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## Original Research Article

# Effect of Pd and Hf co-doping of aluminide coatings on pure nickel and CMSX-4 nickel superalloy



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## ARTICLE INFO

## Article history:

Received 13 February 2018

Accepted 26 May 2018

Available online

## Keywords:

Aluminide coatings

Palladium and hafnium co-doping

TEM analysis

## ABSTRACT

Microstructure of palladium and hafnium co-doped aluminide coatings deposited on pure nickel and CMSX4 nickel super alloy by the CVD method was examined by the SEM and TEM methods. Both coatings have a double layer structure: additive and interdiffusion zones. The additive zone is formed by the hafnium doped  $\beta$ -(Ni,Pd)Al phase. The interdiffusion zone on pure nickel contains the palladium and hafnium doped  $\gamma'$ -Ni<sub>3</sub>Al phase, whereas that on CMSX-4 superalloy the hafnium doped  $\beta$ -(Ni,Pd)Al phase with precipitations of Topologically Closed-Pack phases ( $\mu$  and  $\sigma$ ) and Al<sub>2</sub>O<sub>3</sub> at adhesive/interdiffusion zones interfaces. Palladium is distributed uniformly in whole coatings. Hafnium forms precipitates that are situated in a Hf-rich belt. In both coatings this belt is in the additive layer, near the line of porosity and Al<sub>2</sub>O<sub>3</sub> precipitates. Palladium and hafnium modified aluminide coatings show better oxidation resistance than those modified only with palladium.

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## 1. Introduction

Aluminide coatings, characterized by low density and high both melting point and oxidation resistance, are applied as protective coatings for superalloys or bond coats in thermal barrier coatings (TBC) [1]. Aluminium from the coatings reacts with oxygen from the air forming an  $\alpha$ -Al<sub>2</sub>O<sub>3</sub> at their surface, which protects the substrate from oxidation and corrosion [2]. However, oxidation of coatings degrades them through deformation and internal weakening. Rising of oxidation resistance of such coatings calls for slowing down the oxide

scale growth and improvement of their adherence to the substrate, what was achieved by alloying them with small amount of noble metals (platinum [3], palladium [4]) or reactive elements (hafnium [5], zirconium [6]).

Platinum introduced to aluminide coatings was shown to be effective not only in reducing of the oxide growth rate and scale adherence, but also in preventing a  $\beta$  to  $\gamma'$  phase transformation eliminating chromium-rich precipitates from the outer layer and diffusion of refractory elements from the substrate [7]. Palladium additions to these coatings were found to act in similar way, as proved Alperine et al. and Hong et al. [4,8]. Yavorska et al [9] showed that platinum or palladium

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<https://doi.org/10.1016/j.acme.2018.05.007>

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modified aluminide coatings deposited on Inconel 713 LC and CMSX 4 superalloys consist mainly of the (Ni,Pt)Al phase and (Ni,Pd)Al phase, respectively. Simultaneously, higher total content of substrate alloying elements such as chromium, molybdenum, tungsten, titanium, tantalum and rhenium in CMSX 4, as compared to the content in Inconel 713 LC, resulted in formation of thinner alumina coatings. The noble metals modification increased the outer zone hardness, what could be ascribed to the presence of  $M_{23}C_6$  carbides in Inconel 713 LC and  $\sigma$  and  $\mu$  phases in CMSX 4. However, it is the hafnium which is the most efficient dopant in reducing the oxide scale growth rate [6]. Hafnium segregates to the scale grain boundaries and alloy-scale interface. Its out-diffusion to the oxide scale surface along oxide grain boundaries is slowed down by the large atomic radii. This slow diffusion stands against the outward diffusion of aluminium and inward diffusion of oxygen [10,11]. In the effect the scale adhesion is bettered and the growth rate of the scale is reduced.

The idea of using more than one dopant, so called 'co-doping', has become the main strategy for improvement of scale adhesion and coating lifetimes [12,13]. The surface of platinum and palladium modified coatings deposited on Inconel 738LC superalloy is less rumpled than that modified just with one of them. Moreover, the coating shows better oxidation resistance than any of the single-doped one [8]. The last, but not least important is that the platinum and palladium modified aluminide coatings exhibit significantly higher  $\beta$ -NiAl phase stability as well as maintain more stable alumina oxide layer [12]. Until now, a number of pairs of co-dopants has been analyzed including Pt + Pd, Y + La, Dy + Hf, Y + Hf, La + Hf, Hf + Zr, or Y + La [14,15]. In all cases oxidation resistance of the co-doped coatings turned out better than the respective single-doped ones. However, the palladium and hafnium co-doping in coatings deposited by the CVD method has not been investigated yet.

Therefore, the aim of this paper is to analyze the microstructure of palladium and hafnium co-doped aluminide coatings deposited on pure nickel and CMSX-4 nickel based superalloy as well as to assess their oxidation resistance.

## 2. Experimental

Palladium layers were electrodeposited on nickel (of technical purity) and nickel-based superalloy CMSX-4 (Ni-61.5, Cr-6.5, Mo-0.6, Ta-6.5, Al-5.6, Ti-1, Co-9, W-6, He-0.1, Re-3%wt.). The specimens were cut from the rod and abraded with the sandpaper (grit 320, 500, 800 and 1000). Prior to the electrodeposition process, the specimens were degreased ultrasonically in acetone ( $t = 5$  min) and etched in a hydrochloric acid aqueous solution (35 wt.%,  $t = 1$  min). The palladium layer on the nickel substrate was produced according to the two-step procedure. The first one was the acidic palladium strike process. The thin ( $\sim 50$  nm), well-adherent layer was obtained after 90 s. The current density was equal to  $5 \text{ mA cm}^{-2}$ ,  $T = 40^\circ\text{C}$ . The composition of the bath is given in Table 1. The pH of the bath was set at the level of 3.9. The bath was stirred during the electrodeposition by the compressed air [16].

The specimens were then rinsed with deionised water and the  $3 \mu\text{m}$  thick Pd coatings were electrodeposited. The current

density was equal to  $10 \text{ mA cm}^{-2}$ ,  $T = 55^\circ\text{C}$ ,  $t = 11$  min. The chemical composition of the bath is given in Table 2, the pH of the bath was set at the level of 12. The bath was stirred during the electrodeposition by using the compressed air [17].

Next, the specimens were rinsed with deionised water and dried in the air. The coatings on the CMSX-4 super alloy were deposited according to the three-step procedure. Degreased and etched specimens were electroplated with nickel. The specimens were anodically etched in the plating bath for 2 min at  $3 \text{ A dm}^{-2}$ , rinsed in the deionised water and electroplated at the same current density for 6 min,  $T = 20^\circ\text{C}$ . The chemical composition of the bath is given in Table 3 [18].

The next two steps were the same as for the nickel specimens i.e. the thin Pd coatings were produced and finally the thick  $3 \mu\text{m}$  coatings. Then, the aluminide coatings were deposited on both samples by the CVD method [19,20]. The BPXPR0325S equipment manufactured by the IonBond company was applied. The CVD process consists of four stages: heating from the room temperature up to  $1040^\circ\text{C}$ , aluminizing at  $1040^\circ\text{C}$  for 360 min, hafnizing with aluminizing at  $1040^\circ\text{C}$  for 360 min and cooling.

Microstructure, chemical and phase composition of the coatings were investigated using scanning and transmission electron microscopy methods (SEM, TEM), while their local chemical composition was measured using energy dispersive spectroscopy (EDS) technique. The oxidation resistance of the CMSX-4 sample was analyzed by placing the sample in the furnace, heating it up to  $1100^\circ\text{C}$  and maintaining it at that temperature for 20 h, followed by slow cooling to the room temperature. After each such cycle the sample was weighed and inspected.

**Table 1 – The chemical composition of the Pd strike bath.**

Constituent	Concentration
Palladium(II) chloride	$1.6 \text{ g dm}^{-3}$
1,2-Diaminopropane	$5.4 \text{ cm}^3 \text{ dm}^{-3}$
Glacial acetic acid	$23.3 \text{ cm}^3 \text{ dm}^{-3}$
Sodium chloride	$60 \text{ g dm}^{-3}$
Deionised water	to $1 \text{ dm}^3$

**Table 2 – The chemical composition of the Pd plating bath.**

Constituent	Concentration
Palladium(II) chloride	$13.3 \text{ g dm}^{-3}$
Diethylenetriamine	$16.2 \text{ cm}^3 \text{ dm}^{-3}$
Phosphate buffer	to $1 \text{ dm}^3$

**Table 3 – The chemical composition of the Ni plating bath.**

Constituent	Concentration
Nickel(II) chloride ( $\text{NiCl}_2 \cdot 6\text{H}_2\text{O}$ )	$240 \text{ g dm}^{-3}$
Hydrochloric acid (35 wt.%)	$31 \text{ cm}^3 \text{ dm}^{-3}$
Deionised water	to $1 \text{ dm}^3$

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