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Effect of palladium diffusion in coatings deposited on the nickel based superalloy

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ABSTRACT

Purpose: In this paper the effect of palladium diffusion in coatings deposited on the surface of nickel based superalloy was evaluated.

Design/methodology/approach: The palladium coatings 3 and 7 μm thick were deposited by the electroplating process on Inconel 713 LC Ni-base superalloy. The heat treatment of electroplated coatings at the temperature 1050°C for 2 h under argon atmosphere was performed. The microstructure investigations of the heat treated coatings were conduced by the use of optical microscope (Nikon Epiphot 300) and a scanning electron microscope (Hitachi S-3400N) equipped with an Energy Dispersive Spectroscope EDS (VOYAGER of NORAN INSTRUMENTS). The phase composition was identified by X-ray (ARL X'TRAX) diffractometer. The surface roughness parameter - Ra of heat treated coatings was evaluated by Perthometer S2 MAHR equipment.

Findings: The microstructure of 3 μm thick palladium electroplated coating after diffusion treatment consists of three phases: AlPd2, Ni3Al, Ni0,52Pd0,475. The increase of palladium thickness from 3 to 7 μm does not influence the phase composition of heat treated coatings. Heat treatment of palladium electroplating coatings increases the surface roughness parameter Ra.

Research limitations/implications: The results will be used in the future investigations to explain the influence of palladium on the oxidation resistance of aluminide coatings.

Practical implications: The palladium electroplating coatings after heat treatment and aluminizing process may be used as an alternative to platinum modified aluminide coatings as coatings for turbine blades of aircraft engines.

Originality/value: The paper includes the results of microstructure and surface roughness investigations of palladium electroplating coatings 3 and 7 μm thick after diffusion treatment. Inconel 713 LC; Palladium electroplating; Diffusion treatment; Surface roughness

Keywords:

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MATERIALS

1. Introduction

Nickel-based superalloys are extensively used as gas-turbine engine components. These components need protection against high-temperature oxidation during operation. Diffusion aluminide coatings are widely used for protection of nickel based superalloys from oxidation and hot corrosion [1-3]. Aluminide coatings have been used since the 1950s. However aluminide coatings based on the NiAl phase deposited by the CVD method do not fulfill the requirement of the long term oxidation resistance at high temperature. Pt-modified aluminide coatings affirm the protection of nickel-based blades from oxidative gases even at gas temperature above 1400ºC [4]. Moreover, platinum causes formation of the $PtAl_2$ phase in the coating microstructure [5-6]. The low ductility of $PtAl_2$ and large difference of thermal expansion coefficients between the substrate and platinum causes the coating degradation under the cyclic thermal stress. Alperine et al. [7-8] showed that the palladium modified aluminide coating has higher oxidation and hot corrosion resistance than the conventional aluminide coating. Lehnert et al. [9] showed that palladium modified aluminide coating is a low protector due to the formation of pores by the Kirkendall effect. Monceau et al. [10] investigated the influence of oxygen partial pressure, heating rate and surface treatment on the high temperature oxidation kinetics of Pd-modified aluminide coating, used as a bond coat for partially stabilized zirconium (PSZ) thermal barrier coatings (TBC).

The important factor of palladium coatings after electroplating is the diffusion treatment. The application of diffusion treatment of palladium coatings provides their good adherence [11].

In this paper the effect of palladium diffusion in coatings deposited on the nickel based superalloy after diffusion treatment was investigated. The influence of palladium coating thickness on the depth and microstructure of heat treated coating was analyzed.

2. Experimental procedure

The superalloy used in this study was Inconel 713 LC. Chemical composition of the used superalloy in Table 1.

Table 1.
Chemical composition of the Inconel 713 LC Ni-base superalloy

Elements content, % mas									
Ni	Cr	C	Mo	Nb	Al	Ti	Co	Fe	S
74.7	12	0.05	4.6	1.96	5.7	0.7	0.08	0.19	0.02

Sandblasting process is an usual surface pretreatment before a palladium electroplating process. As analysis of the grit-blasted surface by secondary ion mass spectrometry (SIMS) reveals this pretreatment leads to contamination of the coatings by various impurities, in particular, the alkali and alkaline-earth metals and titanium [12]. These impurities become incorporated into the growing alumina scale during oxidation of palladium-aluminide coatings and adversely affect the oxidation behavior of the aluminide coatings [12]. In this paper surface treatment before palladium electroplating process was grinding.

Process of palladium electroplating was performed in Warsaw Institute of Precision Mechanics. The palladium coatings 3 and 7 μm thick were deposited by electroplating process. The material surface preparation for palladium electroplating includes: degreasing by means of ultrasound, electrolytic etching and surface activation.

The palladium electroplating process was conducted in the bath of palladium chloride $PdCl_2$, sulfamates acid H_2NSO_3, the hydrochloric acid and ammonium chloride NH_4Cl. The samples after electroplating were heat treated at the temperature 1050ºC for 2 h under the argon atmosphere [13].

The palladium electroplating samples after diffusion treatment were cut to study the cross-section. Polished sections were etched by the use of reagent with chemical compositions as follows: 100 ml HNO_3, 4 ml HF, 11 ml H_2O. Microstructure investigations of samples after diffusion treatment were performed by the use of light microscope Nikon 300 and scanning electron microscope (SEM) HITACHI S-3400N equipped with EDS spectrometer. Evaluation of phase composition of the investigated coatings was performed by ARL X'TRA-ray diffractometer, equipped with filtered copper lamp with the voltage of 45 kV and heater current of 40 mA. Measurements were made in the range from 20 to 120º.

The surface roughness parameter - Ra was evaluated by Perthometer S2 MAHR. The average value of surface roughness parameter and a standard deviation were calculated.

3. Results and discussion

The good quality palladium coatings 3 and 7 μm thick were obtained by means of the electroplating process (Figs. 1 a, b).

a)

b)

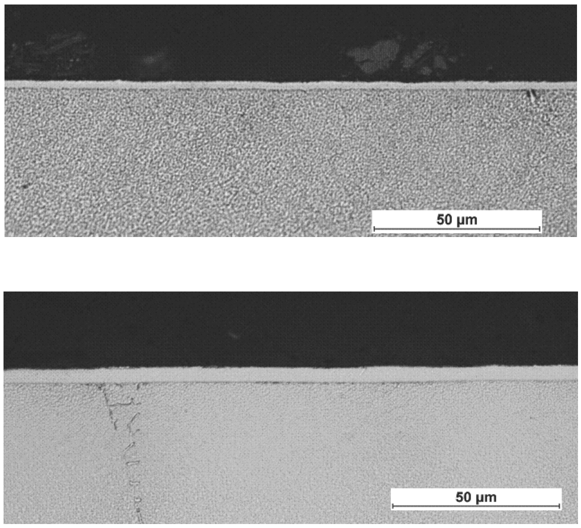


Fig. 1. Palladium electroplating coatings 3 μm (a) and 7 μm (b) thick

The diffusion annealing of palladium electroplating coating 3 μm thick at 1050 ºC for 2 h leads to form of the diffusion zone at the cross section of the coating (Fig. 2). The thickness and microstructure of the heat treated coatings were investigated. The depth of diffusion zone is approximately 10 μm. Analysis of the chemical composition on the cross-section proved the interdiffusion of palladium and substrate elements, e.g. Ni diffused into palladium layer as a result of diffusion zone formation (Fig. 2). The largest palladium content – 11.68 % at. was found at point 1 at the cross-section of heat treated 3 μm thick palladium electroplated coating. Surface morphology characterized a fine-grain structure (Fig. 3a). The high content of aluminum, nickel and palladium was confirmed on the surface of heat treated 3 μm thick electroplated coating (Fig. 3b). The chemical composition on the cross-section of palladium coating after heat treatment showed outward diffusion of nickel, chromium and aluminum and inward diffusion of palladium (Fig. 4).

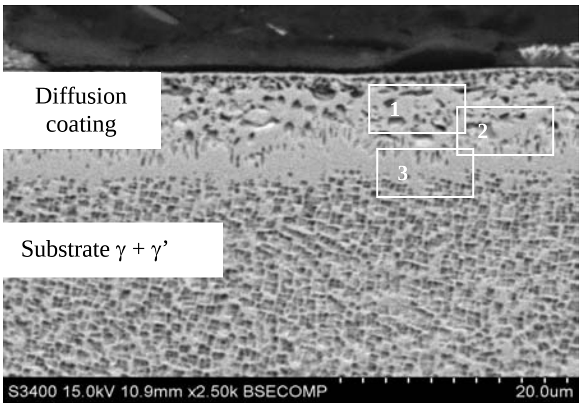


Fig. 2. Cross-section of palladium electroplating coating 3 μm thick after heat treatment process

Table 2.
The results of EDS analysis at the area presented in Fig. 2

Point	Elements content, % at			
	Al	Cr	Ni	Pd
1	6.51	12.01	69.8	11.68
2	3.97	13.49	74.62	7.92
3	5.46	13.73	78.89	1.92

The heat treatment of palladium coating 7 μm thick causes the formation of the diffusion zone. The depth of diffusion zone is about 14 μm (Fig. 5). The largest palladium content – 12.0 % at was found at point 1 at the cross-section of heat treated coating 7 μm thick palladium coating. The chemical composition on the cross-section of 7 μm thick palladium coating after diffusion treatment confirmed outward diffusion of nickel, chromium and aluminum and inward diffusion of palladium. This phenomena is similar to the platinum coating after heat treatment [4].

The phase of Ni_3Al, $AlPd_2$ and $Ni_{0,52}Pd_{0,475}$ were determined in the palladium coating 3 and 7 μm thick after heat treatment process (Fig. 6). Fine grained structure is formed on the surface of the treated sample. The surface is enriched mainly in palladium, chromium and aluminum (Figs. 7 a, b).

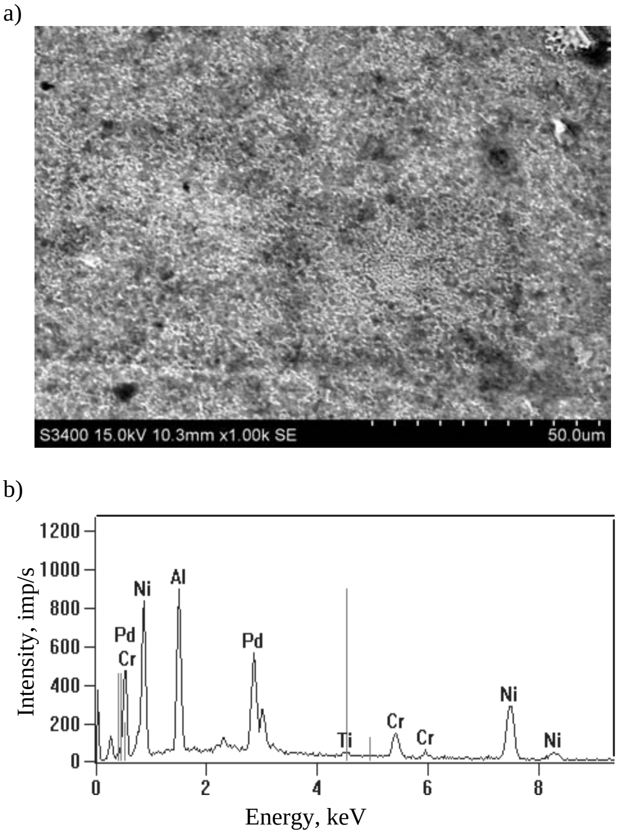


Fig. 3. Microstructure (a) and EDS analysis results (b) of the surface of palladium electroplating coating 3 μm thick after heat treatment process: showing a fine grain structure

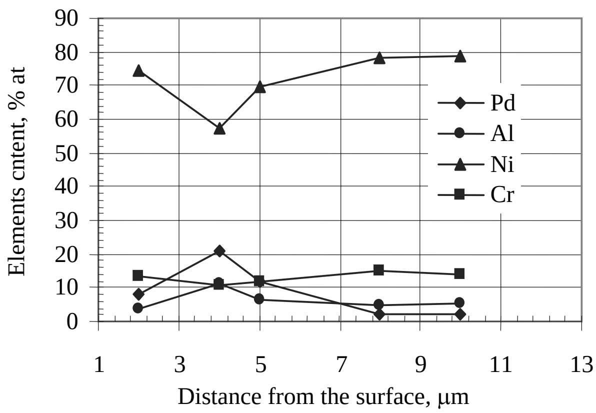


Fig. 4. Chemical composition on the cross section of palladium coating 3 μm thick after heat treatment

The surface roughness parameter of specimens decreases with the increase of palladium deposition thickness (Table 5). Heat treatment of palladium electroplating causes the increase of surface roughness with the increase of palladium thickness. This phenomena may be due to unequal mass flow and internal stresses caused by interdiffusion of palladium and nickel [4].

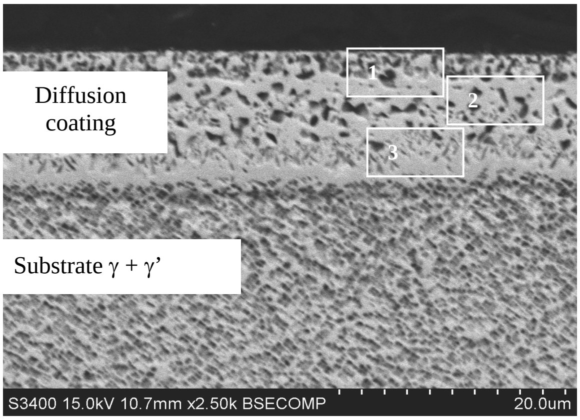


Fig. 5. Cross-section of palladium electroplating coating 7 μm thick after heat treatment process

Table 4.
The results of EDS analysis from area presented in Fig. 5

Point	Elements content, % at			
	Al	Cr	Ni	Pd
1	4.37	12.83	70.8	12.0
2	4.21	9.56	78.24	7.99
3	7.47	12.63	73.26	6.64

Table 5.
Values of surface roughness parameter after palladium electroplating and diffusion treatment

Nickel alloy	Ra				
Inconel 713 LC	0 μm	Palladium electroplating		Diffusion treatment	
		3 μm	7 μm	3 μm	7 μm
Average value	1.080	0.832	0.710	2.034	2.707
Standard deviation	0.0144	0.0179	0.0678	0.033	0.1109

4. Conclusions

Palladium coatings 3 and 7 μm thick were produced by electroplating method. Electroplated samples were heat treated at the temperature 1050 ºC during 2 h under argon atmosphere. After the heat treatment the 3 μm palladium coating diffused for 10 μm whereas 7 μm coating diffused for 14 μm. It was found, that palladium coatings consist of three phases - $AlPd_2$, $Ni_{0.52}Pd_{0.475}$ and Ni_3Al. The chemical composition on the cross-section of palladium coating after diffusion treatment showed outward diffusion of nickel, chromium and aluminum and inward diffusion of palladium. Heat treatment of palladium coatings causes the increase of surface roughness parameter Ra with the increase of palladium thickness as a result of unequal mass flow of palladium and nickel.

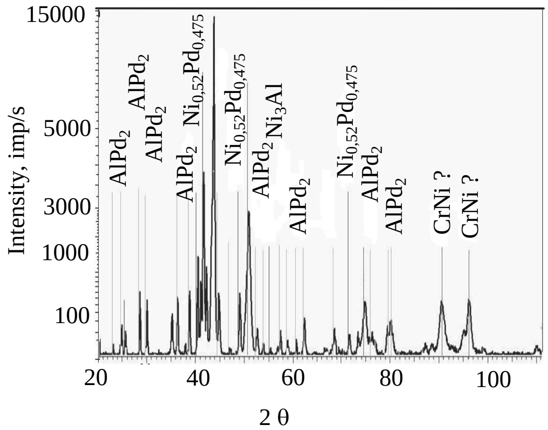
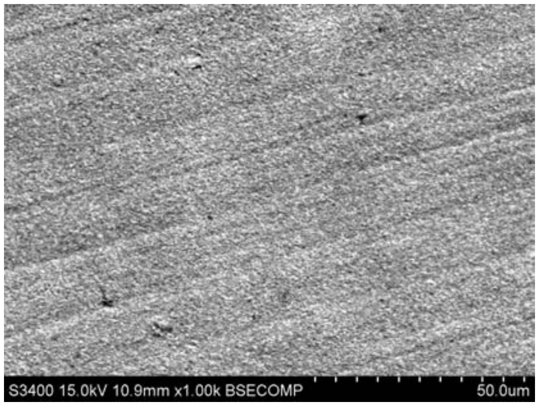


Fig. 6. Diffraction results of palladium coating 3 and 7 μm thick after heat treatment process

a)



b)

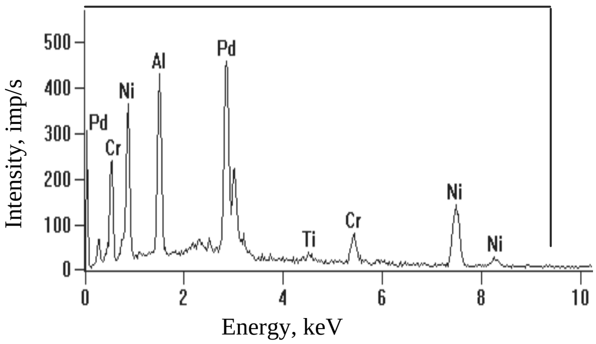


Fig. 7. Microstructure (a) and EDS analysis results (b) of the surface of palladium coating 7 μm thick after heat treatment process: showing a fine grain structure

Acknowledgements

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