Protection of Crack Generation by Functionally Graded Layer at the Interface of Hard-facing Iron Alloys*1

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The protection of crack formation by a functionally graded layer at the interface of surface-hardened iron alloys has been investigated using a modified reactive diffusion process which consists of the two-stage diffusion heat-treatment for diffusion species. From the stress analysis on thermal shock test and the microstructure observation, it was known that cracks formed on the thermal shock test were onion cracks arising from a radial stress σr and radial cracks due to a circumferential stress σθ. In the specimens subjected to the modified reactive diffusion heat-treatment, a functionally graded layer was formed at the interface between the surface carbide layer/iron substrate, which effectively suppressed the generation of cracks by shifting a location of the maximum tensile stress toward the substrate and relaxing a stress concentration at the interface.

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1. Introduction

In the metal/ceramics bonding and the ceramics coating on metallic materials, a significant difference in thermal expansion between metals and ceramics causes thermal stress at the metal/ceramics interface which may result in a poor interface strength and interfacial cracking. Such an imperfect interface bonding leads to a reduction in mechanical properties and the reliability of the products. Hitherto, many works on the reactive diffusion method for wear resistant surface modification have been reported, where a carbide forming element, such as chromium, titanium and vanadium, diffuses from the surface of a steel and reacts with carbon in the steel to form a carbide on the surface.1–3) However, the diffusivity of carbon is much greater than carbide forming elements in steels, and hence carbides formed on the surface have a clear interface with the substrate. On cooling from the process temperature to ambient one, a large thermal stress would be generated at the interface, possibly causing interfacial cracking. In a modified reactive diffusion process, considering the difference in the diffusivity of diffusion species and employing an iron intermediate layer as a diffusion media, a carbide forming element diffuses in the iron intermediate layer to build a gently graded concentration profile during the first-stage heat-treatment performed at higher temperatures, and then a diffusion bonding process (the second-stage heat-treatment) is performed at lower temperatures where main diffusion species is carbon. A functionally graded (F.G.) distribution of a carbide is formed at the carbide/substrate interface by the “two-stage diffusion process” and such a F.G. layer is expected to suppress thermal stress generated at the surface coating/substrate interface in coated tools and dies suffered from a thermal cycle. In our earlier works,4,6) the two-stage diffusion process to built a F.G. distribution of titanium, vanadium and chromium carbides at the carbide/substrate interface has been proved to be effective to suppress interfacial cracking by diminish a stress concentration at the interface. However, the mechanism for the protection of cracking by the F.G. layer has not been clarified.

In the present work, the effect of F.G. layer on the formation of cracks in the surface chromium carbide layer of hard-facing iron alloys has been investigated on the basis of thermal shock test and stress analysis on quenching.

2. Experimental

The interface modification process with a F.G. layer introduced in the present work consists of the two-stage diffusion heat-treatment: the first-stage diffusion process where chromium diffuses in the iron intermediate layer and the second-stage diffusion bonding process where chromium in the iron intermediate layer reacts with carbon which diffuses from the steel substrate to form chromium carbide. In an earlier work,6) it has been suggested that there is a critical thickness of the F.G. layer to suppress the generation of cracks and that the control of the F.G. layer thickness is possible by choosing a suitable condition in the first-stage diffusion process, since the thickness of F.G. layer coincides with the diffusion distance of chromium in the first stage diffusion heat-treatment.

In the present work, the specimens with different thickness of the F.G. layer were prepared by changing the holding time in the first-stage diffusion heat-treatment. The sandwich specimens of pure iron foils with 150, 250 and 500 μm thick coated with a chromium-plated layer with 90, 100 and 200 μm thick, respectively, were set between the upper and lower tool steel rods in the vacuum furnace shown in Fig. 1a. The load applied on the specimen was 30 N, the sum of the tool steel rod and the weight. The first-stage diffusion heat-treatment was carried out under argon gas atmosphere at 1273 K for 3.6–18 ks followed by furnace-cooling (Fig. 1b). The second-stage heat-treatment for carbon diffusion and diffusion bonding of the first-stage sandwich specimens with...
a tool steel 3 mm thick was performed at the same temperature for 3.6 ks followed by furnace cooling. For comparison, a sandwich specimen, Cr-plated iron foil/SK3 tool steel ($t = 3$ mm) and Cr-plated SK3 tool steel ($t = 3$ mm) specimens were subjected to the heat-treatment at 1273 K for 7.2 ks, as shown in Fig. 1c (Simultaneous D.B. specimen) and Fig. 1d (Direct D.B. specimen), respectively.

Thermal shock test was performed to examine the effect of F.G. layer to suppress cracking. After holding 0.6 ks at 1273 K, the three kinds of specimens subjected to the two-stage D.B., Simultaneous D.B. and Direct D.B. heat-treatment were quenched into iced water. The microstructures on the vertical section and in the surface carbide layer were examined by optical microscope observation. The stress analysis during quenching was carried out using a finite element commercial program MARC.

3. Results and Discussion

Microstructures on the section of the specimens subjected to direct D.B., simultaneous D.B. and two-stage D.B. heat-treatments are shown in Fig. 2. In the two-stage D.B. specimens, F.G. layers about 15 $\mu$m thick (Fig. 2c) and about 50 $\mu$m thick (Fig. 2d) are observed inside the surface chromium carbide layers 15–20 $\mu$m thick. A thinner F.G. layer about 5 $\mu$m thick is formed in the simultaneous D.B. specimen and no F.G. layer is seen in the direct D.B. specimen. Many cracks shown by arrows are observed in the direct D.B. specimen, while the number of cracks is significantly reduced in the specimens with F.G. layer, especially in the two-stage D.B. specimens having a wider F.G. layer. The microstructure on the horizontal section of the surface chromium layer is shown for the direct D.B. specimen in Fig. 3. It is known that cracks formed on the
thermal shock test are radial cracks and onion ones. From the morphology of the cracks, it is suggested that the radial cracks are generated by a circumferential tensile stress $\sigma_\theta$ and the onion cracks by a radial tensile stress $\sigma_r$. To elucidate the effect of F.G. layer to suppress a crack generation, stress analysis was performed using meshed elements given in Fig. 4, where the F.G. layer was assumed to consist of four layers 25 $\mu$m thick, having a stepwise decrease in the fraction of the chromium carbide. The materials’ properties used for the stress analysis are given in Table 1. The results of stress analysis are shown in Figs. 5 and 6. Figure 5 shows variation of tensile stresses, along the radial direction $\sigma_r$. 

Fig. 3  Microstructures of the surface carbide layer for Direct D.B. specimen subjected to thermal shock test.
along the circumference $\sigma_\theta$ and along the thickness direction $\sigma_z$ on the edge of the surface carbide layer (A to D), as a function of time. A large tensile stress is generated just after quenching into iced water and then reverses to compressive stress. Since the stress $\sigma_z$ is negligibly small compared to $\sigma_r$ and $\sigma_\theta$, it is suggested that cracks generated on thermal shock test are radial cracks caused by the $\sigma_\theta$ stress and onion cracks by the $\sigma_r$ stress. A large maximum $\sigma_\theta$ stress generated at the edge of the carbide/substrate interface decreases rapidly from the edge (Location A) to Location B $\rightarrow$ Location C $\rightarrow$ the center (Location D) where the $\sigma_\theta$ stress is equal to the $\sigma_r$ stress. On the other hand, the variation of $\sigma_r$ stress is relatively small and $\sigma_r$ distributes homogeneously on the interface. It is, therefore, indicated that radial cracks tend to form near the circumferential edge on the interface and onion cracks on the whole area of the interface. From the results of stress analysis, cracks observed in Fig. 3 are considered to be initially generated at the interface under these stresses and grow through the carbide layer toward its surface. Figure 6 shows the influence of the thickness of F.G. layer on the stress distribution at the time of 0.09 s after quenching where the maximum tensile stress is generated on thermal shock test. In the case of the specimen without F.G. layer compared to those with F.G. layer, a large stress concentration exists at

**Table 1** Materials' properties used for the stress analysis.

<table>
<thead>
<tr>
<th>Material</th>
<th>$E$ (MPa)</th>
<th>$\nu$</th>
<th>$\rho$ (g/cm$^3$)</th>
<th>$\alpha_T$ (lower: $T &lt; 453 K$, upper: $T &gt; 473 K$)</th>
<th>$\lambda$ (W/m K)</th>
<th>$c_p$ (J/g K)</th>
</tr>
</thead>
<tbody>
<tr>
<td>100% SK3</td>
<td>20.9</td>
<td>62.8</td>
<td>6.8</td>
<td>1.0 x $10^{-5}$</td>
<td>20.9</td>
<td>62.8</td>
</tr>
<tr>
<td>80% SK3, 20% Carbide</td>
<td>23.4</td>
<td>61.9</td>
<td>6.96</td>
<td>1.1 x $10^{-5}$</td>
<td>23.4</td>
<td>61.9</td>
</tr>
<tr>
<td>60% SK3, 40% Carbide</td>
<td>25.9</td>
<td>61.1</td>
<td>7.12</td>
<td>1.2 x $10^{-5}$</td>
<td>25.9</td>
<td>61.1</td>
</tr>
<tr>
<td>40% SK3, 60% Carbide</td>
<td>28.5</td>
<td>60.2</td>
<td>7.28</td>
<td>1.3 x $10^{-5}$</td>
<td>28.5</td>
<td>60.2</td>
</tr>
<tr>
<td>20% SK3, 80% Carbide</td>
<td>31.0</td>
<td>59.4</td>
<td>7.44</td>
<td>1.4 x $10^{-5}$</td>
<td>31.0</td>
<td>59.4</td>
</tr>
<tr>
<td>SK3</td>
<td>33.5</td>
<td>58.6</td>
<td>7.6</td>
<td>1.5 x $10^{-5}$</td>
<td>33.5</td>
<td>58.6</td>
</tr>
</tbody>
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*estimated
the sharp interface between the carbide layer and the steel substrate. The maximum stress reduces gently and the position of the maximum stress shifts toward the substrate with increasing the thickness of F.G. layer. This indicates that cracks observed in the direct D.B. specimen should be caused by a stress concentration at the sharp and uneven interface and that the F.G. layer in the two-stage D.B. specimens suppresses cracking by shifting a large tensile stress to the interface between F.G. layer/iron intermediate layer where a ductile iron matrix reduces the stress generated by plastic deformation. The ductile nature of the F.G. layer decreases with increasing the fraction of brittle carbide, i.e., approaching the carbide/F.G. layer interface. Figure 7 shows the distribution of area fraction of \( M_7C_3 \) carbide and tensile stress distribution of \( \sigma_r \) on the section of the two-stage D.B. specimen having a F.G. layer 50 \( \mu \)m thick. Carbide network (skeleton structure) is formed in the F.G. layer more than the carbide area fraction \( f_{\text{carbide}} \) (considered to be nearly equal to the volume fraction) of 0.5 where ductility may rapidly reduced.4) The \( \sigma_r \) value at the location of \( f_{\text{carbide}} = 0.5 \) amounts to about 500 MPa which is nearly half of the maximum stress (~1000 MPa) generated at the interface between F.G. layer/iron intermediate layer. A deflective strength of TiC–Ni cermet is 1200–1800 MPa7) and that of TiC carbide may be a similar order. The strength of \( M_7C_3 \) carbide is roughly estimated to be of the order of 1000 MPa which is much higher than the \( \sigma_r \) stress less than 500 MPa generated at the locations where \( f_{\text{carbide}} \) is greater than 0.5. In such a condition, crack formation may be extensively suppressed. On the other hand, in the direct D.B. specimen shown in Fig. 8, the maximum tensile stress is generated in the substrate adjacent to the well-defined and uneven carbide/substrate interface. Such a stress concentration promotes crack formation.

4. Conclusions

From the stress analysis on thermal shock test and the microstructure observation, it was known that cracks formed on thermal shock test were onion cracks arising from a radial
stress $\sigma_t$ and radial cracks due to a circumferential stress $\sigma_0$. The functionally graded layer in the specimens subjected to the two-stage diffusion heat-treatment effectively suppresses a generation of cracks by shifting the maximum tensile stress generated during the thermal shock test toward the ductile substrate and relaxing a stress concentration through plastic deformation of the ductile phase.

REFERENCES