



## Study on behavior of NiAl coating with different Ni/Al ratios



Qiong Wu, Shusuo Li, Yue Ma, Shengkai Gong\*

School of Materials Science and Engineering, Beihang University, Beijing 100191, China

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### ABSTRACT

$\beta$ -NiAl coatings with different Ni/Al ratios were deposited on K403 superalloy substrates via magnetron sputtering. The phase transformation and diffusion phenomenon of the NiAl/Ni-based superalloy system after vacuum annealing at 900 and 1000 °C were analyzed using X-Ray diffraction (XRD), field emission scanning electron microscope (FE-SEM) and energy dispersive X-ray spectrometry (EDS). The effect of coating concentrations on the outward diffusion behavior of substrate elements was discussed. The high Cr concentrations in the Al-rich NiAl coatings were caused by the intense interdiffusion between Al and Cr. The Ti, W and Mo partitioned to  $\gamma'$ -Ni<sub>3</sub>Al in the coatings. Several possible reasons for the formation of  $\gamma'$ -Ni<sub>3</sub>Al at the surface of Ni-rich NiAl coating were identified, including: diffusion behavior of W and Mo in  $\beta$ -NiAl, destabilizing effect of substrate elements on  $\beta$ -NiAl, and diffusion rates of Ni and Al in  $\beta$ -NiAl. The volume change in  $\beta \Rightarrow \gamma'$  transformation process shows Ni uphill diffused to the  $\gamma'$ -Ni<sub>3</sub>Al islands at the surface of Ni-rich NiAl coatings. The IDZ (interdiffusion zone) thickness and precipitates in IDZ were related to the Al initial concentrations in the coatings.

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### 1. Introduction

The advanced turbine engine requires higher operating temperatures for more complete combustion of fuels to increase efficiency. To protect engine components made from Ni-based superalloys from thermo-mechanical failure, thermal barrier coating systems (TBCs) have been developed to improve their oxidation resistance at elevated temperatures. The bond coating (BC), one of the essential parts of TBCs, is deposited on superalloy substrates to mediate the differences in thermal expansion and to form a dense and protective  $\alpha$ -Al<sub>2</sub>O<sub>3</sub> scale (known as thermally grown oxide (TGO)) as well.  $\beta$ -NiAl has attracted increasing attention because of its potential application as metallic protective coatings to operate at temperatures above 1150 °C [1,2], while the traditional MCrAlY coatings can only withstand temperatures no more than 1150 °C [3–9]. However, the  $\beta$ -NiAl bond layer tends to suffer from Al depletion, leading to a phase transformation from  $\beta$ -NiAl to  $\gamma'$ -Ni<sub>3</sub>Al, which inevitably causes volume changes and reductions in anti-oxidation properties [10–16]. Formation of the  $\gamma'$ -Ni<sub>3</sub>Al phase was observed at both TGO/BC interface and BC/substrate interface [17–24]. In the former case, surface “rumpling” of the bond coating is induced [24,25], which in turn, causes spallation of the  $\alpha$ -Al<sub>2</sub>O<sub>3</sub>

scales. In the latter case, structural integrity is deteriorated, which degrades thermo-mechanical properties of the superalloys [26,27]. In addition, the interdiffusion causes the formation of precipitates at the BC/superalloy interface, which declines the mechanical properties of the superalloys.

Most studies about the behavior of  $\beta$ -based coating focused on Pt modified Al coating [28–30] and stoichiometric Ni–Al coating. Angenete et al. [19–23] systematically compared the properties of four  $\beta$ -based Pt modified Al coatings (with different Ni/Al ratios) on a single crystal superalloy, including the transformation of  $\beta$ -NiAl to  $\gamma'$ -Ni<sub>3</sub>Al in the coatings, the interdiffusion of coating with the substrate, oxidation behavior, etc. Wu et al. [31] investigated the effects of bond coat composition (three BC systems: two  $\beta$ -structured Pt–Al types and a  $\gamma + \gamma'$  type) on the interstitial degradation mechanisms of TBCs. Pint et al. [32] studied the effect of Pt–Al coating composition on the cyclic oxidation lifetime of TBCs. The study of Ni–Al coating concentrated on the oxidation and interdiffusion behavior of stoichiometric NiAl coatings with reactive elements, such as Y, Zr, Dy and Hf [33–37], or some other additives, such as Ir [38], Cr [39].

However, most of the microstructure evolutions of the  $\beta$ -based coatings at high temperature were characterized under oxidation, the effects of oxidation and/or interdiffusion on coating behavior are hard to distinguish, especially in terms of surface microstructure evolutions. In addition, deviation from the NiAl stoichiometry is inevitable during the service of the coating, leading to its distinct diffusion and phase transition behavior. This area, however,

\* Corresponding author. Tel.: +86 10 82339003; fax: +86 10 82338200.

E-mail addresses: [queong@126.com](mailto:queong@126.com), [queong@gmail.com](mailto:queong@gmail.com) (Q. Wu), [gongsk@buaa.edu.cn](mailto:gongsk@buaa.edu.cn) (S. Gong).

**Table 1**  
Nominal composition of K403.

Composition	Ni	Cr	Al	Ti	Co	Mo	W	C	Zr	B	Ce
Wt%	66.41	11	5.6	2.6	4.7	4.2	5.2	0.15	0.1	0.02	0.02
At%	63.84	11.94	11.71	3.06	4.50	2.47	1.60	0.70	0.06	0.10	0.01

has received considerably less attention. In this work,  $\beta$ -NiAl coatings with various Ni/Al ratios were deposited on a Ni-based superalloy using magnetron sputtering. Then the coatings were annealed under vacuum at high temperature to exclude the role of  $\alpha$ -Al<sub>2</sub>O<sub>3</sub> growth. The effects of interdiffusion on phase transitions and especially on the surface structural evolutions are highlighted.

## 2. Experimental procedure

The coatings were deposited via direct current (DC) magnetron sputtering. The nominal compositions of the three binary Ni–Al targets are Al contents in 40 at%, 50 at% and 55 at% with balanced Ni. The Ni–Al alloys were separately melted in an arc furnace under argon atmosphere using a tungsten electrode and re-melted four times. Then the ingots were wire cut into  $\varnothing 60 \times 4$  mm targets. The coatings were deposited on Ni-based superalloy as well as glass substrates. The nominal composition of the superalloy substrate K403 is shown in Table 1. Prior to the deposition, the chamber was evacuated to a residual pressure of about  $8.0 \times 10^{-4}$  Pa. The pressure of the working gas Argon was set at 1.3 Pa, and the deposition power was set at 91 W. The  $\sim 2$   $\mu$ m thick coatings were deposited for 24 min and 10  $\mu$ m thick for 2 h. The substrates were not intentionally heated during the depositions. The as-deposited coatings were then annealed under vacuum ( $<3.0 \times 10^{-3}$  Pa) at 900 °C and 1000 °C for times ranging from 1 to 100 h in a tube furnace. The microstructures of the coatings were investigated

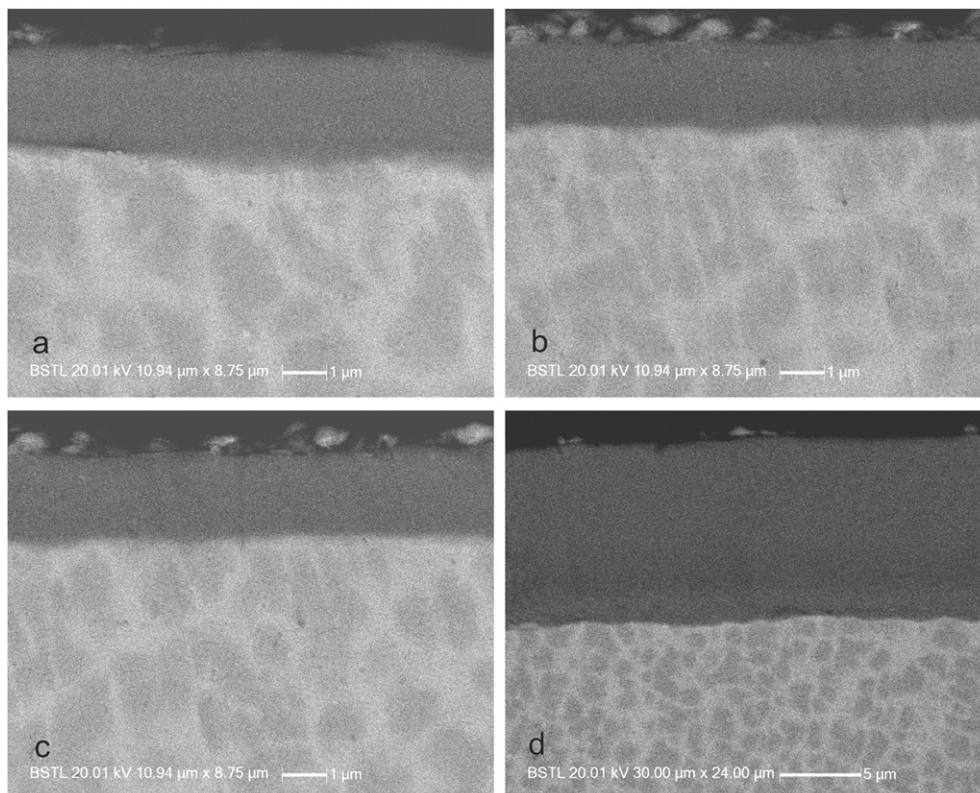
using emission scanning electron microscope (FE-SEM, Apollo 300, Camscan, Cambridge, England) equipped with an Oxford energy dispersive X-ray spectrometry system (EDS). The structures of the coatings and substrates were determined via X-Ray diffraction (XRD).

## 3. Results

### 3.1. As-deposited coatings

Fig. 1 shows the backscattered electron images of the three 2  $\mu$ m thick  $\beta$ -NiAl coatings and 10  $\mu$ m thick Ni-rich NiAl coating in the as-deposited condition. The interfaces between coatings and substrates were clearly distinguished. The average compositions of the targets and coatings as identified by EDS are shown in Table 2. The compositions of the coatings were nearly the same with the targets.

Fig. 2 shows the XRD patterns of the coatings (2  $\mu$ m) as deposited on glass and Ni-based superalloy substrates. The coatings were deposited on different substrates for comparison. The XRD patterns of the coating on glass show all coatings consisted of  $\beta$  phase. The XRD patterns of the coating on superalloy show the XRD detection depth exceeds 2  $\mu$ m, reaching the substrates. The Ni-rich NiAl coating exhibited (110) preferred orientations, and stoichiometric NiAl exhibited random orientations, while Al-rich NiAl coatings showed weak (111) and (211) preferred orientations. The different crystallographic textures may be relevant to the



**Fig. 1.** Cross-section electron backscatter micrographs of the coatings in the as-deposited condition: 2  $\mu$ m thick  $\beta$ -NiAl coating (a) Ni-rich NiAl; (b) stoichiometric NiAl; (c) Al-rich NiAl coatings, and (d) 10  $\mu$ m thick Ni-rich  $\beta$ -NiAl coating.

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