

# Microstructural stability of AlN diffusion barrier for nanocomposite Ni + CrAlYSiHfN coating on single crystal superalloy at high temperatures



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## ARTICLE INFO

### Article history:

Received 7 September 2015

Received in revised form 12 October 2015

Accepted 15 October 2015

Available online 19 October 2015

### Keywords:

Diffusion barrier  
Nanocomposite coating  
TEM  
High temperature

## ABSTRACT

An AlN interlayer was fabricated by filtered vacuum arc evaporation as a diffusion barrier (DB) between Ni + CrAlYSiHfN composite coating and K417G superalloy. Microstructure changes of the AlN DB were investigated after exposure at 1000 and 1100 °C. Amorphous structure was found in the as-deposited AlN DB, which went through crystallization when exposing at high temperature. The AlN DB suppressed interdiffusion between overlayer and substrate effectively after 200 h exposure at both 1000 °C and 1100 °C. A few substrate element precipitates were observed within the grain boundary of the AlN DB after exposure at 1100 °C for 200 h.

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## 1. Introduction

The gas turbine blades protected by MCrAlY coatings (M = Ni, Co, or their combination) offer long service-lifetime when used below 1000 °C. Above 1000 °C, the interdiffusion between coating and superalloy substrate becomes a serious problem. Firstly, the interdiffusion decreases the Al and Cr contents in the coating due to inward diffusion of Al and Cr to the substrate and outward diffusion of Ti, Mo, W from the substrate, which may shorten the lifetime of coatings [1]. This problem may be solved by over-aluminizing MCrAlY coating [2–4]. Secondly, for secondary generation or more advanced single crystal superalloys designed for working at 1050 °C or above, the inward diffusion of Al from the coating results in formation of massive needle and fine granular topologically close-packed (TCP) phases in the secondary reaction zone (SRZ) [5]. The formation of TCP phases and SRZ deteriorates the ductility and creep resistance of Ni-base superalloys [6], which are regarded as the most serious problems in high temperature protection of superalloys. Besides, a potential harm for SRZ formation is the nucleation associated with surface damage [7]. The second problem became serious in case that MCrAlY coating was replaced by NiAl(Pt) [5,8–16].

Many studies in efforts to minimize the interdiffusion and prohibit the TCP formation in SRZ on coated superalloys have been

reported. They might be divided into three groups: nanocrystalline (NC) coating, EQ coating and diffusion barrier (DB). The sputtered NC coating, which was firstly for cast superalloy K38G reported in 1992 [17] and applicable to single crystal superalloys [18], have the same chemical compositions as the substrates. Therefore, all the sputtered NC coatings avoid interdiffusion with the alloy substrates in addition to possessing good oxidation protection. A more attractive merit of sputtering NC coating is that due to the columnar structure, the thermal or grown stresses transported from oxide scale will be easily relaxed by deformation of the NC coating, rather than by cracking of the oxide scale. The EQ coating was developed in which stable phases that are thermodynamically in equilibrium with the substrate, such as  $\gamma'$  phase, have been used as coating materials [19]. With this coating, SRZ formation between the coating and the substrate is suppressed and interdiffusion is minimized because chemical potentials of the alloying elements in the substrate and coating in equilibrium state are equal.

The DB has been extensively studied because it is working without modification of coating itself. They can be divided into metallic refractory, ceramic and active diffusion barriers. The Re–Cr(Mo)–Ni layer was stable and effectively retarded the interdiffusion between the Al reservoir layer and the alloy, and improved oxidation resistance of a Ni<sub>3</sub>Al-based superalloy IC6 and a single-crystal (SC) Ni-based superalloy TMS-82+ [20,21]. A Ni–W base diffusion barrier (DB) on a fourth generation Ni-base superalloy was found to be able to reduce the Al depletion in the  $\beta$ -(Ni,Pt)Al coating and to delay the phase transformations in the coating and the SRZ formation [22]. Cr-based interlayers have also been demonstrated to be

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capable of improving high temperature oxidation properties of MCrAlY coatings on Ni-base superalloy, which are primarily ascribed to the inhibition effect on the inward diffusion of Al [23]. The ceramic DB, such as Cr-O-N [24], Al-O-N [25], Cr<sub>23</sub>C<sub>6</sub> [26],  $\gamma$ -Al<sub>2</sub>O<sub>3</sub> [25] and  $\alpha$ -Al<sub>2</sub>O<sub>3</sub> [27,28], exhibited better diffusion barrier effects than the metallic DB. The ceramic DB, however, may suffer thermal shock problems because of its relatively lower coefficient of thermal expansions (CTE) than both the coatings and the substrates. The active DB is a thin oxide layer which is less thermodynamically than  $\alpha$ -Al<sub>2</sub>O<sub>3</sub>, e.g. yttria partially stabilized zirconia (YSZ). The active DB reacted with Al from superalloy substrate and coating, and transformed into sandwich structured  $\alpha$ -Al<sub>2</sub>O<sub>3</sub>/metal/ $\alpha$ -Al<sub>2</sub>O<sub>3</sub> during service or post vacuum annealing, and therefore was able to suppress interdiffusion between substrate and coating effectively while possessed excellent adherent [29].

The CTEs of nanocomposite Ni + CrAlXN (X is one or more reactive elements, such as Y, Si, Hf) coatings are higher than MCrAlY coatings, so the cyclic oxidation resistance of the nanocomposite coatings on superalloy is better than MCrAlY coatings [30–33]. And, hcp-AlN was found to be useful as DB for nanocomposite Ni + CrAlXN coatings [31]. The AlN DB not only prohibited the coating/substrate interdiffusion, but also promoted formation of exclusive  $\alpha$ -Al<sub>2</sub>O<sub>3</sub>. In the present work, microstructural stability of AlN DB, which is important for long-term performance, for nanocomposite Ni + CrAlYSiHfN coating on single crystal superalloy at 1000 and 1100 °C will be investigated.

## 2. Experimental procedures

Nickel based superalloy K417G, with nominal composition of Ni – (8.5–9.5) Cr – (4.8–5.7) Al – (9.0–11.0) Co – (4.1–4.7) Ti – (0.6–0.9) V – (2.5–3.5) Mo, was used as substrate. Specimens were cut into dimensions of 15 × 10 × 2 mm<sup>3</sup> and ground to 2000 grit SiC paper, which was followed by polishing with diamond paste (2.5  $\mu$ m). The polished samples were then ultrasonically cleaned in ethanol and acetone.

At the first step, AlN diffusion barrier was fabricated by filtered vacuum arc evaporation with an aluminum target (99.99% wt.%) [33]. Then, the Ni + CrAlYSiHfN nanocomposite coating was deposited by a middle-frequency planar reactive magnetron sputtering system with two same target of Ni-25Cr-10Al-0.5Y-0.5Si-0.5Hf (in wt.%). Prior to depositing the nanocomposite coating at the second step, the base vacuum was pumped below 6.0 × 10<sup>-3</sup> Pa. During deposition of the Ni + CrAlYSiHfN composite coatings, N<sub>2</sub> (99.99%) and Ar (99.99%) were introduced to the chamber through two independent mass-flow controllers. The detailed deposition parameters are presented in Table 1.

To study the evolution and degradation mechanism of AlN diffusion barrier during exposure at high temperature, the coated samples were annealed at 1000 °C and 1100 °C in air. The AlN interlayer cross-section of both annealed and as-deposited specimens were observed by scanning electron microscopy (SEM). The as-deposited and heat-treated samples in cross-section were examined by a Jeol JEM 2010F transmission electron microscopy (TEM) equipped with a Tracor EDS. A combination of scanning transmission electron microscopy (STEM), selected area diffraction (SAM), high resolution electron microscopy (HREM) and EDS micro-analysis was performed by the transmission electron microscope.

## 3. Results

Fig. 1 shows the cross-sectional back-scattered electron (BSE) images of the as-deposited Ni + CrAlYSiHfN/AlN nanocomposite coating system. A uniform AlN diffusion barrier, about 1  $\mu$ m in

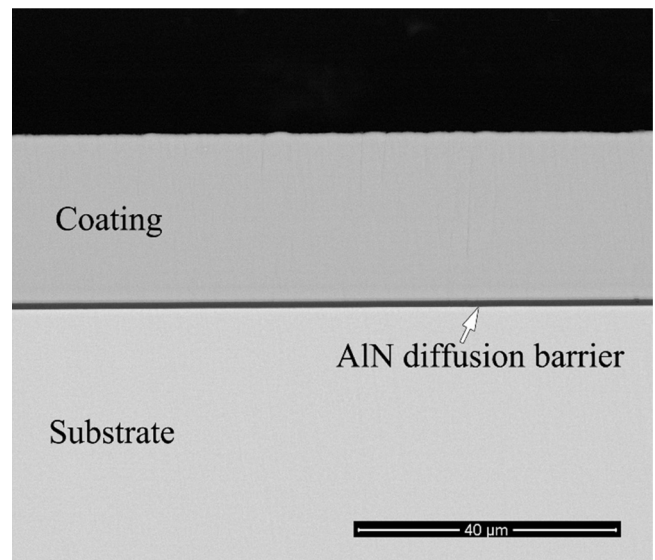


Fig. 1. Cross-sectional BSE image of the as-deposited nanocomposite coating system showing both layers: AlN diffusion barrier and Ni + CrAlYSiHfN top coating.

thickness, was deposited between the Ni + (CrAlYSiHf)N composite coating and the superalloy.

Fig. 2a displays the cross-sectional STEM images of the as-deposited AlN interlayer. It can be seen that the interface between the AlN interlayer and the substrate/coating was rather clear and smooth. Fig. 2b shows the diffraction ring of the as-deposited AlN interlayer. The fragmentary rings indicated preferential growth of fine AlN grains occurred possibly during deposition. Columnar grains with preferential crystalline orientation have been found frequently in magnetron sputtered films. The HREM image (Fig. 2c) shows an amorphous AlN layer about 4 nm thick existed (marked area), and the size of AlN grains is in the size of nano-meters.

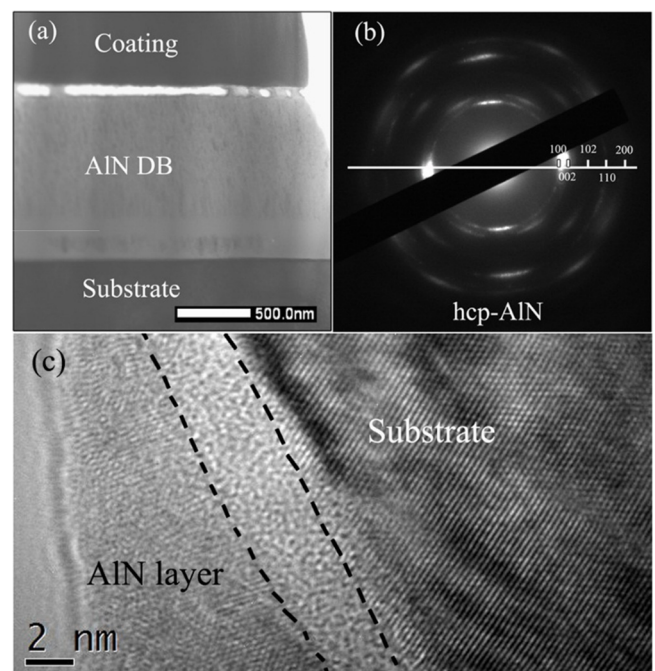


Fig. 2. (a) STEM images of the as-deposited AlN DB, (b) electron diffraction rings of the as-deposited AlN DB, (c) the HREM image of the interface between the AlN DB and K417G substrate.

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