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## THE INFLUENCE OF NICIALY COATINGS ROUGHNESS ON OXIDATION KINETICS DURING STATIC OXIDATION TEST AT TEMPERATURE 1000°C AND 1100°C

### WPŁYW PRZYGOTOWANIA POWIERZCHNI NA KINETYKĘ UTLENIANIA WARSTW TYPU NiCrALY POD WPŁYWEM STATYCZNEGO UTLENIANIA W TEMPERATURZE 1000°C I 1100°C

The article presents the results of research related to the impact of pretreatment plasma sprayed NiCrAlY coating on the kinetics of the oxidation. The analysis covered the shell subjected to thermal spraying. The test were performed at a temperature of 1000°C and 1100°C the samples were removed from the furnace after 25, 300, 500, 750 and 1000 hours. The investigations range included analysis of top surface of coatings by XRD characterization oxides formed types and microscopic investigations of coatings morphology.

Keywords: NiCrAlY coatings, oxidation kinetics, surface condition

W artykule zaprezentowano wyniki badań odnoszące się do wpływu przygotowania powierzchni natryskanej plazmowo powłoki NiCrAlY na kinetykę jej utleniania. Analiza obejmowała powłoki poddane natryskiwaniu cieplnemu. Testy wykonano w temperaturze 1000°C i 1100°C próbki zostały wyjęte z pieca po 25, 300, 500, 750 i 1000 godzinach. Przy analizie stanu warstwy wierzchniej powłok wykorzystano badania dyfrakcyjne określające skład fazowy i badania mikroskopowe umożliwiające ocenę budowy warstwy.

### 1. Introduction

The time-life of thermal barrier coatings (TBC) is related to many different factors. Most research presents that the main processes degradation of TBC coatings take in the TGO (thermally grown oxide) zone in bond-coat [1, 2]. The literature describes two mechanisms which influence the process of destruction. In the case of flat surfaces of bond-coat there are buckling mechanism involving on bendig of the oxide layer are TGO. This is the cause of deformation and generating of tensile stress on edges of oxide zone. This is characteristic mechanism of the thin and dense oxide layer of low adhesion to the substrate. The second mechanism is wedging, which occurs with thicker and more defected layers of oxides but also characterized by high adhesion to substrate [3].

Bond-coat obtained by APS method (atmospheric plasma spraying) characteristic in high roughness of surface. During cooling process of APS coat the compressive stress in the depression of bond-coat profile of roughness. The tensile stresses are observed in the case of peak of profile of roughness. This kind of stress generated the microcracks in the peak of bond-coat and in consequence starts of delamination process of TBC system. The value of the stress generated is dependent on the thickness of the TGO zone, TGO zone adhesion to the bond-coat, as well as the radius of profile curvature [4,5]. TBC

coatings often kinetics of crack propagation In the ceramic layer is lower than the nucleation of cracks and consequently delamination of the coating are on the top TGO roughness profile. It is lead to typical for this process morphology of TGO zone. Oxides present on this zone are thick and cracked. This area is characterized by the column structure and an increase in diraction of the substrate [5-7].

The role of thermal barriers coating bond-coat are MCrAIY type or aluminide coating that protects against base oxidation and corrosion in an environment containing the sulfur compounds. Additionally have a low tendency to form brittle transitions and high resistance to diffusion of alloying layer and the substrate [8-10]. MCrAIY type coating is used in order to ensure the heat resistance of components made not only with nickel alloys but also the alloys matrix phase of Ti-Al [11].

#### 2. Experiment description

Material used for testing was a nickel based superalloy Inconel 625 with additional NiCrAlY (Ni22Cr10AlY) coating. The coating in obtained by plasma spraying in air. In addition an undercoat layer was processed by mechanical grinding. The treatment is conducted to reduce the surface roughness of the

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coating realative to that obtained after the plasma spraying. Two types of surface conditions enabled the observation of differences in the mechanism of oxidation. Characterization of observed phenomena's was described by XRD (diffractometr JEOL JDX-7s) phases analysis of oxidation products on topsurface of coatings and SEM (Hitachi 3400N microscopy) investigations oxide layer morphology on top surfaces and cross section.

### 3. Results

The tests were performed in the two furnaces in air. The samples were heated in the furnace (5°C/min) up to a temperature of 1000°C and 1100°C. Samples were removed from the furnace after 25, 300, 500, 750 and 1000 hours. In Fig. 1 shows the average thickness measurement of oxides layer in the depression and in the peak of bond-coat profile of roughness the tests performed at 1000°C and 1100°C. After 25 hours of the test, irrespective of the temperature of oxidation process can be seen that the oxide layer is thicker in the depression of the roughness profile than at the peak. In the following test, the oxide layer formed in the peak of the roughness profile is thicker than the layer formed in the depression of the profile. The samples were oxidized at 1100°C are noticeably thicker oxide layer and increase its thickness is steeper from samples for which the test was run at 1000°C. With the passage of time is an increase in the thickness of the oxide layer. The kinetics of this process is greater in the peak of the roughness profile of the bond-coat. In the case of an oxide layer formed on the tops of the profile after 1000 hours

test, showing a sharp drop in temperature regardless of the thickness of the oxidation was carried out. This is due to the oxide layer falls off part of the TGO.

Fig. 2 presents an analysis of the chase composition of the samples after 25, 500 and 1000 hours at temperature 1000°C and 1100°C oxidation obtained using the XRD method. The samples removed from the furnaces after 25 hours, regardless of the test temperature is dominated by strong reflections Ni3Al phase. In the case of staying the sample at 1000°C can further be noted the presence of oxide phases reflection: NiAl<sub>2</sub>O<sub>4</sub> and NiCr<sub>2</sub>O<sub>4</sub>. After 500 hours of testing at 1000°C appeared reflections of the phases: NiAl<sub>2</sub>O<sub>4</sub> and NiCr<sub>2</sub>O<sub>4</sub>. In the sample held at a temperature of 1100°C appeared relatively high reflections of Al<sub>2</sub>O<sub>3</sub> phase. In both cases, dominate reflections of Ni3Al phase. After the next 500 hours of testing in both samples is dominated by strong reflections Ni3Al phase and weaker reflections: NiAl<sub>2</sub>O<sub>4</sub>, NiCr<sub>2</sub>O<sub>4</sub> and Al<sub>2</sub>O<sub>3</sub> phases. Reflections oxide phases are much stronger in a sample staying in the furnace at 1100°C. The analysis of the distribution of elements on the surface of 25, 500 and 1000 hours of oxidation is presented in Fig. 3,4 and 5. For samples residing in a furnace at a temperature of 1000°C can be seen as regular distribution of elements on the surface, in particular Ni, Cr and Al. Surface oxidation of the samples treated at 1100°C a noticeable formation of local large clusters that have a tendency to extend with the passage of time. Areas with a high content of Al indicate the presence of Al<sub>2</sub>O<sub>3</sub>. In the case of the samples subjected to oxidation at 1100°C it can be seen that the focus of the Al<sub>2</sub>O<sub>3</sub> occurs around areas where there are large quantities of both Ni and Cr. This indicates the formation of this area of oxide type: NiCr<sub>2</sub>O<sub>4</sub> and NiAl<sub>2</sub>O<sub>4</sub>.



The thickness of oxide layer in the depression of the roughness profile of the bond-coat at a temperature of 1000°C



The thickness of oxide layer in the peak of the roughness profile of the bond-coat at a temperature of 1000°C



The thickness of oxide layer in the depression of the roughness profile of the bond-coat at a temperature of 1100°C



The thickness of oxide layer in the peak of the roughness profile of the bond-coat at a temperature of 1100°C

Fig. 1. The kinetics of growth of the oxide layer thickness in the depression of roughness and in the peak of roughness profile of bond-coat a temperature 1000°C and 1100°C



Fig. 2. XRD diffraction patterns of coated by grinding process after 25,500 and 1000 hours at temperature 1000°C and 1100°C



Fig. 3. Mapping of selection elements of as-sprayed after 25 hours at temperature  $1000^\circ$ C and  $1100^\circ$ C



Inconel 625/NiCrAlY 1100°C / 500h

Fig. 4. Mapping of selection elements of as-sprayed after 500 hours at temperature 1000°C and 1100°C

# 2676



Inconel 625/NiCrAlY 1100°C / 25h

Fig. 5. Mapping of selection elements of as-sprayed after 25 hours at temperature 1000°C and 1100°C



Cr Inconel 625/NiCrAlY 1100°C / 500h

Fig. 6. Mapping of selection elements of as-sprayed after 500 hours at temperature 1000°C and 1100°C

# 2678



Cr Inconel 625/NiCrAlY 1100°C / 1000h



25 µm

25 ym

**25** ymm

**26** µm

Al

Cr

Al



NiAl<sub>2</sub>O<sub>4</sub> **25 pm** 



Fig. 7. Mapping of selection elements of as-sprayed after 1000 hours at temperature 1000°C and 1100°C



Inconel 625/NiCrAlY 1000°C / 25h



Inconel 625/NiCrAlY 1000°C / 500h



Inconel 625/NiCrAlY 1100°C / 25h



Inconel 625/NiCrAlY 1100°C / 500h



Inconel 625/NiCrAlY 1000°C / 1000h



Inconel 625/NiCrAlY 1100°C / 1000h

Fig. 8. Morphology of oxides temperature 1000°C and 1100°layer on as-sprayed after the test at C

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The microstructure of the oxidation layers, depending on the residence time in the furnace the sample is presented in Fig. 6. After 25 hours, the test shows formed the island-like of oxide layers. Along with the process it is evident that the oxide layer is formed on the entrie surface of the layer. An oxide layer formed by oxidation at 1000°C is more compact than the layer was formed at 1100°C. An oxide layer after 500 and 1000 hours residence at 1100°C is heavily cracked as a result of the stress generated [12,13].

### 4. Summary

The surface condition has influence on the rate of formation of the oxide layer and its morphology. In the bottom of the roughness profile of the bond-coat there is a close to linear increase in thickness as a function of time scale. These are relatively compact layer, consisting mainly of  $Al_2O_3$  and small amounts of complex oxide network type of: NiCr<sub>2</sub>O<sub>4</sub> and NiAl<sub>2</sub>O<sub>4</sub>. In the case of oxide layer formed on the tops of the roughness of bond-coat it has a faster growth and greater thickness of the layer. However, it has a tendency to dropout, which has been observed in the presented research. The composition of the oxide layer mainly consists of  $Al_2O_3$ , located close to the substrate and oxide type of NiCr<sub>2</sub>O<sub>4</sub> and NiAl<sub>2</sub>O<sub>4</sub>, which occur in the outer zone of the oxide layer, and a much larger amount than the bottom of the roughness profile.

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