COMPARISON OF THERMAL FATIGUE BEHAVIOR OF PLASMA NITRIDING WITH COMPOUND LAYER AND WITHOUT IT OF H13 STEEL

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Abstract

With the Uddeholm self-restricted method, the effect of compound layer of plasma nitriding on thermal fatigue behavior of H13 steel was studied by the way of adding Ar during plasma nitriding to remove compound layer. The results show that the compound layer of plasma nitriding can delay the nucleation of heat crack and hold back the propagation of heat crack from surface to substrate to some extent because of its high hardness and strength. On the other hand, the heat checking expands faster with compound layer on the surface than that without it. After 3000 cycles of thermal fatigue test, both heat cracks with the compound layer are wider than the latter ones and the number of heat crack of the former is more from the view of crosssection. Otherwise, the X-ray residual stress analysis results display that the compressive stress of conventional plasma nitriding specimen with the compound layer is higher than that of without it, but it descends more rapidly than the latter one’s during thermal fatigue test.

Keywords: H13 steel, plasma nitriding, compound layer, thermal fatigue behavior, residual stress

INTRODUCTION

AISI H13 steel is one of the most popular hot working die steels. As a major casting die material of aluminum die, it has to endure thermal and mechanical impact of molten aluminum at elevated temperature, which results
in failures such as: heat checking; corrosion or soldering, erosion wear by molten aluminum and catastrophic failure [1], in which, 60% failures result from heat checking [2].

Plasma nitriding treatment can not only improve surface strength of casting dies and enhance their wear resistance, erosion resistance and soldering resistance but also retain the strength and toughness of their cores. There is no doubt that the compound layer of plasma nitriding plays a very important role during service since it has the advantages of hardness, erosion resistance and soldering resistance to the liquid aluminum. However, the effect of the compound layer on thermal fatigue behavior is uncertain: on one hand, the toughness of $\varepsilon$-phase ($\text{Fe}_{2-3}\text{N}$) of the compound layer is low, whilst its expansion coefficient is high, which increases the thermal stress of casting dies during service alternately heated and cooled and promotes nucleation and growth of heat checking; on the other hand, that the strength and corrosion resistance of the compound layer inclines to alleviate wear, erosion and soldering would produce a delay in crack nucleation and growth, which benefits to thermal fatigue resistance of H13 steel [3, 4, 5, 6, 7].

In this paper, the Uddeholm self-restricted thermal fatigue experiment was employed to investigate the effect of the compound layer of plasma nitriding on thermal fatigue behavior of H13 steel by the way of adding Ar during plasma nitriding to remove the compound layer and comparing with the conventional plasma nitriding.

**EXPERIMENTAL DETAILS**

**MATERIALS AND HEAT TREATMENTS**

For the investigation, the test samples were taken from the premium quality AISI H13 steel block, which were electro-slag-remelted, multi-direction-forged and extra-refined, whose chemical composition is shown in Table 1. Thermal fatigue test samples were the standard size of the Uddeholm self-

<table>
<thead>
<tr>
<th>C</th>
<th>Si</th>
<th>Mn</th>
<th>Cr</th>
<th>Mo</th>
<th>V</th>
<th>P</th>
<th>S</th>
</tr>
</thead>
<tbody>
<tr>
<td>0.42</td>
<td>0.98</td>
<td>0.30</td>
<td>4.93</td>
<td>1.40</td>
<td>0.87</td>
<td>0.018</td>
<td>0.005</td>
</tr>
</tbody>
</table>
restricted method (Fig. 1), acquired a hardness of 47 HRC after the treatment of 1020 °C vacuum quenching and double tempering at 610 °C. Before plasma nitriding treatment, the specimens were gradually ground with sandpaper and polished with diamond paste to avoid cracks prefabricated. The size of the plasma nitriding treatment samples were 14 mm × 9 mm × 7 mm.

![Figure 1. Size of the thermal fatigue samples.](image)

**PLASMA NITRIDING TREATMENT**

Plasma nitriding treatment processes are listed in Table 2.

<table>
<thead>
<tr>
<th>Code</th>
<th>Plasma nitriding treatments</th>
</tr>
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<tbody>
<tr>
<td>A</td>
<td>Conventional plasma nitriding</td>
</tr>
<tr>
<td>B</td>
<td>Adding Ar during conventional plasma nitriding</td>
</tr>
</tbody>
</table>

**THERMAL FATIGUE TEST**

With the Uddeholm self-restricted method, a specimen was cycled in a high frequency induction heating position and a water shower from 18 °C to 700 °C. The heating and cooling time, controlled by Single Chip Micyoco (SCM), lasted 3.6s and 8s respectively. A, B samples were subjected to 3000 cycles.
OPTICAL MICROSCOPE OBSERVATION AND VICKERS MICRO-HARDNESS TEST

Microstructure of the nitrided layer was observed by Neophet-2 Optical Microscope. And the surface and cross sectional cracks were examined by 4X Optical Microscope. Vickers micro-hardness of the nitrided layer was measured by HX-1000 Micro-sclerometer, before and after testing.

MEASUREMENT OF X-RAY RESIDUAL STRESS

The residual stresses of the nitrided layers before 600 cycles were measured by PSP/MSF X-Ray Residual Stress Analyzer and obtained from the following equation 1 and 2:

\[
\sigma = - \frac{E}{2(1 - \nu)} \cot \theta_0 \cdot \frac{\Delta 2\theta}{\Delta \sin^2 \psi} \quad (1)
\]

\[
\sigma = - K \cdot M \quad (2)
\]

where, \(\sigma\) is the surface residual stress, \(E\) is modulus of elasticity (205.8 GPa), \(\nu\) is Poisson ratio (0.28), \(\theta_0\) is the diffraction angle of \(\alpha\)-Fe\{211\}, \(\theta\) is the diffraction angle of the samples; \(\psi\) is the angle formed by the sample surface normal line. \(K\) is the Stress Constant (-317.9 MPa/deg), \(M\) is the slope of \(2\theta - \sin^2 \psi\).

The residual stress of the nitrided specimens was measured by a X-ray method and was analyzed by \(\alpha\)-Fe\{211\} diffraction, not by Fe\(_2\sim3\)N diffraction due to nitrides because nitrides disappear and decompose by heating during thermal fatigue test [8].

RESULTS AND DISCUSSION

RESULTS

Metallographical Observation. After plasma nitriding treatment, the surface micro-hardness and compound layer were measured in Table 3 and the microstructure of the nitrided layers were shown in Fig. 2. Table 3 showed that the surface hardness of A was higher than that of B and correspondingly, compound layer was present on A sample in Fig. 2. The difference of micro-hardness gradient between A and B before thermal cycle test was also presented in Fig. 5.
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Table 3. Surface hardness plasma nitrided layer

<table>
<thead>
<tr>
<th>Code</th>
<th>Surface hardness (HV$_{0.05kgf}$)</th>
</tr>
</thead>
<tbody>
<tr>
<td>A</td>
<td>1138</td>
</tr>
<tr>
<td>B</td>
<td>965</td>
</tr>
</tbody>
</table>

Figure 2. Microstructures of the nitrided layers.

After 100 cycles, an oxidation layer appeared to some extent on the surface of each sample. But there was no apparent heat checking on A sample’s surface. After 600 cycles, heat checking of B samples had grown up, at the same time, there were leading cracks which were sparse on A. After 1000 cycles, all the cracks grew and their width enlarged. Crack morphology of A was not changed, while cracks in sample B conglomerated and leading cracks became prominent. The heat checking is shown in Fig. 3, after 600 and 3000 cycles.
Figure 3. Surface heat checking after 600 cycles and 3000 cycles.
The cross-section view of heat cracks was shown in Fig. 4 after 3000 cycles and hardness gradient of nitrided layer of specimens after and before thermal fatigue test was presented in Fig. 5.

The cracks developing in the depth, shown in Fig. 6, were studied and counted under microscope at magnification 100×. It was obviously that A’s short cracks between 0.013–0.067 mm were more than B’s, only one long crack penetrating the residual nitrided layer whose length was more than 0.13 mm, which was much less than B’s.

**Residual stress analysis.** Figure 7 presented the axial residual stress changed with thermal fatigue test before 600 cycles. It showed that the initial compressive residual stress of A was much higher than that of B due to compound layer. But it descended more rapidly than that of B.

**DISCUSSION**

**Micro-hardness and crack morphology analysis.** From the observation above, heat checking nucleation of A sample with the compound layer was delayed compared with the other samples. It can be inferred as follows: with the high frequency of induction heating, the heating area was concentrated on the surface layer of the samples [9], where the thermal stress of outmost surface of the specimens was top high. On the basis of the thermal stress of cylinder at axile symmetry, finite length and free ends [10], supposing that τ is the temperature along the radius. Depth of the cylinder is the average penetrating thickness of the whirlpool in heat and cool states at the frequency: ra is radius of inner surface layer, ra = 4.5 mm; rb is radius of outer surface layer, rb = 5.0 mm. With cylindrical coordinates, thermal stresses (σ_ττ, σ_θθ, σ_zz) in three dimension can be deduced respectively as in the expressions in equations (3) – (5):

Thereinto: α – thermal expansion coefficient; E – modulus of elasticity; ν – Poisson ratio

\[
\sigma_{\tau\tau} = -\frac{E\alpha(\tau_a - \tau_b)}{2(1-\nu)} \cot \left[ \frac{\ln(b/r)}{\ln(b/a)} - \frac{(b^2/r^2) - 1}{(b^2/a^2) - 1} \right] \tag{3}
\]

\[
\sigma_{\theta\theta} = -\frac{E\alpha(\tau_a - \tau_b)}{2(1-\nu)} \cot \left[ \frac{\ln(b/r)}{\ln(b/a)} + \frac{(b^2/r^2) + 1}{(b^2/a^2) - 1} \right] \tag{4}
\]
Figure 4. Cross-sectional views of heat checking after 3000 cycles.

Figure 5. Hardness gradient of nitrided layers of specimens before and after thermal fatigue test.
\[
\sigma_{zz} = -\frac{E\alpha(\tau_a - \tau_b)}{2(1 - \nu)} \cot \left[ \frac{2\ln(b/r) - 1}{\ln(b/a)} + \frac{2}{(b^2/a^2) - 1}\right]
\] (5)

According to the references of AISI H13 steel from Uddeholm AB, Swedish Institute for Metals Research, and ASM [11, 12, 13], the data were gotten: \(\alpha = 13.1 \, \mu m/mK, \nu = 0.29, E_{room\, temp} = 202.5 \, GPa\) and from [14], \(E_{700\, ^\circ\, C} = 160 \, GPa\). Take the data above into equations (3) – (5), and then we can get the surface thermal stress at 700 \(^\circ\)C when a specimen was heated: \(\sigma_{\theta\theta} = \sigma_{zz} = -976.2 \, MPa, \sigma_{rr} = 0\); at room temperature when a specimen was cooled, \(\sigma_{\theta\theta} = \sigma_{zz} = 1399.3 \, MPa, \sigma_{rr} = 0\). Thus it can be seen that the thermal fatigue samples endure tensile stress when cooled, while compressive stress when heated. As we know from references [12, 13]: at 700 \(^\circ\)C, the yield strength \(\sigma_{0.2}\) of H13 steel is 430 MPa, while at room temperature, the tensile yield strength of it is 1200 MPa. Therefore, the compressive and tensile stresses which the thermal fatigue samples bear exceed the yield strength of H13 steel when they were heated and cooled alternatively. However, the tensile strength of the compound layer can be reckoned from the tensile strength of it [15]: \(\sigma_b = 8.61 \times 10^4/(100-HRC)\) (MPa), then \(\sigma_b\) is almost 3000 MPa. For the compound layer’s brittleness, it can be drawn that the yield strength of the compound layer is close to its tensile strength and that the thermal stress is less than the yield strength of it. And then, at the beginning of the test, the compound layer could protect the surface of the samples by postponing the nucleation of the thermal cracks. Along with the circulation of cooling and heating, the more cycles, the more fatigue damages on the surface of samples. At last, the compound layer crazed because of its brittleness.

Figure 3 showed that heat checking of A was sparser than that of B sample, distortion on the surface was less than that on B samples. The long and straight cracks of A displayed that the propagation of it was faster than that of B samples. The phenomena that the average width of cracks of A was larger than that of B, Fig. 3, and the cracks’ morphology A was not changed from 600 cycles to 3000 cycles, Fig. 3, could be explained by the brittleness of the compound layer. The higher elastic energy spent for their nucleation, the lower the energy available for the propagation [6].

The brittleness of the compound layer could not make the cracks close when the stress relaxes at the tip if cracks. As a result not only cracks would not vanish once they nucleate but also their morphology would not damage. Furthermore, the number of cracks increases with the cycles.
is evident that with the increasing of repeatedly heating and cooling, the toughness of the compound layer cannot satisfy the deformation at request of the substrate; only to widen the cracks and increase the number of cracks does the compound layer correspond with the substrate to deform in-phase.

Figure 3 showed that after 3000 cycles, the compound layer of A had dissolved and most of the cracks of it were within the nitride diffusion layer, whereas most of the cracks of B were longer, penetrating the diffusion layer. It could be deduced from Fig. 4: though the compound layer of A had dissolved, its hardness of sub-surface was higher than that of B. To some extent, the compound layer could prevent the cracks propagation to the substrate. No sooner did the cracks penetrate the diffusion layer, than they grew faster for the sake of stress concentration.

**Residual stress analysis.** Figure 7 showed that axial compressive residual stress changed with thermal fatigue test before 600 cycles. As expected, the initial compressive residual stress of A was much higher than that of B due to the compound layer.

With the increasing of cycles, the compressive residual stress of A drops abruptly, in a sharp contrast, the compressive residual stress curve of B is flat and smooth. This phenomenon demonstrates that the residual stress of the nitrided layer of A is unstable [16] owing to the dissolution of the compound layer under the adopted experimental conditions before 600 cycles. Otherwise, due to error of the residual stress measurement, the point of intersection of the two curves is less than 100 cycles, Fig. 7; however, before 75 cycles, the compressive residual stress of A is higher than that of B. Accordingly, at the first of the cycling, the compound layer is able to protect the surface of A sample and the number of heat checking of A is less than that of B.

In a summary, there is duplex effect of the compound layer on the thermal fatigue behavior. On one hand, the compound layer can produce a delay in crack nucleation. After 3000 cycles, the compound layer can prevent the propagation of cracks to some extent. On the other hand, in service condition, the compound layer has to contact with aluminium liquid and endures both thermal and mechanical stresses, the advantages, such as wear resistance, erosion resistance and soldering resistance, of the compound layer would be counteracted by its spallation because of its wide, long and straight cracks. As a result, it would be cautious of employing the compound layer on aluminium die casting die of H13 steel.
SUMMARY

In the present work thermal fatigue tests have been carried out on plasma nitrided H13 hot work die steel. The role of the compound layer of plasma nitriding has been studied. Under all the adopted experimental conditions:

1. The compound layer produces a delay in thermal crack nucleation and prevents the cracks propagation to the substrate to some extent since the thermal stress is less than the yield strength of the compound layer.

2. The brittleness of the compound layer makes the thermal cracks wider, longer and more straight than that without it. And the cracks cannot close as well as those without the compound layer. From this point of view, it plays a negative role during the in-service conditions.

3. The compressive stress of conventional plasma nitriding specimen with the compound layer is higher than that of without it, but it descends more rapidly than the latter one’s during thermal fatigue test.

4. It would be cautious of employing the compound layer on the aluminium casting die of H13 steel.

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REFERENCES


Figure 6. Statistics of the cracks on the different samples after 3000 cycles of thermal fatigue test.

Figure 7. Relationship between axial residual stress and thermal fatigue test cycles before 600 cycles.