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EXPERIMENTAL INVESTIGATION ON THE INFLUENCE OF SURFACE ENGINEERING ON THERMAL FATIGUE OF A HOT WORK TOOL STEEL

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ABSTRACT

Thermal fatigue is an important life-limiting factor in die casting moulds. It is observed as a network of fine cracks on the surface exposed to thermal cycling. The crack network degrades the surface quality of the tool and, consequently, the surface of the casting. Surface engineered materials are today successfully utilised to improve the erosion and corrosion resistance. However, their resistance when exposed to thermal cycling is not fully understood.

In this work, surface treatments (boriding and Toyota diffusion to give CrC) and physically vapour deposited (PVD) coatings of CrN, as single-layered or duplex-treated (nitriding followed by PVD coating), on hot work tool steel specimens were compared with the untreated reference material by experimental simulation of thermal fatigue. The test is based on induction heating and surface strain measurements by a non-contact laser speckle technique, which enables studies of the surface strain during thermal cycling.

Thermal fatigue cracking of a surface engineered tool steel is 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 show a tendency to 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.

KEYWORDS

Thermal fatigue, Heat checking, Die casting, Coating, Surface treatment, Hot work tool steel.

INTRODUCTION

During a die casting cycle, molten alloys of e.g. aluminium, zinc, magnesium or copper-based alloys are forced into an internally cooled mould by the application of pressure [1-3]. The molten metal flows with high velocity during injection and provides rapid filling of the die. A fill time in the order of milliseconds is a distinguishing characteristic of die casting. The high velocity injection allows high rate manufacturing of products in a wide range of thin-walled and complex shapes, typically 100 parts per hour. When the casting has solidified and cooled, the die is opened and the casting is ejected. Subsequently, the die may be externally cooled and lubricated by spraying. The die is normally preheated to a temperature within the range of 150 to 350 °C, depending on the type of casting alloy. For aluminium and brass, the melt temperature is approximately 700 and 970 °C, respectively. During injection, the entrance velocity of the liquid metal is typically within the range of 20 to 60 m/s, but can be significantly lower (1 to 10 m/s) for brass. The metal pressure during injection can exceed 70 MPa. Hot work tool steels, such as AISI H11, H13, H21 and H22, are frequently used as die materials.

Thermal fatigue (or heat checking) is an important life-limiting factor in die casting moulds [1-3]. It is a fatigue process caused by the cycling of stress, strain and temperature in the die surface through the thermal cyclic nature of the casting process. Thermal fatigue of die casting moulds is a low cycle fatigue phenomenon basically controlled by the plastic strain imposed on the surface. However, both creep and oxidation may significantly contribute to damage and in some cases they can be the dominant failure mechanisms [4-7]. Thermal fatigue damage is often observed as a network of fine cracks on the surface exposed to thermal cycling. The crack network degrades the surface quality of the tool and, consequently, the surface finish of the cast products, and may eventually cause rejection of the casting. In addition to thermal fatigue cracking, gross fracture, erosion, corrosion and local adherence of the casting alloy (soldering) are other important failure modes which limit the life and performance of die casting dies.

Surface engineering is today successfully introduced to improve the erosion and corrosion resistance as well as to reduce soldering of dies and die materials [2, 8-13]. In addition, tests show that surface engineered materials may increase or decrease the resistance to thermal fatigue cracking as compared to an untreated material [11-13]. However, the resistance of surface engineered materials exposed to thermal cycling is not fully explored.

To increase the understanding of the thermal fatigue behaviour of surface engineered hot work tool steels exposed to thermal cycling it is necessary to perform experimental simulations. Since thermal fatigue cracks are usually limited to a thin surface layer of the tool, the surface response during the thermal cycling is of particular interest. In this study the following conditions of a hot work tool steel were evaluated: quenched and tempered (reference), treated by boriding and Toyota diffusion (CrC), CrN-coated and duplex-treated (CrN), respectively. The test is based on induction heating and surface strain measurements through a non-contact laser speckle technique. This enables studies of the changes of the response of the surface during thermal cycling.

EXPERIMENTAL

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 bal., 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 of (R_a-value) 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 of (R_a-value) 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.

TABLE 1

MECHANICAL PROPERTIES OF THE MATERIALS

	Reference	Boriding	TDP CrC	PVD CrN	Nitriding + PVD CrN
Substrate hardness [HV_{30}]	507 ± 4	519 ± 2	522 ± 2	495 ± 1	507 ± 2
Surface hardness [$HV_{0.025}$]	-	1740 ± 100	1970 ± 70	2000 ± 100	2060 ± 100
Nitriding hardness [$HV_{0.025}$]	-	-	-	-	915 ± 15
Diffusion depth [μm]	-	29 ± 2	29 ± 1	-	159 ± 3
Coating thickness [μm]	-	-	-	6.1 ± 0.1	4.5 ± 0.2

Characterisation

Substrate hardness was assessed by macro-hardness (Vickers) measurements on polished cross-sections, using a load of 30 kg. Surface roughness (R_a-value) was measured using an optical surface profilometer.

For the specimens treated by boriding, TDP CrC and nitriding, the depth of the diffusion zone profiles were assessed by micro-hardness (Vickers) measurements on polished cross-sections, using a load of 25 g. The diffusion depth was defined as the depth beneath the coated surface at which the hardness was 50 $HV_{0.025}$ higher than the substrate hardness. For the specimens treated by boriding the depth is given excluding the outmost compound layer. PVD coating thicknesses were determined by light optical microscopy (LOM) on polished cross-sections.

Relative hardness of the surface treatments and surface coatings was assessed by micro-hardness (Vickers) indentations on polished cross-sections and in the surface of the coatings, respectively, using a load of 25 g. For the specimens treated by boriding, the surface hardness was measured below the compound layer.

Thermal fatigue testing

The test equipment is based on induction heating and surface strain measurements through a non-contact laser speckle technique, which makes it possible to calculate the strains induced in the specimen surface during thermal cycling. The test specimens are hollow cylinders with a diameter of 10 mm, 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 by circulating silicon oil of 60 °C through the specimen, but also externally with argon, which also 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. More information is presented elsewhere [14].

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 [14]. 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 pyrometer temperature control during heating. The reference specimens were pre-oxidised by electrochemical oxidisation in a NaOH-solution at 70 °C, followed by heat treatment, as described elsewhere [14]. All surface treated and surface coated specimens were pre-oxidised at 600 °C for 0.5 hour. In addition, a K-type (Chromel-Alumel) thermocouple with a wire diameter of 0.13 mm was welded to the specimen to measure the surface temperature during testing. Finally, to obtain a good speckle pattern for the surface strain measurements, an area of approximately 10×10 mm located in the middle of the specimens was roughened by a 1000 mesh abrasive paper or 3 μm diamond paste.

Evaluation of thermal fatigue

Induction heating using a frequency of 3 MHz give rise to heating of only a thin zone of the surface ('skin-effect'). During the thermal cycling, the total strain of the surface ε_{tot} is obtained by the laser speckle technique from the change in the specimen dimensions. The mechanical strain ε_{mech} generates the stress which is necessary for any surface cracking. It is due to the fact that the heat is inhomogeneously distributed through the specimen during thermal cycling. Through knowledge of the temperature cycle and the coefficient of thermal expansion of the tool material, it is possible to estimate the contribution from the thermal expansion/contraction ε_{th} of the unevenly heated/cooled specimen surface at any time. The mechanical strain is obtained as [15]:

$$\varepsilon_{mech} = \varepsilon_{tot} - \varepsilon_{th} \tag{1}$$

Crack propagation is represented as maximum crack length versus the number of thermal cycles. The thermal fatigue resistance (as maximum and mean crack length) and the crack density (the number of cracks per unit of length) was evaluated from crack length measurements after 5000 cycles to 700 °C. All evaluation of cracks is based on cracks larger than ~5 µm, where all measurements were performed on polished axial cross-sections by LOM. In addition, the crack pattern on the surface was studied using scanning electron microscopy (SEM). Finally, the mechanism of crack propagation was studied using LOM.

RESULTS AND DISCUSSION

Surface response

Typically, a linear increase in compressive ε_{mech} occurred with temperature, followed by a reduction of ε_{mech} during the first part of the cooling, see Fig. 1a and b. Subsequently, ε_{mech} moved toward an increasing or a decreasing compressive strain. The response of the surface during heating is a consequence of the constraint conditions due to the cooler bulk material, which retains the expansion of the surface. During the first part of the cooling, contraction of the surface occurs simultaneous with expansion of the bulk and, subsequently, both the surface and the bulk are contracting but at different rates. However, the response of the surface changes with increasing number of thermal cycles, represented by a decreasing residual mechanical strain range $\Delta\varepsilon_{residual}$, defined in Fig. 1a. Note that ε_{mech} at the peak temperature is approximately the same for all cycles.

Finally, each strain loop ends with a residual mechanical surface strain, which range is larger in the axial than in the tangential direction, see Fig. 1b. A difference in constraint conditions between the axial and tangential direction is probably the explanation. This indicates that the accumulated strain imposed on the surface during one thermal cycle is larger in the axial than in the tangential direction. Consequently, it is expected that the initial growth of the crack network is perpendicular to the axial direction of the specimen. The axial $\Delta\varepsilon_{residual}$, as normalised with respect to cycle 10, followed the same decreasing trend with increasing number of cycles for the surface engineered specimens as for the reference material, see Fig. 1c. The residual strain represents a cyclic creep strain and is governed by the plasticity of the material and the constraint conditions [16]. This indicates that the surface strain response during thermal cycling depends on changes of the mechanical properties of the substrate, as a consequence of for example cyclic and thermal softening, and a type of shakedown effect.

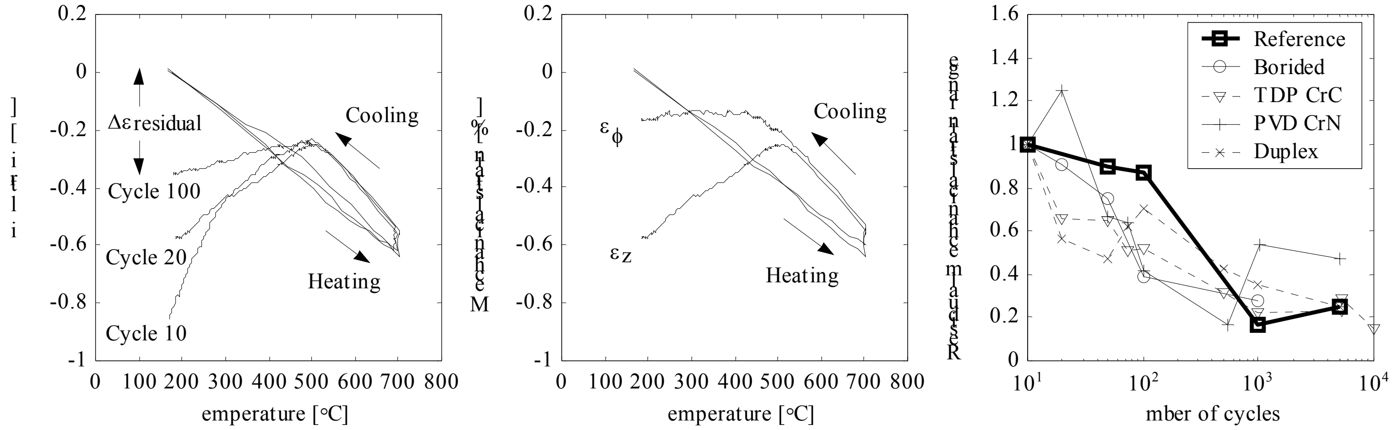

Fig. 1. Examples of the surface response during the 700 °C thermal cycling. a) Evolution of the axial ε_{mech}, including definition of the residual mechanical strain range ($\Delta\varepsilon_{residual}$). b) Comparison between axial strain (ε_z) and tangential strain (ε_ϕ) at cycle 20.
c) Comparison between the normalised axial $\Delta\varepsilon_{residual}$ versus the number of cycles for the reference and the surface engineered specimens.

Development of the thermal fatigue crack network

This study indicates that the thermal fatigue cracks observed on the CrN coatings, after 5000 cycles to 700 °C, initiate at local coating defects, see Fig. 2a, followed by predominant growth perpendicular to the direction of the largest $\Delta\varepsilon_{residual}$, see Fig. 1b and 2b. Initially, the preferred circumferential crack growth is expected since the accumulated damage during one cycle is larger in the axial direction, as indicated previously. In addition, it was frequently observed that the crack network developed through the growth of cracks connected by local coating defects, see Fig. 2c, where the defects probably act as points of initiation in the coating. Crack initiation and propagation during thermal cycling to 10 000 cycles results in a random network of cracks, see Fig. 2d. This crack pattern corresponds well to that observed on die casting dies.

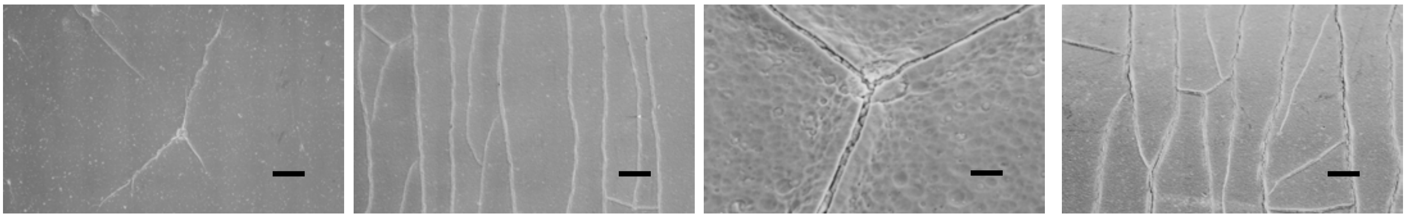

Fig. 2. Typical crack pattern observed using SEM on a CrN coated specimens after treatment with 700 °C cycles. a) Local surface crack at a local surface defect found on a duplex-treated specimen after 5000 cycles b) Cracks predominantly in the tangential direction observed on a CrN coated specimen after 5000 cycles. c) Close-up of b revealing coating defect as connection point for the cracks. d) Crack network observed on a CrN coated specimen after 10 000 cycles. (The axial direction is horizontal in all pictures.)

Crack growth

The crack growth was strongly dependent on the number of cycles and the maximum temperature during each cycle, see Fig. 3a. Since the crack propagation rate was very high during the 850 °C cycles, it was necessary to limit the number of these cycles to 1000. Studies of the mechanism of crack propagation using LOM on borided specimens revealed crack branching after exposure to the 850 °C cycles, whereas no branching of the cracks was observed after the 700 °C cycles, see Fig. 3b and c. The increased crack propagation rate for the 850 °C cycles is assumed to be an effect of increased plastic strain, increased effective crack driving force, or a decreased crack resistance.

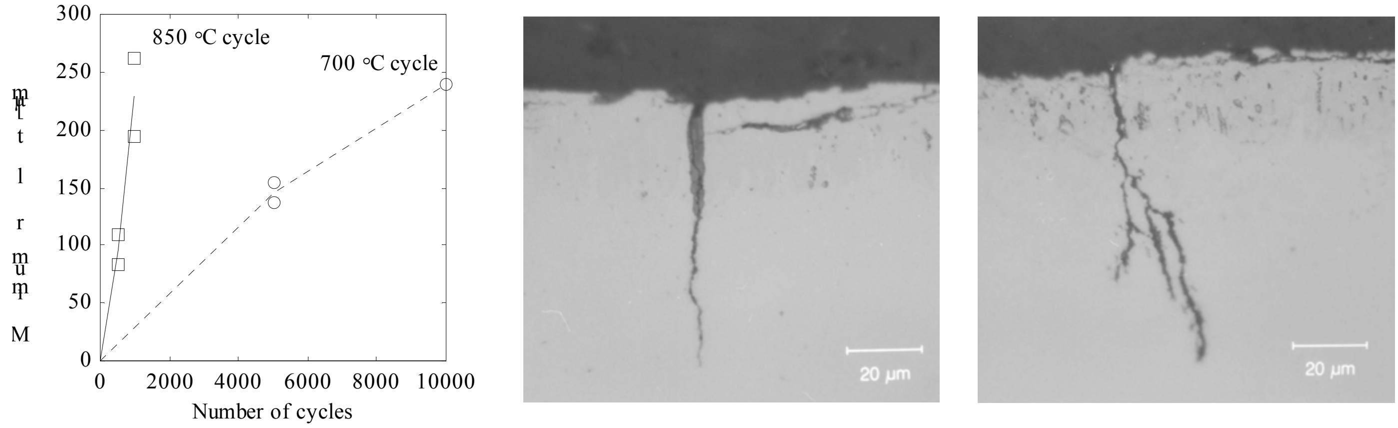

Fig. 3. Crack growth and mode of crack propagation of the borided specimens observed in LOM. a) Crack growth rate. b) Mode of propagation revealed after 5000 cycles to 700 °C. c) Crack propagation by branching revealed after 500 cycles to 850 °C.

In general, the boriding, TDP and CrN coating shows a tendency to decrease the resistance to thermal fatigue cracking as compared to the reference material, see Fig. 4. Additionally, the resistance to cracking among these seems to increase with the surface hardness. The density of cracks was significantly lower for the TDP than for the borided material which probably correlates to its higher surface hardness. 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 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 probably affects the mechanical properties of the substrate differently as compared to that of the nitriding and PVD processes. When considering the maximum crack lengths in the borided and TDP treated materials, it is observed that they are well beyond the diffusion depths. This implies that the subsurface and substrate properties are different. However, the difference between the thermal fatigue resistance and the crack density between the two categories of surface engineerings 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. 4. 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 [16]. 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.

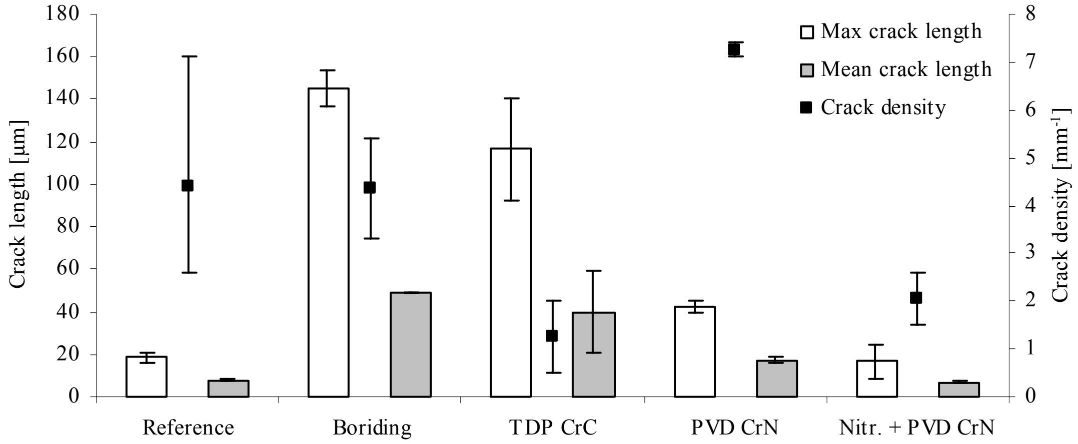

Fig. 4. Maximum and mean crack length as well as crack density after 5000 cycles to 700 °C. Three reference and two specimens of each treatment were tested. The error bars indicate the maximum and minimum value.

CONCLUSIONS

In this study, thermal fatigue of a tool during die casting of aluminium and brass was experimentally evaluated. A selection of surface treatments and PVD coatings of a reference hot work tool steel were included. The following conclusions can be drawn.

- Through induction heating the test equipment makes it possible to simulate failure of surface engineered tools during thermal cycling, as well as to deduce the surface strains responsible for the failure.
- Thermal fatigue cracking of a surface engineered tool steel is 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 show a tendency to 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.
- The initiation and progress of the crack network in CrN coatings on top of tool steel has been described.
- It is clearly demonstrated that the crack growth rate is rapidly increasing with the maximum cycle temperature above 700 °C. This indicates that an attempt to increase the life of tools exposed to thermal cycling should aim at decreasing the maximum temperature.

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