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M. GÓRAL*, M. PYTEL*, K. DYCHTOŃ*

MICROSTRUCTURAL CHARACTERIZATION OF SEVERAL COATINGS DEPOSITED ON TiAlNb INTERMETALLIC ALLOY

CHARAKTERYSTYKA MIKROSTRUKTURY WYBRANYCH WARSTW WYTWORZONYCH NA PODŁOŻU >STOPU TYTANU NA OSNOWIE FAZY MIĘDZYMETALICZNEJ TiAlNb

Titanium alloys based on the γ-TiAl intermetallic phase are a type of material which may replace nickel supperalloys in high temperature applications. Not enough resistance to corrosion at temperatures above 900°C remains the biggest limitation in industrial practices.

The paper presents results of preliminary research into application of new type heat resistant coatings on the basis of γ-TiAl alloy. MeCrAlY and Si coatings were formed by pack cementation method. Aluminide coatings were deposited by pack cementation method and VPA. Research results have shown that except for the coating formed by VPA, coatings were characterized by high porosity and therefore would not ensure appropriate protection against oxidation. Further research will be necessary into application of the VPA method in protecting γ-TiAl titanium alloys in order to improve their heat resistance.

Keywords: TiAl intermetallics, slurry coatings, pack cementation, vapour phase aluminizing

Stopy na osnowie fazy międzymetalicznej γ-TiAl stanowią materiał, który może stanowić zamiennik nadstopów niklu w aplikacjach wysokotemperaturowych. Nadal największym ograniczeniem w ich przemysłowym zastosowaniu jest niewystarczająca odporność na utlenianie w temperaturze powyżej 900°C. W artykule przedstawiono wyniki wstępnych badań nad zastosowaniem nowych rodzajów powłok żaroodpornych na podłożu stopu γ-TiAl. Wytworzono powłoki MeCrAlY oraz Si metodą zawiesinową a także powłoki aluminidkowe metodą pack cementation oraz VPA. Wyniki badań wykazały że poza powłoką wytworzoną metodą VPA pozostałe charakteryzowały się dużą porowatością przez co nie mogą zapewnić odpowiedniej ochrony przed utlenianiem. Wskazano na konieczność dalszych badań nad zastosowaniem metody VPA do ochrony stopów tytanu γ-TiAl w celu poprawy ich żaroodporności.

1. Introduction

Titanium alloys based on the γ intermetallic phase (TiAl) are characterized by high tensile strength and good heat resistance up to 800°C. The $\gamma-$TiAl intermetallics (TiAl) may replace nickel superalloys in some applications, e.g. turbine blades in low pressure aircraft engine [1,2], automotive turbocharger rotor [3]. One of the barriers to extend application of these materials are high costs of production in comparison to various types of protective coatings [4]Titanium alloys based on γ (TiAl) intermetallic phase have good physical and chemical properties, and also mechanical properties which allow for use of these alloys in aviation and automotive industries. Research into $\gamma-$TiAl titanium alloys is focused on improving their mechanical properties and resistance to oxidation as well as reducing production costs. Use of γ titanium alloys results in the reduced total mass of an engine. γ titanium alloys (TiAl) are also used in high quality car engines, turbocharger rotors and valves. They help to increase the engine power, car acceleration and reduce fume toxicity [1-3]. Primary limitations for otherwise extensive application of γ titanium alloys (TiAl) for industrial purposes are high production costs as well as insufficient resistance to corrosion at the temperature 900-1000°C and low resistance to hot corrosion. At present, it is not feasible to determine a chemical composition of γ titanium alloys (TiAl) which would ensure a good level of resistance to hot corrosion at reduced production costs. That is why, as in the case of developing nickel superalloys and modifying γ titanium alloys (TiAl), suitable protective coatings have been devised [15]. During the oxidation process of titanium alloys based on γ (TiAl) intermetallic phase, in spite of introducing alloy additions, no Al_2O_3 scales are formed. Al_2O_3 oxides ensure formation of a homogeneous and dense protective coating, which prevents further corrosion. Research into application of various protective coatings for the base of γ titanium alloys (TiAl) has been conducted. In the case of these materials, numerous approaches to the base protection against corrosion are applied.

In order to improve resistance to corrosion, passivation processes by means of 4% phosphoric acid anodizing are now commonly used. Modification of the γ alloy (TiAl) outer coating has been achieved through forming of an amorphous phosphate layer. Results of oxidation trials have shown increased resistance to corrosion of the coating anodized on the γ titani-

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* RZESZOW UNIVERSITY OF TECHNOLOGY, DEPARTMENT OF MATERIALS SCIENCE, POWSTANCOW WARSZAWY 12, 35-959 RZESZÓW, POLAND

um alloy at the temperature of 800°C [5]. Ion implantation is one of the most common techniques used in order to improve heat resistance of γ titanium alloys (TiAl) [6,7]. Taniguchi [8-11] has shown that silicon implantation into the base layer forms intermetallic phases, e.g. $Ti_7Al_5Si_{12}$. At the same time it improves aluminium activity. No SiO_2 oxide, which is normally formed during oxidation, has been detected in the formed scales. It has been shown that in the outer coating of the silicon-implanted Ti50Al-alloy base, Ti_5Si_3 titanium silicides are formed in the first stages of oxidation at the temperature of 900°C. One may assume that silicides act as a diffusion barrier for oxygen. It has also been shown that increased corrosion resistance of γ titanium alloys (TiAl) has been achieved through simultaneous implantation of niobium and silicon oxides with the use of the ion implantation technique. Heat resistant coatings formed on the NiAl-phase have been prepared in order to protect nickel superalloys' base. [12].

Protective coatings on the $TiAl_3$-phase structure containing a large amount of aluminium are formed on the γ titanium alloy (TiAl), commonly by pack cementation. The coating is formed through in-core diffusion of aluminium, which is an ingredient of powder adjoining the base material, with the use of a halogen activator. The diffusion coating comprises an outer zone made from $TiAl_3$-phase grains and an inner zone made from $TiAl_2$-phase grains. Cracks in the external zone may appear during cooling. This is caused by brittleness of $TiAl_3$-phase particles. High corrosion resistance at the temperature of 1100°C may be increased by fluorides produced by an activator during the process [13]. Xiang and Rose [14] have proven that use of chloride $AlCl_3$ as an activator helps to control the coating growth rate and prevents microcracks. During tests on resistance to oxidation of aluminide coatings at the temperature 800-1000°C a significant growth of the intermediate phase, consisting of $TiAl_2$-phase grains was observed, as well as gradual reduction of the $TiAl_3$-phase area. The entire disappearance of the $TiAl_3$-phase area takes place at the temperature of 900°C after a period of 300h. In spite of this, the Al_2O_3 scales layer $<2\mu$m thick consists of Al_2O_3 oxide. Niobium addition in γ titanium alloys (TiAl) influences the microstructure and diffusion properties of $TiAl_3$ type aluminide coatings made by gas-contact method. Coatings formed on TiAlNb alloys are characterized by higher resistance to oxidation in comparison to the γ alloy (TiAl) base without alloy additions. It has been found that resistance to oxidation increases as the niobium content in the base material is higher. Niobium increases thickness of coatings formed under the same conditions. At the same time it causes grain size reduction in the coating. Reduction of grain size, particularly in the $TiAl_3$/TiAl intermediate area, as well as presence of Al_3Nb traces slows down inward aluminium diffusion. Niobium content in the formed coating improves its mechanical properties, i.e. it decreases hardness, increases ductility and reduces microcracks [15].

Use of silicon in order to improve resistance to titanium and its alloys corrosion has been prompted by the following factors [16]:(a) it dissolves in the TiO_2 oxide layer, (b) it reduces the diffusion rate of oxygen atoms and leads to concentration of oxygen vacancy (c) it causes stress relaxation in scales. SiO_2 oxides in the TiO_2 oxide layer reduce its recrystallization and stratification, and therefore prompt formation of dense and low porous scales. Xiang and Rose in their paper [17] presented a possibility of simultaneous silicon and aluminium deposition by means of the gas-contact method. In a thermodynamic analysis of several types of halogen activators, hydrated chloride $CrCl_3*6H_2O$ was proven to have the best properties. An attempt to form a coating in the powder mixture containing Al-2% wt. %, Si-4% wt. %, Al_2O_3-90% wt. %, and $CrCl_3*6H_2O$ acting as an activator, failed to render results. This is due to the fact that in reaction with gaseous chlorine, vapour from the activator turns into hydrochloric acid HCl. Acid has caused degradation of the base material surface prior to forming of the coating. For purposes of further research $AlCl_3$ chloride characterized by high stability at a high temperature and low stability at room temperature. During the tests there was used a mixture of powders containing Si 1-4% wt. %, Al 1-6% wt. % – an activator, $AlCl_3$ – 1-6% wt. % The process was carried out in argon atmosphere at the temperature of 1100°C. Coating was formed on the titanium alloy (TiAl) base with added tungsten 8.2% at. Analysis of chemical and phase composition has proven that on the surface of silicide zone there has been formed a dense coating consisting of high silicon content, $TiAl_2$-phase grains, while below it there is a highly porous, silicide Ti_5Si_4 zone. Ti_5Si_3, TiSi silicides as well as pure silicon were also observed in the coating [18]. Xiang [19, 20] explained a sequential growth model for this coating. He observed that at the preliminary stage aluminium with a little silicon addition is deposited on the base. $TiAl_3$ phase is formed due to inward aluminium diffusion. During the next stage aluminium concentration close to the base lessens. Simultaneously, silicon activity in the coating formation increases – titanium silicides are formed due to inward Si diffusion at the last stages of the diffusion coating formation. Oxidation tests on the formed coating were carried out at the temperature 800-850°C. Parabolic dependence of the oxidation process was determined. No intermediate layer, characteristic of aluminide coatings with no silicon addition, was reported. During oxidation of the outer zone of the coating with silicon content there takes place total conversion of Ti_5Si_4 silicides into Ti_5Si_3 silicides and forming of SiO_2 oxide. It is possible to form a range of other titanium silicides (from Ti_5Si_3 to $TiSi_2$) due to preferential silicon oxidation. Oxidation products are aluminium, titanium and silicone oxides where the content ratio equals 1:2:7 [20]. Trial forming of protective coatings on the γ titanium alloys (TiAl) base in liquid alloy Al.-Si was carried out [21]. There were used sub-eutectic siluminum (10% wt. %) and eutectic silumin (Si 12.8 % wt. %). The obtained homogeneous coating contained mostly three-element $Al1_2Si_3Ti_5$ and $Ti_7Al_5Si_{12}$-phase as well as a low content of titanium silicides. Tests on cyclic and isothermal oxidation at temperatures: 900 i 1000°C have shown higher resistance of the formed protective coating. Silicon oxide was reported to be the main oxidation product. Oxide concentration in the layer was 45-64 % at. Found in the layer were also TiO_2 and Al_2O_3 oxides. Preparation of several γ titanium alloys (TiAl) coatings that would increase corrosion resistance is the aim of currently run research. Silicon-modified aluminide coatings have good heat resistance. They are produced by Arc-PVD method through vaporization of silumin targets (alloy AK11) and an exothermal reaction [22]. An analysis of the coating's chemical composition run immediately after the coating's deposition has shown aluminium content. This prompted further 2-hour-long vacuum heat processing done at the temperature of 950°C.

The formed coating is approx. 40 μm thick. In the coating's microstructure there was observed an outer zone – a framework consisting of $TiAl_3$-phase grains, pure Al grains and column-type Ti_5Si_3 silicides in the $TiAl_3$-phase framework. Under the outer zone there is an intermediary zone [23]. Tests into kinetics of coating growth at the temperature of 950°C have proven a fast growth rate for the intermediary zone consisting of TiAl-phase equiaxed grains and a zone consisting of $TiAl_2$ -phase grains. Short- and long-term cyclic oxidation trials carried out at the temperature of 950°C have confirmed good heat resistance of such protective coatings – trials were terminated after 920 hours [24]. Coatings made by slurry methods also display good properties [25]. For instance, aluminide coating was formed through deposition of non-organic slurry containing Al and Si-powders and further thermal diffusion processing under an argon atmosphere at the temperature of 1050°C. In the formed coating, approx. 50 μm thick, there were three distinguishable zones. Microstructural examination and chemical composition analysis have shown that the outer zone contained $TiAl_3$-phase grains and a small amount of Ti_5Si_3 titanium silicides. The intermediate phase (approx.15 μm thick) contained the same phase ingredients. Different morphology of titanium silicides was reported – a large number of column crystals has been reported. Below this zone, there is an internal zone consisting of $TiAl_2$-phase grains (approx. 2 μm thick). The formed coatings enjoy good resistance to corrosion. What is more, there is a possibility of an easy transfer of this technology to industrial conditions, where the existing equipment could be used and low production costs would be ensured. The aluminization process can be carried out with the use of the VPA method. So far, research has shown a possibility of forming aluminide coatings consisting of $TiAl_2$ and $TiAl_3$-phase grains ranging from several to several dozen μm [26,27].

2. Experiment

For research purposes, type Ti46Al5Nb two-phase alloy immediately after casting was used. Prior to deposition of all coatings samples were sandblasted with abrasive 220 mesh size and then cleaned and degreased with ethanol in ultrasonic cleaner. Slurry deposition was carried out with the use of silicon powder, 20 μm grit size and 99.9% purity as well as MeCrAlY powder.

An inorganic chromic and orthophosphoric acid solution available for commercial purposes (approx. 1 wt. %) was used as a binder. Samples with deposited slurries were subjected to diffusion annealing at the temperature of 1080°C for 2 hours in vacuum. Formed by pack cementation method, aluminide layer was made from powder containing FeAl and NH_4Cl activator. The pack cementation aluminizing process was carried out at the temperature of 1050°C for 4 hours in heat resistant metal boxes. Vapour phase aluminizing (VPA) was carried out at the temperature of 1050°C for 4 hours in an argon atmosphere. As an aluminium source, Cr-Al granulate with 44 wt. % Al was used, while AlF_3 was used as an activator. Examination of the microstructure was performed with the use of scanning electron microscope S-3400 by Hitachi equipped with EDS detector by Thermo for chemical composition analysis.

3. Results

3.1. Aluminide coating formed by VPA method

The surface of the sample aluminized by VPA method appeared to be entirely covered by aluminide coating. Thickness of the coating was less than 20 μm. No cracks in the coating were observed. Chemical composition analysis has shown high Al content in the sub superficial area (area 1 in Fig. 1). Aluminium content below it was approx. 64 at. % (areas 2,3 in Fig. 1). Close to the base material aluminium content was smaller, that is approx. 55 at.%. In all analyzed spots from 2 to 4 at.% niobium content was determined. A linear analysis of chemical composition performed at the cross-section of the coating (Fig. 2) has proved the presence of three distinguishable zones: a 5 μm thick outer zone, consisting of the $TiAl_3$,-phase, below it a 12 μm phase consisting of the $TiAl_2$-phase, and finally a thin (2 μm) TiAl zone.

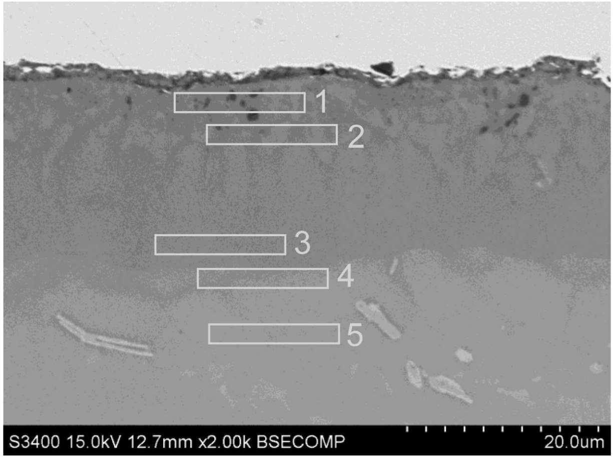

Fig. 1. Structure of aluminide coating deposited by VPA method on γ-TiAl intermetallic alloys

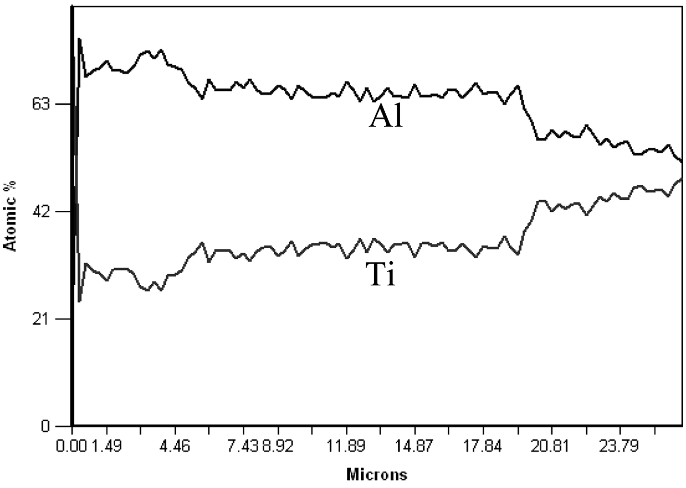

Fig. 2. The linear chemical composition analysis through aluminide coating deposited by VPA method on TiAl intermetallic alloy

TABLE 1
The results of chemical composition analysis from the areas presented in Fig. 1

area	*Element amount (at.%)*			
	Al	*Ti*	*Cr*	*Nb*
1	68.67	28.75	0.01	2.57
2	63.37	34.35	–	2.28
3	63.39	33.88	–	2.73
4	55.44	40.96	-	3.60
5	44.95	51.32	–	3.73

3.2. Pack cementation

The coating formed by pack cementation method had highly porous surface. Microstructure examination showed high porosity on the entire cross-section of the coating and presence of two distinguishable zones (Fig. 3). A chemical composition analysis proved high Al content in the outer zone in the areas 1-4 (over 70 at.%) and a small amount of titanium (up to 22% in the spot indicated in Fig.3). In the bottom part of this zone aluminium content decreased to approx. 50 at.% in the area 3. Similar content of this element was determined in the internal zone (area 4 in Fig. 3). Titanium concentration in areas 3 and 4 (Fig. 3) was higher, that is approx. 40 at.%. In the area 5 chemical composition was in line with chemical composition of the base material.

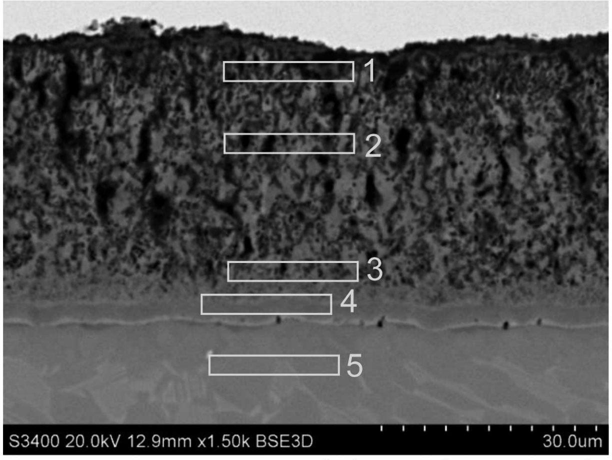

Fig. 3. Microstructure of aluminide coating deposited by pack cementation method on TiAl intermetallic alloy

TABLE 2
Results of chemical composition analysis from areas presented in Fig. 3

area	*Element amount (at.%)*			
	Al	*Ti*	*Ni*	*Nb*
1	75.04	17.42	0.92	6.62
2	71.38	22.81	–	5.81
3	54.09	42.49	–	3.42
4	51.23	43.07	–	5.70
5	45.30	50.24	–	4.45

3.3. Si and MeCrAlY slurry coatings

Tests on forming a heat resistant coating on the basis of silicon powders and MeCrAlY alloy with a standard binder have proven, that obtaining a homogeneous diffusion coating is difficult. The microstructure of formed layers was quite porous (Fig. 4, 5). Application of nickel before preparing metallographic specimen has shown penetration of deposited Ni deep into the coating (can be observed as bright precipitates in Fig. 4 and 5).

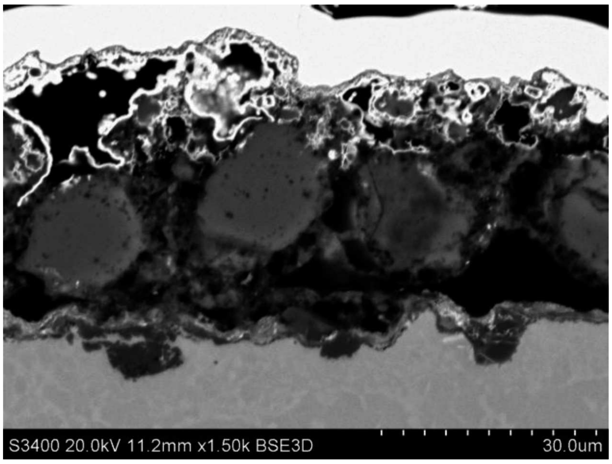

Fig. 4. Microstructure of silicide coating deposited by slurry method on TiAl intermetallic alloy

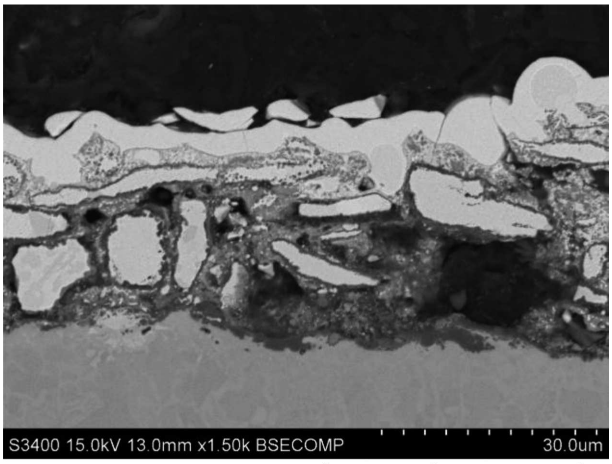

Fig. 5. Microstructure of MeCrAlY coating deposited by slurry method on TiAl intermetallic alloy

4. Summary

The recent work presents results of trial deposition of heat resistant coatings on the basis of the γ-TiAl type titanium alloy. Significant difficulties in forming layers by the slurry method occurred when the original binder was used. Quality of coatings disqualified them for further tests due to significant thickness differences and the porosity appearance, discussed earlier in this paper. It is therefore necessary to prepare a new binder that will ensure coatings of required properties be formed. Layers deposited through pack cementation with the use of Fe-Al source had high aluminium content (more than 70 at. %). At the same time high porosity was observed;

it disqualified the coating for protection of the γ-TiAl type surfaces of titanium alloys. Out of the examined heat resistant coatings, the vapour phase aluminizing method is the most promising technology to improve oxidation resistance of TiAl intermetallics. Though rather thin (less than 20 μm), the coating was characterized by high aluminium content. No cracks or delamination were observed. This is thanks to the coating that has been formed on the basis of $TiAl_2$-phase on the alloy surface. The coating showed lower level of brittleness. High niobium content in the coating may also have positive influence on the granularity and improving quality of the coating. Further research into deposition of aluminide coatings with the use of new activators is necessary, as well as examination of the influence the activators have on the microstructure of the coating. Research into resistance to cyclic and isothermal oxidation is also important. Results of the tests carried out so far point to a possibility of applying this type of coatings in industrial practices, with a view to improving resistance to oxidation of alloys on the basis of the γ-TiAl intermetallics framework.

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