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The effect of thermal annealing on the adherence of Al₂O₃ films deposited by LP-MOCVD on several high alloy steels

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Abstract—Thin alumina films, deposited at 280°C on several high alloy steels by low-pressure metal–organic chemical vapor deposition (LP-MOCVD), were annealed at 0.17 kPa in a nitrogen atmosphere for 2, 4, and 17 h at 600 and 800°C. Film adhesion was studied by scanning scratch testing (SST) and Auger electron spectroscopy (AES). The best adhesion properties were obtained with commercial oxide dispersion-strengthened (ODS) high-temperature alloys, especially PM 3030. Among the ‘normally’ high alloy stainless steels, type AISI-321 showed the best adhesion. The other stainless steel–alumina combinations exhibited a reduced critical load, Lc, after thermal treatment. Alumina on ODS alloys exhibited an increased adhesion. AES studies revealed that this increase could be explained by: (1) the presence of sulfur-trapping elements, preventing segregation of sulfur at the interface; and (2) titanium and carbon enrichment at the interface, resulting in an anchoring effect between the oxide and the substrate.

Keywords: Metal–organic chemical vapor deposition (MOCVD); thermal annealing; alumina films; scanning scratch test (SST); adherence; Auger electron spectroscopy (AES).

1. INTRODUCTION

In addition to the chemical properties of thin films which are often used as protective coatings, optical filters, wear resisting coatings, and insulating layers, their mechanical properties are also of major importance. During the last 10 years, more attention has been paid to characterization techniques for determining the mechanical properties of thin films, microhardness, and scratch adhesion test measurements [1]. The adherence between thin oxide films and metallic substrates depends on several material properties, such as the thermal expansion coefficient and Young’s modulus of the film [2]. The presence of stresses, often built up in the film during deposition,
as well as post-deposition thermal treatment may influence the adhesion characteristics [3, 4]. It was previously found that the adherence between thin alumina films and AISI-304 was significantly reduced after thermal annealing at 600 and 800°C for 2, 4, or 17 h [5]. This detrimental effect of annealing on adherence may be explained by an increased thermal stress and sulfur segregation at the metal–oxide interface. In order to investigate the effect of the composition of the underlying substrate, eight different alloys, including four ‘normally’ high alloy steels and four oxide dispersion-strengthened (ODS) high-temperature alloys, were tested. The ODS alloys contain small amounts of a finely dispersed oxide phase, mostly yttria [6]. Quadakkers [6] reported that these alloys are promising regarding selective oxidation of the scale forming elements, scale growth rate, and scale adherence. Improved scale adherence was attributed to the incorporation of yttria in the oxide scale, especially at grain boundaries. Ikeda and Yata [7] gave an excellent overview of several mechanisms of the yttria effect on sulfur segregation.

The aim of the research described in this paper was to investigate the effect of substrate composition on the adherence of thin alumina films deposited by metal–organic chemical vapor deposition. The adhesion characteristics were measured using a scanning scratch tester. The resulting scratch was also observed optically in order to estimate the minimum load at which delamination took place [8]. Segregation and diffusion of elements at the interface were investigated by AES (Auger electron spectroscopy).

2. EXPERIMENTAL

Depositions were performed on several commercially available alloys (Table 1). The specimens (discs: 20 mm in diameter and 1 mm thick) were ground on SiC paper with

<table>
<thead>
<tr>
<th>Alloy</th>
<th>Fe</th>
<th>Ni</th>
<th>Cr</th>
<th>Al</th>
<th>Ti</th>
<th>Si</th>
<th>Mn</th>
<th>C</th>
<th>S</th>
<th>Y2O3</th>
<th>Other</th>
</tr>
</thead>
<tbody>
<tr>
<td>MA 956</td>
<td>Bal.</td>
<td>—</td>
<td>19.8</td>
<td>4.6</td>
<td>0.3</td>
<td>—</td>
<td>0.09</td>
<td>0.04</td>
<td>0.004</td>
<td>0.52</td>
<td>—</td>
</tr>
<tr>
<td>MA 754</td>
<td>0.33</td>
<td>Bal.</td>
<td>19.6</td>
<td>0.2</td>
<td>0.3</td>
<td>—</td>
<td>0.002</td>
<td>0.04</td>
<td>0.0007</td>
<td>0.57</td>
<td>—</td>
</tr>
<tr>
<td>Inc. 800H</td>
<td>Bal.</td>
<td>31.7</td>
<td>19.9</td>
<td>0.38</td>
<td>0.7</td>
<td>0.5</td>
<td>0.7</td>
<td>0.08</td>
<td>0.006</td>
<td>—</td>
<td>—</td>
</tr>
<tr>
<td>AISI-316</td>
<td>Bal.</td>
<td>10.68</td>
<td>16.62</td>
<td>—</td>
<td>—</td>
<td>0.28</td>
<td>1.15</td>
<td>0.044</td>
<td>0.016</td>
<td>—</td>
<td>P: 0.03</td>
</tr>
<tr>
<td>AISI-304</td>
<td>Bal.</td>
<td>8.88</td>
<td>17.39</td>
<td>—</td>
<td>—</td>
<td>0.70</td>
<td>1.27</td>
<td>0.044</td>
<td>0.017</td>
<td>—</td>
<td>P: 0.033</td>
</tr>
<tr>
<td>AISI-321</td>
<td>Bal.</td>
<td>10.13</td>
<td>17.45</td>
<td>—</td>
<td>0.40</td>
<td>0.52</td>
<td>1.55</td>
<td>0.048</td>
<td>0.003</td>
<td>—</td>
<td>P: 0.037</td>
</tr>
<tr>
<td>PM 2000</td>
<td>Bal.</td>
<td>—</td>
<td>20</td>
<td>5.5</td>
<td>0.3</td>
<td>—</td>
<td>—</td>
<td>0.01</td>
<td>—</td>
<td>0.5</td>
<td>—</td>
</tr>
<tr>
<td>PM 3030</td>
<td>Bal.</td>
<td>17</td>
<td>6</td>
<td>—</td>
<td>1.0</td>
<td>—</td>
<td>0.05</td>
<td>—</td>
<td>1.1</td>
<td>—</td>
<td>Mo: 2.0</td>
</tr>
</tbody>
</table>
a final size of 4000 grit, followed by polishing with Al₂O₃ (3 μm) and subsequently cleaned ultrasonically for 30 min in hexane and ethanol. Finally, the samples were immersed in Struer’s etching fluid (5% solution of 3 M nitric acid in ethanol) for 15 min, washed with pure ethanol, and dried in hot air.

Al₂O₃ was deposited by pyrolysis of aluminium-tri-s-butoxide (ATSB, Janssen Chimica) in a low-pressure chemical vapor deposition (LPCVD) system. Details of the experimental procedure have been reported earlier [8]. Standard conditions for a film thickness of 0.5 μm were deposition temperature 280°C, partial pressure of ATSB 3.7 x 10⁻⁴ kPa (ATSB saturation temperature 134°C), reactor pressure 0.17 kPa, and linear gas velocity 2.6 m/s. After deposition, the ATSB flow was stopped, and the furnace was heated at a rate of 5°C/min to the desired temperature. The samples were exposed to a 0.17 kPa nitrogen atmosphere at 600 and 800°C for 2, 4, and 17 h. Following annealing, the temperature was lowered at 5°C/min to below 200°C before the samples were removed from the furnace.

Scanning scratch adhesion measurements were performed with a SST-101 scanning scratch tester developed by Shimadzu. With the SST-101 tester, the adhesion of thin films is evaluated by the load applied with a hard stylus (critical load), corresponding to the site where the film starts to delaminate. A detailed description of the SST-101 is given elsewhere [8]. Specific test parameters were stylus tip radius 25 μm, amplitude 50 μm, indentation velocity 1 μm/s, and stage drive velocity along the y-axis 20 μm/s.

Chemical composition and establishment of the depth profiles were performed by means of a Perkin-Elmer PHI 600 scanning Auger multiprobe. Alternate sputtering and Auger analysis were used for in-depth analysis.

The morphology of the specimens was investigated by means of optical and scanning electron microscopies (JEOL M 35 CF), the latter equipped with an Energy Dispersive X-ray Microanalysis (EDX) system (Kevex Delta — class III).

3. RESULTS

3.1. Scanning scratch tests

Figure 1 shows the critical load (Lc) as a function of the post-deposition annealing time and temperature for thin alumina films deposited on different substrates. The critical loads were obtained from the scratch loading curves corresponding to the position of the first sudden increase of output voltage. For each sample, ten scratches were made so that the average and the standard deviation could be calculated. Optical and scanning electron microscopies revealed that for the nonannealed samples no significant delamination or spallation took place. This type of failure mode is also known as conformal cracking [11]. It occurs when the coating remains fully adherent, despite the elastic and plastic deformation of the coating and substrate induced by the indenter. In this case, only an increase in scatter of the load—output voltage curve was found. It was sometimes hard to detect the transition from complete adherence to conformal cracking of the film. After thermal treatment, the sudden increase in
Figure 1. Critical load ($L_c$) of alumina films as a function of the annealing time and temperature ($\Delta = 600^\circ C; \blacksquare = 800^\circ C$).

Output voltage is much more pronounced, as illustrated in Fig. 2 for an alumina film deposited on AISI-304 without thermal annealing (a) and after annealing for 2 h at 800°C (b). In the case of AISI-304, AISI-321, AISI-316, and Incoloy 800H, the films showed spallation and delamination after reaching $L_c$. All these specimens showed
a reduction in $L_c$ after thermal treatment, except for AISI-321 annealed at 800°C. The ODS alloys showed an increase of the critical load after annealing, depending on the annealing time and temperature. The extent of spallation was also significantly reduced, and in some cases, only plastic deformation of the oxide film was detected. This plastic deformation can be explained by the fact that next to alumina, boehmite was also one of the deposition products [8]. Details of the scratch results are as follows:

AISI-321. The nonannealed specimens showed that after reaching the critical load, only very small particles were removed locally. Therefore it was difficult to determine $L_c$. After annealing at 600°C, adhesion failure was more pronounced and the critical load was reduced. For annealing at 800°C, a clear change from adhesion to de-adhesion of the oxide film was observed. From Fig. 1a it is clear that the critical load, necessary to induce spalling and/or delamination, increased with increasing annealing temperature. The effect of annealing time for both annealing temperatures was insignificant.

AISI-304. The nonannealed specimens exhibited no clear $L_c$. A change in output voltage corresponding to adhesion failure by buckling of the oxide film was used as $L_c$. This change became more pronounced after thermal annealing at 600°C. The
Figure 3. SEM micrographs of the scratch morphology of alumina films on different substrates. (a) AISI-304 (2 h, 600°C); (b) AISI-304 (4 h, 800°C); (c) AISI-316 (2 h, 600°C); (d) AISI-316 (4 h, 800°C).
Effect of thermal annealing on the adherence of \( \text{Al}_2\text{O}_3 \) films

Figure 3. (Continued) (c) PM 3030 (2 h, 600°C); (f) PM 3030 (4 h, 800°C); (g) MA 956 (2 h, 600°C); (h) MA 956 (4 h, 800°C).
critical load decreased with longer annealing times and a higher annealing temperature (Fig. 1b). Figures 3a and 3b show that after reaching the critical load, the delamination of the oxide film was more pronounced for the specimen annealed for 4 h at 800°C than for the specimen annealed for 2 h at 600°C.

**AISI-316.** $L_c$ for the nonannealed sample was comparable to that measured for AISI-304 (Fig. 1c). Adhesion failure was manifested by the removal of small oxide particles. After thermal annealing at 600°C for 17 h and at 800°C for 2 h or longer, the critical load decreased to about 30 mN. Prior to scratch testing, the surface of the specimens exhibited many cracks, indicative of poor adherence. This was more pronounced for the specimens annealed at 800°C. Figures 3c and 3d show local spallation in the specimen annealed for 2 h at 600°C. The specimens annealed at 800°C showed a significant change following adhesion failure, with pre-existing cracks presumably inducing spallation.

**Incoloy 800H.** The nonannealed samples showed a higher critical load compared to AISI-304 and AISI-316 (see Fig. 1d). The critical load corresponds to film buckling without spallation or delamination. After annealing at 600°C, $L_c$ decreased to about 70 mN and remained constant with annealing time. Adhesion failure corresponded to a sharp increase in voltage output. After annealing at 800°C, $L_c$ decreased to about 40–50 mN and extensive surface cracking was observed, similar to that for AISI-304 (Figs 3a and 3b).

**PM 2000.** In this case, $L_c$ is much higher compared with the aforementioned alloys (see Fig. 1e). In many cases, only an increase in plastic deformation of the alumina film occurred without any peeling. Therefore $L_c$ was defined as the load at which the coating was extensively plastically deformed. For the nonannealed specimens, minor peeling occurred. In all cases, $L_c$ increased after annealing. No improvement was observed after annealing at 800°C. The critical load varied between 200 and 240 mN.

**PM 3030.** The critical load increased after thermal treatment at 600°C (Fig. 1f). No further effect was found as a function of the annealing time. A delamination phenomenon occurred in the nonannealed sample, resulting in a sudden increase of output voltage. For the annealed samples, plastic deformation occurred at the end of the scratch, as for the PM 2000 samples, making the determination of $L_c$ difficult. After annealing at 800°C, $L_c$ increased significantly to almost 400 mN. After 4 and 17 h annealing, locally very small particles of the oxide film were detached. Figures 3e and 3f show the scratch morphology of the specimens annealed at 600 and 800°C for 2 and 4 h, respectively. The local delamination of the film, annealed at 800°C is remarkable.

**MA 956.** For the nonannealed sample, delamination and spalling beyond a well-defined critical load occurred, which did not alter significantly with annealing at 600°C (Fig. 1g). In samples annealed for 4 and 17 h, delamination occurred only at sites where also very small cracks had formed. In the sample annealed for 2 h at 600°C, only local delamination and plastic deformation were observed (Fig. 3g). The specimens annealed at 800°C exhibited only plastic deformation (Fig. 3h) with no delamination.
MA 754. Annealing at 600°C resulted in a significant increase in $L_c$ from about 160 mN in the absence of annealing to more than 210 mN (Fig. 1h). Only after longer exposure times did $L_c$ decrease slightly. The scratches were characterized by plastic deformation of the oxide film with internal microcracks. After annealing at 800°C, only plastic deformation of the film was observed, accompanied by the removal of very fine particles of the oxide film in samples annealed for 17 h. The critical load increased after thermal treatment, but decreased slightly after longer exposure times.

3.2. Auger electron spectroscopy (AES)

The alumina–substrate interface was investigated by means of AES. Generally, the AES analyses showed for the ‘normally’ high alloy steels AISI-321, AISI-304, AISI-316, and Incoloy 800H that after thermal treatment the interface was no longer sharp. A certain amount of chromium had penetrated into the alumina film.

The Auger depth profile of the thermally treated alumina–AISI-321 specimen is shown in Fig. 4a. After the heat treatment, only a small enrichment at the interface of manganese and titanium was observed.

In the case of the alumina–AISI-304 specimens, it was found (see Fig. 4b) that after heating to 800°C, small amounts of sulfur and silicon were segregated at the interface.

Figure 4c shows the AES depth profile of the thermally treated alumina–AISI-316 specimen. A small enrichment of sulfur and manganese was observed near the interface. No carbon segregation was found, corresponding also to the observations for AISI-321. The sulfur enrichment for specimens AISI-304 and AISI-316 can be due to the high sulfur content of AISI-304 and AISI-316, nominally 0.016 and 0.017 wt%, respectively, as shown in Table 1.

Depth profile analyses of Incoloy 800H showed no segregation of specific elements, as already found for the aforementioned stainless steels. Hence, the Auger depth profile analyses indicated that if sulfur or titanium and carbon were present to a certain level within these steels, they would segregate at the interface after heat treatment.

The AES depth profiles of the ODS alloy PM 2000, before and after thermal treatment, are shown in Figs 5a and 5b, respectively. Before annealing, no titanium or carbon enrichment was observed at the interface (the amount was below the detection level), as was in the annealed specimen. The thickness at which an increased carbon and titanium concentration was found was about 20–30 nm. In the case of the annealed specimen PM 3030, only very small amounts of carbon could be detected at the interface. No other element enrichments were found by AES. Figures 5c and 5d show the AES depth profiles of the alumina–MA 956 specimens, which correspond well to those of PM 2000, exhibiting also an increased concentration of titanium and carbon at the interface. The same results were obtained for the alumina–MA 754 specimens. Hence, except for alumina–PM 3030, which does not contain titanium, the Auger data revealed that for ODS alloys titanium and carbon segregated to the interface after thermal annealing, whereas sulfur did not.
Figure 4. AES depth profiles of (a) alumina–AISI-321 substrate, (b) alumina–AISI-304 substrate, and (c) alumina–AISI-316 substrate (all annealed for 17 h at 800°C). Sputter depth 0 corresponds to the gas–oxide interface.
Figure 5. AES depth profiles for alumina–PM 2000, (a) before annealing and (b) after annealing for 17 h at 800°C, and for alumina–MA 956, (c) before annealing and (d) after annealing for 17 h at 800°C. Sputter depth 0 corresponds to the gas–oxide interface.
4. DISCUSSION

From the results it is clear that the adhesion strength between the alumina film and the underlying non-ODS high alloy steels was reduced by annealing. This reduction in $L_c$ after thermal annealing processes may be explained by (1) the presence of thermal stresses in the thin alumina films as a result of the difference in the coefficient of thermal expansion of the film and the substrate, and (2) modification in the interfacial structure and/or composition.

Assuming that intrinsic stresses have fully disappeared by the thermal treatment [5], the total stress is now equal to the thermal stress:

$$\sigma_{\text{total}} = \sigma_{\text{thermal}} = E_f \Delta T \Delta \alpha,$$

where $E_f$ is Young’s modulus of the film, $\Delta T$ is the difference between the deposition and the actual (room) temperature, and $\Delta \alpha$ is the difference between the thermal expansion coefficients.

An increasing difference between the annealing and the actual temperatures leads to an increased thermal stress, which eventually will reach the critical stress for adhesion failure. Earlier experiments [4] have shown that metal–organic chemical vapor-deposited alumina films at reduced as well as at atmospheric pressure suffer from internal stress which can lead, under extreme circumstances, to cracking and delamination. Furthermore, these films start to crack and delaminate beyond a critical thickness, due to a high intrinsic (growth) stress. This may be explained by phase transformation reactions of boehmite to $\gamma$-alumina, which is more pronounced at the interface than at the outer part of the film. This might be explained by a longer residence time of the inner part in the reactor during deposition. Thermal annealing experiments have shown [8] that the intrinsic stress is significantly reduced after 4 h (or longer) at 700 °C (or higher). Scale failure after annealing can only be attributed to temperature changes and different values of the coefficients of thermal expansion, $\alpha($alumina$) = 8 \times 10^{-6} \text{ K}^{-1}$ and $\alpha($steel$) = 12 \times 10^{-6} \text{ K}^{-1}$ (assuming that the bulk values can also be used here). It is possible to determine the thermal stress from equation (1). Because the thermal expansion coefficient of the metallic substrates is larger than that of the alumina film, the oxide film will be subjected to compression after cooling. Scale adherence failure by the presence of a compressive stress gradient is often related to the energy required for the production of two new surfaces. For equal film thicknesses, the higher the critical stress for spalling and/or delamination, the higher the fracture toughness of the film/substrate combination will be. Hence, the presence of high thermal stress may have a detrimental effect on scale adherence.

A modification of the structure of the alumina film may also affect the interfacial structure of the specimens. It was found [8] that the untreated alumina films, analyzed by XRD, show an ‘amorphous’ structure. After annealing, small peaks of $\gamma$-alumina were detected. This transformation to a harder, more brittle layer might also explain why the delamination of the oxide film was more pronounced after thermal treatment.

The AES analyses revealed that thermally treated alumina-coated AISI-304 and AISI-316 showed at the interface an increased concentration of sulfur and silicon,
and of sulfur and manganese, respectively. In the case of AISI-321, where the critical load increased after thermal treatment at 800°C, no sulfur was detected. Only small amounts of titanium, manganese, and carbon were found near the interface. The only characteristic observation for the thermally treated alumina–Incoloy 800H specimen was the penetration of chromium into the alumina film. Sulfur segregation at the interface and chromium penetration into the alumina film might have a detrimental effect on adhesion. On the other hand, if titanium and carbon segregate at the interface, the adhesion strength is beneficially influenced. From the scanning scratch test results it is clear that scale adhesion or fracture toughness of the interface depends on the substrate composition and thermal treatment. Sulfur segregation near the interface, which has a deleterious effect on adhesion, has been reported elsewhere [2, 9, 10]. Lees [11] and Briant and Mulford [12] suggested that sulfur would have an embrittling effect when segregated at the interface. Sigler [13] investigated the influence of sulfur on the adherence of alumina grown on Fe–Cr–Al alloys. It was found that after a long annealing or after certain annealing cycles sulfur was removed from the foil, leading to an improved adherence. Although these results are valid for self-growing alumina scales [2, 11–13], this sulfur migration could presumably apply also to alumina films on metallic substrates. Other substrate elements which improve the adherence of alumina films are, for example, titanium and carbon. Especially for alloy AISI-321, containing titanium and carbon, and a very low sulfur concentration compared to AISI-304 and AISI-316, an increased adherence was determined after 17 h annealing at 800°C. The beneficial effect of titanium and carbon on the adherence of alumina films on steels has also been reported by Tosa et al. [2], who attributed this effect to the formation of TiC within the alumina film, resulting in an anchoring effect. The concentration gradient of TiC near the interface can also relax the thermal stress. A third mechanism, proposed by Tosa et al. [2], is that Al–O–Ti bonding forms between the alumina film and the precipitated TiC. On the other hand, if only graphite precipitation were to take place, a local volume expansion of the precipitate at the interface would push up the film, causing cracking and delamination. Furthermore, Yoshitake et al. [10] suggested that the precipitation of TiC may form Ti–O bonds, suppressing the segregation of sulfur which would otherwise break the Fe–O bonds. Although several suggestions concerning the TiC effect are given here, no detailed mechanism has been proposed yet which clearly explains the increased adherence of alumina films on metallic substrates. Their results are consistent with those reported here. With respect to the effect of the migration of chromium into the alumina matrix on adherence, nothing has been reported. The diffusion of chromium took place during annealing, due to the miscibility of chromia and alumina. From the AES and the scanning scratch test results, it can be assumed that this miscibility will have a detrimental effect on adherence. This is also based on the fact that no significant segregation of certain elements was observed for AISI-321 after annealing.

With respect to the ODS alloys, it was found that the critical load increased after annealing at 800°C. From Table 1 it can be seen that the nominal sulfur concentration of the MA- and PM-type ODS alloys is very low, indicating that the sulfur effect can be eliminated. Also, the AES analyses revealed that no sulfur segregation occurred within the limits of detection. For the type PM 2000, MA 956, and MA 754 alloys,
it was observed that after thermal treatment at 800°C for 17 h, the interface was enriched in titanium and carbon, probably in the form of titanium carbide. The absence of sulfur at the interface could be explained by its low concentration in the alloy, or by the presence of sulfur-trapping elements, such as yttrium. This was, for example, not the case for Incoloy 800H. This alloy does not contain any yttrium. This theory of trapping S by Y was first proposed by Smeggil et al. [14, 15]. They studied the effect of Y for Ni–Cr–Al alloys by AES. In addition to the incorporation of yttrium into the metallic matrix, yttria dispersions had a beneficial effect on the adherence of alumina layers. Ikeda and Yata [16] studied alumina films which were applied to ODS alloys by magnetron sputtering. They gave an excellent review of several mechanisms of the ‘yttria’ effect on sulfur segregation, including (1) sulfur trapping by yttria, (2) retardation of diffusion of elements such as manganese and chromium into the alumina films, and (3) increased plasticity of alumina doped with yttria. Self-forming alumina scales by oxidation of ODS alloys exhibit an excellent delamination resistance [6]. Due to the presence of a well-adherent alumina film induced by the presence of yttria particles, these oxide films can also be used as excellent diffusion barriers against aggressive gas compounds [17]. For example, it was found that pre-oxidation in air of MA 956 produced an alumina scale with excellent resistance against sulfidation. Retardation of the diffusion of chromium by the presence of yttria was also found in the present investigations. From Figs 4 and 5 it can be seen that the amount of chromium that migrated into the alumina matrix is less pronounced for the ODS alloys.

The increased adherence of alumina films to ODS alloys may be attributed to (1) the low sulfur concentration of the alloy which decreases the susceptibility for segregation near the interface, (2) the presence of yttria as a sulfur-trapping compound, (3) the retardation of migration of chromium into the alumina matrix, and (4) the formation of titanium carbide near the interface (anchoring affect). The fracture toughness of the interface is related to the chemical bonds between the metallic substrate and the oxide film, and can thus be affected by segregation of elements from the matrix to the interface.

5. CONCLUSIONS

- Thermal annealing at high temperatures results in high thermal stresses which may detrimentally affect the adhesion between alumina films and the substrate.
- Phase transformation of the amorphous alumina to γ-alumina may increase the brittleness of the oxide film, affecting the critical load for cracking or delamination.
- Sulfur segregation at the oxide–substrate interface can reduce the critical load for oxide spallation. Titanium carbide probably influences the adherence beneficially.
- Alumina layers on ODS alloys exhibited an increased critical load, due to the presence of sulfur-trapping elements and titanium carbide segregation near the interface.
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