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## Thickness apropos stoichiometry in Pt-aluminide (PtAl) coating: Implications on the tensile properties of a directionally solidified Ni-base superalloy



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#### ABSTRACT

The effect of thickness and stoichiometry of a PtAl coating on the tensile properties of a directionally solidified superalloy has been examined in the temperature range between room temperature (RT) to 1000 °C. The coating thickness was varied as 60, 90 and 180 µm. A concomitant change in the inherent stoichiometry, from Ni-rich to Al-rich, occurred for the constituent B2-(Ni,Pt)Al phase of the coating with the increase in thickness. The brittle-to-ductile-transition-temperature (BDTT) of the coating increased relatively with increase in thickness and, correspondingly, ductile deformation in the coatings occurred at higher temperatures of 700, 870 and 1000 °C. The high BDTT and brittle fracture in the thick coating till high temperatures had a negative bearing on the tensile properties of the superalloy substrate. In the brittle fracture regime for the respective coatings, the tensile properties of the substrate deteriorated more with the application of thick coatings. The formation of long through-thickness cracks and higher crack penetration depths in thick coating induce greater deterioration in tensile properties of the superalloy. On the other hand, ductile failure in the coatings retards the formation of through-thickness cracks, preserves the tensile strength, and improves the ductility of the substrate.

#### 1. Introduction

Oxidation resistant diffusion Pt-modified aluminide coatings (PtAl) are applied on gas turbine engine components for enhancing their high temperature capability. The oxidation resistance is derived from the constituent B2-NiAl phase which forms a stable, protective, and regenerative layer of alumina on the surface [\[1](#page--1-0)–4]. However, the B2 phase is inherently brittle and renders a high brittle-to-ductile transition temperature (BDTT) to the coating [5–[10\].](#page--1-1) Recent studies involving microtensile testing of standalone PtAl bond coats have shown that the BDTT of the coating can be as high as  $700 \degree C$  [5–[8\].](#page--1-1) Cracks formed in the coating at temperatures below BDTT are known to degrade the overall strain tolerance of the coated components [\[1,4,9](#page--1-0)–15]. Nevertheless, the PtAl coatings are indispensable because of their excellent oxidation resistance at high temperatures of 1000– 1100 °C.

In the modern-day thermal barrier coating (TBC) systems, the thickness of the PtAl bond coat used is  $~60-100 \text{ µm}$  [\[1,4\].](#page--1-0) From the point of view of enhancing the oxidation resistance, increasing the coating thickness can provide protection over prolonged durations. The PtAl coatings are graded in composition and formed by aluminization of the superalloy using the pack cementation or chemical vapor deposition processes  $[1-3,16-18]$  $[1-3,16-18]$  $[1-3,16-18]$ . The thickness of the PtAl coating can be increased by increasing the aluminization temperature and the concomitant Al concentration in the vapor phase during aluminizing, and vice versa [\[2,3,16](#page--1-2)–18]. Sharp changes in curvature of aerofoil components, such as that at leading and trailing edges in turbine blades [\[1,4\],](#page--1-0) are associated with change in diffusion flux of Al and Ni may and cause concomitant variation in thickness of the aluminide coatings. However, alteration in the coating thickness has implications on the stoichiometry of the constituent B2-NiAl phase. With the increase in coating thickness, the stoichiometry of the constituent B2 phase can be expected to vary from Ni-rich (hypo-stoichiometric) to Al-rich (hyperstoichiometric). In bulk NiAl, the change in stoichiometry has significant effects on the mechanical and fracture properties [\[19,20\]](#page--1-3). Increase in Al content is associated with the formation of vacancy defects in the B2 lattice which causes a greater extent of hardening and decrease in fracture toughness when compared to that of Ni-rich B2 where the lattice contains Ni-antisite defects [\[19,20\].](#page--1-3) The effects of stoichiometry and the associated defect structure on the mechanical properties of bulk B2-NiAl have been extensively studied and welldocumented in several reviews [\[18,19\]](#page--1-4). However, the effect of stoichio-

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metry of micron-scale B2-NiAl based coatings, such as that of PtAl, on the mechanical properties of a coating-superalloy ensemble is yet to be reported in the open literature.

The present paper examines the effect of PtAl coating thickness and the associated change in stoichiometry of the coating on tensile properties of the Ni-base directionally solidified (DS) superalloy, CM-247LC. The thickness of the coating has been varied between 60– 180 µm and the tensile properties evaluated at various temperatures between room temperature (RT)−1000 °C. The variation in tensile properties of the substrate superalloy with temperature and coating thickness has been correlated with that of the fracture surface features, cracking behavior, and stoichiometry of the coating.

#### 2. Experimental details

The DS CM-247LC superalloy was available in the solution heat treated condition in the form of 12 mm diameter rods. The direction of orientation of the grains [001] was along the longitudinal axis of the rods. The nominal chemical composition of the alloy is (in wt%) Ni-9.2Co-8.1Cr-9.5W-5.6Al-3.2Ta-1.5Hf-0.7Ti-0.005Zr-0.5Mo-0.05B-0.07 C. Some of the rods were given a two-stage aging heat treatment in vacuum (1080 °C/4 h followed by 870 °C/20 h) to achieve the required γ-γ′ structure. In the fully heat treated condition, the superalloy microstructure exhibits the typical  $\gamma$ - $\gamma'$  structure of superalloys, i.e. cuboidal γ′-Ni<sub>3</sub>Al precipitates were dispersed in a matrix of γ-Ni [\[21\].](#page--1-5) The average size and volume fraction of the  $\gamma'$  precipitates was ~0.5 µm and 0.65, respectively, as reported in our earlier studies [\[22,23\]](#page--1-6).

Round tensile samples (ASTM E8M [\[24\]](#page--1-7)) were machined out from the rods using a computerized numerically controlled (CNC) machine. The tensile specimens fabricated from the solution treated rods were applied with the PtAl coating. The coating was processed using the following sequence: (i) electroplating of a 5 µm thick layer of Pt on the specimens, (ii) diffusion heat treatment in vacuum at  $1080 \text{ °C}/4$  h, (iii) pack aluminization, (iv) post aluminization heat treatment in vacuum at 1080 °C/4 h, and (v) aging heat treatment at 870 °C/20 h in vacuum to form the γ-γ′ structure of substrate superalloy. Details on the microstructure evolution of the coating during each of the above mentioned processing steps can be obtained elsewhere [\[3\]](#page--1-8). The pack aluminization treatment was carried out using a powder mixture (in wt %) 15NiAl-3NH<sub>4</sub>Cl-82Al<sub>2</sub>O<sub>3</sub> in a medium of Ar for a 5 h. The aluminization temperature was varied as 600, 700 and 800 °C for achieving coatings of 60, 90 and 180 µm in thickness. The corresponding Al pick-up values, indicated by the specific weight gain after aluminization, were 7.4, 10.6 and 23.2 mg cm<sup>-2</sup>. In the subsequent text, the samples with coating thickness of 60, 90 and 180 µm have been referred to as C60, C90 and C180, respectively. The uncoated sample has been termed as UC.

Tensile testing of the uncoated and coated specimens was carried out at five different temperatures, i.e. RT, 500, 700, 870 and 1000 °C, using a 200 kN Walter-Bai tensile testing machine. For the high temperature tests, a split-type furnace was used for heating the sample which was fit into grips. The sample was soaked at the desired test temperature for 15 min prior to the start of tensile loading in order to ensure temperature uniformity. At least two samples of each type were tested for any given condition for ensuring consistency in the results. The nominal cross head speed was maintained at  $1 \text{ mm min}^{-1}$  for the tests.

Standard metallographic procedures involving sectioning of the samples using a low speed saw, mounting, and polishing were adopted for preparing the cross-section samples for microstructural examination. In order to prevent rounding of the sample edges and damage to the coating while polishing, a 10 µm thick Ni-plating was applied on the samples prior to sectioning of the samples. A Quanta 400D scanning electron microscope (SEM) operating at 20 kV was used for observing the microstructure and the fracture surface. The composition of the coatings was analysed by wavelength dispersive spectroscopy

(WDS) using a Cameca SX-100 electron probe microanalyzer (EPMA) operating at 20 kV. The variation in hardness across the coating thickness was measured using the instrumented Anton-Parr microhardness testing machine. A maximum load of 10 mN and a micro-Vickers indenter with a tip radius of  $0.1 \mu m$  was used for the indentation tests.

#### 3. Results

#### 3.1. Microstructure and composition of coatings

The cross-sectional microstructure of the coating in C60, C90 and C180 specimens is shown in [Fig. 1.](#page--1-9) The coating was uniform and the thickness was  $60 \pm 2$ ,  $87 \pm 6$  and  $185 \pm 5$  µm, respectively. All the coatings had a three-layer structure. The outer layer contained  $PtAl<sub>2</sub>$ and B2-NiAl phases. In addition, numerous fine W-rich precipitates were also present in the outer layer. The intermediate layer was constituted of a single-phase B2-NiAl and fewer W-rich precipitates were present in this layer than that of the outer layer. The matrix B2- NiAl phase in the outer and intermediate layers also contained some amount of Pt in solid solution, evident from the bright hue in the back scattered electron (BSE) images ([Fig. 1](#page--1-9)). The innermost layer was the heavily precipitated interdiffusion zone (IDZ) that is typically observed in diffusion aluminide coatings  $[1-3,25]$ . The thickness of the outer and intermediate layers increased with the increase in coating thickness. The thickness of the outer layer was 15, 30 and 80 µm in the C60, C90 and C180 coatings. The corresponding thickness of the intermediate layer was 25, 45 and 80 µm. The thickness of the IDZ was similar for all coatings at  $\sim$  20  $\mu$ m. The similar thickness of the IDZ can be expected since this layer forms during the post aluminization heat treatment [\[3\]](#page--1-8) which was the same for all coatings, as mentioned earlier.

The concentration plots for Ni, Pt and Al in the coatings are shown in [Fig. 2.](#page--1-10) For the coating in C60, the Ni content increased from 41 to 49 at%, Pt decreased from 9 to 3 at%, and Al decreased from 45 to 39 at % over a depth of 35 µm from the coating surface, i.e. across the outer and intermediate layers. The concentration profiles for Ni, Pt and Al also showed a similar trend for the coating in C90: the Ni concentration increased from 23 to 49 at%, Pt decreased from 11 to 1 at%, and Al decreased from 55 to 25 at% across the outer and intermediate layers (over depth of 75 µm from the surface). For the coating in C180, the Ni concentration was initially constant at ~35 at% across the outer layer (over a depth of 80 µm from the coating surface) and then increased from 35 to 47 at% across the intermediate layer (for depth from the coating surface between  $80 \mu m$  and  $160 \mu m$ ). The Al content was constant at ~57 at% in the outer layer and subsequently decreased to 50 at% across the intermediate layer. The Pt content decreased from 11 to 0.5 at% across the outer and intermediate layers. For all coatings, scatter in the Ni, Pt and Al concentration in the IDZ was observed because of the presence of numerous complex precipitates in this layer.

#### 3.2. Tensile properties

The tensile properties for the UC, C60, C90 and C180 specimens that were evaluated at various temperatures are shown in [Fig. 3](#page--1-9). For the UC sample, the 0.2% yield strength (YS) was 925 MPa at RT, increased to 970 MPa and 1000 MPa at 500 and 700 °C, respectively, and subsequently decreased to 850 MPa at 870 °C and 400 MPa at 1000 °C [\(Fig. 3\(](#page--1-9)a)). The anomalous strengthening behavior, i.e. increase in YS between RT-700 °C, can be ascribed to the well-known phenomenon of onset of cross slip of dislocations from octahedral to cube planes which gives rise to the formation of immobile Kear-Wilsdorf (KW) locks [\[26](#page--1-11)–28]. On the other hand, the decrease in strength at higher temperatures occurs due to thermally activated glide of dislocations overcoming the KW locks [\[26](#page--1-11)–28]. The YS for C60, C90 and C180 also increased with increase in temperature up to 700 °C and subsequently decreased. For instance, the YS for C90 increased from

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