



## Improvement of oxidation resistance of Ti–47Al–2W–0.5Si alloy modified by aluminizing method



Agnieszka Szkliniarz<sup>a,\*</sup>, Grzegorz Moskal<sup>a</sup>, Wojciech Szkliniarz<sup>a</sup>, Radosław Swadźba<sup>b</sup>

<sup>a</sup> Department of Material Science and Metallurgy, Silesian University of Technology, Krasińskiego 8, 40-019 Katowice, Poland

<sup>b</sup> Institute for Ferrous Metallurgy, Miarki 12-14, 44-100 Gliwice, Poland

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### ABSTRACT

The presented study defines the impact of aluminizing process by out-of-pack method on the oxidation resistance of cast Ti–47Al–2W–0.5Si alloy after homogenising and hot isostatic pressing. The surface condition was characterized, as well as chemical and phase composition of scale formed on examined alloy with and without protective coating, when subjected to cyclic oxidation at 950 °C. It was established that the generated hermetic and compact aluminium diffusion coating with a thickness of about 7.5 μm, consisting mainly of TiAl<sub>2</sub> phase, has a significant effect on the improvement of cyclic oxidation resistance in the case of investigated alloy. It was confirmed that the scale formed after the oxidation process on the surface of alloy with protective coating was composed mainly of Al<sub>2</sub>O<sub>3</sub> oxide, contrary to the scale formed on the alloy without protective coating, which was mainly consisting of TiO<sub>2</sub> oxide.

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### 1. Introduction

One of the major barriers limiting the suitability of TiAl intermetallic alloys for use at elevated temperature is their susceptibility to degradation as a result of oxidation [1–7]. Low resistance to oxidation of these alloys is connected with the lack of possibility of the formation of Al<sub>2</sub>O<sub>3</sub> oxide protective coating on their surface during the oxidation, which is a barrier against further progress of oxidation. It is a result of similar value of aluminium oxide and titanium oxide free formation energy. Under long-term high-temperature operating conditions, TiAl intermetallic alloys form a scale on the surface, consisting of the mixture of titanium oxide and aluminium oxide with low protective capacities at high temperature. It is an effect of much higher kinetics of titanium oxide growth in comparison to aluminium oxide [1–10]. The low protective capacity of the scale is caused by poor adhesion of TiO<sub>2</sub> to the substrate and its fast build-up rate as a result of a much easier diffusion of oxygen through defected anion sublattice of TiO<sub>2</sub> than in case of Al<sub>2</sub>O<sub>3</sub>. The additional important factor affecting the oxidation of these alloys is the presence of nitrogen in the atmosphere. In case of alloys oxidised in the pure oxygen, the content of aluminium from the range of 47–49 at.% is required to ensure formation of the protective Al<sub>2</sub>O<sub>3</sub>

oxide layer. If alloys are oxidised under air, the required “safe” aluminium level increases to 60–70 at.%. This fact is associated with the formation of TiN titanium nitride layer in the scale, which facilitates the formation of heterogeneous TiO<sub>2</sub>–Al<sub>2</sub>O<sub>3</sub> oxide mixture and thus increases the rate of oxidation [1–10].

The second-generation TiAl intermetallic alloys reveal properties that predestinate them to serve as a construction material for the structural components of aircraft and automotive engines, operating at temperatures not exceeding 760 °C [1,4,6,7]. The increase in this temperature up to 800–850 °C was obtained in the third-generation TNB alloys [4–7] by the introduction of heavy niobium (up to 10 at.%) into their chemical composition and optimisation of the microstructure. Further increase in the admissible working temperature of TiAl intermetallic alloys to 950 °C, which would significantly increase the possibility of using them as substitutes for heavy and expensive nickel superalloys requires the use of protective coatings [4–7].

For the protection of TiAl intermetallic alloys' surfaces against oxidation three types of coatings are applied: the aluminide coatings based on high-aluminium TiAl<sub>2</sub> and TiAl<sub>3</sub> phases from Ti–Al system, MCrAlY coatings and silicon-modified coatings [2–5,8–10]. Similar effects were obtained for Al<sub>2</sub>O<sub>3</sub> and SiO<sub>2</sub>-based coatings [4,10–15]. Resistance to oxidation is also improved by CaTiO<sub>3</sub>, SrTiO<sub>3</sub>, BaTiO<sub>3</sub>, SrTiO<sub>3</sub> and AlTiO<sub>5</sub> coatings [4,8–15]. The most often methods used for production of coatings include: pack cementation method, out-of-pack method, slurry method as well as CVD (Chemical Vapor Deposition) and arc-PVD method [4,11–22].

The out-of-pack aluminizing method involves placing coated components in a container such that they do not contact the powder

\* Corresponding author at: Department of Material Science and Metallurgy, Institute of Materials Science, Silesian University of Technology, Krasińskiego 8, 40-019 Katowice, Poland.

E-mail addresses: [agnieszka.szkliniarz@polsl.pl](mailto:agnieszka.szkliniarz@polsl.pl) (A. Szkliniarz), [grzegorz.moskal@polsl.pl](mailto:grzegorz.moskal@polsl.pl) (G. Moskal), [wojciech.szkliniarz@polsl.pl](mailto:wojciech.szkliniarz@polsl.pl) (W. Szkliniarz), [rswadza@imz.pl](mailto:rswadza@imz.pl) (R. Swadźba).

mixture. The samples are placed in a retort or vacuum furnace and heated. Over the course of the coating deposition process, a neutral carrier gas is fed into the container, which enables the transfer of the coating gases created during the process. The main advantages of the out-of-pack process are: no contact between the coated material and powder, which notably improves coating surface; increased control during the deposition process; increased purity of the process (in comparison with powder technologies); as well as the fact that aluminide coatings may be modified by a number of elements to increase their oxidation resistance [4,21,22].

The purpose of the investigations was the characteristics of resistance to cyclic oxidation for Ti–47Al–2W–0.5Si casting alloy with and without protective coating obtained as a result of aluminizing using out-of-pack method.

The Ti–47Al–2W–0.5Si was developed for maximum creep resistance. The addition of tungsten is known to increase creep resistance and also improved oxidation resistance. Additionally W has a very strong effect of stabilizing the  $\beta$  phase in Ti–Al alloys. Solubility of silicon in the  $\gamma$  phase is reported to be lower than 0.5 at.% at 1000 °C and that  $Ti_5Si_3$  particles precipitate along interfaces. As a result, silicon in  $\gamma$ -TiAl based alloys increases the toughness, lowers primary creep strain, enhances the creep resistance up to 850 °C, stabilizing lamellar structure to inhibit dynamic recrystallization and also improves high temperature oxidation resistance [4,11,23,24].

## 2. Material and methods

Ti–47Al–2W–0.5Si casting alloy with chemical composition as specified in Table 1 was used for investigations. This alloy is characterized by high strength, good creep and oxidation resistance and satisfactory castability, which is of particular importance when producing this alloy from thin-walled castings, e.g. aircraft engine turbine blades [4,23–27].

The studied alloy was manufactured from the commercial pure Ti (Grade 1) and elements such as Al, W, Si which were added as Al–W–Si master alloy. The alloy was melted in vacuum induction furnace using a special crucible made of isostatic pressed graphite [28,29] and cast as an ingot with 45 mm diameter and approx. length of 350 mm, into a graphite mould. The ingot was subjected to vacuum homogenising at 1400 °C for 1 h and hot isostatic pressing at 1260 °C and 170 MPa for 4 h in the hot isostatic press chamber filled with argon of utmost purity.

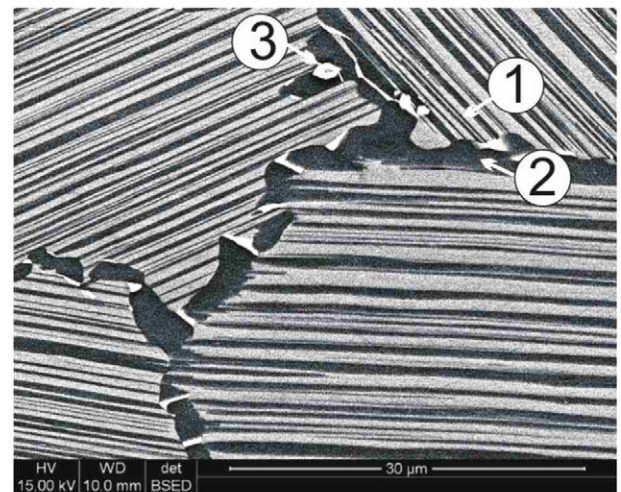
A series of cylindrical test pieces of 14 mm in diameter and 4 mm in height was made out of the ingot. The protective coating was produced on the surface of test pieces by aluminizing using out-of-pack method. Aluminizing process was made in internal Surface Engineering Laboratory in Institute of Materials Science of Silesian University of Technology. Aluminium granules and ammonium fluoride activator were used to rich in aluminium layer deposition. The aluminizing process was made at temperature 900 °C for 5 h in Ar atmosphere. For investigation of the oxidation resistance of alloy without coating and with applied coating the method of cyclic oxidation conducted at a temperature of 950 °C, which is similar to the expected maximum working conditions for this group of alloys, was used. It consisted in placing the test pieces in ceramic gutters in the furnace heated up to the test temperature. After each 23 h cycle, with one-hour cooling of test piece to room temperature, the weight of the test pieces was measured with an accuracy of

0.0001 g, and on this basis the curves of weight gain with every consecutive cycle were determined. The total number of cycles was 100.

Standard metallographic techniques were employed for microstructure observation using stereomicroscope Nikon SMZ 745T and scanning electron microscope Hitachi 3400N with the Noran System Six software. The specimens for microstructure examination were prepared by mechanically polishing and etching in reagent consisted of 30 ml of lactic acid, 15 ml of  $HNO_3$  and 5 ml of HF. The microstructure characterization of alloy substrate, coating and oxide scale was performed on the cross section of specimens. The obtained coating was characterized by X-ray diffraction (XRD) analysis to yield the phase composition of the aluminized surface (JEOL JDX 7S diffractometer) and energy dispersive X-ray (EDX) spectrometry microanalysis to determine the chemical composition. Electron backscatter diffraction (EBSD) experiments were conducted using the Hitachi 3400N microscope with the HKL Channel 5 software. TEM specimens were prepared by either ion beam thinning or twin jet polishing using a solution consisting of 30 ml of  $HClO_4$ , 175 ml of butony-ethanol and 300 ml of  $CH_3OH$ . During polishing, the voltage was maintained at 15 V. TEM was carried out on FEI TITAN 80–300 transmission electron microscope.

## 3. Results and discussion

The microstructure of investigated Ti–47Al–2W–0.5Si alloy after casting, homogenising and hot isostatic pressing consists of coarse grains of the primary  $\alpha$  phase filled with alternately arranged lamellar precipitates of  $\alpha_2$ -Ti<sub>3</sub>Al and  $\gamma$ -TiAl phases (Fig. 1). These treatment resulted in fully lamellar microstructure with an average interlamellar spacing of  $\sim 0.2 \mu m$ . The large Al-rich precipitates of  $\gamma$  phase at the colony boundaries and very small amounts of blocky phase also appeared in the intercolony region. EDX analysis showed that it was rich in tungsten (about 13%) compared to 2.6% in the  $\gamma$  phase and 4.1% in the  $\alpha_2$  phase (Fig. 1). According to [23], both W addition and lower Al content favour the formation of the  $\beta$  phase. The analysis of Ti–47Al–2W–0.5Si alloy phase composition using X-ray phase analysis methods confirmed partially these observations and revealed the predominating participation



Point	Chemical composition, at.%			
	Ti	Al	W	Si
1	59.2	36.0	4.1	0.7
2	50.5	46.5	2.6	0.4
3	53.8	33.0	12.4	0.8

**Table 1**  
Chemical composition of investigated alloy.

Element content							
at.%				ppm			at.%
Al	W	Si	C	O	N	H	Ti
46.0	2.33	0.43	0.34	836	120	21	Balance

**Fig. 1.** Microstructure and chemical composition (points 1–3) on the cross-section of Ti–47Al–2W–0.5Si alloy in the initial state.

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