Acta Materialia 105 (2016) 438-448

Contents lists available at ScienceDirect

Acta Materialia

journal homepage: www.elsevier.com/locate/actamat

Full length article

Growth mechanism of the interdiffusion zone between platinum modified bond coats and single crystal superalloys



Acta MATERIALIA

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A R T I C L E I N F O

Article history: Received 21 November 2015 Accepted 5 December 2015 Available online xxx

Keywords: Nickel-based single crystal superalloys Intermetallics (iron-aluminides) Precipitate Diffusion mechanism TEM

ABSTRACT

Pt-modified β -NiAl bond coats are applied over the superalloys for oxidation protection in jet engine applications. However, as shown in this study, it also enhances the growth of the interdiffusion zone developed between the bond coat and the superalloy along with brittle precipitates. Location of the Kirkendall plane indicates that a precipitate free sublayer grows from the bond coat, whereas another sublayer grows from the superalloy containing very high volume fraction of precipitates. With increasing Pt content, thickness of both the sublayers increases because of an increase in diffusion rates of the components. Quantitative electron probe microanalysis indicates high concentration of refractory components in the precipitates. Transmission electron microscopy shows that René N5 superalloy produces TCP phases μ and P, whereas CMSX-4 superalloy produces μ and σ in the interdiffusion zone. With increasing Pt content in the bond coat, the average size of the precipitates decreases when coupled with René N5. Precipitates become much finer when the same bond coats are coupled with CMSX-4.

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1. Introduction

Ni-based superalloys are used in gas turbine engines. Many refractory elements such as Co, Fe, Mo, Re, Ru, Ti, Nb, Ta, V, W etc. are added to these alloys for providing high temperature mechanical strength and creep resistance [1-3]. These are partitioned into two phases, namely γ -Ni(Al) solid solution and γ' -Ni₃Al intermetallic compound. The aluminide bond coat is applied over the superalloy for protection against oxidation and yttria stabilized zirconia (YSZ) is used as top coat for thermal protection. Bond coat acts as an Al reservoir from which Al diffuses out for the formation of Al₂O₃ at the interface of bond coat and top coat. Oxygen diffusion rate through Al₂O₃ is very low making it suitable for protection of the superalloy substrate from oxidation. Pt-modified β -NiAl is one of the commonly used bond coats [4,5]. Although the exact mechanism due to which Pt plays a beneficial role is not very clear, it is reported that the presence of Pt decreases the sulfur segregation at the bond coat/Al₂O₃ interface and improves the adherence of the oxide layer. It also improves the lifespan of the bond coat by producing the Al₂O₃ layer at a faster rate on the exposed bond coat after spallation caused by thermal mismatch. Therefore, diffusion of

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http://dx.doi.org/10.1016/j.actamat.2015.12.014

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Al out of the bond coat for the growth of the oxide layer might be enhanced because of Pt-addition. This is, in fact, in agreement with the theoretical analysis showing an increase in point defects concentrations (such as vacancies and antisites) because of Pt-addition to β -NiAl facilitating higher diffusion rate of components [6]. Recently, this is validated based on interdiffusion studies conducted by the diffusion couple technique [7].

During operation, diffusion of components occurs from both the sides of the bond coat. As already mentioned, towards the top coat side Al diffuses out from bond coat to react with oxygen for the formation of the oxide layer. On the other side, an interdiffusion zone (IDZ) grows between the bond coat and the superalloy substrate by diffusion of Al from the Al-rich bond coat and diffusion of Ni from the Ni-rich superalloy. The loss of Ni from the superalloy leads to the growth of the topologically close-packed (TCP) precipitates, which are rich in refractory elements. These brittle precipitates are one of the sources for mechanical failure. Many studies are available in literature that discuss the effect of Pt on microstructural developments of nickel aluminide coatings by pack cementation [8–11], oxidation rate of the bond coats [12–15], nature of the oxide scale [16–19], degradation of the coating phases [20–22] and rumpling of the oxide scale [23–25]. On an industrial scale, Pt is first electroplated over the superalloy and then subjected to aluminization. Following this, β -(Ni,Pt)Al is grown through an interdiffusion process between layers of Al, Pt and the superalloy



substrate. The β -(Ni,Pt)Al coatings obtained following this process exhibit a varying concentration of Pt across them, thus making it difficult for a systematic study delineating the role of Pt on evolution of IDZ and the TCP phases.

Therefore, the aim of the present study is to examine the role of Pt on the growth of IDZ and the TCP phases. Diffusion couple technique is followed, which mimics the real structure with an added possibility for quantitative analysis. Two sets of bond coats $(Ni_{50-x}Pt_x)Al_{50}$ and $(Ni_{60-x}Pt_x)Al_{40}$ are prepared with Pt content of x = 0, 5, 10 and 15 at.%. These are coupled with two different types of single crystal superalloys: René N5 and CMSX-4. The interdiffusion zones and precipitates are characterized with the help of Scanning Electron Microscope (SEM), Electron Probe Micro-Analyzer (EPMA) equipped with Wavelength Dispersive Spectrometer (WDS) and Energy Dispersive Spectrometer (EDS), and Transmission Electron Microscope (TEM).

2. Experimental procedure

The average compositions of different components in René N5 and CMSX-4 are listed in Table 1. The only difference is in the Ti content. René N5 is free of Ti, whereas, CMSX-4 has small amount of Ti. β -(Ni,Pt)Al alloys with desired compositions of (Ni_{50-x}Pt_x)Al₅₀ and $(Ni_{60-x}Pt_x)Al_{40}$ where x = 0, 5, 10 and 15 at.% were melted in an arc melting furnace under an Ar atmosphere using weighed quantities of Ni (99.95 wt%), Pt (99.95 wt.%) and Al (99.9 wt.%). Melted samples were then homogenized at 1200 $^{\circ}C$ (±5 $^{\circ}C$) for 100 h in a calibrated vacuum furnace ($\sim 10^{-4}$ Pa). The compositions of the alloys were measured randomly at many points and standard deviations from average were found to be around ± 0.5 at.%. Following, these were EDM (Electro Discharge Machine) cut to the dimensions of 5 mm \times 5 mm \times 1 mm for coupling with superalloys of similar dimensions. These were then bonded and annealed at 1100 °C (\pm 5 °C) for 25 h in the same vacuum furnace. After the experiment, samples were removed, cross-sectioned and prepared metallographically. Final polishing of these cross-sectioned samples was carried out using colloidal silica (~0.04 µm). Quantitative WDS composition mapping was performed in an electron probe micro-analyzer (EPMA) equipped with Field Emission Gun (FEG). For identification of the phases present, characterization using a field emission tipped transmission electron microscope (F30, FEI) was carried out. Samples for transmission electron microscopy were made using focused ion beam (FIB) milling.

3. Growth of precipitates between $\beta\text{-(Ni,Pt)Al}$ bond coats and René N5 superalloy

3.1. General features of the interdiffusion zone

As already mentioned two sets of bond coats were prepared, type 1: $(Ni_{50-x}Pt_x)Al_{50}$ and type 2: $(Ni_{60-x}Pt_x)Al_{40}$, where x = 0, 5, 10 and 15 at.%. According to the Ni–Pt–Al ternary phase diagram [26] (see supplementary file), type 1 bond coats have the stoichiometric compositions of β -(Ni,Pt)Al *i.e.* (Ni + Pt):Al \equiv 50:50. Type 2 bond coats have compositions close to the (Ni,Pt)-rich side of the phase boundary. These were diffusion coupled with single crystal superalloys, i.e., René N5 and CMSX-4. Superalloys are the phase

Table 1
Compositions of René N5 and CMSX 4 superalloys.

Component (at. %)	Со	Cr	Al	Ti	Та	Мо	W	Re	Ni
René N5	7.7	8.1	13.8	-	2.2	0.9	1.6	1	64.7
CMSX 4	8.7	7.1	11.8	1.2	2	0.3	1.8	1	66

ppm levels: Y, Zr, Hf, S, C, O, P.

mixtures of γ -Ni(Al) solid solution and γ' -Ni₃Al intermetallic compound containing many refractory components in them, as listed in Table 1. Fig. 1a shows schematic representations of the interdiffusion zone developed between two types of bond coats and superalloys in general. Fig. 1b-d show the SEM micrographs and composition profiles of different parts of the IDZ developed between type 1 bond coat with 15 at.% Pt and René N5 annealed at 1100 °C for 25 h. Fig. 1e shows the composition profile of precipitate free IDZ between type 2 bond coat and René N5 annealed at 1100 °C for 25 h, since the other part is similar to the IDZ when type 1 bond coat was coupled. As shown in Fig. 1b and c, the location of the Kirkendall marker plane (shown as K) is detected by the presence of line of pores [27,28], which was evident in all the diffusion couples. Following the physico-chemical model [29,30], it indicates that on either side of this plane, IDZ has grown differently as two different sublayers from two different sides of the diffusion couple. Precipitate-free IDZ is grown from the bond coat and the extent of this zone could be understood from the composition profile, as shown in Fig. 1d. Precipitate-containing zone is grown from the superalloy, as shown in Fig. 1b. A focused area, as indicated by a dotted rectangle comprising mainly the precipitates, is shown in Fig. 1c. It indicates that precipitate-containing zone has two different parts. β -NiAl and a phase mixture of β -NiAl and γ' -Ni₃Al are present along with precipitates. From the composition profile of the precipitate-free part, as shown in Fig. 1d and quantitative WDS

components. On left-hand side of the Kirkendall marker plane, precipitatefree IDZ is grown from the bond coat by the loss of Al and Pt from the bond coat and gain of Ni from the precipitate-containing IDZ. Other side of the marker plane *i.e.* IDZ with precipitates is grown from the superalloy. As already mentioned, many refractory components are added in the superalloy for imparting superior physico-mechanical properties. Concentrations of these components are maximized depending on their respective solubility limits in the γ -Ni(Al) and γ' -Ni₃Al phases of the superalloy. Solubility is high especially in the γ -Ni(Al) solid solution. With the loss of Ni or addition of Al, first the phase mixture of γ -Ni(Al) and γ' -Ni₃Al of superalloy gets converted to the phase mixture of β -NiAl and γ' -Ni₃Al. This leads to the growth of precipitates by rejection of refractory components because of dissociation of the γ -Ni(Al) phase. With further loss of Ni and addition of Al, β -NiAl and γ' -Ni₃Al phase mixture gets converted to a single phase β -NiAl. Much higher volume fraction of precipitates in this part indicates that the solubility limits of the refractory components in this β-NiAl phase are much lower. The average compositions of Co and Cr in the precipitate-containing IDZ is not much less compared to the superalloy. Therefore, these components diffuse from this part to the precipitate-free IDZ. Diffusion of refractory components to the precipitate-free IDZ is very small indicating that these are mainly consumed by the growth of the precipitates and also diffusion rates of these components in the β -NiAl phase must be very low.

analysis of the precipitate-containing part, we can analyze the

growth mechanism of the phases with respect to the diffusion of

When type 2 bond coats are diffusion coupled with René N5, the main difference in terms of general feature is found for the precipitate-free IDZ. This can be realized from the composition profile measured in (Ni₄₅Pt₁₅)Al₄₀/René N5 couple, as shown in Fig. 1e. Since the composition of these bond coats are very close to the phase boundary composition in the (Ni,Pt)-rich side (see Ni–Pt–Al phase diagram in the supplementary file), it does not grow very prominently like in the previous example of type 1 bond coat. The IDZ containing precipitates is similar in nature in terms of phases; however the thickness is different as discussed in the next section. The micrographs of the entire interdiffusion zone are shown in Fig. 2.

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