INFLUENCE OF SURFACE ENGINEERING ON THE PERFORMANCE OF TOOL STEELS FOR DIE CASTING

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Abstract
Thermal fatigue cracking and wear by erosion and corrosion are important life-limiting failure mechanisms in die casting dies. To develop new and more resistant tool materials for this application detailed knowledge of the casting conditions, the failure mechanisms and their aggressiveness are essential. Experimental simulations have successfully been applied to study the failure mechanisms and also to evaluate the resistance of tool steels and surface engineered materials against failure in die casting.

This study elucidates the thermal conditions during actual die casting of brass. In addition, thermal fatigue and corrosive wear of surface engineered hot work tool steel specimens were experimentally evaluated. Thermal fatigue cracking was evaluated for the following conditions of a hot work tool steel: quenched and tempered (reference), treated by boriding and Toyota diffusion (CrC), respectively, PVD CrN-coated and duplex-treated topped with a PVD CrN-coating, respectively. A special study of corrosive wear of CrN PVD coatings applied on hot work tool steel specimens after treatment in an aluminium melt was also performed.
Temperature profiles in the surface layer of the mould were recorded and
details of the thermal cycling during actual die casting were obtained. With
the exception of duplex-treatment, all variants of surface engineering had
a tendency to decrease the resistance against thermal fatigue cracking as
compared to the reference steel. The mechanisms of initiation and progress
of liquid aluminium corrosion of CrN coated tool steels have been explained.
The corrosion resistance of CrN coatings improve with the coating thickness.
Finally, since the duplex-treated PVD CrN coating shows a high resistance to
surface cracking, and the corrosion resistance can be significantly improved
by CrN coatings, it is concluded that there is a potential to improve life and
performance of die casting tools by surface engineering.

**Keywords:** Thermal fatigue, Corrosive wear, Failure, Temperature, Die casting.

**INTRODUCTION**

Die casting involves injection of molten metal, for example aluminium,
zinc, magnesium and copper based alloys, into a mould [1, 2, 3]. Prior
to casting aluminium and copper alloys, the die is normally preheated to
a temperature of 250-300°C and 300-350°C, respectively, and the average
temperature is usually kept at those levels through internal cooling. High
velocity of the liquid metal during injection provides rapid filling of the die
cavity. For aluminium alloys, the entrance velocity during injection of the
melt is typically 20-60 m/s and the melt temperature is approximately 700°C,
whereas those for copper alloys is about 1-10 m/s and approximately 970°C.
When the casting has solidified, the die is opened and the casting is ejected.
Thereafter, the die may be externally cooled and lubricated by spraying. Hot
work tool steels, such as AISI H11, H13, H21 or H22, are frequently used
as die materials.

The life and performance of die casting dies is limited because of a num-
ber of reasons such as thermal fatigue cracking (heat checking), erosion,
corrosion, local adherence of the casting alloy to the tool (soldering), and
gross fracture [1, 2, 3]. Thermal fatigue cracking results from the cycling
of the tool temperature, which may induce stresses high enough to impose
plastic strain in the tool surface during each cycle. Surface cracks appear
already after a few thousand castings, or even earlier, and are, therefore,
formed in the low-cycle fatigue range [4]. However, it is reported that creep
and oxidation may significantly contribute to cracking [5, 6, 7]. The thermal
fatigue damage is often observed as a network of fine cracks on the tool
Erosion is induced by the high velocity of the incoming melt and partially due to solid particles in the molten casting alloy. The erosive damage is primarily seen where the molten metal jet first hits the die surface. Corrosion damage originates from dissolution of the tool material into the liquid metal. Erosion and corrosion may cause significant loss of surface material. The mentioned failure mechanisms degrade the surface finish and geometrical tolerances of the tool and, therefore, also those of the cast products, and may eventually cause rejection of the casting. Consequently, die failure increases the production costs.

Surface engineering is today successfully introduced to improve the erosion and corrosion resistance as well as to reduce soldering of dies and die materials [8, 9, 10, 11, 12, 13, 14, 15]. In addition, it has been shown that surface engineered materials may show increased or decreased resistance to thermal fatigue cracking as compared to untreated materials [11, 12, 13, 14, 15]. However, the mechanisms behind these discrepancies are not fully understood. Experimental techniques have successfully been utilised to study failure mechanisms and also to evaluate the resistance of tool steels and surface engineered materials.

To develop new and more resistant tool materials for die casting detailed knowledge of the casting conditions, the failure mechanisms as well as their aggressiveness are essential. In this study, the temperature variations in the surface layer of hot work tool steel, as heat treated or surface engineered, were experimentally recorded during actual brass die casting. Thermal fatigue and corrosive wear of surface engineered hot work tool steel specimens were experimentally evaluated. Various types of surface engineering were studied including surface treatment (boriding and Toyota diffusion to give CrC) and physically vapour deposited (PVD) coatings of CrN used as a single-layer or as the top layer in duplex-treatment (nitriding followed by PVD coating). The untreated hot work tool steel was used as a reference material. Corrosive wear of PVD coatings of CrN applied on hot work tool steel specimens and treated in an aluminium melt was also studied.
EXPERIMENTAL TEMPERATURE RECORDING DURING ACTUAL DIE CASTING OF BRASS

Field test equipment. For the temperature measurements, a relatively simple tool used for production of tube couplings in brass was selected. It was used in a 1.5 MN cold chamber machine in actual production runs. The temperature of the brass melt was 980°C and the total cycle time 30 s during which the die was closed 10 s and open 20 s. Water at 20°C was continuously circulated through cooling channels in the tool. In addition, the tool surfaces were lubricated but not intentionally cooled by spraying. The total shot weight of each casting was 1.6 kg, the peak casting pressure 164 MPa, and the entrance velocity of the melt was about 1.5 m/s. Note that the tool in this study was not preheated.

Four cylindrical measurement probes (Ø 16 mm) were designed, each including a small cylindrical test specimen (Ø 8 mm) and a K-type (Chromel-Alumel) thermocouple with thin wires (Ø 0.13 mm), spot welded to the back of the specimen. The thin wires enable rapid response of any change in temperature. The thickness of the four test specimens was 0.25, 0.50, 2.0 and 5.0 mm, respectively. Consequently, the temperature variations at these depths below the surface are obtained. A hot work tool steel (Uddeholm QRO 90 Supreme) of about 520 HV_30 was used both in the tool and the probes.

The temperature data was collected simultaneously from all thermocouples with a sampling rate of 500 Hz, using a customised data acquisition system. More information is presented elsewhere [16].

Materials. A hot work tool steel, Uddeholm QRO 90 Supreme, with the nominal chemical composition (wt. %) 0.38 C, 0.30 Si, 0.75 Mn, 2.6 Cr, 2.25 Mo, 0.9 V and Fe balance, was used as test material. The specimens were hardened and tempered (austenitizing 30 min at 1030°C and tempering 2 × 2 h at 625°C) to a hardness of 500 ± 5 HV_30.

The same steel, heat treated as above, was also borided (~25 h at ~850°C) and CrN-coated by PVD, respectively. Prior to surface engineering, the specimens were ground and polished with 1µm diamond paste in the last step. The boriding process was followed by the same heat treatment as above. The PVD-CrN coating was applied to the hardened and tempered steel, at
a process temperature of 300°C. All treatments resulted in a martensitic microstructure for the steel. The thickness of the diffusion zone profile of the borided specimens was 27 ± 4 µm, and the PVD coating thickness was 4.6 ± 0.3 µm.

The brass cast alloy CuZn33Pb2Si-C (Ametal C, Tour & Andersson designation) was used in the tests. It has a nominal liquidus temperature of 887°C and a solidus temperature of 844°C, and an approximate chemical composition (wt. %), obtained by X-ray spectroscopy on a casting, of 64.1 Cu, 32.3 Zn, 1.9 Pb, 0.71 Si, 0.35 Fe, 0.05 As, 0.03 Al. More information is presented elsewhere [16].

**THERMAL FATIGUE TESTS**

**Materials.** A hot work tool steel, Uddeholm QRO 90 Supreme, with the nominal chemical composition (wt. %) 0.38 C, 0.30 Si, 0.75 Mn, 2.6 Cr, 2.25 Mo, 0.9 V and Fe balance, was used as test material. The reference specimens were hardened and tempered (austenitizing 30 min at 1030°C and tempering 2 × 2 h at 625°C), followed by fine grinding to a surface roughness (Rₐ) of 0.38 ± 0.05 µm.

Prior to surface engineering, the specimens were ground and polished with 1 µm diamond paste in a last step to a surface roughness (Rₐ) of 20 ± 14 nm.

The specimens were surface treated by boriding (~25 h at ~850°C), Toyota diffusion to generate CrC (TDP CrC) (6 h at 1030°C) or plasma nitriding (15 h at 480°C), to produce a diffusion zone without any iron nitride compound layer. The boriding process was followed by hardening and tempering (at 1030°C and 2 × 2 h at 625°C, respectively), while the TDP treatment was followed by tempering 2 h at 625°C and 2 h at 600°C. All plasma nitrided specimens were duplex-treated with a PVD CrN coating on top of the nitrided layer. The PVD CrN coatings were produced in a multi-arc process, with a deposition temperature of 300-400°C. The five treatments resulted in different mechanical properties, see Table 1. More information is presented elsewhere [17].

**Thermal fatigue testing.** The test specimens are hollow cylinders with a diameter of 10 mm and a length of 80 mm, and have a 3 mm axial hole for internal cooling. An induction unit (25 kW, 3 MHz) heats the specimen surface. Continuous cooling is performed internally by circulating silicon oil of 60°C through the specimen, but also externally with argon, which also
Table 1. Mechanical properties of the test materials

<table>
<thead>
<tr>
<th></th>
<th>Ref.</th>
<th>Boriding</th>
<th>TDP CrC</th>
<th>PVD CrN</th>
<th>Nitr.+PVD CrN</th>
</tr>
</thead>
<tbody>
<tr>
<td>Substrate hardness [HV&lt;sub&gt;30&lt;/sub&gt;]</td>
<td>507 ±4</td>
<td>520 ±2</td>
<td>522 ±2</td>
<td>495 ±1</td>
<td>507 ±2</td>
</tr>
<tr>
<td>Surface hardness [HV&lt;sub&gt;0.025&lt;/sub&gt;]</td>
<td>—</td>
<td>1740 ±100</td>
<td>1970 ±70</td>
<td>2000 ±100</td>
<td>2060 ±100</td>
</tr>
<tr>
<td>Nitriding hardness [HV&lt;sub&gt;0.025&lt;/sub&gt;]</td>
<td>—</td>
<td>—</td>
<td>—</td>
<td>—</td>
<td>915 ±15</td>
</tr>
<tr>
<td>Diffusion depth [µm]</td>
<td>—</td>
<td>30 ±2</td>
<td>30 ±2</td>
<td>—</td>
<td>160 ±3</td>
</tr>
<tr>
<td>Coating thickness [µm]</td>
<td>—</td>
<td>—</td>
<td>—</td>
<td>6.1 ±0.1</td>
<td>4.5±0.2</td>
</tr>
</tbody>
</table>

decreases oxidation during the thermal cycling. The specimen surface represents the surface of the die and the induction heating and cooling simulates the temperature cycles during die casting.

Two temperature cycles were used to simulate aluminium and brass die casting conditions, respectively. They include a steep ramp to the maximum temperature, followed by a short hold time (<0.1 s), and subsequent cooling to the minimum temperature. To simulate aluminium and brass die casting, the maximum temperatures were set to 700°C and 850°C, respectively. The minimum temperature for both cycles was set to 170°C. The heating times in the 700°C and 850°C cycles were 0.4 and 2.5 s, respectively, and the total cycle times were 14.4 and 26.5 s, respectively.

Prior to testing, the specimens were pre-oxidised in order to get a thin oxide layer, which facilitates the pyrometer temperature control during heating. More information is presented elsewhere [17, 18].

**CORROSION TESTS**

**Materials.** The substrate material used in the test specimens was a premium grade AISI H13 hot work tool steel, ORVAR SUPREME (Uddeholm Tooling designation), with the nominal chemical composition (wt. %) 0.39 C, 1.0 Si, 0.4 Mn, 5.2 Cr, 1.4 Mo, 0.9 V and Fe balance. Prior to coating the steel was hardened and tempered to a nominal hardness of 45–48 HRC.
Table 2. Substrate temperature, deposition time and resulting thickness of the CrN coatings

<table>
<thead>
<tr>
<th>Designation</th>
<th>Substrate temperature [°C]</th>
<th>Deposition time [min]</th>
<th>Coating thickness [µm]</th>
</tr>
</thead>
<tbody>
<tr>
<td>LL</td>
<td>230</td>
<td>50</td>
<td>2.9 ±0.2</td>
</tr>
<tr>
<td>LH</td>
<td>230</td>
<td>175</td>
<td>13.6 ±0.4</td>
</tr>
<tr>
<td>ML</td>
<td>280</td>
<td>50</td>
<td>4.2 ±0.3</td>
</tr>
<tr>
<td>MH</td>
<td>325</td>
<td>175</td>
<td>20.1 ±1.4</td>
</tr>
<tr>
<td>HL</td>
<td>440</td>
<td>40</td>
<td>3.2 ±0.3</td>
</tr>
<tr>
<td>HH</td>
<td>430</td>
<td>70</td>
<td>18.9 ±1.1</td>
</tr>
</tbody>
</table>

aluminium alloy used in the tests was ISO Al-Si8Cu3Fe with the nominal chemical composition (wt. %) 3 Cu, <1.2 Fe, 9 Si and Al balance. This alloy is similar to the common die casting alloy AA380.0.

The PVD CrN coatings were made in a similar way to those of the thermal fatigue test. By varying the substrate temperature and deposition time, see Table 2, six coating series were produced, representing six combinations of coating thickness and defect density. Of the coating designations given in Table 2, L denotes both low temperature and short time, M denotes medium temperature, and H represents both high temperature and long deposition time. Each series consisted of five specimens, except test series HH, which consisted of three. More information is presented elsewhere [19].

Corrosion testing. The experimental set-up consisted of a resistively heated crucible for the aluminium melt, and a circular specimen holder for three specimens, and a thermocouple. The melt temperature was controlled to 710 ± 10°C. To maintain uniform temperature and concentration during testing, the liquid aluminium was stirred by a ceramic plate rotating at 80 rev./min. Cylindrical specimens, 90 mm in length, 13 mm in diameter, and with the tip rounded to a 6.5 mm radius, were used. During each test three specimens and the thermocouple were clamped symmetrically in the circular specimen holder and submerged 1 h in the liquid aluminium. More information is presented elsewhere [19].
RESULTS

RECORDED TEMPERATURE PROFILES IN ACTUAL BRASS DIE CASTING

The measured temperature profiles consisted typically of a steep ramp from the minimum to the maximum temperature, followed by a more gradual decrease in temperature. During the first cycles (less than 20), the minimum temperature increases from room temperature to about 300°C, where after the temperature cycles are almost identical. A representative temperature recording after equilibrium is shown in Fig. 1. As expected, the maximum temperature and the heating time were strongly dependent on the distance from the surface, while the minimum temperature appeared to be relatively independent of the depth. Note that the minimum temperature is approximately the same at the beginning and at the end of a cycle at each depth in the tool which proves that steady-state is reached. Finally, note that the temperature decreases rapidly, especially near the surface when the die opens and the casting is ejected. No additional cooling effect from the lubrication can be observed.

The thin surface layers from the boriding or CrN treatment did not have any notable effect on the thermal conditions.

SURFACE CRACKING AFTER THERMAL FATIGUE TEST

Thermal fatigue cracking of the surface engineered tool steel after thermal cycling is exemplified in Figs. 2a and 2b. The crack growth was strongly dependent on the number of cycles, and it was significantly faster during the 850°C cycles as compared to the 700°C cycles. The resistance to cracking (as maximum and mean length of cracks such as that of Fig. 2b and crack density (as number of cracks per unit of length) differs significantly between the surface engineering, see Fig. 3. It is seen that the boriding, TDP, and CrN coating show a tendency to impair the resistance to thermal fatigue cracking as compared to the reference material. It is also seen that the duplex-treatment proved comparable to the reference material, but gave an increased resistance to surface cracking as well as a reduced density of cracks as compared to the single-layered CrN coating.
Figure 1. Typical temperature profiles at different depths from the surface obtained at steady-state condition (Boronized specimens at cycle 20.).
Figure 2. Example of thermal fatigue cracking after treatment with the 700°C cycle (borided specimens).

Figure 3. Maximum and mean crack length as well as crack density after 5 000 cycles to 700°C. Three reference specimens and two specimens of each treatment were tested. The error bars indicate the maximum and minimum value.
COATING CHARACTERISTICS AFTER CORROSION TEST

Coating damage in the form of localised circular bulges was frequently detected by SEM, see Fig. 4a. Fractured cross-sections revealed a corrosive attack on the substrate material under the bulge, see Fig. 4b. Note the pinhole defects in the centre of the bulges.

Macroscopic corrosive damage was observed in the SEM as local corrosion pits in the coating surface, see Fig. 5a. Note that the damage consists of circular corrosion pits, as well as pits that have grown together and formed larger cavities. Local areas without any detected corrosive attack on the coating material was observed even though the loss of the coating was large, see Fig. 5b.

The fraction of corroded area occupied by the pits differs significantly between the series, see Fig. 6. It is seen that the thick coatings (group LH, MH and HH) show a low amount of corroded area, while the thin coatings except ML show a high fraction.

DISCUSSION

TEMPERATURE CYCLING OF DIE SURFACE DURING ACTUAL BRASS DIE CASTING

A typical heat distribution in the tool surface layer during one casting cycle is seen in Fig. 1. When the 980°C melt makes contact with the tool, the tool
material is heated within about 0.35 s from around 300°C to a maximum temperature of around 750°C at a depth of 0.25 mm. Deeper below the tool surface the maximum temperature is lower, and the heating rate is reduced. Until the tool is opened, cooling occurs by heat conduction into the bulk of the tool. Die opening and simultaneous cast ejection give rise to an additional heat loss through irradiation and convection, which naturally is most notable at the 0.25 and 0.5 mm depths.

Detailed knowledge of the thermal cyclic nature in the surface layer of a die casting tool during actual service conditions can be used to perform realistic imitations of the heat cycling in, for example, experimental and numerical simulations of thermal fatigue. Since the thermal cyclic conditions is most severe during the first phase of the casting cycle (when the die is closed, cp. Fig. 1), knowledge of the cyclic nature in the surface layer during this period is of greatest interest. For the 0.25–5.0 mm surface layer, the following approximate values were found: Maximum temperature 750-450°C, minimum temperature ∼300°C, heating time 0.35-4 s, and heating rate 1250–40°C/s.

From measured temperature profiles such as those of Fig. 1, the maximum surface temperature and surface heat flux can be estimated to approximately 826°C and about 9.8 MW/m², respectively [16].
Figure 6. Fraction of corroded area vs. the coating thickness.
THERMAL FATIGUE CRACKING

In general, the boriding, TDP and CrN coating show a tendency to decrease the resistance to thermal fatigue cracking as compared to the reference material, see Fig. 3. Additionally, the resistance to cracking among these seems to increase with the surface hardness, which is indicated by the fact that the density of cracks was significantly lower for the TDP than for the borided material. To resist thermal cracking, a material should, for example, have a high hot hardness or hot yield strength, but also sufficient ductility, since the hot yield strength controls the plastic strain for a given temperature cycle, and the ductility represents the ability to resist plastic strain without cracking [2]. The engineered surfaces have higher hardness levels than the reference material, and it is consequently expected that their hot yield strength is higher and their ductility is lower than for the reference material. However, the high deposition temperature of the boriding and the TDP processes seem to deteriorate the mechanical properties of the substrate more than the nitriding and PVD processes do, even if this is not reflected by the substrate hardness numbers. This is supported by the fact that the maximum crack lengths in the borided and TDP treated materials are well beyond the diffusion depths. Consequently, the subsurface and substrate properties are very different. However, the difference between the thermal fatigue resistance and the crack density between the two categories of surface engineering is explained by the combined effect of differences in plastic response, residual stress state of the surface zone, as well as differences in these properties of the substrate.

Finally, it is clearly demonstrated that the duplex-treatment results in an increased resistance to surface cracking as well as a reduced density of cracks as compared to the single-layered CrN coating, see Fig. 3. This indicates that the plasma nitriding process prior to coating plays a dominant role to inhibit crack initiation and to slow down the crack propagation. The initiation and growth of cracks is probably slowed down as a consequence of the increased strength and the compressive stresses generated in a zone beneath the surface during plasma nitriding. Simplified, this reduces the surface plastic strain and the tensile stress intensity range during thermal cycling and, consequently, the driving force for crack initiation and growth.
CORROSION

Previously, the corrosive damage of coated materials exposed to liquid aluminium has been observed as local attacks in the form of corrosion pits, while other areas seemed to be completely unaffected by the exposure. In addition, it is indicated that the corrosion pits result from local coating defects rather than from any intrinsic deficiency [9, 10]. Finally, the unaffected areas between the corrosion pits (cp. Fig. 5) indicate that the coatings themselves are inert to the exposure of liquid aluminium [9]. Since the coating material is inert, the defects behind the corrosion must penetrate through the coating.

The mechanisms of initiation and progress of liquid aluminium corrosion of CrN coated tool steels have previously been explained [9, 19]. It was observed that the corrosive attack is initiated through pinhole defects, which act as small channels for the liquid aluminium through the coating down to the substrate (cp. Fig. 4), where intermetallic phase transformation and volume expansion due to diffusion of aluminium occurred. The volume below the defect continues to expand and local coating detachment occurs when the deflection of the coating is excessively large. In the following, the craters coalesce to aggravate the corrosion as evidenced by Fig. 5a [9].

From Fig. 6 it is evident that the resistance to corrosive wear is improved by increasing the coating thickness. This is supported by previous observations that the density of defects through the coating (pinhole defects) decrease with increasing coating thickness [19].

CONCLUSIONS

In this study, the temperature variations in a surface layer of hot work tool steel, as heat treated or surface engineered, were experimentally recorded during actual brass die casting. In addition, thermal fatigue and corrosive wear of surface engineered hot work tool steel specimens were experimentally evaluated. The following main conclusions can be drawn.

- The temperature profiles in the surface layer of the mould were accurately recorded and details of the thermal cyclic conditions were obtained.

- Thermal fatigue cracking of a surface engineered tool steel is primarily influenced by the modification of the mechanical properties of the substrate which occurs during the engineering process.
With the exception of duplex-treatment, all variants of surface engineering decrease the resistance to thermal fatigue cracking as compared to the reference steel.

However, the fact that the duplex-treated PVD CrN coating increased the resistance to thermal fatigue cracking as well as reduces the density of cracks as compared to the single-layered CrN coating, the potential to improve the life and performance in for example die casting applications still prevails.

Liquid aluminium corrosion of CrN coated tool steels occurs as follows. Initially, through-the-coating defects act as channels and cause the liquid aluminium to locally attack the steel. The subsequently formed corrosion pits coalesce and the corrosive attack aggravates.

The corrosion resistance of CrN coatings is improved by increasing their thickness.

The fact that the defect free areas of the CrN coating did not show any corrosion indicates that the potential of further improvement the CrN/tool steel system is high.

Since the duplex-treated PVD CrN coating showed the highest resistance to surface cracking, and the corrosion resistance can be significantly improved by CrN coatings, there is a potential to improve life and performance of die casting tools by surface engineering.

ACKNOWLEDGMENTS

The authors like to acknowledge the Swedish Knowledge Foundation, Uddeholm Tooling AB, Tour & Andersson AB, and Bodycote Heat Treatment AB for their financial and material support.

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Influence of Surface Engineering on the Performance of Tool Steels for Die Casting