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

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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 β-(Ni,Pd)Al phase. The interdiffusion zone on pure nickel contains the palladium and hafnium doped γ'-Ni$_3$Al phase, whereas that on CMSX-4 superalloy the hafnium doped β-(Ni,Pd)Al phase with precipitations of Topologically Closed-Pack phases (μ and σ) and Al$_2$O$_3$ 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$_2$O$_3$ 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 α-Al$_2$O$_3$ 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 β to γ' 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 all [9] showed that platinum or palladium

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 σ and μ 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 that 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 β-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 electrode-position 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 (∼50 nm), well-adherent layer was obtained after 90 s. The current density was equal to 5 mA cm^{-2}, $T = 40$ °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 μm thick Pd coatings were electrodeposited. The current density was equal to 10 mA cm^{-2}, $T = 55$ °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 A dm^{-2}, rinsed in the deionised water and electroplated at the same current density for 6 min, $T = 20$ °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 μm 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 °C, aluminizing at 1040 °C for 360 min, hafnizing with aluminizing at 1040 °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 °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 g dm^{-3}
1,2-Diaminopropane	5.4 cm^3 dm^{-3}
Glacial acetic acid	23.3 cm^3 dm^{-3}
Sodium chloride	60 g dm^{-3}
Deionised water	to 1 dm^3

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

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

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

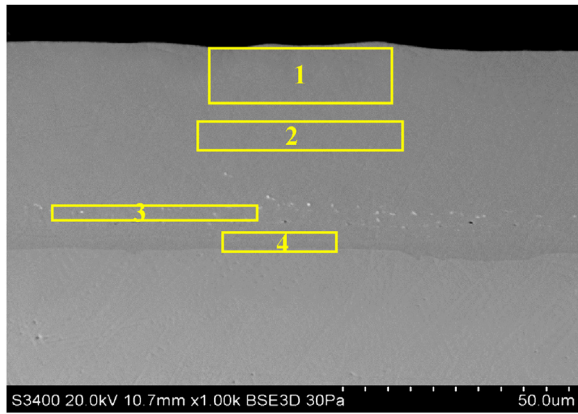| Constituent	Concentration
Nickel(II) chloride ($NiCl_2 \cdot 6H_2O$)	240 g dm^{-3}
Hydrochloric acid (35 wt.%)	31 cm^3 dm^{-3}
Deionised water	to 1 dm^3

Fig. 1 – SEM microstructure on cross-section of palladium and hafnium modified aluminide coating deposited on nickel substrate.

Table 4 – Chemical composition on cross-section of palladium and hafnium modified aluminide coating deposited on the nickel substrate (in at.%).

Spot	Al	Ni	Pd	Hf
1	35.8 ± 0.3	59.3 ± 0.5	4.8 ± 0.1	0.1 ± 0.1
2	33.2 ± 0.3	62.8 ± 0.5	3.7 ± 0.1	0.3 ± 0.1
3	23.4 ± 0.3	75.7 ± 0.5	0.8 ± 0.1	0.1 ± 0.1
4	–	100	–	–

3. Results

3.1. Palladium and hafnium modified aluminide coating on pure nickel

Palladium and hafnium modified aluminide coating on pure nickel has a double-zone structure (Fig. 1, Table 4). In the first, additive zone, on the top of the coating, the proportion of Ni do

Al corresponds to the β-NiAl phase (Table 4, Point 1). Moreover, some palladium (4.8 at.%) and hafnium (0.1 at.%) was identified. This indicates formation of the hafnium doped β-(Ni,Pd) Al phase. The thickness of this zone is ~45 μm. The second, interdiffusion zone, situated below is much thinner (~3 μm). Its chemical composition suggests formation of the palladium and hafnium doped γ'-Ni_3Al phase (Table 4, Point 3). Bright, hafnium rich precipitates were observed in the additive zone (Fig. 2, Points 1,2). EDS spectrum of particle 1 and 2 shows presence of aluminium, nickel, palladium and hafnium peaks (Fig. 2b, c). The nickel substrate/hafnium doped γ'-Ni_3Al as well as the hafnium doped γ'-Ni_3Al/hafnium doped β-(Ni,Pd)Al interfaces were relatively flat (Fig. 3a). Kirkendall-like porosity was observed within the hafnium doped β-(Ni,Pd)Al near hafnium doped γ'-Ni_3Al phase (Fig. 3b). Kirkendall-like pores form because of the unbalanced diffusion of elements into and out of the alloy. The area of significant porosity is formed between additive and interdiffusion zone of the aluminide coating. According to Kirkendall–Frenkel theory, there is an unbalanced flow of nickel and aluminium atoms in the diffusion zone. The value of nickel diffusion coefficient is bigger than aluminium ($D_{Ni} > D_{Al}$). The unbalanced flux of nickel and aluminium atoms results in the differences in microvolume and causes stress in the diffusion zone. The microvolume is reduced in the area of higher nickel concentration and vacancies are formed. When the number of vacancies are big, vacancies coagulate, pores are formed and Kirkendall porosity is generated [21]. The globular and plate-like Hf-rich precipitates (0.1–1.7 μm) were found co-existing with a layer of porosity or oxides in the hafnium doped β-(Ni,Pd)Al phase (Fig. 3b). Fig. 4 shows the uniform distribution of nickel, aluminium and palladium in the first zone, on the top of the coating, confirming the presence of the (Ni,Pd)Al phase. The presence of oxygen and aluminium in the light contrast particles allowed to identify them as Al_2O_3 (Fig. 4). The globular and needle-like precipitates showed only presence of hafnium peaks, but in such materials the oxygen lines could be easily absorbed by the surrounding material. Therefore, they

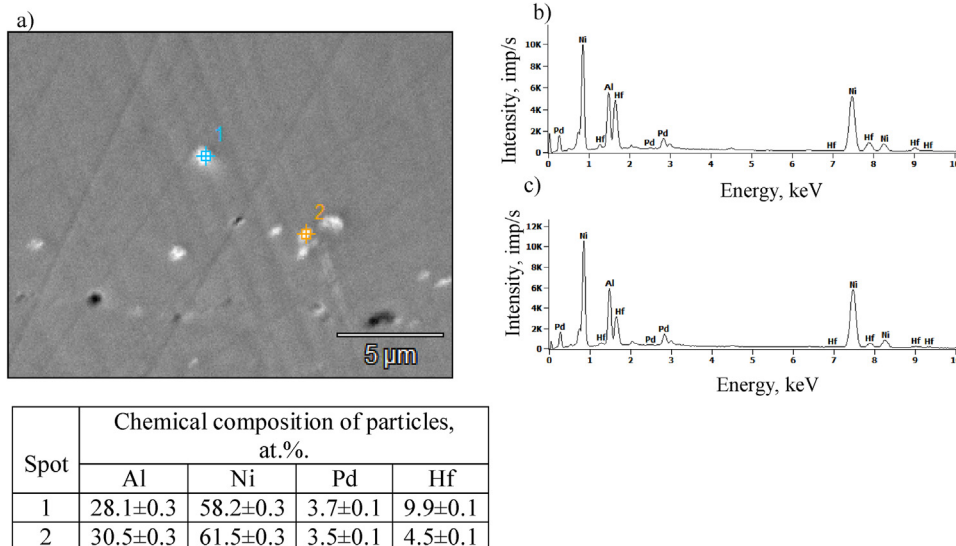| Spot	Chemical composition of particles, at.%.			
	Al	Ni	Pd	Hf
1	28.1±0.3	58.2±0.3	3.7±0.1	9.9±0.1
2	30.5±0.3	61.5±0.3	3.5±0.1	4.5±0.1

Fig. 2 – SEM microstructure of additive zone (a) with EDS spectrum of particle 1 (b) and particle 2 (c) of palladium and hafnium modified aluminide coating on the nickel substrate and their chemical composition.
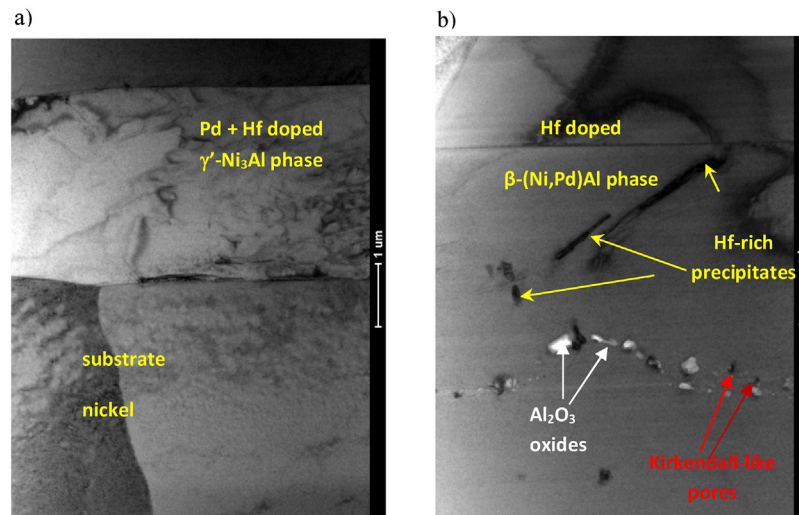
Fig. 3 – TEM microstructure of Pd and Hf modified aluminide coating on nickel: (a) substrate/Hf doped γ′-Ni₃Al(Pd) interface and (b) Hf-rich precipitates in Hf doped β-(Ni,Pd)Al.
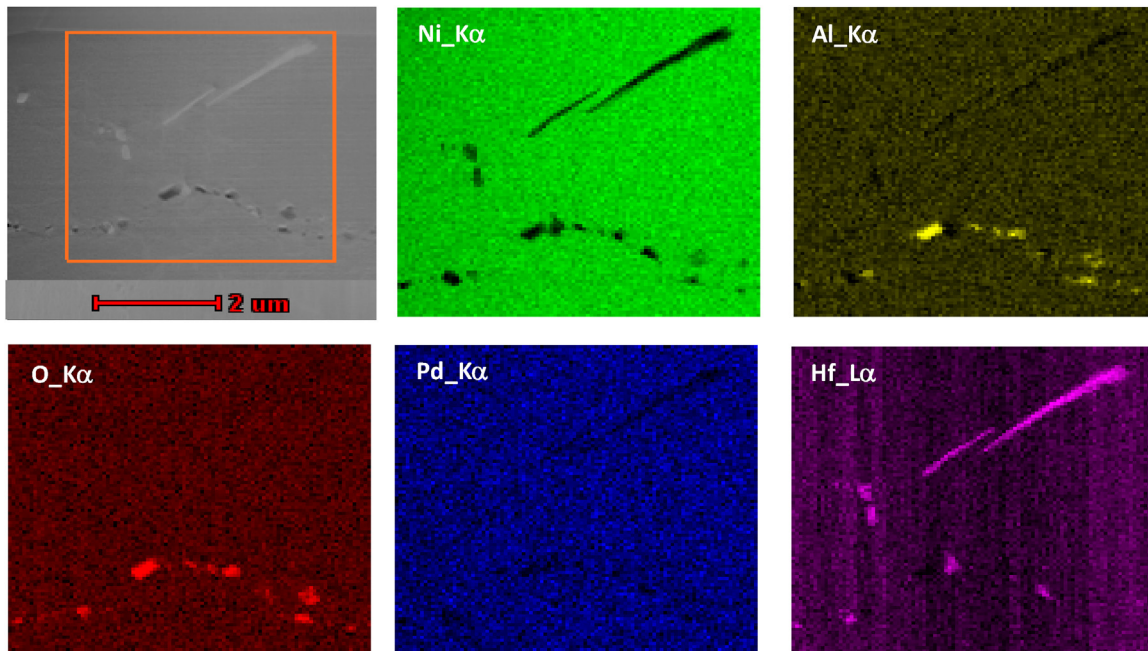

Fig. 4 – STEM-HAADF microstructure of cross-section of Pd and Hf modified aluminide coating on nickel (a) with corresponding maps presenting distribution of Al, Ni, Hf, Pd and O.

should be described either as hafnium or hafnium oxide precipitates. As the oxygen content is low, the presence of pure hafnium is more probable.

Fig. 5 shows the X-ray diffraction results for the palladium and hafnium modified aluminide coating deposited on the nickel substrate. The dashed line presents standard 2θ angles of the NiAl phase. As compared with the standard NiAl, the leftward-shifting trend for the palladium and hafnium modified coating is significant. It indicates, that the NiAl crystal lattice had been enlarged due to the co-doping of Pd and Hf. The shifting behaviour seems to be more evident at higher index crystal planes, because the mismatch at higher indexes is proportional to the basic mismatch of the crystal lattice. The XRD results confirmed the successful doping of Hf in the single phase β-(Ni,Pd)Al coating.

3.2. Palladium and hafnium modified aluminide coating on CMSX 4 superalloy

The palladium and hafnium modified aluminide coating on CMSX 4 superalloy has a double-zone structure, similarly to the one on pure nickel (Fig. 6a). In the first one (18 μm thick),
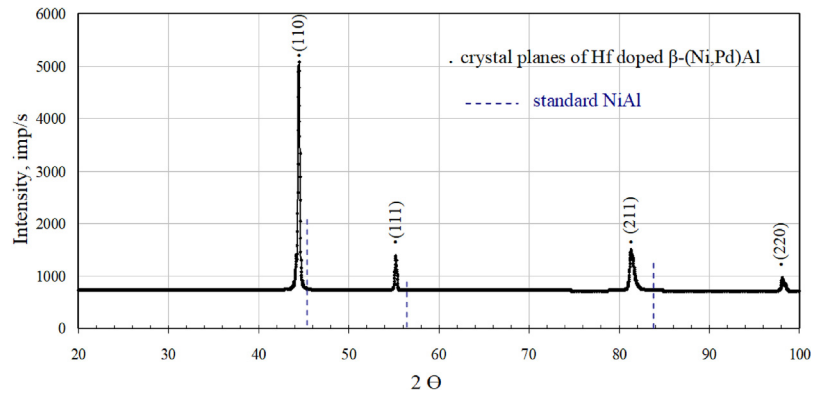
Fig. 5 – XRD diffraction pattern of the palladium and hafnium modified aluminide coating deposited on the nickel substrate.
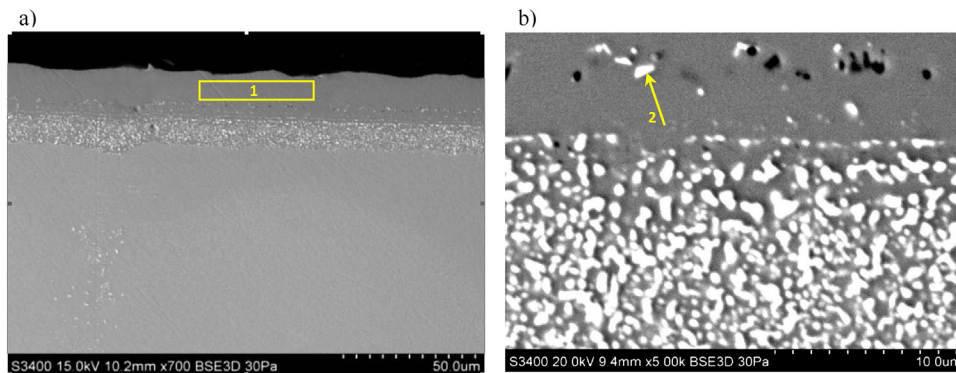

Fig. 6 – SEM microstructure on the cross-section of the palladium and hafnium modified aluminide coating deposited on the CMSX 4 superalloy (a) and Hf-rich precipitates (b).

Table 5 – Chemical composition on the cross-section of the palladium and hafnium modified aluminide coating deposited on CMSX 4 superalloy (in at.%).						
Spot	Al	Cr	Co	Ni	Pd	Hf
1	37.2 ± 0.3	2.2 ± 0.1	5.6 ± 0.1	46.3 ± 0.5	8.6 ± 0.1	<0.1 ± 0.1
2	32 ± 0.3	–	5.8 ± 0.1	48.9 ± 0.5	6.3 ± 0.1	7.0 ± 0.1

called the additive zone and located on the top of the coating, the proportion of Ni do Al corresponds to the β-NiAl phase (Table 5, Point 1), even though palladium (8.6 at.%) and hafnium (<0.1 at.%) were also identified. Such chemical composition indicates the hafnium is doping β-(Ni,Pd)Al phase. In the second, interdiffusion zone (12 μm thick), residing just below the first one, Topologically Closed-Pack phases containing refractory elements were identified in the β-(Ni,Pd)Al matrix, i.e. like in Rh modified aluminide coatings [20]. Bright, hafnium rich precipitates were observed predominantly at the first and second zone interface (Fig. 6b, Table 5, Point 2). The EDS measurements indicated, that they contain up to ∼7.0 at.% hafnium (Table 5).

TEM observations of the cross-section of the palladium and hafnium modified aluminide coating revealed that the Topologically Closed-Pack phases are occupying the substrate/interdiffusion zone interface as well as grains interiors and

boundaries of the β-(Ni,Pd)Al phase matrix (Fig. 7a, b). The EDS spectrum of TCP precipitates indicates that they are built of Ti, Cr, Co, W, Ta and Mo (Fig. 7d). The presence of fine aluminium oxides and Hf-rich precipitates are situated at or near hafnium additive and interdiffusion interface was documented (Fig. 7c).

The maps of local chemical composition revealed a uniform distribution of nickel aluminium and palladium in the first additive zone, on the top of the coating, confirming the presence of (Ni,Pd)Al phase (Fig. 8). The precipitates located at the additive and interdiffusion zone interface or near by are mostly composed either of hafnium, tantalum and to lesser amount of titanium or of aluminium and oxygen. In the latter case it indicates the presence of Al_2O_3 reproducing situation on the nickel substrate. The other precipitates, i.e. those with hafnium showed only occasionally co-presence of oxygen (especially the smaller ones), probably due to strong absorption of a soft oxygen radiation.
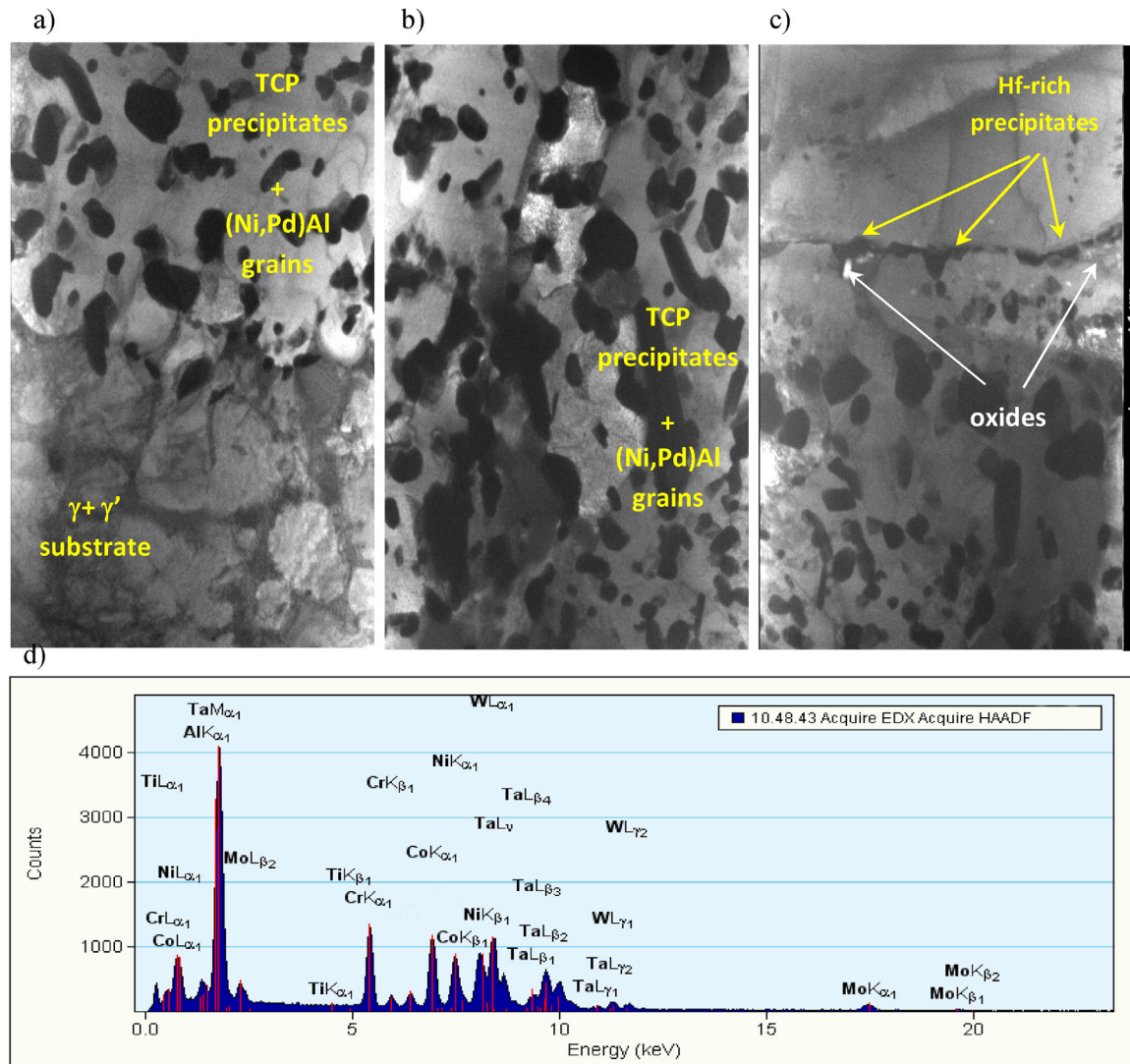
Fig. 7 – TEM microstructure of Pd and Hf modified aluminide coating on CMSX 4: substrate/second zone interface (a), distribution of TCP precipitates in the β-(Ni,Pd)Al phase (b), the first/second zone interface (c) and EDS spectrum of TCP precipitates (d).

3.3. Oxidation behaviour of palladium and palladium and hafnium modified aluminide coating on CMSX 4 superalloy

Palladium and hafnium modified aluminide coatings show better oxidation resistance than those modified only with palladium (Fig. 9). The weight change of two coatings from 0 to 107 h is similar. During the initial 40 h, the weight gain increases significantly. The coatings, exposed to the high temperature, oxidize quickly. Then from 40 h to 107 h of oxidation the weight gain becomes stable. Dense protective oxide layers have been formed on after initial oxidation. Similar oxidation behaviour of PtAl and PtZrAl coatings on single crystalline Ni-based superalloy was observed by Fan et al. [22]. Increasing of the oxidation time from 107 h to 160 h leads to the weight gain (to about 0.3 mg/cm^2) of the palladium and hafnium modified aluminide coatings and leads to weight loss (to about 0.1 mg/cm^2) of the palladium modified aluminide coatings. After 107 h of oxidation, the internal Hf-rich belt in the palladium and hafnium modified aluminide coating

may effectively act as a diffusion barrier to block the inward diffusion of aluminium and the outward diffusion of substrate elements, thus oxidation resistance of the palladium and hafnium modified aluminide coating is better than only palladium modified one. Similar phenomenon was observed by Yang et al. [23]. The constant K_p value of the oxidation rate of the palladium and hafnium modified aluminide coatings obtained using the slope of the fitted lines is close to 7×10^{-4} mg^2/cm^4 h (Fig. 10).

The above proves the existence of a positive synergistic effect between hafnium and palladium as compared with the lifetimes of Pd-modified and Pd + Hf-modified coatings, i.e. similarly to the effect observed for β-NiAl(Cr) coatings [26].

4. Discussion

Palladium and hafnium co-doped aluminide coatings were successfully deposited on pure nickel and CMSX-4 nickel
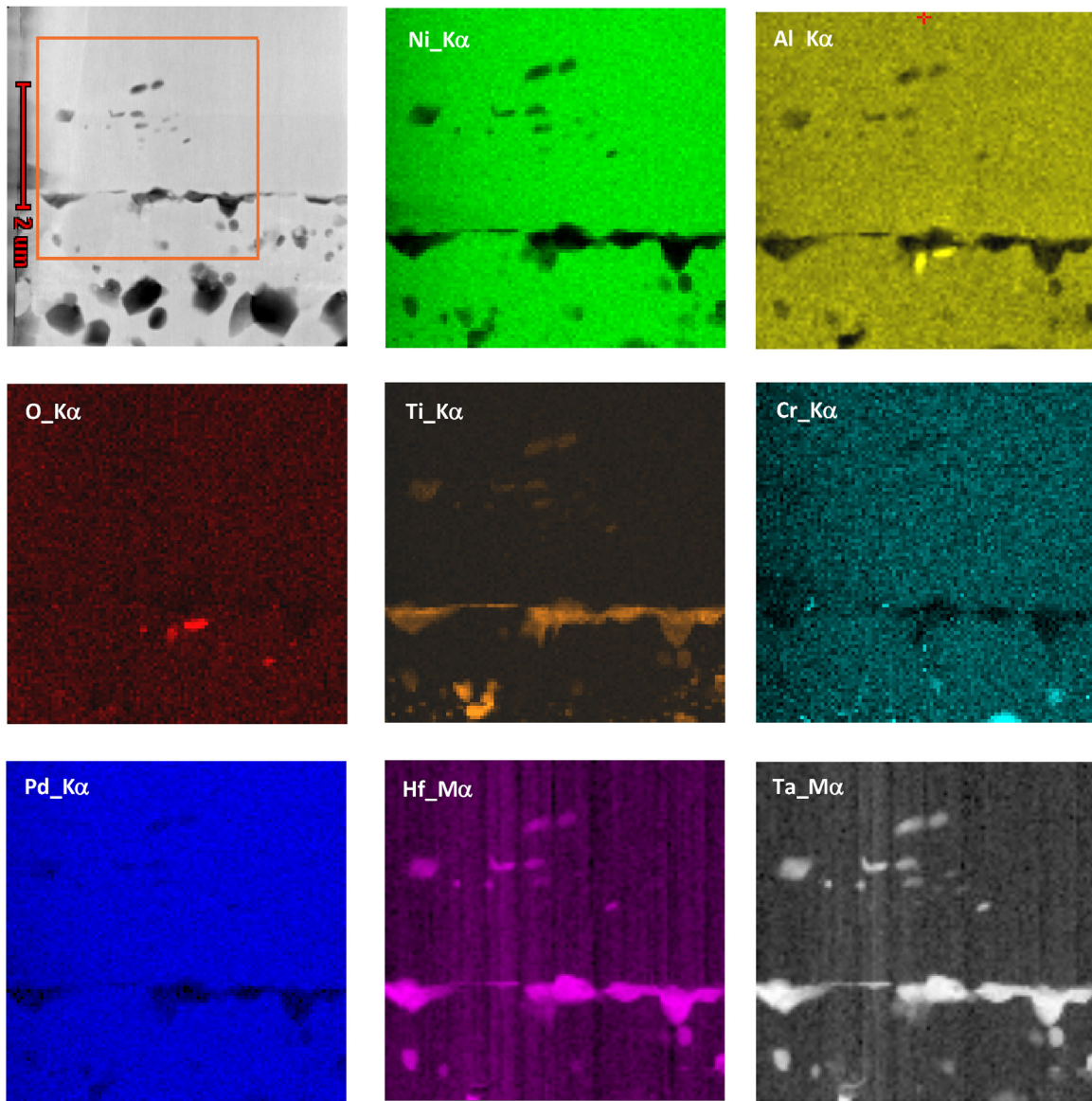
Fig. 8 – STEM-HAADF micrograph of Pd and Hf modified aluminide coating deposited on CMSX 4 with maps presenting distribution of Al, Hf, Ni, Cr, O and Pd.
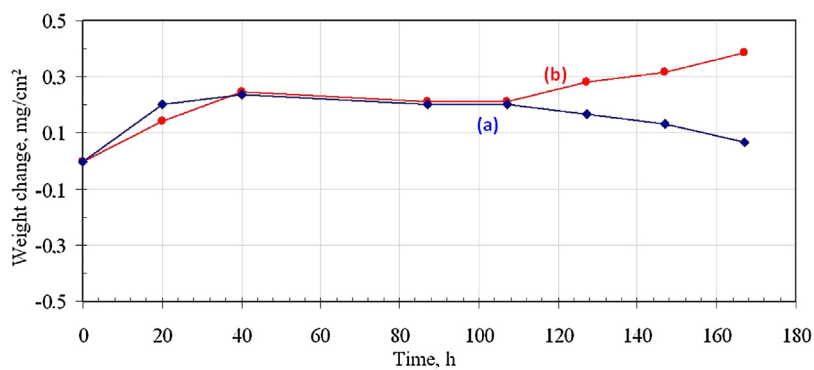

Fig. 9 – Weight change versus oxidation time of the palladium modified (a) and palladium and hafnium modified (b) aluminide coatings deposited on the CMSX 4 superalloy.
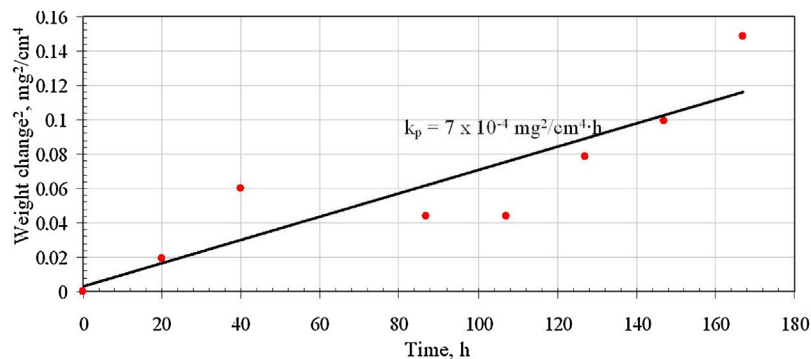
Fig. 10 – Square of weight change versus oxidation time of the palladium and hafnium modified aluminide coatings deposited on the CMSX 4 superalloy.

superalloy. Coatings deposited on both type of substrates consisted of two zones, i.e. additive and interdiffusion one. In each case the additive zone is formed by the hafnium doped β-(Ni,Pd)Al phase. The interdiffusion zone on pure nickel contains the palladium and hafnium doped γ'-Ni_3Al phase, whereas that on CMSX-4 superalloy the hafnium doped β-(Ni,Pd)Al phase. Low solubility of alloying elements in the hafnium doped β-(Ni,Pd)Al results in precipitation of Topologically Closed-Pack phases (μ and σ). Moreover, presence of Al_2O_3 at or in the vicinity of adhesive/interdifusion zones interfaces, i.e. located in the original surface of coated materials may be caused by presence of remnants of native oxide at the treated surfaces. It corresponds to results obtained by Hong et al. [24] for palladium doped aluminide coatings deposited on Inconel 738 LC superalloy by the pack aluminizing method. In this case the outer zone was also composed of one phase β-NiAl containing Pd. Presence of porosity at adhesive/interdifusion interface was attributed to interaction of Pd and Ni during the aluminizing process.

Palladium is distributed uniformly in whole coatings. This phenomenon may be attributed either to full palladium solubility in the β-NiAl phase and high maximum solubility of 10–12 at.% in the γ'-Ni_3Al phase at the temperature range between 1000 and 1150 °C [25]. It is due to the existence of the stable β-PdAl phase and the absence of Pd_3Al phase. Palladium stabilizes the Al-rich β-NiAl phase for longer oxidation lifetimes [26]. 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_2O_3 oxides. In coatings deposited by the 'above-pack' method and modified with Pt and Hf by the electroplating method the hafnium-rich belt was also observed, but it was situated between the additive and interdiffusion zones [23].

The weight gain of the palladium and hafnium modified aluminide coatings deposited on CMSX 4 superalloy is about 0.3 mg/cm² after 160 h of oxidation. This is 0.06 mg/cm² less than the weight gain of the Hf-doped (Ni,Pt)Al coating deposited on the nickel-based single crystal (SC) after 160 h oxidation [23]. This also is 0.36 mg/cm² less than the weight gain of the (Ni,Pt)Al coating deposited on the nickel-based single crystal (SC) after 160 h oxidation [23].

5. Conclusion

- Palladium and hafnium co-doped aluminide coatings on pure nickel and CMSX-4 nickel superalloy. Consisted of two zones, i.e. additive and interdiffusion one.
- The additive zone is formed by the hafnium doped β-(Ni,Pd)Al phase.
- The interdiffusion zone on pure nickel contains the palladium and hafnium doped γ'-Ni_3Al phase, whereas that on CMSX-4 superalloy the hafnium doped β-(Ni,Pd)Al phase.
- Low solubility of alloying elements in the hafnium doped β-(Ni,Pd)Al results in precipitation of Topologically Closed-Pack phases (μ and σ) are formed in the hafnium doped β-(Ni,Pd)Al.
- Palladium is distributed uniformly in whole coatings.
- Hafnium forms precipitates that are situated in a Hf-rich belt.
- Oxidation resistance of the palladium and hafnium modified aluminide coating is better than the palladium modified one.

Acknowledgment

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