HEAT AND SURFACE TREATMENT OF HOT-WORK TOOL STEELS FOR OPTIMUM IN-SERVICE PERFORMANCE

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Abstract

The aim of this work is to study the influence of mechanical and metallurgical properties of hot-work tool steels determined by heat treatment and thermal-chemical surface treatment on in-service life of pressure die-casting tooling. The study is performed on Uddeholm hot-work tool steels; Dievar, Orvar Supreme, QRO 90 Supreme, and Vidar Supreme. A number of heat treatment parameters, such as austenitizing temperature, quenching speed, heat treatment system, and different tempering approaches is studied. Thermal-chemical surface treatments such as nitriding and oxidation are also studied. To characterize the material, the hardness and toughness are measured and metallographic examination is performed. A test apparatus is developed for assessment of thermal fatigue resistance of the materials, which is estimated by the surface area of cracks developed on the surface of test specimens subjected to cyclic thermal loading. Material characterization is performed at different stages of the thermal fatigue test to study the evolution of mechanical and metallurgical properties throughout the service life. Temperature transients at different locations of test specimens are measured and used in computation of transient stresses performed by finite elements. A relationship between the thermal fatigue resistance and surface stresses is developed. It is also expressed as a function of the initial material properties and the material type. The results are applied at STEEL Group heat-treatment facility to optimize the heat treatment and thermal-chemical surface treatment parameters in order to provide best performance and in-service life of pressure die-casting tooling.

Keywords: High-pressure die casting, hot-work tool steel, heat treatment, thermal fatigue, toughness, hardness, microstructure, finite element modeling.

INTRODUCTION

Both metallurgical and mechanical properties of hot-work tool steels critically depend on heat treatment. Different standard procedures have been developed specifying minimum heat treatment requirements [1, 2]. Although most hot-work tool steels possess high hardenability and can be hardened in air, it is wellknown that significant improvement in mechanical properties can be achieved by increasing the quenching speed. In addition to hardness, these processes define the material toughness and tempering resistance. In die-casting industry, for example, where extreme temperature cycling at the tool working surface is one of the most frequent causes of the material failure, the optimization of these material properties is crucial. To lessen the detrimental effects of gross cracking, material toughness is maximized. This material property may vary significantly for a given hardness dependent on
the heat treatment performed. In addition, it is shown that material resistance
to tempering significantly depends on heat treatment performed [3]. Higher
tempering resistance prevents a rapid hardness loss, which is a contributing
factor to thermal fatigue cracking at the die working surface [4].

In the last decade, significant advancements in development of the heat-
treating equipment have been achieved. Heat treatment systems that allow
for high quenching velocities are available on the market. These range from
relatively simple salt-bath quenching systems, modern vacuum systems with
high-pressure nitrogen or helium quenching, and up to most sophisticated
dual chamber vacuum systems [5].

Rapid quenching causes high thermal gradients resulting in high transient
stresses as well as residual stresses and distortion of the treated part. There-
fore, a part of given geometry should be quenched at an optimal speed, high
enough to provide the benefit of rapid quenching to material properties, but
not too high to prevent the risk of excessive distortion or cracking. Different
approaches can be chosen to determine this optimum quenching speed
for a given part. These range from empirical approaches, [2], to more so-
phisticated finite element modeling approaches developed to optimize heat
treatment processes of certain materials [6, 7, 8]. An advanced optimization
software-tool for heat treatment of tool steels based on a pre-calculated data
using finite elements is also presented [9].

This paper is reporting the status of an ongoing research of the effect of
heat treatment on the mechanical and metallurgical properties of different
hot-work steels. The main idea is to establish a correlation between the
basic mechanical properties, hardness and toughness, and the thermal fatigue
resistance. In order to accomplish this, a special apparatus is designed to
perform thermal fatigue testing in conditions that are similar to those at the
working surface of a die in operation. The apparatus and the thermal fatigue
specimens are described in detail. Furthermore, for appropriate evaluation
of the thermal fatigue resistance, the temperatures at different locations of
the thermal fatigue specimen is measured and the finite element computation
of stresses is performed. To evaluate the severity of the thermal fatigue test
in comparison to the conditions at the die surface, a modeling study of a
critical die at in-service conditions is performed.
EXPERIMENTAL

The main idea of this research is a critical examination of different steels subjected to different heat and surface treatments utilized for pressure die-casting tooling in terms of mechanical and metallurgical properties. It is also the objective to perform thermal fatigue testing on these materials and establish a correlation between the basic mechanical properties, hardness and toughness, and thermal fatigue resistance. The effect of surface treatment on thermal fatigue resistance is evaluated separately.

A thermal fatigue testing apparatus is developed and the tests are performed on specially designed test specimens, see Figs. 1, 2, and [10]. Especially designed specimens are prepared to allow temperature measurement at different axial and through-thickness locations. The measured temperature cycles in the specimens are used for: (1) comparison with the intensity of thermal gradients measured at critical points in a pressure die-casting die, and (2) to compute the thermal stresses in the specimen during a test cycle.

Figure 1. Thermal fatigue testing apparatus
EXPERIMENTAL PROGRAM

A comprehensive test program is designed to study the material properties and resistance to thermal fatigue of Uddeholm Dievar, Orvar, QRO 90, and Vidar subjected to different heat treatments and thermochemical surface treatments. The austenitizing temperature and some particular quenching and tempering parameters are studied for different heat treatment systems. The surface nitriding and oxidation treatments are applied to some specimens. Three test specimens are prepared for each heat and surface treatment performed; a block of $15 \times 55 \times 60$ mm is used to machine three Charpy V-notch specimens and specimens for hardness and microstructure testing; two $25 \times 25 \times 150$ mm specimens for thermal fatigue testing are machined.
After the heat treatment, the Charpy V-notch specimens are machined and the impact testing performed. The hardness is measured and the microstructural characteristics are evaluated at the same specimens.

The thermal fatigue specimens are subjected to 16000 thermal cycles. A program of inspection and evaluation is developed that defines the tests performed every 4000 cycles. At 4000 cycles the specimens are inspected for number of cracks and edge rounding at a pre-determined edge section. At 8000 cycles, in addition to parameters evaluated at 4000 cycles, one of the specimens is sectioned to inspect the average crack length, the microhardness profile, and the microstructural characteristics of the material. At 12000 cycles, the same inspection as at 4000 cycles is performed. At 16000 cycles, the second specimen is used for complete examination.

Temperature measurement is performed by thermocouples (TC) inserted to 1.6 mm diameter holes drilled next to the inner and outer surface of the specimen. The outer TC is positioned at the specimen edge with the centerline at 1.2 mm from both outer surfaces, whereas the inner TC’s centerline is also placed 1.2 mm from the inner specimen surface. Three specimens were prepared with sets of holes to the depth of 70 mm, 90 mm, and 110 mm from the top specimen surface. The edge section between 70 and 110 mm, measured from the top of the specimen is the evaluation region for the thermal fatigue cracking, edge rounding, and the microhardness profile.

THERMAL FATIGUE TESTING APPARATUS

Figure 1 shows the testing apparatus developed to simulate thermal cycling conditions that occur at the surface of a high-pressure die-casting (HPDC) die. The test specimen is immersed into molten aluminum at 700 °C, then cooled at air, and finally immersed into a water solution of graphite or sprayed with an emulsion to prevent aluminum from attacking to the specimen surface. The duration of a typical cycle is between 30 and 40s; 8-12s of immersion in aluminum, 2-4s of immersion in water or spraying, and 20-24 of air cooling. Four specimens are mounted simultaneously on a specially designed fixtures connected to cooling system, providing a continuous internal cooling of the specimens. The specimen fixtures are fastened on a plate driven by the pneumatic cylinders in vertical and horizontal direction, so the furnace with molten aluminum and the basin with coolant/lubricant is reached. The pneumatic system is controlled by a personal computer hav-
ing the possibility to define the speed of each movement and the time of permanence in the aluminum and the cooling bath.

Despite the lubrication of the test specimens, there is a tendency of the aluminum to attach to the specimen surface. This represented a serious problem for the testing: (1) the aluminum attached on the surface sometimes formed a relatively thick, pot-shaped attachment, that brought a certain amount of water into the molten aluminum causing dangerous explosions, and (2) although the aluminum attached remelted in each cycle, it significantly affects the temperature gradient in test specimen. This is because the heat from the aluminum bath is not transmitted to heat the specimen, but to melt the aluminum attached on the surface. Such test is inefficient and can not be evaluated using the established thermal fatigue criteria. To prevent this behavior, a system was developed to clean the aluminum from the specimen surface at the time it leaves the aluminum bath.

Figure 2 shows the specimen, which is a 25×25 mm square section, 150 mm long with a 10 mm diameter axial hole to the depth of 140 mm. Inside the hole is inserted a tube connected to the cooling circuit that brings the cooling water of 20°C to the bottom of the test specimen. The water then flows upward between the tube and the specimen inner wall and continuously cools the inner specimen surface. This cooling assures the high temperature gradient between the inner and the outer surface of the specimen while in contact with molten aluminum. The high thermal gradient causes high axial stresses that peak at the specimen edge. Finite elements are used to calculate the temperature distribution in the specimen and the corresponding axial stresses throughout the heating and cooling transient.

**COMPUTATIONAL**

**THERMAL STRESS ANALYSIS OF THE SPECIMEN**

A typical thermal cycle measured in the thermal fatigue test specimen is modeled using the ABAQUS finite element code [11]. The objective of modeling study is to determine the axial stresses in the specimen throughout the heating and cooling transient. The stress transient represents a basis for evaluation of thermal fatigue resistance of the material.

A 2D finite element model is developed of the specimen cross-section. The model and the applied boundary conditions are shown in Fig. 3. The Cartesian coordinate system is used with the origin in the center of the inner
hole, and the geometric symmetry in both axes is considered. The section is modeled with 360 linear eight-node elements. In mechanical analysis, which is performed separately from the thermal, the corresponding plane-strain elements were used.

The heat transfer coefficient between the specimen and the aluminum bath, the air, and the emulsion fluid is computed based on the comparison between the experimental and computational results. The computed heat transfer coefficients are then used to calculate temperature distribution in the specimen throughout the transient.

The mechanical analysis was performed in a separate run, uncoupled with the thermal analysis. The elastic-plastic constitutive model was used and the stress-strain curve was characterized by elastic modulus, the yield stress, the ultimate stress and the elongation, thus defining the work hardening properties of the material. A strain rate independent model with the Von Mises yield criterion, isotropic hardening, and a Poisson’s ratio of 0.3 was used in all analyses. Temperature dependent mechanical properties were used in computations.

*Figure 3.* The finite element mesh
ANALYSIS OF THE TEMPERATURE CYCLES AT A CRITICAL HPDC TOOL

In a separate study a thermal analysis of a critical HPDC die is performed for the first 15 cycles of filling, solidification, die cooling and emulsion spraying. Two different cases are studied: (1) the die is preheated to 200°C before the first cycle starts, and (2) the first cycle starts without preheat, i.e. cold start is performed. The temperatures are recorded at 16 different positions in the die. At each position, the temperature is recorded at the surface, at 3 mm and at 6 mm below the surface. The surface temperature is recorded at 0.1 to 0.3 mm beneath the surface depending on the discretization density of the finite-difference model established by the MAGMA Soft computer code used in computations. The results are used to determine the severity of temperature gradients at different locations in the die and to compare them with the temperature gradients measured in thermal fatigue testing specimens. This way the results of thermal fatigue testing can be used to make predictions of in-service life of actual HPDC tooling.

RESULTS AND DISCUSSION
TEMPERATURES IN THE THERMAL FATIGUE SPECIMEN

Temperature measurement in the thermal fatigue specimen is performed at two different cooling regimes. In the first regime both the continuous cooling with the cooling water inside the specimen and the intermediate cooling achieved by 2-4s immersion of the specimen into the cooling basin are applied. In the second regime, only the continuous cooling inside the specimen is applied. Figure 4(a) shows temperatures of a few cycles measured using the two TCs at the outer and inner surface. The peak temperature measured at the outer surface is 460°C, whereas the maximum at the inner surface is about 330°C, giving a maximum through-thickness gradient of 130°C (the distance between the TCs is about 8 mm). The minimum cycle temperature is about 85°C for both surfaces. The maximum temperature transient is 375°C and 245°C for outer and inner surface respectively. Figure 4(b) shows the temperature measured for the case of inside cooling only. The temperatures are higher: 500°C at the outer surface, 370°C at the inner surface, with the minimum cycle temperature of 145°C. The through-thickness gradient
is about the same as in the first case, whereas the maximum temperature difference is lower for both surfaces. Note that the TC’s centerline is positioned at about 1.2 mm beneath the surface, which means that the peak temperature for the outer surface is higher, and lower for the inner surface. The exact surface temperatures are to be calculated using the finite element model developed.

Figure 5 shows the temperature distribution in the cross-section of the thermal fatigue specimen before exiting from molten aluminum. The high temperature gradient between the outer surface and the inner surface causes high axial stresses. The temperature at the outer surface edge is considerably higher than in the rest of the outer surface. Therefore, the highest axial stresses are expected at the outer surface edge (compressive when in aluminum bath and tensile when in cooling bath), which is also the location of the first cracking expected.

**TEMPERATURES AT A HPDC DIE SURFACE**

Figure 6(a) shows the temperatures at a critical location of a HPDC die at the first temperature cycle. The temperatures are computed for cold-start, which means the die is not preheated before stamping, and hot-start, where the die is preheated to 200 °C. The temperatures are recorded at the surface, 3 mm and 6 mm below the surface at the exit from the feeding channel. The maximum surface temperature is 470 °C and 515 °C for cold-start and
Figure 5. Temperature distribution in the thermal fatigue test sample before exiting from the molten aluminum
preheated die respectively. Figure 6(b) shows the same temperatures for the cycle number 14, where the die is already at in-service regime. This is confirmed by the minimum effect of initial die preheat on the peak surface temperature, which are for some 20°C higher from the peak temperatures of the first cycle.

![Figure 6](image)

*Figure 6.* Temperature transient in a HPDC die measured at the exit from feeding channel at the surface, 3 mm and 6 mm below the surface.

The detrimental effects of the cold-start are shown in Fig. 7(a), which shows the temperature difference between the surface TC and the TC positioned 6 mm below the surface. The temperature gradient in the first 6 mm underneath the surface reaches 350 °C, which is 100 °C higher than the temperature gradient of the first cycle made with the preheated die. Figure 7(b) shows that temperature gradient lowers to 200 °C when the die reaches the stable in-service regime, regardless of the preheat temperature.

The conditions at this critical location compare very well to the conditions at thermal fatigue test, bearing in mind that the temperature at the test specimen is measured at 1.2 mm beneath the surface. The temperature cycle computed at the exit from the feeding channel is extremely severe. Both the peak temperature and the temperature gradient of all other locations examined at the die are considerably less severe.
MECHANICAL AND METALLURGICAL PROPERTIES

Figure 8 demonstrates the effect of quenching speed on the mechanical properties of Dievar. The two specimens quenched in oil and in air are tempered to the same hardness of 48 HRc. The toughness of the oil quenched specimen is higher for 25%. A similar effect of the cooling speed is expected on tempering resistance. The evaluation will be performed after completion of thermal fatigue testing.

Figure 9(a) shows a homogenous martensitic microstructure of the specimen quenched in oil. Figure 9(b) is a micrograph of the air cooled specimen of Dievar. The difference between the two microstructures is obvious. The coarse grain microstructure with evident grain boundaries of air cooled specimen explains the lower toughness obtained.

THERMAL FATIGUE EVALUATION

At this stage a preliminary evaluation of thermal fatigue damage is performed on an Orvar Supreme specimen after completion of 6000 cycles on the thermal fatigue testing apparatus. As expected the cracking first appeared along the specimen edge. The cracks are perpendicular to the specimen axial direction. Therefore, the cracking is caused by high axial stresses at the specimen edge, which are the result of high through-thickness temperature gradients and temperature cycling at the edge.
Figure 8. Hardness and toughness of Uddeholm Dievar subjected to different heat treatment cycles

(a) subjected to rapid oil quenching  
(b) subjected to air cooling. Nital 4% etch

Figure 9. Micrograph showing the structure of Uddeholm Dievar

A series of parameters is used to evaluate the thermal fatigue resistance of the materials studied. The parameters evaluated are: (1) the number of cracks, (2) edge rounding, (3) average crack depth, (4) microhardness profile from the surface. The parameters are evaluated along a 40 mm segment of the edge, between 70 mm and 110 mm from the specimen top side.
Figure 10(a) shows the specimen sectioned in transverse direction to evaluate the edge rounding. Note the 0.1 mm deep region of the edge material that is severely damaged by thermal fatigue and exposure to high temperatures. Exposure to high temperatures resulted in a tempered structure of a hardness inferior to that of the original specimen. The microhardness measured at the damaged edge region is 410 HV$_{0.2}$, at the distance of 0.1 mm from the surface is at 470 HV$_{0.2}$, whereas at 0.2 mm the microhardness equals to that of original material which is 550 HV$_{0.2}$.

Figure 10(b) shows cracking along the specimen edge sectioned in axial direction. At this stage an average of 10 cracks per millimeter are found with the length between 10 and 300 µm, and the average crack length of 69 µm.

Figure 10(c) shows a preferential transgranular cracking not affected by the microstructural characteristics of the material. The crack propagates in a direction perpendicular to the specimen surface. Figure 10(d) shows the tip of the same crack at higher magnification.

**CONCLUSIONS**

A comprehensive test program is designed to study the material properties and resistance to thermal fatigue of Uddeholm hot-work tool steels subjected to different heat treatments and thermochemical surface treatments. The results of a preliminary evaluation of Dievar specimens show a 25% higher toughness for the case of rapid oil-cooling with respect to slow air cooling at the same hardness level. The microstructure analysis confirms the mechanical property results.

An apparatus is made to perform thermal fatigue testing by immersion to molten aluminum of specially designed specimens with a continuous internal water cooling. A preliminary testing is performed and thermal fatigue resistance of a specimen is evaluated using a series of parameters: number of cracks, average crack length, specimen edge rounding and microhardness profile.

Temperature measurement at different locations in the thermal fatigue specimen is performed at two different cooling arrangements. The measured temperature transient are used in development and verification of finite element model used to analyze the stresses in the specimen throughout the thermal transient.

A thermal analysis of a critical HPDC die is performed by a commercial computer code. Temperatures are recorded at a number of locations at the
Figure 10. Micrographs used for evaluation of damage caused by thermal cycling after 5000 cycles.

(a) edge rounding and cracking
(b) typical cracks at specimen edge sectioned in axial direction
(c) a typical crack at specimen edge, 4% Nital etch
(d) tip of a typical crack at specimen edge, Fry’s solution etch
die surface. The temperature gradient underneath the die surface is also analyzed. Two startup regimes are studied: (1) cold start, and (2) start with die preheated to 200°C. The results are used to compare the thermal conditions in the thermal fatigue specimen with conditions at an actual die working-surface. The results reveal detrimental effects of the cold-start on the die material.

The evaluation of a thermal fatigue test specimen at 6000 cycles shows numerous transgranular cracks of an average length of 69 µm, propagating perpendicularly into the material. The test thermal conditions correspond to those at critical spots at the die working surface. Note that the thermal fatigue specimen is not subjected to pressure nor the wear of high-velocity aluminum flow.

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