Meeting standards for die heat treatment

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Problems with ensuring the quality of large tools have led to the creation of several processing standards the most widely applied of which is from NADCA. The basis of this standard is briefly described along with the vacuum furnace equipment capable of achieving it. Results of experimental validation show that this equipment can easily meet the current and future needs of the tooling industry.

Keywords: Large dies, Vacuum heat treatment, Standards

Introduction

Problems with ensuring the quality of large tools (moulds and dies) have led to the creation of several processing standards. The most widely known studies in this area were published by the American association NADCA (North American Die Casting Association)¹ and the leaders of automotive industry, among others companies such as Ford,² General Motors³ and Toyota. These standards relate mainly to steel X37CrMoV5-1 and X40CrMoV5-1 (DIN, EN) and modifications thereof. They refer to the quality inspection of initial material, guidelines for conducting and controlling of the heat treatment process and for measuring its results. No such complex approach to tool manufacturing has been observed in Europe; nevertheless such standards are also developed on an industrial level, especially by automotive concerns and steel manufacturers. It is not surprising that these standards are based on NADCA guidelines.

NADCA guidelines for heat treatment of hot work tool steels

The North American Die Casting Association became deeply involved in the issues of manufacturing hot work steel tools. As a result of that involvement, a guidebook was produced entitled 'Special quality die steel & heat treatment acceptance criteria for die casting'. The study focuses on the issues of initial material quality, vacuum heat treatment and welding methods.

Before heat treatment, the parameters and quality of the steel have to be confirmed through:

- (i) classifying steel grade with respect to the chemical composition of alloy additions and the contents of sulphur and phosphorus (grades from A to E)
- (ii) measurement of hardness after annealing (below 235 HB)
- (iii) analysis of the contents of microimpurities

- (iv) checking whether there are any internal defects such as cracks, presence of oxides, porosity, segregation, etc. (ultrasound examination)
- (v) defining grain size (above 7 according to ASTM E112)
- (vi) examination of microstructure (ferrite with evenly distributed spheroidal carbides).

According to the NADCA criteria, the heat treatment process should be performed in a vacuum furnace (Fig. 1) with a high pressure gas quench while monitoring and controlling the surface and core temperature of the processed piece (workload thermocouples have precisely preset locations).

Preheating to austenitising temperature is done gradually not allowing excessive temperature differences. The first hold occurs at approximately 590-680°C and lasts until temperature difference between core and surface is below 110°C (much less in practice). The next hold is preset at the temperature of 815–860°C and lasts until temperatures are uniform with a difference not bigger than 14°C. Finally, the austenitising temperature of 1030°C is reached, at which point the load is held for a minimum of 30 min to obtain temperature uniformity (with allowable temperature differences below 14° C) or for maximum 90 min until 1030°C is obtained on the surface. These guidelines limit thermal deformation and excessive growth of austenite grains. Figure 2 shows a graph illustrating the proper heating of a die according to those criteria.

Dies are hardened by quenching at maximum speed down to the temperature of 150° C in the core. The average cooling rate for the surface from 1030 down to 540° C should be at least 28° C min⁻¹. In the case of large dies (cross-sections above 300 mm) interrupted quenching (isothermal hold) at surface temperature of 400– 450° C is applied when the core temperature diverges by more than 110° C. The interrupted quench is completed when one of the following conditions occurs:

- (i) core temperature differs from surface temperature by less than $110^{\circ}\mathrm{C}$
- (ii) surface temperature drops below 400°C
- (iii) 30 min has passed from the start of the interrupted quench
- (iv) interrupted quenching is shown in Fig. 3.

FORD and GM have similar requirements concerning interrupted quench with the only difference being that

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1 Loading large die into vacuum furnace (courtesy of SECO/WARWICK)

FORD shortens the time to maximum 15 min (Fig. 4) while GM to only 5 min (Fig. 5) and, at the same time, accelerates the quenching rate to 39° C min⁻¹ (28° C min⁻¹ for NADCA).

Quenching is continued until 150° C is reached in the core (50° C on the surface) and is immediately followed by tempering. The workpieces should not be cooled down below the temperature of 33° C. The required cooling rate is significant due to the risk of excessive grain boundary precipitation of carbides that result in worse impact strength. Interrupted quenching limits the temperature difference between surface and core and thus reduces stress and deformation, protects the workpiece against cracking while at the same time preventing the creation of a pearlitic structure.

The first tempering is carried out at a minimum temperature of 565°C holding for the time that depends on tool cross-section (1 h/25 mm), though not less than 2 h. This is followed by cooling to ambient temperature



2 Guidelines and progress of real austenitisation and interrupted quench with monitoring of furnace temperature and of die surface/core temperatures (in accordance with NADCA guidelines)



3 Guidelines and progress of actual interrupted quench with monitoring of furnace temperature and of die surface/core temperatures (in accordance with NADCA guidelines)

and second tempering at a minimum temperature of 550°C. A third tempering is not usually necessary and is applied only for final adjustment of hardness. Tempering reduces internal stress and ensures dimensional stability as well as proper structure and required hardness, usually within the range of 42–52 HRC.

Vacuum furnace for heat treatment of tools

The requirements for heat treatment of moulds and dies, dictated by NADCA, FORD, GM and others, can be achieved in a single chamber vacuum furnace equipped with high pressure cooling system in inert gas (type HPGQ – high pressure gas quench).^{4–8} Typical examples of such furnaces especially designed for the heat treatment of tools are shown in Fig. 6.

These furnaces have a compact design and, due to lack of emission of contaminants and other noxious substances, may be installed and operated in clean rooms and production facilities. They are equipped with a graphite heating chamber that provides for heating the workload to maximum temperature of 1300° C with temperature uniformity of $\pm 5^{\circ}$ C and better. This is facilitated by heating elements that work by radiation in vacuum and inert gas convection at low temperatures (heating and tempering). The furnaces use a high pressure inert gas quench (15 bar) with closed circuit circulation enforced by a blower. The high velocity



4 Guidelines and progress of actual interrupted quench with monitoring of furnace temperature and of die surface/core temperatures (in accordance with GM guidelines)



5 Guidelines and progress of actual interrupted quench with monitoring of furnace temperature and of die surface/core temperatures (in accordance with Ford guidelines)

cooling gas is directed onto the workload where the heat is transferred and collected to an internal heat exchanger. The system ensures very high intensity of cooling in nitrogen comparable to cooling in free oil (heat transfer coefficient up to $800 \text{ W m}^{-2} \text{ K}^{-1}$) and uniformity throughout the entire working space as well as very good penetration potential in densely packed loads. A typical system is illustrated in Fig. 7. This cooling system enables interrupted quenching by controlling cooling intensity through blower rotation and gas pressure, depending on the surface temperature of a processed workpiece.

In the case of tools of defined shape it is possible using the type of furnace shown in Fig. 7 to programme a sequence of directed flows of cooling gas – dynamic cooling. The choices include gas flow from all directions, from top and bottom, from both sides, 270° (four options) and from the front, all as shown in Fig. 8. The process may progress statically or change dynamically thus allowing practically unlimited number of combinations. This permits adjustment the cooling system operation depending on the geometry of workpieces and configuration of the workload in order to improve cooling uniformity and reduce deformation.

Experimental verification

Experiments using this type of furnace have shown that cooling with circumferential 270° inflow, dynamically



7 Cooling gas circulation at quenching phase shown on cross-section of SECO/WARWICK VECTOR furnace

changed at appropriate time sequence, is particularly effective. A significant acceleration of cooling rate was achieved as well as better equalisation of temperatures on workpiece surfaces.

The effectiveness of gas quench in these furnaces was proved on a reference steel block sized 400/400/400 mm (Fig. 9). Surface cooling rates substantially exceeding the minimum requirements for both NADCA – 28° C min⁻¹ and GM – 39° C min⁻¹ were obtained. Depending on the size of furnace's working space the following cooling rates of the steel block surface were achieved by quenching in nitrogen at 14 bar:

- (i) $1200 \times 1200 \times 1800 \text{ mm}; > 40^{\circ} \text{C min}^{-1}$
- (ii) $900 \times 800 \times 1200 \text{ mm}; >55^{\circ}\text{C min}^{-1}$
- (iii) $600 \times 600 \times 900 \text{ mm}; > 80^{\circ}\text{C min}^{-1}$ (over 200°C min⁻¹ for 24 bar He).

A vacuum furnace can provide the entire process cycle to be effected in a single piece of equipment without transferring the workload, in a single work cycle, by performing the sequence of: preheating for austenitisation, interrupted quenching, mutiple tempering and also nitriding. The process may be monitored by workload thermocouples located at a critical place in the processed tool. Carrying out the treatment in vacuum and inert gases facilitates maintaining an ideal surface on the workpieces (Fig. 10).



6 Horizontal vacuum furnaces for tool treatment (VECTOR vacuum furnaces from SECO/WARWICK, size 900/800/ 1200 mm and 1200/1200/1800 mm, 15 bar guench)



8 Basic gas inflow options during quenching in VECTOR furnace. Top pictures: all around 360°, side-side, top-bottom. Bottom pictures: four options of 270°

Tool steel quench simulator

Defining interdependencies of structure, processing and operating properties is of key importance for proper practice in tool manufacturing. Today the traditional trialand-error method of optimising product properties and technological parameters is often replaced with computer based simulation and prediction methods that permit design of both the product and its processing. It is also in the area of thermal and thermochemical processing that we observe an increased interest in the applications for modelling and simulation of such phenomena. This pertains both to the progress of the process and to the final properties of the processed elements.^{9–15}

The mathematical basis of quench process and the dependence of material hardness from cooling time used by the G-Quench Pro software (Fig. 11) were drawn up following the research performed at the Technical University of Lodz, Poland and SECO/WARWICK as well as available literature. The direct result of the simulation is a cooling curve in given conditions. Determination of cooling curve is done on the basis of the parameters of the material, the process and the physical workpiece such as quenching temperature, type and pressure of quench gas, dimensions of the workpiece and

its shape, workload density in the cooling chamber. Combined with individual phase diagram for the material, the curve provides data on the phases through which the steel passes in the course of quenching. The ultimate effect of the simulation is defining the quench rate and expected final hardness of the material at a given depth.

The individual parameters of the quenching equipment largely determine the actual progress of the process, thus, at the installation phase the software is configured to suit a given furnace. In this way the individual characteristics of the furnace are taken into account when calculating the final properties of the product.

The software monitors the quench process in real time (on-line monitoring). In this mode the software is connected to the furnace control system and shows the cooling curve on a phase diagram on the basis of actual temperature measurements obtained from workload thermocouples. This option permits assessment of correctness of quench process while the latter is still in progress and allows amendments to be introduced if necessary.

Summary

Worked out by NADCA, the criteria and standards for production and exploitation of hot working steel are



9 Quench rate test in accordance with NADCA guidelines on reference steel block 400/400/400 mm



10 Die in vacuum furnace chamber following complex heat treatment



11 Overall view of software for simulation and control of tool steel quenching process

commonly applied or adopted in both American and European industry. The guidelines cover an entire spectrum of processes and process control referring to tool manufacturing, beginning from raw material, then heat treatment to application and repairs.

Heat treatment of hot working tools should be carried out in vacuum furnaces with gas quench and isothermal hold. The treatment should be performed at appropriate heating and cooling speed and should be monitored with workload thermocouples to control temperature difference within the material.

The vacuum furnaces described provide an ideal solution to meet NADCA, FORD, GM and other requirements concerning complex heat treatment of hot working tool steel. They also have potential to meet more restricted requirements in terms of cooling speed and uniformity in the future. The system of dynamic cooling enables the programmed (sequential and temporal) definition of quench gas flow direction that positively influences cooling uniformity and reduces deformation. The simulator software provides prediction of process results and optimum adjustment of cooling parameters to a given workpiece and furnace that ensures the appropriate outcome.

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