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Pre-Nitriding: A Means of Significantly Increasing Carburizing Throughput

By Thomas L. Hart

Pre-nitriding is a technology that addresses grain growth and creates a finer grain structure in the carburized case at higher temperatures. Case studies show how carburizing productivity has doubled, and sometimes tripled, using pre-nitriding.

CARBURIZING IS A SURFACE-HARDENING METHOD that uses heated metal in a carbon-rich environment to increase carbon levels on the metal, via diffusion, for added hardness. Power transmission parts (gear boxes, gears, shafts, etc.) are a good example of components that benefit from carburizing.

Traditional/endothermic (“endo”) atmosphere carburizing, which takes place in a controlled atmosphere, has been a common technique used for decades.

Low-pressure carburizing (LPC), performed in a vacuum furnace, is a process advancement that enables a user to carburize steels at higher temperatures with precision and accuracy. Working in conjunction with Lodz Technical University, a patent for a unique method of LPC was created. With U.S. and European patents, LPC is a process that has been patented for both the control system and unique carburizing atmosphere mixture. Compared to endo carburizing, LPC produces a more precise, uniform case for difficult shapes and dense, packed charges. LPC eliminates oxidation at the grain boundaries, is environmentally friendly, and allows for a practically unlimited supply of carbon at the surface.

Additionally, developed in conjunction with Lodz Technical University, the LPC process itself can be further enhanced by the support of pre-nitriding surface technology. When using pre-nitriding, there is a significant reduction to the carburizing time, even up to two- to three-fold. The time reduction benefits are dependent on carburizing thickness (effective case depth — ECD), type of material, and structural requirements. LPC increases productivity and reduces overall manufacturing costs by shortening the heating cycle.

PRE-NITRIDING LPC TECHNOLOGY

The next step in LPC technology development is the addition of pre-nitriding surface technology. This technology uses ammonia dosing during the initial heat-up phase of a carburizing cycle (see Figure 1). This process forms nitrides at the required density and depth to allow for future high-temperature carburizing of the load, such that the resulting case will be of a sufficiently fine-grain structure while having minimal negative impact to the crystalline structure and mechanical properties traditionally experienced at high temperatures.

Pre-nitriding expands the use of LPC technology toward higher carburizing temperatures. Pre-nitriding technology is backed by material science and engineering research and is currently being used in industrial applications.

As illustrated in Figure 1, the technology is based on ammonia gas dosing in a vacuum furnace chamber during continuous heating of a charge in the temperature range of 750°F (400°C) to 1,300°F (700°C). With the introduction of ammonia, nitrogen is absorbed into the surface layer, which supports the carburizing process by creating nucleation sites for new austenitic grains.

As a result, the carburized layer, processed at higher temperatures, does not demonstrate grain growth as compared to traditional endo processing. Additionally, due to the higher process temperature, even up to 1,930°F (1,000°C), the overall process can be completed in a shorter amount of time by increasing the diffusion of carbon.

These advantages allow for the reduction of process time by increasing the carburizing temperature. The resulting carburized layer has proper microstructure and mechanical properties when compared to lower temperature traditional endo processing.

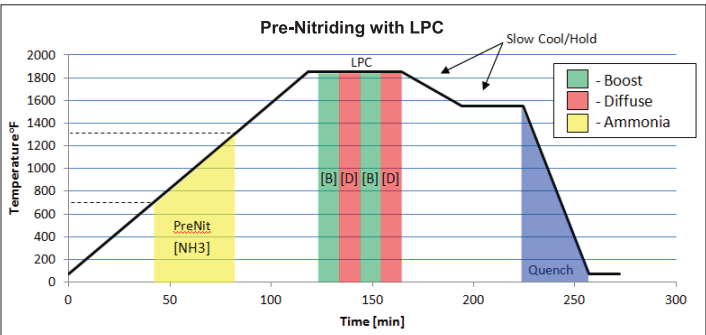


Figure 1: Pre-nitriding — process characteristic

Various Carburizing Methods - Process Time					
ECD = 0.024" ECD Carbon % = 0.4% Surface Carbon % = 0.8%					
Type of Carburizing	Endothermic	LPC	PreNitLPC		
Temperature	1690°F	1690°F	1740°F	1800°F	1830°F
Boost time	167 min	23 min	17 min	13 min	11 min
Diffusion time		92 min	64 min	58 min	43 min
Total Carburizing Time	167 min	135 min	81 min	61 min	54 min
Carburizing Time Reduction	0%	19%	51%	63%	68%

Figure 2: Comparison of endo, LPC and pre-nitriding times for different ECD and temperature

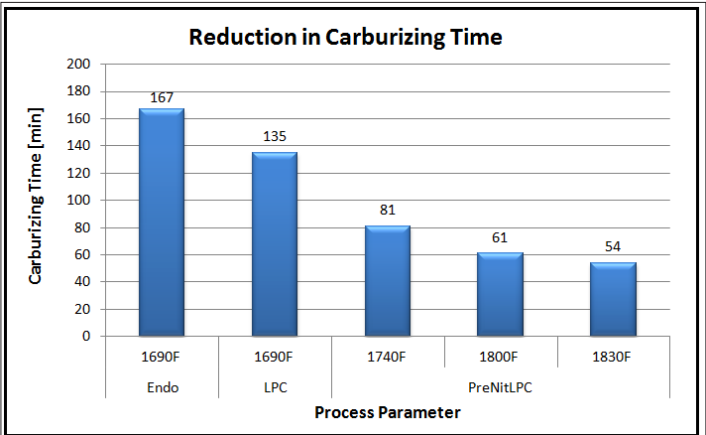


Figure 3: Pre-nitriding – time reduction chart

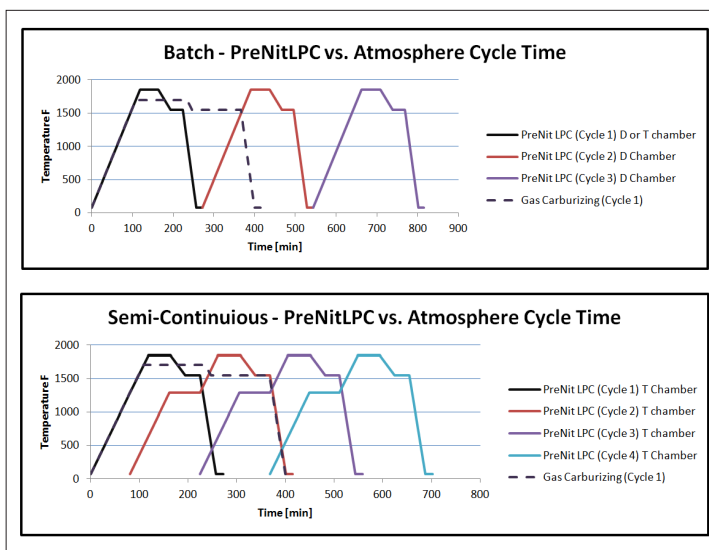


Figure 4: Pre-nitriding – time savings graphs comparing 1,700°F endo carburizing to 1,850°F pre-nitriding

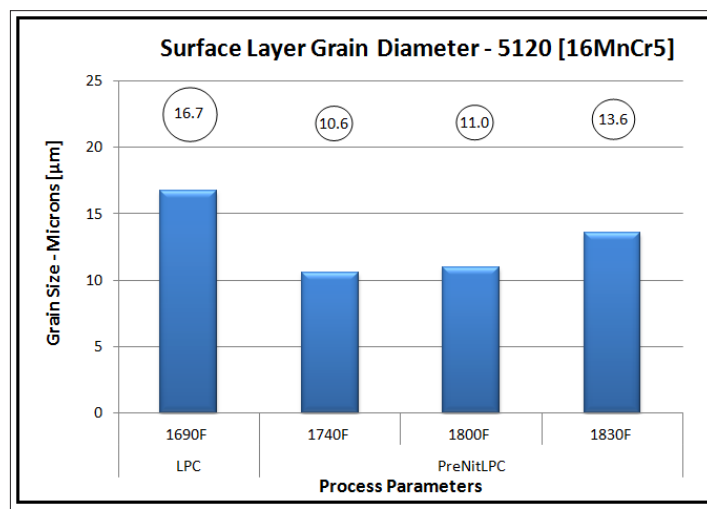


Figure 5: Pre-nitriding – grain size comparisons

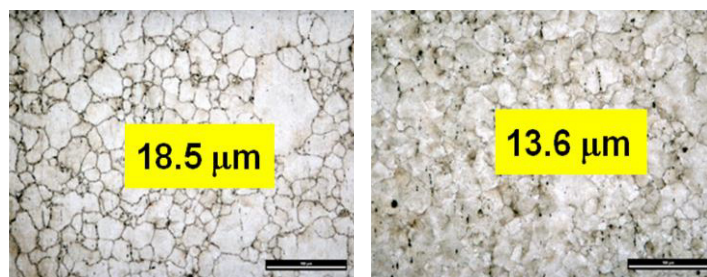
An additional benefit of LPC processing is the fact that it is a much more environmentally friendly process as compared to endo solutions, and LPC technology is becoming a more commonly used solution in a wide range of today's industry.

As LPC is successfully replacing traditional technologies, there is a great amount of interest to broaden LPC applications and to optimize LPC process techniques. The most promising approach to broadening and optimizing LPC processes is the addition of nitrogen together with carbon in the carburized surface layer. This combination leads to improved functional properties and economic benefits.

RESEARCH AND DEVELOPMENT

A series of LPC and pre-nitriding processes were completed at different temperatures in order to compare the structure and properties of carburized layers.

Two types of steel were carburized, 5120 (16MnCr5) and 4317 (17CrNi6-6) with an ECD of 0.024 inches (0.6 mm), where an acetylene-ethylene-hydrogen gas mixture was applied for the carburizing atmosphere and nitrogen, obtained from ammonia dissociation, for the pre-nitriding option. The carburizing and ammonia atmospheres were dosed according to procedures described in the LPC and pre-nitriding patents, respectively. Ammonia was introduced during the heat-up stage of the cycle in the temperature ranges of 750°F (400°C) to 1,300°F (700°C).



LPC @ 1830°F

Pre-Nitriding @ 1830°F

Fig 6: AISI 5120 (16MnCr5) LPC vs. pre-nitriding grain size

Additionally, an endo process was completed showing a comparison of LPC and pre-nitriding against the traditional carburizing method, as illustrated in Figure 2.

Figure 2 represents how temperature can strongly influence the time required for vacuum carburizing, mainly due to the exponential rate in which the carbon diffuses in steel at higher temperatures. Using endo carburizing at 1,700°F (930°C) as a base reference point, the carburizing process time can be reduced 63 percent by raising the process temperature to 1,800°F (980°C). Additionally, and depending on the ECD requirements, it is possible to further reduce the cycle 2 or 3 times at temperature 1,890°F (1,030°C). When processing 5120 (16MnCr5) steel, with a required ECD of 0.080 inches (2.0 mm) at 0.35 percent C, 550HV, and oil quench, the carburizing process will last 21 hours at a temperature of 1,700°F (930°C), while at 1,890°F (1,030°C), carburizing time requires less than 10 hours.

Figure 3 shows the shortest carburizing process time achieved with pre-nitriding at 1,830°F (1,000°C). The ECD was 0.024 inches (0.6 mm) only after 54 minutes of LPC treatment. Time advantages are 68 percent lower in comparison to conventional carburizing and 60 percent lower when compared to LPC, when done at 1,690°F (920°C). This is promising from an economic point of view, provided the mechanical properties of obtained layers at 1,830°F (1,000°C) were at least the same as those at 1,690°F (920°C).

Further research of microstructure and properties of those layers was done in order to verify this outcome.

RESULTS — SHORTENING OF CARBURIZING TIME

Carburizing at a higher temperature decreases the process time due to the much higher carbon diffusion coefficient experienced at the elevated temperatures. The process time for different conditions is demonstrated in Figures 2 and 3. Typical process solutions for LPC can be achieved in batch, semi-continuous, and most recently, single-piece flow furnaces. As illustrated in Figure 4, one can see the process flow advantages of pre-nitriding in batch and semi-continuous solutions over a traditional endo carburizing processing. With a semi-continuous solution, the PreNitLPC technology lends itself to a much higher rate of production due to a “straight-through” approach vs. an “in-and-out” batch approach.

GRAIN STRUCTURE

Using the pre-nitriding method, it is possible to carburize at temperatures of 1,830°F (1,000°C) and above, while maintaining an appropriate grain size in the case, just like in traditional low temperature processes.

Figure 5 charts and illustrates the relationship between endo and pre-nitriding, and even at higher temperatures, grain sizes in the carburized layer are smaller when using pre-nitriding vs. endo technologies.

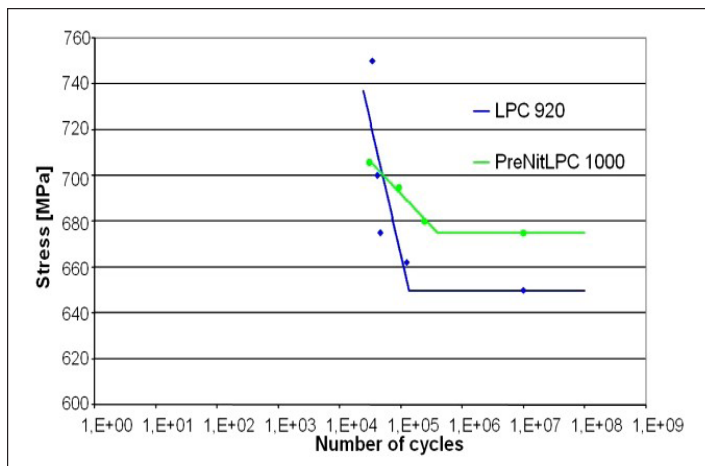


Figure 7: Wohler's curves showing cycle fatigue and fatigue limit strengths for LPC and pre-nitriding, 4317 (17CrNi6-6) steel

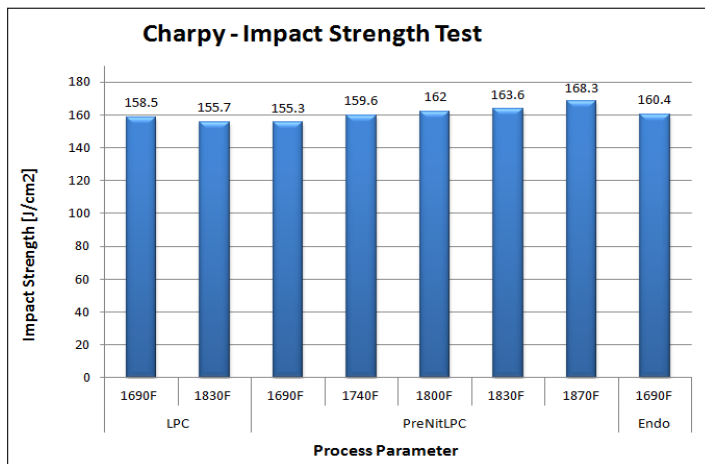


Figure 8: Impact strength of 5120 (16MnCr5) specimens, V-notch by Charpy's test



Figure 9: Pre-nitriding – single-chamber vacuum furnace system



Figure 10: Pre-nitriding – multi-chamber vacuum furnace system

Additionally, when using PreNit technology, the grain structures become more tightly refined than that of traditional LPC processes. AISI 5120 (16MnCr5) material was carburized, and as Figure 6 shows, the grain sizes are refined using pre-nitriding (13.6 μm) in comparison of traditional LPC (18.5 μm).

STRENGTH PROPERTIES

An important component of the analysis was to define the carburized layer mechanical properties in order to determine the potential applications for pre-nitriding in relation to other carburizing methods.

When using pre-nitriding at higher temperatures on 5120 (16MnCr5) steel, the hardness profile in the carburized layer showed comparable results to traditional LPC. Fatigue strength for bending was measured to assess the hardness profiles. Figure 7 represents the calculated Wöhler's curves, showing high/low cycle fatigue and fatigue limit for LPC 1,690°F (920°C) and pre-nitriding 1,830°F (1,000°C) processes. Test


data concluded the fatigue limit bending strength is higher when using pre-nitriding technology for 4317 (17CrNi6-6) steel.

IMPACT STRENGTH TESTS

Charpy impact strength tests were performed on V-notched test parts made from 5120 (16MnCr5) steel and were 0.39 inches \times 0.39 inches \times 2.16 inches (10 mm \times 10 mm \times 55 mm) in size and carburized under different conditions.

The Figure 8 Charpy test that used 150 J of energy shows a final result where all conditions had impact test measurements above 150 J/cm². The pre-nitriding processed parts show higher impact strength values from 155 J/cm² at 1,690°F (920°C) to 168 J/cm² at 1,870°F (1,020°C) — not a significant difference, but improvement, due to the use of pre-nitriding technology.

SUMMARY

- Higher carburizing temperatures, applied while using pre-nitriding technology at 1,830°F (1,000°C), resulted in a smaller grain size in the case as compared to traditional LPC at 1,690°F (920°C).
- The strength properties (hardness, fatigue, and impact) achieved via pre-nitriding technology are comparable and slightly higher than those properties achieved in standard processes.
- Pre-nitriding technology may be used at much higher temperatures than that of traditional LPC or endo processes without detriment to the mechanical properties of the case.
- Due to the higher carburizing temperature, it was possible to significantly reduce the carburizing process time required to achieve the desired ECD.
- Pre-nitriding at high temperature enables a reduction of carburizing time up to 2-plus times that of traditional LPC or endo carburizing processes, offering an alternative solution to the carburizing heat-treatment process that is technically equivalent and economically attractive.
- Pre-nitriding is an advanced, accelerated, and efficient low-pressure carburizing system designed for high temperature that can be equipped on single-chamber (Figure 9), double-chamber, multi-chamber (Figure 10), and single-piece flow vacuum furnace systems. 

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ABOUT THE AUTHOR: Thomas Hart is the vacuum product manager at SECO/VACUUM Technologies

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