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Comparative tensile behavior of freestanding γ - γ' and β -(Ni,Pt)Al bond coats and effect on tensile properties of coated superalloy

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ABSTRACT

The tensile behavior of freestanding 40 μm thick γ - γ' and 50 μm thick β -(Ni,Pt)Al coatings was evaluated at room temperature (RT) and 870 °C by microtensile testing method. Simultaneously, the effect of coatings on the tensile properties of the directionally solidified (DS) CM-247LC superalloy was also examined. Both the freestanding coatings had lower strength than that of the superalloy and the yield as well as the ultimate tensile strength (YS and UTS) of the superalloy substrate was lowered by the application of either coating. At RT, the freestanding γ - γ' coating was significantly stronger (YS = 660 MPa) and ductile than that of the brittle β -(Ni,Pt)Al coating which fractured at ~300 MPa with negligible ductility. Comparatively, the β -(Ni,Pt)Al coating caused more deterioration in substrate tensile properties (especially ductility) than that of the γ - γ' coating at RT. On the contrary, at 870 °C, albeit their similar YS, the γ - γ' coating exhibited brittle intergranular fracture and limited ductility whereas the β -(Ni,Pt)Al coating showed ductile failure. Numerous sharp cracks formed by the de-cohesion of the γ and γ' phase boundaries within the γ - γ' coating and their penetration into the substrate aggravated degradation in the substrate ductility than in case of the ductile β -(Ni,Pt)Al coating.

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1. Introduction

Oxidation resistant diffusion aluminide coatings are applied on Nisuperalloy gas turbine engine components for enhancing their durability and performance at high temperatures [1–4]. Pt-modified β -NiAl bond coats, also known as β -(Ni,Pt)Al, serve as excellent oxidation barriers and are used in advanced thermal barrier coating (TBC) systems [1–4]. However, these bond coats are incompatible, both in composition and phase constitution, with that of the superalloy substrate. The coating contains high Al (40 at.%) and low Ni (35 at.%) when compared to the low Al (15 at.%) and high Ni (90 at.%) in the superalloy. Furthermore, the coating is constituted of the intermetallic B2-NiAl phase [1–4] while the substrate is comprised of FCC γ -Ni matrix containing coherent ordered L1₂ γ '-Ni₃Al precipitates [5,6].

During service at high temperatures, the composition gradient causes interdiffusion of Al and Ni between the β -(Ni,Pt)Al coating and the substrate, resulting in dynamic phase changes within the coating and at the coating-substrate interface [4,5,7–11]. The stresses associated with the phase transformations accentuate rumpling in the bond coat and spallation of the TBC [12]. In order to overcome these limitations, diffusion Pt-aluminide bond coats having a γ - γ ' phase constitution

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rather than the conventional B2-NiAl structure are being developed [13–18]. These γ - γ' coatings are enriched in Pt but have similar Al and Ni content as that of the superalloy [13–18]. Recent studies have shown that the γ - γ' coatings possess good oxidation resistance as well as microstructural stability during high temperature exposure [13–18].

Considering the use of coated components in gas turbine engines, the evaluation of mechanical properties of coatings is crucial. The β -(Ni,Pt)Al coatings undergo brittle fracture at low temperatures and have a high brittle-to-ductile transition-temperature (BDTT), typically above 650 °C [19–21]. Cracks formed in the β -(Ni,Pt)Al coating are known to deteriorate the mechanical properties of the coated superalloy components at low temperatures [1,3]. Though information on the tensile behavior and properties of the β-(Ni,Pt)Al coating exists [19– 22,24,25], however, the tensile behavior of γ - γ' coating has not been reported in the open literature. Therefore, the objective of the present study is to evaluate the tensile property of a freestanding γ - γ' coating (i.e. without any substrate attached to it) and compare with that of the β -(Ni,Pt)Al coating. Microtensile testing of the freestanding coatings [19-25] has been carried out to evaluate the representative tensile properties of the coatings without any interference from the substrate. The implications of the tensile behavior of the freestanding coatings on the tensile properties of the Ni-base superalloy, CM-247LC, have also been examined. The temperatures for the tensile tests were

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selected as room temperature (RT) and 870 $^{\circ}$ C because of the relevance of these temperatures in benchmarking of the superalloy tensile properties.

2. Experimental details

Directionally solidified (DS) CM-247LC alloy, with a nominal composition (in wt.%) 9.2 Co-8.1 Cr-9.5 W-5.6 Al-3.2 Ta-1.5 Hf-0.7 Ti-0.015 Zr-0.5 Mo-0.15 B-0.07 C-balance Ni, was available in the form of 13 mm diameter rods. The $\langle 001 \rangle$ solidification direction was along the length of the rods. The average grain size perpendicular to the solidification direction was 2.1 mm whereas the primary dendritic arm spacing and the secondary dendritic arm spacing were about 350 and 50 μm , respectively. The rods were given a two-stage solution heat treatment in vacuum: 1230 °C for 30 min followed by 3 h at 1260 °C. The vacuum level was maintained at 10^{-5} mbar during the various heat treatments mentioned in the study. Subsequently, the solution heat treated rods were used as substrates for the deposition of coatings. Details on the coating deposition, fabrication of freestanding coating microtensile samples, macrospecimens, and tensile testing are mentioned below:

2.1. Fabrication of freestanding coating samples and microtensile testing

Strips, 0.5 mm in thickness, were cut out from the solution treated rods by electro-discharge machining (EDM) such that the longitudinal axis of the strips was parallel to the length of the rods, i.e. parallel to <001> solidification direction. Subsequently, the strips were grit blasted using a medium of zircon sand (average particle size of -325 mesh) and compressed air (pressure of 0.5 bar). The low grit blasting pressure did not cause any deformation of the strips and generated a surface having average roughness (Ra) of 0.6 µm adequate for the adhesion of Pt layer that was deposited subsequently by electroplating. For the formation of the γ - γ' coating, a 5 μ m thick layer of Pt was electrodeposited on the grit blasted strips and subsequent vacuum diffusion treatment carried out at 1080 °C for 4 h. The β -(Ni,Pt)Al coating was formed by aluminizing of some of the above Pt plated and diffused (i.e. γ - γ ' coated) strips using the powder mixture (in wt.%) 15Al-2NH₄Cl-83Al₂O₃ at 600 °C for 5 h and subsequent vacuum diffusion heat treatment at 1080 °C for 4 h. All the coated strips were then subjected to an aging heat treatment at 870 °C for 20 h in vacuum.

The microtensile samples used in the present study had the dimensions: gage length of 2 mm, gage width of 0.5 mm, fillet radius of 0.5 mm and an overall length of 8 mm. The above dimensions were established using parametric linear elastic finite element method (LE-FEM) simulations [24]. The microtensile samples were machined out from the coated strips by EDM. The gage length of each of the samples was parallel to the longitudinal direction <001> of the strips. Each cut-out sample was thinned from one side by precision polishing using 600, 1000, 1500, 2500 grit polishing paper (in that sequence). Final polishing was done on films containing 2, 1 and 0.5 µm diamond particles (in that sequence). The extent of thickness reduction was monitored during polishing using a precision micrometer [21,24]. Polishing was continued till the entire substrate was removed and the sample thickness became equal to the coating thickness, i.e. 40 μ m for the γ - γ' coating and 50 μ m for the β-(Ni,Pt)Al coating. Subsequently, Pt markers were deposited on the gage length of the micro-specimens using a Quanta 200 3D focused ion beam (FIB) machine. These markers enabled the in-situ measurement of strain in the specimens during tensile testing by means of a non-contact optical extensometer.

A 500 N Walterbai-Ag microtensile testing machine was used for testing of the freestanding coating samples. Since the samples were fragile, slotted grips were used to hold the samples during the test (shown in the inset in Fig. 2) [24]. Tensile testing was carried out at RT and 870 °C at a nominal strain rate of 10^{-3} s⁻¹. For high temperature testing, the microsample was resistively heated in ambient surroundings by passing DC current. The temperature of the specimen gage

was monitored using a two-color ratio infrared radiation (IR) pyrometer, having an accuracy within $\pm\,10$ °C. After the sample attained the temperature, tensile loading was carried out. An optical video extensometer, having a strain resolution of $10\,\mu\text{-strain}$, was used for continuous measuring of strain in the specimen during testing. The displacement of the Pt markers, tracked using a non-contact extensometer, enabled the measurement of longitudinal strain directly in the micro-specimens during testing. At least three samples were tested for any given condition to ensure consistency in the results. The stress-strain plot was recorded in a computer connected with the machine.

2.2. Fabrication of coated superalloy macrosamples and tensile testing

Round tensile samples having gage diameter and gage length of 4 and 50 mm, respectively, as per ASTM E8M standard [26], were machined from solution heat treated superalloy rods using a computerized numerically controlled (CNC) milling machine. Prior to the coating formation, the samples were subjected to the same grit blasting process as mentioned earlier. The γ - γ' and the β -(Ni,Pt)Al coatings were formed on the tensile samples in the same manner as that formed on the strips, as discussed before. The coated tensile samples were aged at 870 °C for 20 h in vacuum in order to form the desired γ/γ' structure of the substrate superalloy. Uncoated tensile samples were also fabricated directly from the fully heat treated superalloy rods, i.e. after the rods had been subjected to the two-stage solution heat treatment and aging heat treatments, as mentioned above.

Tensile testing of the coated and uncoated samples was carried out at RT and 870 °C in an Instron 4400R machine. For the high temperature tests, the sample was heated in air using a split furnace equipped with a thermocouple and temperature controller. A nominal strain rate of $10^{-3}~\rm s^{-1}$ was used for the tensile tests. For any given condition, three samples were tested to ensure consistency in the results.

2.3. Characterization

The microstructure and fracture surface of the samples were studied using a Quanta 400 scanning electron microscope (SEM) operating at 20 kV. A Cameca SX-100 electron probe micro-analyzer (EPMA) operating at 20 kV was used for chemical analysis of the coating.

3. Results

3.1. Microstructure

Fig. 1(a) shows the cross-section of the $\gamma\text{-}\gamma'$ coated superalloy. The coating thickness was approximately 40 μm and its $\gamma\text{-}\gamma'$ phase constitution was confirmed by XRD [27]. The bright and the gray phases in the coating correspond to the γ' and γ phases, respectively, as shown in Fig. 1(a). In addition to γ and γ' phases, several Kirkendall porosities were also present in the coating. Such porosities were generated due to unequal mass transfer and interdiffusion between the Pt layer and the substrate alloy during coating formation, i.e. during the vacuum diffusion heat treatment at 1080 °C for 4 h [4,27]. The concentration of Pt and Al in the coating was approximately 20 and 10 at.%, respectively, as reported in our earlier study [27].

The β -(Ni,Pt)Al coating exhibited a two-layer microstructure, as shown in Fig. 1(b). The outer layer consisted of B2-(Ni,Pt)Al, i.e. Pt was present in solid solution in the B2-NiAl phase, as evident from the bright hue in the BSE image (Fig. 1(b)). The B2-NiAl phase of the outer coating layer was confirmed by XRD [4,21]. The inner layer was the interdiffusion zone (IDZ), consisting of a B2-NiAl matrix dispersed with numerous W and Cr rich precipitates (Fig. 1(b) and (c)) [1–4,28]. The thickness of the coating was approximately 50 μ m. The microstructural details and the mechanism of formation of the above high activity β -type Pt-aluminide coatings have been well reported [1–4,28]. As

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