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THERMAL FATIGUE IN HOT WORK TOOL STEELS - A REVIEW

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Abstract

Thermal fatigue is a dominant mechanism that causes premature failure in components exposed to high temperature. In order to extend the useful life of tools for hot work, studies have been conducted trying to understand the mechanisms involving thermal fatigue. Thus, different types of materials combined with different parameters of thermal and surface treatments have been investigated using thermal fatigue tests. This review addresses the main aspects of thermal fatigue as well as the main alternatives used to increase the resistance of the material to this type of failure.

Keywords: thermal fatigue, thermal cracks.

1 Introduction

During operations involving high temperatures, tools are subjected to thermal heating and cooling cycles which generate temperature gradients. These gradients cause locally different thermal expansions, inducing the generation of stresses and cyclic mechanical deformation that can cause thermal fatigue failure [1, 2].

Thermal fatigue is one of the most common mechanisms for crack initiation in hot work tool steels [2-4].

With the objective to extend the useful tool life, studies have been performed to further understand thermal fatigue mechanisms. Different heat treatment procedures have been evaluated using thermal fatigue tests. Additional treatments such as nitriding, post-oxidation, cryogenic and surface coatings have also been used to extend tool life.

Although the effects related to mechanical stress also play a significant role in the damage generated by tools for hot work, this review addresses only issues related purely to thermal fatigue, considering only the stresses caused by internal constraints on thermal expansion.

2 Discussion

2.1 Thermal fatigue

According to Spera (5), thermal fatigue is a gradual deterioration. It leads to cracking of the material by alternating heating and cooling during which the free thermal expansion is partially or completely constrained, causing a thermal stress that may initiate and propagate fatigue cracks. Although the process may be caused by an excessive temperature gradient, thermal fatigue can still develop under uniform temperature conditions of the specimen. In this case, instead of the internal constraints caused by the temperature gradient, the stresses are generated due to the different grain orientations in the microstructure or due to the anisotropy of the coefficient of thermal expansion of certain non-cubic crystals [6].

Thermal shock present in the thermal fatigue process induces a high tensile stress on the component which rapidly decreases through its thickness. Generally, in order for the thermal fatigue process to occur, it is necessary to have a restriction of the free expansion and thermal contraction which may be external or internal. External constraint is understood to mean those forces applied to the surface of the specimen being heated and cooled [5, 6].

In many applications where the components are subject to thermal fatigue, there is also loading by mechanical forces [1]. In this case, the thermally induced mechanical loads, and the loads resulting from the mechanical forces acting on the matrix, overlap in a very complex way causing strain and deformation that varies according to its intensity and orientation over time and also according to the location of the matrix. A detailed analysis of the loading condition of the matrices during the casting cycle is, even for simple geometry, only possible by the application of numerical methods [7].

The fatigue generated when external constraints are present during the thermal cycles is termed as thermomechanical fatigue, which is established by the combination of thermal cyclic loading with the mechanical cyclic load which generate synergistic damage to the components [5, 8].

There is also isothermal fatigue, which can be considered as a particular case of thermomechanical fatigue where the nominal temperature remains constant during the test. Although the thermomechanical fatigue test is of greater importance for real applications, a larger research database involving isothermal fatigue exists because thermomechanical fatigue experiments are more expensive and difficult to perform. The use of isothermal fatigue to predict the performance of the material under thermomechanical fatigue, however, has been shown to be inconvenient since most of the deformation and damage under thermomechanical fatigue cannot be predicted on the basis of isothermal fatigue information. Many existing models do not consider the interaction of mechanical deformation with temperature since this interaction is quite complex and not yet well understood [6].

2.2 Types of failures caused by thermal fatigue

In die-casting, forging, rolling and hot extrusion, tools are subjected to high temperatures and loading which may result in various surface failures. These failures can be caused, for example, by thermal fatigue, erosion, corrosion, deformation and soldering, which influence the productivity and quality of the surface thereby reducing the useful life of tools and dies [1, 9-13].

Among the various damage modes to which hot work tools are subjected, thermal fatigue is one of the most common failure mechanisms for crack initiation corresponding to more than 70% of the die failures [2, 3]. Cracks originate when a thermal stress at some point in the component exceeds the fracture resistance of the material, causing the component to fracture. Failures in a heating and cooling cycle occur when the material cannot deform plastically to relieve the thermal stress [14].

Cracks from thermal fatigue are characterized by their network formation, occurring predominantly on the surfaces exposed to thermal cycles, as shown in Figure 1 [4, 15-17]. Le Roux and collaborators have reported a detailed description in terms of geometry and topology characteristics of the thermal fatigue crack pattern, presenting different types of images and morphologies of network cracks [15]. It is important to note, however, the pattern of cracks formed by thermal fatigue may vary according to the material used. Thus, considering that the thermal fatigue behavior of steels varies according to their properties, steels must be carefully selected accounting for the type of mold and its shape [18].

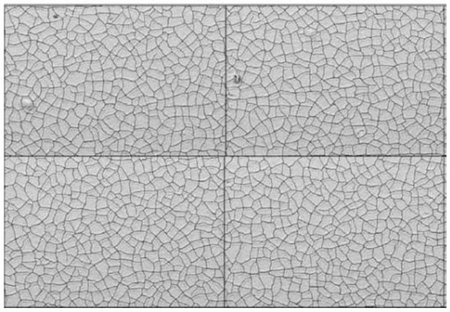

*Figure 1. Network cracking observed in the surface of probes made by*X38CrMoV5 *(AISI H11) steel after thermal fatigue tests. Source: adapted from Le Roux [15].*

Usually cracks caused by thermal fatigue appear after a few thousand cycles, or even before (low cycle fatigue), depending on the variables and process conditions and die design [4]. The effects of the atmosphere on the nucleation and propagation of cracks in tool steels subject to thermal fatigue have also been investigated which have shown that initial crack growth is facilitated by oxidation of the surface crack [19-22]. According some authors, there is an important relationship between thermal fatigue and steel oxidation during hot work. However, there is still a lack of detailed knowledge about the influence of oxidation on the initiation and propagation of thermal cracks [23].

Another factor that contributes to the initiation of surface cracks is the intense heating on the surface of the tool. This heating causes a tempering in the martensitic structure near the surface causing a decrease in the hardness which contributes to crack initiation and subsequent propagation [24]. Failures caused by thermal fatigue may also influence the occurrence of other damage to the surface of materials. Although there is, for example, no intrinsic relationship between thermal fatigue, soldering and erosion, it is known that cracks generated by thermal fatigue increase surface roughness which contributes to the increase in the occurrence of soldering and erosion on the surface of the molds [13].

During thermal fatigue, progressive changes in structure precede the formation of localized faults which may eventually reach a macroscopic size. The thermal fatigue process can be divided into three stages that partially overlap [25]. The first step would be the hardening and / or cyclic softening of the material since cyclic loading in metals and alloys can cause changes in their structures and consequently changes in their properties. In the second stage, crack nucleation occurs which is associated with the accumulation of local plastic surface deformation typical of low-cycle fatigue. In the case of aluminum die casting molds, another reason for crack nucleation could be the welding of fractions of the aluminum alloy on the surface of the tool, producing a corner, and thus facilitating crack nucleation [13, 26]. Finally, the propagation step occurs which is facilitated by the oxidation of cracks on the surface. In the case of aluminum casting dies, the subsequent growth of the cracks is further facilitated by the filling of the cracks with the molten material, by the oxidation, and by the softening of the tool material [4, 22, 27, 28].

Similar observations were pointed out by Mellouli and collaborators when investigating the process of initiation and propagation of thermal fatigue cracks in injection molding under pressure. The tests performed by the authors revealed that thermal fatigue was the first failure mechanism to occur and that crack initiation occurred due to local accumulation of plastic deformation at the die surface (typical of a low-cycle fatigue process). During the beginning of crack formation, the presence of surface oxidation was observed. It was further observed that crack growth was facilitated by filling with the molten alloy, by further oxidation, and by softening of the matrix material [29].

Damage from thermal fatigue is also heavily temperature dependent and may influence the way faults occur. As the maximum temperature of the cycle increases, crack transition from transgranular to intergranular type is usually observed. In this case, a possible explanation for this transition would be attributed to the possible weakening of the grain boundary due to the tension directed to the high diffusion activity. Another possibility would be that impurities and precipitates could accumulate in grain boundaries [17, 30]. In die casting, one of the main reasons for failure is the thermal stress resulting from the surface temperature gradient through the die section. Periodic thermal cycles lead to cracks in the surface and in the die. The magnitude of the thermal stress depends on the mechanical and thermal properties of the material, the heating and cooling rates, and the maximum and minimum temperature of the cycle [10, 31, 32].

Thermal fatigue cracks produced in the surface of the molds are replicated in their castings causing a deleterious effect in the quality of the component [22, 33]. Figure 2 shows replicated cracks on the surface of an aluminum part which was cast in a die made of H13 steel subjected to thermal fatigue [34].

Figure 2. Surface of an aluminum part which was cast in a die having cracks in the mold. Source: Price [34].

Crack lengths are strongly dependent on the number of thermal cycles, however, the crack density saturates in a relatively low number of cycles [35]. The temperature plays an important role in the intensity of damage caused by the thermal fatigue so that the increase in temperature implies increasing intensity of the generated cracks [11].

In general, the heating and cooling cycles are responsible for the thermal expansion and contraction of the material which generate fields of tension and deformation. These plastic deformations, when locally accumulated, lead to localized faults resulting in crack propagation as the die is used [11, 22, 27, 36, 37].

Faults generally begin to appear in the critical locations of the die, i.e. in places where shape and geometry change abruptly such as small-radius corners and also in places where there are residual stresses resulting from the manufacturing process [11, 31]. These critical locations are still subject to a stress generated by the pressure of the molten metal which further contributes to decreasing useful life of the tool [27].

Thermal fatigue effects can be reduced by controlling process parameters. Reduction of the thermal gradient generated during the process can extend the service life of a tool in service. In pressure casting, for example, it would be possible to minimize the damage by adopting some procedures such as: die design should avoid sharp corners and notches; residual stresses during die manufacture should be minimum; during the work process, uniform cooling should be maintained to prevent localized overheating that may affect the mechanical properties of the material; preheating the mold die to lower the temperature gradient; lowering the temperature of the molten alloy and the time of injection of each cycle; low injection rate should be applied to decrease the temperature gradient and erosion at the tool surface, and, finally, preventing rapid cooling of the die surface during deposition of the emulsion [33, 38]. It is important to note that damage to tools from contact with hot materials is primarily due to thermal fatigue but also mechanical and chemical loads must be considered [1]. Although the effects of time at maximum temperature on thermal cycles appear small when surfaces in service are maintained at high temperatures for a much longer time than those from experimental thermal cycles, the effects of thermal fatigue can be more severe. The reason for the disagreement between the results obtained in laboratory tests and the useful life of the tool in service may be partially due to this time effect [14].

Although tools for hot work are cited more often, the appearance of thermal cracks is also observed in other applications. Thermal fatigue cracks, for example, are one of the most common phenomena observed in many types of pressurized equipment, electrical, nuclear installations, steam turbines, among other applications [9].

2.3 Thermal Fatigue Test

Thermal fatigue test is an assay where the surface of the specimen is suddenly heated and cooled cyclically. This test is generally used when it is desired to evaluate the ability of the material to withstand the development of cracks on its surface. In these tests, thermal fatigue cracks appear on the surface of the specimen after a defined number of cycles, therefore, the number of cycles required to produce a visible crack is generally considered to be a characteristic of the material's resistance to thermal fatigue. During the thermal cycling process, the number of cracks and their lengths increase until a net number of cracks is produced. The increase in the maximum temperature of the test reduces, in most cases, the number of cycles before the development of cracks. Figure 3 shows the results of a typical thermal fatigue test relating the maximum temperature of the thermal cycles to the number of cycles required for the onset of cracking.

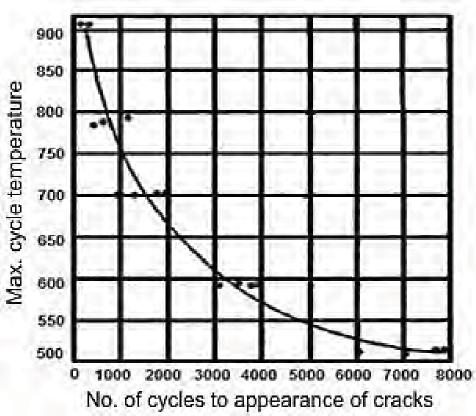

Figure 3. Results from thermal fatigue test in a Ni alloy for different temperatures. Source: adapted from Balandin [30].

There are different methods of thermal fatigue testing, which are characterized by varying the heating source on the surface of the specimen. One of the possible heating methods is through the use of a laser pulse. This method, in addition to enabling the assay to be performed within a vacuum chamber to inhibit oxidation, has also the advantage of providing high precision in the control of the temperature cycle. On the other hand, the small width of the laser beam limits the area of the surface to be heated, making difficult to observe and evaluate the damage caused by thermal fatigue [17].

Another type of thermal fatigue test is performed through repeated and alternate immersion of the test specimen, first into a hot liquid metal, followed by immersion into a cooling fluid, for example, water. After a certain number of heating and cooling cycles, the surface of the test specimen is analyzed to verify the extent of the damage caused by the thermal fatigue [4, 12, 31, 38]. This method provides good agreement with the real situation for die casting under pressure, but it is sensitive to the geometry of the test specimen and furthermore, due to the extreme surface coating by the metal, analysis of the potential surface damage of the test-piece can be compromised.

Another often applied method uses electromagnetic induction to simulate the heating of the surface of the test specimen [16, 17, 39, 40]. This heating system is usually composed of a coil, which is positioned around the test specimen for heating. In order to enable effective surface heating of the test specimen, an extremely high induction frequency is desirable, otherwise the use of a lower frequency will more effectively heat the inside of the test specimen. An advantage of this method is the high heating speed provided by electromagnetic induction thus minimizing the thermal cycling time and consequently the total testing time [40].

Another possibility of testing would be the heating through the use of convection ovens, however this method can be quite time consuming [9]. There are also other methods for heating the surface of test specimens, which are not very common, such as flame heating, friction heating, oil immersion heating, and fluidizing bed heating [41].

Since the 1970s, Howes had already noted the lack of standardization of thermal fatigue testing [41]. Unfortunately, almost half a century later this lack of standardization still persists which has made it difficult to compare the results of different investigations mainly due to the different geometries of the test bodies that are used in the tests which can influence the results as well as the variation of heat absorption [38]. Thus, due to the absence of standardized tests, it is suggested that comparative data should only be used for specific test conditions and specimen geometries and the other parameters should be constant [30]. For specific applications, the use of simple tests makes it very difficult to compare the thermal fatigue strength for different materials. To reproduce, for example, a loading situation in comparable tests between different materials, as well as for the practical application of the tools, it is necessary to perform numerical simulations [1].

2.4 Desirable properties for resistance to thermal fatigue

Inhibition of crack formation by thermal fatigue can be possible through the use of materials with thermomechanical and thermophysical properties suited to the working conditions. While there are several properties that should be considered, some are difficult to combine. It is generally agreed that for thermal fatigue resistance, the material must possess a low coefficient of thermal expansion, high heat resistance, good resistance to tempering, high creep resistance, adequate ductility, good toughness and high thermal conductivity [3, 6, 16, 42]. The temperature gradient is a function of the thermal conductivity of the material. As the thermal conductivity increases, the thermal gradient decreases, leading to a decrease in the maximum temperature during the heating process. As a result, less tension and deformation occur in the material, which leads to an increase in tool performance [1, 33].

Regardless of the application, there are some basic properties that hot work tool materials must have in order to perform well. Toughness and hardness are fundamental and serve to prevent the instant fracture of the tools or their edges, and the hardness must be high enough to avoid local plastic deformation [43].

The order of importance of the most suitable properties for thermal fatigue resistance cannot be established since material properties may vary according to the intended application [3, 44]. In die casting, where the useful life of the mold is often determined by thermal fatigue, ductility exhibits a greater effect on tool life than toughness [43]. Although a sufficiently high hardness prevents plastic deformation and fatigue, the prevention of instantaneous tool failures is linked with a critical hardness level which should not be exceeded for a given application. Molds for injection of aluminum under pressure, for example, typically exhibit hardness in the range of 45 ± 1 HRC [3].

In general, the material properties are interrelated and cannot be analyzed in isolation. The level of hardness, for example, competes directly with the toughness, since its increase can imply a loss of toughness and an increase in crack propagation [3, 43]. Experimental results for AISI H11 steel, subjected to several thermal treatments showed that toughness decreases and the rate of crack propagation increased with increasing hardness [45]. In another thermal fatigue study involving different materials, it was found that toughness was not the most significant property since the material with the highest thermal fatigue damage resistance also exhibited the lowest impact strength [40].

The hardness and toughness in hot work tool steels depend on the heat treatment procedure so that the hardness is closely linked with ductility and with the toughness. In the case of a hot working steel such as AISI H11, because it is used at high temperatures, it must be tempered at high temperatures (above the secondary hardening peak) in order to achieve the required hardness and toughness [43]. Although all of these desirable properties are well established, thermal fatigue resistance is a very complex material property since it strongly depends on the loading conditions (heat flow inside and outside the surface as a function of time, physical properties) and materials response to these loading conditions in terms of cyclic stresses and cyclic plastic and elastic deformations. It is important to consider that if plastic deformations occur, the loading conditions are gradually changed during the test or during the application of the tool significantly influencing the failure process [1].

2.5 Effects of heat treatment on the thermal fatigue

Thermal treatment is very important to achieve the required properties in each material, in this way, the parameters of these treatments must be adjusted according to the desired final properties required by the final application.

Since the microstructures of materials may be substantially modified by heat treatments and that each material property can be affected in a different way, studies have been conducted to determine heat treatment on thermal fatigue resistance [43, 46-48]. By comparing the effects of different structures obtained by different thermal treatments, it was observed that a purely martensitic microstructure exhibits better thermal fatigue resistance than the mixture of martensitic and bainitic microstructures [1].

Tool steels for hot working are generally supplied in the annealed state and their microstructure is formed by a ferritic matrix containing defined quantities of globular carbides. In order to enable the alloying process, it is necessary to dissolve most of these carbides in the matrix by a suitable heat treatment [49]. After the quenching and tempering, tool steels for hot work usually exhibit a microstructure consisting of a martensitic matrix with incorporated primary and secondary carbides. The size and volume fraction of the hardened secondary carbides exhibit the main influence on the hardness and thermal stability of the material [50, 51].

In addition to the influence of the microstructure, the residual stress state also strongly influences the behavior of the materials in relation to thermal fatigue [39, 52]. A study using test specimens with different mechanically treated surfaces showed that on surfaces with higher traction residual stress, the cracks appeared after relatively few cycles. However, when the same test specimens were previously subjected to stress relief, the occurrence of cracks was delayed [52]. The study, however, found a similarity in the rate of crack propagation for all test specimens regardless of the level of residual stress.

To improve materials properties, including thermal fatigue, additional treatments such as nitriding and surface coatings are being used increasingly although they are generally restricted to smaller matrices and components [1].

2.5.1 Influence of austenitizing temperature and tempering temperature on the hot work tool steel performance

Studies have shown that the use of different austenitizing temperatures in heat-hardened tool steels results in large differences in the thermal stability of the material [53, 54]. In addition, the effect of austenitizing temperatures on the appearance of cracks due to thermal fatigue has also been investigated [43, 45-47, 49]. Changes in the austenitizing and tempering temperatures modify and control the structure of the material at several different levels. These two parameters are somewhat independent with synergetic effects, both in the microstructure and in the fracture characteristics. On a microstructural scale, the austenitizing temperature influences the size of the martensitic lathes as well as the morphology and volume fraction of the primary carbides [45, 54]. On the nanostructural scale, the tempering temperature influences the secondary carbides, as well as the structural dislocations caused by tempering. The austenitizing on temperature may also alter the fraction of secondary carbide volume by modifying the amount of solubles in the austenite prior to quenching. Ductility, for example, can be improved by decreasing the amount of carbide present in the alloy, but on the other hand, can be substantially reduced with the growth of the austenitic grain [45].

Studies on the influence of austenitizing temperature on steels for hot work, shows a correlation with modifications of the microstructure. High temperatures affect toughness and hardness because of the carbide dissolution and its subsequent precipitation [43, 48, 49, 53, 54].

Austenitizing temperature also affects the amount and type of carbides that are dissolved which determines the alloying elements present in the austenite and the resulting martensite. The types of carbides precipitated during annealing affects performance of the tool and is closely related to the temperature selected [47, 53].

Hardening treatment of most tools can be a challenge for commercial heat treatments for different reasons. One of these problematic aspects would be the permanence of the material at a higher temperature during the quenching process, which occurs due to the difference in the exposure time at the austenitizing temperature between the surface and the core. This may result in increased austenitic grain size which is detrimental to the mechanical properties of the material [28]. Results of thermal fatigue studies indicate that the number of cycles required to initiate cracking decreases with increasing grain size [30]. Figure 4 shows the correlation between grain size and number of cycles for cracking nucleation in a typical thermal fatigue test.

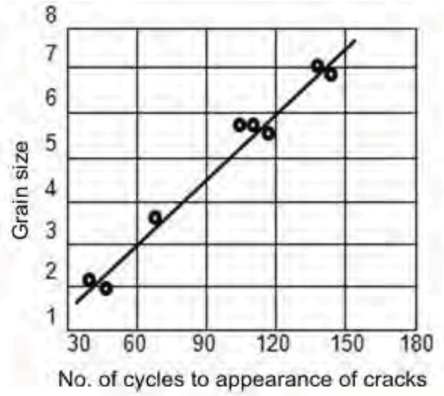

Figure 4. Effect of the grain size on the thermal fatigue resistance of Kh20N80 alloy. Max. temperature 850 ^{0}C, min. temperature 200 ^{0}C. Source: adapted from Balandin [30].

Under certain situations, the thermal fatigue conditions may not vary within a specific range of austenitizing temperature. In tests performed on AISI H11 steel, it was shown that an increase in austenitizing temperature from 990 ºC to 1050 ºC increased the grain size from 22 µm to 127 µm but did not influence the propagation rate of cracks and toughness [45]. The austenitizing temperature is material dependent and is generally recommended by the steel manufacturer. In specific cases, a confirmatory investigation should be performed as well.

The relationship of the tempering temperature on the hardness and toughness of the tool steel has shown that modifications in tempering that exhibits an indirect impact on the hardness of the alloys can result in a drastic change in the tenacity and rate of crack propagation [43]. Studies using AISI H13 and AISI H11 steels, toughness and hardness varied inversely as a function of increasing tempering temperature [44, 55].

These studies showed that with increasing tempering temperature, toughness decreases to a minimum value, then increasing at higher temperatures. Conversely, the hardness first increases to a maximum value and then gradually decreases with increasing temperature thus confirming that hardness and toughness behave in an opposite manner as shown in Figure 5.

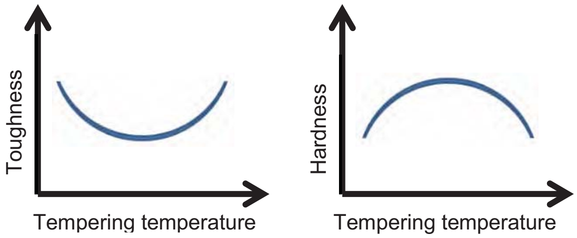

Figure 5 – Toughness and Hardness behavior as function of tempering temperature

In general, the hardness tends to decrease with increasing tempering temperature. An increase in the tempering temperature from 580 ºC to 640 ºC, a typical range for the second tempering may cause a decrease in hardness from 52 HRC to 40 HRC [56].

Another study investigating the influence of austenitizing and tempering temperatures on AISI H11 steel properties has also demonstrated that increasing tempering temperature leads to an increase in the precipitation of secondary carbides which increases toughness and reduces hardness [53].

In applications where the service temperature does not continuously exceed 600 ºC, there is no need to have a high resistance to tempering, instead, high ductility and toughness should be prioritized [17].

Although higher hardness and strength as well as a higher resistance to tempering can provide a better resistance to the thermal crack initiation, this may also lead to an increase in tensile stress concentration which can accelerate the crack propagation after nucleation [23].

2.6 Effects of the surface treatments on the thermal fatigue

Generally, all surface damage processes occurring in molds and dies including: thermal fatigue, plastic deformation, abrasive wear and erosion originate in the surface layer of the material. Thus, for higher durability of molds and dies, it is desirable that better properties are created in this layer which can be achieved with the use of coatings and layers with suitable characteristics [57]. Although the purpose of such treatments is usually to increase hardness and surface wear resistance while the core of the material remains ductile, other properties such as: fatigue strength, corrosion resistance and oxidation resistance in high temperatures can also be achieved.

2.6.1 Effects of the nitriding

Several thermochemical processes have been used to improve material properties. Among the most used processes are carburizing, nitriding, cyanidation, carbonitriding, nitrocarburizing, boronizing and thermo-reaction diffusion (TRD) [58]. Among these, nitriding (gas and plasma [60]) is one of the most common surface treatments for hot working steel dies and is mainly used for improving wear, corrosion and fatigue resistance [59]. Generally, the effects of nitriding on material properties depends on material composition and the final hardness gradient in the nitride layer which varies with process parameters such as temperature, average nitrogen composition and nitriding time [59, 61].

The effects of nitriding on thermal fatigue have shown varied results [39, 60, 62-64]. Changes in the process parameters exert a strong influence on thermal fatigue. However, the compound layer (white layer) that is generated may play a negative role during the conditions in service [39, 64]. Studies on the influence of gas and plasma nitriding on thermal fatigue resistance revealed that the brittle iron nitride layer facilitated crack nucleation [60]. In another study, however, the opposite results were obtained when plasma nitriding, even without the presence of compound layers, failed to offer any improvement in thermal fatigue at elevated temperatures. According to the authors, the life-limiting factor for nitrided tool steel is attributed to the extensive oxidation suffered during thermal cycling [65].

The low toughness of the diffusion layer which is deleterious to the thermal fatigue resistance may be compensated by the higher compression stress of the white layer. However, a study on thermal fatigue in plasma nitrided AISI H13 showed that the compression stress of the white layer, although initially higher, decreases more rapidly during thermal cycling [64].

As with the compound layer, the diffusion layer is also characterized by low toughness which favors rapid crack propagation from the surface to the core which has the original microstructure. This occurs because the nitrided surface induces a loss of toughness in the diffusion layer which facilitates crack propagation throughout the layer. However, this brittleness can be reduced by performing nitriding treatments with low N2 / H2 ratios for the formation of thinner diffusion layers with lower micro hardness values. In any case, an excess of nitriding should always be avoided once a thicker layer and inhomogeneous compound layer shows a tendency to reduce thermal fatigue resistance [63]. Recent studies have shown that nitriding performed using low pressures have promoted a high thermal fatigue resistance with low crack density [35].

Thermal fatigue of nitrided tool steel may also be affected by the softening due tempering. It has been shown that surface hardening by plasma nitriding was completely removed during thermal fatigue. In this case, the annealed martensitic structure was replaced by fine equiaxed ferritic grains suggesting a dynamic recrystallization process during thermal cycling. The thermal tensions generated on the surface during the tests were responsible for the structural change [62]. It has also been shown that the effects of nitriding on material properties can be improved when combined with PVD coatings [2, 57, 59, 66, 67].

2.6.2 Effects of the post-oxidation

Post-oxidation is a thermochemical treatment to increase corrosion resistance. It is a process widely used in aluminum injection molds and dies to increase corrosion resistance and decrease the surface adhesion of aluminum. There are, however, many published works relating the influence of post-oxidation treatment on thermal fatigue. In this process, the treatment time depends on the type of material, microstructure, and the depth of the desired oxide layer. By variation of these parameters, it is possible to obtain an oxidation layer that produces mechanical resistance for each application. The process is usually applied after nitriding or carbonitriding to provide high hardness and an excellent combination of properties.

Significant improvement in wear and corrosion resistance has been reported when the post-oxidation treatment is accompanied by nitriding [68-71].

In a study on the influence of the post-oxidation treatment on thermal fatigue, it was found that the post-oxidation treatment at 500 ° C produced a dense and adherent oxide film on the compound layer from plasma nitriding. However, this oxide layer failed to improve the oxidation resistance so that the tool steel with post-oxidation treatment failed in almost the same way as the tool steel without the treatment [62]. According to the study, the stresses acting on the surface during much of the thermal cycle led to a descaling of the oxide, exposing new surfaces to additional oxidation. The repetition of this sequence over the thermal cycles led to a substantial loss of material in the center of the front face of the samples. Thermal fatigue, in turn, could not be evaluated in this study because of the extensive damage caused by the oxidation which caused the thermal fatigue test to be interrupted after only 500 cycles before any evidence of cracking was observed.

In a more recent thermal fatigue test, where thermal cycling was performed by immersion of the test specimen in an aluminum alloy, the efficiency of the nitriding and post-oxidation treatments in protecting the surface of the samples against welding of the alloy, soldering, corrosion resistance, and thermal fatigue prevention [4].

A comparative study between carbonitriding in a salt bath and gas nitriding followed by post-oxidation showed that both surface treatments induced a compressive residual stress field and significant surface hardening. Carbonitriding produced a higher compressive residual stress although the depth was smaller compared to nitriding and post-oxidation treatment. In contrast, the surface hardness was almost equal for both treatments [72].

2.6.3 Effects of surface coatings

Various types of surface treatments and coatings applied to tools for hot work were evaluated to compare the effects of coating properties on tool performance [59]. Among the coating processes used to obtain resistant surface layers in tools, two are most commonly used; PVD (Physical vapor deposition) and CVD (Chemical vapor deposition). PVD processing is done under high vacuum and temperatures ranging from 150° to 500°C while the CVD process is performed at higher temperatures exceeding the tempering temperatures of the tools which may cause the substrate to soften. A further negative aspect of the process is its limitation in the coating of die-castings for injection under pressure, especially relative to large and geometrically complex steel molds [73].

PVD coatings are currently the most commonly applied for hot working dies. Technical obstacles to the application of the PVD coatings include: low corrosion protection caused by its thin layer, columnar structure, occluded droplets and dust particles. In addition, the thin layer of the PVD coating (30 μm versus 0.3 mm of the traditional coating) replicates the surface of the substrate without any smoothing or leveling [74]. A disadvantage of the PVD process would be the difficulty of its application in heavy and complex molds since the process of depositing or adjusting the mold assembly requires its movement which complicates the process and increases the cost. An alternative that has been used for the homogeneous coating of geometrically complex and heavy molds, without the need for movement or rotation of the mold, is the use of the plasma-assisted chemical vapor deposition (PACVD) [73].

CVD and PVD processes have advantages when used for dies but applications on surfaces exposed to thermal fatigue have not been successful in preventing damage. Most of the success has been in reducing welding of cast alloy in the mold (soldering) and in the reduction of erosion [75]. Several studies have indicated that PVD coatings improve material properties when combined with nitriding [2, 57, 59, 66, 67].

Surface treatment by multilayer deposition has also shown some benefits. AISI H13 steel coated with a triple layer (oxide / TiAlN / Ti) showed that the outer layer was effective in reducing cracks during thermal cycling. The results showed the importance of the adequacy of the behavior of the substrate with the coating [75].

Another coating process that has been studied is aluminizing combined with post-oxidation. According to a recent study, surfaces treated by aluminizing and post-oxidation exhibited improved thermal fatigue properties. When compared to the untreated surfaces, the thermal fatigue cracks of the aluminized and post-oxidation treatment samples, in addition to having a lower propagation rate, took a longer time to nucleate [76].

2.6.4 Effects of duplex coating

CVD and PDV coatings have failed to improve thermal fatigue strength in steels for hot working of dies. These coatings fail prematurely due to thermal cycling [75]. Among coating possibilities, duplex treatment approaches have shown some benefits. Duplex coating is an optimal combination between surface treatments of plasma nitriding followed by PVD coating or PACVD. Several studies have demonstrated the potential of duplex processes [57, 73, 77, 78]. PVD coatings combined with nitrided layers results in a mutual and synergistic combination [57, 78]. This is because the nitrided layer between the PVD coating and the tool steel increases the surface hardness and its resistance to plastic deformation, protecting the coating against loss of cohesion and adhesion to the base material [57]. In addition, the nitrided layer may further increase the low cycle fatigue strength. The PVD coating, in addition to reducing the wear of the matrices, also plays an important role as an insulation layer for the nitrided base, reducing the influence of external factors on its wear process [2].

The application of the nitrided layer / coating composition by PVD to neutralize the process of plastic deformation, oxidation, erosion or abrasive wear has been confirmed [21, 66, 67]. One study showed that the Nitriding / TiN coating applied on AISI H13 steel was able to inhibit thermal fatigue. The mechanism most likely involved increasing thermal fatigue by retardation of crack nucleation and its growth [2]. In another study performed with AISI H13 steel coated with various nitride layer combinations with PVD coatings, it was shown that the low thermal conductivity of the PVD coating plays an important role in reducing thermal shock intensity during thermal fatigue in addition to effectively decreasing the intensity of the substrate tempering [57]. In another study involving the combination of nitriding with plasma by a PACVD process, it was found that the nitride layer matrix improved the stability of the thermal stress [73].

In general, the improvement in the thermal fatigue strength is closely related to the higher hot-hardness of the substrate and to the high residual compression stress of the coating, thereby producing a delay in cracking.

The difference in the coefficient of thermal expansion between the coating and the substrate is one of the causes of coating failure. This imbalance allows the brittle coating to be placed in a state of stress during thermal cycling resulting in cracking nucleation [63]. Another relevant factor to be observed is the temperature involved in the process since the low thermal stability of the compound layer may cause oxidation of the iron nitrides with subsequent fragmentation of the PVD coating. This suggests the need to remove the compound layer in applications where the temperature and atmosphere cause oxidation [63]. In a study of thermal fatigue in AISI H11 steel with PVD coating, it was shown that after 120 thermal cycles, in the same temperature range, a complete oxidation- induced pickling of the samples occurred. This shows that high oxidation resistance of coatings becomes a fundamental requirement for applications involving thermal fatigue in aggressive environments [21].

It is important to consider the possibility that coating failures are associated with other specific sources. The cause of the failures may be attributed, to the inclusion of sulfur on the surface of the matrix since this element may be present in the grinding stones used in the final polishing operations of the matrices. In this case, the rough surface produced at the end of the die manufacturing operation immediately prior to grinding may exhibit a significant influence on the failure mechanism since it can provide locations for trapping abrasive particles with high sulfur content. Thus, a poor coating adhesion to the sulfur inclusion can occur, leading to its detachment. Thus, without the coating, the surface of the substrate is exposed favoring crack nucleation [79].

It is also important to note that all types of surface treatment and coating technologies can form a compound layers on the surface. Therefore, it is always important to consider the thermal property difference between these compound layers and the substrate can lead to a stress concentration which reduces the thermal fatigue resistance of the tool [76].

2.7 Effects of the deep cryogenic treatment

Deep Cryogenic Treatment (DCT) is a supplementary process to conventional heat treatment. This process typically consists of slowly cooling a mass of parts to -196 ° C, keeping them at this temperature for hours or tens of hours, and then slowly heating until they reach room temperature again [80-83]. Unlike conventional surface treatments, it is a one-step treatment, and it affects the properties of the component's core [84]. According to the literature on DCT in tool steels, improvement of mechanical properties can be attributed to various phenomena including: reduction or elimination of retained austenite from the quenching process, precipitation of finely dispersed small carbides in martensite, and the removal of residual stresses [83, 85-87]. DCT causes a permanent change in the kinetics of carbide precipitation causing the material to possess a higher volume fraction of carbides than a material that has been subjected to only conventional heat treatment and tempering. In addition, the martensite phase will consequently exhibit a lower carbon content giving rise to greater toughness [72, 81, 87]. Studies involving DCT showed a significant increase in the wear resistance of AISI H11 steel at elevated temperatures. However, the DCT soaking time at temperature of DCT is fundamental [81]. Although the effects of DCT on the material properties, few studies have reported the effects of DCT on the thermal fatigue of tool steels for hot work. One of the few papers reporting the effects of DCT on hot working (AISI H11) thermal fatigue properties reveals that cryogenic treatment may delay the crack nucleation process without increasing its propagation. However, the study observed that the mean length and maximum crack length remained practically unchanged for the test specimens with and without DCT [88].

Although a greater amount of data is needed to evaluate the direct effects of DCT on thermal fatigue resistance, it is possible to correlate other more frequently investigated properties such as toughness and hardness which are desirable elementary properties to obtain thermal fatigue resistance. Studies have shown that toughness is considerably affected by DCT while hardness is minimally affected. Studies involving AISI H13 steel subjected to conventional tempering and cryogenic bath annealing showed that the material improved the impact energy by 20% when compared to non-cryogenic treatments. On the other hand, the cryogenic treatment did not influence the hardness [89].

The performance of different thermal treatments combined with DCT has been investigated. The study analyzed AISI H13 subjected to a combination of DCT with two different tempers (gas and oil). The results showed that, in both gas tempering and quenching tests, combined or not, with DCT, the effects were not significant for the change in mechanical properties of traction and hardness. However, with the application of DCT, the tenacity of AISI H13 steel increased considerably. In the treatments with cryogenic stages for gas and oil tempers, tenacity increased 22.5% and 24%, respectively, when compared to non-cryogenic treatments [87]. Another investigation was conducted to study the effects of DCT on the hardness and wear behavior of AISI H13 steel coated with TiC layer and without this coating. The study revealed that the DCT applied to the uncoated material resulted in a 5.8% increase in hardness and 33% in wear resistance compared to conventional heat treatment. When DCT was applied to the coated material, the values increased to 9.6% for hardness and 60% for wear resistance [85].

2.8 Effects of surface roughness

There are several published works addressing the effects of surface roughness on the performance of materials, however, there is no direct correlation of the effects of roughness with the thermal fatigue of tool steels for hot work. However, it is possible to correlate the influence of roughness with other more frequently investigated properties, such as fatigue, coating adhesion, erosion resistance, corrosion resistance and welding of the molten alloy in the mold (soldering), which are properties that Influence thermal fatigue. Welding fractions of the aluminum alloy on the surface of the tool, for example, produces a living corner that facilitates cracking. The rough surface exerts a great effect on the fatigue strength, since the cracks start predominantly on the free surface of the material. Studies have shown that a surface with less roughness has a greater resistance to fatigue, while the surfaces with greater roughness show relatively a lower life in fatigue. In this way, the polishing of the surfaces can increase the life in fatigue of components [90].

The performance of the tool coatings is affected by the rough surface of the substrate, so that the decrease in roughness results in an improvement of the mechanical and adhesion properties of the coatings. Studies involving Ti / TiN / Zr / ZrN multilayer coatings applied to substrates with different surface roughness have indicated that the corrosion resistance of the coating can be decreased with increasing substrate roughness. In addition to altering the corrosion resistance, it was further observed that the erosion resistance and the adhesion strength of the coating increased with the decrease of the roughness of the substrate. In this case, the high adhesion strength and the strong interfaces between adjacent layers were able to reduce crack nucleation and its propagation, inhibiting coating faults [91].

It has been observed in previous studies that the adhesion of the PVD coating also depends on the pre-treatment of the substrate. A ground surface, for example, exhibits better adhesion of the coating than surfaces blasting with micro-abrasives, as the adhesion of the coating is lower with a higher substrate roughness, especially on blasted surfaces [92].

Another study relating surface roughness and corrosion resistance also indicated an improvement in corrosion resistance for a low substrate roughness. The study used substrates with varied roughness (between 0.2 μm and 17 μm), covered by Ti and TiN coatings. The results indicated an increase in corrosion resistance for a roughness (Rmax) of less than 1 μm for Ti films, and less than 3 μm for TiN film [93].

The tribological properties are influenced mainly by the rough surfaces of the material. These properties in some form, direct or indirect, are also related to thermal fatigue. Thus, it is expected that the decrease in surface roughness, as with tribological properties, will also be able to increase the resistance of the tools to thermal fatigue. However, further studies are needed to confirm the direct correlation between surface roughness and thermal fatigue.

3 Conclusions

Thermal fatigue is a primary failure mode in tools and dies for hot work. Failures originate in the surface layer of the material and influence the productivity and quality of the surface thereby reducing the useful life. Thus, for higher durability tools and dies, it is desirable that better properties are created in the surface layers.

The heat treatments of quenching and tempering are typical in hot work steel. Different austenitizing temperatures can result in large differences in thermal stability of the material.

The tempering temperature influence on secondary carbides engaged are a primary influence on hardness and thermal stability and the volume fraction of small precipitates directly influences mechanical resistance to high temperatures.

Although nitriding can improve various surface properties, to improve the resistance to thermal fatigue requires caution since, the compound layers generated during nitriding can negatively affect in-service conditions. The negative aspects of nitriding on thermal fatigue can be reduced by using plasma nitriding. Low-pressure nitriding can promote a high performance thermal fatigue-related crack density.

The potential of the duplex (nitriding + PVD) process to inhibit thermal fatigue is related to high residual compression stress of the coating and higher hot-hardness of the substrate. Thermal fatigue properties have also been improved on surfaces treated by the combination of aluminizing and oxidation but these studies are relatively limited.

Nitriding accompanied by post-oxidation produces an improvement in wear and corrosion resistance, however, the influence of post-oxidation on the thermal fatigue is still not well established.

Limited work has been reported on the effect of DCT on thermal fatigue properties but there is evidence that the cryogenic treatment can delay crack nucleation without increasing crack propagation.

There are different thermal fatigue tests, but the lack of standardization has hampered comparison between results therefore, results should be carefully compared based on specific test and material conditions. In addition, care is needed when laboratory tests results are intended for real applications, especially where mechanical and chemical loads are present. Simulation of in-service conditions has some limitations; therefore, direct field testing in service is preferred.

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Boriding of AISI 440B Stainless Steel and Coating Characterization

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Abstract

The AISI 440B (DIN 1.2210, X90CrMoV18) steel is one of the hardest among martensitic stainless steels. This type of steel is used in a variety of industrial applications where wear and corrosion are determinant, such as molds, parts and tools for the automotive and biomedical industries. Their superior mechanical properties are due to its high carbon (0.75-0.95 % C) and chromium (16-18% Cr) contents. Suitable coatings can increase wear resistance and expand these materials usability range. Boride coatings, with their high hardness and wear resistance are good candidates for this purpose. Boride layers were obtained by boriding treatment in a salt bath (a mixture of sodium borate and aluminum). The layer properties, such as hardness, thickness, layer/substrate interface morphology and phases formed are influenced by steel composition. In this work, the layers produced on AISI 440B steel were harder, thinner, with a smoother interface when compared to plain carbon steels due the larger amount of alloying elements. In order to evaluate mechanical properties of borided layers in samples of stainless steel AISI 440B, Optical Microscopy (OM) microstructural analysis, Vickers microhardness tests and micro-adhesive and micro-abrasive wear resistance tests were performed. The layers produced exhibited a hardness close to 2250 HV and excellent wear resistance far superior to that of substrate.

Introduction

Surfaces of tools and mechanical components are often subjected to static and cyclic stresses, friction, wear and corrosive environments. Thus, in order to prevent failures and increase the useful life of these engineering materials, the development and application of surface modification processes is very important from an economic and performance point of view [1]. Among the various processes used to improve the surface resistance of steels, boriding stands out for its simplicity and production of layers with abrasion and friction resistance greater than other thermo-chemical treatments such as carburizing and nitriding [2].

In the boriding treatment, boron atoms diffuse into the substrate at high temperatures then reacting with the base metal to form borides. In ferrous alloys, the layer produced generally consists of two sublayers; the first one consisting of Fe_2B adjacent to the substrate, followed by FeB in the upper portion of the layer [3]. Several methods can be used for this treatment, gaseous substances such as diborane or boron halides, liquid media such as borax or solid boriding agents [4]. Layer thicknesses are determined by temperature and time of treatment. Layer hardness may be 1650-2000 HV which is in the range of low alloy steels [5, 6] and above 2100 HV in chromium-alloyed steels [7]. Boriding can be applied on materials including: carbon steels, low alloy steels, tool steels, nickel and cobalt alloys, molybdenum, tungsten, niobium and titanium. [8-13].

AISI 440B is a martensitic stainless steel with high hardness and is used in a variety of industrial applications where wear and corrosion resistance are necessary such as molds, parts and tools for the automotive and biomedical industries [14]. The excellent mechanical properties of this steel are attributed to its high carbon (0.75-0.95% C) and chromium (16-18% Cr) contents. However, for hard tribological applications, the properties of AISI 440B may not be sufficient to meet performance requirements requiring the use of surface layers with appropriate characteristics [15].