocarburizing), material hardenability and chemical composition.

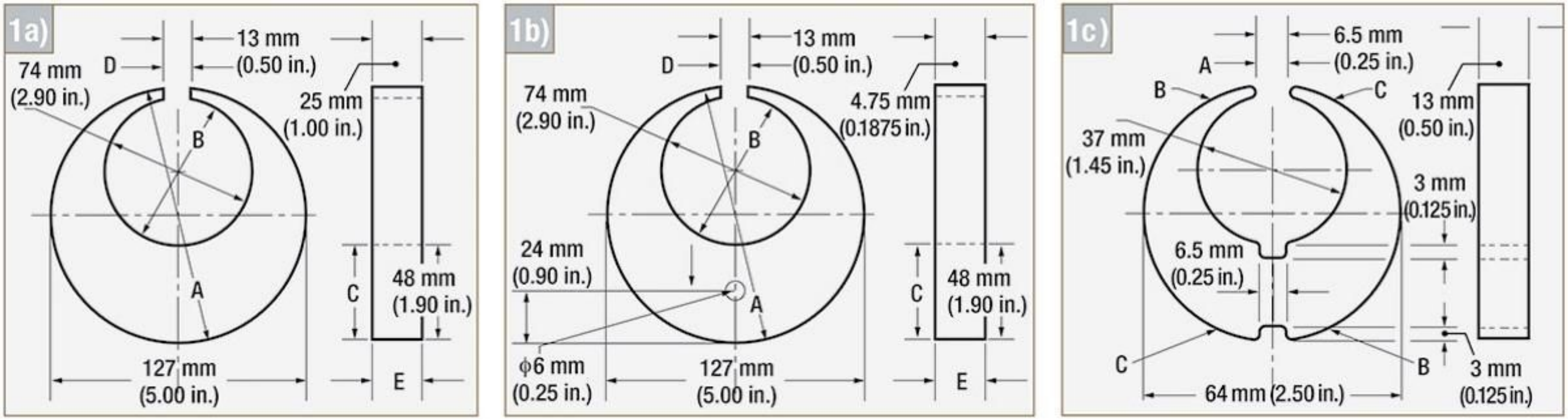

Fig. 1. Typical Navy C-rings; (a) – (c) Examples of size/type variation in Navy C-ring specimens (a) original design (b) original with hole (c) custom half-size with keyways.

Investigating these factors[1] has revealed that the rate of cooling from carburizing is extremely important. Very fast cooling rates (e.g., water quenching) completely outweigh the effects of major changes in composition. By contrast, while the carburizing process reduces dimensional movement in low-alloy steels, it has a lesser effect in steels of high hardenability.

Steel composition is more complex, and distinctions must be made between the effect of composition on increasing hardenability and the effect on the depression of the martensite-start temperature in fully hardenable grades. Both aspects must be fully understood to correlate dimensional movement over a wide range of compositions. To illustrate this point, the distortional behavior of boron steels is entirely different from non-boron steels of comparable hardenability.

The specification of steel chemistry to restricted-hardenability grades has been reported to reduce the variability of distortion. Even greater effect has been found by the use of steels in which the hardenability is greater than that required for through-hardening for a given section size.

The Navy C-ring has been effectively used to evaluate the final distortion produced by quenching of steel parts. This simple specimen can provide distortion information as a function of heat-treatment condition and position within the workload with respect to changes in the:

- ID
- OD
- Gap width
- Thickness
- Flatness
- Cylindrical dimensions
- Roundness
- Bore (if an optional hole is machined in the sample)
- Change in size on deep freeze or cryogenic treatment and single or multiple tempers (as a function of both temperature and time)

In addition, one can understand the effect of material (composition) and initial microstructure on size and shape distortion, retained austenite and residual stress.

Furthermore, the test can help evaluate the effectiveness of prior hardening processes such as (mill) annealing or normalizing prior to carburizing. Retained austenite evaluation of carburized components is another parameter of interest, especially in carburized steels that can contain varying amounts of retained austenite in the quenched-and-tempered condition – depending on the material (i.e., alloying elements) and process parameters used (e.g., carburizing and hardening temperature, carbon potential, cooling rate). Retained austenite can influence near-surface hardness and dimensional stability over time.

Heat-treat processes also create residual stress in a material, which results in dimensional variation (i.e., size and shape changes) both within a given part and from location to location within a workload.

## Summary

In this day and age of uncompromising part quality, the inclusion of Navy C-ring testing on a quarterly basis will prove an invaluable aid in reducing both equipment- and process-related variability. Testing of rings for other than just dimensional change can serve as confirmation of process stability with regard to such items as temperature variation throughout the load, quench effectiveness, surface and core hardness differences as a function of position and overall material hardenability. The results of these tests can help qualify process capability, evaluate process (recipe) changes, confirm the validity of control instrumentation, direct maintenance activities and act as an invaluable quality-control tool (especially when statistical data analysis is performed). As they say, “Just do it.” You’ll be glad you did.

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# Tempering Revisited

The Doctor is often asked questions about tempering, from something as fundamental as "Is it necessary?" to something a bit more complex like "What are the rules when re-tempering parts?" Other frequent questions include, "How long do I have after quenching before I temper?" or "How many tempers should I perform?" These are all important questions, and every heat treater must know the answers. Let's learn more.

## What is tempering?

While the end-use application of a component determines its heat treatment, as heat treaters we are constantly striving to achieve a delicate balance between the properties of strength and ductility. Nowhere is this fine line more evident than in the tempering (aka drawing) process for a given steel, where precise control of time, temperature and (in some instances) cooling rate are critical.

Essentially, tempering (Fig. 1) is the modification of the newly formed hardened microstructure toward equilibrium. For this reason (if no other), tempering should always be performed. Almost all steels that are subjected to any type of hardening process are tempered – subjected to a subcritical heat treatment that alters their microstructure and properties (Fig. 2). In general, tempering lowers strength and hardness while improving ductility and toughness of the as-quenched martensite.[3]


Fig. 1. Typical load exiting a tempering furnace (courtesy of MacLean-Fogg)

Precipitation-hardened alloys, including many grades of aluminum and superalloys, are tempered to precipitate intermetallic particles, which strengthen the metal. Tool and high-speed steels are often tempered multiple times to achieve proper hardness while transforming retained austenite first to untempered martensite and on subsequent tempers to tempered martensite.

## Snap (aka Safety) Temper

Low-temperature tempering in the range of 135-150°C (275-300°F) prior to conducting a standard temper operation is often referred to in the heat-treatment industry as a "snap" or "safety" temper. It is also known out on the shop floor as "taking the curse off" the material. It is a step added to the process recipe when the time between the quench and temper operations will be longer than 1-2 hours or no longer than 15-30 minutes after quenching for high-hardenability steels. A snap temper should always be performed at a temperature lower than the final tempering temperature. Snap tempering is also commonly performed prior to an initial deep-freeze operation to minimize stress, negate geometry effects and avoid cracking.

## Retempering

Retempering is an operation in heat-treat shops that is often added to a process sequence for one of several reasons. One of the more common reasons for retempering is the as-hardened and tempered product exceeds the hardness aim (or range) and is concomitantly low in ductility and/or toughness. This can be a somewhat common occurrence, especially in the early history of a component's manufacture, until an empirical dataset can be established over many cycle runs and heats of material. Most manufacturers would "rather retemper than requench." No one wants to repeat multiple operations if a simple retemper will resolve the issue. If the initial results are out of range or too far from desired target values, the parameters for a retempering operation can be readily determined.

Retempering is performed over a wide variety of temperatures and times relative to the original tempering operation, depending on a host of material and process factors. As previously noted, tempering is a time-temperature-dependent process, and an added temper (or multiple tempers) enhances the effects of the original tempering operation.

The Hollomon-Jaffe and/or Larson-Miller time-temperature tempering-parameter equations are widely used and have been refined over the years.[6] These equations are extremely useful and can aid the heat treater in making estimates of the effects of the original temper and the cumulative effects of any subsequent tempers on many different material

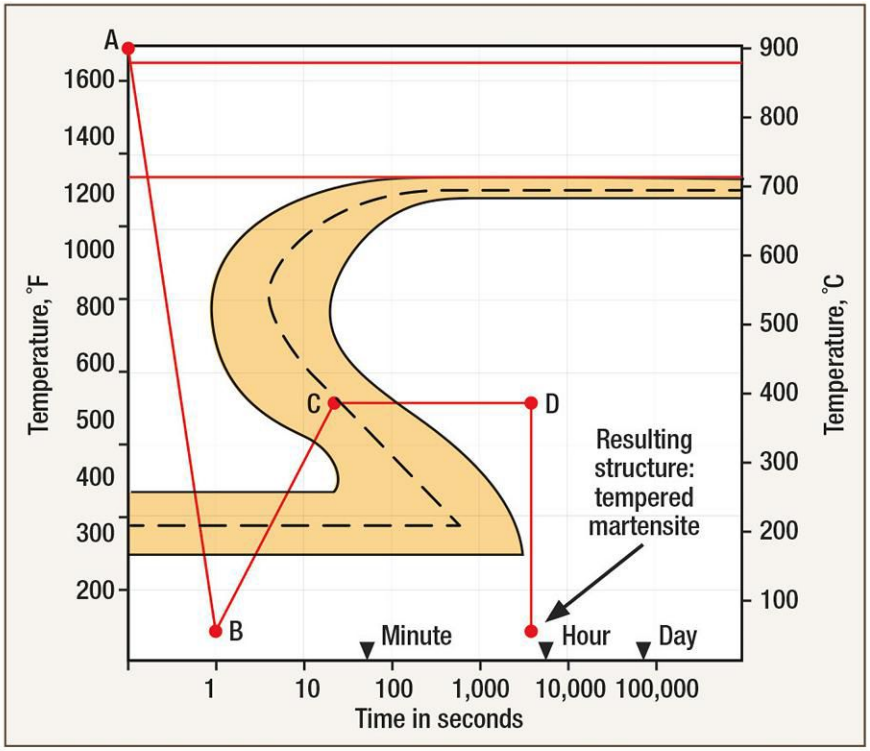

Fig. 2. Conventional tempering cycle[5]

properties (e.g., strength, hardness, ductility, toughness, microconstituent details). This is crucial to ensure the retempering operation will not only provide the desired corrective effects on the property of interest but will also not create any foreseen or unforeseen detrimental effects.

Caution must be exercised that a retemper does not raise the tempering temperature into the tempered-martensite-embrittlement (TME) zone. This would not only embrittle the steel, but – since TME is an irreversible phenomenon – the components would have to be re-hardened. This has the potential to create many additional problems, and it is a very easy processing trap to fall into. While the hardness and strength could be readily adjusted to a desirable level, the toughness could be significantly compromised.

A second common reason for retempering is to recover tensile yield strength when a product is cold deformed in a compressive manner during its manufacture. The working in compression during a cold-forming, cold-drawing or cold-straightening operation can trigger the Bauschinger Effect, which results from strain-hardening effects. The result is that when quenched-and-tempered material is strained in compression, the yield strength in tension decreases. Fortunately, the phenomenon is reversible and can generally be resolved via a retempering operation. Such a retemper is usually conducted at a temperature 28-56°C (50-100°F), or more, lower than the initial temper. This is done to preserve the ultimate strength/hardness while recovering the tensile yield strength by eliminating the strain-hardening directionality effects.

Caution must also be exercised when processing quenched-and-tempered steels that are susceptible to temper embrittlement (TE). A susceptible steel initially tempered at 590°C (1100°F), cold worked and then stress-relief tempered at 540°C (1000°F) could be detrimentally impacted.

Similarly, one must be aware when retempering is conducted in the range where secondary hardening can occur in the more highly alloyed steels or in precipitation-hardening materials in various alloy systems. An engineering review of the tempering characteristics of any workpiece material should always be conducted and appropriate heat-treating plans designed. This is especially true for instances where the alloy content can result in the "fourth stage of tempering"[4] and provide significant effects to the resulting properties.

The question naturally arises as to the acceptability of retempering in a given operation. One must understand the extent to which retempering is to be considered a rework/reprocess operation. It is important to establish the proper protocols for advising specific parties of a retemper when one is conducted. One must be aware of the governing specification(s) for the product and operations at hand, those of the processor and those of the customer. It is good practice to have these protocols predetermined.

An excellent example is provided by AIAG in the current CQI-9 specification,[7] which states the following relative to heat-treat processing and retempering. "Question: Is the OEM customer to be notified when parts are reprocessed?" Requirements and Guidance (partial list, paraphrased):

- The OEM is to be notified when parts are reprocessed
    - Notification on a case-by-case basis is preferred
- Some reprocessing (such as, but not limited to, retempering operations) may be preapproved
    - To be preapproved, the heat treater:
        - Shall submit a reprocessing procedure for approval by the OEM
        - The procedure shall describe process characteristics for which reprocessing is permissible and those for which it is not
        - Any reprocessing activity requires a new processing control sheet, which denotes the necessary heat-treat modifications
        - Records shall show when and how the material was reprocessed

Retempering can be, and often is, a commonly employed process in heat-treatment operations for intermediate and final products for a large number of industries and various alloy systems. The heat treater must know and understand the metallurgical properties and thermal-processing characteristics of their workpiece materials if retempering is employed.

## Conclusion

When it comes to tempering, there seems to be resistance to performing multiple tempers simply due to the delay involved in releasing the final product back to manufacturing. In this writer's eyes, multiple tempers are always advantageous, unless very long tempering times are involved.

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# Stress Relief

Stress relief is a heat-treatment process that relies on slow cooling to achieve its desired effect, and it is influenced by a number of factors including the internal stress induced into the parts from the various manufacturing methods (e.g., bending, shearing, forging, sawing, machining, grinding, milling, turning, welding, etc.) and prior processing. The application end use ultimately defines the allowable stress state. So how does one perform a stress-relief operation? Let's learn more.

Processes that depend on slow cooling (e.g., annealing, normalizing, stress relief) do so for a number of reasons – to relieve stresses, improve chemical homogeneity, soften a material for subsequent operations (e.g., machining), refine grain size and for such reasons as embrittlement relief or magnetic properties.[1] As a general rule, the larger or more complex the part, the greater the amount of internal stress present.

Stress relief can be differentiated from other slow-cooling processes in that it is most often performed below the lower critical temperature ($Ac_1$). Time at temperature depends on such factors as the complexity of the part, and enough time must be allowed in order to achieve the desired reduction in residual-stress level. Following stress relief, the steel is cooled at a sufficiently slow rate to avoid formation of or reintroduction of excessive thermal stresses. No microstructural phase changes occur during the stress-relief process..

## How slow is slow?

Stress relief most often requires a "still-air cool" so as not to reintroduce stress into a material, but what does this really mean? A still-air cool (quench) can be defined as cooling at a rate of 40°F (22°C) per minute or faster to 1100°F (593°C) and then at a rate of 15-25°F (8-14°C) per minute from 1100-300°F (593-150°C). Below 300°F (150°C) any cooling rate may be used.

## How do we perform a stress-relief operation?

For carbon steels, stress-relief operations are typically performed at 105-165°F (40-75°C) below the lower critical temperature – in the range of 930-1200°F (500-650°C). It is also important to understand that the elimination of stress is not instantaneous, being a function of both temperature and time for maximum

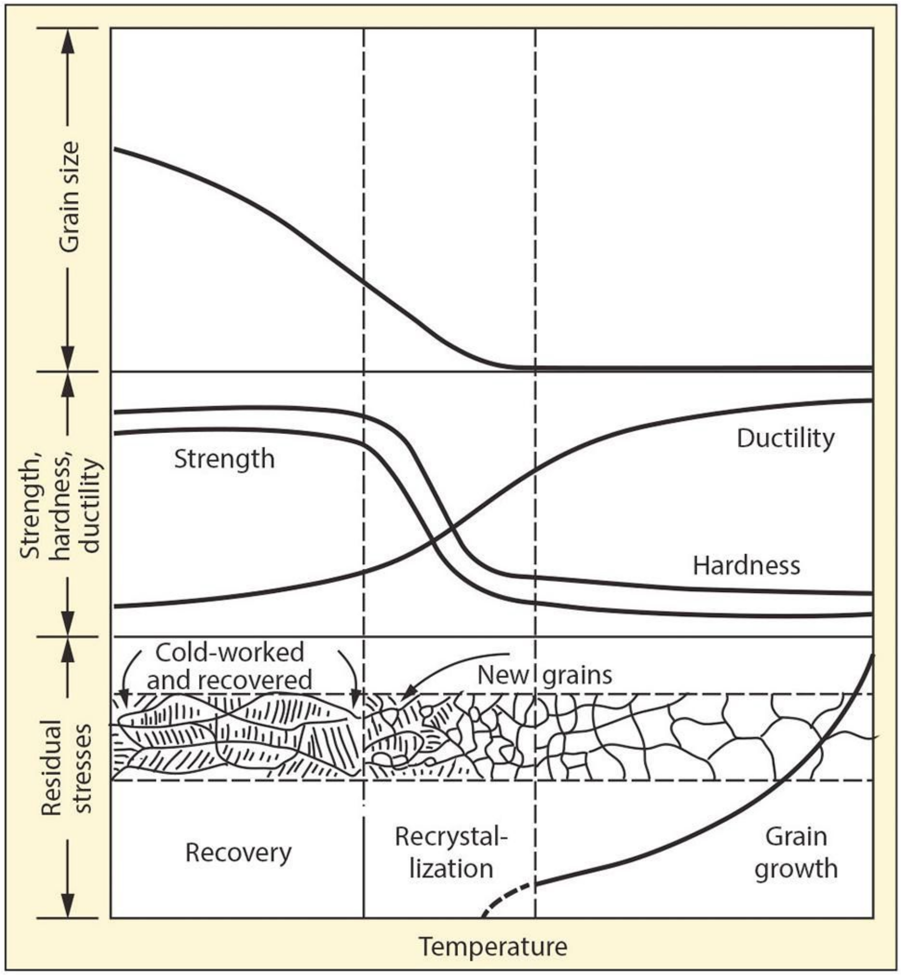

Fig. 1. Effects of recovery and recrystallization on grain structure

benefit. Typically, one hour per inch (25 mm) of maximum cross-sectional area (once the part has reached temperature) is required. After removal from the furnace or oven, the parts are air cooled in still air. Rapid cooling will only serve to reintroduce stress, and this is the most common mistake made in stress-relief operations. This cycle is estimated to remove more than 90% of the internal stresses. Stress relief on alloy steels is often done at (slightly) higher temperatures.

For tool steels, the process is similar. It is common to perform a stress-relief operation in the temperature range of 500-550°C (925-1025°F), allowing the parts to slowly cool to room temperature before subsequent operations.

For stainless steels, the situation is more complex.[2] Stress relief is done in the range of 290-425°C (550-800°F), which is below the sensitization range. Stainless stress relief depends on the form of the material, the operation being performed (e.g., machining) or whether a completed assembly will be stress relieved.

## Poor Man's Stress Relief

In hardening, rapid cooling/quenching alone or in combination with pre-existing internal stresses can result in unwanted distortion, brittle fracture and, if near welds in certain grades of metal, stress corrosion cracking. For this reason, a number of heat treaters introduce a "stress-relief hold" during hardening or case-hardening treatments. This involves heating of a workload to an intermediate temperature – in the range of 1000-1300°F (538-705°C) – and soaking for a period of time equivalent to one hour per inch of maximum cross-sectional area. The idea is to allow for stress relaxation so that more predictable dimensional change occurs on quenching.

## Stress Relief of Springs

Stress relief is one of the most common heat-treating processes used in spring manufacturing as well as the manufacture of other wire-formed products. Drawing, forming and machining induce stresses in all wire products. These stresses can cause loss of tolerance, cracking and distortion and contribute to in-service failures. For these reasons, stress relieving is often necessary and, in many cases, mandatory.

In addition to removing stresses, stress relief returns the material to a strength level approximately equivalent to where it was prior to forming. Studies have shown[3] that the interstitial elements pin the lattice defects in the atomic structure of the metal, resulting in this increase in mechanical strength.

To completely eliminate residual stresses in helical springs through stress relief, the material must be heated high enough to fully recrystallize. This is not practical in spring manufacturing since the recrystallization process significantly reduces the material's strength and, therefore, its usefulness in spring applications. On the other hand, an elevated-temperature recovery process (i.e. stress relief) can eliminate the majority of residual stresses without significantly deteriorating the material's strength (Fig. 1). The temperature required to accomplish the recovery process depends on the material type and processing history (i.e. carbon steel vs. alloy steel, cold drawn vs. oil tempered, etc.) The SMI Encyclopedia of Spring Design[4] provides recommendations for proper recovery.

Temperature, time and time at temperature are key process variables, and these have been documented elsewhere.[5] In general, heating steel to a temperature of about 165°F (75°C) below the transformation temperature ($Ac_1$) for about one hour (or until the entire part reaches temperature) will allow for the removal of most internal stresses. Typical temperature ranges are:

- 1025-1200°F (550-650°C) for unalloyed and low-alloy steels
- 1115-1300°F (600-700°C) for hot-work and high-speed tool steels

For many alloy steels, little or no stress relief occurs at temperatures less than approximately 500°F (260°C) while approximately 90% of the stress is relieved by 1000°F (540°C). The maximum temperature for stress relief is limited to 55°F (30°C) below the tempering temperature used after quenching from the hardening process. After removing from the furnace or oven, the wire must be cooled in still air. If cooled in any other manner, stresses are reintroduced into the part.

Many other severely cold-worked or bent shapes can be heated between 400-800°F (205-425°C) for a relatively short time to help reduce internal stresses. Alternative stress-relief processes (e.g., vibratory stress relief, rapid tempering/stress relief) are covered in the references.[6,7,8]

## The Final Word

The influence of internal stress can be positive or negative, which means that one must understand the design application in order to apply the proper stress-relief operation (at low or high temperature). In lightly stressed parts where dimensional tolerances are not critical, the presence of internal stress is not as great a concern as a highly stressed component that must hold dimensional stability over time or where the service application is such that excessive distortion or even fracture may occur.

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# Low-Temperature Vacuum Heat-Treatment Processes

Low-temperature vacuum heat treatment is one of The Doctor's favorites, offering unique advantages over other types of low-temperature processing since component parts (Fig. 1) are placed in a controlled environment designed to minimize surface interactions. Let's learn more.

Applications for this technology vary widely but generally fall into the following categories:

- Aging
- Annealing
- Oxidizing/Bluing
- Stress relief
- Tempering

Fig. 1. Annealing of nickel-based alloy jet-fighter afterburner assemblies

Typical materials include:

- Alloy and high-carbon steels (including maraging grades)
- Beryllium copper and beryllium nickel
- Brass
- Copper
- Inconel
- Specialty alloys (Elgiloy®, NiSpan C, Nitralloy)
- Stainless steels, including precipitation-hardening grades
- Titanium alloys
- Tool steels

Low-temperature vacuum heat treatment is used by both captive and commercial heat treaters and spans such diverse markets as aerospace, automotive, electronics, optics, housewares, industrial products, tool & die, military/defense and farm implement to name a few.

Most processes run in the temperature range of 175-730°C (350-1350°F). Special applications extend these ranges down to as low as 120°C (250°F) and up to as high as 925°C (1700°F), but this is unusual. Temperature uniformity (Table 1) in dedicated vacuum furnaces is considered excellent throughout the

**Table 1. Field data on low-temperature vacuum-furnace uniformity[1]**

| Control temperature, °C (°F) | Deviation from control, °C (°F) | Total spread, °C (°F) |
|---|---|---|
| 176.7 (350) | +1.2, - 0.2 (+2.0, - 0.5) | 1.4 (2.5) |
| 204.4 (400) | +1.1, - 0.0 (+2.0, - 0.0) | 1.1 (2.0) |
| 246.1 (475) | +1.9, - 0.3 (+3.5, - 0.5) | 2.2 (4.0) |
| 315.6 (600) | +1.1, - 2.0 (+2.0, - 3.5) | 3.1 (5.5) |
| 371.1 (700) | +2.8, - 0.5 (+5.0, - 1.0) | 3.3 (6.0) |
| 565.6 (1050) | +3.9, - 1.1 (+5.0, - 2.0) | 5.0 (7.0) |
| 676.7 (1250) | +2.2, - 0.6 (+4.0, - 1.0) | 2.8 (5.0) |
| Notes: a. Class 1 per ASM 2750D (Pyrometry) | | |

standard temperature ranges listed.

It is also worth noting that clean and/or bright work is most often associated with vacuum processing. Since "clean" and "bright" are very subjective terms and difficult to define in a universal way, we tend instead to say that the part surface is not metallurgically damaged. If a change occurs, it is generally a positive one. In all cases, the surface condition of the parts being processed are said to be improved.

## Vacuum Designs

Vacuum furnaces for low-temperature processing can be batch or continuous, stand-alone, integrated into continuous vacuum furnace systems or a separate "module" incorporated into a cellular system. For example, the basic operation for a batch vacuum furnace is as follows. Mechanical vacuum pumps, optionally equipped with blowers, produce vacuum levels down to 1.3 x 10-3 mbar (0.001 torr) with 6.7 x $10^{-3}$ mbar (0.005 torr) common. This is normally achieved within $10^{-30}$ minutes of the start of cycle, depending on the size of the pumping systems and the nature of any contaminants present on the workload. The unit is then backfilled in the range of 66.7 x $10^{1}$ mbar (500 torr) negative pressure to 0.10 bar (1.5 psig) positive pressure with an inert gas such as nitrogen, argon or a mixture of nitrogen/hydrogen (3% maximum) and heating begins. Double pumpdown cycles are often found to be advantageous to speed the overall cycle time. After reaching setpoint and soaking at temperature, the cooling cycle is initiated.

The materials of construction in the heating chamber are such that the furnace can be opened and unloaded at any required temperature. In most cases, however, surface condition is important and the workload must be cooled to at least 150°C (300°F) and more commonly to below 65°C (150°F), as measured by a thermocouple positioned in the workload itself. These units can be either gas fired or electrically heated. Fiber insulation is typical, often in the form of a "hard pack" or rigidized so as to withstand the high velocities produced by the convection fan. These design features translate into rapid heat-up and cool-down rates (Figs. 2 & 3).

## Summing Up

Low-temperature vacuum processing of workloads is becoming increasingly more common due to a need for improved surface quality, better process repeatability, control of process and equipment variability, and the ability to predict quality results. Designs capable of meeting these needs are available and perform well.

Some of the key considerations for choosing low-temperature vacuum processing can be summarized as:

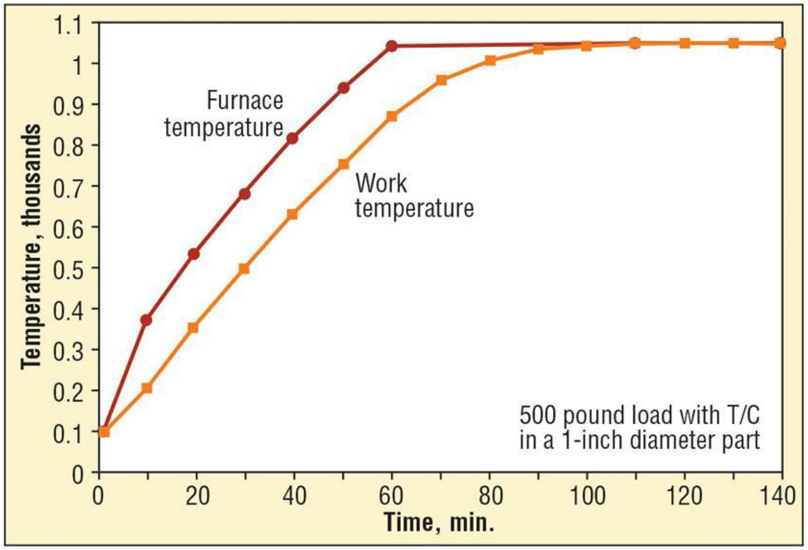

Fig. 2. Typical heating-rate performance data[1]

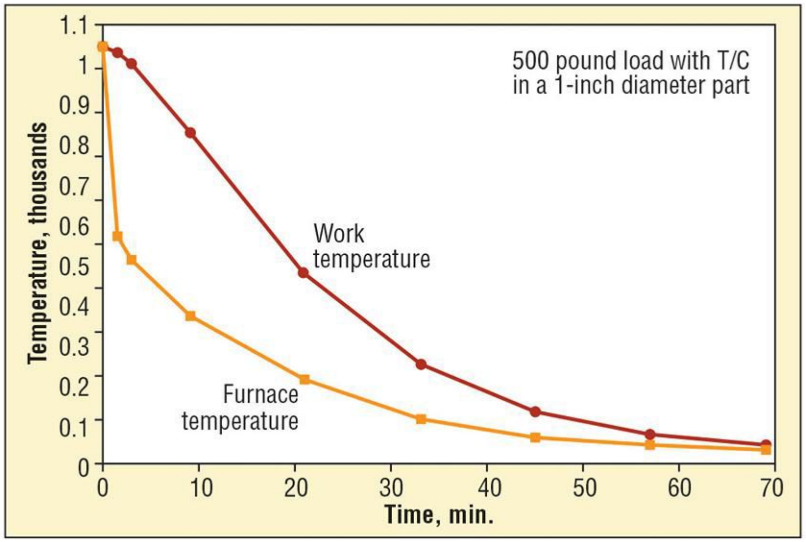

Fig. 3. Typical cooling-rate performance data[1]

1. Vacuum heat treating is mandatory for parts that must be processed without surface damage (e.g., oxidation). Parts at all stages of the manufacturing process, not just finished surfaces, benefit from this type of treatment.
2. Heating and cooling is uniform and fast with minimum energy consumption.
3. High productivity requirements are best achieved by vacuum processing. The ability to heat slightly more rapidly and, especially, to cool much more rapidly in a positive pressure reduces cycle time. Gas-fired equipment is especially beneficial in this regard.
4. Minimum atmosphere consumption. Once partial pressure or backfill gas is introduced, only small amounts of make-up gas are needed. Even in cases where repeated gas flushing is required, far less atmosphere is needed.
5. Process control is absolute, including the ability to upload recipes and download process and equipment variables in real time. Planned preventative-maintenance practices and a complete history are simple and straightforward.

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# The Heat Treatment of Fasteners – The Practical Side

Heat treating, while perhaps not the most significant cost element in the manufacture of fasteners (Fig. 1), is critical to achieving proper quality and performance. As heat treaters we are often faced with high volumes and tight margins. Let's learn more.

Fasteners come in almost every conceivable shape and size. Fasteners have different thread types (e.g., coarse, fine, extra fine), come in various lengths, with grip or no grip (shank), have different types (e.g., hex, 12 pt, carriage), and different coatings (i.e. passivated, cadmium, dry film lube, hot dip tin, etc.). There are various classes of fit (i.e. class 3) and multiple grades (e.g., grade 2, 5, 8). Fasteners come with left- or right-hand threads, metric or SAE, different number of threads per inch (e.g., 20 or 28 for the same size fastener) and various versions of those (e.g., UNF vs. UNJF).

Fasteners are also made from a variety of different materials (Table 1). Irrespective of their form or material, the heat treatment of fasteners is most often done using mass-production methods (Fig. 2), which presents unique heat-treating challenges.

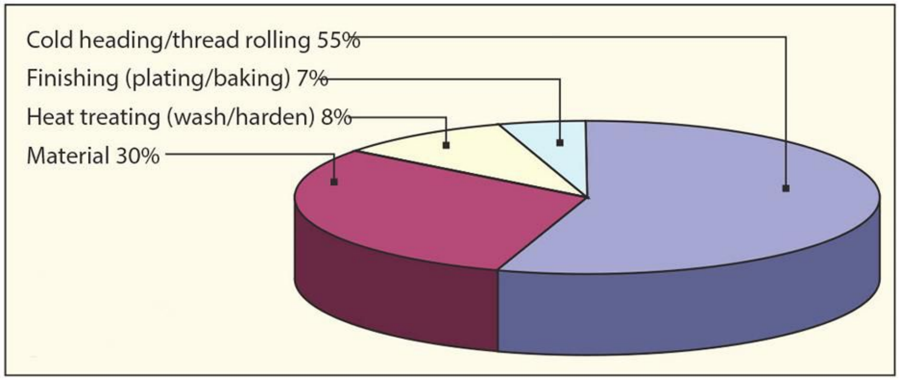

Fig. 1. Domestic fastener production costs, No. 8 (M3–M4) screw

## Types of Heat Treatments

he most common heat treatments for fasteners involve through or selective hardening and case hardening (carbonitriding and carburizing). Case depths are shallow – typically 0.0015–0.015 inch (0.0038–0.038 mm) specified in ranges of 0.005 inch (0.127 mm). Quench media runs the gamut from brine, water, polymer, oil and salt depending on specification requirements.

In mesh-belt conveyor furnaces (Fig. 3) it is not uncommon to see fasteners loaded between ½–2½ inches (12.7–63.5 mm) deep. Also, whether we like it or not, the reality is that fasteners typically enter the furnace wet and with a certain amount of oil, soap or

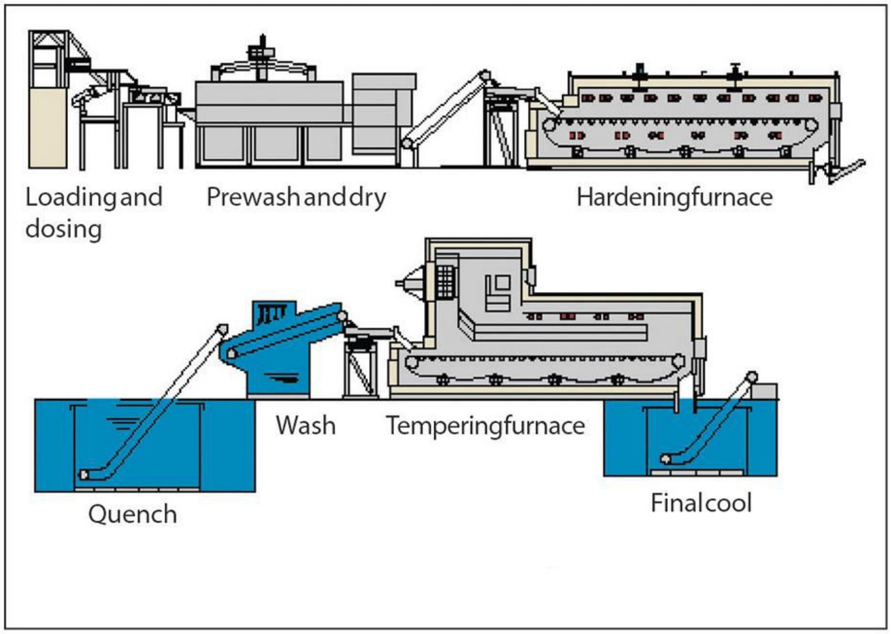

Fig. 2. Typical mesh-belt furnace system with oil-quench capability

phosphate residue. If you were to pick up a handful of fasteners off the belt, your hand would be soaking wet and slippery from the amount of oil present. As such we are taxing our furnace atmosphere severely, and atmosphere direction, flow rate and control are critical considerations, as is the performance of the preheat zone. It is not uncommon to see multiple-zone furnaces. For example, a four-zone mesh-belt furnace will normally have circulating fans in the first three zones with zone 1 and 3 running in the same direction and zone 2 running in the opposite direction. Needless to say, the type of fan and fan speed are important considerations.

Induction-heating techniques (Fig. 4) can also be used to heat treat fasteners at high production rates. For example, high-frequency (10–50 kHz) systems can draw back 8620 bolt heads after carburizing to improve toughness at a rate of 1–2½ pieces/second. Similarly, 4140 seat and seat-belt retention bolts – run head down, threads up at 200 kHz – are case hardened to depths up to 0.020 inch and surface hardness of 40–45 HRC to impart both strength and toughness.

## Most Common Problems Encountered

Furnace atmosphere problems – often resulting in non-uniformity of case depth or sooting – are the common symptom when the furnace atmosphere is unstable. These problems are due to:

- Part cleanliness (oils, cleaning-compound residue, water, phosphate coatings)
- Air infiltration or products of combustion (air leaks, radiant-tube leaks)
- Zonal separation (temperature, atmosphere)
- Control-thermocouple placement
- Temperature uniformity (side-to-side)
- Temperature profile (down the length of the furnace)
- Atmosphere sampling tools (dew point, CO/CO$_2$/CH$_4$,

Table 1 Fastener Materials

| Category | Common Materials | Tensile Strength Ranges (ksi) | Typical Applications |
|---|---|---|---|
| Low carbon and boron steels | 1010, 1018, 1020, 1021, 10B21, 1117, 11L17, 12L14, 1215 | 60 | Machine screws, nuts, carriage bolts, and other fasteners without critical strength requirements |
| Mediumcarbonand boron steels | 10B30, 1038 or 10B38, 15B30, 15B35, 1041, 1042, 1045, 1050 | 105 – 150 | Bright cap screws, specialty fasteners for high-strength |
| Alloy steels | 4037, 4140 – 4145H, 50B35, 5115, 5141, 8637, 8740 | 170 - 190 | High-strength bolts, studs, nuts, cap screws |
| Stainless steels | 304, 316 | 70 – 100 | Accessory bolts and studs |
| Tool steels | L19 | 230 – 260 | Connecting Rod Bolts |
| Custom Alloys | Inconel 718, AerMet 100, | 220 - 300 | Connecting Rod Bolts |

oxygen probe)
- Sample port location and insertion depth
- Circulating fans (number, type, location, speed)

### Important Heat-Treat Considerations

For heat treaters, fastener properties such as strength, toughness and corrosion resistance are important, as is avoiding brittleness and of course cost.

Brittleness is always a concern, and in bolts it is defined as failure at stresses below the strength of the bolt material with little or no evidence of plastic deformation. Typically, fasteners are not brittle below 180 ksi ultimate tensile strength. Nearly all fasteners are considered ductile except some made from certain precipitation-hardening stainless grades (e.g., 15-5 Mo, 17-4 and 17-7).

Toughness is also an important feature of a fastener. It is the opposite of brittleness and gives you an idea of how much abuse a fastener will take without being damaged and eventually weakening – fatigue being a prime consideration. One way to “measure” toughness is by looking at the hardness rating of a fastener. The higher the number (Brinell, Rockwell), the harder the material is and the tougher (more resistant) it is to damage.

Fatigue usually doesn’t play a big part in grade 5 or 8 fasteners since most steels are good for 2 million to 10 million cycles. Almost all fastener fatigue failures are the result of improper (almost always too low) torque. Too low a torque will cause the fastener to pick up more load more often and eventually cycle it to failure. Lubricated threads significantly change the

Fig. 3. Typical load of fasteners entering a mesh-belt furnace

actual preload on the fastener and the risk of over-torque.

### Other Important Considerations

Good heat treating can only get us so far with respect to fastener quality and performance. Attention to design, manufacturing and application (service) issues are very necessary. Key items are the number of exposed threads (to avoid lowering ductility), geometry of the radius under the head (to avoid cracking), geometry of thread run-out (to avoid brittleness),

Fig. 4. Induction bolt-head draw system

rolling of threads after heat treatment (to increase fatigue life by an order of magnitude and avoid over-packed threads and tensile stresses), lubricity and clamp load.

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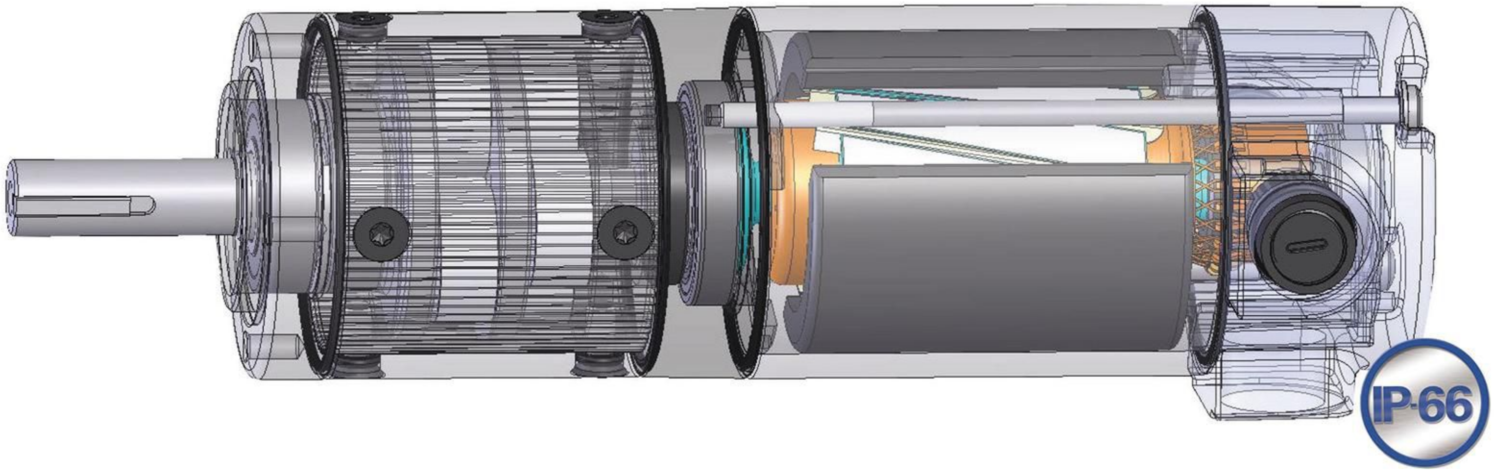
2. Sisson, Richard D., Heat Treatment of Steel Fasteners, FastenerTech 2007, Rosemount, IL.


IP-66

# Just What Do You Call Dirt on Mars, Earth?

The Doctor has always been curious about questions such as this one, which stimulate thought and promote an exchange of new ideas. Most, if not all, heat treaters – whether they like to admit it or not – enjoy the challenges that present themselves when confronted by a new material or the need to develop a new process recipe to achieve specified properties or results. Here, we will consider an example of doing just that. Let's learn more.

Fig. 1. Arno Bernard Knives: Bush Baby Series – Giraffe Bone, N690 steel (courtesy of CollectorKnives)

A question was asked of The Doctor as to the best heat-treatment practice to develop Rockwell hardness in the low 60s for a Böhler N690 material. Being somewhat unfamiliar with this particular grade and what products are commonly manufactured from it, it was natural to do an Internet search to glean some general insights followed by a more detailed investigation of what heat-treat recommendations were available from the manufacturer. In addition, it made good sense to reach out to several commercial heat treaters to learn from their experience. As a result, several heat-treat recipes were developed.

## Composition

Böhler N690 is classified as a stainless steel – more specifically, a martensitic chromium stainless steel with cobalt, molybdenum and vanadium additions (Table 1). The cobalt addition in particular is responsible for many of this alloy's most desirable properties. This material is intended for applications requiring high hardness in combination with good corrosion and wear resistance, along with excellent surface-finish and edge-retention characteristics in the finely ground and highly polished condition.

**Table 1. Selected materials for comparison to Böhler N690**

| Steel Name | N690 | VG-10 | 154CM | CPM154 |
|---|---|---|---|---|
| **Manufacturer** | **Böhler** | **Takefu** | **Crucible** | **Crucible** |
| **Form** | **Cast** | **Cast** | **Cast PM grade** | **PM grade** |
| **Composition** | | | | |
| Carbon | 1.08 | 1.00 | 1.05 | 1.05 |
| Chromium | 17.3 | 15.0 | 14.0 | 14.0 |
| Cobalt | 1.50 | 1.40 | - | - |
| Manganese | .40 | .50 | - | - |
| Molybdenum | 1.10 | 1.05 | 4.0 | 4.0 |
| Phosphorous | - | 0.03 | - | - |
| Silicon | .40 | - | - | - |
| Vanadium | .10 | .20 | - | - |

**Table 2. Mechanical properties at room temperature**

| Condition | Hardness |
|---|---|
| Annealed | 285 HB (maximum) |
| Hardened | 60-62 HRC |
| Hardened and tempered | 58-60 HRC |

## Applications

Knife blades are one of the most common uses of this grade. Other applications include surgical (cutting) instruments, rotary knives for the meat-processing industry, plate and knife-edge fulcrums, corrosion-resistant roller bearings, valve needles and pistons for refrigeration systems.

Many knives are made of 440C steel, given that this alloy is easy to re-sharpen and has an excellent price point and good general-duty performance. However, the ability to hold a sharp edge in demanding field use and a desire to improve overall performance drives knife manufacturers to investigate alternative steels. As such, literally dozens of types of steel are used to create knife blades. Some of the more popular material grades include 420HC, 1095, AUS-8, 18Cr13MoV, X50CrMoV15 (DIN 1.4116), 154CM, S30V, VG10, D2, Elmax®, CPM3V, ZDP189 and SM100. Another popular choice is Böhler N690 (Fig. 1).

Most knife makers use VG10 to compare the performance of N690 rather than 440C. N690 belongs to a class of materials of similar chemistry (Table 1), which are all available in sheet form to facilitate manufacturing (i.e., stamping of the material).

## Heat Treatment

Manufacturers typically provide general heat-treat instructions for their stainless steels with expected results in terms of microstructure and hardness (Table 2). Examples are:

- Annealing: 800-850°C (1470-1560°F). Resultant microstructure is ferrite and carbide.
- Hardening (furnace): 1030-1080°C (1885-1920°F) followed by rapid forced-air cooling or oil quenching (for thick sections). Tempering: 100-200°C (210-390°F). Resultant microstructure is martensite and carbide.

From this type of data, one can develop specific heat-treat recipes based on a given steel. For N690 knife blades this would be as follows:

1. Preheat to 650-705˚C (1200-1300˚F) and equalize (i.e., soak until parts and load are uniform in temperature, typically 15-30 minutes).
2. Heat to 815-870˚C (1500-1600˚F) and equalize (i.e., soak until parts and load are uniform in temperature, typically 10-20 minutes).
3. Heat to 1060˚C (1940˚F) and soak for 15-30 minutes. Do not over soak; alternate hardening temperature: 1070˚C (1960˚F).
4. Quench in air using rapid forced convection cooling.
5. Deep freeze (for retained austenite conversion) at -84˚C (-120˚F) for a minimum of 2 hours with 3-4 hours recommended. The minimum treatment is -70˚C (-95˚F) with a soak of 3-4 hours at temperature; -185˚C (-300˚F) is also permitted; deep freeze within 30 minutes of quench completion.
6. Temper immediately upon return to room temperature. Double tempering is required to achieve desired hardness and maximize stability. The minimum time at temperature is 2 hours.

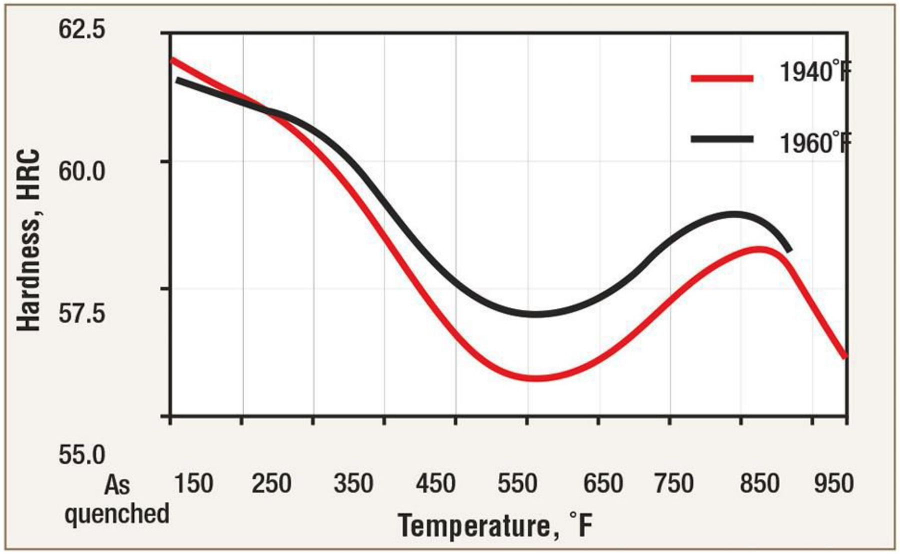


Fig. 2. Tempering curves for Böhler N690a for indicated hardening temperatures. Note: [a]Based on a sample size of 20 mm (0.5 inch) diameter, double tempering for 1 hour at temperature.

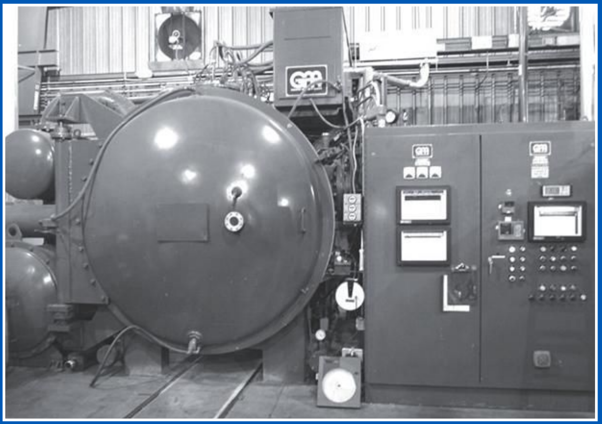
Hardness results vary somewhat with the choice of austenitizing temperature and tempering temperature (Fig. 2). For example, to achieve 60-61 HRC, one would double temper N690 austenitized at 1060°C (1940°F) for two hours at a temperature of 99-135°C (210-275°F). By contrast, using an austenitizing temperature of 1070°C (1960°F), one finds that a tempering temperature of 125-150°C (255-300°F) is required to achieve the same result.

## Summary

What is important for the heat treater to focus on is the process of determining the best heat-treatment practice for a given steel. This involves understanding the end-use application, researching the material properties, and then creating an optimized recipe based on input from the steel supplier and the experience of the heat-treat community.

## References

1. Herring, Daniel H., Vacuum Heat Treatment, BNP Media, 2012
2. Böhler N690 Technical Data Sheet DE 12.2007 EM-WS
3. Wikipedia (wikipedia.org)



# A Curious Case of Variable Part Hardness

The Doctor was recently called in to investigate a variable-hardness issue involving rotary cutting dies. The process used to diagnose what was wrong and how to correct the problem offers a number of important lessons. Let's learn more.

## The Problem

Areas of variable hardness (i.e., hard and soft spots) were identified along the blade length (i.e., cut edge) of 4150 rotary cutting dies (Fig. 1). The hardness problem had been occurring, off and on, for a number of years. Some parts came out very good, and others did not. No apparent pattern existed. For example, two mirror-image dies (same size, same material, same manufacturing method, same production time frame) yielded one good and one bad part.

The definition of "hard" and "soft" is the response of material to hand sharpening using carbide tools. Soft areas are where the tools "dig in." The steel will form burrs and produce a serrated surface, which is virtually impossible to sharpen. Hard areas are those in which the tool "slides across" with minimal resistance. If the part is too hard, however, cut edges are brittle, causing cracks and resulting in scrap parts. The targeted hardness for the dies is 56-62 HRC. The part diameters vary from 50-200 mm (2-8 inches), and the length ranges from 140-635 mm (5.5-25 inches).

The heat-treatment process involved induction hardening and polymer quenching (Fig. 2) followed by induction tempering. After air cooling, the dies were visually inspected for cracks, a hardness check was done and life was tracked throughout the product's service life in the field.

## Variables

After thorough analysis, the main focus areas appeared to be: material chemistry (higher-than-normal certified sulfur levels were found) part geometry (given the raised areas on the dies); austenitizing temperature (not measured) and time; quench parameters (polymer concentration, flow, pressure, spray angle, spray area); reheating of a localized area after initial quenching; and tempering temperature (not measured) and time.

Fig. 1. Typical rotary die

Action Plan

The following action plan was established to investigate the problem.

- Obtain a "good" and "bad" sample for metallurgical analysis.
- Monitor the induction heating parameters (power/frequency settings, scan speed, dwell time, etc.) for variation (part-to-part, operator-to-operator).
- Determine hardening and tempering temperature (via Tempil® paint and infrared thermography).
- Check the quench spray pattern.
- Determine pressure/flow characteristics during quench.
- Send parts out for neutral (furnace) hardening, oil quench and oven temper, then check for soft/hard spots.
- Analyze the parts in the lab as well

## Metallurgical Analysis Work

The surface hardness of the "good" dies averaged 61 HRC. On the "bad" dies, a hardness difference of approximately 1.5 HRC was found between the hard and soft blade areas.

The microstructure of the hard area of the blade consisted of tempered martensite with some ferrite observed. The microstructure in the soft area revealed slightly more ferrite to be present.

The depth of hardening (visually) in the hard area was 2.39-2.41 mm (0.094-0.095 inch) beneath the surface, while the depth of hardening in the soft area was 2.34-2.36 mm (0.092-0.093 inch). The height of the blade above the surface on the dies was approximately 1.32 mm (0.052 inch).

The conclusion reached was that optimizing the process parameters (adequate soak time at temperature, proper quench concentration, spray pattern, quench pressure and flow pattern) would eliminate this hardness differential.

## Field Analysis Work

The induction equipment being used was approximately 10 years old; a 150-kW, 10-kHz unit running 218-291 volts, one phase, 7,260-7,424 amps. The range of adjustment was as follows.

1. Scan distance: 0-915 mm (0-36 inches)
2. Scan speed:0-150 mm/second (0-6 inches/ second)
3. Dwell time:0-99.99 seconds
4. Power level:0-120%
5. Quench:on/off
6. Rotation:0-350 rpm

Concerns

The questions raised prior to running the process were as follows.

- Is the power level fluctuating?
- Is the part rotation proper?
- What is the (air) gap between the quench ring and the die? Is it adequate?
- What are the characteristics of the quench?
  - Is the quench duration too long or too short?
  - What is the effectiveness of quench (i.e., analysis of quench variables – supply pressure, flow, polymer concentration, etc.)?
  - Spray quench versus dip quench – which is better?
  - Is 8% the proper variable polymer concentration?
- Observations

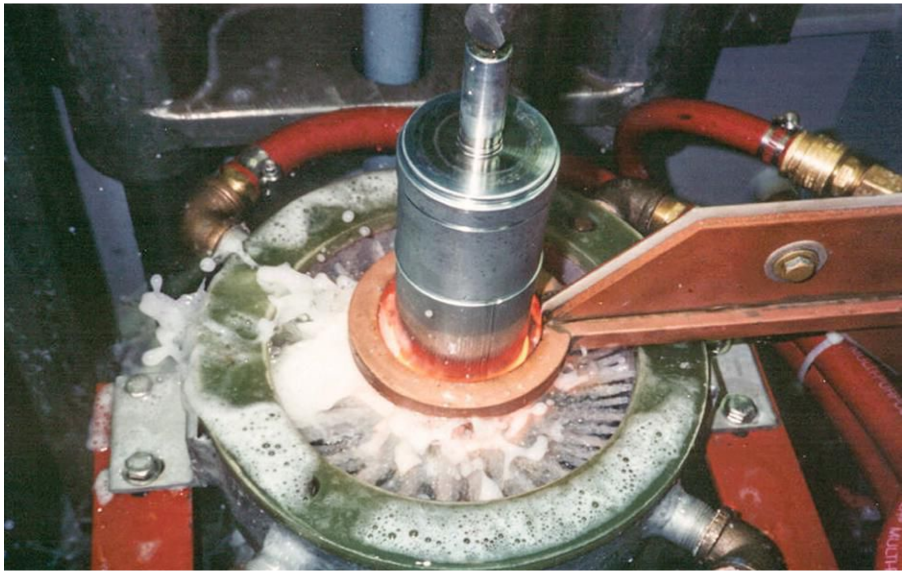


Fig. 2. Induction hardening process in action after process optimization

- An abbreviated outline follows. Full details are available online.
- Output power regulation
  - Constant output power is required.
  - The output power limit was not reached.
- Austenitizing temperature
  - Temperature at existing settings varies.
  - Decrease scan speed to increase scan time.
- Part wobble
  - Minor but noticeable.
- Polymer quench
  - Less backwash and more cascading are required.

  - Refractometer readings were being improperly performed.
  - Some quench holes were plugged.
- Residual part heat
  - The amount of residual heat present on the part varied.
  - Variation in temperature due to part mass.
- Tempering
  - Recipes were not being used for tempering.
- Barber-pole effect
  - Slight (visually)
  - Increase RPM to reduce effect
- Other
  - Water overflows onto shop floor.

## The Findings

The root cause of the problem was poor quenching. Some of the quench holes were partially plugged with mineral deposits. The polymer being used had a definite odor to it, and the quench tank itself had a significant amount of scale buildup. The heat exchangers were also suspected of having mineral-deposit buildup and were in need of cleaning (i.e., acid flushing).

During the course of this investigation all of the process parameters were better understood, and the process recipe was optimized.

Conventional oven tempering was also recommended over induction tempering. The dies should be immediately placed in a tempering oven running at 150˚C (300°F) allowing 1.5 hours (minimum) heat-up time and one hour per inch for soak.

## Summary

The lessons learned, through a combination of laboratory and shop-floor testing, show how to best solve heat-treat problems.

## References

1. Herring, Daniel H., Atmosphere Heat Treatment, Volume I, 2014

# Decarburization

There is a group of recurring questions asked of The Doctor, all of which are centered around: "How much surface oxidation is allowable on a steel part?" The latest was an innocent enough request for "a 'decarburization' chart (or article), which shows the effect on steel, if normalized, in a non-inert-gas environment." The fulfillment of this request provides some valuable information for the heat treater. Let's learn more.

While the question appears straightforward, there are a number of important issues that arise from it, and some interpretation is needed to fully understand what is being asked. For example, what type of steel is involved, in what form is it being purchased and what is the manufacturing sequence used to create the component parts? From a metallurgical perspective, are we talking about total or partial decarburization? How will it be measured, and what is the application end use of the product so that an evaluation can be made of the impact of decarburization on the design?

Decarburization varies with material grade, hardenability, furnace atmosphere, carbon potential and the type of heat-treatment process being performed (e.g., temperature, time). Decarburized parts exhibit lower (surface) hardness, reduced wear resistance and lower fatigue life, which affects their service life. One example is an automotive manufacturer whose steering columns were found to be loosening on the assembly line due to the presence of a 0.025- to 0.075-mm (0.001- to 0.003-inch) partial decarburization layer on the shaft retaining ring.

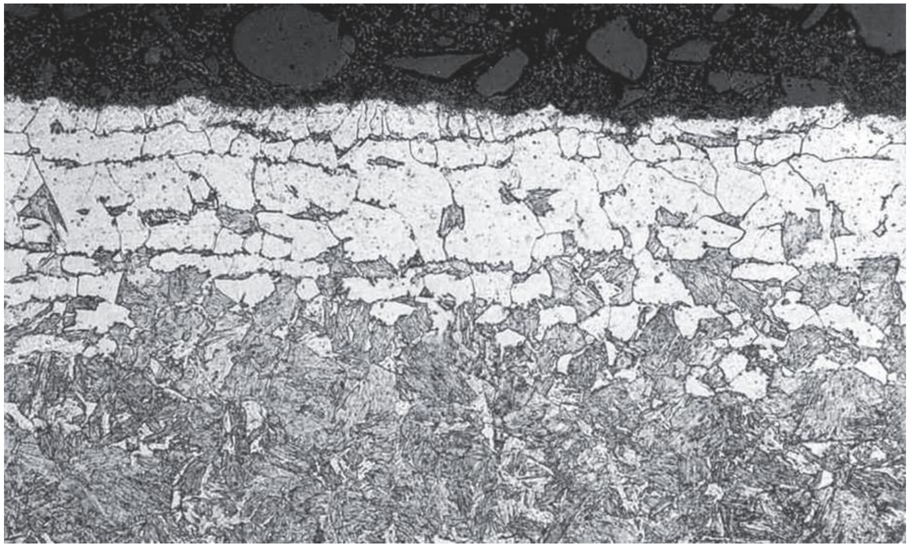


Fig. 1. Decarburized steel microstructure (free-ferrite is white)

If one simply refers to AMS 2759/1 or /2,[2,3] four part types are defined that, in turn, dictate the class of atmosphere permitted or prohibited when heating parts

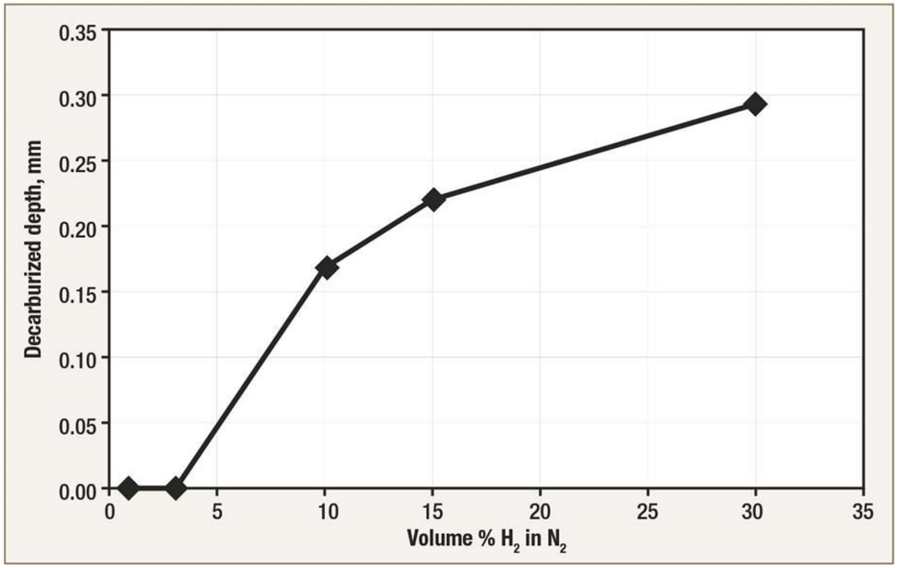


Fig. 2. Surface decarburization as a function of hydrogen content of the atmosphere[6]

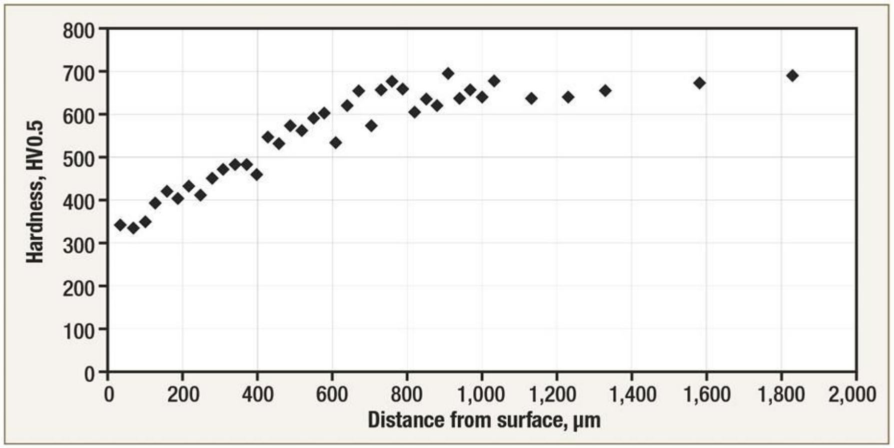


Fig. 3. Representative hardness profile for 300M sample 2, in air[7]

above 677˚C (1250˚F). The type of starting surface (e.g., hot-finished, cold-drawn) and the amount of metal to be removed by partial or finished machining, whether greater or less than 0.51 mm (0.020 inch), are the important criteria. These specifications go further by discussing surface contamination and providing limits on such items as partial decarburization, ≤ 0.13 mm (0.005 inch), and intergranular attack, ≤ 0.018 mm (0.0007 inch), as well as defining the method of measurement and rejection criteria.

Being told what to do is one thing, but understanding why it must be done is another, which was the real question being asked here.

## A Little Theory[4,5]

Total decarburization (aka type-1 decarburization) is the depth to which the surface microstructure is free-ferrite; that is, the depth to which there has been a 100% loss of carbon (Fig. 1). Partial decarburization (aka type-2 or type-3 decarburization) is that depth from the surface where some loss (greater than or less than 50% respectively) of carbon has occurred, but there is no measurable depth of complete decarburization.

The loss of carbon from the near surface has been found to occur above 700˚C (1290˚F) when the furnace atmosphere contains carbon dioxide, water vapor, oxygen and hydrogen (Fig. 2).

Carbon present in the steel will interact with the furnace atmosphere, and it will leave the surface in a gaseous phase under the right conditions. This results in a change in concentration causing migration of carbon from the interior to the surface, which continues until a balance has been re-established, thus creating a maximum depth of decarburization. Depending on whether the steel is between the lower ($Ac_1$) and upper critical ($Ac_3$) temperatures or above the upper critical, carbon diffusion rates vary. Temperature and composition are the principal factors involved, and their influence varies depending on the process (e.g., annealing, normalizing) being performed.

## A Little Deeper[7]

As stated, decarburization begins to occur as the rate of carbon at the surface decreases due to its reaction with oxygen as this reaction exceeds the growth rate of scale (iron-oxide) formation. The scale contributes to the decarburization depth (e.g., Equation 1 being typical).

$$\mathrm{FeO} + \mathrm{C}_{\mathrm{Fe}} \rightarrow \mathrm{Fe} + \mathrm{CO(g)} \qquad (1)$$

Decarburization is not only dependent on the presence of oxygen but also the interaction with other oxidizing gases in the atmosphere, most notably water vapor and carbon dioxide. In a controlled trial,[7] samples were decarburized and results measured for both low oxygen-level atmospheres and air (Table 1). The loss of hardness was carefully documented (Fig. 3). These tests found the maximum decarburization depth to be around 0.51 mm (0.020 inch), which correlates well with the AMS information.

## Summary

The choice of the correct furnace atmosphere, temperature and to a lesser extent time at temperature are important variables to offset the effect of decarburization.

As it turned out in this particular instance, the question involved the need for either nitrogen as a blanketing atmosphere during normalizing at 955˚C (1750˚F) or if the process could be done in a gas-fired box furnace with the work protected by the products of combustion. Since the minimum stock removal was 1mm (0.039 inch) and the soak times were four hours

**Table 1. Selected decarburization results for 300M, in air[7]**

| Sample number | Austenitizing temperature,[a] °C (°F) | Ferrite depth, µm (visual) | Scale depth,[b] µm | Surface hardness, HV0.5 | Subsurface hardness, HV0.5 | Total decarburization depth, µm |
|---|---|---|---|---|---|---|
| 1 | 800 (1472) | 46.3 | 114 | 327 | 710 | 500 |
| 2 | 900 (1652) | 136.1 | 85 | 345 | 675 | 825 |
| 3 | 1000 (1832) | 208.8 | 112 | 367 | 710 | 995 |

Notes: a. Soak time at temperature was two hours.
b. Scale refers to the iron-oxide layer present on the steel surface.

or less, the deleterious effects of decarburization could be avoided with either atmosphere. Conducting trials using a furnace running the process cycle(s), however, is always strongly recommended to determine the actual decarburization levels.

## References

1. Herring, Daniel H., Atmosphere Heat Treatment, Volume II, BNP Media, 2015
2. AMS 2759/1, "Heat Treatment of Carbon and Low-Alloy Steel Parts Minimum Tensile Strength Below 220 ksi (1517 MPa)," SAE International, (Rev. E), 2009
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5. SGS MSi (www.msitesting.com)
6. "Furnace Atmospheres No. 8: Sintering of Steels," Special Edition Booklet, Linde 2011
7. Mayott, Steven W., "Analysis of the Effects of Reduced Oxygen Atmospheres on the Decarburization Depths of 300M Alloy Steel," Thesis, Rensselaer Polytechnic Institute, 2010

# A Curious Case of Carbon Pickup

Over the years, instances of carbon pickup resulting in subtle hardness variations in 0.30% and 0.40% carbon steels after neutral hardening in an endothermic gas atmosphere have been reported and often result in rework or scrapped loads. It's time to discuss what causes this and what steps can be taken to prevent it. Let's learn more.

## A Real-World Example

Take a recent case of the heat treatment of extremely thin parts, approximately 1 mm (0.040 inch) thick, manufactured from 4130 strip stock. The load consisted of 740 pieces, which were well spread out in a basket and covered with a screen to retain heat during transfer to quench. The net load weight was only 1 kg (2.2 pounds). The parts were run in an integral-quench furnace at 870˚C (1600˚F), held for two hours at temperature with an oxygen probe setpoint of 0.30% C and then oil quenched.

During microstructural inspection for decarburization, the parts were found to have picked up carbon at the surface. A thin "case" was developed, which was confirmed by microhardness testing (Table 1) and metallurgical analysis (Fig. 1). Optical microscopy revealed subtle differences in the martensitic microstructure between the near surface, within the first 0.20 mm (0.008 inch), and the core.

**Table 1. 4130 hardness values**

| Depth below surface, mm (inches) | Hardness, (HRC)[a] | Hardness increase above core, (HRC) |
|---|---|---|
| 0.050 (0.002) | 51.8 | 1.1 |
| 0.075 (0.003) | 53.1 | 2.4 |
| 0.100 (0.004) | 52.6 | 1.9 |
| 0.125 (0.005) | 52.0 | 3.2 |
| 0.150 (0.006) | 52.1 | 1.4 |
| 0.175 (0.007) | 51.7 | 1.0 |
| 0.200 (0.008) | 51.8 | 1.1 |
| Core | 50.7 | - |

Notes: [a] Converted from $HK_{500}$

The most likely explanation advanced was that the component part picked up additional carbon from the furnace atmosphere. A review of the furnace charts, however, confirmed that the carbon

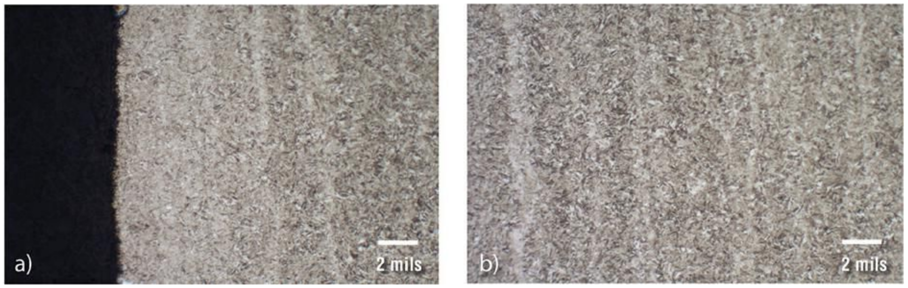

Fig. 1. 4130 microstructure; (a) Surface (200x, 3% Nital); (b) Core (200x, 3% Nital)

setpoint was maintained throughout the cycle and that the austenitizing time and temperature were correct. The temperature uniformity of the furnace was checked and found to be within ±5.5˚C (±10˚F). The endothermic gas atmosphere had remained consistent as well with an incoming dew point of +6˚C (+43˚F).

While the calculation of carbon from the millivolt signal of an oxygen probe depends on a multitude of factors, including the age and condition of the probe and burn-off history, a variance of 5 mV (a conservative estimate) equates to only a 0.03% carbon change given the above process parameters.

If we consider hardness variation as a function of carbon content in the material, we find that a nominal 0.30% C steel quenched to 99.9% martensite (Fig. 2) would have a resulting hardness of 50 HRC, while a 0.32% C would be 51 HRC and a 0.34% C would be 52 HRC. Hence, a subtle variation in the carbon content of the steel itself could explain this phenomenon.

## Sources of Hardness Variation

In order to fully investigate a hardness variation such as the one reported, we must consider all of the factors that may have been involved.

### Chemical Composition/Variations in Carbon Content

Variations in steel composition (and resultant hardenability) can occur in component parts. Each steel type has an acceptable range for each of the elements it contains. In the case of 4130 material, the range of allowable carbon is 0.28-0.33%. Variations in carbon can arise from micro-chemical segregation effects within a given heat, heat-to-heat variation in carbon content and from carbon pickup or decarburization experienced during the heat-treatment cycles. Relying solely on the material certification sheet may be misleading. It is always best to analyze the material in question so that the actual carbon content can be used in predicting results.

### Underheating

This can be due to insufficient temperature and/or insufficient time at temperature. The result is that the component or some region within a component never exceeds the upper critical temperature on heating for a sufficient enough time during the austenitizing portion of the hardening cycle. For example, the lower critical temperature on heating ($Ac_1$) for 4130 steel is approximately 749˚C (1380˚F), while the upper critical temperature ($Ac_3$) is approximately 802˚C (1475˚F). The normal austenitizing temperature specified for this grade is 870˚C (1600˚F). If the austenitizing is insufficient, portions or all of the cross section of the part will not be martensitic upon quenching.

## Furnace Loading

In an effort to maximize productivity, we run the risk of overloading the furnace or, in the case of certain continuous furnaces, piling parts atop one another in large clumps. In these instances, there is a high probability that some of the parts will be underheated. One way to know this is happening is to have a database of microstructures (and corresponding hardness values) that represent properly austenitized and underheated product based on empirical data gathered over time or designed experiments.

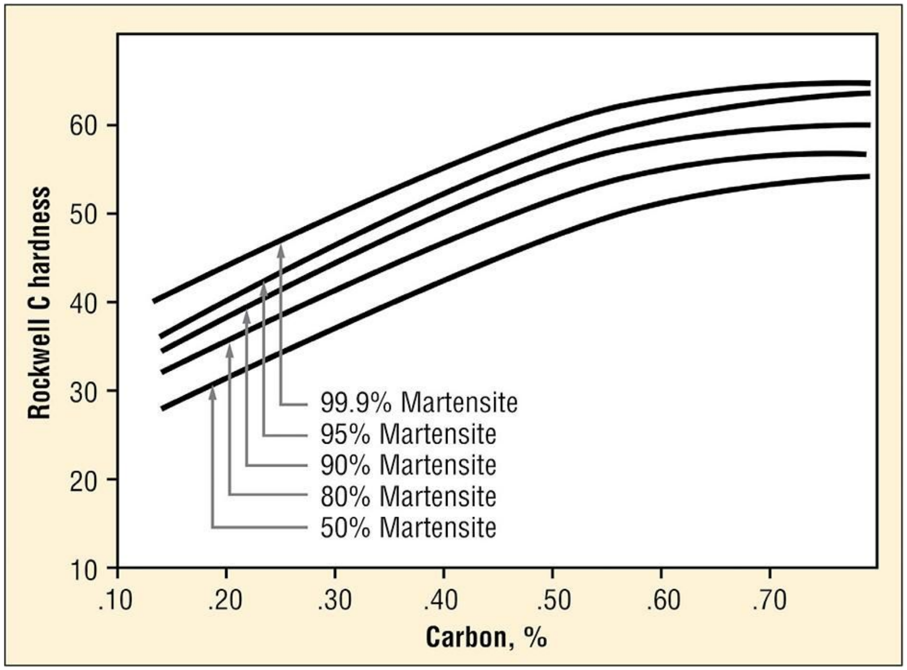

Fig. 2. Hardness variation as a function of carbon content[3]

## Overheating

Exceeding the recommended austenitizing temperature for a given steel is never a good practice, especially for steels that have carbon levels where significant amounts of retained austenite may form in the hardened microstructures. Retained austenite is softer than martensite, and increasing amounts will result in lower hardness values. Excessively high temperatures can also promote grain growth. If this occurs prior to quenching, the hardenability will increase and the resultant microstructures and hardness levels will vary accordingly. This is particularly important because of current emphasis on increased throughput by reducing cycle times through the use of higher austenitizing temperatures.

### Transfer Time from Heat to Quench

We need to consider the upper critical temperature on cooling ($Ar^3$) and the lower critical temperature on cooling ($Ar^1$) as well as the TTT (Time-Temperature-Transformation) and/or CCT (Continuous Cooling Transformation) diagrams. These will provide guidelines on the temperatures that must be maintained prior to quenching. The cooling rate determines if a fully martensitic microstructure will develop and the corresponding hardness levels that will occur.

If the components are held in the $Ar_3$ – $Ar_1$ inter-critical range or cooled insufficiently through it, pro-eutectoid ferrite will form. If the cooling rates are insufficient, various amounts and combinations of non-martensitic transformation products (NMTPs) with correspondingly lower hardness levels will result. Figure 2 is an invaluable tool for estimating the relative hardness that can result, and one sees that for a given carbon value, say 0.30% C, the hardness can vary from 50 HRC (99.9% martensite) to 37 HRC (50% martensite).

## Summing Up

The challenge in talking about subtle changes in hardness is that the variability can be induced by so many factors. For every instance discussed here, the reader can likely think of examples from their experience that were caused by other factors. It is the discussion that is important, however. It brings to light the fact that, whether large or small, changes in hardness need to be explored and the root cause determined so that our processes are kept under control

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# Surface White-Layer Formation in Vacuum Oil Quenching

The news is full of reports on the role of Russia in American politics and society. After years of speculation, The Doctor has found the answer to a seldom seen and often confusing, yet fascinating, problem in of all places Soviet science and good old-fashioned modern-day research. Let's learn more.

## The Problem

Perhaps a half-dozen times over the course of The Doctor's career have vacuum furnace users reported the presence of a white layer on the surface and near-surface of steel and stainless steel component parts after vacuum hardening and/or carburizing followed by vacuum oil quenching (Fig 1). This white layer is most often mischaracterized as surface decarburization.

The phenomenon is far from common and, as it turns out, is not new. In point of fact, it was investigated decades ago and even documented,[1] but the explanation as with many things in Soviet science was not readily accessible to the greater metallurgical community. In these past investigations, it was found that these white layers have much higher carbon content, more carbide-forming elements and higher levels of retained austenite than the bulk microstructure of the steel.

In testing conducted both then and now, white layers of 0.0002-0.0007 inches (5-18 mm) thick were produced and analyzed. Chemical analysis showed a high carbon content in these layers directly related to carbon saturation of the surface zone of the steel. Originally believed to be related to the process of oil quenching, it is now found to be due to oil vapors present in the vacuum environment.

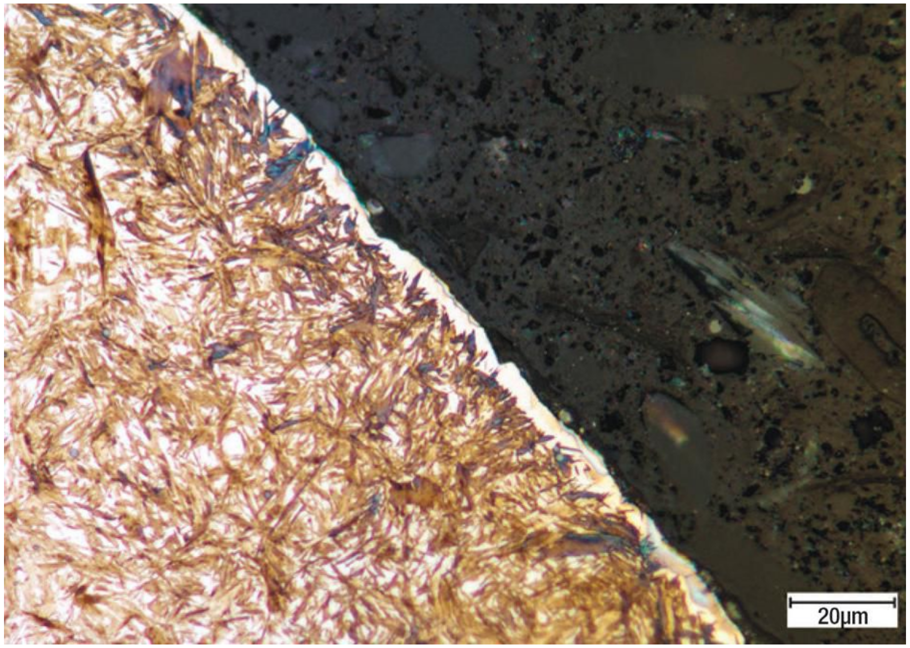

Fig. 1. Example of white-layer formation (500X, 2% Nital)

## The Phenomenon

It was previously established[2] that these white layers were always found after oil quenching, but when quenching from 1400-1650°F (750-900°C) these layers are very thin and cannot be easily identified without very careful sample preparation and surface-analysis techniques. They are more readily identified after quenching from above 1800°F (1000°C). Over the years, the phenomenon has also been seen in vacuum furnaces on loads that are gas cooled above the oil.

A very thin surface layer forms when the temperature of the metal surface is high enough. Metallographic examination revealed a zone of cementite-austenite on the surface of the carburized layer.[1] The thickness of this layer is dependent on temperature, cooling rate and/or time in contact with the cooling medium above a minimum reaction temperature. The layer can be uniform, even on surfaces of complex configuration, but is most often sporadic in nature along the surface – within a given part and from part to part throughout the load.

## The Investigation

The white-layer phenomenon occurs in both vacuum furnaces with and without vacuum-sealed inner doors (Fig. 2). Tests were conducted to more accurately determine the root cause of this uncontrolled carburization (and the resultant transfer of carbon or carbon-based compounds into the vacuum environment) and subsequently their reaction with hot parts to form the observed white layer. A furnace with a vacuum-sealed door was used in the testing.

A thorough investigation of the variables that might contribute to the formation of this white layer on carburized 5120 steel was undertaken. It included:

- Checking the tightness (leak rate) of the furnace
- Checking the operation and functionality of the carburizing system
- Looking for the presence of quench-oil vapor in the hot zone
- Checking work transport sequence and oil-quench chamber

It is well known[4] that a large leak can cause ingress of air (oxygen) into the vacuum furnace chamber. If the temperature is high enough when this occurs, a reaction will take place between graphite and air that results in the formation of carbon monoxide, a carburizing gas. The leak rate of the test furnace was checked and found to be within acceptable limits (<20 microns/hour).

Leakage in the carburizing gas mixture injection system would be another source of carburizing gas in the vacuum furnace. As part of this test program, mass-flow controller functionality and control/

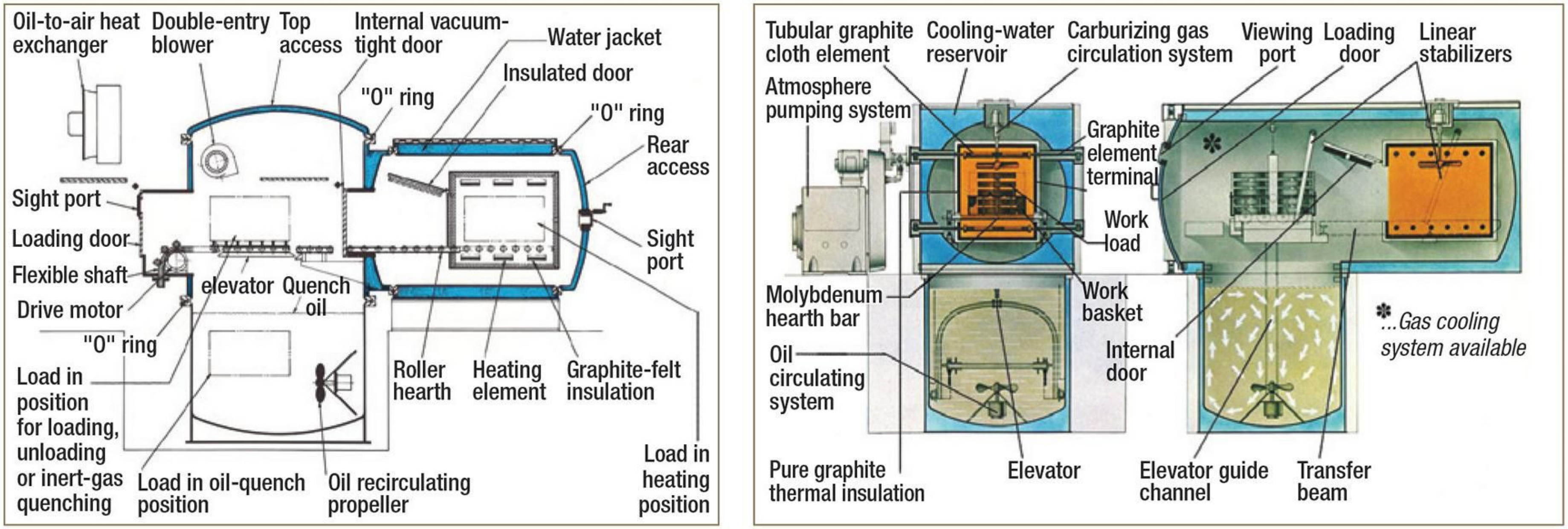

Fig. 2. Two-chamber vacuum oil-quench furnace cross-section designs: (left) with vacuum internal door; (right) without vacuum door (courtesy of C.I. Hayes)

shutoff valves were verified to be operating properly, which eliminated them as a potential cause of the uncontrolled carburizing problem.

Subsequent actions then focused on the possible impact of the quench oil and more specifically the oil vapor. The entire load transport process from the heating chamber to immersion in oil was thoroughly and systematically analyzed and included: the sequence of load transport from the heating chamber into the quenching chamber; the sequence of internal door opening and closing; vacuum level during load transportation for quenching; the moment of backfill (with nitrogen) and gas dispersion during the backfill process in the quenching chamber; pressure above the oil; load immersion rate; oil agitator activation and rotational speed and quench-oil temperature.

As a result of the optimization of these factors, the intensity and quantity of the white layer was reduced and its presence limited to isolated areas in the microstructure, but it was not completely eliminated. The next step was to change the quench oil knowing that different oils have different vapor pressures (and the ability to produce varying amounts of oil vapors). Oil from a different supplier was used. Further testing did not solve the problem. The white layer still appeared occasionally but perhaps to a slightly lesser degree.

Unable to find a definitive solution to the problem during load transfer and in the quench chamber itself, the focus then turned to the heating chamber, particularly to that portion of the cycle where the temperature is lowered to final hardening temperature (the drop temperature stage in carburizing) and load stabilization just before load transfer to the quench.

It was discovered that, in some recipe settings, both the furnace and quench chambers were being pumped simultaneously by the same pumping system. In this arrangement both chambers are connected, and, theoretically, migration of oil vapors from the oil chamber to the heating chamber is possible. The test was repeated with a revised approach excluding simultaneous pumping of the two chambers. When this was done, the white layer disappeared completely. The tests were repeated multiple times with the same positive result, revealing that oil migration into the heating chamber was the root cause of the observed white-layer phenomenon.

## The Solution

The root cause of surface and near-surface white-layer formation is uncontrolled carburizing due to quench-oil vapor. This may occur in different stages of the process: during soak periods in the hot zone before transfer to the quench; during cooling of a hot load over the quench oil; and during load immersion into the oil. Which of these stages has the most influence on white-layer formation remains an open question.

In vacuum furnaces equipped with oil-quench tanks, the operational and process parameters should be set up and configured in such a way as to minimize the possibility of contact of quench-oil vapors with the hot load and to reduce the possibility of quench-oil vapor transfer to the heating chamber.

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# High-Temperature Oxidation – A Case Study

The effect of hydrogen on aluminum alloys can manifest itself in a phenomenon known as high-temperature oxidation (HTO), also known as high-temperature deterioration (HTD). A case study involving 2024 and 7075 aluminum aerospace fasteners in which hydrogen-induced damage was found after solution heat-treatment and aging operations serves as an excellent example. Let's learn more.

In simplest terms, HTO (Fig. 1) is a form of hydrogen diffusion that affects surface layers of a part during elevated-temperature treatment. This condition is often due to moisture contamination in the furnace atmosphere and is sometimes aggravated by sulfur or other furnace refractory contamination. The most common manifestation of HTO is surface blistering (Fig. 2). However, it may also appear in the form of surface voids or internal discontinuities. The symptoms of HTO are nearly identical to those of high gas content in ingots due to improper mill practices. The 7xxx-series alloys are the most susceptible followed by the 2xxx alloys.[2]

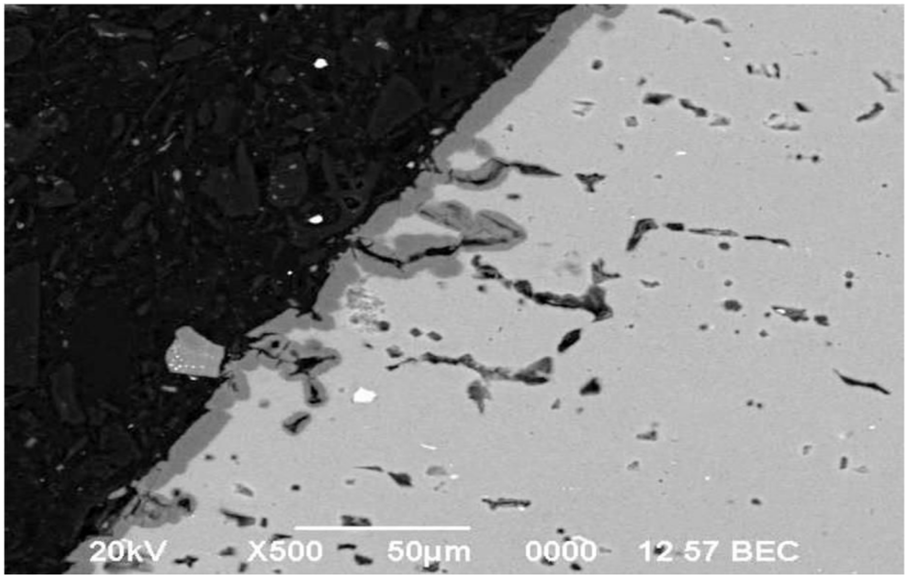

Fig. 1. High-temperature oxidation manifesting itself as surface and subsurface porosity

## Background

Aerospace fasteners were being manufactured from 2024 and 7075 wrought aluminum bar stock. After heat treatment and during assembly, the hex end of multiple fasteners fractured and separated from the body close to the specified torque level. The quality department quarantined the parts in question. Visually, some surface pitting could be observed. Samples were analyzed, both in-house as well as by an independent

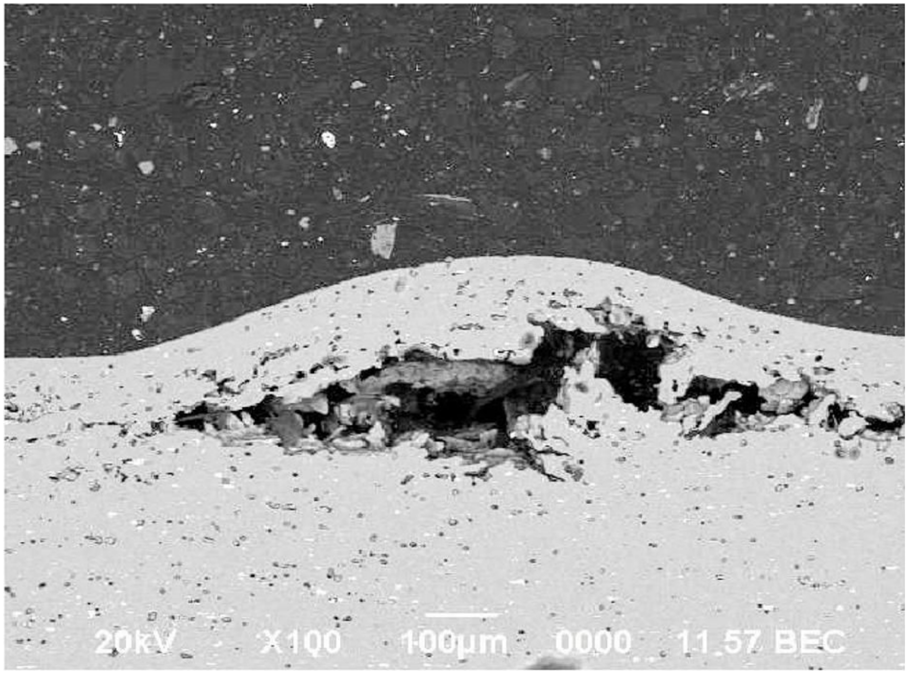

Fig. 2. Backscatter electron image of the blistered surface of the part showing a large blister with subsurface porosity

laboratory to identify the root cause of failure.

The heat treatment of the two different fastener materials involved heating to 465˚C (870˚F) and 495˚C (920˚F), respectively, in an electrically heated air-circulation oven and holding for 75 minutes. The temperature uniformity of the oven was ±5.5˚C (±10˚F).

After soaking at temperature, a trap door opened beneath the single-basket load, which was then tilted so that the fasteners tumbled out of the basket into a chute that led to a water quench tank located below the oven. The loose parts were then collected in a perforated basket located beneath the chute. The water in the quench tank was kept at 32˚C (90˚F) via a heat exchanger before the introduction of parts. It was observed that the water was rising to 60˚C (140˚F) during the quenching operation.

The quench chute itself included an area that consisted of fine-mesh screening located around the circumference and extended for about 2 feet below the water level so as to allow water in the tank to move freely through the quench-chute area. A pipe with a series of holes designed to spray water across the top surface of the tank was located just at the water line in order to prevent steam from rising and entering the heating chamber as the parts were dropped from above.

## Laboratory Investigative Work

Samples were gathered in the field and analyzed both as received using stereomicroscopy and after preparation by both optical and scanning electron microscopy. Samples were cut using a precision cutoff machine, and metallographic mounts were prepared in accordance with ASTM E3 using a conductive mounting material suitable for use in the SEM.

The scanning electron microscope was equipped with energy dispersive spectroscopy (EDS) capability. The EDS was capable of characterization of the near surface using both secondary electron imaging and backscatter electron imaging for evaluation of compositional variation (Fig. 3). Results from this analysis were documented through acquisition of digital photography, including EDS results of the corresponding spectra for each area analyzed. Multiple locations on each sample were evaluated to determine the degree of consistency in composition and morphology.

The result of the analysis was that the near-surface condition observed on a number of suspect parts was that of HTO. The presence of the subsurface porosity induced by HTO required the parts in quarantine to be scrapped and not used for production.

## Field Investigative Work

Based on laboratory findings, water vapor present in the heating chamber was suspected to be the root cause. If water vapor rose from the quench-tank chute, it could enter the furnace through the trap door in the heating chamber. A high-humidity atmosphere was suspected as the source of hydrogen, which subsequently entered the part surface during the soak period.

Close inspection of the quench-tank area revealed

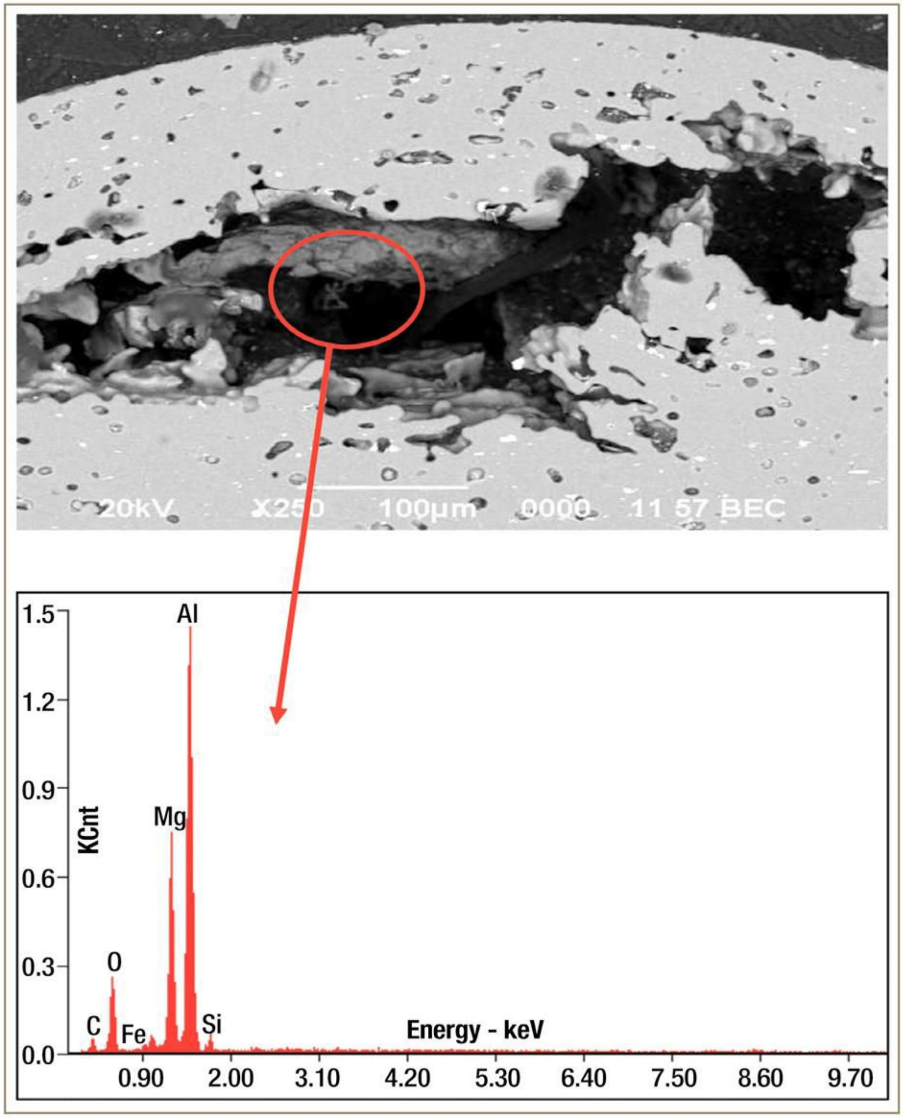

Fig. 3. Representative spectra from subsurface showing primarily aluminum (Al) and magnesium (Mg) but high levels of oxygen (O), silicon (Si) and carbon (C)

two distinct problems. First, the exiting spray located at the water line of the tank was blocked, which limited spray and, in some cases, prevented spray from coming out of the holes. Disassembly found the pipe and holes to indeed be partially or fully clogged by mineral deposits and debris. Well water was being used to supply the system. Although not analyzed, it is a known source of such mineral deposits.

Second, the fine-mesh screen used to allow flow of water from the tank to the quench-chute area was completely blocked. This created a localized temperature rise in the chute area, creating steam that then rose into the oven proper. All components were cleaned, and a preventive-maintenance schedule was established. The problem did not reoccur.

## Conclusion

Case studies are invaluable and offer us the opportunity to share practical lessons learned.

Hydrogen-induced damage in fasteners is an industry concern most often negated by bake-out cycles. Phenomena such as the case of high-temperature oxidation, however, underscore the need for the heat treater to be ever diligent.

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# Processing Magnesium: The Importance of Proper Process Control

Anyone who has ever seen the bright, blinding light of burning magnesium will never forget it. Here is a case of where a combination of process and equipment decisions resulted in catastrophic consequences –and one that will serve as a valuable reminder of how important our safety systems are. Fortunately, no one was injured. The drying oven involved, however, was a complete loss. Let's learn more.

## The Drying Process

The oven in question was a typical truck oven produced by a reputable company. The unit was designed to process a 19,090-kg (44,000-pound) load of magnesium ingots (Fig. 1). The objective was to dry the ingots prior to melting. Otherwise, rapid vaporization of the latent moisture in the ingots will cause an explosive reaction when the damp metal meets the molten magnesium at 600°C (1100°F).

The process to ensure dryness was to heat the ingots to 204°C (400°F), well above the boiling temperature of any latent water in the ingots. In order to accelerate the heating process, however, the oven was set at 426°C (800°F). This "heat head" greatly reduced the overall cycle time, especially when bringing the ingots up to the process temperature.

Fig. 1. Typical load of magnesium ingots[2]

Some facts about magnesium are important here. In addition to being highly flammable, magnesium burns at a temperature of approximately 3100°C (5610°F), and the auto-ignition temperature of magnesium is 473°C (883°F).

## Oven Heating/Recirculation System

The drying oven used a common combination airflow. The ingots were loaded on top of a traveling load car that rolled inside the oven, after which the doors were closed, and the heating/recirculation system was turned on. The heated air was then delivered from supply ducts located on both sides of the heating chamber, passed through the load of ingots and then returned to the heating/recirculation system on the oven roof (Fig. 2).

## Control System and Thermocouple Locations

As in many industrial ovens, a type-J control thermocouple located in the recirculated air stream was used. The thermocouple was wired to a control instrument that sensed the air temperature and adjusted the burner output higher or lower to maintain the desired setpoint temperature of the heated air. It also includes a separate high-limit thermocouple and instrument set at 482°C (900°F), per NFPA standards.

The temperature-control thermocouple was located in the oven, just outside the supply duct, where the heated air first enters the oven chamber after leaving the duct (Fig. 2). This location allows precise control of the process temperature because the air temperature is being sensed just prior to impinging on the load. The burner output is adjusted via a 4-20 mA signal from the controller to maintain the exact burner output required to accurately sustain the temperature of heated air as it enters the load of magnesium.

On the ill-fated magnesium drying oven, the high-limit thermocouple was also located in the heating chamber just outside the supply duct. This "convenient" location near the front of the furnace was less than ideal because the air temperature was not being sensed at the hottest location in the oven. It also left the high-limit thermocouple exposed to the influence of the load itself and also vulnerable to entrainment of cold air entering through the oven doors should they not seal properly or be left open. This location of the thermocouples near the over door turned out to be a critical error.

## The Fatal Scenario

During one particular heating cycle, the oven doors were inadvertently left partially open (Fig. 2). This allowed cold air from the factory to begin to enter the oven during heating. At first, this was not a problem because it took several hours for the oven to heat up due to the large mass of magnesium, the load cart, the oven interior sheet metal and structure.

As the oven continued to heat, however, the area inside the front of the oven, where the control thermocouple was measuring, remained colder

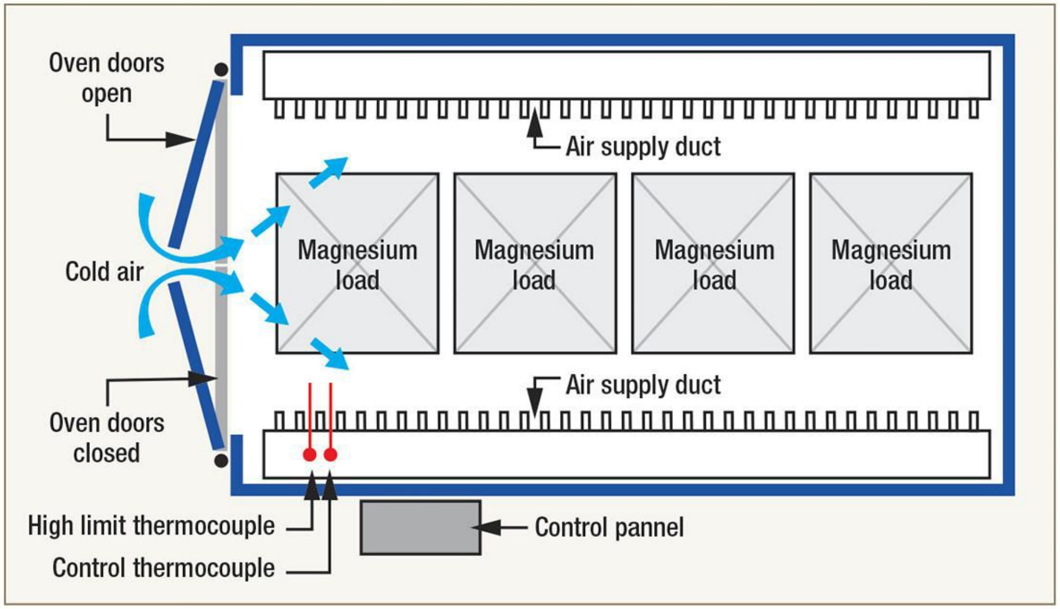

Fig. 2. Oven plan view showing thermocouple locations and air from the factory entering through partially open doors

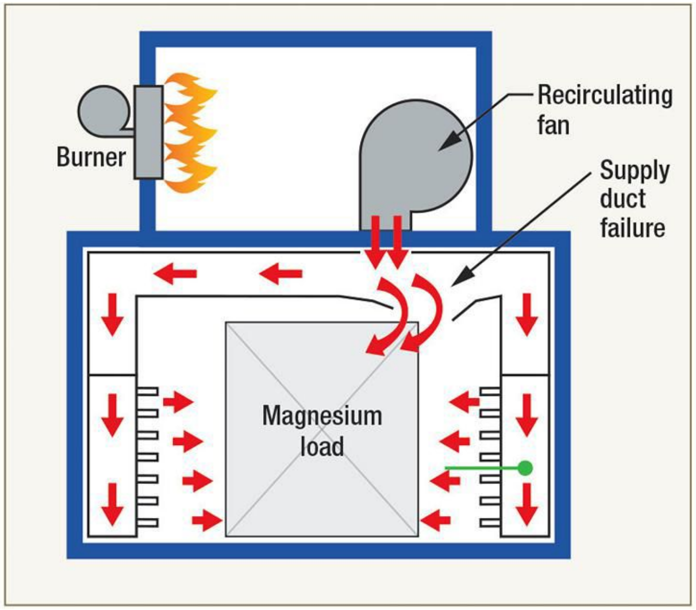

Fig. 3. Supply-duct failure directs overheated air to the magnesium

than everywhere else in the oven. This caused the temperature controller to continue to demand heat from the burner in an unsuccessful effort to bring the control thermocouple up to the setpoint temperature.

The burner, located in a housing above the workload area, was rated at 0.6 million kilocalories (2.4 million BTU/hour), and it continued firing at 100% output. With the doors still partially open, the control thermocouple continued to mistakenly believe the oven interior was below setpoint, and the oven temperature continued to rise. Since the high-limit thermocouple was also located near the front of the oven, it did not sense the over-temperature condition and failed to turn off the heat.

The oven interior temperature continued to climb and reached 593°C (1100°F). This caused the supply-duct sheet metal to fail and split open right above the load of magnesium near the center of the oven (Fig. 3). As a result, extremely hot air exited the heater house (where the blower and burner are located) and directly impinged on the magnesium. The combination of overheated air and radiant heat from the burner caused the magnesium to reach its ignition temperature of 664°C (1227°F). An irreversible reaction began at this point, and the 19,090-kg (44,000-pound) load of magnesium ignited and burned with an intense, white heat.

As the magnesium continued to burn, ultimately reaching a temperature of 2200°C (3992°F), the oven was entirely engulfed in flames. Considering that magnesium has a heat of combustion of 25.1 MJ/kg, the 19,090-kg load contained over 47,000 MJ (44.6 million BTU) of energy, the equivalent of 1,545 kg (3,400 pounds) of coal. The fire burned so intensely, it was impossible to put out. The factory was evacuated, and the fire was allowed to burn until its magnesium fuel was entirely exhausted, which took over 12 hours!

## Lesson Learned

If closed-loop control is used in a heating process, one must pay special attention to the location of both the control and high-limit (excess temperature) thermocouples. During an overheating situation, the high-limit thermocouple must be able to sense a runaway-temperature condition and signal the stand-alone high-limit instrument to shut off heat to the unit. In this case, the high-limit thermocouple location combined with the use of a heat head to reduce cycle time, prevented the over-temperature system from realizing the temperature was becoming dangerously high. As such, it was unable to protect the oven or the work.

The recommended location for a high-limit thermocouple is in the hottest spot inside the unit. Considering the design of this oven, this would have been either near the burner or directly at the immediate outlet of the recirculation fan. Either location would have sensed an overheating condition regardless of the oven doors being open and would have prevented a magnesium oven fire.

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# Quench Cracking

It is important for heat treaters to understand the mechanisms associated with quench cracking and to take all prudent steps necessary to avoid in-service product failures (Fig. 1). To accomplish this task, we must work with design and manufacturing engineers on materials selection, manufacturing methods (including heat treatment) and safety allowances. One of the first steps in this process is to understand how flaws in materials initiate failures and how heat treatment might contribute to the problem. Let’s learn more.

## Flaws

The critical flaw size in a material is defined as the size of a flaw that will cause failure of the component at the expected operational stress level. Flaws exist in most engineered materials and may be characterized as cracks, voids, inclusions, weld defects or design/manufacturing discontinuities acting singularly or in combination with one another (Fig. 2). Simply stated, flaws are responsible for most parts failing.

Flaws are stress concentrators. It is also important to understand that surface cracks and internal cracks are not the same (large surface cracks being worst) and long, thin cracks are especially bad (since they have a lower radius of curvature and propagate under lower stress conditions). Any applied stress at the surface rises to a maximum value near the crack. In addition, applied loads will not distribute themselves over cracks. The size, orientation and distribution of cracks in a material influence which cracks will grow under stress and to what extent. Remember, once initiated, cracks propagate at the speed of sound.

There are three classic modes of fracture associated with cracks: tensile (Mode I), sliding (Mode II) and tearing (Mode III). Stress intensity (e.g., KI for Mode I) is a function of loading (i.e. applied stress), crack size and geometry. If KI represents the level of stress at the tip of a crack, the fracture toughness, KIC, is the highest value of stress intensity that a material can withstand (under very specific conditions) without fracture. Fast fracture occurs in a stressed material either when the crack reaches a critical size or a critical stress value. Perhaps surprisingly, the combination of critical stress and critical crack length at which fast

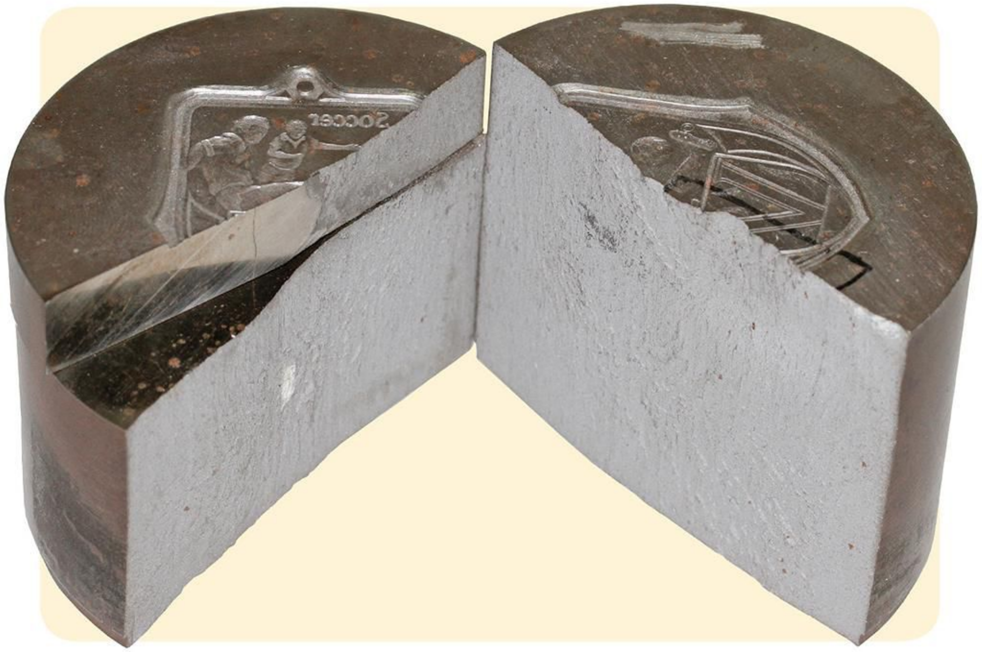

Fig. 1. In-service **D2 tool-steel component failure due to quench cracking** (Photograph courtesy of Aston Metallurgical Services Co., Inc.)

fracture occurs is a material constant.

Ductile fractures are usually more desirable than brittle fractures since they normally provide some form of warning before failure, whereas brittle failures do not since there is little or no plastic deformation at strain rates typically under 5%. In general, temperature determines the amount of brittle or ductile fracture that can occur in a material. At higher temperatures, the yield strength is lowered and fracture tends to be more ductile in nature. On the opposite end (at lower temperatures) the yield strength is greater and fracture tends to be more brittle in nature. At moderate temperatures (with respect to the material), the material exhibits characteristics of both types of fracture.

Failure types depend on both temperature and stress. For noncyclic stress conditions and temperatures under 0.4 times the melting point, failure stress decreases with increasing maximum flaw size, rate of loading or decreased temperature. For temperatures greater than 0.4 times the melting point, time to failure decreases as stress or temperature increases.

## Quench Cracking

Certain heat-treatment processes such as hardening and quenching tend to increase the internal stress state of a material. Improper heating to austenitizing temperature can result in thermally induced stress, which may cause a flaw to open up into a crack. As the material is heated, it undergoes volumetric changes due to phase transformation as well as thermal expansion. Rapid heating only accentuates this condition. Cracks occurring during heating are most often related to material imperfections, seams and inclusions.

Rapid or uneven cooling, especially when

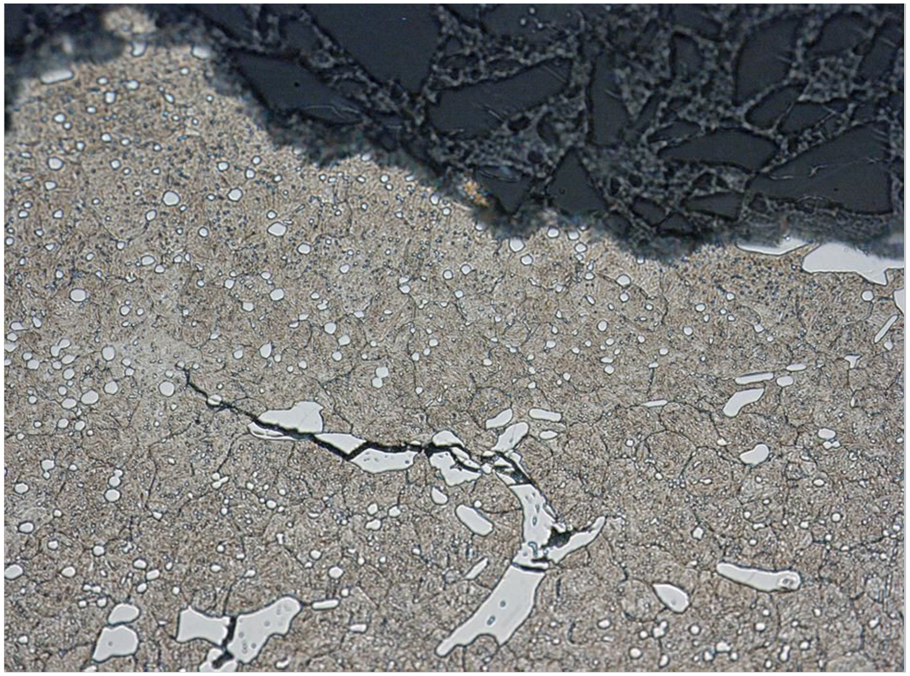

Fig. 2. Fracture path in D2 tool steel (Photograph courtesy of Aston Metallurgical Services Co., Inc.)

transforming the microstructure to martensite, also creates additional internal stresses. Holes, sharp edges, grooves, slots and corners can all be potential stress risers and crack-initiation zones. At a sharp edge or edge of a hole, for example, the heating and cooling rates can be substantially higher than the surrounding areas, putting tremendous strain on the material in these regions. While those features may be necessary in the component, it is important to exercise good engineering practices and properly chamfer or radius those areas to prevent sharp corners and edges. In induction heating, for example, certain materials may be placed in holes and other critical areas to help act as a heat sink and dampen the shock during the quenching operation. This can be costly, however, and the efficiency of the heat-treat operation may suffer.

The common ways in which quench cracking can occur from heat treating include:[4]

- Improper steel selection: Selecting a steel with too high a hardenability can result in susceptibility to quench cracks and excessive core hardness.
- Improper part design: Sharp changes of section, lack of radii, holes, sharp keyways and unbalanced sectional mass create stress risers in locations where cracking is likely to occur.
- Inadequate stock removal: During original machining, remnants of seams or other surface imperfections act as a nucleation site for quench cracking during subsequent thermal operations.
- Overheating: Temperature overshoot during the austenitizing portion of the heat-treatment cycle can coarsen normally fine-grained steels. Coarse-grained steels increase hardening depth but are more prone to quench cracking than fine-grain

steels. Overheating and excessively long dwell times should be avoided while austenitizing.

- Improper quenchant selection: Using an overly aggressive quenchant (e.g., water, brine or caustic) when a less severe quench media (e.g., polymer, oil) will work is a common cause of part cracking.
- Improper fixturing and entry of the part into the quenchant: A part should enter the quench medium with as little interference as possible. Differences in cooling rates can be created, for example, if parts are nested together in a basket, resulting in the parts along the edges cooling faster than those in the mass in the center. Part geometry can also interfere with quenchant delivery and effectiveness.
- Long lag times between quenching and tempering: Tempering parts as soon as practical will help avoid internal stresses from building up and being relieved by cracking. Certain high-hardenability materials, such as 4340, are particularly prone to quench cracking and must be tempered immediately (usually within 15 minutes of quenching) to avoid problems.

Material and process selection can contribute to cracking as well. For example, high-carbon and alloy steels with high hardenability often exhibit a greater tendency to initiate and propagate cracks. Even if they do not crack, materials that are heat treated to very high strength levels may contain localized concentrations of very high residual stress. If these stresses are aligned in the same direction as the applied load in service, catastrophic failure can occur. Care must also be taken to temper all materials before subsequent operations.

Cracking can also be encountered in materials such as iron, nickel and cobalt superalloys (the phenomena is called “fire cracking” or “strain-age cracking” or “stress cracking”) and occurs in both age-hardenable and solid-solution alloys.[5]

Causes for this condition have been traced to such items as high residual tensile stresses on the part surface, a strain-intolerance microstructure and the presence of stress risers. Steps to prevent cracking in superalloy materials include: reducing (or eliminating) residual surface tensile stresses (e.g., shot peening); modifying or redesigning part geometry (to eliminate stress risers); and addressing surface-roughness issues.

## Summing Up

Avoiding premature failure of component parts, whether due to improper material selection, design, manufacture or heat treatment, is a goal shared by all. It is critical, therefore, that heat treaters understand enough fracture mechanics (the study of the propagation of cracks in materials) and engineers understand the effect of heat treatment so as to eliminate in-service performance issues.

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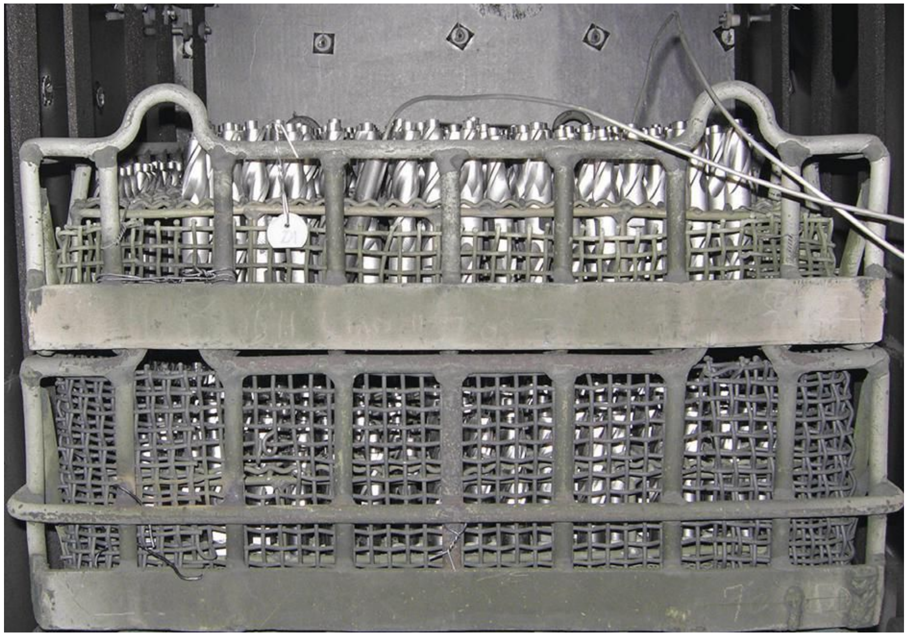
# Simplified Temperature Uniformity Surveys Using Ceramic Technology

Heat treaters have been searching for ways to simplify furnace temperature uniformity surveys. Using proven technology developed in the ceramics industry, this is now a reality. With emphasis today on the importance of meeting the requirements of AMS 2750D, CQI-9 and Nadcap, the heat-treating, brazing and sintering industries now have a tool to help them focus on the uniform and efficient delivery of heat to their products to ensure consistent and repeatable quality.

For many years the ceramics industry has known that predictable shrinkage can be obtained by various nonmetallic, inorganic materials when exposed to a known amount of thermal energy. This technology offers a simple, economical option for the measurement and recording of temperature (Fig. 1). Various types of ceramic temperature-measurement devices are in common usage throughout the ceramics industry.[1]

## Product Development for Heat Treating, Brazing and Sintering

Starting from product mixes known to work in the ceramics industry, various blends of inorganic materials were developed and tested to determine the best fit for the rapid heating and cooling cycles utilized in the metals industry. Also necessary was the investigation of the various furnace atmospheres used in the heat-treating, brazing and sintering industries, including air, various hydrogen/nitrogen mixtures, endothermic and exothermic gas and even gases used for vacuum partial-pressure and high-gas-pressure quenching (up to 20 bar). Material



TempTab® discs placed in proximity to workload thermocouples

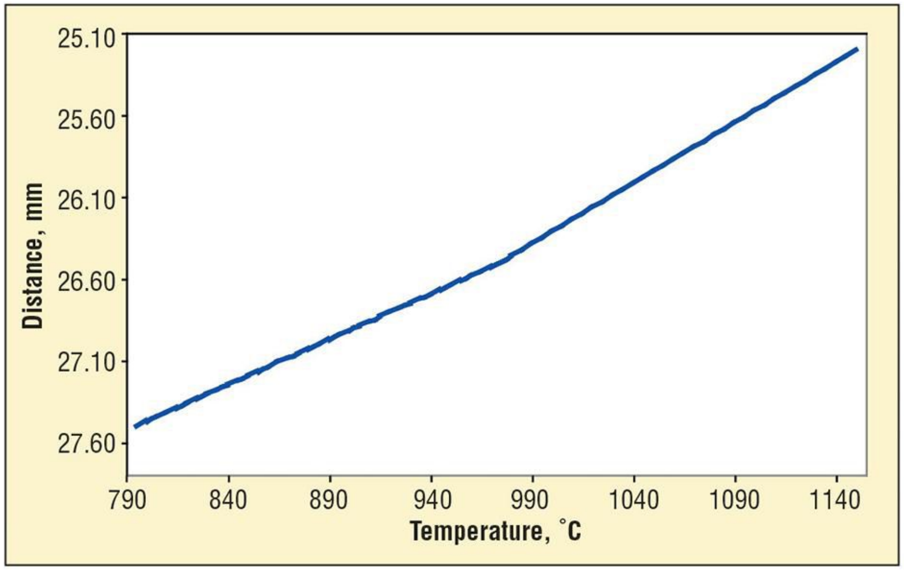


Fig. 1. Typical relationship between temperature and final dimension for ceramic temperature-measurement devices

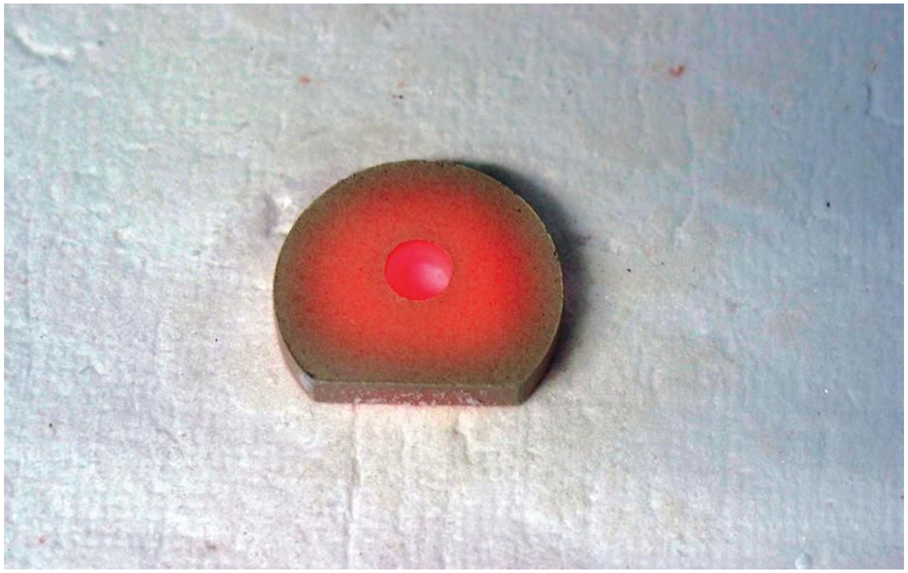


Fig. 2. Final TempTab® shape seen cooling in air

selection also took into account the need to prevent contamination of the work or the furnace from an atmosphere with unwanted contaminates released from inorganic compounds. As a result, a number of blends of materials were investigated and an optimum mix selected to give the best performance over the wide range of temperatures necessary to meet the requirements of the metals industry. The ceramic-disc product developed is called a TempTab®.[2]

Once the blends were established, the size and shape of the measurement device was carefully chosen to provide ease of use and to maximize its ability to withstand the thermal shock of rapid heat-up and quench rates (Fig. 2).

Dry pressing was the forming method of choice due to the ability to control size and density of the finished piece. Once pressed, the product is further dried to remove processing water and then calcined to remove the binder used during forming, bound-chemical water and other volatiles, resulting in a product with no contaminate evolution on subsequent exposure to temperature. Each batch is then analyzed to develop a unique temperature look-up table (Fig. 3) to account for the shrinkage characteristics in the raw materials.

The ceramic-disc location within a load or furnace can be marked with an identifier simply by using a high-temperature marking pen or in many cases a Sharpie®. Once the heat-treating process is complete, the discs are collected and measured (in millimeters to two decimal places) using a digital micrometer and a gauge fixture (Fig 4).

The measurements are taken across the widest dimension of the disc and then converted to a TempTab temperature using the look-up table supplied with each batch. The accuracy of temperature can be measured within +/-10°F (5.5°C). Therefore, variations in temperature within the furnace can easily be detected and quantified. TempTab discs are so easy to use, the furnace operator is able to run temperature uniformity checks with each load, when they are experiencing problems or before a formal temperature uniformity survey. Statistical-process-control charts can be created for each furnace, for each part number run or for each type of load. In this way, the discs serve as an early warning device to detect temperature variation and to plot trends (Fig. 5). TempTab discs, however, are not intended to be used as thermocouple-calibration devices.

The fact that these ceramic discs can be run with or alongside the work is a real convenience because temperature uniformity survey checks

| Time at Temperature | | | | | |
|---|---|---|---|---|---|
| 10 Minutes | | 30 Minutes | | 60 Minutes | |
| Temp C | mm | Temp C | mm | Temp C | mm |
| 1150 | 25.45 | 1150 | 25.25 | 1150 | 25.21 |
| 1145 | 25.55 | 1145 | 25.28 | 1145 | 25.25 |
| 1140 | 25.60 | 1140 | 25.31 | 1140 | 25.28 |
| 1135 | 25.66 | 1135 | 25.34 | 1135 | 25.32 |
| 1130 | 25.72 | 1130 | 25.37 | 1130 | 25.36 |
| 1125 | 25.78 | 1125 | 25.43 | 1125 | 25.39 |
| 1120 | 25.85 | 1120 | 25.49 | 1120 | 25.43 |
| 1115 | 25.92 | 1115 | 25.53 | 1115 | 25.47 |
| 1110 | 26.00 | 1110 | 25.57 | 1110 | 25.50 |
| 1105 | 26.03 | 1105 | 25.61 | 1105 | 25.54 |
| 1100 | 26.06 | 1100 | 25.66 | 1100 | 25.58 |
| 1095 | 26.10 | 1095 | 25.72 | 1095 | 25.62 |
| 1090 | 26.13 | 1090 | 25.80 | 1090 | 25.65 |
| 1085 | 26.16 | 1085 | 25.86 | 1085 | 25.69 |
| 1080 | 26.20 | 1080 | 25.92 | 1080 | 25.73 |
| 1075 | 26.25 | 1075 | 26.00 | 1075 | 25.76 |
| 1070 | 26.30 | 1070 | 26.04 | 1070 | 25.80 |
| 1065 | 26.32 | 1065 | 26.09 | 1065 | 25.84 |
| 1060 | 26.35 | 1060 | 26.13 | 1060 | 25.87 |
| 1055 | 26.38 | 1055 | 26.17 | 1055 | 25.91 |
| 1050 | 26.40 | 1050 | 26.21 | 1050 | 25.95 |
| 1045 | 26.42 | 1045 | 26.26 | 1045 | 25.98 |
| 1040 | 26.44 | 1040 | 26.28 | 1040 | 26.02 |
| 1035 | 26.46 | 1035 | 26.29 | 1035 | 26.06 |
| 1030 | 26.48 | 1030 | 26.31 | 1030 | 26.09 |
| 1025 | 26.50 | 1025 | 26.33 | 1025 | 26.13 |
| 1020 | 26.52 | 1020 | 26.35 | 1020 | 26.17 |
| 1015 | 26.54 | 1015 | 26.37 | 1015 | 26.21 |
| 1010 | 26.56 | 1010 | 26.39 | 1010 | 26.24 |
| 1005 | 26.59 | 1005 | 26.41 | 1005 | 26.28 |
| 1000 | 26.61 | 1000 | 26.43 | 1000 | 26.32 |
| 995 | 26.64 | 995 | 26.45 | 995 | 26.35 |
| 990 | 26.66 | 990 | 26.47 | 990 | 26.39 |
| 985 | 26.68 | 985 | 26.49 | 985 | 26.43 |
| 980 | 26.70 | 980 | 26.52 | 980 | 26.46 |
| 975 | 26.72 | 975 | 26.54 | 975 | 26.50 |

| Time at Temperature | | | | | |
|---|---|---|---|---|---|
| 10 Minutes | | 30 Minutes | | 60 Minutes | |
| Temp C | mm | Temp C | mm | Temp C | mm |
| 970 | 26.74 | 970 | 26.57 | 970 | 26.53 |
| 965 | 26.76 | 965 | 26.60 | 965 | 26.56 |
| 960 | 26.79 | 960 | 26.64 | 960 | 26.59 |
| 955 | 26.82 | 955 | 26.66 | 955 | 26.61 |
| 950 | 26.84 | 950 | 26.69 | 950 | 26.64 |
| 945 | 26.87 | 945 | 26.72 | 945 | 26.67 |
| 940 | 26.90 | 940 | 26.74 | 940 | 26.70 |
| 935 | 26.92 | 935 | 26.77 | 935 | 26.72 |
| 930 | 26.95 | 930 | 26.81 | 930 | 26.75 |
| 925 | 27.00 | 925 | 26.84 | 925 | 26.78 |
| 920 | 27.05 | 920 | 26.87 | 920 | 26.81 |
| 915 | 27.11 | 915 | 26.91 | 915 | 26.83 |
| 910 | 27.16 | 910 | 26.95 | 910 | 26.86 |
| 905 | 27.21 | 905 | 26.98 | 905 | 26.89 |
| 900 | 27.26 | 900 | 27.06 | 900 | 26.92 |
| 895 | 27.32 | 895 | 27.15 | 895 | 26.95 |
| 890 | 27.37 | 890 | 27.19 | 890 | 26.97 |
| 885 | 27.38 | 885 | 27.23 | 885 | 27.00 |
| 880 | 27.39 | 880 | 27.28 | 880 | 27.03 |
| 875 | 27.40 | 875 | 27.31 | 875 | 27.06 |
| 870 | 27.41 | 870 | 27.34 | 870 | 27.08 |
| 865 | 27.42 | 865 | 27.37 | 865 | 27.11 |
| 860 | 27.43 | 860 | 27.41 | 860 | 27.14 |
| 855 | 27.45 | 855 | 27.44 | 855 | 27.17 |
| 850 | 27.46 | 850 | 27.45 | 850 | 27.20 |
| 845 | 27.47 | 845 | 27.46 | 845 | 27.22 |
| 840 | 27.48 | 840 | 27.47 | 840 | 27.25 |
| 835 | 27.49 | 835 | 27.48 | 835 | 27.28 |
| 830 | 27.51 | 830 | 27.49 | 830 | 27.31 |
| 825 | 27.52 | 825 | 27.50 | 825 | 27.33 |
| 820 | 27.53 | 820 | 27.52 | 820 | 27.36 |
| 815 | 27.54 | 815 | 27.53 | 815 | 27.39 |
| 810 | 27.55 | 810 | 27.54 | 810 | 27.42 |
| 805 | 27.56 | 805 | 27.55 | 805 | 27.44 |
| 800 | 27.57 | 800 | 27.56 | 800 | 27.47 |
| 795 | 27.58 | 795 | 27.57 | 795 | 27.50 |

Fig. 3. Batch-specific table for relating final dimension to temperature

can be conducted without interrupting normal production. To conduct a temperature uniformity check, one needs only to place the TempTab discs in locations of interest within the furnace, such as where you would normally place thermocouples if conducting a survey.

## Equipment Diversity

TempTab discs have been successfully used to conduct temperature profiles and temperature uniformity surveys in mesh-belt conveyor furnaces (Fig. 6). They have also been used for brazing steel, stainless steel, copper and even brass alloys in various atmospheres, including dissociated ammonia and various nitrogen/hydrogen atmospheres supplied from cylinders, tube trailers and cryogenic tanks.

These ceramic shapes have been tested in integral-quench furnaces, which were hardening, carburizing and carbonitriding gears and other types of critical components. They are used to provide an indication of the uniformity of temperature within a large batch load. Ceramic discs are well suited to the sintering of powdered-metal products and were field tested in mesh-belt conveyor furnaces running at a variety of belt speeds. This provided, for example, an indication of temperature uniformity across the belt as well as peak part temperature. Used in pusher-

Fig. 4. Digital dimension measurement tool

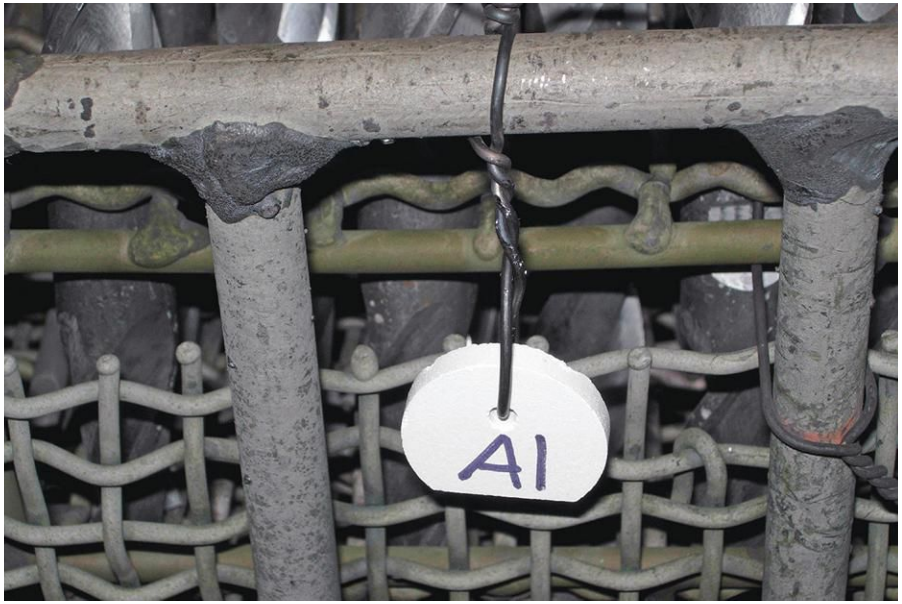


Fig. 5. Typical TempTab® position in a furnace load

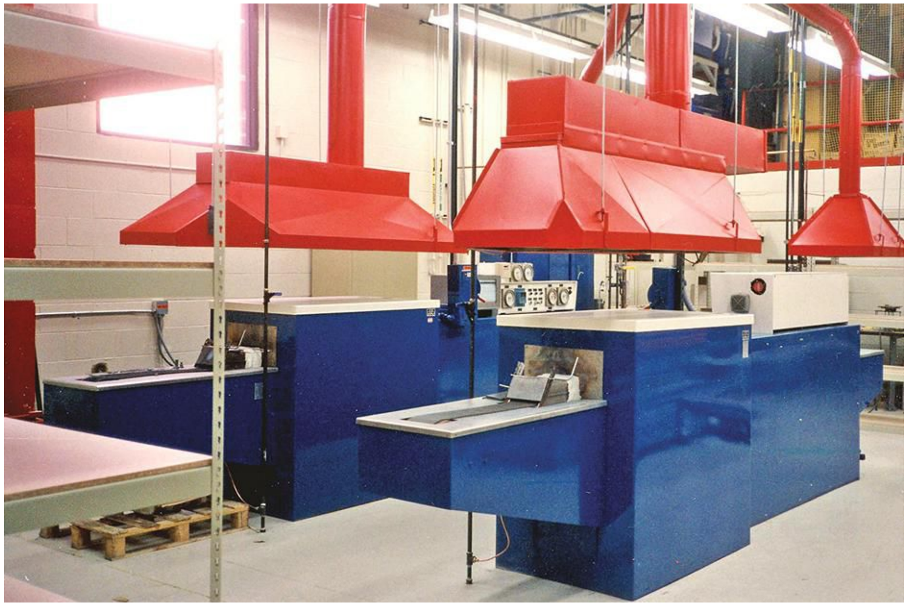


Fig. 6. Typical mesh-belt brazing furnaces
(Photograph courtesy of C.I.Hayes)

Fig. 7. Typical vacuum-furnace application
(Photograph courtesy of Specialty Heat Treating, Inc.)

style furnaces, these discs help determine the heat work associated with the proper delivery of thermal energy over time.

The discs have also proven to be reliable in temperature uniformity surveys in single-, dual- (Fig. 7) and multi-chamber vacuum furnaces for various applications, including low-pressure vacuum carburizing of automotive parts with a high-pressure nitrogen quench.

## Application Diversity

### Case Study 1 – Brazing and Annealing of Brass, Copper and Stainless Steel

In a large annealing and brazing operation with 22 atmosphere mesh-belt furnaces, TempTab discs and other ceramic temperature-measurement shapes were placed on the belt alongside the product as it was going through the furnace (Fig. 8). The dwell time in the furnace was approximately 40 minutes with the parts being exposed to maximum temperature for approximately 10 minutes. Depending on belt width, either two or three discs were placed on the belt – left side, right side and

Fig. 8. Various ceramic temperature devices running with production parts

center, if necessary. Once through the process, the discs were measured and the look-up table used to determine the maximum (peak) temperature they were exposed to as a function of location.

As expected in a well-run operation, the temperature uniformity of many of the furnaces was relatively close, within ±10°F (5.5°C). These tests revealed, however, that several furnaces had large temperature differences across the belt, in one case as much as 122°F (50°C). Finding this large difference told the process engineer he needed to immediately

work with his maintenance group to find and correct the source of the temperature variation, and he alerted the quality engineer to conduct more extensive testing of product being produced from those specific furnaces.

Running discs at the beginning of every shift provided a record of the thermal energy being applied to the product, and this was used as a diagnostic tool to locate problems. The use of this product can alert personnel to a problem with temperature or assist when trying to solve problems with higher than normal reject rates.

### Case Study 2 – Brazing of Stainless Steel

Product-contamination tests were conducted on small stainless steel components run on graphite boards through mesh-belt conveyor furnaces in which the operating dew point of the dissociated-ammonia atmosphere was in the -50 to -60°F (-45 to -50°C) range. In addition to determining the peak temperature achieved during the brazing operation, part contamination was carefully evaluated using X-ray photoelectron spectroscopy (XPS). This analysis method revealed that the TempTab discs did not cause any type of part surface contamination to occur.

### Case Study 3 – Annealing of Stainless Steel

Ceramic discs were tested in a commercial heat-treat facility in a vacuum furnace dedicated to bright annealing of stainless steel automotive parts. The discs were run along with survey thermocouples attached to a data logger at several temperatures, and the resulting data showed good correlation against thermocouple readings. The discs were wired in place in and around the parts in a stack of five wire trays used to hold the parts to be annealed. The batch furnace was brought to temperature and the parts were loaded into the vacuum chamber. Once evacuated, the parts were exposed to temperatures of 1740°F (950°C) for three hours. The temperature was quickly lowered using a 2-bar nitrogen quench. Temperature differences were identical from both testing methods, and a 30°F differential was found from one location to another.

### Case Study 4 – Hardening of Tool Steels

Tests were run in two identical single-chamber batch vacuum furnaces processing M-series (M2, M3 Type 1 & 2, M7, M42), T-series (T-1, T-15) and specialty tool steels (ASP 23, ASP 30, CPM M4, REX 23, REX 76) at temperatures between 2050°F (1120°C) and 2315°F (1270°C) to check disc integrity as a function of process recipe, load configuration, ramp rate, partial-pressure settings, austenitizing temperature and quench severity (5- and 6-bar nitrogen). The discs were unaffected by changes in any of the process variables. The results from these tests proved that running a temperature uniformity survey before its scheduled date was unnecessary, and a good correlation was achieved with the two load thermocouples routinely run with each workload.

**Case Study 5 – Sintering of Powder Metal**

A major producer of automotive powder-metal engine components ran tests to check the side-to-side temperature uniformity as well as peak temperature values in multiple mesh-belt conveyor furnaces having both open-chamber and muffle-type designs. The impact of delubrication and changes in furnace-atmosphere dew point as a function of location within the furnace were evaluated. The discs were unaffected, and comparison to standard temperature uniformity surveys showed excellent correlation.

## Conclusion

The importance of temperature control in the heat-treatment, brazing and sintering industry cannot be overstated. Furnace temperature uniformity surveys have and always will be necessary, but they have never been as simple as now. TempTab® discs offer the first truly simple and affordable way to monitor thermal processing on a daily basis. Early detection of problems allows more time for thought as to the best path for corrective action and promotes preventative maintenance.

Although these discs are not a complete replacement for temperature uniformity surveys, they are very capable of providing a record of the temperature history within a furnace without interrupting production schedules and without incurring additional cost.

# The Role of Metallurgical Analysis in Solving Heat-Treat Problems

As a young boy growing up in the neighborhoods of Chicago, one of the Heat Treat Doctor's most trusted friends was a mythical conjurer by the name of Mandrake, The Magician. If a task seemed impossible to accomplish, or when all else failed, all one needed to do was summon Mandrake, and "voila," the impossible became possible!

As heat treaters, we often seek answers to processing problems or component part failures from the metallurgical community without fully understanding what they need to do their job properly. What do we need to provide, whether it be accurate background information, a representative set of samples (good and bad) for comparative analysis, or even something as simple as protecting the surface of the component to be analyzed from further damage? Often, we don't communicate our expectations in precise terms and thus do not know what to expect from an analysis. It's time for us to learn what we can do to assure that accurate information and reliable facts result from whatever testing or analysis is performed, upon which we can make informed decisions. Mandrake would be proud. Let's learn more.

## The Role of Photography

In this day and age of digital photography, a picture can indeed be worth the legendary 1,000 words (or more). Provide photographs of everything, from multiple angles, and remember to use good lighting and high resolution. Handle parts carefully so as not to induce damage and resist the temptation to refit mating fracture surfaces together. Note part orientation and other salient features.

## Processing History/Background Information

Don't assume that someone knows your process or product, or its intended service application, better than you do. Communicate the history of the part or process; separate assumptions from facts; provide necessary drawings, including mating components if appropriate as well as required specifications; and explain in detail the design requirements. In other words, take the guesswork out of the analyst's job. Document anything of importance and give this information to the metallurgist, even if it means having a confidentiality agreement in place before you start.

If it turns out that the component is being returned from the field, extreme care must be taken to ensure a representative sample and to avoid further damage (see "The Do's and Don'ts of Field Failure Analysis," *Industrial Heating*, January 2006).

## Incoming (Raw) Material Analysis

Too often we are forced to begin an analysis making assumptions about the raw material. Provide the laboratory with a copy of the mill's material certification sheets. In addition to the chemical constituents, the metallurgist will glean information based on the form of the raw material, its grain size, cleanliness and prior mill processing. Doing an actual chemical analysis is not necessarily redundant. For example, trace-element chemistry can play a significant role when investigating certain phenomenon, such as temper embrittlement.

## Laboratory Procedures

Discuss with your metallurgist or outside laboratory what type of tests will be conducted and in what order. Understand what will be achieved at each step in the analysis process so that you can ask questions or offer suggestions. (This will also help explain the time or expense involved.) In this way, you will be better able to interpret the final results.

Be aware there is nothing more frustrating in the

Fig. 1. The Doctor's boyhood companion

laboratory than to work hard on a job only to find out that it is not the right sample or that the damage observed was induced by extraneous factors. This translates into lost time and money. Also, be conscious of the fact that once the investigator has begun to work on your project, it is best that the analysis move forward uninterrupted. So be sure to define the scope of work and clarify the boundaries of what he or she is allowed to do once the investigation is under way. (Oftentimes, a "not to exceed" figure works well for this part of the investigation.)

Selecting the proper tests may involve trade-offs due to cost or time. Be sure you understand the cost/benefit relationship of each test and what the expected outcome might be so that the right choices can be made. Insist on specificity to avoid open-ended analysis efforts. Here are some examples of what can be done in the laboratory.

- Stereomicroscopy
- Nondestructive testing
  - Eddy current
  - Ultrasonic
  - Pressure testing (hydrostatic, pneumatic)
  - Surface finish
- Macroetching
- Mechanical testing
  - Hardness/microhardness testing
  - Tensile testing
  - Impact testing (e.g., Charpy testing)
  - Fatigue testing
  - Torque/torque-tension
  - Shear and double shear strength
  - Torsion testing
  - Creep
  - Stress rupture and stress durability
  - Vibratory testing
- Sample preparation
  - Unetched part examination
  - Etched part examination
- Optical microscopy
  - Microstructural determination
  - Grain size
  - Micro cleanliness
  - Intergranular attack
  - Inclusion characterization
  - Alpha case
- Image analysis
  - Plating depth (layer thickness)
  - Defect measurement
  - Grain size
- Scanning electron microscopy
  - Fractography
  - Feature/character recognition

- Energy-dispersive X-ray spectroscopy
    - Qualitative element analysis
    - Inclusion characterization
    - Elemental distribution (dot) mapping
- Corrosion testing

## Selecting the Right Laboratory

Not all laboratories are created equal, either in the talent of their researchers or in tools available to do the job right. Talk to people you trust in the industry to help in the selection process. Be aware that many labs are better at some things than others and subcontract certain tasks to other labs. Be sure that you understand when and why this is being done and determine if you are better off going direct.

## Comparative Analysis (Good vs. Bad)

If good parts exist, they can be invaluable aids in understanding why a bad part failed. Taking the seemingly extra step (and expense) of testing a good part along with a bad one will yield tremendous insight into the problem at hand. Do this whenever possible.

## Timing

In an effort to get answers, avoid the temptation to push the lab to the point where steps are skipped or time is not taken to investigate secondary factors that may prove to be major contributors. Ask for verbal reports at key milestones in the analysis work, but avoid taking up valuable analysis time by "checking in" too often. Meeting in person to begin a project is always beneficial.

## Lab Reports

Metallurgists tend to write reports for other metallurgists, a noble but often frustrating problem for the heat treater. If you need the report "translated" into layman's terms, be sure to tell the lab. Yes, there is a delicate balance here between

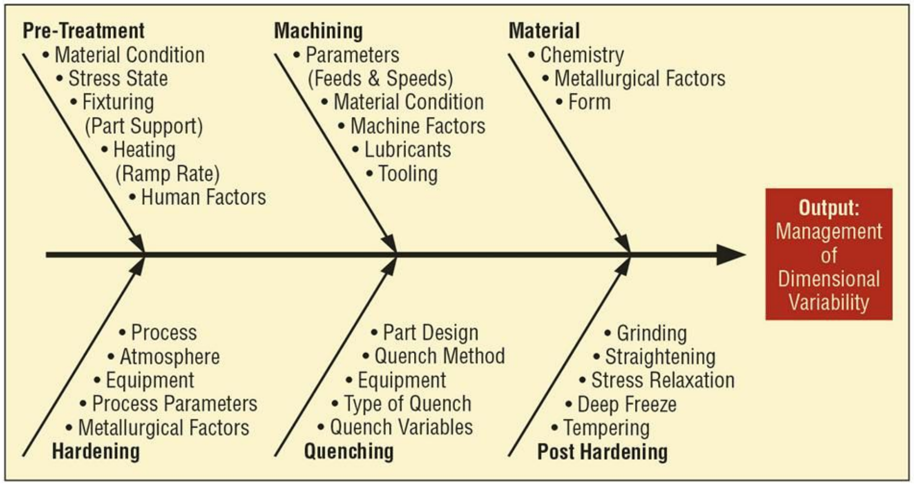


Fig. 2. Ishakawa diagram – quenching

the facts and their interpretation, but this can often be handled by placing the interpretation in a "Discussion" section of the report. The trend today, due to liability concerns, is to simply report the facts and rely on the client to interpret them. If necessary, hire outside experts to put the information in the proper context in order for you to determine the right course of action. There is nothing worse than paying good money for a report you don't understand.

## Root-Cause Determination

"What caused the problem, and how can I avoid its reoccurrence" should be the objective of any analysis effort. There are often multiple contributory factors and removing any one of them might avoid a part failure, even though defects may still exist. While it may or may not be possible to establish the root cause, it should always be the goal. The use of Ishakawa (fishbone) diagrams (Fig. 2) or other diagnostic methods listing all of the variables impacting a successful outcome can be a big help. Sometimes it's the thought process itself and a discussion among various company departments that leads to the solution to be implemented.

## The Bottom Line: To Analyze or Not to Analyze

A cost/benefit analysis should be performed before and after any analysis/testing work. Knowledge is strength, and assumption is weakness. When in doubt, do the metallurgical analysis. It will amaze you what can be revealed. And remember that Mandrake is alive and well, living within each and every metallurgist!

# The Value of Metallographic Interpretation

A wise professor of metallurgy once quipped, "The microstructure tells the whole story." As heat treaters, if we want to determine if a particular part is "good" or "bad," there is only one place to look – at the microstructure. This is not to say that there is no value in hardness or mechanical testing or other analysis techniques, but as all metallurgists know, the microstructure doesn't lie! Let's learn more.

Over the years, many Heat Treat Doctor columns have featured photomicrographs to illustrate concepts, provide insights or point out why we are able to draw certain conclusions. This is no accident. Most informative technical articles and scholarly works rely on microstructures to provide evidence, confirm conclusions or prove (without many words) certain facts. The key, however, is for the observer to understand the significance of what he is seeing. Therefore, metallographic interpretation becomes an invaluable skill.

Metallurgists secretly like destroying things. If we had our way, we would cut up every part in a load just to confirm they were all good. The problem, of course, is that there would be no parts to ship to the customer. Not our problem, as metallurgists like to say! So, we make the argument that other types of testing only give an indication that all is well but cannot make a definitive statement one way or the other. We must also be careful to draw conclusion only from a truly representative sample. There is no greater sin than

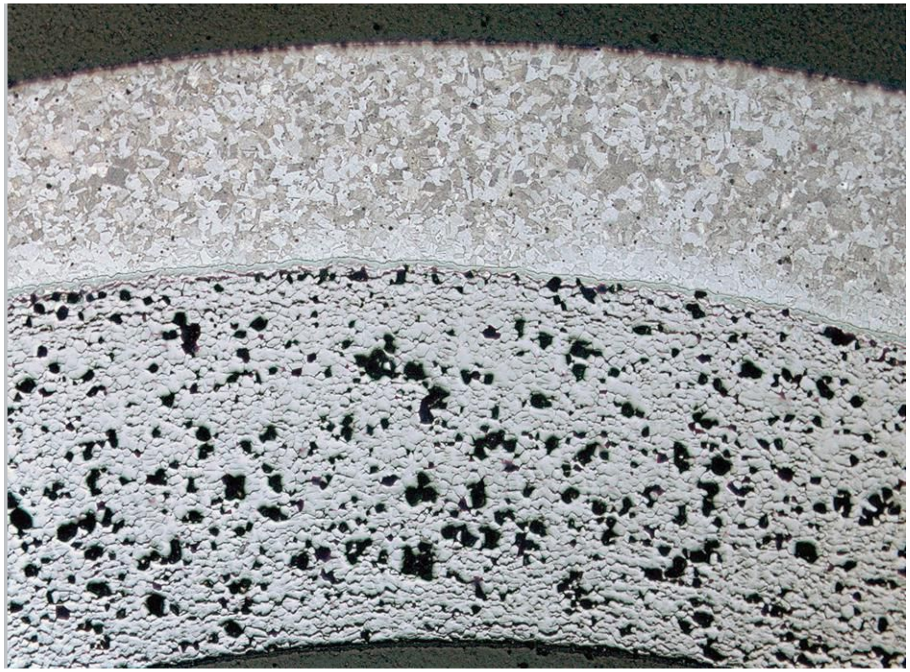


Fig. 1. 440C stainless steel (composite topographical image, 30X) example of thread damage from machining – Leika Model M205C digital composite rendering (Courtesy of Aston Metallurgical Services Co., Inc.)

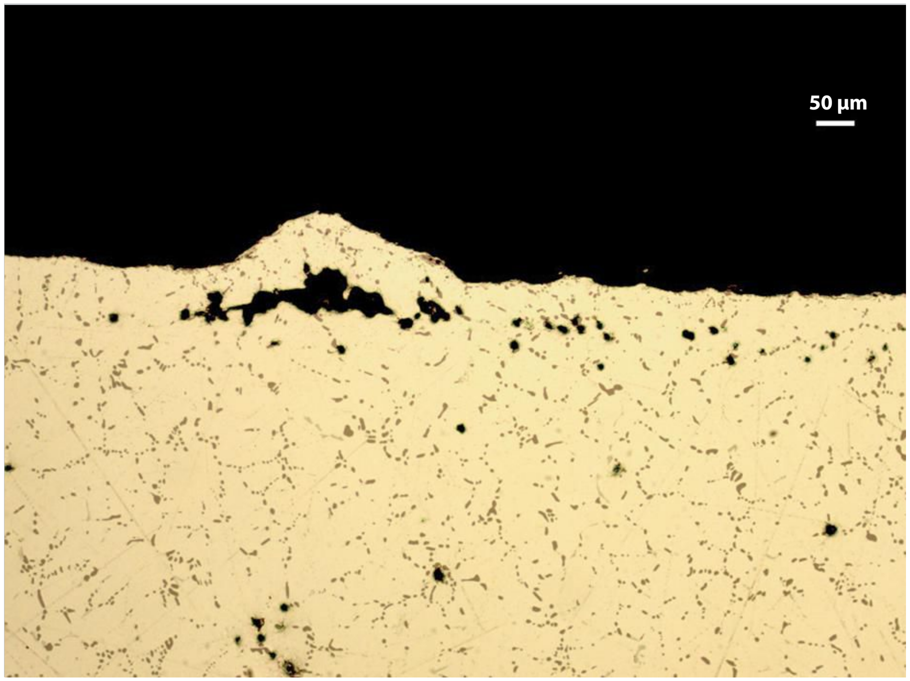


Fig. 2. A356.0 aluminum casting showing blistering due to hydrogen embrittlement (unetched, 125X)

spending considerable time and effort only to reach a false conclusion, a red "herring" if you will pardon the pun.

## Metallography Secrets

Any analysis starts with a good visual or low-magnification (5-50X) stereographic inspection of a component part (Fig. 1). This helps to set the direction for further investigation and often provides a clue as to the best starting point, which may be missed if we began the analysis at the microscopic level.

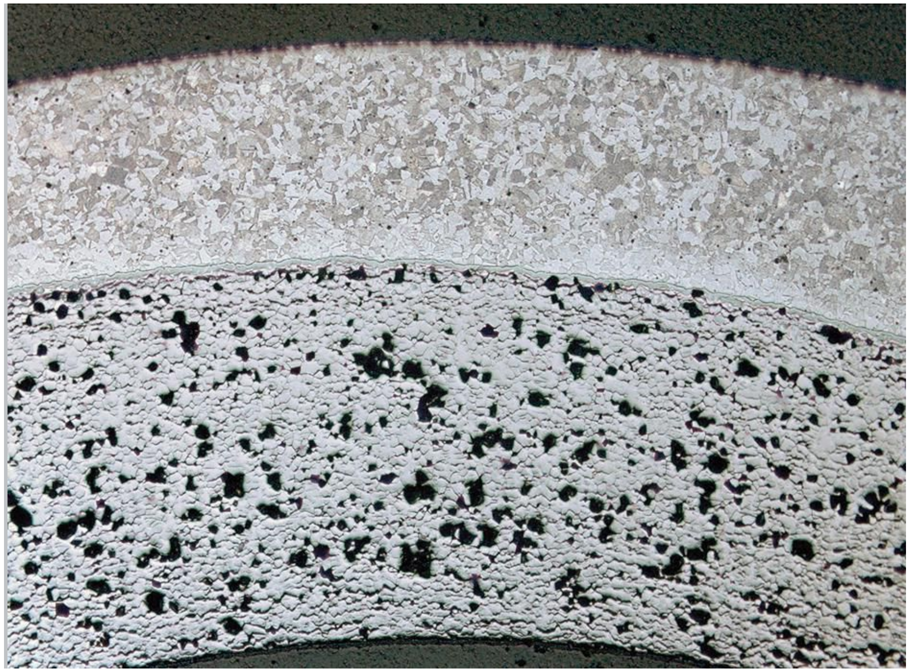


Fig. 3. Inconel 600 retort failure on the raw ammonia (inlet side) of an ammonia dissociator; effect of gaseous nitriding (inside to outside) after seven years of service (Kallings, DIC, 25X)

Metallography, whether it be via optical microscopy (Figs. 2-5) or through the use of more advanced tools such as a scanning electron microscope (Fig. 6), is oftentimes the easiest, fastest, most direct and most reliable way to determine the acceptability of a component part, solve a problem or determine the root cause of a failure. In the hands of a skilled metallographer, answers come quickly, and positive

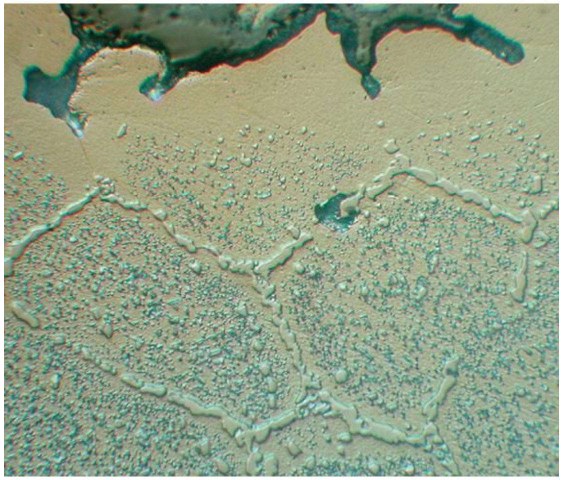


Fig. 4. 330 stainless steel alloy illustrating catastrophic carburization with radiant-tube failure in the area where the tube passed through the insulation (Kallings, 1000X)

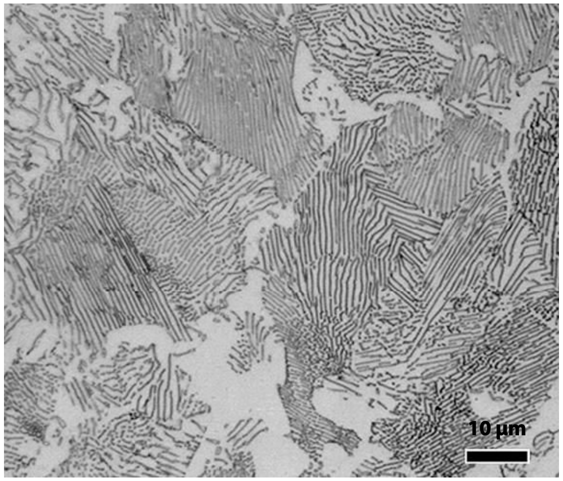


Fig. 5. SAE 4140 showing coarse pearlite and proeutectoid ferrite (1000X, 4% Picral) formed on delayed cooling from austenite (Courtesy of George Vander Voort)

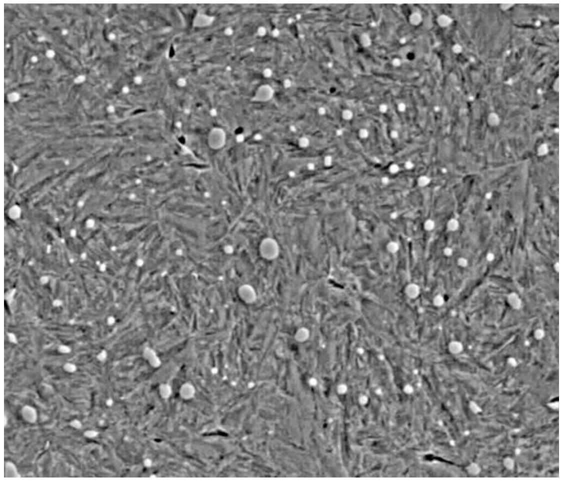


Fig. 6. 52100 bearing race that was vacuum hardened and 20-bar high-pressure gas quenched, showing fine carbide distributed in a matrix of tempered martensite (3680X, 2% nital).

conclusions can be drawn.

One of the secrets to success in metallographic interpretation is the ability to spot something in the microstructure that is abnormal. Management often doesn't understand why metallographic equipment costs so much, why more and more sophisticated tools are necessary or why it takes so long to reach a conclusion. The metallographer is akin to a detective constantly piecing together clues until the crime is solved. To the uninformed, however, getting answers is an exasperating experience, as metallurgists never seem to have enough evidence to conclude with absolute certainty.

## The Role of Comparative Metallography

It is a practical reality that many companies find themselves without a metallurgist or with fewer metallurgists having less time to support production on the shop floor. As such, technicians or those less skilled in the art are called upon to make daily decisions, and this is where comparative metallography comes in. Having examples of "acceptable" and "unacceptable" microstructures allows non-metallurgists to make decisions. The key is to have these comparisons prepared by a metallurgical laboratory that knows and understands what is needed.

For example, there is no need for color metallographic images, if the people doing the work day in and day out do not use this technique, or for providing photomicrographs at a magnification not in use.

## Metallurgical Reports

A comment about reports is also in order. If you request an analysis from an outside metallurgical laboratory, recognize that their reports are written by metallurgists, for metallurgists. Interpretation by those less skilled is sometimes difficult (if not impossible) in many cases. Outside testing laboratory reports, in particular, often do not attempt to interpret the facts. This is understandable given that, in most cases, not enough background information is available to understand the full scope of the problem under investigation. Asking the right questions of them and, if necessary, bringing in third parties for help in deciphering the results is of critical importance.

## A Lost Art?

Courses in metallographic interpretation are offered by universities, technical societies, suppliers of metallographic equipment and independent third-party consultants. These are both important and necessary to teach the basic skills, but they are no substitute for hands-on application of the lessons learned.

Metallography is a practical science gained by doing and doing over and over again. While one etchant or method may reveal the desired answer, experimenting with other techniques – perhaps a different edge-retention method, polishing cloth or etchant – may produce new insights. So don't be afraid to try new things. You will like what you find!

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# Interpreting Carburized Case Depths Part 2: Methods for Measuring Case Depth

We continue our discussion on how to interpret carburized case depths by focusing on measurement techniques. These methods are also applicable to cases produced by nitriding, nitrocarburizing, boronizing, and induction or flame hardening. Let's learn more.

Methods used to determine the depth of case can be categorized as visual, chemical or mechanical in nature. Sample parts or representative test specimens are most often of the same grade of steel as that being case hardened and ideally from the same heat of steel. At the very least, one should know the chemistry and/or properties. Some companies, however, prefer to use a single steel (e.g., SAE 8620 for carburizing) and perform tests on it for comparative purposes with the added benefit of gaining insights into furnace performance over time.

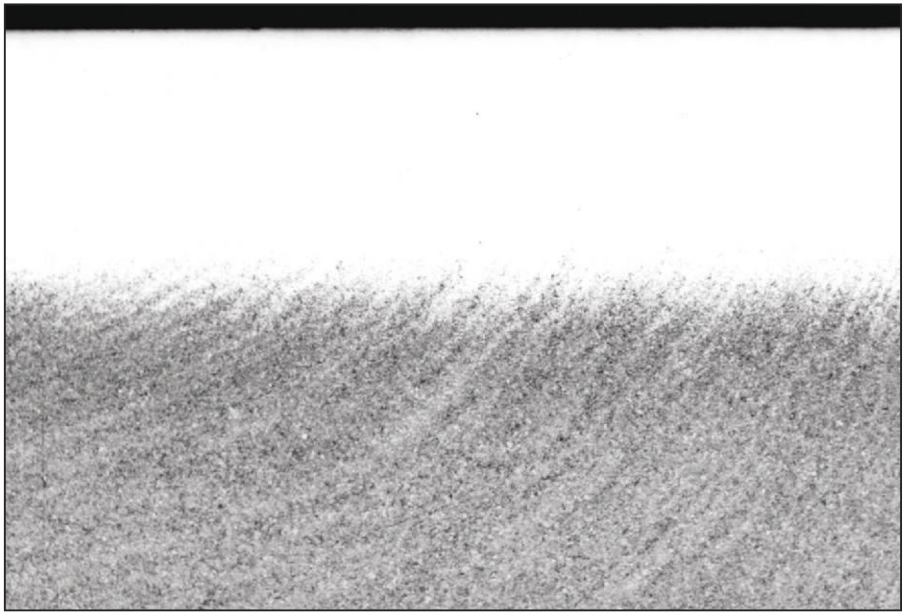

Figure 1. Example of demarcation line in the $M_s$ method

## Visual Methods

Visual interpretation falls in two broad categories: macroscopic and microscopic, and both are valuable.

### Macroscopic Techniques

Macroscopic methods are often used on the shop floor for routine process control, primarily because of their simplicity and the short time required for determination. They are typically done using the unaided eye, a loop or with a stereomicroscope up to magnifications of 40X. Accuracy of results can be improved by correlation with other methods used to measure the case depth of the parts being processed.

Visual methods are normally applied to hardened specimens. Induction- and flame-hardened samples are prime examples since they usually have excellent contrast between case and core when macroetched (e.g., 10% Nital). Other methods include the use of fracture bars, which is an efficient and quick way to test every case-hardened load. The depth of hardening is well defined and easily interpreted by fracture methods. The outside has a flat but slightly grainy appearance associated with brittleness, while the inside has an irregular, rather fibrous appearance associated with toughness.

The important point is that the fracture changes from one to the other quite abruptly. Case depth can be measured by a Brinell scope or with a scale on a stereomicroscope. If the core is soft, the fractured surface will exhibit good contrast between case and core. If the core is hard, bluing the fracture on a hot plate[3] can enhance the contrast.

The $M_s$ method[4] is another, but more involved, visual technique. It is based on the fact that the martensite-start temperature ($M_s$) varies with carbon content. Quenching (typically in a salt bath) and then holding the steel for a short time at the $M_s$ temperature corresponding to a given carbon content tempers the martensite formed at all lower-carbon levels. Subsequent water quenching transforms austenite at all higher-carbon levels to untempered martensite. Polishing and etching reveals a sharp line of demarca-tion between tempered and untempered martensite (Fig. 1).

## Microscopic Techniques

Microscopic methods are commonly used for determining case depth and have been described in detail by others.[3-5] What is often overlooked is that their accuracy depends on the nature of the case and core microstructures. For example, carburized depth is easier to evaluate in an unhardened sample while nitrided cases are, in general, difficult to estimate. It is important that the sectioning of the sample be perpendicular. Otherwise, the taper angle must be known. Perhaps the single-most overlooked step is to ensure good edge retention by use of proper mounting methods and procedures.

Microscopic techniques require that specimens first be given a full polish and etch before the evaluation, usually at 100X. Effective case-depth determination of hardened specimens relies on comparison to metallographic structures found to be equivalent to 50 HRC by other methods. A structure that is approximately 85% tempered martensite and 15% mixed transformation products often corresponds to 50 HRC. Total case depth is the demarcation line between the case and core (between dark and light regions after etching). This line is far from distinct for alloy steels.

## Chemical Methods

These methods usually rely on analysis of chips from turned bars. Test specimens must be carburized with the parts or in a manner representative of the process. If the parts and test specimens are quenched after carburizing, the specimens should be tempered at approximately 595-650˚C (1100-1200˚F) and straightened to 0.04 mm (0.0015 inch) maximum TIR (total indicator runout) before machining is performed. The time at temperature should be kept to a minimum to avoid excessive carbon diffusion even at these low temperatures.

Machining intervals between 0.05 and 0.25 mm (0.002 and 0.010 inch) are typically chosen depending on the accuracy desired and expected depth of case. Chips from each increment must be kept separate and analyzed individually for carbon content in a carbon analyzer or other suitable device. In some cases, especially for deep-case carburizing, taper bars can be used. They are machined, and spectrographic analysis is performed along the length of the bars at a spacing of at least one turn diameter apart.

## Mechanical Methods

These methods are preferred for an accurate determination of effective case depth and for determining total case depth in parts that have been shallow case hardened. The use of this method is based on obtaining and recording hardness values at specific intervals through the case. The sample is considered through-hardened if the hardness level does not drop below the effective case-depth value.

Considerable care should be exercised during preparation of specimens for case-depth determination by any of the mechanical methods. Serious errors can be introduced if the specimen has not been properly prepared. In the case of microhardness measurements, it is important to avoid cutting or grinding burns. It is always a good idea to use an etchant for burn detection as a general precaution, although this is almost never done in practice.

If the specimen is to be tested directly on a Rockwell scale, the cutoff technique done on it is critical. The hardness indentations must be made perpendicular to the surface, and in no case can the angle from parallel of the top and bottom surfaces be greater than 2 degrees. Otherwise, the readings will be erroneous.

When using microhardness methods, surface finish of the specimen is important and is a function of the indenter load. For accurate readings, the hardness impressions must not be affected by the surface condition. For example, a Knoop (500-gram) hardness profile can be performed on a specimen that was final polished on 600-grit (15-micron) paper. (Remember, the larger grit numbers correspond to smaller particle size and smoother surface finish with finer scratches.) The lighter the indenter load, however, the finer the polish necessary. Also, the hardness traverse should be started far enough below the surface of the case to ensure proper support from the metal between the center of the impression and the surface. A common error is to use too heavy an indenter load too close to the edge of the specimen, which results in deflection at the edge and a false (low) hardness value.

Another common error is to bunch the readings too close together. Making an indentation cold works the surface in the vicinity of the impression. If a subsequent reading is taken too close to a previous one, the resultant hardness value will be distorted (too high). For light and medium cases, up to 0.75 mm (0.030 inch), the indentations should be spaced along a 45-degree diagonal, a minimum of one indenter width apart. For deeper cases readings under one another are acceptable.

A typical Vickers or Knoop (500-gram) microhardness traverse would have an initial reading at 0.06 mm (0.0025 inch) and subsequent readings at 0.13-mm (0.005-inch) intervals to 0.75 mm (0.030 inch) and then at 0.25-mm (0.010-inch) intervals until readings above and below the 513 HV value are observed. Interpolation or additional indentations can be done to determine the exact value.

## Summary

Regardless of the technique used to determine total and effective case depth in carburized components, it is important that the method be consistent, accurate and correlate to actual physical and mechanical properties as they relate to the performance and characteristics of the part in service.

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# Tensile Testing

Hardness testing is one of the oldest and most reliable measures of whether or not a component part has been successfully heat treated. However, it should not be the only test conducted to confirm this is true. One of the most reliable mechanical tests to help us to predict the behavior of a component under various operating conditions is the simple tensile test. Let's learn more.

The tensile test (Fig. 1) allows us to measure a material's response to loading and deformation. By measuring the force required to elongate a specimen to its breaking point, material properties can be determined that will allow engineering designers and quality managers to predict how their materials and products will behave in their intended end-use applications.

Examples of products and industries that use tensile testing include fasteners (e.g., bolts, nuts, screws), seat-belt components, and tubing and pipe manufacturers to name a few. Tensile tests can predict pull-off force, peel and tear resistance and adhesion/bond strength. In the test, one end of a specimen is typically clamped in a load frame while the other end is subjected to a controlled displacement or a controlled load. A transducer or servo-drive connected in series with the specimen provides information about the displacement (d) as a function of the load (P) applied (or vice versa).

## A Little Theory

The concept of stress, strain and strength of materials is at the core of the engineering discipline. Mechanical properties such as yield strength, tensile strength, ductility, toughness, impact resistance, creep resistance, fatigue resistance, stiffness and others all influence the design, fabrication and service life of equipment.

The engineering measures for stress (se) and strain (ee) are determined from load and deflection readings using the original specimen cross-sectional area (AO) and length (LO). Formulas for these values are given as follows:

$$\sigma_e = \frac{P}{A_0} \qquad 1)$$

$$\varepsilon_e = \frac{\delta}{L_0} \qquad 2)$$

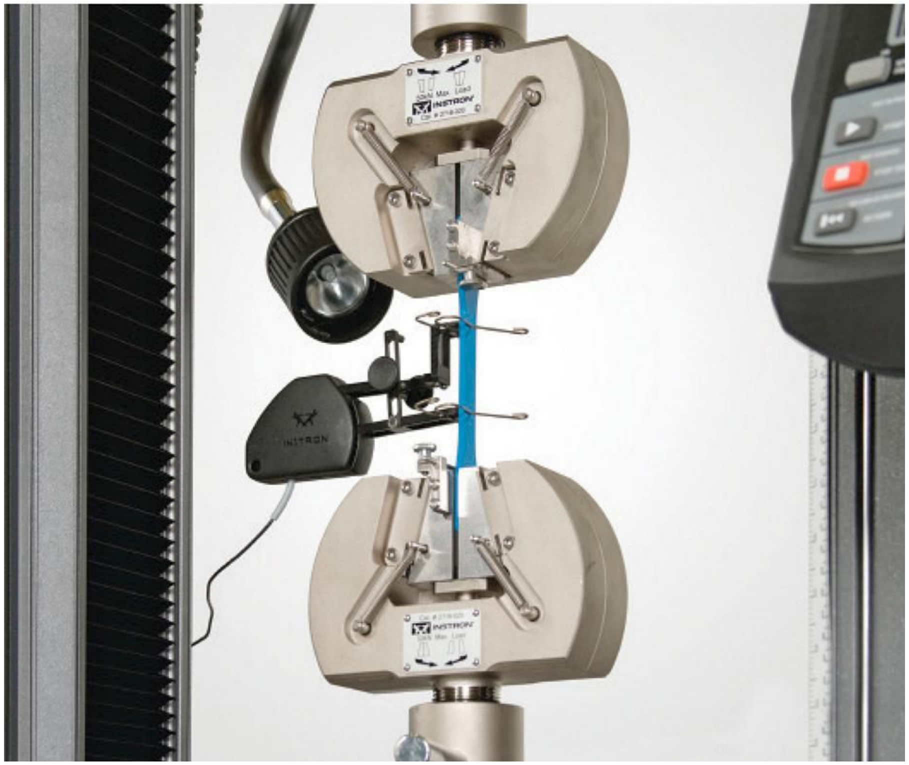

Fig. 1. Tensile-testing apparatus for fasteners[2]
(Photograph courtesy of Instron®)

## Stress-Strain Curves

Stress-strain curves (Fig. 2) can then be generated and divided into "regions" that are descriptive of what is happening on the microscopic level, namely:

- A. Elastic region
- B. Plastic region
  1. Yielding
  2. Strain hardening
  3. Necking
- C. Failure (fracture)

The shape and magnitude of the stress-strain curve of a metal (Fig. 3) will depend on its composition, heat treatment, prior history of plastic deformation and strain rate, temperature, and state of stress imposed during the testing. The parameters that are used to describe the stress-strain curve of a metal are the tensile strength, yield strength or yield point, percent elongation, and reduction of area. The first two are strength parameters, and the last two indicate ductility.

In the early (low strain) portion of the curve, many materials obey Hooke's law, which states that the deformation is, within a reasonable approximation, linearly proportional to the stress. As a result, the stress is proportional to strain with the constant of proportionality being the modulus of elasticity (i.e. Young's modulus).

As strain is increased, many materials eventually deviate from this linear proportionality. The point of departure is known as the proportional limit. This nonlinearity is usually associated with so-called "plastic" deformation (flow) in the

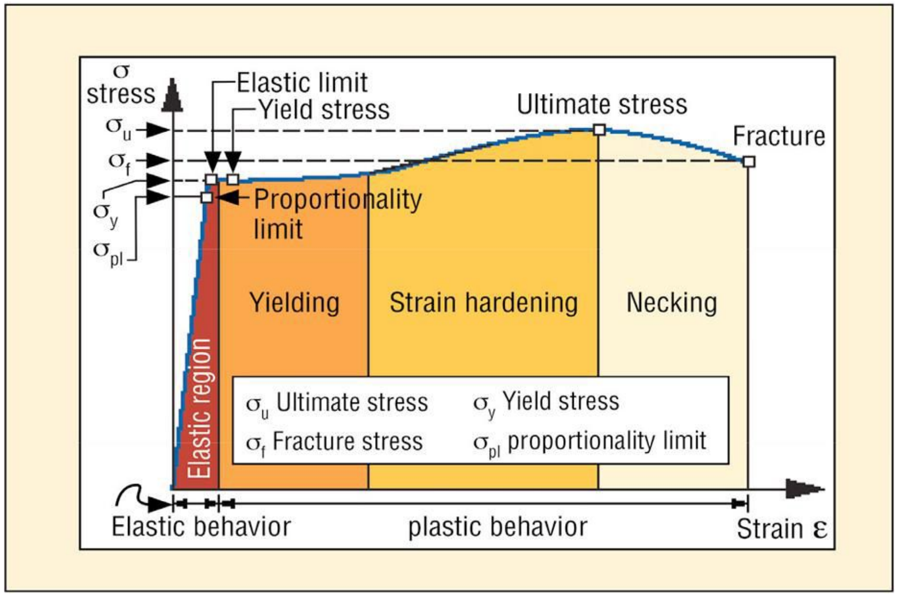

Fig. 2. Various regions and points on the stress-strain curve[3]

specimen. In this region, the material is undergoing rearrangement of its atoms (being moved to new equilibrium positions). The degree of plastic flow depends on the mobility mechanism, which in metals (i.e. crystalline materials) can arise from dislocation movement. Materials lacking this mobility (e.g., by having microstructural features that block dislocation motion) are usually brittle rather than ductile. The stress-strain curves for brittle materials are typically linear over their full range of strain, eventually fracturing without appreciable plastic flow.

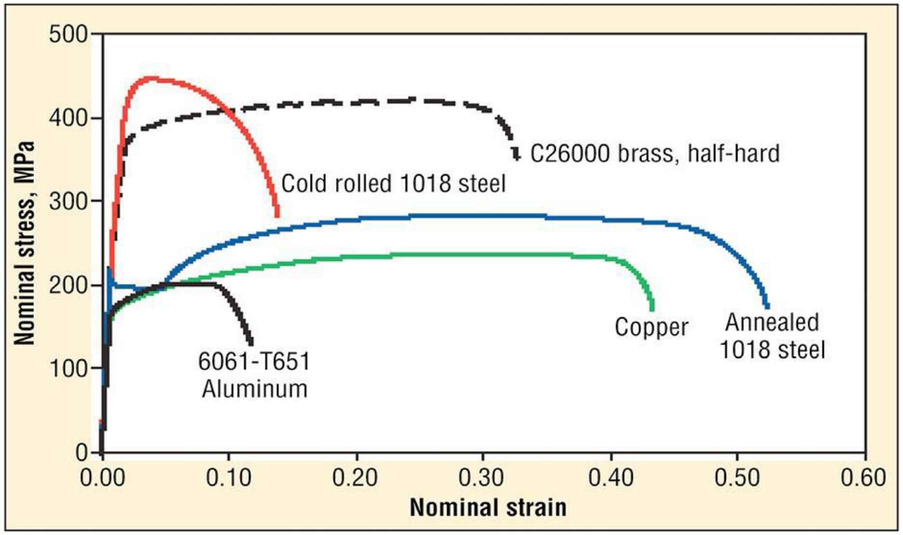

Fig. 3. Typical stress-strain curves for various materials[6]

As the plastic deformation continues above the yield strength, the engineering stress reaches a maximum point – the (ultimate) tensile strength (T.S.) of the material. The plastic deformation produces dislocations within the region in the curve between the yield strength and the tensile strength. The increasing dislocation density makes the plastic deformation harder.

This phenomenon is strain hardening, and it is a key factor in shaping the material by cold work. The microstructural rearrangements associated with plastic flow are usually not reversed when the load is removed, so the proportional limit is often the same as or at least close to the material's elastic limit. Elasticity is the property of complete and immediate recovery from an imposed displacement on release of the load, and the elastic limit is the value of stress at which the material experiences a permanent residual strain that is not lost on unloading.

The strain at failure occurs after the specimen fractures. It is the interaction point of the plastic recovery region in strain axis. It is known as ductility. The ductility is the percent elongation at failure and indicates the general ability of the material to be plastically deformed.

A closely related term is the yield stress (sy) of a material, which is the stress needed to induce plastic deformation. Since it is often difficult to pinpoint the exact stress at which plastic deformation begins, the yield stress is taken to be the stress needed to induce a specified amount of permanent strain, typically 0.2%, the so-called "offset" yield stress.

As most heat treater's know, for steel, hardness can be used to approximate the tensile strength of the material (Equations 3, 4) where HB is Brinell hardness (3,000 kgf load). This relationship is far from exact, however, and only a tensile test can reveal the true nature of the stress-strain relationship of a material.

$$(3a)\ TS(MPa)=3.55 \bullet HB (HB \le 175)$$
$$(3b)\ TS(psi)=515 \bullet HB (HB \le 175)$$

and

$$(4a)\ TS(MPa)=3.38 \bullet HB (HB > 175)$$
$$(4b)\ TS(psi)=490 \bullet HB (HB > 175)$$

Finally, recall that toughness measures the ability of a material to absorb energy and withstand shock up to fracture (i.e. the ability to absorb energy in the plastic range). In other words, toughness is the amount of energy per unit volume that a material can absorb before rupture and is represented by the area under the stress-strain curve. For further information, refer to The Heat Treat Doctor columns in Industrial Heating entitled "Toughness" (December 2010) and "Toughness Revised" (March 2011).

## In Layman's Terms

The stress-strain curve is logically divided into two distinct deformation regions corresponding to elastic deformation and plastic deformation. The elastic deformation is temporary and fully recovered when the load is removed. By contrast, the plastic deformation is permanent and not recovered when the load is removed (even though a small portion of the elastic part in the deformation is recovered).

A simple analogy to explain these concepts is the rubber band. When a rubber band is stretched and then released, we say that it is in the elastic region of the curve if it returns to its original shape. The point at which the force we use is great enough to prevent the rubber band from returning to its original shape is the yield point, beyond which we are in the plastic deformation region. Finally, when we stretch a rubber band beyaond the material's ultimate tensile strength, it snaps and we reach the fracture point.

## Tensile-Testing Standards

There are a number of tensile-testing standards, including:

- ASTM B913, D76, D1876, D3822, D412, D638, D828, E8
- BS 5G 178, BS EN 1895
- ISO 37, 527, 1924, 13934
- MIL-C-39029, MIL-T-7928

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# Toughness

Improving the strength/toughness relationship increases the usefulness of any engineered material (Fig. 1). Steel is no exception. As heat treaters, we need to understand what influences steel toughness and ask ourselves what tests best measure it. Let's learn more.

## What is Toughness?

Toughness is a fundamental material property measuring the ability of a material to absorb energy and withstand shock up to fracture; that is, the ability to absorb energy in the plastic range. In other words, toughness is the amount of energy per unit volume that a material can absorb before rupturing and is represented by the area under the (tensile) stress-strain curve (Fig. 2). In service, this loading often occurs in the form of (sudden) impact.

When considering toughness, one must make the distinction between impact toughness, which most often occurs under high strain-rate loading above the yield point, and fracture toughness, which generally occurs under lower strain-rate loading. All steels have different strength and ductility characteristics as a function of their composition (i.e. alloy design), and the key to good toughness is a balance between these properties and overall life-cycle cost.

Tough materials can absorb a considerable amount of energy before fracture, while brittle materials absorb very little. Comparing areas under each stress-strain curve reveals this difference. A material with high strength and high ductility will have more toughness than a material with low strength and low ductility. Recall that brittle materials may be strong, but they are not tough due to limited strain values.

## Why is Toughness Important?

Catastrophic failures are caused by a combination of inadequate material properties, improper design, poor manufacturing or fabrication processes, uneven or excessive loading, and pre-existing flaws. These failures can most often best be addressed by understanding the strength and toughness characteristics of a given material, including both its impact and fracture toughness.

## What Influences Toughness of a Material?

The general factors that influence toughness

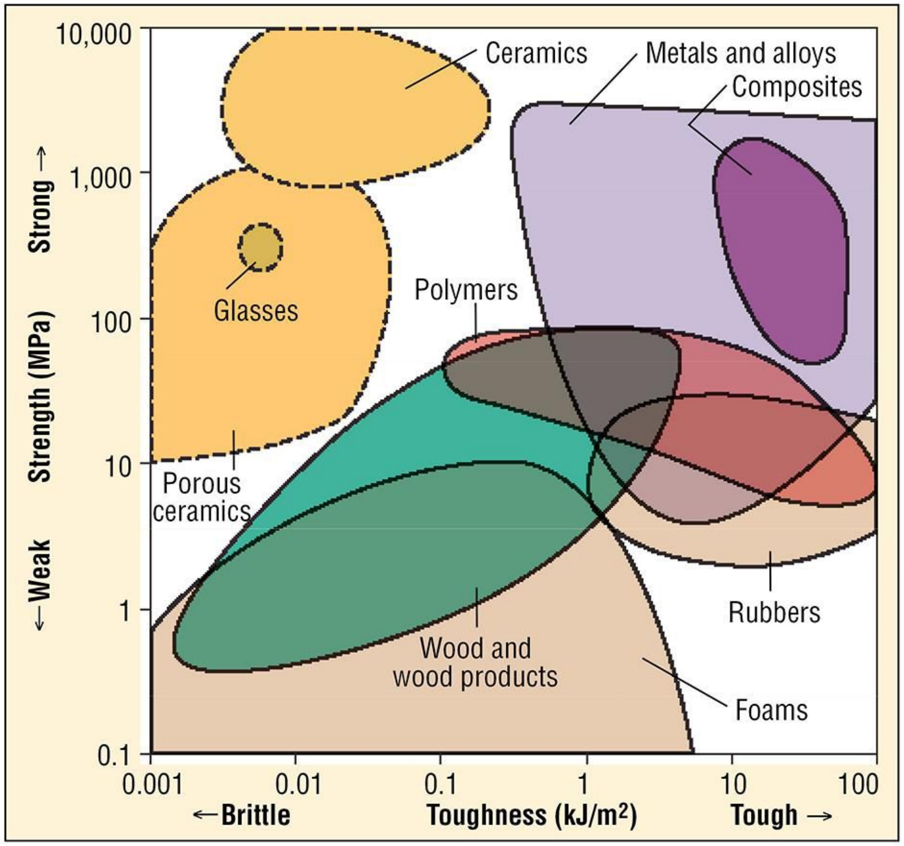

Fig. 1. Relative toughness (Gc) of various materials[1]

are alloying elements, fabrication techniques, microstructure, temper condition and service application (e.g., temperature, strain rate, strength-to-ductility ratio and the presence of stress concentrators).

There are several variables that have a profound influence on the toughness of a material. These include rate of loading (i.e. strain rate), temperature, distribution of stress, surface topography, and the presence or absence of any pre-existing flaws or cracks and their stress intensity.

A steel may possess satisfactory toughness under static loads but may fail under dynamic loads or impact. In general, ductility and, as a consequence, toughness decrease as the rate of loading increases. Most materials are more brittle at lower temperatures and more ductile at higher temperatures. Finally, the distribution of stress is critical. A material might display good toughness when the applied stress is uniaxial, but when a multiaxial stress state is produced due to the presence of a notch the material might not withstand the simultaneous elastic and plastic deformation in the various directions.

As a general rule, the lower the hardness and strength, the higher the ductility and toughness of a microstructure. However, embrittlement phenomena (e.g., quench embrittlement, temper embrittlement) are exceptions to this rule. For example, tempered martensite embrittlement (TME) lowers ductility and toughness as hardness decreases within a particular range of tempering temperature (Fig. 3). This is why after tempering of certain alloy steels such as 4140 or 4340 at temperature of 480–750°F (250-

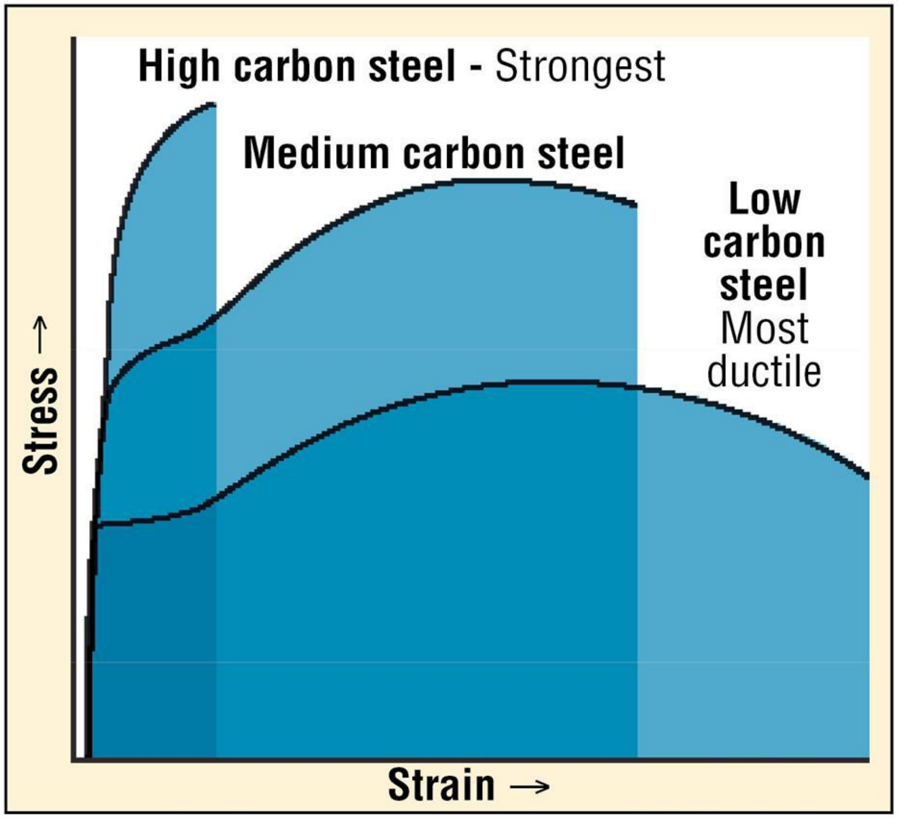

Fig. 2. Strength/toughness relationship[2]

400°C) the impact toughness is lower than that obtained on tempering at temperatures below 480°F (250°C) or above 750°F (400°C).

In crystalline materials, the toughness is strongly dependent on crystal structure. Face-centered-cubic (FCC) materials are typically ductile, while hexagonal-close-packed (HCP) materials tend to be brittle. Body-centered-cubic (BCC) materials often display dramatic variation in the mode of failure with temperature. Steels with ferritic microstructures have inherently lower toughness when tested below their ductile-to-brittle transition temperatures.

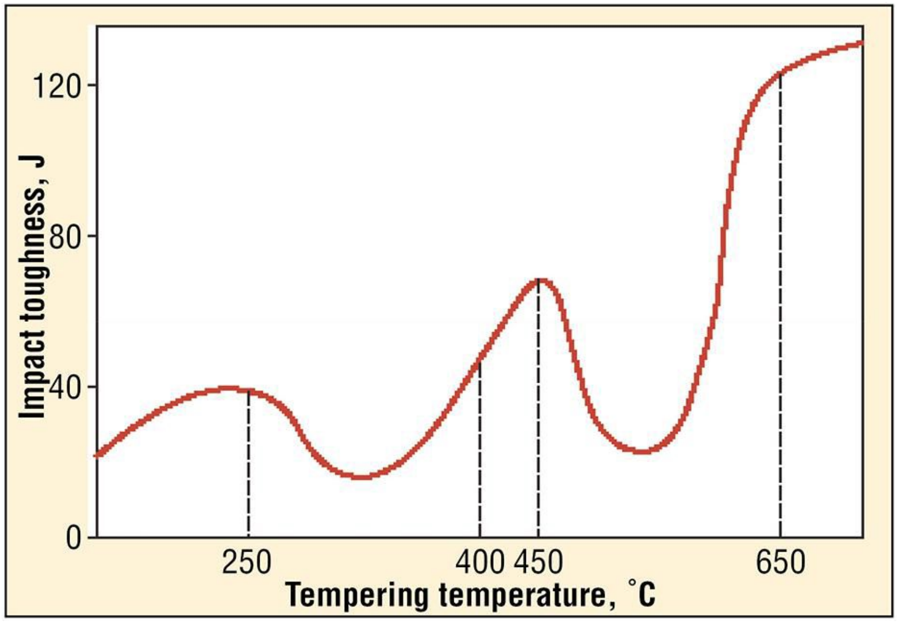

Fig. 3. Effect of tempering temperature on impact properties[3]

## Testing Methods

The toughness of a material can be measured by tensile testing, where the total area under its stress-strain curve measures, at low strain rates, reduction

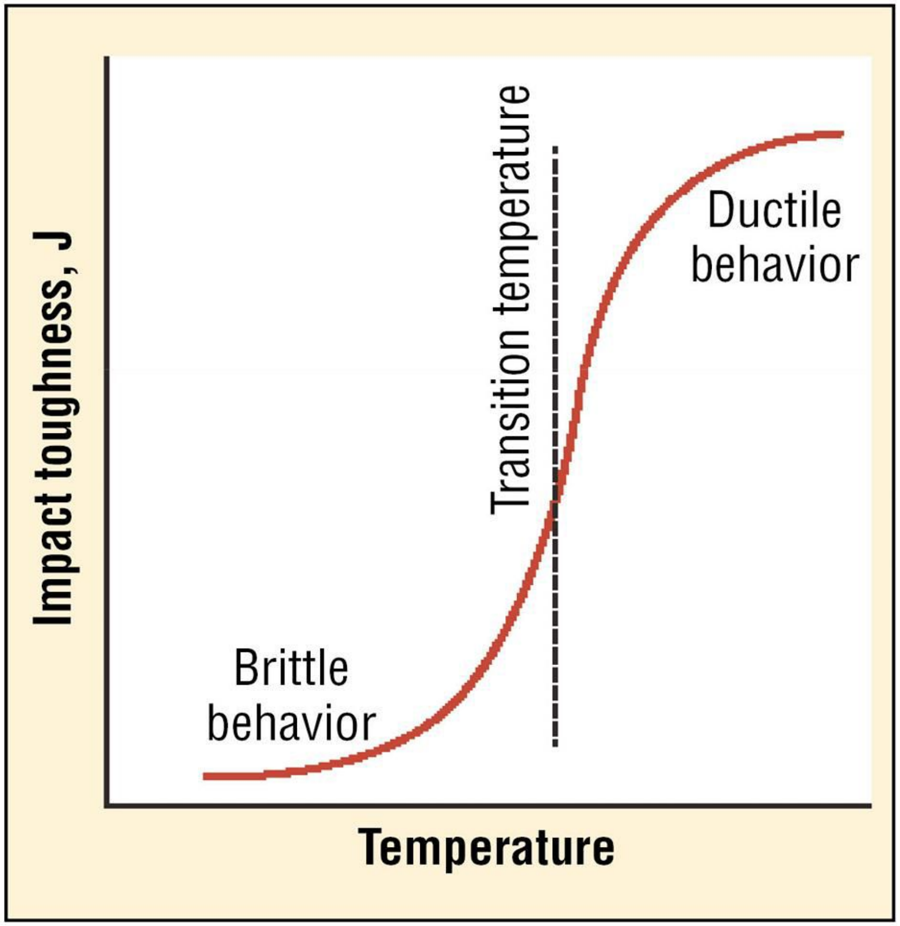

Fig. 4. Impact toughness as a function of temperature[2]

of area and total elongation – both parameters sensitive to fracture. This value, the so-called material toughness, equates to a slow absorption of energy by the material, and it has units of energy per volume.

A comparison of the relative magnitudes of the yield strength, ultimate tensile strength and percent elongation of different material will give a good indication of their relative toughness. Materials with high yield strength and high ductility have high toughness. Integrated stress-strain data is not readily available for most materials, so other test methods have been devised to help quantify toughness (Fig 4).

### Impact Toughness

The most common test for toughness is the Charpy V-notch impact test (previously Izod testing), which evaluates the effect of high strain-rate loading and a sharp notch on the energy absorbed for fracture. Impact toughness is the ability that a material possesses to absorb energy either with or without the presence of a stress intensifier such as a notch. Since toughness is greatly affected by temperature, these tests are often repeated several times with each specimen tested at a different temperature. The ductile-to-brittle transition temperature (DBTT) is often an extremely important consideration in the selection of a material. The use of alloys below their transition temperature is avoided due to the risk of catastrophic failure.

## Fracture Toughness

Fracture toughness (K1c) testing evaluates stress intensities required to propagate unstable fracture in front of a sharp crack under conditions of maximum constraint of plastic flow. It is an indication of the amount of stress required to propagate a pre-existing flaw due to processing, fabrication or end-use application. Flaws may appear as cracks, voids, non-metallic inclusions, weld defects, hot shortness, discontinuities, overheating during forging or a combination of several types. Fracture toughness generally depends on temperature, environment, loading rate, the composition of the material and its microstructure, together with geometric effects (constraints). Although it is possible to correlate Charpy energy with fracture toughness, a large degree of uncertainty is associated with correlations because they are empirical.

It is preferable to determine fracture toughness in a rigorous fashion, in terms of K (stress intensity factor), CTOD (crack tip opening displacement) or J (the J integral). ASTM E1290 covers CTOD testing, ASTM E1820 includes K, J & CTOD (including R-curves) and ASTM E1921 covers J testing to determine TO for ferritic steels.

## Future Column

Being a complex subject, there is more to discuss, such as the influence of alloying elements (e.g., carbon, nickel), microstructure, heat treatment, embrittlement phenomena and service conditions on toughness.

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# Toughness Revisited

Last time we learned something about the relationship between strength and toughness (Fig. 1).[1] A material may be strong and tough, exhibiting high strains if it ruptures under high loads. By contrast, brittle materials may be strong, but with limited strain values they are not tough. Generally speaking, strength indicates how much load/stress the material can sustain, while toughness indicates how much energy a material can absorb before rupturing.

In our previous discussion on toughness we also talked about the need to discuss the influence of alloying elements, microstructure, heat treatment, embrittlement phenomena and service conditions. Let's learn more.

## Effect of Alloying Elements on Toughness

Alloying elements play an important role in influencing a host of mechanical properties (Table 1), not the least of which is toughness. As we all know, steels contain various alloying elements (and impurities) that influence the formation of new phases as well as interact with the crystal structures of austenite, ferrite and cementite. Some of the alloying elements are austenite stabilizers (e.g., manganese and nickel), some are ferrite stabilizers (e.g., silicon, chromium and niobium) and still others are strong carbide formers (e.g., titanium, niobium, molybdenum and chromium). Of these effects, certain alloying elements (e.g., manganese, sulfur, aluminum, calcium and silicon) as well as oxygen influence the type, distribution and morphology of inclusions (e.g., oxides, sulfides, silicates and nitrides). There is a strong effect of inclusions on fracture and mechanical properties. Coarse inclusions also serve as fatigue initiation sites and have been reported to initiate cleavage fracture.

Here's a look at each individual element affecting toughness:[4]

- Calcium improves steel cleanliness by influencing the size, morphology and total number of inclusions; desulfurizes; and reduces the tendency toward directional properties. Calcium also modifies the shape of any remaining sulfide inclusions so as to be less detrimental to mechanical properties. Calcium has no effect on transformations occurring

**Table 1.** Alloying elements with a favorable influence on mechanical properties (alphabetical listing)[3]

| Hardenability | Strength | Toughness | Machinability |
|---|---|---|---|
| Boron | Carbon | Calcium | Lead |
| Carbon | Cobalt | Cerium | Manganese |
| Chromium | Chromium | Chromium | Phosphorous |
| Manganese | Copper | Magnesium | Selenium |
| Molybdenum | Manganese | Molybdenum | Sulfur |
| Phosphorous | Molybdenum | Nickel | Tellurium |
| Titanium | Nickel | Niobium | |
| | Niobium | Tantalum | |
| | Phosphorous | Tellurium | |
| | Silicon | Vanadium | |
| | Tantalum | Zirconium | |
| | Tungsten | | |
| | Vanadium | | |

during heat treatment.

- Cerium, while itself a potent deoxidizer and desulfurizer, is added to control the shape of inclusions in steel that has already been deoxidized and desulfurized by other additives. Cerium does not take part in heat-treating reactions.
- Chromium has a tendency to form hard and stable carbides. Chromium strongly affects hardenability, wear resistance, corrosion resistance, resistance to hydrogen attack and resistance to softening at elevated temperature (i.e. greater creep and stress-rupture properties). Chromium has a strong affinity for both carbon and nitrogen.
- Magnesium desulfurizes steel, and by its ability to influence sulfide inclusions improves ductility, formability and directional uniformity.
- Molybdenum is a potent hardenability agent that retards softening at elevated temperature and improves corrosion resistance. During heat treatment, molybdenum steels have a tendency toward surface decarburization. Molybdenum is a strong carbide former and reduces the tendency toward temper embrittlement.
- Nickel is an austenite stabilizer, that is, the $A_3$ temperature will be depressed and in the presence of carbon so too will the $A_1$ temperature. Nickel is a solid-solution strengthener, a weak hardenability agent and promotes high toughness (often in combination with chromium and vanadium), especially at low temperatures. While its effects are not strong, nickel does have some influence on heat-treating transformations retarding both pearlite and, to a greater extent, bainite reactions.

- Niobium has two principal uses in steels: as a grain refiner and for the formation of extremely hard and stable carbides and carbonitrides. Niobium has a strong ability to remove carbon from solid solution and thus has a negative effect on hardenability.
- Tantalum improves strength and forms fine precipitates, but increasing tantalum content has a negative effect on ductility.
- Tellurium improves machinability but has no effect on the transformations occurring during heat treatment. Tellurium is a grain refiner and can be added to steel to influence the size, shape, distribution and morphology of sulfide inclusions, the result of which is an improvement in transverse toughness and certain mechanical properties.
- Vanadium promotes fine grain size (i.e. retards grain growth during austenitizing), increases hardenability (when dissolved in austenite, although its effectiveness is diminished somewhat by its high affinity for carbon) and improves wear resistance through the precipitation of carbides and nitrides.
- Zirconium forms stable compounds with oxygen, sulfur, nitrogen and carbon, thus helping to control nonmetallic inclusions and the fixation of nitrogen (primarily in boron steels). The presence of zirconium in quenched-and-tempered steels also reduces grain coarsening/sensitivity and permits the use of higher hardening or carburizing temperatures. Zirconium raises the yield/tensile ratio and improves ductility and impact strength, particularly in the transverse direction. In highly alloyed steels, ductility decreases while hardness improves. Its usefulness is limited for economic and processing reasons.

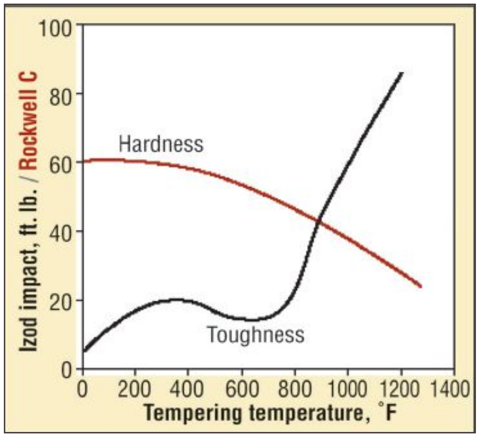

Fig. 1. Relationship of hardness and toughness of 4140 steel after tempering for one hour[2]

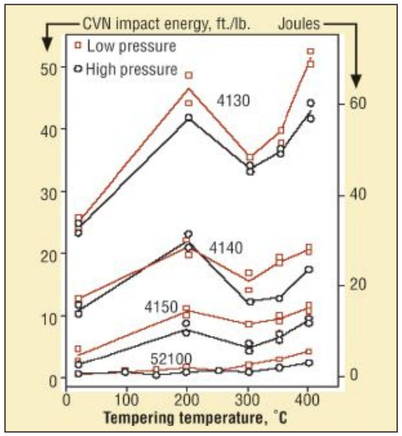

Fig. 2. Charpy V-notch energy absorbed in fracture of 41xx-series and 52100 steels tempered at various temperatures[5]

## Embrittlement Phenomena[5]

High-strength quench and-tempered steels are

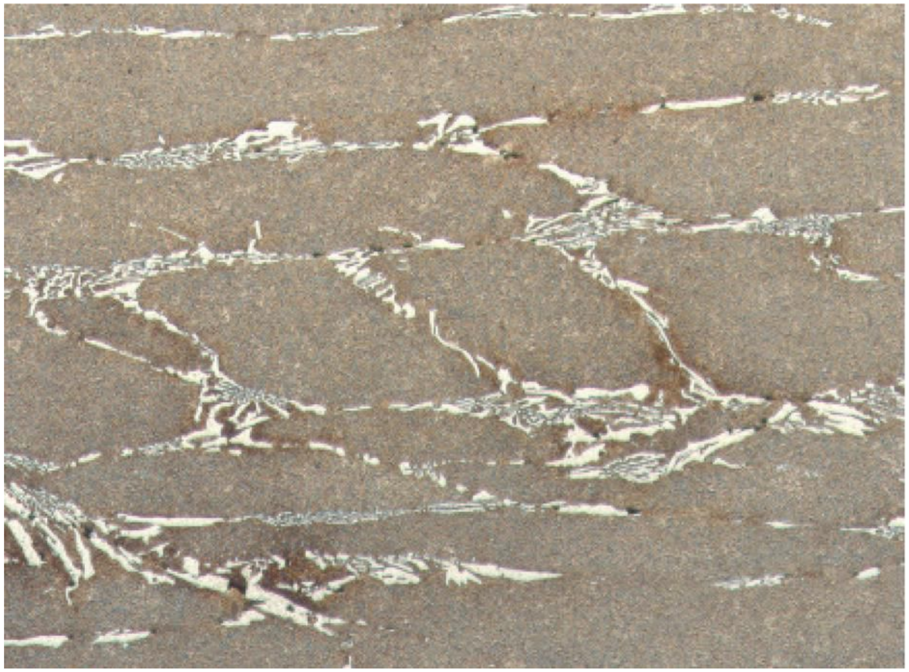

Fig. 3. Hot-working problems on a D2 roll (Photograph courtesy of Aston Metallurgical Services Co., Inc.)

subject to a variety of embrittlement phenomena including quench embrittlement, tempered-martensite embrittlement, temper embrittlement, hydrogen embrittlement and liquid-metal embrittlement, to name a few. In addition, stainless steels are affected by sigma-phase embrittlement.

The steel's carbon content has a significant impact on toughness under various tempering conditions (Fig. 2). The effects on toughness can be categorized as follows:

- Tempered martensite (blue) embrittlement (TME) is irreversible and results in a loss of room-temperature impact toughness and fracture resistance. It manifests itself by ductile, cleavage and intergranular modes of fracture. Tempering in the range of 250-400°C (480-750°F) can result in TME. The interactive effect of both tempering temperature and carbon level on the magnitude of the temper-embrittlement effect is seen in Figure 2.
- Temper embrittlement (TE) results in the loss of ductility (or the increase in the ductile-to-brittle transition temperature) after tempering in the range of 375-575°C (700-1070°F) or slow cooling through this range. Impurities such as phosphorous, tin, arsenic and antimony strongly influence the susceptibility of a material to TE. In general, heating to temperatures above the embrittlement range for several hours followed by rapid cooling can reverse the effects.
- Quench embrittlement is an intergranular mechanism of brittle fracture, especially in high-carbon (≥ 0.50% C) steels during austenitizing and quenching. Tempering is not required. It is similar to quench cracking, which is due to high surface tensile stresses during quenching. Carbon and phosphorous play a significant role.

- Sigma-phase embrittlement results from the precipitation of an iron-chromium compound after holding austenitic or ferritic stainless steels for long periods of time in the range of 560-980°C (1050-1800°F). Slow cooling through the range of 1040-650°C (1900-1200°F) produces the same effect as does quenching from this range followed by subsequent heating in the range of 560-850°C (850-1560°F).

## Effects of Primary Processing on Toughness

In addition to a large number of inclusions, cracking during solidification and hot working may introduce flaws that compromise performance. In the case of hot working, incipient melting, precipitation of particles or ferrite formation in the austenite grain boundaries and resultant microvoid formation are believed to be the mechanisms involved. By way of example, the microstructure of a hot-worked 5,000-pound roll (Fig. 3) reveals large chromium carbides in an intergranular network, creating a part with extremely poor fracture toughness.

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# Impact Testing (Part 1)

Strength and toughness relationships are perhaps the most important ones when it comes to advances in materials and engineered products. Toughness is often determined by impact testing, and every heat treater needs to know how these tests are performed and what they mean. Let's learn more.

## Brittle Failure

One of the surprising phenomena in the metals industry is that normally ductile materials, such as mild steel, can become brittle under certain conditions. There are three basic factors that contribute to a brittle-type (or cleavage-type) fracture, namely: the presence of a triaxial stress state (defined later), a low temperature (relative to the ductile-to-brittle transition temperature) for the material in question and a high strain rate or rapid rate of loading.[2]

Interestingly, not all three need be present at the same time for a brittle fracture to occur. For example, the presence of low temperature, stress risers – such as sudden changes in part geometry – or other "metallurgical notches" – such as nonmetallic inclusions or surface defects/imperfections (seams, laps, etc.) – is often responsible for the brittle failures experienced in service. Other three-dimensional (volumetric) defects (e.g., porosity or other macro-imperfections), while they create a lesser stress notch effect, can also amplify stress since they reduce the load-bearing area. The following characteristics must be taken into account when assessing the significance of a defect:[2]

- Size
- Sharpness
- Orientation (with respect to the principle working stress and residual stress)
- Location

Furthermore, the tendency toward brittle fracture cannot necessarily be determined by tension or torsion tests, which are usually conducted at slower strain rates. Materials that have similar tensile properties can show pronounced differences in toughness. For this reason, impact tests at high rates of loading have been developed.

## Triaxial Stress

Engineered components often experience more than one type of stress at the same time. This is known

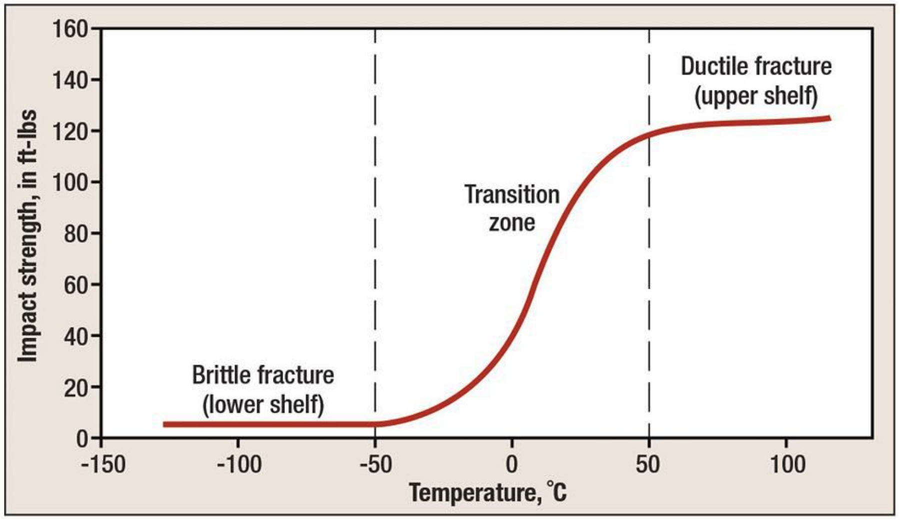

Fig. 1. Ductile-to-brittle fracture transition curve[6]

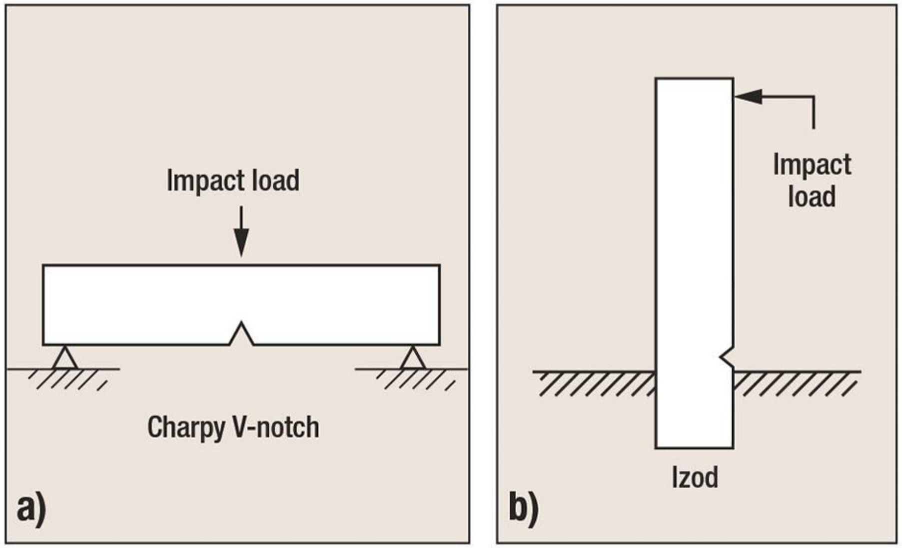

Fig. 2. Method of loading for impact tests[2]
(a) Charpy; (b) Izod

as the combined stress state. In normal and shear stress, the magnitude of this stress is maximum for surfaces that are perpendicular to a given loading direction (and, in some instances, zero across any surfaces that are parallel to that direction). Uniaxial, biaxial and triaxial stresses refer to conditions where stress is applied on one, two or all three of the principal axes of a component. Thus, triaxial stress has normal and shear stresses that are applied in three dimensions or planes (i.e., the stress is nonzero across every surface element).

## Impact Tests

Impact testing provides us with a simple method of ascertaining the change in the fracture mode of a material as a function of temperature (Fig. 1). Note that in the graph shown there isn't a sharp transition from ductile to brittle failure modes but rather one that occurs over an extended range of temperatures. An analysis of the fracture surface of an impact specimen can characterize the fracture mode.[4,5]

Impact tests measure both the energy required and the resistance to failure of a material subjected to a sudden applied load. The test measures the impact energy; that is, the energy absorbed by the material prior to fracture. The two most common tests are the Charpy test and the Izod test.

Fig. 3. Instrumented impact pendulum system for Charpy testing (courtesy of Instron)

## The Charpy Test

This test is named for its inventor, Georges Augustin Albert Charpy (1865-1945). The Charpy test measures the energy absorbed by a standard notched specimen while breaking under a three-point bending impact load. The most common method of measuring impact energy in steels today is the Charpy V-notch test (Fig. 2a). Other notch configurations (U-notch, keyhole, etc.) can be used. The importance of the Charpy impact tests lies in the fact that it can reproduce the ductile-brittle transition transformation (DBTT) in essentially the same temperature range as it is actually observed in engineering structures.[5]

## What does the Charpy test involve?

The Charpy tester (Fig. 3) involves striking a suitable test piece with a shaped tup mounted at the end of a pendulum. The test piece is fixed at both ends in a horizontal orientation and the striker impacts the test piece immediately behind a machined notch. In this case the "V" notch points opposite the direction of load approach. A significant amount of testing is conducted at room temperature. When DBTT testing is required, the testing usually involves six temperatures, one of which is room temperature. The non-room-temperature testing involves removing a test specimen from its medium and positioning it on the specimen supports. The pendulum is to be released as vibration-free as possible within five seconds after the material is removed from the thermal medium.

Fracturing the specimen removes energy from the hammer, and the height at which the hammer rises after the specimen is broken is measured on the tester. For a ductile fracture, the energy expended is high, and the energy will be low for a brittle fracture. The information obtainable from this test includes the impact energy, lateral expansion and fracture appearance.

## The Izod Test

The Izod impact test was named for its inventor, Edwin Gilbert Izod (1876-1946), and consists of a pendulum with a determined weight at the end of its arm swinging down in a circular arc and striking a specimen while it is held securely in a vertical position (Fig. 2b). It is a cantilever beam test in which the notch is oriented to point in the direction of load approach. The impact strength is determined by the loss of energy of the pendulum as calculated by precisely measuring the loss of height in the pendulum's swing. The specimen that is usually notched is gripped at one end only, which is the principal difference between it and the Charpy test.

The principal advantage of this test is that a single specimen can be used multiple times since the ends are broken off one at a time. The principal limitation is the lengthy setup time required. Thus, both elevated- and low-temperature testing are not options, which is a major disadvantage due to the importance of the ductile-to-brittle transition temperature characteristics of most steels.

The size and shape of the specimen varies according to what materials are being tested. Specimens of metals are usually square, and polymers are usually rectangular and struck perpendicular to the long axis of the rectangle. The specimen-holding fixture is usually part of the machine and, as such, cannot be readily cooled (or heated). For this reason, Izod testing is not recommended at other than room temperature. The information obtainable from this test includes the impact energy, lateral expansion and fracture appearance.

**Next Time:** A discussion about the variables that affect transition temperature and a look toward the future of impact testing.

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# Impact Testing (Part 2)

The subject of impact testing continues from last month's discussion by looking at the Charpy "V" notch test in more detail. Let's learn more.

## Charpy Test Specimens

Charpy test specimens normally measure 55 mm x 10 mm x 10 mm and have a notch machined across one of the larger faces. The basic types of notches are the "V" notch, keyhole notch and "U" notch (Fig. 1). The V-shaped notch is 2 mm deep, with 45° included angle and 0.25-mm radius at its root. The U-shaped notch is 5 mm deep with vertical sides and a 1-mm radius U-shaped root. The keyhole notch is also 5 mm deep and contains a 2-mm-diameter hole and vertical sides. The notch shape, depth and tip radius are, therefore, important test parameters.

Since the base of the notch is in a state of triaxial tension (Part 1) and the rate of loading is 10 million times faster than a standard tensile test, the notch serves as a stress concentration zone. Some materials are also more sensitive to the presence of a notch than others.

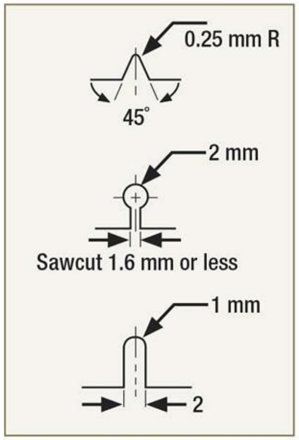

Fig. 1. Types of notches[8]

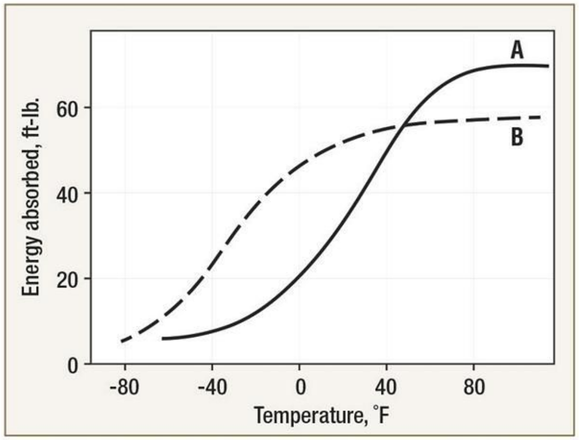

Fig. 2. The influence of temperature on determining the transition temperature[2]

## What is impact energy?

Impact energy is a measure of the work done to fracture a test specimen when subjected to a high instantaneous shock load. In a Charpy test, when the striker impacts the specimen, the specimen absorbs the energy of impact and begins to yield, with plastic deformation (and subsequent work hardening) occurring at the notch. When the specimen can absorb no more energy, fracture occurs.

## Determination of Charpy Impact Energy

At the point of impact, the striker has a known amount of kinetic energy. The impact energy is calculated based on the height to which the striker would have risen if no test specimen was in place compared to the height to which the striker actually rose. Prior to fracture, tough materials absorb relatively higher amounts of energy than brittle materials, which absorb much lower amounts of energy.

Factors that affect the Charpy impact energy of a specimen include:

- Yield strength and ductility
- Stress intensity for the type, size and depth of notch employed
- Temperature
- Strain rate
- Fracture mechanism

## Yield Strength and Ductility

For a given material, the impact energy will generally decrease if the yield strength is increased (i.e., if the material undergoes processing of some nature that makes it more brittle and less able to resist plastic deformation). Such processes may include various types of heat treatments, cold working, etc.

Instrumented impact tester (courtesy of Instron)

## Temperature and Strain Rate

Most of the impact energy is absorbed by means of plastic deformation during the yielding of the specimen. Therefore, factors that affect the yield behavior and ductility of the material, such as temperature and strain rate, will influence the impact energy. This type of behavior is more prominent in materials with a body-centered cubic structure (e.g., martensite, ferrite), where lowering the temperature reduces ductility more markedly than for face-centered cubic structures (e.g., austenite).

The notch-bar impact test over a range of temperatures is more meaningful than at a single temperature. Multi-test temperatures at above and below room temperature are employed to determine the ductile-to-brittle transition temperature of a given material (Fig. 2). Steel A shows higher notch toughness at room temperature, but its transition temperature is higher than that of steel B. The material with the lowest transition temperature is generally preferred.[2]

## Fracture Mechanism

Metals tend to fail in overload by one of two fracture modes: either ductile rupture by microvoid coalescence or brittle fracture via cleavage. Microvoid coalescence occurs when voids form and strain increases, causing these voids to eventually join together, and failure results. By contrast, cleavage occurs via shear along specific crystal planes. Of the two fracture modes, cleavage involves far less plastic deformation and hence absorbs far less fracture energy.[4,5]

## Variables Affecting Transition Temperature

The transition temperature in steel can be affected by a number of factors, one of the most interesting of which is the microstructure. If we compare a quenched-and-tempered microstructure with that of a normalized-and-tempered microstructure, we can see the effect of these different heat treatments on the impact strength and ductile-to-brittle transition temperature (DBTT) characteristics of 4340 medium-carbon alloy steel with high hardenability (Fig. 3). While both heat treatments produced an identical hardness, the microstructure of the quenched-and-tempered material consisted of tempered martensite and fine spheroidized alloy carbides. The normalized structure may consist of some combination of pearlite (in this case), bainite or ferrite, depending on the size and cooling rate of the heat-treated component. As one quickly observes, the transition temperature is approximately 150˚C (300˚F) lower for the quenched-and-tempered microstructure.

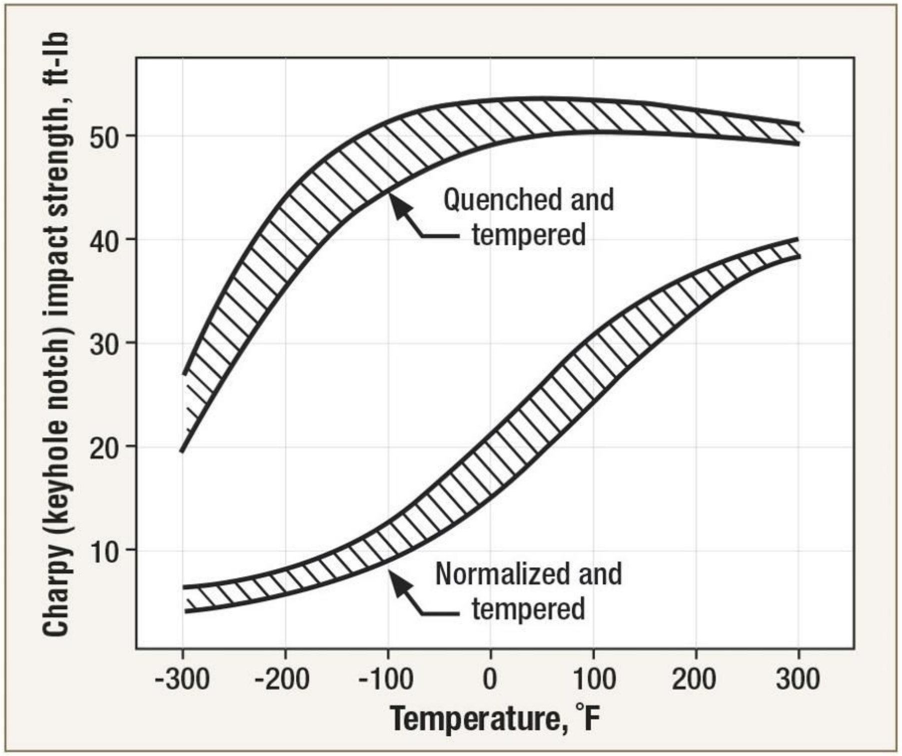

Fig. 3. Effect of microstructure/heat treatment on impact strength (note: bands indicate scatter of experimental measurements.)[3]

The chemistry of the steel also plays a prominent role. For example, the transition temperature for a 1% manganese, 0.30% silicon mild-carbon steel rises rapidly with increasing carbon content. Thus (all other factors being equal), the higher the carbon content, the more prone a material is to brittle fracture, especially near room temperature and below. Phosphorous has an even stronger effect on raising the DBTT, while increasing manganese contents tends to lower it. Other elements may play important roles, depending on the composition of the steel.

## Trends

Impact testing has changed very little over the decades. Until the mid-20th century, there was greater use of Izod testing and keyhole-notch Charpy testing than today. Charpy V-notch is currently the predominant test method, although some U-notch and unnotched specimens are also used in specific applications. The key difference today is the way in which the data that comes from the test equipment is handled.

ASTM E23 (Standard Test Method for Notched Bar Impact Testing of Metallic Materials), perhaps the most widely adopted standard on metals impact testing, establishes guidelines for test specimens (e.g., configuration, orientation, machining), preparation of the apparatus, test-temperature conditions and actual test procedures.

While mechanical pointers are still in use, many equipment manufacturers also include a rotary encoder on the machine axle to collect data. This data can then be used not only to determine the height to which the hammer rose but also to calculate the impact speed of the hammer. The impact values can instantly be displayed on the screen.

One new trend, which is currently being driven by the nuclear industry, is toward a fully instrumented impact tester. In these machines, the striking tup is equipped with strain gauges that are linked to high-speed data-capture equipment. A typical impact test is over in approximately 10 milliseconds or, quite literally, the blink of an eye. With this sophisticated equipment, however, we can capture up to 20,000 data points during this test. This data can be gathered to generate a graph that represents the striking tup position as it travels through the specimen.

Finally, it is interesting that this new technology will not fundamentally change the way in which the test is performed or the equipment required to perform it. Instead, newly developed technology will provide us with more repeatability, greater accuracy and higher resolution, as well as improved speed with respect to interpretation of results.

**Next Time:** How to compare Charpy and Izod values.

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# Impact Testing (Part 3): Comparing Izod and Charpy Data

The Doctor has always been intrigued by the question of how to compare the impact values obtained by Izod testing methods with those of Charpy testing methods. A great deal of data, albeit older (mid-20th century), has been collected using Izod testing, and it would be of great value to be able to use it today in some fashion. The time has come to find out how. Let's learn more.

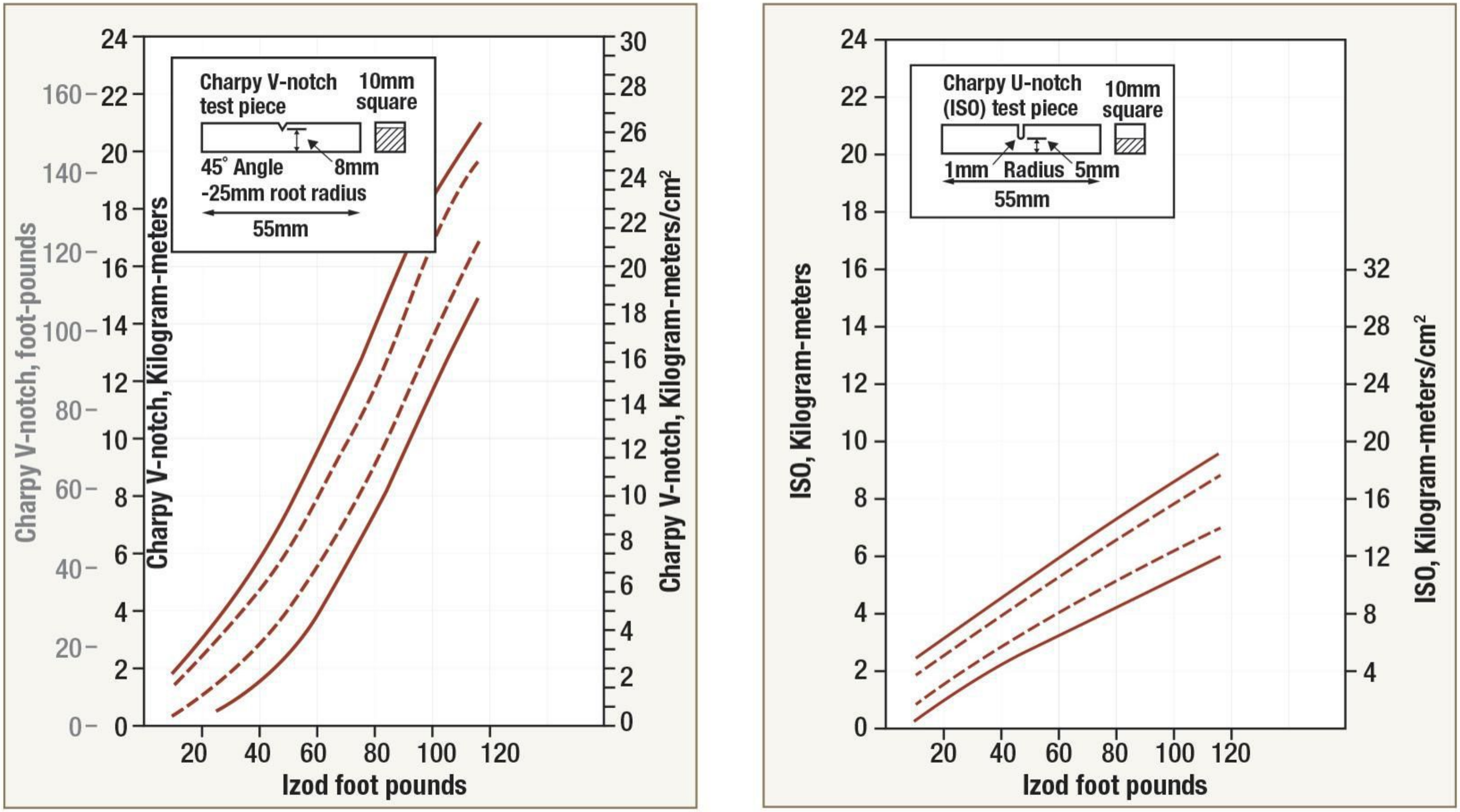
In the studies presented here, a cautionary emphasis must be placed on interpreting them. While we are dealing with specimens of the same cross section, the size of the cross section under the notch may be different as well as the stress intensity of each given notch geometry. The information in these investigations can be very helpful in rank ordering materials with individual processing conditions/properties and making educated estimates.[7]

## English Steel Study

The English Steel Corporation[7] performed one of the most comprehensive comparison studies. Izod data was compared with Charpy data from four different types of Charpy specimens (V notch, Mesnager U notch 2 mm deep, DVM U notch 3 mm deep, and the commonly used ISO U notch 5 mm deep), two of which are presented here (Figs. 1-2). The study also emphasized that a definite relationship between methods does not exist, the graphs must be considered approximate relationships and the data should not be used for comparing or compiling specifications.[7]

## TimkenSteel Study

A study was conducted in the 1940s when both Izod and Charpy keyhole specimens were used extensively. It is unique in that the data are one-to-one values obtained on the exact same orders/heats of steel and heat treatment (liquid-quenched and tempered). For several months when an order was produced requiring one of the test types, both specimen types were machined and tested. When examining this data (Fig. 3) one may ask, Why would the Charpy U-notch data have a lower value than the Charpy V-notch data? The answer lies in the volume of material being tested – the U-notch being 5 mm deep while the V-notch is only 2 mm deep. The reader is directed to ASTM E 23 for more in-depth discussion. Note also that under brittle conditions up to 13.5-20.4 J (10-15 foot-pounds), the two methods were nearly identical.[9]



Figs. 1 and 2. Comparison of impact values obtained with the British Standard Izod and various alternative methods of testing (Charpy V-notch and Charpy U-Notch 5 mm deep). The inner bands represent the area within which 50% of the data is expected to fall, while the outer bands cover approximately 80% of the expected results.[8] (reprinted by permission of Elsevier Publishing)

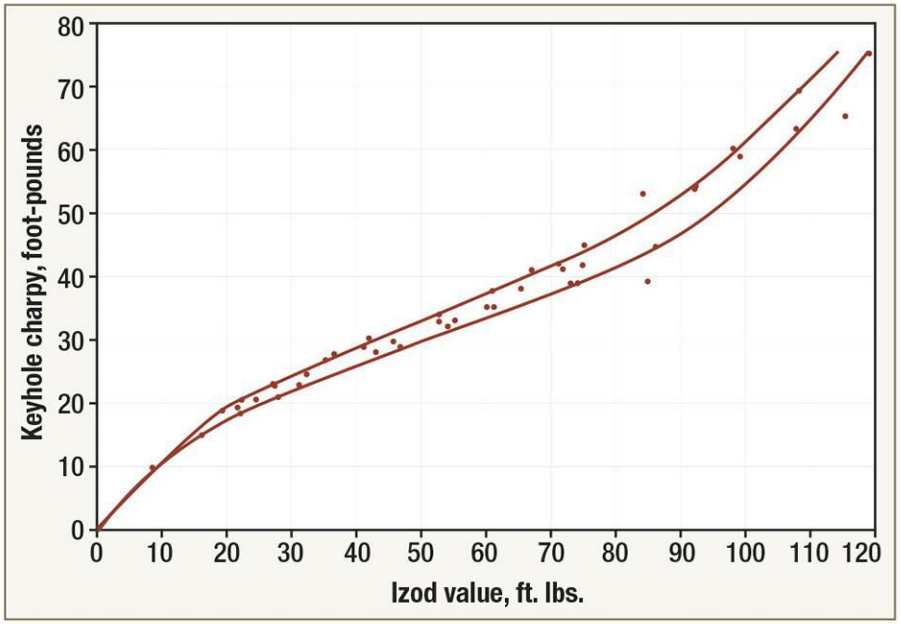


Fig. 3. Comparison of Izod and Charpy keyhole impact results for quenched-and-tempered steels[9]

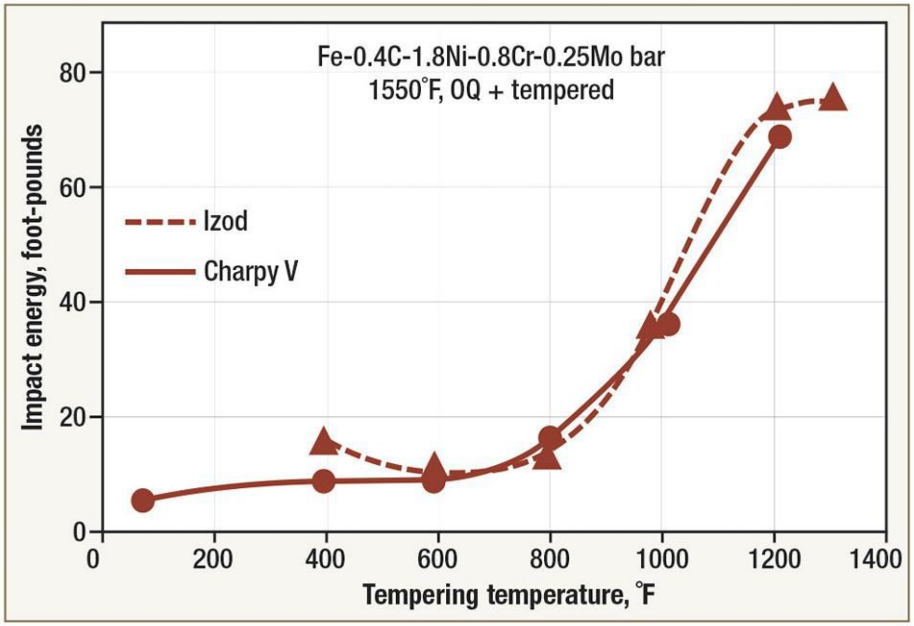


Fig. 4. Effect of tempering temperature on impact properties of oil-quenched bar at room temperature[10] (reprinted by permission of CINDAS LLC)

## 4340-Type Steel Study

TimkenSteel also conducted a one-off study of the effect of tempering temperature on the toughness of 4340-type steel using Charpy V test data. They then compared it to Izod data that had been published in the Alloy Digest. This graph was published in the Aerospace Structural Metals Handbook[9] and is reproduced here (Fig. 4). In this instance, the data were highly correlated, but one must keep in mind that the data represents different heats of steel produced years apart (1982 vs. 1954) and heat treated in separate processing equipment.

## Special Note

Discussion within ASTM Standard E 23[7] notes the following in regard to the comparison of Izod and Charpy data, namely: “General correlation between the energy values obtained with specimens of different size or shape is not feasible, but limited correlations may be established for specification purposes on the basis of special studies of particular materials and particular specimens.”

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## Final Thoughts

A few of the comparative studies between Izod and Charpy Impact values have been described. As the graphical representations show, the data will generally not fall in a one-to-one relationship. There are many reasons for the variances that can and will likely be experienced. Nonetheless, the data are both interesting and quite useful when applied with appropriate caution and consideration. The reader is urged to consult ASTM E 23 (latest revision) for additional detail and study the references provided (in particular numbers 8 and 10), which are outstanding sources for a tremendous variety of mechanical-property data.

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# Understanding Component Failures (Part 1: Mechanisms)

When a component part fails, it is only natural to ask why and then strive to determine the root cause. Gathering all possible information about the damage event and performing a thorough failure analysis is a critical first step in the process. This type of information helps the heat treater create a set of do's and don'ts that are invaluable in avoiding a repetition of the problem. Let's learn more.

In simplest terms, a failure is the inability of a component part to perform its intended function (Fig. 1). In service, components experience different types of conditions/environments, damage mechanisms and applied loading, including tension, compression, bending, torsion and mixed modes (combinations). The failures that result may be categorized in a broad sense as those related to fracture, wear, corrosion and dimensional change/distortion. As heat treaters, we must also consider that residual stresses can often play an important role.

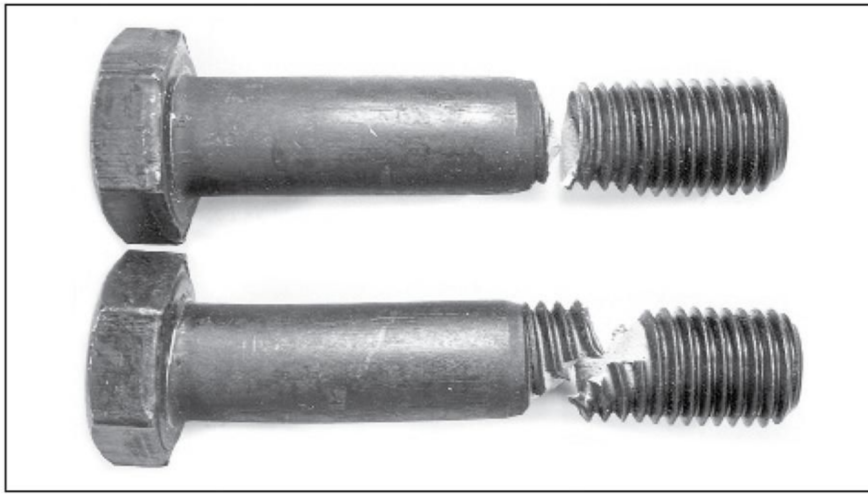
(Courtesy of George Vander Voort Consulting)

Fig. 1. Failed ASTM A490 bolts from structural beams in a power plant

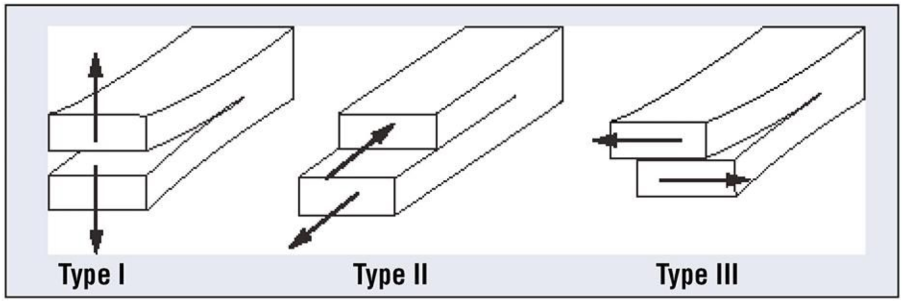

Fig. 2. Examples of fracture loading/opening modes (Type I = tearing, Type II = shear via edge sliding, Type III = shear via screw sliding)[2]

## Types of Fracture: Macroscopic Scale[4]

Applied loads (Fig. 2) may be unidirectional or multi-directional in nature and occur singularly or in combination. The result is a macroscopic stress

Fig. 3. A crack surface illustrating multiple initiations (origins), each with their own fatigue propagation (Courtesy of Aliya Analytical, Inc.)

state comprised of normal stress (perpendicular to the surface) and/or shear stress (parallel to the surface). In combination with the other application conditions, the result is one of four primary modes of fracture: dimpled rupture (also called microvoid coalescence), cleavage, decohesive rupture and fatigue. These will be discussed in more detail in Part 2.

## Types of Fracture: Microscopic Scale

Virtually all engineering metals are polycrystalline. As a result, the two basic modes of deformation/fracture (under single loading) are shear and cleavage (Table 1). The shear mechanism, which occurs by sliding along specific crystallographic planes, is the basis for the macroscopic modes of elastic and plastic deformation. The cleavage mechanism occurs very suddenly via a splitting action of the planes with very little deformation involved. Both of these micro mechanisms primarily result in transgranular (through the grains) fracture.

## Ductile and Brittle Fractures

Numerous factors influence whether a fracture will behave in a ductile or brittle manner (Table 2). In ductile materials, plastic deformation occurs when the shear stress exceeds the shear strength before another mode of fracture can occur, with necking typically in evidence before final fracture. Brittle fractures occur suddenly and exhibit very little, if any, deformation before final fracture.

Ductile fractures typically have the following characteristics:[1]

- Considerable plastic or permanent deformation in the failure region

**Table 1. Differences between shear and cleavage fracture**[1]

| Metric | Shear | Cleavage |
|---|---|---|
| Movement | Sliding | Separation by snapping or tearing |
| Occurrence | Gradual | Sudden |
| Deformation | Yes | No |
| Behavior | Ductile | Brittle |
| Fracture appearance | Dull and fibrous | Bright and sparkling |
| Fractographic feature | Dimpled rupture | Cleavage |

- Dull and fibrous fracture appearance
- Brittle fractures typically have the following characteristics:[1]
- Lack of plastic or permanent deformation in the region of the fracture
- Principle tensile stress is perpendicular to the surface of the brittle fracture
- Characteristic markings on the fracture surface pointing back to where the fracture originated

When examined under a scanning electron microscope, fracture surfaces seldom exhibit entirely dimpled rupture (i.e. ductile fracture) or entirely cleavage (i.e. brittle fracture), although one or the other may dominate. Other fracture modes include intergranular fractures, combination (quasi-cleavage) fractures and fatigue fractures.

## Fatigue Fractures

Beachmarks (aka stop marks, arrest marks, clamshell marks, conchoidal marks) are a classic fracture feature (Fig. 3) found during failure analysis, and they identify fatigue failures by their presence. These manifest themselves as visible ridges and are indicative of interruptions in crack propagation. Beachmarks (as well as

**Table 2. Typical characteristics of ductile and brittle fractures[1]**

| Parameter | Ductile Characteristic | Brittle Characteristic |
|---|---|---|
| Temperature | Higher | Lower |
| Rate of loading | Lower | Higher |
| Type of loading | Torsion | Tension or compression |
| Geometry | Absence of stress concentrators | Presence of stress concentrators |
| Size | Smaller, thinner | Larger, thicker |
| Pressure | Higher | Lower |
| Material strength | Lower | Higher |

**Table 3. General categories of wear[1]**

| Type of Wear | Key Characteristic(s) | Solution(s) |
|---|---|---|
| 1. Abrasive | Material removal from the surface (cutting). | Increase surface hardness; remove particles causing abrasion; replace worn parts. |
| a) Erosion | Material removal, grooving or channeling of the surface. | Rounding corners; eliminating bends; changing flow characteristics. |
| b) Grinding | Cutting, plowing or grooving of the surface. | Eliminate high-stress locations such as points and edges; use coatings or case hardening. |
| c) Gouging | Battering or impact damage by hard or abrasive products. | Replaceable parts; substitution of more resistant material. |
| 2. Adhesive | Microwelding of sliding interfaces due to frictional forces, scoring, scuffing, galling, seizing and tearing. | Lubrication; use of smooth surfaces, chemical films, insoluble contact materials. |
| 3. Fretting corrosion | Wear/oxidation of mating surfaces under vibrational load (slight relative motion). | Eliminate or reduce vibration; reduce slip at the interface; lubrication; elastomeric materials in the joints. |
| 4. Contact stress fatigue | Pitting/spalling under rolling and/or sliding motion (e.g., bearings, gears). | Improve metallurgical properties (e.g., cleanliness), change geometry and improve finish; Lower loads. |
| a) Sub-surface-origin | Pitting/spalling initiating subsurface in regions of maximum shear stress. | Cleaner steel to eliminate hard, brittle inclusions and subsurface defects; improve load distribution. |
| b) Surface-origin | Multiple causes including sliding and rolling-induced (small surface) pitting. Dents, poor finish, poor geometry. | Improve geometry, surface finish, lubrication and load distribution; minimize sliding. |
| c) Subcase-origin | Initiates below the case in case-hardened or induction-hardened parts when applied shear stress exceeds some percentage of available shear strength (believed to be 55% or higher). | Increase case depth and/or core hardness. |
| d) Cavitation | Pitting by collapse of vapor bubbles at dynamic liquid-metal interfaces. Enhanced by vibration and corrosion. | Minimize vibration and vapor formation; increase stiffness and hardness; and improve surface finish. |

striations) identify the position of the tip of the fatigue crack at any given point in time. They are formed by plastic deformation at the crack tip and by differences in the time of corrosion (when present) in the propagating crack. Beachmarks expand outward from the fatigue origin and are often circular or semicircular in appearance. They will not be present if the part saw only brief interruptions in service.

## Wear

Wear (Table 3) is a type of surface destruction that involves the removal of material from the surface of a component part under some form of contact produced by a form of mechanical action. Wear and corrosion are closely linked, and it is important not only to evaluate the failure but to take into consideration design and environment and have a good understanding of the service history of a component..

## Corrosion

Corrosion is the destruction of a material or component by the actions of chemical or electrochemical reactions with the component environment. The major types of corrosion include galvanic action, uniform corrosion, crevice corrosion, stress-corrosion cracking and corrosion fatigue. The mechanisms and effects created by each of these are well documented in the literature.[8, 9] It is critical to understand that the effects of corrosion are present to some degree in every failure analysis, which is one of the reasons why protecting fracture surfaces is so critical to performing a proper failure analysise.

## In Summary

This brief introduction to the types and mechanisms involved with the failure of component parts in service is intended to invite the reader to learn more about this subject as it relates to his/her specific area of interest. One of the important takeaways is that no product failure should be treated lightly, and determination of the (single) root cause is critical to the success of any engineering design.

Part 2 will address failure-analysis methods and introduce the subject of fractography as a tool in determining root cause.

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# Understanding Component Failures (Part 2: Analysis Methods)

We continue our discussion of component failures by talking about how to analyze what went wrong and some of the laboratory tools we use. If our analysis is accurate, one of the fracture mechanisms discussed in Part 1 will be consistent with the cause. If the mechanism is not properly understood or the analysis incomplete or inadequate, all contributory factors and, therefore, the root cause will not be properly identified, making corrective action ineffective. Let's learn more.

## The Role of Fractography

Fractography is the term used to describe the study of fracture surfaces. Fractographic methods are routinely used to help determine the root cause of a failure. One of the goals of fractography is to establish and examine the origin of cracks in an attempt to reveal the cause of crack initiation. Initial fractographic examination is commonly carried out on a macro scale using stereo microscopy and oblique lighting techniques (e.g., low angle, often from various sides) to identify the extent of cracking, possible modes and likely origins. Optical or scanning electron microscopy is then used to pinpoint the nature of the failure and the causes of crack initiation and growth (if the loading pattern is known).

Common features that may cause crack initiation are inclusions; voids or holes in the material; contaminants; and stress concentrators such as dents, bruises, coarse surface finishes, sharp corners, insufficient radii or other abrupt changes in cross section. Lines on fracture surfaces often show crack direction. Some modes of crack growth leave characteristic marks on the surface that identify the mode of crack growth and the origin on a macro scale (e.g., beachmarks or striations on fatigue cracks). Also revealing are areas that exhibit subcritical cracks (i.e. cracks that have not grown to completion). They can indicate that the material was faulty when loaded or, alternatively, that the sample was overloaded at the time of failure. Other clues such as cusps, which typically form where brittle cracks meet, may be present.

## Fracture Modes

Fracture in most engineered materials occurs either by intergranular (along the grain boundaries) or transgranular (through the grains) fracture paths. As

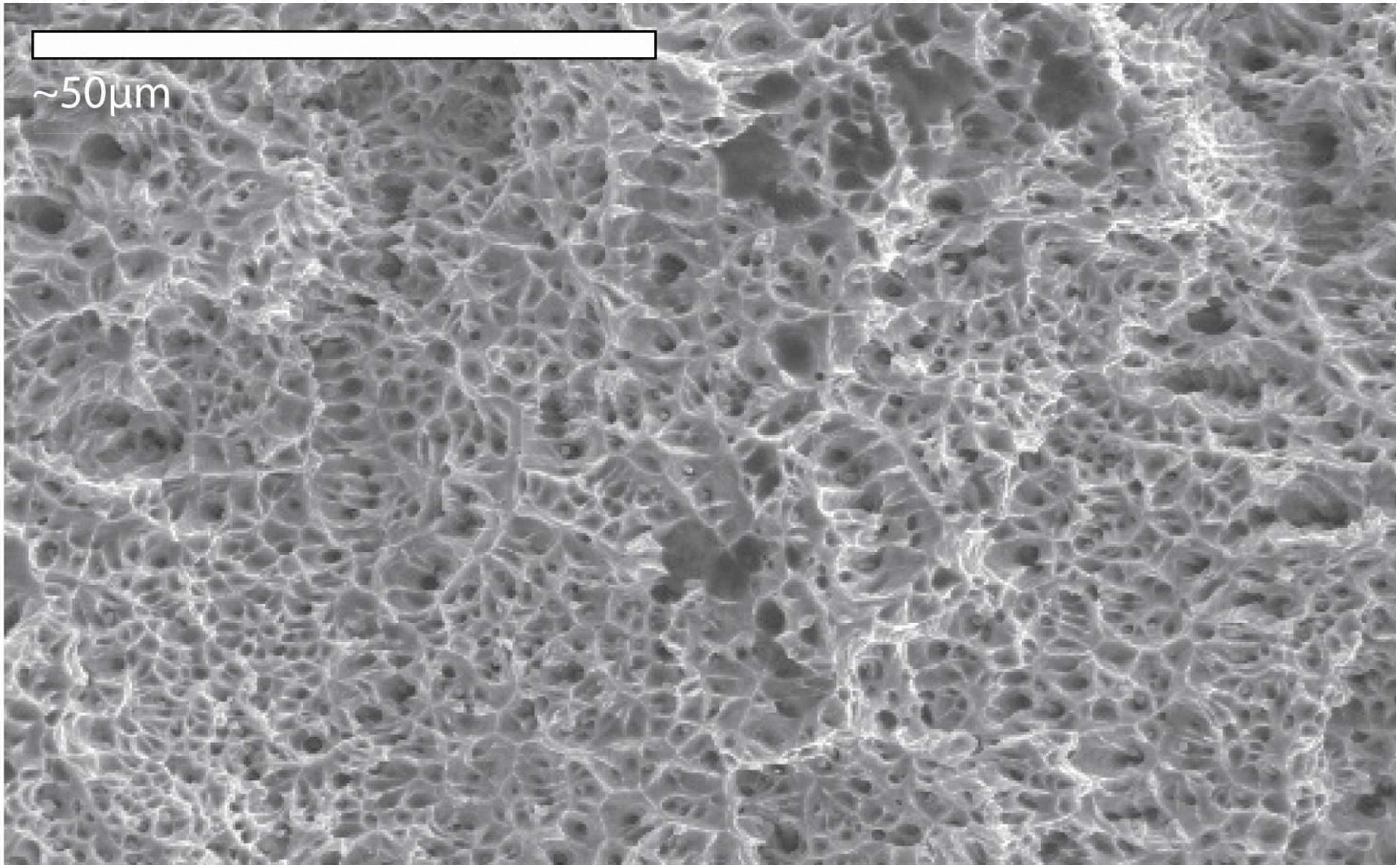

Fig. 1. Example of a dimpled rupture-type failure (Courtesy of Aliya Analytical, Inc.)

mentioned in Part 1, the type of loading experienced produces different types of stresses in the material, which result in different modes of fracture. These can be classified as dimpled rupture, cleavage, decohesive rupture and fatigue.

**Dimpled rupture** (aka microvoid coalescence, or MVC) is essentially transgranular and ductile in nature. This type of fracture exhibits cuplike depressions (Fig. 1) commonly referred to as dimples. Dimple rupture is the mode of fracture when overload

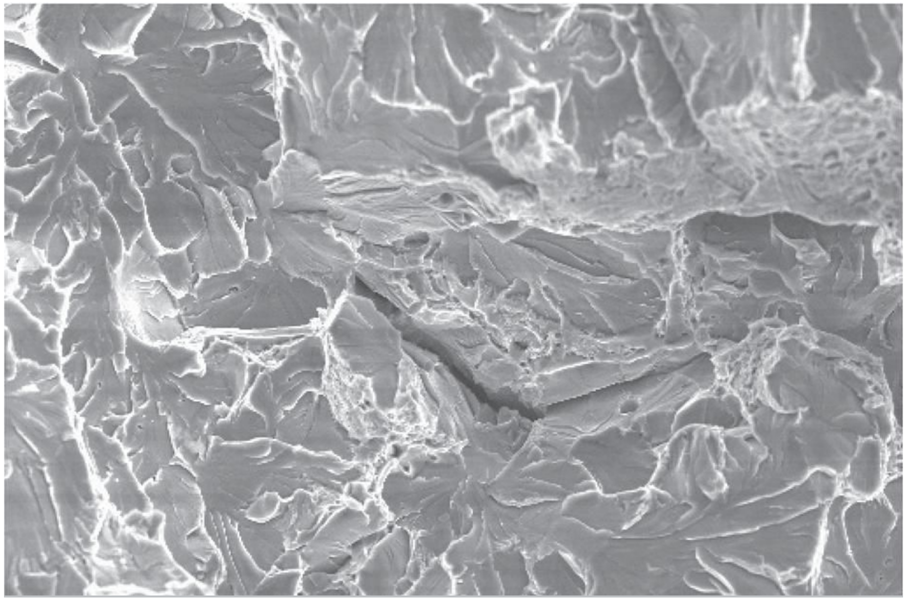

Fig. 2. Example of a cleavage-type failure (Courtesy of Aliya Analytical, Inc.)

is the principal cause of failure in a ductile manner and typically proceeds in three stages: nucleation, growth and coalescence of microvoids. The dimple shape is dictated by the stress state of the material and can be caused by particle cracking or interfacial failure between precipitate particles and the matrix. It usually occurs under single load or tearing.

When a material is put under uniaxial tensile loading, equiaxed dimples appear that have complete rims. Under tear loading, the dimples are elongated, the rims of the dimples are not complete and the dimples are in the same direction as the loading when viewing the mating fracture surfaces. Shear loading has the same features as tear loading except the dimples are in opposite directions on the mating surfaces. Microvoids grow during plastic flow of the matrix, and microvoids coalesce when adjacent microvoids link together or the material between microvoids experiences necking.

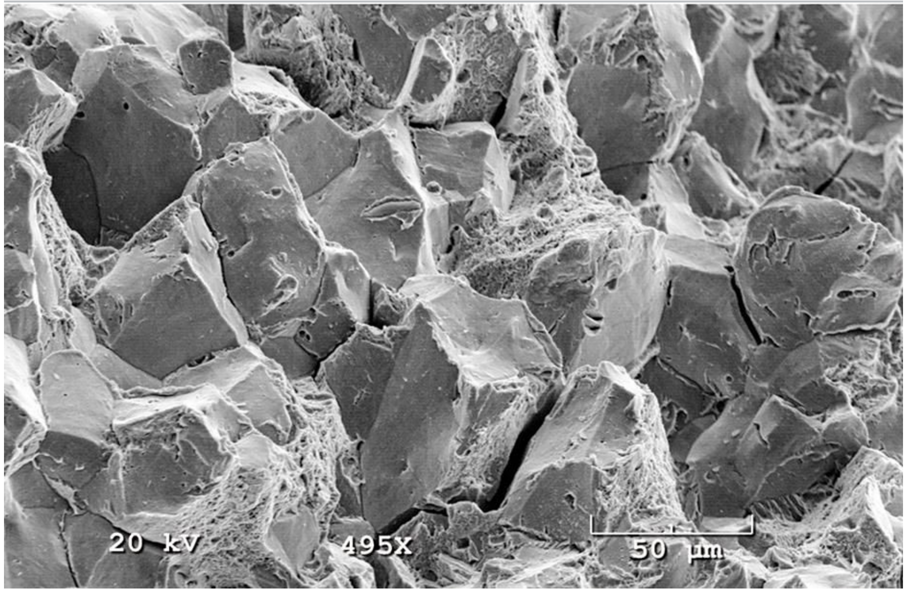

Fig. 3. Example of a decohesive rupture-type failure (Courtesy of Aston Metallurgical Services Company, Inc.)

**Cleavage** is also essentially transgranular and brittle in nature being a low-energy fracture propagating along well-defined crystallographic planes. It is normally flat with shallow features. Cleavage occurs in brittle materials at low temperature and generally results from high stress along multiple axes with a high rate of deformation. Characteristics of cleavage are cleavage steps (i.e. a cleavage facet joining two parallel cleavage fractures); feather markings (i.e. very fine, fan-like markings on a cleavage fracture); chevron patterns; herringbone structures; tongues and microtwins; Wallner lines (i.e. distinct V-shape created by pattern intersection of two groups of parallel cleavage steps, primarily found in brittle materials); and quasi-cleavage (i.e. cleavage with micro-dimples).

**Fatigue failures** (c.f. Part 1) are essentially transgranular brittle fractures and the result of repetitive or (high or low) cyclic loading. The observed characteristic striations (clamshell marks, beachmarks, etc.) show the propagation of the crack front.

**Decohesive rupture** is almost exclusively intergranular in nature and typically occurs by one of several phenomena in weaker areas such as those between the individual grains.

## Tools of the Trade

### SEM

The scanning electron microscope (SEM) uses a focused beam of high-energy electrons to generate a variety of signals from the surface of a specimen. These signals reveal information such as external morphology (texture), chemical composition, crystalline structure and orientation. In most applications, data are collected over a selected area of the surface of the sample and a two-dimensional image is generated that displays spatial variations in these properties. Areas ranging from approximately 1 cm to 5 microns in width can be imaged in a scanning mode using conventional SEM techniques (magnification ranging from 20X to approximately 30,000X and spatial resolution of 50-100 nm). The SEM is also capable of performing analyses of selected point locations on the sample. This approach is especially useful in qualitative or semi-quantitative analysis, helping to determine chemical compositions (using EDS), crystalline structure and crystal orientations (using EBSD).

### TEM

The transmission electron microscope (TEM) operates on the same basic principles as the light microscope but uses electrons as a “light source” because their lower wavelength makes it possible to obtain a resolution 1,000 times better than with an optical light microscope. The TEM is generally used to study ultrathin sections (50-60 nanometers), allowing the user to observe and analyze internal microstructures.

Stained areas of the sample absorb or scatter the beam, producing dark spots; unstained areas appear light.

### STEM

Scanning transmission electron microscopy (STEM) combines the principles of TEM and SEM and is increasing in popularity for failure analysis. Like the TEM, a STEM requires very thin samples and looks primarily at a focused beam of electrons. One of its principal advantages over TEM is in enabling the use of other of signals that cannot be spatially correlated in TEM, including secondary electrons, scattered-beam electrons, characteristic X-rays and electron energy loss. The STEM looks like a TEM but produces images one spot at a time, as in the SEM, rather than all at one time as in the TEM. Like the SEM, the STEM technique scans a very finely focused beam of electrons across the sample, which is correlated with beam position to build a virtual image in which the gray level at the corresponding location in the image represents the signal level at any location in the sample. Its primary advantage over conventional SEM imaging is the improvement in spatial resolution.

## What the Heat Treater Needs To Know

The important aspect of failure analysis for the heat treater to understand is that tools and investigative practices exist to help us pinpoint where and how a component failed. If it happens that heat treatment is the root cause, the information obtained by failure analysis allows us to make informed decisions regarding changes to recipes, processes or equipment as part of the corrective action.

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# Failure Triangles – A Diagnostic Tool

Avoiding product failure is the mission of everyone involved in engineering and manufacturing. "What must the part do in service?" is a question that needs to be answered when designing any component or system. This leads to the establishment of the engineering requirements that will allow the product to perform its intended function. In manufacturing we ask ourselves, "How was or should the part be made?" Finally, understanding the circumstances under which a part may or did fail is a necessary step in the process. Let's learn more.

Failures can be traced to deficiencies in design, materials, processing, product characteristics and quality, known and unknown application factors, and to human error. Examples include excessive distortion, buckling, ductile or brittle fracture, creep, rupture, cracking, fatigue, shock, wear, corrosion, misalignment, poor geometrical design and literally hundreds of other factors. Whatever the source, it is important to recognize that it is impossible to separate the product from the process and, as such, material, design and processing applications are all interrelated.

When considering ways to prevent failures from occurring, one determines the factors involved and whether they acted alone or in combination with one another. We ask ourselves questions such as "Which of the various failure classifications were the most important contributors?" and "Was the design robust enough and the safety factors properly chosen to meet the application rigors imposed in service?" Having a solid engineering design, coupled with understanding the application, loading and design requirements, is key to avoiding failures. If failures do happen, we must know what contributed to the damage.

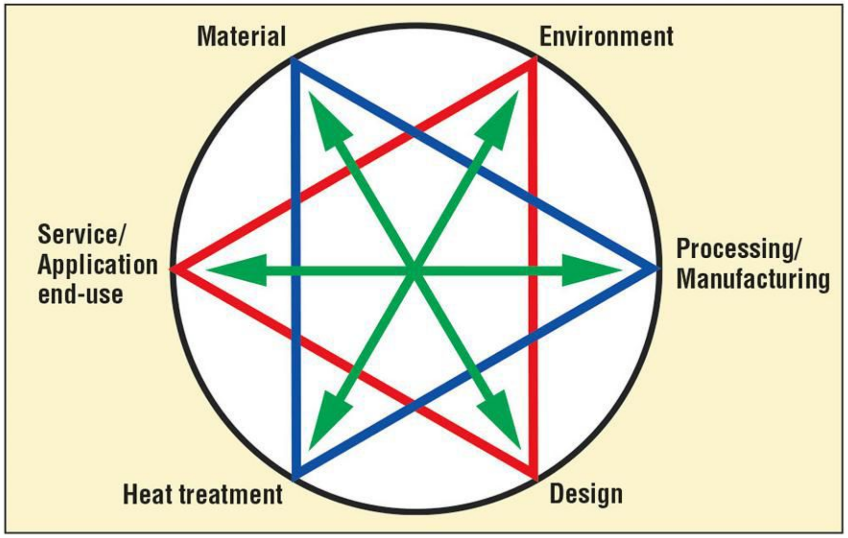

Fig. 1. External influences: application factors (red); Internal influences: manufacturing factors (blue); Interrelationships (green)

**Table 1. Typical service application influences**

| Issue | Examples |
|---|---|
| Overloading | Improper application of load (e.g., offset loads), inadequate stress analysis, unexpected loads or conditions |
| Operating/exposure conditions | Temperature, vibration, impact, collision |
| Contamination | Chemical, mechanical, electrical |
| Maintenance or repair issues | Improper maintenance, servicing or substitution of improper parts |
| Type of forces | Bending, axial load, static load, fatigue |
| Human error | External influence to the form-fit-function |

**Table 2. Typical material influences**

| Issue | Examples |
|---|---|
| Alloy design/ selection | Use of known engineering performance criteria |
| Form/condition | Forging, casting, bar, plate, wire |
| Microstructural characteristics | Grain size, matrix microconstituents and secondary phases |
| Chemistry | Primary compositional elements as well as residual-element effects |
| Properties | Mechanical (e.g., strength/toughness, fatigue strength), physical or metallurgical (e.g., hardenability, formability, machinability, weldability) |
| Internal quality | Inclusion types, amounts, sizes, morphologies and distribution |

## Failure Classifications

There are a multitude of different types of failures and failure classifications, each requiring a detailed analysis of both the external influences and internal influences that contribute to either the success or failure of a product. Failure triangles (Fig. 1) help us visualize these basic classifications and the types of interactions that might take place when failures occur. In most cases, failures happen when one or more of these variables act alone or in combination with one another. These include:

**Service/Application End-Use Factors:** What forces and conditions a part is subjected to in service (Table 1), coupled with an understanding of those factors that might contribute to a failure, are important considerations. Service conditions may change over time, making this a difficult area to predict.

**Material Factors:** The type and form of material selected for a given application (Table 2) are critical to its performance in a given application.

**Design Factors:** Fitness for purpose is the principal focus of design (Table 3) and must be innately valid such that the expected service life, service conditions and loading can be safely accommodated.

**Table 3. Typical design influences**

| Issue | Examples |
|---|---|
| Design factors | Stress state/type, loading (static or dynamic), stress risers (geometrical and/or metallurgical) |
| Raw material | Form, hardness, mechanical/physical/metallurgical properties, cost |
| Performance characteristics under specific conditions | Duty cycle, impact loading, offset loading, misalignments, transient or unexpected loads, environmental factors |
| Safety factors | Anticipation of unforeseen service conditions |

**Processing Factors:** Manufacturing practices must be robust and can also contribute to product failures if not properly performed (Table 4).

**Heat-Treating Factors:** Heat-treatment issues can contribute to failures in a number of ways (Table 5). Caution must be observed to avoid blaming heat treatment for revealing a problem the root cause of which may lie outside the contribution of heat treatment to the condition observed.

**Environmental Factors:** Environmental-induced factors (Table 6) can play a significant role in field failures and are one of the most difficult aspects to anticipate or control.

**Table 4. Typical processing (manufacturing) influences**

| Issue | Examples |
|---|---|
| Equipment type and availability | Use of wrong equipment or processes |
| Manufacturing methods | Improper machining (feeds and speeds), improper selection of pre- and post-heat treatment or other manufacturing operations |
| Assembly methods | Use of improper tools or poor techniques introducing undesired stress into a component through forced fit-up, nicks, dings, etc. |
| Welding or joining methods | Introduction of residual stress, voids or cracks (surface or subsurface) and/or undesirable microstructures |
| Improper heat treatment | Case/core properties, introduction of distortion or undesirable residual stress |
| Surface finish | Proper or improper use of grinding, polishing, peening or coatings |
| Residual stress | Proper or improper use of heat treatment or surface treatments (shot blasting/shot peening) |

## Final Thoughts

Accurate record keeping and careful documentation of failures if/when they occur are of critical importance to assist in determining the root cause of a particular product failure and to avoid its reoccurrence in the future.

**Table 5. Typical heat-treatment influences**

| Issue | Examples |
|---|---|
| Heating and/or cooling | Shape changes, volumetric expansion |
| Changes in residual stress state | Differential expansion due to thermal gradients, transformational (phase change) induced stress |
| Quenching | Improper or inadequate quenching (e.g., wrong quenchant, poor agitation, improper equipment design) leading to distortion and/or cracking |
| Hardness | Improper hardness for a given application |
| Microstructure | Issues related to or arising from grain growth, microcracking, microsegregation, internal oxidation, alloy depletion, formation of high-temperature transformation products, decarburization, retained austenite, precipitation of unwanted phases and excessive carbide formation |
| Post-heat-treatment methods | Improper post-heat-treatment operations such as grinding, straightening, shot blast/shot peening or plating |
| Equipment/process selection | Use of the wrong equipment or process introducing unwanted variability |

## More to Come...

Failure triangles and their interactions will be the subject of a future article (Reference 1). Stay tuned.

**Table 6. Typical environmental influences**

| Factor | Examples |
|---|---|
| Humidity/moisture | Environmental influences accelerating failure due to corrosion |
| Chemical attack | Corrosion (e.g., stress, fatigue, microbial) or embrittlement (e.g., hydrogen, liquid metal) |
| Lubrication | Viscosity, temperature, oxidation state, additive condition |
| Wear | Erosion, galling, seizing, pitting |
| High temperature | Warping, creep, oxidation, localized melting |
| Site changes | Introduction of unexpected conditions such as vibration, shock impact or exposure to external elements (e.g., radiation) |

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# Stress Corrosion Cracking

Components fail for a variety of reasons, which includes a corrosion phenomena characterized by the fact that stress (and/or deformation) is present to provide a trigger that leads to sudden crack formation, propagation and failure. Let's learn more.

Stress corrosion cracking (SCC) is the type of failure mechanism caused by a combination of environmental, material and stress conditions (Fig. 1). It is generally considered the most complex of the failure modes since it can attack soft or hard parts; ferrous or nonferrous materials; ferritic or austenitic structures; and materials in the unalloyed or alloyed state. Cracks may propagate in a transgranular or intergranular fashion or in a combination of the two. The stress, however, must be in the form of tensile stress above some minimum (i.e. threshold) value, usually below the yield stress of the material and in the presence of a corrosive environment that includes sulfides, chlorides, caustics and hydrogen. Temperature is a significant environmental factor affecting crack formation, and pitting is commonly associated with SCC phenomena. In addition, catastrophic failure can occur without significant deformation or obvious (surface) deterioration of the component.

The SCC phenomena can be affected by many factors in addition to stress level, including alloy composition, microstructure, concentration of corrosive species, surface finish, micro-environmental surface effects, temperature, electrochemical potential and the like. Further complications are initiation and propagation phases and the observation that in some cases cracks initiate at the base of corrosion pits.

## Mechanisms

There is no identified single mechanism explaining SCC, but several theories have been proposed.

- Active path propagation: Localized preferential corrosion (a.k.a. dissolution) at the crack tip, along a susceptible path, with the bulk of the material remaining in a more passive state. The rate of metal dissolution can be several orders of magnitude higher when an alloy is in its active state compared to its passive condition.
- Hydrogen embrittlement: High hydrogen concentrates in highly stressed regions, such as at the crack tip or other stress concentrators, leading to localized embrittlement.

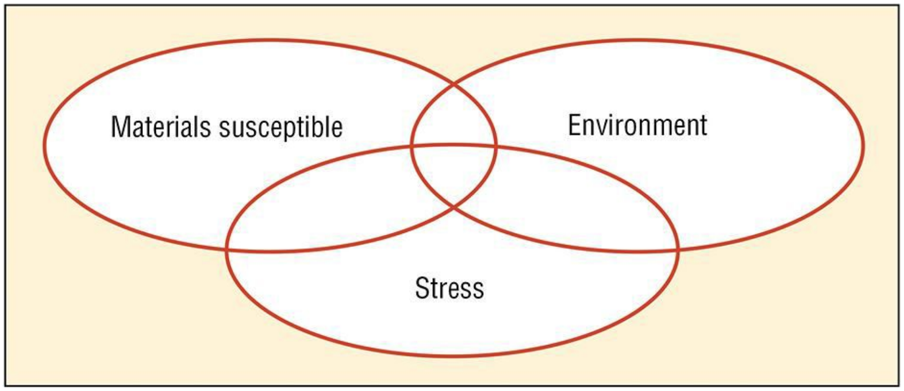

Fig. 1. Factors contributing to stress corrosion cracking

- Brittle film-induced cleavage: Cracks initiated in a brittle surface film may propagate (over a microscopic distance) into underlying, more ductile material before being arrested by ductile blunting of the crack tip. If the brittle film re-forms over the blunted crack tip (under the influence of corrosion processes), such a process can be repeated over and over again.

## Negating the Effects of SCC

A combination of good design, correct selection of SCC-resistant materials, environment management, maintenance and inspection can effectively control this type of corrosion. Stresses to consider include:

- ✓ Operational conditions
  - Applied (tensile) stresses
- ✓ Thermally induced factors
  - Temperature gradients
  - Differential thermal forces (expansion and contraction)
- ✓ Buildup of corrosion products
  - Volumetric dependent
- ✓ Assembly issues
  - Poor fit up (tolerance problems)
  - Tightening/torqueing
  - Press and shrink fits
  - Fastener interference
  - Joining method
- ✓ Residual stresses (from the manufacturing processes)
  - Joining (welding, brazing, soldering)
  - Forging or casting
  - Surface treatment (plating, mechanical cleaning, etc.)
  - Heat treatment (quenching, phase changes)
  - Forming and shaping
  - Machining
  - Cutting and shearing

One of the most important considerations to negate the effects of SCC is choosing the proper alloy. It is relatively simple to choose a component with adequate strength and good (general) corrosion

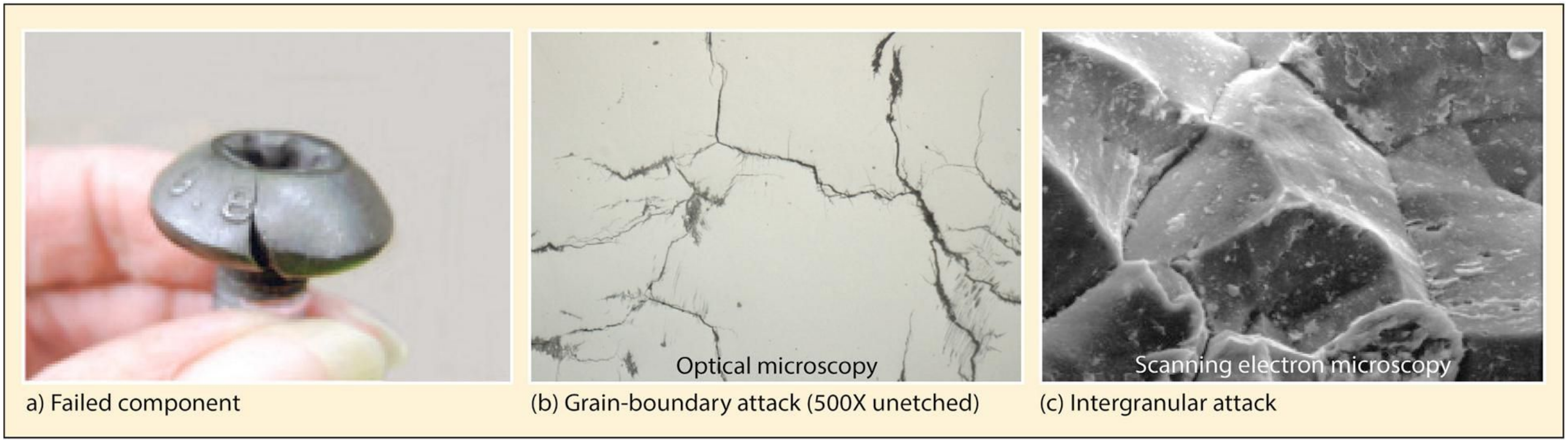

Fig. 2. Fastener failure – Stress corrosion cracking[2]

resistance. However, knowing the particular type of SCC issues that may be at work in the application is an important step in achieving a resistant material. In certain environments, it may be necessary to choose a material that will experience some general corrosion since general corrosion is visually evident, and, with proper preventative maintenance, general corrosion can be seen and components replaced as necessary. On the other hand, SCC is rarely visually apparent and often occurs without warning (Fig. 2). When it does, a catastrophic failure often follows.

Other methods include removing the corrosive environment or changing the manufacturing process or design to reduce the tensile stresses. A combination of good design, careful selection of stress corrosion-resistant grades (e.g., stainless steel) and effective management, including maintenance and inspection, all can effectively control corrosion. Specific steps can be taken to prevent the onset of SCC and minimize its consequences when it does occur by:

- Consideration of the potential for SCC during the design and fabrication of components
- Selection of appropriate material grades
- Maintaining a chemical balance of the environment
- Ensuring that the potential for (organic or inorganic) contamination is minimized
- Maintaining proper environmental conditions (e.g., air quality)

- Regular inspections of components for signs of corrosion and SCC

## Importance of Material Selection

In many applications, austenitic stainless steel fasteners (e.g., ASTM A193 grade B8) of 304 and 316 stainless steels provide good general corrosion resistance and are commonly requested. However, in marine environments where stainless steel would seem to be the logical choice, alloy-steel fasteners are preferred due to SCC concerns. Chlorides, fluorides and other halogens are known catalysts for chloride SCC. In order to reduce their susceptibility to general corrosion, alloy-steel fasteners such as grade B7 are usually provided with some type of protective coating (e.g., zinc or cadmium plating). However, the designer must still be aware that this can lead to another form of corrosion due to environmental stress cracking in the form of liquid metal embrittlement (LME) or a related failure mode, solid metal induced embrittlement (SMIE). Therefore, appropriate cautions must be taken.

In addition to SCC, other forms of embrittlement include: (a) environmentally induced cracking due to such factors as cold work (i.e. residual stress), welding, grinding, thermal treatment or service conditions; (b) hydrogen embrittlement from plating, welding, cathodic protection and as a by-product of general corrosion; (c) corrosion fatigue; and (d) liquid-metal embrittlement.

## In Conclusion

Careful consideration of the factors discussed above as well as taking the time to understand how and where a component will be used in service can help minimize stress corrosion cracking in most applications

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# Sigma-Phase Embrittlement

Many stainless steels and other iron-chromium alloys are susceptible to a grain boundary phenomenon known as sigma-phase embrittlement. This type of embrittlement has been shown to cause severe loss of ductility, toughness, and corrosion resistance resulting in cracking (Fig. 1) and failure of components, especially those subjected to impact loads or excessive stress. As heat treaters we need to know more about what sigma-phase embrittlement is and how to avoid its occurrence. Let's learn more.

Prolonged exposure in the temperature range of 565-925°C (1050-1700°F) results in chromium depletion from the grain boundaries, making them susceptible to intergranular corrosion. The most rapid sigma-phase formation occurs in the range of 700-900°C (1290-1650°F). Alloy elements such as molybdenum, titanium and silicon promote the formation of sigma phase, while nitrogen and carbon reduce its tendency to form.

Sigma phase is an intermetallic compound consisting of chromium and iron, which is hard, brittle and non-magnetic. Pure sigma (σ) forms between 42% and 50% chromium and is one of the equilibrium phases in the iron-chromium phase diagram (Fig. 2).

Fig. 1. Section of a cast HH (25% Cr, 12% Ni) stainless steel furnace load-lifting hook that failed due to sigma-phase embrittlement. (Photograph courtesy of George F. Vander Voort, Vander Voort Consulting LLC)

A duplex structure (sigma and alpha phases) has been found to form in alloys with as little as 20% chromium and as much as 70% chromium when exposed to the critical temperature range noted above. At chromium contents of less than 20%, sigma phase is difficult to form, but the presence of molybdenum, silicon, manganese or nickel have a tendency to shift the lower limit down.

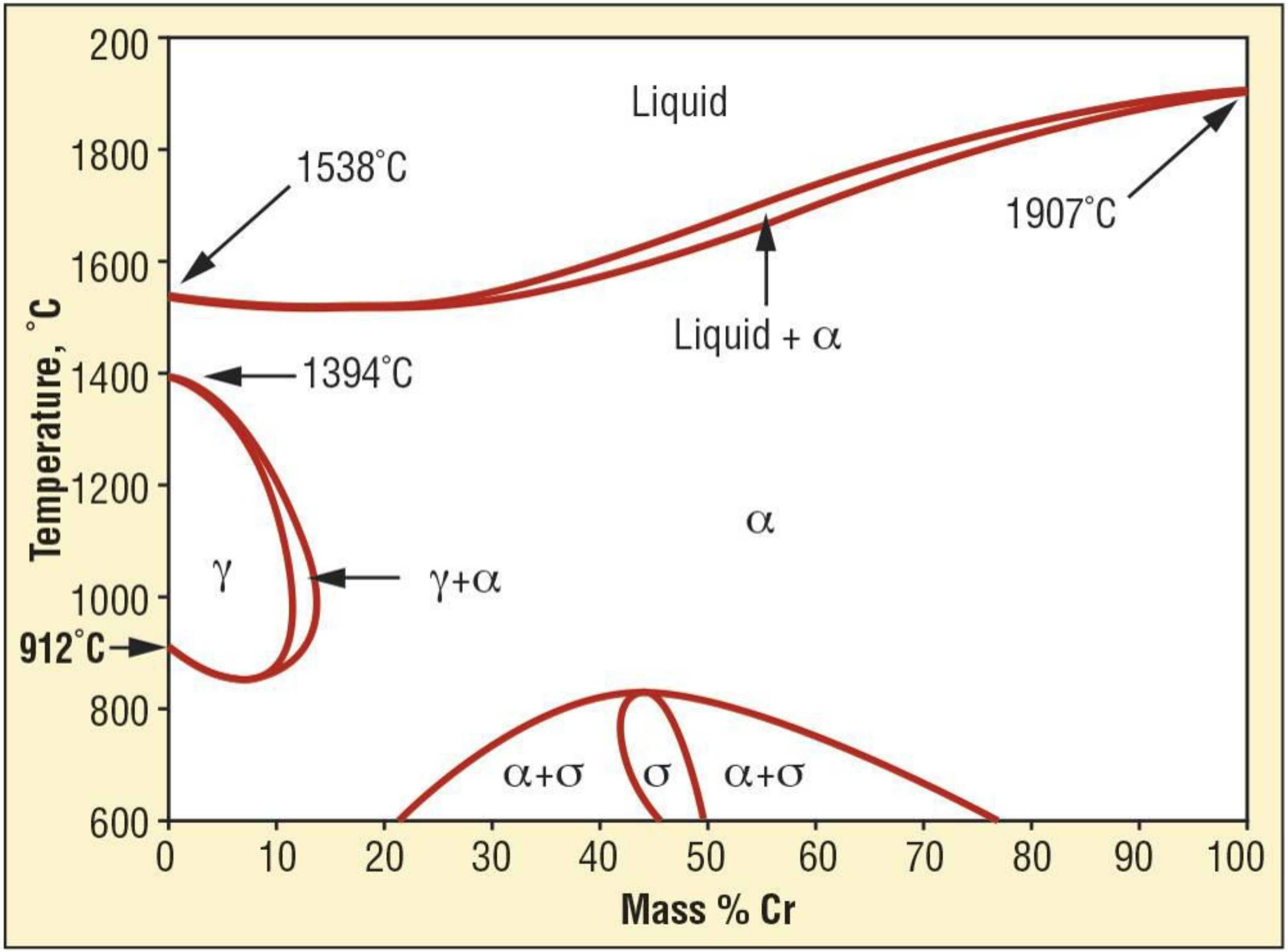

Fig. 2. Iron-chromium phase diagram[1]

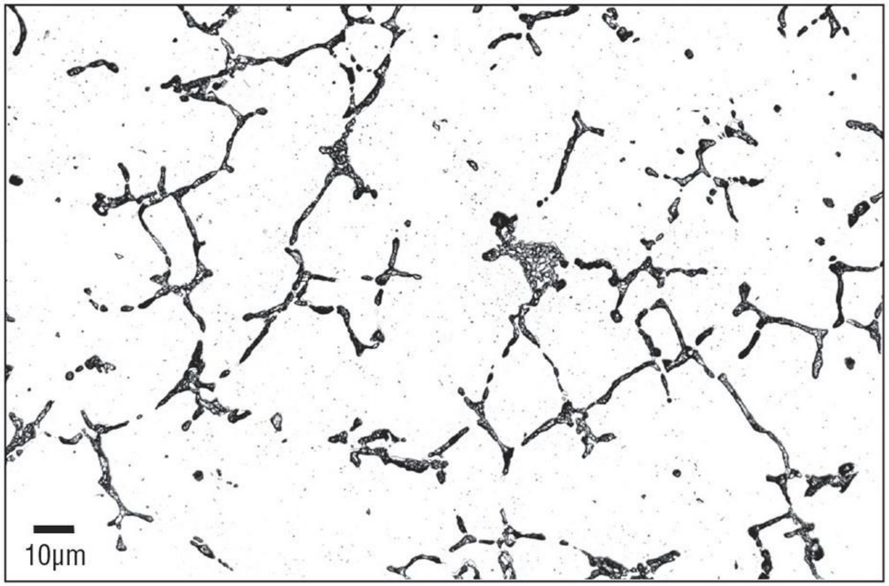

Fig. 3. Sigma phase (dark areas) precipitated from excessive ferrite in the cast HH stainless steel furnace hook, causing it to fracture extensively. (Photograph courtesy of George F. Vander Voort, Vander Voort Consulting LLC)

Molybdenum reportedly promotes sigma-phase formation much more effectively than chromium, particularly at temperatures around 900°C (1650°C). This is why, in the HH cast stainless example shown, the molybdenum content of the alloy is deliberately kept around 0.5%. Austenite-forming elements such as nickel or nitrogen can also accelerate the nucleation and growth of the sigma phase, although these elements may reduce the total amount formed because of the smaller volume fraction of ferrite. Sigma typically nucleates in the austenite-ferrite grain boundaries and grows into the adjacent ferrite. Additional austenite often forms in the areas of chromium depletion adjacent to the sigma phase.

Although the formation of sigma phase is sluggish, cold working enhances the precipitation rate considerably, and sigma phase has even been found in the air-cooled, as-cast structures in very high chromium content alloys. Sigma phase usually appears as a continuous network in the microstructure. Since sigma has a significantly lower corrosion resistance compared to the ferrite matrix, its presence can be detected by etching in a metallographic examination (Fig. 3).

The temperature range of rapid sigma formation coincides with the normal temperatures used for annealing ferritic stainless steels. Consequently, highly alloyed ferritic stainless steels must be annealed in the 1050°C (1925°F) range and rapidly cooled through the critical range to avoid sigma-phase embrittlement. Any sigma phase already formed can be dissolved again by a solution-annealing process performed above 800-850˚C (1470-1560˚F) for relatively short times – approximately an hour (once the entire part has reached temperature) – followed by air cooling.

## Other Forms of Embrittlement[2]

### 475˚C (885˚F) Embrittlement

Iron-chromium alloys containing 15-70% chromium may exhibit a pronounced increase in hardness accompanied by severe loss of ductility and corrosion resistance if exposed to the temperature range of 400-540˚C (750-1005˚F) for significantly shorter time periods than is required for sigma-phase formation. The name of this phenomenon comes from the fact that the peak hardness usually occurs at 475˚C (885˚F). In fact, it can occur during slow cooling from an elevated temperature as well as during elevated-temperature service. For alloys containing 18% Cr, the onset of embrittlement is fast enough to require rapid cooling from the annealing temperature to extend below 400˚C (750˚F) in order to ensure optimal ductility.

In service, alloys containing greater than 16% Cr should not be used at 375-540˚C (707-1004˚F) for extended periods of time or cycled from room temperature through this critical range. This embrittlement phenomenon is believed to be due to the formation of a submicroscopic, coherent precipitate that is induced by the presence of a solubility gap below approximately 550˚C (1020˚F) in a chromium range where sigma phase forms at higher temperatures. Cold work intensifies the rate of 475˚C (885˚F) embrittlement, especially for the higher-chromium alloys. Reheating the alloy to above 550˚C (1022˚F) for a few minutes completely removes this form of embrittlement.

## High-Temperature Embrittlement

Medium- and high-chromium ferritic alloys containing moderate amounts of carbon and/or nitrogen develop high-temperature brittleness if cooled slowly from above 950˚C (1740˚F). The mechanism is similar to that of sensitization and leads to severe intergranular corrosion. Work on two wrought ferritic stainless steels containing 18 and 25% Cr, respectively, has shown that the maximum amount of carbon plus nitrogen tolerable for good room-temperature toughness is 0.055% for the 18% Cr alloy and 0.035% for the 25% Cr alloy.

## Duplex Steels Not Immune

In general, the presence of a high percentage of sigma phase is undesirable in duplex stainless steels due to its detrimental influence on corrosion (e.g., pitting) and mechanical properties.[3] Duplex and super-duplex stainless steels are ferrous alloys with up to 26% chromium, 8% nickel, 5% molybdenum and 0.3% nitrogen and are intended for service in corrosive applications.[4] The metallurgy of duplex and super-duplex stainless steels (especially castings) is complex due to high sensitiveness to sigma-phase precipitation on cooling from solidification temperature as well as from heat treatment.

The hardness of these materials is a strong indication of the presence of sigma phase in the microstructure. It has been found that the material hardness is inversely proportional to the heat-treatment temperature. When the heat-treatment temperature during solution treatment increases, the sigma-phase content in the microstructure decreases. Consequently, the material hardness diminishes. When the sigma phase is completely dissolved by the heat treatment, the material hardness is influenced only due to ferrite and austenite contents in the microstructure.

The soak temperature also influences the percentage of sigma phase present in solution (as well as in the volumetric concentrations of the ferrite and austenite phases). The ferrite percentage increases with the increasing heat-treating temperature. From 1060˚C (1940˚F) and up, the sigma-phase quantity is eliminated and the volume fractions of ferrite and austenite each approach 50%.

## Summing Up

The presence of sigma phase in stainless steels and iron-chromium alloys should be cause for concern among heat treaters, but awareness of what can trigger this form of embrittlement and what can be done to negate its effects are worth our time and effort.

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# Hot Gaseous Corrosion

Data and information on the effects of corrosion on engineered materials are available in many forms and from many sources.[1–15] The focus for most corrosion engineers is on aqueous corrosion, an important topic in and of itself. As heat treaters, however, the effects of hot gaseous corrosion in our heat-treat furnaces are of more immediate concern. Let's learn more.

## Corrosion Basics

We begin with the realization that all materials are chemically unstable in some environments, and corrosive attack will occur. It can often be predicted or modeled by studying thermodynamic data and knowing which of the many corrosion-related chemical states are active. In the real world, however, it is important to recognize the various forms of corrosion:

- Uniform (or general) attack
- Intergranular attack
- Galvanic (or two-metal) action
- Erosion
- Dezincification (or parting)
- Pitting
- Stress corrosion
- Electrolytic (or concentration) cells

When it comes to corrosion, the greater the metal's solubility, the greater the degree and severity. There are many important variations on the above, and two of the most important are (a) localized corrosive attack (e.g., pits, intergranular attack, crevices, galvanic action) and (b) interaction with mechanical influences (e.g., stress, fatigue, fretting). These actions are frequently very rapid and have catastrophic effects.

There are also a number of ways to combat corrosion, including: alloying to produce better corrosion resistance; cathodic protection (via sacrificial anodes); coatings (metallic or inorganic); organic coatings (e.g., paints); metal purification; alteration of the environment; nonmetallics and design (i.e. physical) changes.

## Heat-Resistant Alloys

Furnace interiors contain numerous examples of heat-resistant iron-nickel-chromium (Fe-Ni-Cr) alloys for such items as radiant tubes, fans, heating elements,

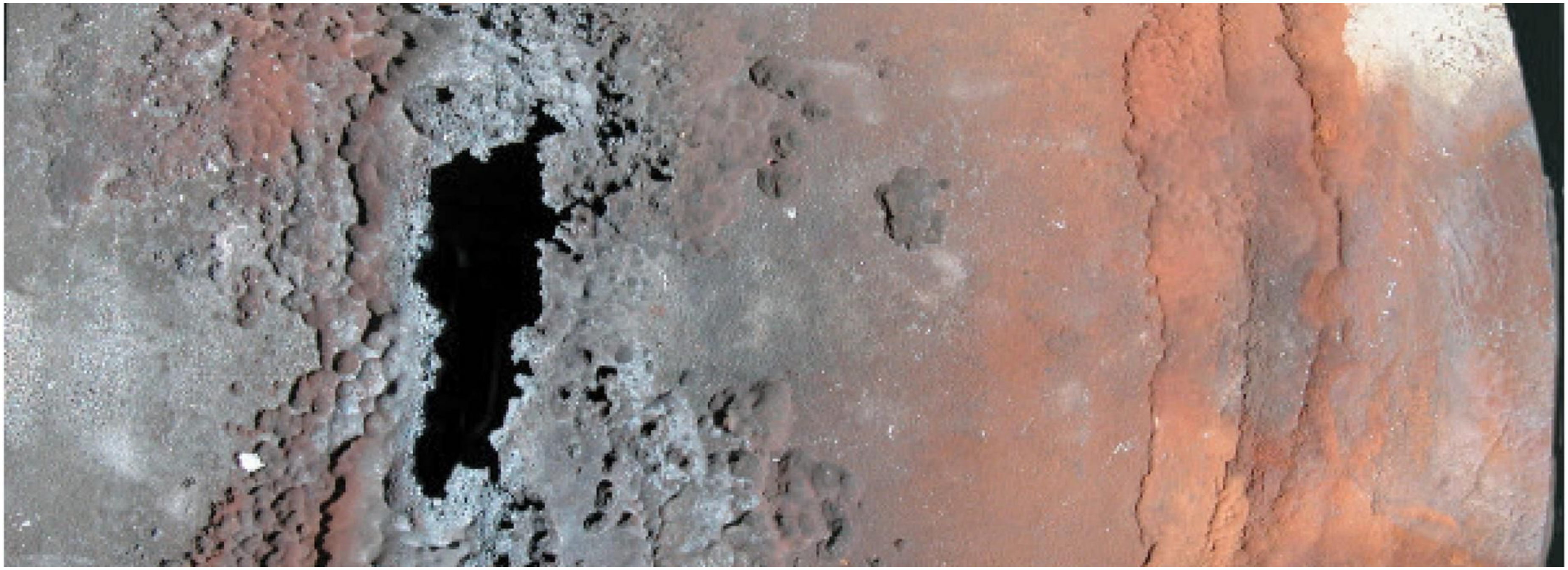

Fig. 2. 330-alloy radiant tube removed after six months of use (rotary retort furnace)

roller rails and rollers, chain guides, and atmosphere inlet tubes to name a few. Baskets, grids and fixtures are other examples. These alloys are normally selected based on their strength (at temperature) and resistance to corrosive attack.

Since these heat-resistant alloy parts are often the most expensive furnace components, heat treaters should have an understanding of how they can be attacked and what can be done to extend their life by minimizing or preventing it.

## Gas-Solid Reactions

A chemical reaction involving a solid and a non-equilibrium gas or gas mixture can be classified as a gas-solid reaction. Examples of intermediate and high-temperature reactions of this type include oxidation, sulfidation, carburization, nitriding, chloridation and the like. The principles are the same for all these types. Only the details differ. As heat treaters, our interest is in controlling, retarding or suppressing these reactions to prevent unwanted corrosion, gasification or embrittlement of the furnace

Fig. 1. Pusher-furnace alloy fan and shaft assembly

alloy or materials being processed.

## Examples of Catastrophic Carburization (a.k.a. Metal Dusting)

Metal dusting (Fig. 1) is a hot gaseous corrosion phenomenon in which a metallic component disintegrates into a dust of fine metal and metal-oxide particles mixed with carbon.

Generally, metal dusting occurs in a localized area. How rapidly the disintegration progresses is a function of temperature, the composition of the atmosphere and its carbon potential, and the material. Other significant factors include the geometry of the system, reaction kinetics, diffusivities of alloy components, the specific volume ratio of new and old phases, and the ultimate plastic strain.

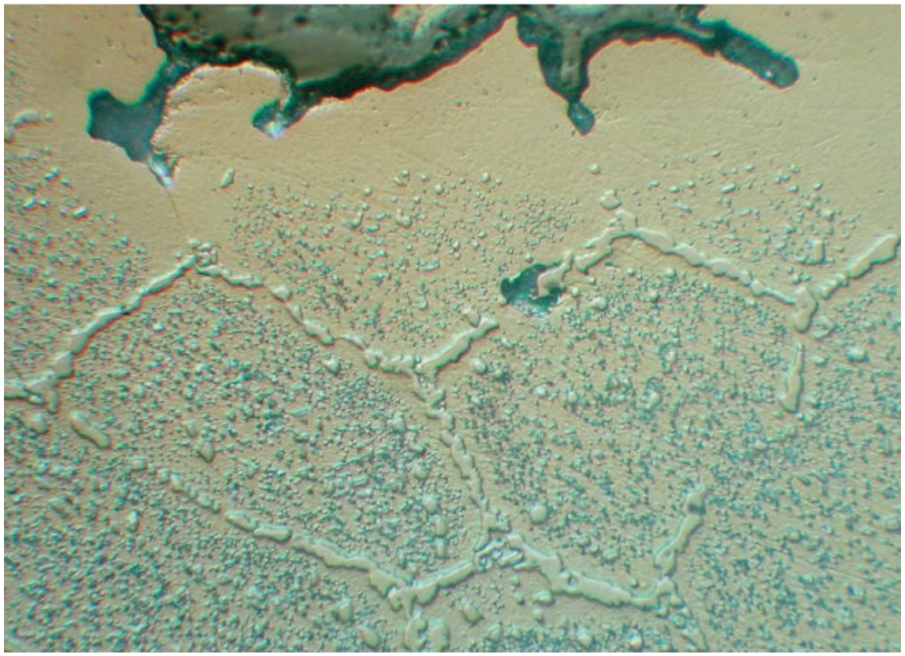

Fig. 3. Microstructural view – catastrophic carburization

Metal dusting usually manifests itself as pits or grooves on the surface or as an overall surface attack in which the metal can literally be eaten away in a matter of days, weeks or months. As an example, the writer has seen a 330-alloy plate mounted underneath a refractory-lined inner door of an integral-quench furnace (where atmosphere passes underneath the door and into the quench vestibule) reduced in thickness from 12.5 mm (0.5 inch) to less than 0.75 mm (0.03 inch) in a little over two months.

In another example, a metallographic investigation of a failed wrought 330-alloy radiant tube (Fig. 2) was conducted. Optical microscopy of the inside (Fig. 3) and outside diameter surfaces in the attacked area revealed evidence of massive carbides. These carbides formed by the reaction of carbon with chromium, depleting the matrix of chromium in regions adjacent to the carbides. Grain detachment and subsequent failure by erosion then occurred.

## How does it occur?

In general, catastrophic carburization of ferrous alloys proceeds via the formation and subsequent disintegration of metastable carbide. The first step in the process is absorption of the gaseous phase on the surface of the metal. The more reactive this phase, the easier it decomposes or is catalytically decomposed (in the case of iron) on the surface. This step is followed by diffusion of carbon atoms from the surface into the bulk metal.

As a result, there is a continuous buildup of carbon within the surface layer. As this layer becomes saturated with carbon, a stable carbide, metastable carbide or an activated carbide complex forms. It then grows until it reaches a state of thermodynamic instability, at which point it rapidly breaks down into the metal plus free carbon.

It's at this stage that the metal disintegrates to a powder as the result of plastic deformation and subsequent fracture in the near-surface layer. The process is controlled by internal stresses due to phase transformation. In other words, competition between stress generation and relaxation exceeds the ultimate strength in this near-surface layer and causes fracture to occur.

In Fe-Ni-Cr alloys, the phenomenon occurs slower (but does not stop) since the disintegration leads to larger metal particles, which are less active catalysts for carbon deposition than the fine iron particles that form with ferrous metals. Therefore, the mass gain from carbon depositing onto high-nickel alloys is much lower. Also, the decomposition of high-nickel alloys occurs by graphitization and not via unstable carbides.

## Pourbaix-Ellingham Diagrams

Thermodynamics can be applied to solid-gas reactions such as oxidation, carburization, sulfidation and nitriding to obtain equilibrium dissociation pressures below which no reactions occur. Diagrams are available of the free energies of formation versus temperature for most metallic compounds. An interesting use of Pourbaix Diagrams (generally reserved for mapping out possible stable equilibrium phases of aqueous, electrochemical systems) as a predictor of stable alloy systems is found by superimposing the various elemental constituents. These diagrams are read much like a standard phase diagram (with a different set of axes).

## Final Thoughts

Hot gaseous corrosion should be an area of focus in an effort to extend the life of alloy components, reduce downtime and save money.

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# Heat-Treat Audits

Audits of the heat-treat department, whether in a captive or commercial shop, are a critical part of any good quality program either as part of a self-assessment, an ISO program or compliance with an industry requirement (e.g., Nadcap, CQI-9, TS16949, etc.). We all recognize that audits are a necessary vehicle for continuous improvement. Instead of fearing what we'll find, we should embrace the audit process. Let's learn more.

## Why Audit?

The goal of a heat-treat audit is to find variability – equipment-induced or process-induced. One of the many objectives of such an audit is to ensure that the parts are processed in accordance with the applicable customer specifications. The "fear" most people or organizations have is that they will interrupt current production, and the implementing of audit findings will further interrupt workflow.

The audit process should be designed to ask basic questions. Who is performing the heat treatment and are they competent? What procedures are being used to carry out the heat-treating operation, and are they adequate to assure proper quality? Where is the work being done, and is the shop capable of performing the required task(s)? When was the last assessment, and was it representative of current practice? Why is an assessment required now (Did a quality issue or problem trigger the event, and if so, will the audit help solve it?)? How will the audit be performed (Are the right personnel in place to reach meaningful conclusions?)?

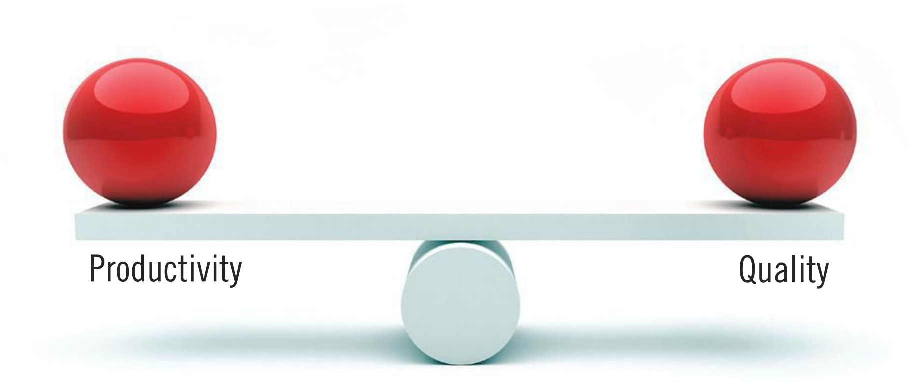

Fig. 2. The goal: a world-class heat-treat shop (Courtesy of ALD Thermal Treatment)

Perhaps surprisingly, most audits that "fail" do so because they do not reveal the true nature of what is happening within the heat-treating shop. By contrast, successful audits find compliance with respect to:

- Quality (e.g., forms, instructions, records)
- Performance (e.g., process or equipment variability)
- Testing (e.g., methods and procedures)
- Specifications (e.g., AMS 2750D, ARP-1962, etc.)

## What Does an Audit Look For?

In general, heat-treat audits look at people, methods, equipment and testing procedures to measure the overall success of a department's quality. Specific areas of focus include:

- General facts (e.g., company/department profile)
- Capabilities (e.g., ability to meet customer requirements)
- Instructions (e.g., recipes, records, processes in-control)
- Sampling guidelines (e.g., testing methods and requirements)
- Continuous improvement programs (e.g., plans, progress)
- Specific compliance (e.g., audit questions/answers)
- Nonconformance (e.g., major and minor deviations)
- Corrective action (e.g., root-cause determination)

## What Does an Audit Achieve?

Too often we are concerned with the heat-treat department's capabilities, either in terms of overall capability, services we can offer to our customers or the size/quantity of equipment in our shops. More importantly, however, we need to focus on our limitations – we need to know what we can't do and set our sights on overcoming these limitations. This is precisely what a good audit achieves.

Most audits should be done in two distinct phases: a general condition assessment and an equipment/process capabilities assessment. Each phase performs a different role.

Phase I: A general condition assessment asks questions such as:

- Where are we today?
- What is the true condition of our equipment?
- Are we monitoring and controlling the right variables?
- Are our maintenance practices adequate?
- Do our methods and control systems measure up?

Phase II: A process/equipment capabilities assessment looks into:

- Process recipes, cycle times and results
- Historical record (e.g., defective parts and corrective actions, types of product failures being experienced and root-cause determination, etc.)
- Safety and environmental conditions
- Customer complaint resolution

## What are the Audit Steps?

A typical audit looks to address the following issues (in sequential order) each time an audit is to be conducted:

- Schedule a kick-off meeting to discuss the audit objectives, timing and report format and distribution. Be sure to include all necessary disciplines and assign an overall audit coordinator.
- Assess the readiness of the heat-treat operation, controls and systems in place for an audit.
- Plan, then test these systems to ensure proper operation.
- Discuss all preliminary observations with management.
- Draft realistic and achievable short-term and long-term corrective actions.
- Discuss with management the draft audit report and their responses, if available, prior to release of the final audit report.
- Follow up on critical issues raised in audit reports to determine if they have been successfully resolved.

## Individual Job Audits

One of the best ways to understand if a general audit is necessary is to perform individual job audits. A job audit is a step-by-step review of all processes conducted on a given batch of parts to evaluate if they fully meet customer requirements. A good heat-treating job audit will cover items such as:

- Incoming part inspection and material verification

- Material specification review
- Component part drawing/heat-treating specification reviews
- Part fixturing/racking method
- Procedure for recipe creation and overall process-cycle review
- Equipment selection/capability
- Process review (including atmosphere requirements)
- Testing requirements/methods/capabilities/certifications
- Shop paperwork – forms/record keeping
- Operator and quality sampling/testing and acceptance criteria

## Additional Audit Requirements

Areas such as data management and operator/maintenance personnel training (in accordance with SAE-ARP-1962) should not be overlooked. Critical questions need to be asked, including if the various heat-treat equipment has the ability to display, achieve, store and retrieve critical process data and if the company has a defined training program and is reinforcement training done on an annual basis?

Finally, companies should consider independent verification of their auditing practices. Personnel conducting such audits would observe processes being run; review equipment and methods in use; talk with supervisors, managers and engineers about policies and procedures; collect evidence that activities are as claimed; and verify operations, including outputs at the various steps.

Internal audits have broad-based objectives, including verification of items such as:

- Special procedures or tests
- Product characteristics and performance
- Implementation of procedures to match the requirements
- Defects or nonconformities and if are they being addressed
- Training, equipment capabilities and process settings

By comparison, external auditors look to verify that:

- All appropriate requirements are being followed, including supplier processes and calibration procedures, heat-treatment methods and maintenance activities
- Supplier product/service characteristics or performance measures
- Conformance to contract requirements
- All nonconformities have been addressed
- Material is as specified, and all sources are traceable

## Audit Follow-Up

Don't forget to conduct a follow-up audit within six months after the initial (internal or external) audit has taken place and the audit report has been issued. This should be scheduled before the initial audit takes places. The purpose of this follow-up is to critically evaluate if corrective action has been taken on the audit issues reported in the original report.

## In Conclusion

The most common excuse for not performing an audit is that we are too busy. However, it always seems there is enough time to do the task over again. Let's do it right the first time and every time. Perform an audit, find areas where improvement is needed and take the necessary corrective action. You'll be happy you did.

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# All About Thermocouples (Part 1)

Accurate temperature control of heat-treating furnaces, ovens and baths depends in large part on the proper choice of thermocouples as well as on ancillary items such as extension (lead) wire, protection tubes and connectors. Specifications like the latest revision to AMS 2750 (Pyrometry) have helped by focusing attention on the critical role of these important temperature sensors. Let's learn more.

## What is a thermocouple?

Thermocouples (Fig. 1) are used to sense temperature in heat-treating furnaces and are a type of electrical sensor that consists of two dissimilar metals joined together that produce an output when subjected to a difference in temperature. The joined end that is placed inside the furnace is called the "hot" (or measuring) junction. The end attached to the connector outside the furnace is called the termination end, and the end attached to the instrumentation is commonly called the "cold" end (Fig. 2). An electromotive force (EMF) is generated. EMF is measured in millivolts that are proportional to the difference in temperature between the hot and cold ends. The different types of materials used to construct thermocouples produce different output signals.

Fig. 1. Typical thermocouples and thermocouple assemblies (courtesy of Pyromation, Inc.)

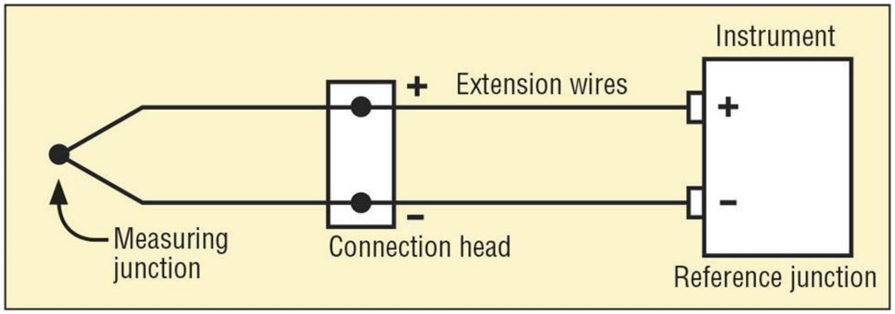


Fig. 2. Schematic of a basic thermocouple circuit[3]

**Table 1. Common thermocouple types[4]**

| ANSI Code | Positive (+) connector | Negative (-) connector | Temperature range | Limits of error | Limits of error | Limits of error |
|---|---|---|---|---|---|---|
| | | | | Range (˚F) | Standard | Special |
| J | Iron (magnetic)<br>Color: white | Constantan<br>Color: red | 0-760˚C<br>(32-1400˚F) | -18 to 293˚C (0-560˚F)<br>293-760˚C (560-1400˚F) | ±2.2˚C (±4˚F)<br>±0.75%[a] | ±1.1˚C (±2˚F)<br>±0.4%[a] |
| K[a] | Chromel<br>Color: yellow | Alumel (magnetic)<br>Color: red | -200 to 1260˚C[d]<br>(-328 to 2300˚F) | -18 to 293˚C (0-560˚F)<br>-200 to 0°C (-328 to 32°F)<br>293-1150˚C (560-2300˚F) | ±2.2˚C (±4˚F)<br>±2.2˚C (±4°F) or ±2%[a]<br>±0.75%[a] | ±1.1˚C (±2˚F)<br>±0.4%[a] |
| T | Copper<br>Color: blue | Constantan<br>Color: red | -200 to 370˚C<br>(-328 to 700˚F) | -200 to 0˚C (-328 to 32˚F)<br>0-370˚C (32-700˚F) | ±3.5°C (±1.8°F) or ±1.5%[a]<br>±3.5°C (±1.8°F) or ±0.75%[a] | ±0.5°C (±0.9°F) or<br>±0.4%[a] |
| E | Chromel<br>Color: purple | Constantan<br>Color: red | -200 to 870˚C<br>(-328 to 1600˚F) | -200 to 340˚C (-328 to 644˚F)<br>340-870˚C (644-1600˚F) | ±1.7˚C (±3.1˚F)<br>±3.1°F or ±1%[a]<br>±0.50%[b] | ±1°C (±1.8°F)<br>±0.4%[a] |
| S<br>or<br>R | Platinum/10% rhodium<br>Platinum/13% rhodium<br>Color: black[c] | Platinum/13% rhodium<br>Platinum<br>Color: red | 0-1480˚C<br>(32-2700˚F) | 0-600˚C (32-1112˚F)<br>600-1480˚C (1112-2700˚F) | ±1.5˚C (±3˚F)<br>±0.25%[a] | ±0.6˚C (±1.1˚F)<br>±0.1%[a] |
| B | Platinum/30% rhodium<br>Color: gray[b] | Platinum/6% rhodium<br>Color: red | 870-1700˚C<br>(1600˚F-3100˚F) | 870-1700˚C (1600-3100˚F) | ±0.50%[a] | ±0.25% |
| N | Nicrosil<br>Color: orange | Nisil<br>Color: red | 0-1260˚C<br>(32-2300˚F) | 0-293˚C (32-560˚F)<br>293-1260˚C (560-2300˚F) | ±2.2˚C (±4˚F)<br>±0.75%[a] | ±1.1˚C (±2˚F)<br>±0.40%[a] |
| C[d] | Tungsten/5% rhenium | Tungsten/26% rhenium | 0-2330˚C<br>(32-4200˚F) | 0-450˚C (32-842˚F)<br>450-2330˚C (842-4200˚F) | ±4.5˚C (±8˚F)<br>±1.00%[a] | - |

Notes: [a] Limited in vacuum use to 1150˚C (2100˚F). [b] When determining the accuracy of thermocouples using a percent of accuracy, the temperature scale used for these calculations is Celsius. [c] The color codes black and gray are used on extension-grade wire. [d] Type R and S use the same extension wire. Type C is now recognized by ASTM E230/E230M-12.

**Table 2.** Common thermocouple applications[5, 6]

| ANSI code | Application usage |
|---|---|
| J | Comparatively inexpensive. Suitable for continuous service to 760°C (1400°F) in vacuum, reducing or inert atmospheres. Reduced life in oxidizing atmosphere. Iron oxidizes rapidly above 540°C (1000°F), so only heavy-gauge wire is recommended for high temperature. Bare elements should not be exposed to sulfur-bearing atmospheres above 540°C (1000°F). Protection tubes should always be used in a contaminating atmosphere and above 480°C (900°F). |
| K | Recommended for continuous oxidizing or neutral atmospheres. Mostly used above 540°C (1000°F) since it provides a more mechanically and thermally rugged unit than platinum/rhodium (type R, S or B) and has longer life than iron/constantan (type J). Subject to failure if exposed to sulfur-bearing atmospheres. Preferential oxidation of chromium (positive leg) at certain low oxygen concentrations causes a phenomenon called "green rot" and large (negative) calibration drifts, most serious in the 815-1150°C (1500-2100°F) range. Applications up to 1260°C (2300°F) are possible using special precautions. Requires protection tubes when used in these temperature ranges. Excellent performance when supplied in mineral-insulated, metal-sheathed cable form. |
| T | Usable in oxidizing, reducing or inert atmospheres. Its stability makes it useful for subzero temperatures, and it has high conformity to published calibration data. Not subject to corrosion in moist atmospheres. Copper oxides above 315°C (800°F). |
| E | Recommended for continuously oxidizing or inert atmospheres since both elements are highly corrosion-resistant. Does not corrode at subzero temperatures. Highest thermoelectric output of common calibration. Stability is unsuitable in reducing atmospheres. |
| S<br>or<br>R | Recommended for higher-temperature environments. Insulators should always be high-purity alumina. Should be protected with nonmetallic protection tubes and ceramic insulators since it is easily contaminated in non-oxidizing atmospheres. Molybdenum or Inconel sheathing can be used. Continued high-temperature usage causes grain growth, which can lead to mechanical failure. Calibration drift (negative) caused by rhodium diffusion to pure leg as well as from rhodium vaporization. |
| B | Better stability than types S or R since it has increased mechanical strength. Recommended for higher-temperature environments. Lower output values. Must be protected with nonmetallic protection tubes and ceramic insulators. Easily contaminated in non-oxidizing atmospheres. Continued high-temperature usage causes grain growth, which can lead to mechanical failure. Calibration drift (negative) caused by rhodium diffusion to pure leg as well as from rhodium vaporization. Calibration (millivolt) tables do not start until above 425°C (800°F). |
| N | Nickel-based thermocouple alloy used primarily at high temperatures. While not a direct replacement for type K, provides better resistance to oxidation at high temperature and longer life in applications where sulfur is present. Used in temperature range of 315-1260°C (600-2300°F), this material is less susceptible to preferential oxidation than type K. It provides excellent performance when supplied in mineral-insulated, metal-sheathed cable form. |
| *** | Material has no oxidation resistance, so its use is restricted to vacuum, hydrogen or (truly) inert atmospheres. |

**Table 3.** Other less-common thermocouple designations[7]

| Designation | Positive (+) connector | Negative (-) connector | Maximum-use temperature | Most-common atmosphere type |
|---|---|---|---|---|
| Nickel/nickel-molybdenum[a] | Ni/18% Mo (20 Alloy) | Ni/1% Co (19 Alloy) | 1370°C (2400°F) | Reducing |
| Platinel II | 55% Pd/31% Pt/14% Au | 65% Au/35% Pd | 1260°C (2300°F) | Oxidizing |
| W | W | W/26% Re | 2300°C (4200°F) | Reducing |
| W-3 | W/3% Re | W/26% Re | 2300°C (4200°F) | Reducing |
| Notes: [a] Limited availability | | | | |

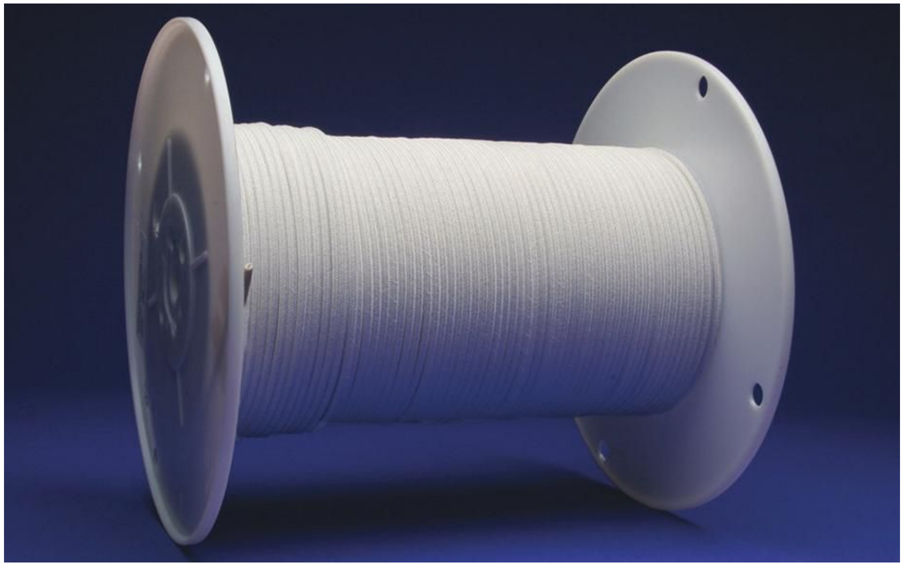


Fig. 3. Insulated spool of thermocouple wire (courtesy of GeoCorp, Inc.)

Fig. 4. Thermocouple extension wire and standard temperature connectors (courtesy of GeoCorp, Inc.)

Extension wire, which must match the thermoelectric characteristics of the thermocouple, is used to connect the termination end to the temperature instrumentation. This wire is generally specified by thermocouple type and, if calibrated, is done so at a specific set of temperatures – for example 0˚C (32˚F), 20˚C (72˚F) and 65˚C (150˚F).

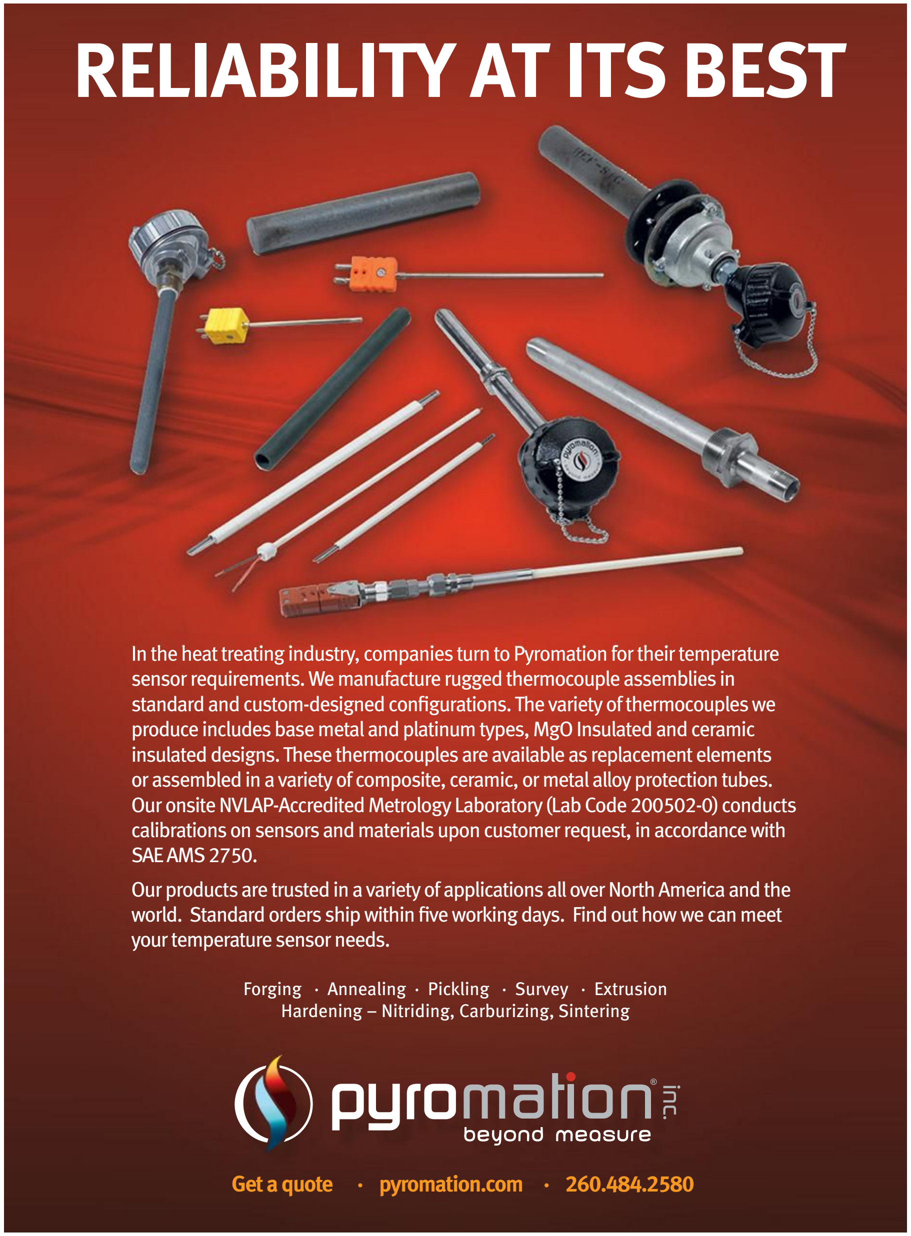
Some audit organizations feel extension wire does not need to be calibrated since the wire is only transferring the signal input from the thermocouple assembly. What is especially important, however, is to avoid creating additional junctions by splicing wire lengths together. This can cause erroneous readings. It is necessary to provide a separate metal conduit housing for the extension lead wire since it isolates or shields the lead wire from stray voltage signals like those produced by other electrical or power wiring or nearby machinery. Shielded extension wire with a so-called drain wire is available if a separate metal conduit is not available.

## What is thermocouple wire?

Thermocouple wire consists of the two dissimilar metals that are joined together at the sensing end of the thermocouple. Different thermocouple types (e.g., J, K, N) use different types of metals for these wires (Table 1 and Table 3) and have different application uses (Table 2). These wires are enclosed in various insulation packages (Fig. 3) including fiberglass, vitreous silica and ceramic fiber to name a few.

## What is extension wire?

Extension wire (Fig. 4) is used to extend a thermocouple's signal from the connection head of the thermocouple back to the instrument reading the signal. The extension-grade wire typically has a lower operational temperature limit (normally ambient temperature) in which the wire may be used. PVC, Teflon®, Kapton® and fiberglass insulators are common. As a rule, thermocouple wire may be used as extension wire, but extension wire may not be used in the sensing point (or probe part) of the thermocouple.



# All About Thermocouples (Part 2)

Our discussion of thermocouples continues this month with more practical advice and a deeper understanding into how to install and maintain thermocouples so as to control the heart of any thermal-processing system.  Let's learn more.

Correct installation techniques and good maintenance procedures are a must. The most common operational problem with furnaces today is inaccurate temperature control due, in large measure, to the fact that the thermocouples have been in the furnace longer than their normal life expectancy.

## How to Install Thermocouples

The one common feature of all furnaces is the fact that within their insulated chambers there are horizontal planes of even heat flow (i.e. areas of equal temperature called isotherms). Factors that cause these temperature planes or gradients to occur include uneven heating, inadequate circulation, uneven distribution of the workload within the furnace, improper location of the heating source and the like. In order for a thermocouple to properly sense furnace temperature it must be oriented parallel to these isotherms and installed into the furnace a minimum of 20 thermocouple diameters. For example, a 3-mm (1/8-inch) diameter thermocouple will need to be inserted 63.5 mm (2.5 inches) into the furnace chamber.

## What is the maximum-use length of a thermocouple?

The two main factors in determining useable thermocouple length are total loop resistance and prevention of electrical signal noise. Since thermocouples are made from dissimilar wire, the resistance will vary based on the type of wire as well as its diameter and length. The allowable loop resistance (typically under 100 ohms) is affected by the input resistance of the amplifier circuit to which it is attached. As a general guideline, 20 AWG or thicker wire is adequate for runs up to 30 meters (100 feet). Thermocouple wire creates a low voltage signal and should not be run near power wires, motors, etc. To help minimize noise pickup, thermocouples used in industrial furnace applications are almost always run in separate metal conduit.

There are three basic types of construction for thermocouples (Table 1). Each has advantages and disadvantages. For example, ceramic-beaded thermocouples (Fig. 1) lack flexibility when

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**Table 1. Common thermocouple construction techniques[7]**

| Construction method | Type |
|---|---|
| Ceramic beaded | High-purity (99.9%) alumina ($Al_2O_3$) |
| | Mullite (70% $Al_2O_3$ + 30% $SiO_2$) |
| | Steatite |
| Insulated | Plastic (polyvinyl, Teflon, etc.) |
| | Glass braid |
| | Ceramic fiber |
| Metal sheathed (mineral insulated: MgO, $Al_2O_3$) | Stainless steel |
| | Inconel<br>Specialty high-performance alloys |

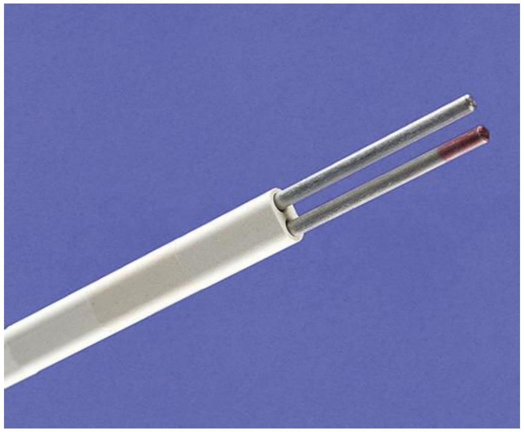

Fig. 1. Ceramic-beaded thermocouple (courtesy of GeoCorp, Inc.)

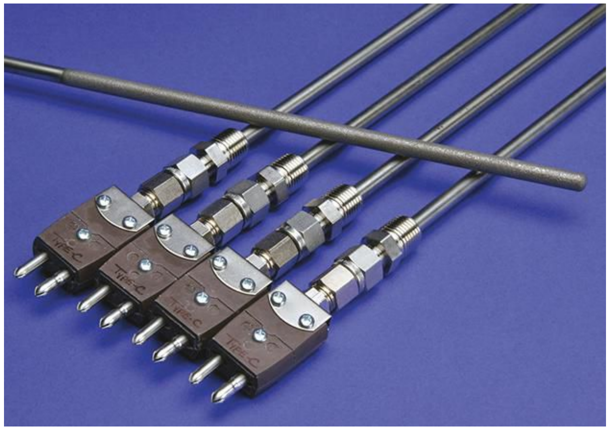

Fig. 2. Metal-sheathed thermocouples (courtesy of GeoCorp, Inc.)

Fig. 3. Typical thermocouple protection tubes (courtesy of GeoCorp, Inc.)

compared to base-metal thermocouples, which can have various types of insulation placed directly on the wire, making them extremely flexible. By contrast, ceramic-beaded thermocouples can often be used at higher operating temperatures than many of their base-metal counterparts. Mineral-insulated, metal-sheathed thermocouples (Fig. 2) provide excellent performance, and they can be used as replacement elements to ceramic-beaded, base-metal elements in many cases. Metal-sheathed cable is offered in many sizes and materials and can be optimized to help provide long-lasting, stable temperature measurements.

## Why protection tubes?

A thermocouple is often inserted into a protection tube (Fig. 3). Protection tubes (Table 2) are used to shield thermocouples from contaminants and/or mechanical damage. The heat treater must be aware that all types of thermocouple protection tubes can crack or distort, potentially damaging or exposing the thermocouple to the environment they were intended to protect it from. In addition, contamination from handling/touching the wires, oils and dirt inside the protection tube and contamination buildup on the outside of the protection tube are some of the many factors that can cause thermocouple error.

## Attaching Thermocouples in Workloads

For stationary loads, one of the best ways to determine part temperature is to use one or more workload

**Table 2.** Types of protection tubes used in the heat-treat industry[4,5]

| Material | Maximum-usage temperature | Typical heat-treat applications | General comments |
|---|---|---|---|
| Steel | 540˚C (1000˚F) | Annealing, drying, stress relief, tempering | Useful in a variety of low-temperature applications in noncorrosive environments |
| Stainless steel (446) | 1090˚C (2000˚F) | Hardening, nitriding, salt baths | Good resistance to corrosion at high temperatures, highly resistant to sulfur attack |
| Inconel 600/601 | 1150˚C (2100˚F) | Carburizing, hardening, nitriding, gas generators, salt baths | Most common materials in use today. Greater mechanical strength than 446 SS; excellent resistance to corrosion and oxidation at high temperatures. Inconel 601 is more resistant to the influence of sulfur than 600. Both materials are subject to hydrogen embrittlement. |
| Metal/ ceramic | 1370˚C (2500˚F) | Sintering and high-temperature heat treatments | Superior oxidation resistance; thermal conductivity equal to that of stainless steel; typical composition 77% Cr/23% $Al_2O_3$ |
| Mullite | 1540˚C (2800˚F) | Annealing, forge furnaces, metallizing ceramics | Impervious to gases at high temperatures. Good thermal shock but poor mechanical shock; often necessary to provide secondary tube protection; vertical orientation recommended; typical composition 65% $Al_2O_3$/35% $SiO_2$ |
| Silicon carbide | 1650˚C (3000˚F) | Soaking pits, kilns | Excellent thermal conductivity for quick response to temperature changes; eliminates possibility of iron pickup; can be used as a secondary protection tube for resistance to thermal shock; typical composition 90.0% SiC/9% $SiO_2$ |
| Alumina | 1870˚C (3400˚F) | Kilns | Fair resistance to thermal and mechanical shock; impervious to gases up to 1760˚C (3200˚F); typical composition 99.7% $Al_2O_3$ |

**Table 3.** Typical industry standards for thermocouples

| Organization | Standard number | Description |
|---|---|---|
| ASTM | E230 | American Society for Testing Materials |
| ANSI | MC 96.1 | American National Standard Institute |
| IEC | 584 – 1/2/3 | European Standard<br>(International Electotechnical Commission) |
| DI | EN 60584 – ½ | Deutschee Industrie Normen |
| BS | 4937.1041 / EN 60584 -1/2 | British Standard |
| NF |  | Norme Francaise |
| JIS | C 1602, C 1610 | Japanese Industrial Standard |
| GOST | 3044 | Russian Commonwealth Standard |

thermocouples. Ideally, you would like to have access to an internal blind hole that would allow you to measure the core temperature of the thickest cross section of the part. Unfortunately, we do not always have this luxury. The next best choice is to use dummy blocks that are predrilled and representative of the maximum cross section of the parts being run. In lieu of a blind hole or dummy block, wiring the thermocouple in intimate contact with the surface of the part is the next best option, using the rule of thumb of one hour per inch of cross-sectional area for soaking the part.

Be aware that some people suggest tack welding a thermocouple tip, but this practice is highly questionable because it can change the millivolt signal and provide inaccurate results. Epoxy or other adhesives fail prematurely in service and are not a good choice. Running a test load under simulated production conditions is often the best way to determine heat-up and soak times for moving loads or in furnaces with internal transfer mechanisms.

## Thermocouples in the Heat-Treat Industry

The heat treater, not the original equipment manufacturer or maintenance department, should be responsible for the selection of the type of thermocouple as well as making sure that they are in the correct position within the furnace (both location and insertion depth) to accurately sense and control furnace temperature. Thermocouples located too close to heating or insulation sources or too close to the workload itself will not represent true furnace temperature. Temperature uniformity checks of the workload areas must factor in deviation from the control thermocouple. Although temperature offsets are allowed in some instances, this practice is highly discouraged.

The majority of heat-processing applications in the metals industry, including virtually all heat-treating processes, occurs in the range of -185 to 1650˚C (-300 to 3000˚F). No one type of thermocouple can span this entire range, and quite often we attempt to use a particular thermocouple well beyond its normal temperature range simply because “it’s available.” This practice should be avoided since it affects both accuracy and, once exposed to abnormal conditions, may alter its life expectancy. As strange as it seems, it is not uncommon to find the wrong type of thermocouple being used to control a critical process.

Thermocouples must be checked regularly for accuracy against a known certified standard (probe) thermocouple, and this procedure should be done (at least) annually. This calibration must take place while the thermocouple is installed in its normal operating location for reliable measurements. The operating life of a thermocouple depends on its operating temperature, time at operating temperature, ambient temperature, cyclic range (high to low temperature variation) and, most often overlooked in heat-treating applications, the influence of contaminants either on an exposed thermocouple or on the protection tube itself. Many thermocouples are replaced on a periodic basis, typically every six to nine months depending on the severity of the end-use application.

## Industry Standards

Thermocouple wire is supplied according to various industry standards (Table 3).

## Final Thoughts

Thermocouples are not, as many people believe, a "set it and forget it" technology. They require constant monitoring and confirmation of accuracy to ensure that the temperature being sensed and controlled is precise. Remember, the quality of your products is highly dependent on these simple and relatively inexpensive devices, which makes their selection, care and replacement critical to your success.

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# Ice Blasting

Baby, it's cold outside. So what better month to talk about ice blasting – a technology that may have a future in the heat-treatment industry? Let's learn more.

## What is ice blasting?

Ice blasting is a cleaning process using dry ice ($CO_2$). It is most commonly associated with cleaning of industrial equipment such as heat exchangers, tanks, and gas and steam turbines (Fig. 1). One of its key advantages is that it can typically be performed without major disassembly of components while avoiding fire or electrical hazards. Ice blasting is commonly used to clean food-processing equipment to effectively decontaminate surfaces to avoid such bacterial agents as salmonella enteritidis, E. coli and listeria.

In fact, the EPA recommends dry-ice blasting as an alternative to many types of solvent-based cleaning. Dry-ice blasting can clean multiple objects with differing, complex geometries at the same time. Due to the blast media sublimating without residue, dry-ice blasting is used as a maintenance tool in a variety of industries, including:

- Automotive
- Food manufacturing/processing
- Power generation
- Plastics manufacturing
- Printing and packaging
- Chemical manufacturing
- Fire restoration, mold remediation and historical preservation
- Petroleum refining (catalytic reduction units)
- Beverage and bottling
- Foundry.

## How it Works

Carbon dioxide ($CO_2$) blasting works by combining three primary factors: pellet kinetic energy, thermal-shock effect and thermal-kinetic effect. Optimized cleaning is a result of controlling the various process parameters, including:

- Compressed air pressure
- Blast nozzle type (velocity distribution)
- Pellet size and density
- Pellet mass rate and flux density (particles per unit area per second)
- Pellet kinetic energy

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Fig. 1. Ice blasting of heat-recovery steam-generator components

### Pellet Kinetic Energy

At high impact velocities and direct head-on impact angles, the kinetic effect of solid $CO_2$ pellets is minimal when compared to other media (e.g., grit, sand, glass). This is due to the relative softness of a solid $CO_2$ pellet, which is not as dense and hard as other projectile media. Also, the pellet changes phase from a solid to a gas almost instantaneously upon impact, which effectively provides an almost nonexistent coefficient of restitution in the impact equation. Very little impact energy is transferred into the coating or substrate, so the ice-blasting process is considered to be nonabrasive.

### Thermal-Shock Effect

Instantaneous sublimation (phase change from solid to gas) of $CO_2$ pellets upon impact absorbs maximum heat from the very thin top layer of surface coating or contaminant. Maximum heat is absorbed due to latent heat of sublimation.

The very rapid transfer of heat into the pellet from the coating top layer creates an extremely large temperature differential between successive microlayers within the coating. This sharp thermal gradient produces localized high shear stresses between the microlayers. The shear stresses produced are also dependent on the coating's thermal conductivity and thermal coefficient of expansion/contraction, as well as the thermal mass of the underlying substrate. The high shear produced over a very brief expanse of time causes rapid microcrack propagation between the layers, leading to contamination and/or coating final bond failure at the surface of the substrate.

### Thermal-Kinetic Effect

The combined impact energy dissipation and extremely rapid heat transfer between the pellet and the surface cause instantaneous sublimation of the solid CO2 into gas. The gas expands to nearly 800 times the volume of the pellet in a few milliseconds in what is effectively a "microexplosion" at the point of impact.

The microexplosion as the pellet changes to gas is further enhanced for lifting thermally fractured coating particles from the substrate. This is because of the pellet's lack of rebound energy, which tends to distribute its mass along the surface during the impact. The $CO_2$ gas expands outward along the surface, and its resulting "explosion shock front" effectively provides an area of high pressure focused between the surface and the thermally fractured coating particles. This results in a very efficient lifting force to carry the particles away from the surface.

## Heat-Treat Case Study

A company in Texas with a cold-wall horizontal vacuum furnace contracted a local ice-blasting service to expedite the cleaning of their furnace interior. The hot zone had been removed so that all surfaces of the cold wall and the front door were easily accessible. A thick buildup of braze residue and soot coated these surfaces.

Using low pressure to avoid removing any of the interior's epoxy-painted surface, the removal process began. It was observed that some exposed areas showed signs of failing paint coverage (flakes and bubbles). The process was able to remove the damaged paint and leave behind a clean surface that could be recoated with epoxy. Altogether, the blasting produced only a dust-like residue at the entrance floor of the furnace. This was easily swept up with a broom and dustpan.

Fig. 2. Portable ice-blasting machine

The entire cleaning process took about five hours of blasting and used a minimal amount of carbon dioxide. The result was a pristine interior. In fact, the process worked so well that the decision was made to attempt to clean a number of other internal parts on the hot zone itself – from graphite to steel – and even the copper power feedthroughs.

The heavy buildup came off of all parts with no damage introduced. Regular high-velocity nozzles and high ice flow were used on the copper and steel. For the graphite, a fragmentation nozzle (makes the $CO_2$ pellets similar to the consistency of table sugar) was used. The process required another 5-6 hours of blast time to remove all contaminants from the individual components that made up the hot zone. Thus, the entire process could be completed in a 10-hour workday.

## Reasons for Using Dry-Ice Blasting

Users have provided the following list of reasons why they feel dry-ice blasting should be considered to simplify and reduce the time involved in common maintenance practices.

1. It has fewer cleaning cycles and more effective cleaning cycles (reduced equipment downtime).
2. Machines can be cleaned in place with minimal, if any, disassembly/reassembly (reduced equipment downtime).
3. It is a dry process with no residue (environmentally friendly).
4. It is a quicker cleaning method than alternatives (reduced equipment downtime).
5. It is nonabrasive, nonflammable and nonconductive: dry ice blasting won't damage most substrates and can be used safely on electrical components provided the power is turned off and remains off until the components have returned to room temperature.
6. There is minimal secondary waste cleanup and disposal (reduced labor costs – temporary containment areas can possibly be reused).
7. It can get into tight spaces that many other methods can not.
8. It meets USDA, FDA and EPA guidelines (environmentally friendly).
9. There is minimal exposure to chemicals or grit media (operator safety).
10. It is not as labor intensive as traditional cleaning methods (reduced equipment downtime, lower cost).

## Summary

The cost, speed and efficiency of ice blasting make it an option that can possibly reduce maintenance time for heat treaters.

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1. Brandon Kelly, Global Ice Blasting (www.globaliceblasting.com), technical contributions and private correspondence

# The Importance of pH

Once upon a time, in a science class far, far removed, the subject of pH was discussed. Little did we know at the time how important these two simple consonants, combined in such an odd way, were to the water systems that cool our heat-treating equipment. Let's learn more.

## The Water Molecule

All substances are made up of millions of tiny atoms. These atoms form small groups called molecules. In water, for example, each molecule is made up of two hydrogen (H) atoms and one oxygen (O) atom (Fig. 1). The formula for a molecule of water is $H_2O$ (there are two hydrogen atoms needed for each oxygen atom to form a stable compound).

### Introduction to pH

The term pH is used to describe a unit of measure to indicate the degree of acidity or alkalinity of a solution. It is measured on a scale of 0-14. The term pH is derived from "p" (the mathematical symbol of the negative logarithm) and "H" (the chemical symbol of hydrogen).

**Table 1. pH chart**[2]

| Concentration of hydrogen ions compared to distilled water | pH level | Examples of solutions at this pH |
|---:|:---:|---|
| 10,000,000 | pH=0 | Battery acid, Strong hydrofluoric acid |
| 1,000,000 | PH=1 | Hydrochloric acid secreted by stomach lining |
| 100,000 | pH=2 | Lemon juice, gastric acid, vinegar |
| 10,000 | pH-3 | Grapefruit, orange juice, soda |
| 1,000 | pH=4 | Acid rain, tomato juice |
| 100 | pH=5 | Soft drinking water, black coffee |
| 10 | pH=6 | Urine, saliva |
| 1 | pH=7 | "Pure" water |
| 1/10 | pH=8 | Sea water |
| 1/100 | pH=9 | Baking soda |
| 1/1,000 | pH=10 | Great salt lake, milk of magnesia |
| 1/10,000 | pH=11 | Ammonia solution |
| 1/100,000 | pH=12 | Soapy water |
| 1/1,000,000 | pH=13 | Bleaches, oven cleaner |
| 1/10,000,000 | pH=14 | Liquid drain cleaner |

The formal definition of pH is the negative logarithm of the hydrogen-ion activity. It is expressed mathematically by the formula:

(1) $$pH = -\log[H+]$$

Thus, pH provides a way of expressing the degree of the activity of an acid or base in terms of its hydrogen-ion

**Table 2. Typical water requirements for open systems[2]**

| Description | Value |
|---|---|
| Hardness (calcium carbonate) | 7-10 grains/gallon[a] (120-170 ppm) |
| Total suspended solids | 10 ppm |
| Total dissolved solids | 200 ppm |
| Iron | 0.3 mg/liter |
| Aluminum | 0.05-0.2 mg/liter |
| Copper | 1.0 mg/liter |
| pH | 7.0-8.0 |
| Odor | 3 threshold odor number |
| Conductance | ≤ 300 μS/cm |
| Maximum water temperature (inlet) | 31°C (88°F) |
| System drain pressure | ≤ 3.5 psig |

Notes: [a] Grains per gallon is defined as 64.8 mg (1 grain) of calcium carbonate per 3.79 liters (1 U.S. gallon) or 17.1 ppm.
[b] For best cooling efficiency and component longevity, the water supply should be treated to prevent corrosion and scale (controlled by phosphonate test; range 15-20 ppm), scum formation, algae and other biological agent buildup and the like.

**Table 3. Water requirements for closed hydronic systems[4]**

| Parameter | Optimal conditions | Comments |
|---|---|---|
| Glycol freeze protection | 30-50% | Below 20% can promote the growth of bacteria. Above 50% will dramatically reduce heat-transfer ability. Glycol seepage can occur at O-rings and seals. |
| Corrosion inhibitor | [A] | Without the addition of nitrite or molybdate inhibitors, corrosion of the metallic compounds will begin and eventually lead to leaks. |
| pH | 9-10.5 | A pH below 9.0 will promote corrosion of steel and above 10.5 will promote the corrosion of brass and copper. |
| Conductivity | 700-3,200 μS/cm | Conductivity above 3,500 μS/cm will cause the water to become physically abrasive and damage O-rings. Addition of chemicals to the water will raise the conductivity. |
| Hardness | 100-300 ppm (5.8-17.5 grains/U.S. gallon) | Artificially soft water can be aggressive to the system. The use of unsoftened water is recommended. Do not use distilled or purified water. |
| Bacteria/mold | None | The growth of bacteria will cause erosion of seals, and the deposit of a bacterial slime will clog the system. Bacteria can attack the O-rings and cause premature failure. Glycol above 20% will kill any bacteria. |

[A]Notes: Molybdate (100-150 ppm) or nitrite inhibitor (800-1,200 ppm)

activity.

The pH value of a substance is directly related to the ratio of the hydrogen ion $[H^+]$ and the hydroxyl ion $[OH^-]$ concentrations. If the hydrogen ion concentration is greater than the hydroxyl ion concentration, the compound is acidic and the pH value is less than 7. If the hydroxyl ion concentration is greater than the hydrogen ion concentration, the compound is basic with a pH value greater than 7. If equal amounts of hydrogen

**Fig. 1.**
**The water molecule**

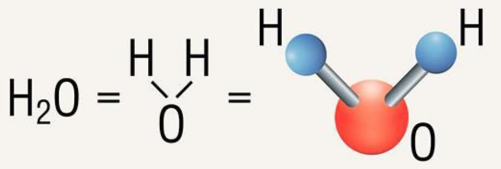
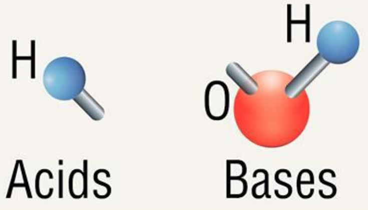


**Fig. 2.**
**Acids and bases**



ions and hydroxyl ions are present, the material is neutral with a pH of 7.

Acids and bases have, respectively, free hydrogen and hydroxyl ions. Since the relationship between hydrogen ions and hydroxyl ions in a given solution is constant for a given set of conditions, either one can be determined by knowing the other. Thus, pH is a measurement of both acidity and alkalinity, even though by definition it is a selective measurement of hydrogen-ion activity. Since pH is a logarithmic function, a change of one pH unit represents a tenfold change in hydrogen-ion concentration (i.e., of both the hydrogen ion and the hydroxyl ion at different pH values, Table 1). Note that each decrease in pH by one pH unit means a tenfold increase in the concentration of hydrogen ions.

Fig. 3. Water problems leading to sludge buildup and flow blockage in the top cool of an integral-quench furnace

The concentration of hydrogen ions in a solution is very important for living things. This is due to the fact that hydrogen ions are positively charged, and they alter the charge environment of other molecules in solution. By putting different forces on the molecules, the molecules can change from their normal shape.

A substance that increases the concentration of hydrogen ions (lowers the pH) when added to water is called an acid. A substance that reduces the concentration of hydrogen ions (raises the pH) is called a base. Finally, some substances enable solutions to resist pH changes when an acid or base is added. Such substances are called buffers.

### Acids and Bases in Water

When an acid is poured into water, it gives up $H^+$ (hydrogen ion) to the water (Fig. 2). When a base is poured into water, it gives up $OH^-$ (hydroxyl ion) to the water.

### Water Quality in the Heat-Treat Shop

Water is used in most of our heat-treat shops for a variety of purposes. Examples include water-cooled bearings on fans and rolls, seals on pit-furnace covers, water-cooled jackets on continuous furnaces, water-cooled jackets for quench tanks, top or side cooling chambers, inner doors and plate coils, endothermic generator "top hats," seals (e.g., oil seals on rotary-hearth furnaces) and makeup water for water systems to name a few.

Water-quality requirements are often defined differently for open (Table 2) and closed (Table 3) systems. Open systems are typically more problematic because the issue of water quality varies. Water is often classified as "soft" or "hard" depending on its mineral content. Soft water has an ideal hardness of approximately 120 ppm (7 grains/gallon). Hard water often results in the formation of mineral deposits, which can lead to blockages in water systems (Fig. 3).

Further, we must ensure that the water being discharged from our heat-treatment operations is clean and meets EPA standards. Finally, we must be especially careful to avoid cross contamination from other sources in the shop (e.g., polymers, quench oils, chemicals, etc.).

## In Conclusion

As heat treaters, if we take our water supply and water systems for granted, unexpected surprises, unwanted downtime and expensive repairs may result. This just goes to show the importance of those two little consonants coming together.

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# Soot

Soot … the very word sends shivers down the spines of even the bravest of heat treaters. The problems this seemingly innocuous black powder creates cause management to bemoan lost productivity, maintenance men to vow to seek out those responsible and, until a permanent solution is found, makes all our lives miserable. So, how can we avoid creating soot in the first place and what can we do if/when it forms? Let's learn more.

## Carbon Penetration

The presence of soot raises many red flags, one of them being that carbon from unstable furnace atmospheres is rapidly absorbed into refractory linings, which are incredibly porous. Infrequent or improper burnouts (so-called flash burnouts) give a false sense of security, as they address only the refractories near surface layer. Once absorbed, carbon will continue to diffuse deeper and deeper

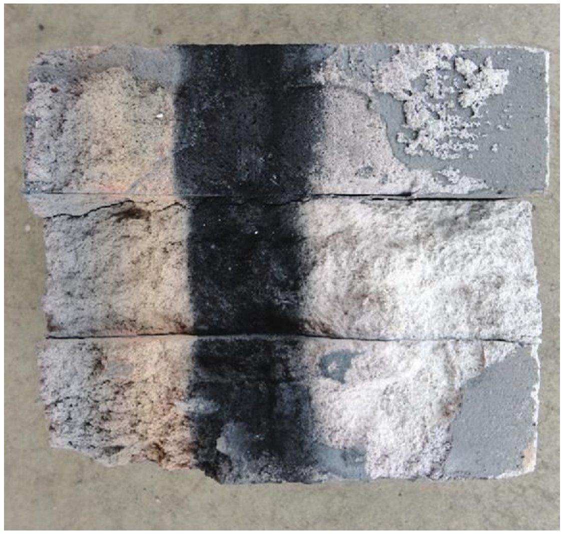

Fig. 1. Carbon-rich subsurface layer inside a refractory wall

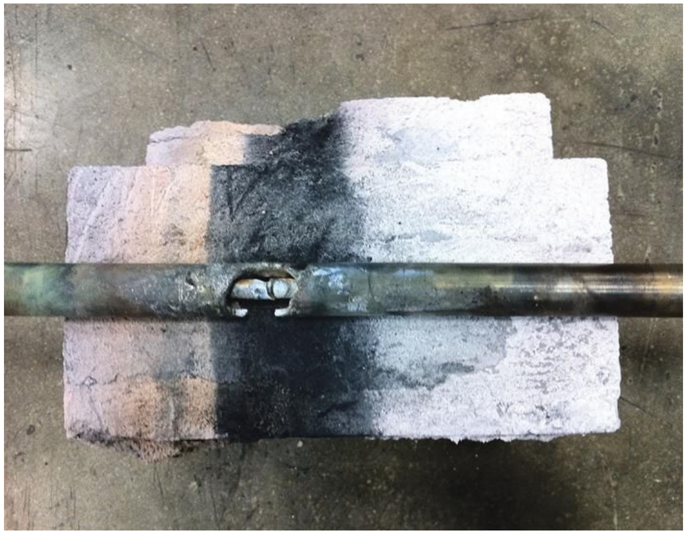

Fig. 2. Oxygen probe and its alloy protection tube destroyed by melting at the exact location of the carbon-rich subsurface layer

Fig. 3. Alloy radiant tube destroyed by soot accumulation in the insulation

into the refractory (Fig. 1). The penetration halts in the temperature range of approximately 900-1300°F (480-705°C).

As more and more carbon is absorbed, the refractory loses its thermal properties and becomes conductive. The result can be damage or melting of alloy components that extend into or through these conductive layers. Heating elements, terminal ends and support hangers in electric furnaces are particularly vulnerable as are atmosphere inlets, sample tubes and even oxygen probes (Fig. 2). Gas-fired furnaces are not immune because radiant tubes are also susceptible to carbon attack and metal dusting (Fig. 3).

Many heat treaters believe box and integral-quench furnaces are only vulnerable to the effects of soot if they are performing deep-case carburizing cycles. This is not true. Furnaces running shallow case depths or even atmospheres neutral to medium and high-carbon steels (e.g., 0.60-0.80%C) are at risk.

The use of low-iron brick (<0.5% Fe) helps to retard carbon absorption. In all cases, a robust and frequent burnout procedure in which air reacts with the soot (carbon) to form carbon dioxide and carbon monoxide gas must be established to return the furnace to good working order.

There are several ways in which this furnace burnout can be accomplished. Each have advantages as well as limitations, and it is important to recognize that, done improperly, air burnouts may cause extensive damage to equipment. Extreme care must be exercised so as to avoid this situation. For integral-quench or pusher-style carburizing furnaces running endothermic gas, the following methods have been found to be effective.

**Method 1:** Raise the dew point of the furnace atmosphere to around +70°F (20°C) with air additions and maintain this condition over a long period of time (typically 24-72 hours).

Fig. 4. Soot depositing on the front load table of a mesh-belt furnace after one hour with the furnace atmosphere out of control

Fig. 5. Typical vacuum-furnace hot zone (Photograph courtesy of ALD-Holcroft)

**Method 2:** Remove the furnace atmosphere and add a fixed volume of air, entering the furnace through a flowmeter, for a prolonged period of time (typically 12-36 hours). This is often combined with opening and closing of the furnace door(s).

**Method 3:** Remove the furnace atmosphere and use an air lance or wand (under highly reduced pressure) directed at locations in the furnace interior with heavy soot deposits. This is normally followed by the introduction of air through a flowmeter for a period of time (typically 4-12 hours). Special note: This is the riskiest of the air burnout methods given the potential to significantly damage the furnace interior, including an alloy fan if present. Only highly experienced personnel should be allowed to attempt this procedure.

Care must be taken to prevent overheating of the furnace during an air burnout. For this reason, the furnace temperature is usually lowered to around 1550-1600°F (845-870°C), and the process is stopped if the furnace temperature rises by 100°F (38°C) or more. Most people are not aware that carbon (soot) burns at over 4500°F (2480°C), which is high enough to melt through any of the materials in the furnace!

Furnace burnouts must be done before carbon has had a chance to build up in the refractory. They must also be frequent enough to ensure carbon diffusion is not occurring. One way to measure the effectiveness of the current burnout procedure is to extract a small section of insulation and inspect it on a frequent basis. If at any point it is determined that the insulation is absorbing an unacceptable amount of carbon, either increase the time of the burnout or the number and/or duration of burnouts or use some combination of the methods explained above.

Soot not only affects alloy life, but it deposits on the work and is then carried into the quench tank, where it negatively influences the performance and life of the quenchant.

Finally, it is a good idea to monitor the carbon potential of the furnace atmosphere during operation (via shim stock, turnbars or other methods). Atmosphere systems must be properly calibrated, and control devices such as oxygen probes should be complemented by three-gas analyzers and/or dew-point meters. Also, an automatic burnout of the oxygen probe during processing is mandatory to make sure the probe does not soot up and give false readings. In addition, the reference-air system should be checked to be sure it is operating properly. Oxygen probes should be changed based on manufacturer's recommendations.

## Furnace Atmospheres Gone Wild

Simply stated, an out-of-control gas generator or furnace atmosphere is a nightmare scenario in which soot can form in seconds and build up significantly in just a few minutes (Fig. 4). Fluctuating dew points, either at the generator or the furnace, are a clear indication of an unstable atmosphere condition.

Today, many endothermic generators are operated in a dew-point range of +40°F to +45°F. Lowering the endothermic-gas generator dew point aids in reducing the amount of hydrocarbon enrichment gas required at the furnace but increases the frequency of maintenance on the generator itself.

## Soot and Tar Formation in Vacuum Carburizing

Two significant technological advances, low-pressure techniques – at or below 20 torr (27 mbar) – and the use of acetylene ($C_2H_2$), have so significantly reduced soot and tar formation in vacuum-carburizing furnaces that only routine maintenance is now required (Fig. 5). Today, typical maintenance of these systems consists of vacuuming out the chamber every 3-8 weeks, and the amount of soot produced is in the order of less than a handful.

The reasons for this are the reduced volume of gas needed, the lower pressure and the fact that acetylene catalytically decomposes. In other words, as acetylene enters the hot zone, it remains thermally stable until coming in contact with the load surface. In addition, the pyrolysis reaction is virtually instantaneous, which allows for carbon to quickly begin diffusing into the steel without the formation of undesirable intermediate compounds unlike so many of the other hydrocarbon gases previously used.

## Final Thoughts

Soot formation inside heat-treating furnaces should be taken very seriously. Methods must be devised to prevent its formation and/or limit its deleterious effects. This is one New Year's resolution worth keeping!

# Atmosphere Furnace Burnouts

As a rookie, the task of performing weekly burnouts of the atmosphere furnaces in our heat-treat shop fell to the Doctor. Perhaps it was the unbridled enthusiasm of youth, but the job was never boring and always filled with excitement as one tried to invent new and innovative ways to perform (and shorten) the seemingly simplest of tasks on a Friday night. It was only later that the importance of a proper burnout on the furnace's ability to maintain precise process control and lower the frequency of furnace maintenance struck home. Over the years, many people have forgotten how or even why we perform this function in the heat-treat world. Let's learn more.

## The Why

Under a variety of operating conditions, carbon, in the form of soot, can build up inside a heat-treat furnace. The presence of soot adversely affects the life of the internal alloy components; is absorbed into the refractory lining, changing its thermal characteristics; and can interfere with effective heat and carbon transfer to the parts. Plugged gas inlets, malfunctioning oxygen probes and inaccurate temperature readings are some of the most commonly reported problems.

Soot formation can also have devastating long-term maintenance consequences, requiring us to change out alloy fans, radiant tubes, roller rails, chain guides and protection tubes. The life of electric heating elements and radiant tubes are often shortened. Left unchecked, refractory life is dramatically reduced as well.

Case-hardening processes such as carburizing and carbonitriding are classic sources of excess carbon, but other processes (such as sintering of powder-metal parts) produce carbon due to the release of organic binders into the furnace atmosphere. A common source of soot is an endothermic or nitrogen/methanol atmosphere that is out of control (Fig. 1).

## The How

For all of the above reasons and more, it is periodically necessary to remove this unwanted carbon from our furnaces. It is important to recognize, however, that the reaction of carbon with oxygen to form carbon dioxide is highly

Fig. 1. Soot formation on the front load table of a mesh-belt furnace after one hour with an out-of-control furnace atmosphere

exothermic. For example, 96 kcal per gram mole of energy is released 925°C (1700°F). Thus, great care must be exercised to avoid overheating and/or creating localized hot spots, which will rapidly and catastrophically damage the furnace.

It is customary to help control the carbon burnout reaction by lowering the furnace temperature in the range of 845-870°C (1550-1600°F) before commencement of the burnout process. If at any time during the burnout procedure the temperature rises 38°C (100°F) or more, the process should be temporarily halted.

Here are the most common ways in which a furnace burnout can be accomplished:

1. With the atmosphere removed from the furnace, use a fixed volume of clean, dry and filtered air entering through a flowmeter (typically at a rate no greater than 20% of the normal atmosphere flow) for a prolonged period of time, typically 3-8 hours.
2. With the protective atmosphere still inside the chamber, raise the furnace dew point into the range of +15 to +21°C (+60 to +70°F) by introducing clean, dry and filtered air or water-saturated air for a prolonged period of time, typically 12-72 hours.
3. With the atmosphere removed from the furnace, introduce clean, dry, filtered and (flow and pressure) regulated compressed air through a steel lance or wand. Manually direct the air stream toward locations of heavy soot deposits for no more than 10-15 seconds at a time. This is then followed by the introduction of air through a flowmeter for a fixed period of time, typically 1-4 hours.

While all methods are used in the industry, method 3 must be performed with extreme care to avoid damaging the furnace interior from overly high-pressure air, long-duration air impingement in a localized area or striking interior components such as the circulating fan (as The Doctor found out one day). Remember, soot (carbon) combusts at approximately 2480°C (4500°F), high enough to melt any of the materials present in the furnace.

It has also been found[3] that carbon dioxide may be used either undiluted or with an inert carrier gas to accelerate the burnout process. The addition of small amounts of water (i.e. saturated nitrogen, air or carbon dioxide) can speed up the process as well. However, the use of oxygen (as opposed to air) is highly discouraged due to the rapid combustion and acceleration of temperature (as the Doctor again knows all too well).

## The When

In the case of medium- to deep-case carburizing, weekly furnace burnouts are typically recommended (dependent to some degree on the dew point of the incoming endothermic gas). Monthly atmosphere burnouts should be adequate in most other cases. To an extent, the time between burnouts (and their duration) is dependent on when the last burnout was conducted.

During normal operation, it is important to try to keep furnaces as soot-free as possible by first making sure they are running the right gas flows at the proper carbon-potential setpoint. The enriching gas additions being used should be limited over the course of the total cycle to an acceptable percentage of the total flow (typically 10-15% in the case of natural gas).

It is noteworthy that most automatic systems add enriching gas in relatively short increments of high flow. These should be timed to determine exactly how much enrichment is actually entering the furnace, and in many cases the peak flow should be limited to prevent too much enriching gas from entering the chamber in too short a time.

One of the many consequences of an improper or partial burnout is that carbon absorbed into the refractory continues to diffuse inward. This creates a situation in electric furnaces where the refractory becomes conductive and element terminals or alloy extending through the refractory may melt.

## The Oxygen Probe

With so many furnaces using oxygen (carbon) probe control systems, it is worth briefly talking about them. The recommended probe burnout frequency, flow and duration vary by manufacturer based on sheath diameter and tip design but are also dependent on the process-carbon-level setpoint. In the case of gas carburizing, every four hours is a common practice using 0.05-0.3 $m^3$/hour (2-10 cfh) of air piped to the burn-off fitting on the head of the probe. Pumped room air or filtered combustion air are most commonly used. Compressed air is not recommended due to the contamination of water and oil often present that can damage the oxygen probe. It is good practice never to exceed 90 seconds to avoid overheating the tip of the probe.

A consistent way to verify a correct burnout is to monitor the oxygen millivolts of the carbon controller during the burnout phase. If a proper burnout is taking place, the oxygen will drop below 200 millivolts. This can also vary based upon the circulation in the furnace and the probe placement.

A possible side effect of extended burnout duration is the oxidation of the tip of the sensor. The problem can manifest itself with oxygen millivolts being elevated over time, which will require a lower CO-factor setting for the same calculation of carbon.

## The Generator

Today, many endothermic gas generators are run at dew points in the +1.5 to +7°C (+35 to +45°F) range. The primary reason for this is to reduce the frequency of generator maintenance. The consistency of the endothermic gas produced at the generator significantly affects all downstream processes. In case hardening, the volume of enrichment gas that is required to achieve a given surface carbon content is affected. The generator should be controlled by a dew-point analyzer and monitored with a three-gas analyzer (CO, $CO_2$, $CH_4$) so as to detect if the generator catalyst is sooted. The $CH_4$ value rising above 0.5% carbon is an indicator that a generator burnout is needed. If the value does not fall below this threshold after burnout, a catalyst change is required.

## The Conclusion

A successful atmosphere burnout will return the furnace to productive service. Once the furnace burnout is complete, it is natural to ask if one can begin running heat-treat cycles immediately afterward. While possible for many processes, it has been suggested[5] that several conditioning runs be performed under neutral atmosphere prior to the onset of carburizing or carbonitriding. The secret is to avoid the control system making unnecessarily high enriching-gas additions to compensate for the reabsorption of some carbon by the furnace after a burnout.

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# Vacuum Furnace Maintenance

We all have haunting memories of the one that got away. Truth be told, the Doctor is still in agony over not attending an excellent ASM Vacuum Maintenance Seminar held in Anaheim, Calif. a few years ago. Proper maintenance of all types of furnaces is important, but maintenance on vacuum furnaces is especially critical to their operation. Let's learn more.

## Common Problems and Solutions

Vacuum processing is designed to keep parts "bright and clean." As such, discolored parts (Fig. 1) or contaminated work (Fig. 2) is just not acceptable. Knowing where to look, what to maintain and how often to do it become key concerns. Discoloration can often be traced to air (oxygen), dirt (debris, oil, cleaning agents) or water vapor.

Sources of air infiltration include dirty or damaged door seals, leaky or cracked O-rings and broken thermocouples. Wiping clean and re-greasing door O-rings prior to every run, cleaning out loose debris from the furnace interior, inspecting thermocouples and power feed-throughs, and performing weekly leak-up tests are simple, yet effective, routine preventive measures to detect and avert problems with air entering the furnace during processing.

Sources of "dirt" include residues left on parts (oils, cleaning agents, metal fines), debris left in hot zones and contaminants deposited on cold vessel walls that can be carried back into the chamber over time by gas circulation and other means (Table 1). Being sure that parts are clean and dry, wiping down cold walls and the inside of the outer door, and routine cleaning of heat exchangers are simple and effective preventive measures.


Fig. 1


Fig. 2

Fig. 1. Example of part discoloration during cooling (13-8 Mo PH stainless steel).
Fig. 2. Example of part contamination due to improper bead blast prior to plasma nitriding (15-5 PH stainless steel after failed humidity and salt-spray test)

**Table 1. Sources of dirt and debris[1]**

| Element | Possible sources |
|---|---|
| Aluminum | Blasting medium, stop-off paints, ceramics |
| Carbon (soot) | Decomposition of hot-zone insulation, residual oils, paper, hydrocarbon decomposition (vacuum carburizing) |
| Chlorine/Fluorine | Water, cleaning agents |
| Copper | Stop-off paints, brazing filler metals |
| Chromium | Vaporization (especially stainless and tool steels) |
| Lithium | Stearates (powder metal), brazing filler metals |
| Manganese | Dealloying (steel) |
| Potassium | Cleaning agents, pickling solutions |
| Silicon | Ceramics, dealloying (steel) |
| Zinc | Dezincification (brass, bronze, nickel silver), stearates (powder metal), brazing filler metals |

Sources of water vapor include moisture in the air, condensate ("sweating") of water jackets and leaks in water-cooled components (e.g., element feed-throughs, heat exchangers, vacuum vessels). For example, a good rule of thumb is that raising the air temperature 10˚C (18˚F) doubles its moisture capacity. This means that air at 30˚C (86˚F) can hold eight times more water than air at 0˚C (32˚F). Closing the outer door between loads, maintaining the chamber water temperature higher than that of the ambient air, and using multiple pump-down and backfill cycles will help mitigate this issue.

Water is generally considered the most problematic of the sources of contamination since water vapor will condense on initial pump down (due to the cooling effect associated with a sudden drop in pressure), creating a "fog" cloud and subsequently ice crystals. During initial heating, the ice will vaporize, and water vapor will be present in the area of the parts.

The dew point of any gas introduced into the chamber during a furnace run must be on the level of -54˚C (-65˚F) or lower to ensure a low parts-per-million (ppm) of potential contaminant in the atmosphere. This is about 24 ppm of water vapor. In reality, gas should consistently run at or below -73˚C (-100˚F), which is about 1.5 ppm.

## When to Perform Maintenance

The frequency of maintenance (i.e. interval between routine repairs) is highly dependent on such factors as the type and number of heat-treating processes performed; the skill of the operators; the equipment type and design; the quality of prior maintenance and type of spare parts used; and the quality of the water system and gas system from the supply to the equipment. Additionally, running clean parts and properly supporting parts in baskets or on grids often help reduce the frequency of maintenance.

When performing maintenance it is important to understand why a particular task is necessary. Furthermore, the work should be signed off upon completion (which includes testing to ensure that the repair was successful).

## How to Perform Maintenance

Each type of furnace is unique, but the following generic conditions should always be met before any repairs are undertaken:

1. Power should be switched off for any repairs not directly involved with the electrical systems, controls or instrumentation. Lockout/tagout procedures should be in place.
2. The furnace should be cool, less than 50˚C (120˚F).
3. The furnace door(s) should be in the open position and secured so that they cannot be closed.
4. Disconnect all utilities, including gases, water and air. Lockout/tagout procedures should be followed.
5. Check that the furnace environment is safe and

that adequate ventilation is in place and functioning properly.

6. Wear protective clothing, including safety glasses and safety shoes.
7. Be sure that all confined-entry procedures are thoroughly understood and followed without exception.
8. Use the buddy system.
9. Before entering the vacuum furnace, confirm that the oxygen level is safe for human exposure.

## Preventive-Maintenance Checks

Setting up a planned preventive-maintenance program will minimize equipment downtime, ensure that proper spares are on hand for repairs and simplify the overall maintenance effort. As a minimum, the following checks should be performed at the specified interval.

### Each Run

1. Inspect the front door O-ring for cleanliness and damage. Clean and re-grease as necessary.
2. Inspect hot-zone insulation and heating elements for signs of damage, deterioration and that connections are snug and secure.
3. Inspect the load thermocouple(s) for damage if appropriate.

### Daily

1. Inspect the exterior and interior of the vacuum furnace for indications of damage, discoloration, dripping fluids and the presence of foreign material (e.g., dirt, grease, oil).
2. Check the water flow and temperature from each drain line.
3. Check the oil level on all pumps.
4. Ballast the vacuum pump (15-20 minutes minimum) before processing the first workload of the day.
5. During operation, inspect for hot spots, leaking fluids, excessive noise and/or vibration.

### Weekly

1. Perform a leak (rate-of-rise) test on the main vacuum vessel and pumping system. The furnace should be clean, dry, empty and outgassed before testing.
2. Check mechanical pump oil for contamination (e.g., dirt, particulates, water).
3. Check instruments for functionality.
4. Inspect the pumping system (pumps, valves, piping).
5. Visually inspect control and over-temperature thermocouples for damage.

**Monthly**

1. Check for hot-zone deterioration (insulation and heating elements), including doors.
2. Check calibration of vacuum instruments.
3. Check all thermocouples (e.g., control, over-temperature, load).
4. Change vacuum pump oil.
5. Check belts for proper tension (e.g., mechanical pump).
6. Change all filter elements.

**Semiannually**

1. Replace or recalibrate all thermocouples.
2. Flush all cooling lines and clean all in-line filters, strainers, etc.
3. Clean and replace diffusion-pump oil if applicable.
4. Inspect all vacuum gauges.
5. Replace door gasket or O-ring seals.
6. Remove, clean and reinstall thermocouple vacuum gauges.
7. Test the pressure-relief valve per manufacturer's instructions.
8. Make all necessary repairs to hot-zone components (including power feed-throughs).
9. Clean all mating flanges.

**Annually**

1. Drain and inspect the cooling water system (including temperature sensors).
2. Check all electrical connections. (Caution: Only a trained, licensed electrician should perform this activity).
3. Service all motors.
4. Clean the furnace heat exchanger if applicable.
5. Drain and filter quench oil if applicable.
6. Check the convection fan and/or oil agitators for proper operation if applicable.
7. Check, remove and inspect vacuum valves for proper operation, sealing and wear if applicable.

## Final Thoughts

Vacuum maintenance should also be performed in such a manner as to return the equipment to full operational service. Never compromise – a job worth doing is worth doing right. This will ensure years of productive service from your vacuum furnace!

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# Is it Safe?

The Doctor made a house call the other day, only to find his patient in a highly dangerous condition. Special care is needed whenever furnaces with combustible atmospheres are running, especially when operating below 760˚C (1400˚F). Let's learn more.

## A Word About Safety

Prevention of industrial accidents involving unwanted fires and gas explosions not only requires knowledge of the flammability characteristics (e.g., limits of flammability, ignition requirements, burning rates) of the combustible gases and vapors likely to be encountered under various conditions of use (and misuse) in the heat-treat shop, but also an understanding of the internal and external conditions that may be present when operating a particular piece of equipment or that exist in the environment surrounding that equipment.

## A Word About Terminology

Confusion exists at times between the words flammable (easily set on fire or that will burn readily) and combustible (capable of catching fire and burning).

In simplest terms, a flammable gas (or liquid) is one that is easily ignited and can burn in the presence of air when brought into contact with heat or flame. The flammable (explosive) range is the range of concentration of a gas or vapor that will burn or explode if an ignition source is introduced. Below the explosive or flammable range – the so-called lower explosive limit (LEL) or lower flammability limit (LFL) – the mixture is too lean to burn, and above the upper explosive limit (UEL) or upper flammable limit (UFL) the mixture is too rich to burn.

NFPA 86 (2011 edition) para. 3.3.65.5 defines an inert special atmosphere (purge gas) as "a special atmosphere of nonflammable gases that contains less than 1% oxygen." In this writer's view, any furnace containing flammable gases (e.g., carrier gases, enrichment gases, generated or synthetic atmospheres, gas mixtures containing percentages of flammable gases and the like) must be properly monitored, controlled and protected from dangerous internal and external influences.

When the chamber temperature (Fig. 1) is at or above 760˚C (1400˚F), as pointed out by NFPA 86, uncontrolled air infiltration could create process

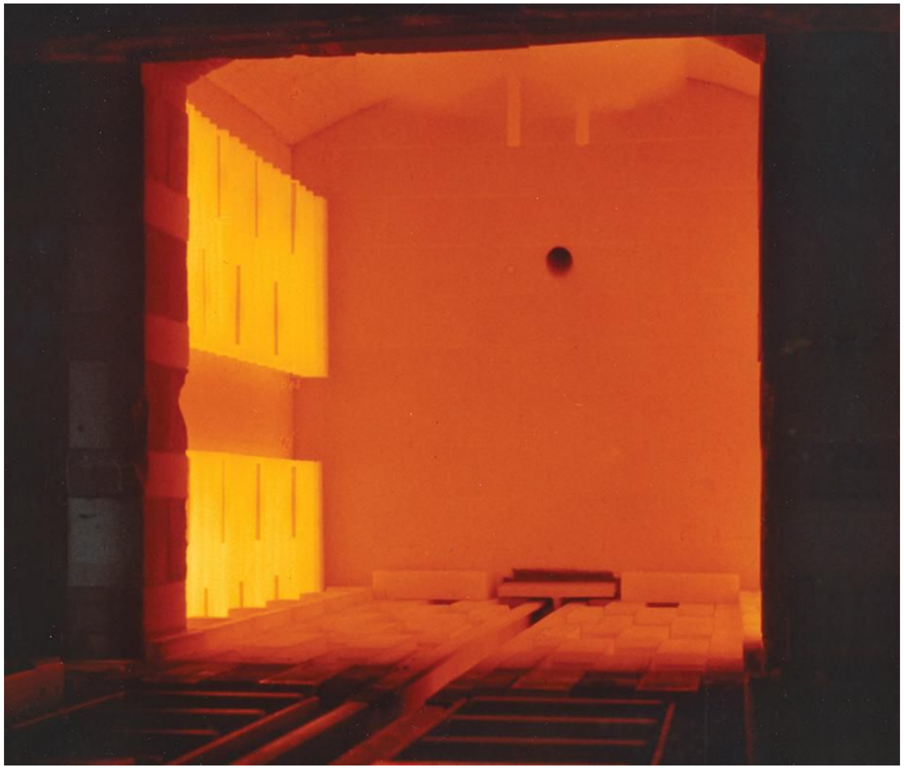

Fig. 1. The Doctor's first true love, an integral-quench furnace

quality issues. However, it is not anticipated this will create safety issues. That being said, when either the furnace temperature or any area within the furnace drops below 760˚C (1400˚F), any introduction of air can create an explosive mixture, and special safeguards must be in place.

Furthermore, any practice that causes a furnace chamber (heated or unheated) to lose positive pressure while running a combustible atmosphere is a major cause for concern and a possible call for immediate action. Examples include power failures, air leaks, loss of the heating system, carrier-gas settings lower than normal, unexpected door openings and loss of furnace temperature. These types of conditions can lead to the uncontrolled infiltration of air into furnace chambers, which could rapidly lead to an unsafe condition – faster in many instances than operators might be able to respond.

Transfer of loads in integral-quench furnaces to the quench-tank area and normal burn-in and burnout of furnace atmosphere are conditions that routinely create loss of positive-pressure conditions. Following manufacturer's instructions and company practices will ensure these situations remain under control despite loss of positive pressure. Other conditions, such as furnace pressure loss during transfers between chambers, should be monitored (Fig. 2), and the burn-off flame recovery observed (and timed) to understand how quickly the furnace positive pressure has been re-established under these conditions.

Processes are often run, either intentionally or unintentionally, below 760˚C (1400˚F). Process examples include loss of temperature inside a furnace chamber when a large cold load is introduced, nitrocarburizing at 580˚C (1075˚F) or stress-relief annealing at 730˚C (1350˚F) that are run below the industry-recognized auto-ignition temperature. In these cases, good practices and certain precautions are necessary. For example, outer doors should never be opened under low-flow conditions or without properly functioning pilots and flame curtains. Another especially critical time in the cycle is during load transfer or introduction of a load into the furnace while another is quenching.

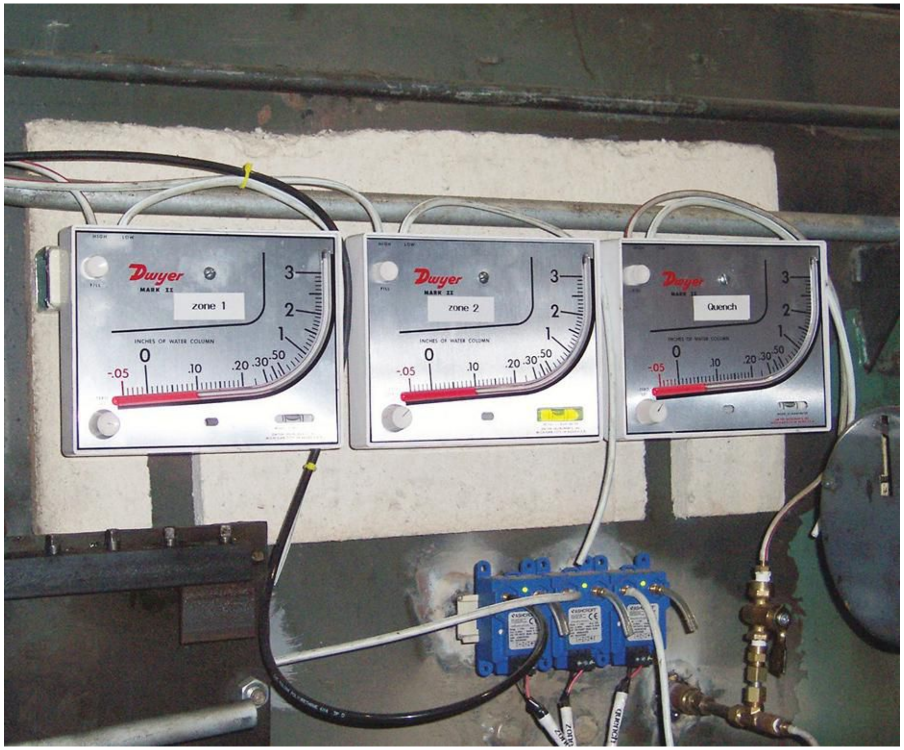

Fig. 2. Internal furnace pressure monitors

## The Importance of a Good Flame Curtain

A robust flame curtain that covers the full width and height of the furnace is mandatory (c.f. "The Flame Curtain – Function, Adjustment and More," Industrial Heating, March 2008). This massive wall of flame often scares the uninformed. However, it acts as a physical barrier designed to minimize air infiltration and disrupt the furnace atmosphere inside. Furnaces where strong wind currents (e.g., near open trucking bay doors, under downdrafts from ductwork or air conditioning vents, or near poorly positioned floor fans) can extinguish front-door pilots or force flame curtains to lie down horizontally, create significant risk of large volumes of unwanted air entering the furnace vestibules.

## Unusual Furnace Sights and Sounds

Any unusual sights – such as the furnace atmosphere pulsating or surging in a sporadic fashion from the burn-off port – or sounds emanating from inside the furnace – such as "wheezing," "whistling" or "popping" – are calls for immediate investigation and action. Trapped air pockets, leaky packing glands on cylinders, damaged door seals, holes in radiant tubes, leaky or missing access plates and even partially clogged nitrogen/methanol spargers are common sources.

One of the scariest situations the writer personally experienced was the integral-quench furnace pictured in Figure 1 howling like the proverbial banshee – so loud that the sound of air infiltrating into the furnace and partial combusting in the vestibule could be heard a block away. At one point the building was abandoned, and we were standing across the street literally seconds from disaster before going back in to take corrective action. This unsafe condition was created by substituting rich exothermic gas (~8% hydrogen) for endothermic gas (~40% hydrogen) and not recognizing the dangerous condition we created. We failed to anticipate what would happen when we discharged the load to the top cool of the furnace and the dilution of the atmosphere by air infiltration when the top-cool fan automatically came on. The situation was corrected, and the operation returned to normal by turning the top-cool fan off and increasing the flow rate. The lesson learned was that not all combustible atmospheres are created equal, and one must take extraordinary care to understand the differences between combustible gases when operating a furnace.

In another highly dangerous situation, we arrived at work in the morning only to find our integral-quench furnace (left unattended at temperature overnight) flowing endothermic gas at room temperature! Without a nitrogen safety purge, we were forced to remove the atmosphere by first extinguishing all sources of ignition, then opening doors sequentially and allowing chambers to purge by dilution with room air – a dangerous but necessary method.

## Moral of the Story

These stories, though scary, had positive outcomes and no one was hurt. As operators, supervisors and managers, we have an obligation to recognize and understand what type of dangerous conditions can (and possibly do) exist, take proper precautions, be well trained and have procedures in place that everyone in the organization is aware of, for every possible safety scenario.

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# “Run Baby, Run” – A Safety Story

Once upon a time (a long, long time ago), The Doctor was called in to investigate an industrial accident that resulted in serious burn injuries to personnel in the heat-treat shop. As part of that investigation, the subject of what to do in the case of a quench-oil fire came up and became legend, when in a late night meeting the infamous phrase, “run baby, run” was uttered. Let’s learn more.

Our story begins with an innocent-looking batch integral-quench furnace, one that sent six people to the hospital suffering from severe burns. This particular furnace was manually operated, and its front (outer) door burn-off exited from a circular hole near the bottom of the door, about 50 mm (2 inches) in diameter. The burning endothermic gas exited horizontally. It was reported that flames suddenly erupted from the opening during production runs, and anyone caught standing in front of the furnace – as far as 4.5 meters (15 feet) away – was in danger of being burned.

Two supervisors, several heat treaters and a fellow from the nearby plating department were unlucky enough to be within range of the flames and were seriously burned. What caused the furnace to erupt the way it did, and what could be done to fix the problem, was the subject of intense debate. Enter The Doctor.

First, this puts renewed emphasis on the fact that no one should stand in front of a furnace door for longer than is absolutely necessary. You would be amazed by how many conversations are held by people standing in close proximity to a furnace door. Don’t do it!

Secondly, a temporary solution to the problem was to change the direction of the burn-off by adding a 90-degree elbow into the outer door, thus changing the flame direction from horizontal to vertically upward. A small hole in the bottom of the elbow allowed the pilot flame to ignite the endothermic gas. This is one of the reasons that many furnace atmosphere burn-off cans are moved up and away from personnel (Fig. 1).

The investigation then began in earnest. A careful check of the furnace and transfer sequence found nothing out of the ordinary. In fact, the flame eruption was occurring mid-cycle and not during load transfer. One rather strange clue was found on the factory floor, an “X” marked in chalk that coincided with the distance the flames traveled horizontally outward from the outer door when the inner door was opened (such

as when a load was being transferred to or from the quench vestibule). A second clue was a small remnant

Fig. 1. Preferred location of burn-off can

of twine wrapped around the manual-activation handle for the inner-door cylinder. Could this be a case of deliberate intent to harm?

It turned out that a disgruntled furnace operator had indeed discovered how to make the furnace belch flames and how far the flame would extend out in front of the furnace. Positioned out of sight behind the furnace when someone he didn’t like “came within

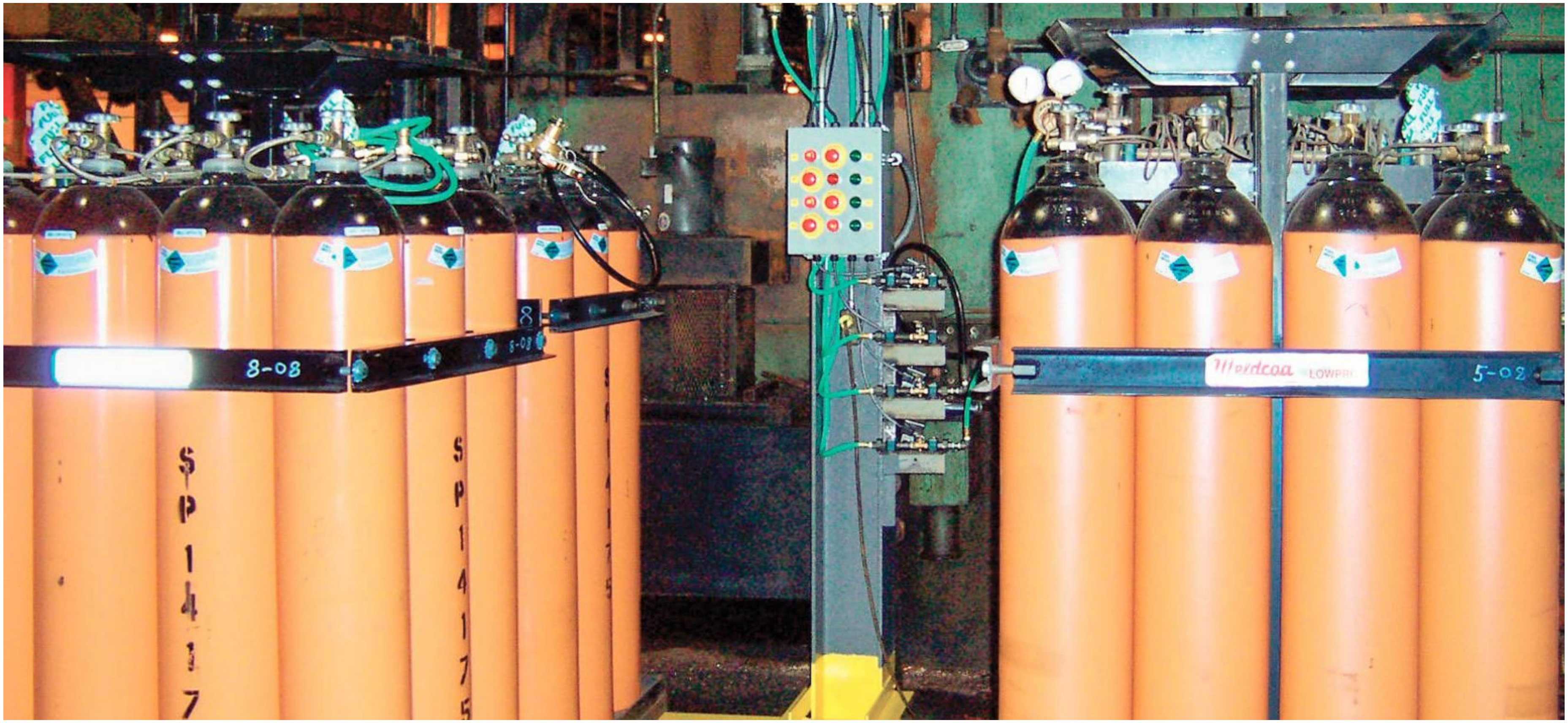

Figure 2. Nitrogen-purge setup with redundant cylinder banks

range," he tugged on the string attached to the inner-door open activation handle, causing the inner door to open with an eruption of flames out the front door. This is only one of two instances of criminal acts The Doctor has ever witnessed in the heat-treat shop. Amazing but true.

As part of the investigation, managers from several operating divisions had gathered to learn from the event. One of the other subjects of intense interest was what to do in the event of a quench-oil fire. What started as a lively discussion over lunch extended far into the evening talking about the do's and don'ts related to quench-oil fires. Since one of the most dangerous conditions is a load jammed half in and half out of the oil, this was the principal focus of everyone's attention. Oil discharge from the overflow pipe into the pit was also mentioned, however. At this time The Doctor related a true story about a similar situation, where up to 100 mm (4 inches) of oil had accumulated in the pit and was ignited by a careless employee, destroying seven integral-quench furnaces and a large portion of a heat-treat department.

A brief recap of the discussions follows.

1. The subject of equipping furnaces with a nitrogen purge (Fig. 2) was raised almost immediately. Everyone agreed it was a good idea, but the reality was that most of the company's integral-quench furnaces were not equipped with this feature. A management action item was undertaken. One interesting part of the discussion centered on a sintering furnace in which the empty nitrogen-purge cylinders had been replaced by oxygen cylinders (despite the fact that they had left-handed threads). One of the engineers stated that it is still the single loudest explosion he has ever heard … and he was in an adjacent building.
2. Also discussed was educating supervisors not to give instructions to "shut everything off" (which included the nitrogen-purge cylinders) in the hopes that the fire will burn itself out. An entire plant was lost because of this mistake.
3. The question was raised if the front door should ever be opened "just to take a peek" while flames are erupting and the hot load is still volatilizing the quench oil. Surprisingly, this sparked a heated debate as to whether this technique should or shouldn't be used. Several managers stated that they gather a crew of trained individuals in proper personal protective equipment; open the door; and, using long rods, attempt to clear the jam in an effort to move the load either down or up out of the oil. Others, including The Doctor, view this as putting personnel in unnecessary danger.
4. Who should be your first call? Is it to your supervisor, his boss or 911? While most fire departments do not train for heat-treat-related emergencies, they are trained professionals that deal with fire events all the time. Since, for example, a grease fire in a kitchen can get out of control in as little a 30 seconds, remember to act quickly and responsibly. Confirm by asking that someone has indeed called the fire department.
5. Should the furnace ever be placed in manual mode and the operator attempt to reposition the load? The consensus was that this is a prudent act to perform provided one can do it safely and it does not involve attempting to open the front door.
6. Where is the control panel in relationship to the

front door? In many instances it is next to it, perhaps convenient during normal operation but unsafe to stand in front of in emergency situations. While relocation of the control panel may not be an easy task, everyone was in agreement it should be on the side of the furnace, away from the door area. Remember that the pressure inside the furnace builds up, and one of the ways it will relieve itself is to push the front door outward away from its mating flange. The Doctor has seen 1-meter (3-foot) flames driven out horizontally under high pressure at the front door during such situations, so beware!

7. How often should grids and baskets be inspected, and do you attempt to weld repair a grid in order to extend its life? Grids and baskets should be inspected daily for cracks, warpage or other signs of damage. If your application is carburizing, weld repairs will fail quickly. Grids that tear apart are a major cause of load jams.
8. What other scenarios can cause fires? There are obvious dangers that most of us are taught to watch out for, such as water in quench oil that can cause a quench tank to erupt into flames. Another subtle but important consideration is the makeup of our workloads. We must pay attention to surface area and remember that whether we are running tiny fasteners in an integral-quench furnace or moving large, heavy parts by crane for quenching into an open tank, the transfer must be smooth, performed in a timely manner and the load must be fully submerged. Huge evolutions of flame may be dramatic, but they are deadly!

## Conclusion

While we all had a good laugh over the fellow who uttered "run baby, run" when faced with immediate danger, his comment hits the mark in many ways. When faced with an emergency, it is only human nature to protect one's life first. This is where knowledge, understanding and training of operators, supervisors and managers helps everyone make informed decisions.

When is the last time your shop had an emergency drill to practice various dangerous scenarios that can arise? Never assume that the person next to you has clearly heard or understands your commands. Asking them to repeat your instructions is always a good idea. The Doctor still remembers shouting "don't spark the pilot" then holding on to a ladder about 20 feet in the air (in a horizontal position to the floor) because the salesperson, trying to be helpful, thought he heard "spark the pilot."

The bottom line is accidents happen, but we don't want people injured because of them. Train, train and train some more.

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# Vacuum Equipment Innovations

In simplest terms, innovation is the introduction of a new method, idea or product into the marketplace that represents a revolutionary (as opposed to evolutionary) change or breakthrough, often leading to a technological advance within its targeted industry. The Doctor loves innovation, and while this article is not intended to be an endorsement of, nor a commercial for, a particular company or product, a few vacuum-furnace designs have recently caught the Doctor's eye and are deserving of our attention. Let's learn more.

As we all know, the criticality of today's applications demands absolute control, process repeatability and the highest possible quality. The following equipment innovations – in no particular order – help heat treaters do just that.

## Innovation 1: Large/Heavy, Complex Parts Processing Solutions

Car-bottom-type vacuum furnaces (Fig. 1) were developed to handle large, heavy product workloads as well as long, horizontal components. These furnaces initially started at 3.6-m (12-foot) hot-zone length, progressed to 7.2-m (24-foot) length and eventually were built to handle 11-m (36 foot) lengths. The major advantage of this type of furnace is to process loads up to 45 tons (100,000 pounds) while incorporating heating elements under the workload as well as heating elements around the circle of the hot zone.


Fig. 1. Load of titanium for vacuum annealing (courtesy of Solar Atmospheres, Inc.)

Recent demands from the aerospace and nuclear industries have extended designs to 14.6 m (48 feet) by 2.1 m (7 feet) in diameter; units capable of handling

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loads up to 68 tons (150,000 pounds) at 1315˚C (2400˚F). Dual-loading cars are provided for loading/unloading from both ends of this furnace to minimize lost production time (proven to increase capacity by up to 30%) when loading/unloading large loads.

Car-bottom furnaces offer a unique ability to thermally process sensitive materials such as titanium, zirconium, precipitation-hardening stainless steels and nickel alloys while meeting the demands for surface cleanliness. This is achieved in part by:

- A highly efficient vacuum system that includes triple mechanical pumps, vacuum boosters, and (Varian 35-inch) diffusion pumps
- A stainless steel inner cold wall, which inherently remains cleaner than a carbon-steel cold wall
- A load car (the portion of the hot zone exposed to air on loading) consisting of less graphite-felt insulation, resulting in less moisture pickup when outside the furnace

## Innovation 2: Near-Net-Shape Processing Solutions

Powder metallurgy represents one of a number of niche markets whose identifying characteristics include rapid growth and product diversity. Within this industry, metal injection molding is one of the near-net-shape technologies used to produce high-performance, precision components such as medical and orthodontic devices, mobile-phone parts, firearms and door-lock internals to name a few. Materials include stainless steels (e.g., 316L, 17-4); low-alloy steels; tool steels and specialty alloys for implantable and magnetic applications; hard metals (e.g., WC-50, WC-10Co); and refractory alloys.

Uniquely designed single-chamber batch vacuum furnaces (Fig. 2) are one solution (continuous vacuum being the other) that allows the processing of the wide range of materials required from the many different types of feedstock available. These designs offer cycle (recipe) flexibility, choices of process atmospheres (e.g., hydrogen, argon) and the ability to do secondary debinding and sintering in one self-contained unit.

## Innovation 3: Lean Manufacturing via Small-Batch Processing Solutions

Integration of heat treatment directly into the mainstream of the manufacturing process has been the goal of heat treaters for a very long time. This enables synchronization with soft-machining operations to produce a small batch of production parts in-line and on-demand to meet the needs of the other manufacturing centers. The goal is to reduce unit cost to produce and improve efficiency.

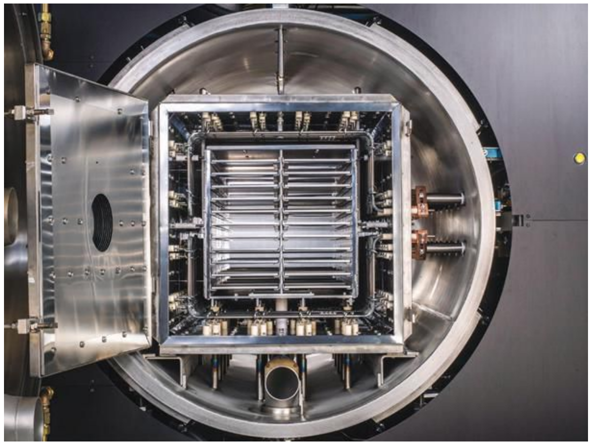

Fig. 2. MIM sintering furnace hot zone (courtesy of Elnik Systems LLC)

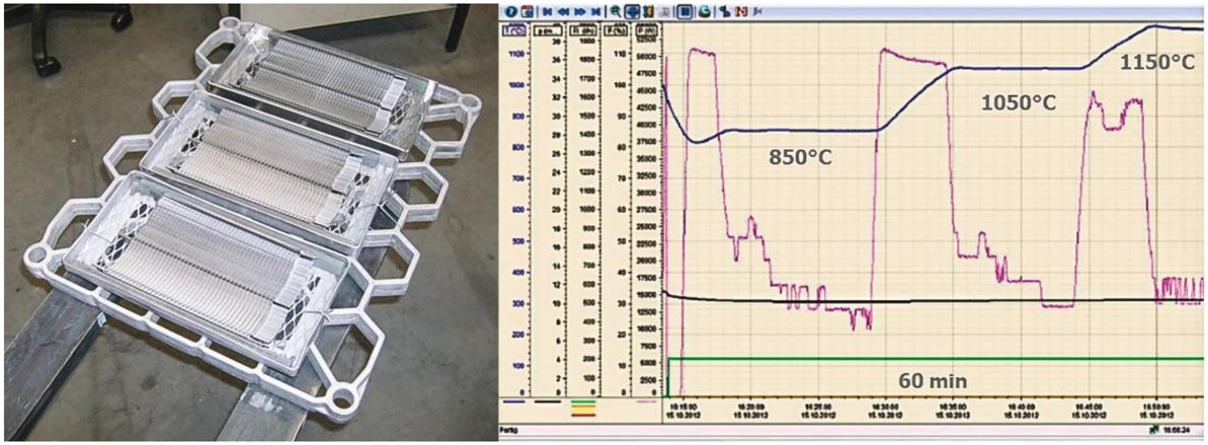

Fig. 3. Hardening of high-speed-steel saw blades (courtesy of ALD-Holcroft)

Important benefits include short cycle times (achieved by optimizing hardening or case-hardening processes for small-batch loads), part surface control, minimizing distortion while maintaining precise temperature uniformity, and cycle reproducibility so as to achieve consistent quality in a 24/7 manufacturing environment.

Parts are loaded onto a single grid, which is transported through the system (Fig. 3) for hardening or case hardening (carburizing or carbonitriding). In the example shown, high-speed-steel saw blades are hardened at 1150-1200˚C (2100-2200˚F) soaking at temperature for 5-7 minutes to produce the desired microstructure on both the high-speed-steel teeth and backing materials. In other applications, such as carburizing of gears, austenitizing temperatures of 1040˚C (1900˚F) are utilized.

## Innovation 4: Single-Piece Part Processing Solutions

Manufacturers continue to strive for true single-piece part flow, and the Unicase Master® (Fig. 4) is designed for hardening or case hardening high volumes of automotive gears and bearing races. The design transports single parts through the entire process to allow individualized part heating and 4D Quenching® where the quench gas follows the contour of a single component part in the quench. The way in which this is done represents a possible breakthrough in quenching deformation control.

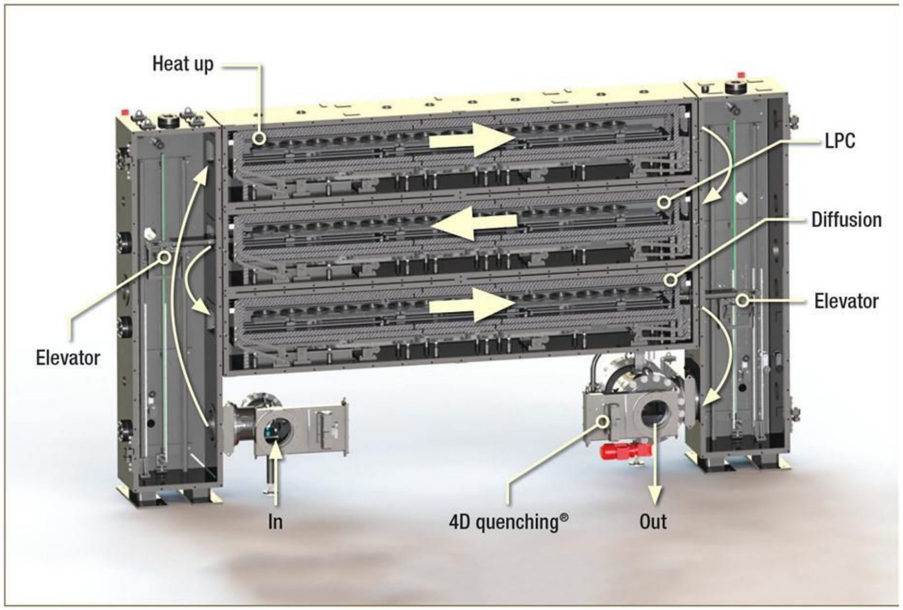

Fig. 4. Load transport in an LPC and HPGQ furnace (courtesy of SECO/WARWICK Corporation)

This green, lean and agile design meets the productivity demands of high-volume gear and bearing manufacturers while optimizing process flexibility and operational speed by applying cutting-edge low-pressure carburizing (LPC) and high-pressure gas-quenching (HPGQ) technologies.

## Final Thoughts

Progress is the movement toward achieving a specific goal, which in the case of the heat-treatment industry is offering the most price-competitive technology solutions to industry. Progress can only be achieved by innovation and the exchange of ideas by those in the heat-treating community. If your product or service was not mentioned here, set the goal of being here next time. The Doctor will be watching.

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Columbia CPM+

# Advancing Technology Through Innovation

It was a grand party – full of music, dancing and laughter. Even fireworks! The European operation of a major furnace company turned 25, and The Doctor was there amidst the festivities. Let's learn more.

## Technical Conference Highlights

In addition to joining in the fun, The Doctor was present, along with some 150 engineers from all over Europe, to give a talk on "Selecting the Best Quenching Method for Distortion Sensitive Gears" at SECO/WARWICK's 19th Heat Treatment Technical Seminar on New Trends in Heat Treatment held at the Mierzecin Palace Resort in Mierzecin, Poland.

The venue was spectacular, and the conference was even more so (leave it to a heat treater to find true beauty in the technical side of things). Of the 14 talks presented, several were especially noteworthy, including:

- "Heat Treatment of Steels According to the Principle of Sufficient Hardenability" (J. Pacyna, AGH University of Science and Technology, Krakow)
- "New Technology of Gas Nitriding – Zero Flow®. The Minimization of Process Media Consumption and the Reduction of Emissions of Process Gases" (T. Przygonski, SECO/WARWICK)
- "PreNit® - The Economic Option for Vacuum Carburizing" (Professor P. Kula, Lodz University of Technology)
- "Improved Efficiency and Productivity – The Achievements of Vacuum Carburizing Applications Based on Vector Vacuum Furnaces – CaseMaster® and UniCase Master®" (Dr. M. Korecki, SECO/WARWICK)
- "Gear Distortion in the Processes of Bainite Nanostructurization" (S. Marciniak, Warsaw University of Technology)

While space prevents discussing all of these excellent talks, several will be highlighted here.

### The Future of Gas Nitriding

ZeroFlow® technology uses only ammonia for nitriding and is designed to minimize ammonia consumption by optimizing the impact of process temperature, chemical composition of the atmosphere in the furnace retort (i.e., nitriding potential), nitrogen concentration in the surface layer and phase/zone microstructure at the near surface.

Fig. 1. SECO/WARWICK Europe CEO Bartosz Klinowski oversees the 25th anniversary festivities.

A rise in the process temperature increases ammonia consumption in a nonlinear relationship to the nitride layer formation. Higher nitriding potential of the atmosphere also increases gas consumption. Furthermore, the thicker the compound layer, the larger (and more dramatic) the increase in gas consumption (i.e., the concentration of nitrogen on the surface of the nitrided layer causes an increase in the consumption of ammonia). ZeroFlow technology reduces the consumption of ammonia by controlling precisely the growth kinetics of the nitrided layer. Case studies using ZeroFlow technology showed how to achieve the lowest overall consumption.

## Advances in High-Temperature Processing

The control of grain growth is especially important in vacuum carburizing and is an active research area to shorten overall cycle time. Material with microalloying elements (e.g., Al, Nb, Ti) that pin the grain boundaries is one such approach.

Another technique involves the addition of nitrogen into the surface of the steel using a prenitriding step (PreNit®) added to the cycle as the work heats to carburizing temperature. The process involves the addition of ammonia to the vacuum furnace chamber during heating of the workload in the temperature interval from around 400-700˚C (750-1290˚F).

Nitrogen diffuses into the surface, forming precipitates that act to pin the grain boundaries and limit austenite grain growth before carbon diffusion takes over this function. The nitrogen then diffuses deeper into the case, which avoids higher retained-austenite levels. As a consequence, it has been reported that low-pressure carburizing temperature may be increased to as high as 1000°C (1850°F) without affecting the microstructure or mechanical properties (e.g., fatigue strength).

When compared to either atmosphere carburizing or low-pressure carburizing without prenitriding, this process controls grain size while case depth and hardness remain essentially the same – even when higher carburizing temperatures (for faster cycle times) are used. Economic comparison with atmosphere (endothermic gas) carburizing is favorable for case depths over 0.4 mm (0.016 inch) with reported savings up to around 60%.

## The Future of Vacuum Hardening and Vacuum Carburizing

Of all the recent technological advances in the heat-treatment industry, the UniCase Master® is very intriguing. I am pleased to report that the concept exists in the real world – and in this writer's opinion will allow our industry to achieve a competitive advantage over competing technologies by offering true one-piece flow, high productivity (one part every 30-60 seconds and up to 1 million gears or bearings a year), uniform and rapid part heating of one piece at a time and individual (contour) quenching in a compact footprint (15-20 seconds). The concept has been introduced previously (c.f., "Vacuum Equipment Innovations," *Industrial Heating*, November 2015), but the big revelation was to both see the unit in productive operation in the R&D facility and to know that it has been working continuously over the last four months carburizing gears.

Fig. 2. UniCase Master® in operation

## A Customer's View

When our customers speak, I pay attention. Representatives from the aerospace industry talked about the advantages they have derived from using vacuum carburizing for aerospace engine components. Aerospace customers have demanding requirements, examples of which would be as follows:

- Effective case depth: 0.5-1.1 mm (0.020-0.045 inches)
- Surface hardness: 81-85 HRA

- Core hardness: 35-48 HRC
- Microstructure: Finely dispersed carbides in a matrix of tempered martensite
- Retained austenite: 10% maximum
- Case profile: Uniform distribution of carbon throughout the case and in particular in the transition (i.e., case/core interface) layer (i.e., no carbides in the grain boundaries and no carbide necklaces)
- Residual subsurface compressive stress: 10 ksi maximum
- Prior austenitic grain size: ASTM 5
- IGO/IGA: ≤ 8 μm (0.003 inches)
- The implementation of vacuum carburizing in the aerospace industry has resulted in the following advantages:
- Surface activation (preoxidation, chemical treatment, blasting) not required
- Shorter carburizing time (up to 30% less) in relation to the same carburizing temperature with conventional (atmosphere) carburizing
- Savings in production costs (up to 15% less) by reducing the amount of carburizing gas
- Reduced energy consumption (up to 15% less) by elimination of atmosphere generators and adaptation of modern technology solutions
- No emissions (e.g., carbon dioxide); no open flames
- Less distortion (up to 15% less) by controlling the speed of heating and cooling
- Improved product quality (i.e., uniformity of properties, improved surface condition)
- Extremely uniform case depth and better homogeneity of the carburized layer on internal and external surfaces
- No internal oxidation
- The ability to direct quench after carburizing
- Elimination of copper plating layer prior to quenching
- Reduction of product lead time (up to 30% less)
- Reduce the post treatment time through a combination of high-pressure quenching and vacuum tempering.

## Summary

While not often called upon to do so, The Doctor enjoyed his role as a roving reporter. While this column is not intended to endorse the host company or their products, one could not help but be impressed by what was seen and heard.

Technological advances are not limited to any one company. It is exciting to know that innovation is alive and well.