



## Effect of Au diffusion in Au-coated ( $\gamma + \alpha_2$ ) titanium aluminides

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### ABSTRACT

In order to understand the role of  $\alpha_2$ -Ti<sub>3</sub>Al precipitates during the oxidation of Au-coated  $\gamma$ -48-2-2-TiAl alloys, the  $\gamma$ -TiAl and  $\alpha_2$ -Ti<sub>3</sub>Al microstructures resulting from Au diffusion at 900 °C were investigated. The as-obtained coatings were characterized using scanning electron microscopy (SEM), combined energy and wavelength dispersive X-ray spectroscopies (EDS, WDS), electron backscatter diffraction analysis (EBSD), X-ray diffraction (XRD) and isothermal oxidation tests. Results showed that Au-coated- $\gamma$ -TiAl alloys presented an upper aluminoformer TiAlAu<sub>2</sub> layer and a lower non-protective TiAlAu layer, both with narrow homogeneity range, and with thicknesses that could be estimated with the use of the Prasad model. A more complex layer structure is obtained after Au diffusion in  $\alpha_2$ -Ti<sub>3</sub>Al alloys, since eight different phases were identified. In the lower layers, two new ternary tetragonal phases (Ti<sub>2</sub>AlAu and Ti<sub>3</sub>Al<sub>2</sub>Au) were found, and a partial diagram for the Al-Au-Ti 900 °C isotherm is proposed. Absence or depletion of Al was found in the upper layers and, in addition, a low oxidation resistance of the Au-coated- $\alpha_2$ -Ti<sub>3</sub>Al alloy was reported.

### 1. Introduction

TiAl-based alloys are continuing to attract attention from the industry as a high-temperature material, due to their exceptional combination of light weight and mechanical properties. Titanium aluminides have been found to exist in three different phases, Ti<sub>3</sub>Al ( $\alpha_2$ ), TiAl ( $\gamma$ ), and TiAl<sub>3</sub>. However, most TiAl-based alloys developed for structural applications are essentially two-phase ( $\gamma + \alpha_2$ ) mixtures. According to the Ti-Al diagram, the aluminum content of the Ti<sub>3</sub>Al ( $\alpha_2$ ) phase is between 22% and 39% (at.%), while the TiAl ( $\gamma$ ) phase contains 48.5%–66% (at.%) aluminum.

The attractive features of titanium aluminides are their lower density, higher stiffness, higher strength, improved creep resistance up to moderately high temperatures, lower coefficient of thermal expansion, higher thermal conductivity, and higher melting temperature compared to the conventionally used Ni-based super-alloys, which makes them suitable for potential high-temperature applications in the aerospace, automotive and power generation industries [1–5].

However, the alloys are known to possess a number of limiting properties, such as poor corrosion resistance to chlorine agents and low oxidation resistance above 700 °C, which have continued to restrict their wider application. Many studies have already been carried out to successfully improve the oxidation resistance of TiAl, as stated in the review by R. Pflumm et al. [6]. To a lesser extent, some corrosion

studies are described in the literature [7–10].

Therefore, to enhance the corrosion resistance of TiAl, we modified its surface composition by adding a noble and inert element. Given its high compatibility with titanium, we developed a specific coating based on a gold (Au) electrolytic deposit to protect a  $\gamma$ -48-2-2 alloy (nominal composition Ti-48Al-2Cr-2Nb in at. %) against corrosion by NaCl and oxidation [11]. The coating obtained after diffusion of 20  $\mu$ m of gold, for 1 h at 900 °C and at 10<sup>-5</sup> Pa, comprised two layers: an aluminoformer upper layer TiAlAu<sub>2</sub> and a lower layer TiAlAu. After 1000 h of cyclic oxidation at 800 °C, the formation of a dense but non-homogeneous protective alumina layer was observed over the entire surface of the sample. Indeed, some more degraded areas appeared locally under the layer of alumina. Since the  $\gamma$ -48-2-2 alloy has a gamma-grain equiaxed microstructure (TiAl) with a few small  $\alpha_2$ -phase precipitates (Ti<sub>3</sub>Al) at the grain boundaries, it was assumed that these degradations could be related to the presence of the  $\alpha_2$  precipitates at the grain boundaries of the alloy.

In order to separate the contributions of each phase, so as to be able to validate the latter hypothesis, the present work investigates the microstructures of Au-diffused coatings formed on single-phased samples: a stoichiometric TiAl alloy (called  $\gamma$ -TiAl), i.e., an alloy whose microstructure is a  $\gamma$ -grain microstructure with no  $\alpha_2$  phase precipitates, and a stoichiometric  $\alpha_2$ -Ti<sub>3</sub>Al alloy without any  $\gamma$  phase.

To check for destabilization of the upper TiAlAu<sub>2</sub> layer formed over

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**Table 1**

Characteristics of the 29  $\mu\text{m}$  Au-coated  $\gamma$ -TiAl specimens. Temperature of the vacuum diffusion treatment: 900 °C. \*Thicknesses measured from SEM micrographs.

$\gamma$ -TiAl	Vacuum diffusion duration (h)	TiAlAu <sub>2</sub> thickness* ( $\mu\text{m}$ ) $\pm$ 2 $\mu\text{m}$	TiAlAu thickness* ( $\mu\text{m}$ ) $\pm$ 2 $\mu\text{m}$
Specimen A	1	48	21
Specimen B	2	43	32
Specimen C	3	37	35
Specimen D	7	24	40
Specimen E	13	15	60
Specimen F	113	0	74

the  $\gamma$ -TiAl alloy after long Au diffusion treatments, the stability of the compositions of the two layers, TiAlAu<sub>2</sub> and TiAlAu, has been studied. For this purpose, the experimental thicknesses of these layers have been compared to those calculated by a typical growth model for layers with a very narrow homogeneity range [12].

In order to characterize the phases formed on the  $\alpha_2$ -Ti<sub>3</sub>Al alloy after Au diffusion, different analysis techniques were combined. Finally, isothermal oxidation tests at 800 °C were conducted to determine the ability of the Au-diffused  $\alpha_2$ -Ti<sub>3</sub>Al alloy to form dense and protective alumina.

## 2. Experimental procedure

$\gamma$ -TiAl and  $\alpha_2$ -Ti<sub>3</sub>Al alloys prepared by double vacuum arc melting, re-melted seven times and annealed, were used as substrates for the coating experiments. Their bulk composition (at.%) was measured to be Ti50Al50 and Ti75Al25 for the  $\gamma$  and  $\alpha_2$  alloys, respectively (induction coupled plasma technique). The alloy microstructures were checked by means of scanning electron microscopy.

Specimens were cut into 10\*15\*2.5 mm parallelepiped blocks by electro-discharge machining. After being ground down to an 800-grid finish using SiC paper, the specimens were sandblasted and then ultrasonically cleaned using alcohol. The Au deposition experiments are described in Reference [11].

After Au deposition, the specimens were heat-treated in a vacuum furnace ( $10^{-5}$  Pa, heating rate of 10 °C/min up to 900 °C with a threshold from 1 to 113 h). The cooling rate was 15 °C/min. Specimens coated according to these steps (Au deposit and vacuum heat treatment) are called Au-coated specimens throughout the remainder of this article.

Surface and cross-sectional examinations were performed using a high-resolution field emission gun scanning electron microscope (FEG-SEM Zeiss Merlin and Gemini). Secondary (SE) and back-scattered electron (BSE) imaging modes were used. Energy dispersive X-ray spectroscopy (EDS-SDD with SAMx microanalysis software [13]) was used to determine the elemental composition and mapping of the regions of interest. In order to ensure state-of-the-art quantitative composition measurements, long-time acquisition and metallic standards (Al, Ti, Au, and Ti-50Al) were used.

X-ray diffraction (PANalytical Empyrean diffractometer, Cu-K $\alpha$  radiation) was also used in the Bragg-Brentano reflection geometry. To identify the crystallized phases of the inner layers of the coating, mechanical grinding down to grid 4000 was performed, followed by surface SEM examinations.

Electron backscatter diffraction (EBSD) is used in this study to locate crystallized phases previously identified by X-ray diffraction. Indeed, EBSD has been proven to be a powerful tool for phase identification in multiphase materials, by analyzing Kikuchi patterns or electron backscattered diffraction patterns (EBSPs) [14]. At each pixel, an EDS spectrum is acquired while the EBSD pattern is collected. The EDS peaks are identified to determine the chemical element present and databases are searched for all crystals that match the chemistry. These tables are compared sequentially with the interplanar angles and lattice parameters measured from the EBSD pattern. The phases having provided the best match and fit are selected and compared with those

determined by surface XRD analysis. All of these experiments were performed on a FEG-SEM ZEISS Merlin with an EBSD system (TSL instrumentation software and NORDIFF detector) and EDS system (SAMx software and SDD detector). Moreover, a dedicated EDS mapping experiment at 8 kV provided not only spectral imaging to qualitatively differentiate intermetallic phases presenting a similar composition, but also – through a special quantification procedure available in the SAMx software – chemical quantitative imaging, as reported in a previous article [15].

800 °C isothermal tests were carried out in an open, horizontal furnace.

All of the compositions are given in at.%.

## 3. Experimental results

### 3.1. Characterizations of Au-coated $\gamma$ -TiAl and $\alpha_2$ -Ti<sub>3</sub>Al specimens

#### 3.1.1. Influence of the heat treatment duration on Au-coated- $\gamma$ -TiAl specimens

The characteristics of the Au-coated  $\gamma$ -TiAl specimens analyzed in this section are given in Table 1.

As already published [11], when vacuum heat treating (900 °C, 1 h) a 29  $\mu\text{m}$  Au coating that was deposited on a  $\gamma$ -TiAl alloy, the initial Au layer was consumed completely and the formed coating was composed of TiAlAu<sub>2</sub> and TiAlAu layers from the surface to the bulk alloy (Fig. 1, Spots 1 and 2, respectively). A zone enriched in Au is present (Fig. 1, Spot 3) at the interface between the lower TiAlAu layer and the  $\gamma$ -TiAl alloy. Semi-quantitative EDS measurements of the layers are given in Table 2. Previous work [11] showed that the upper TiAlAu<sub>2</sub> layer, which contains around 25% at. of Al, transformed into an adequately dense and protective alumina layer during oxidation at 800 °C, but the TiAlAu layer did not effectively provide good resistance to oxidation.

The thicknesses of the TiAlAu layer and of the diffusion zone enriched in Au increased according to the duration of the diffusion dwell period, while that of the TiAlAu<sub>2</sub> layer decreased (Fig. 2). The duration of the diffusion treatment leading to the total consumption of the TiAlAu<sub>2</sub> layer was estimated at 20 h (Fig. 3). Even after long diffusion treatments, no additional phase was identified.

It should be noted that although the layer thicknesses vary, their chemical composition remain constant over a long diffusion dwell time.

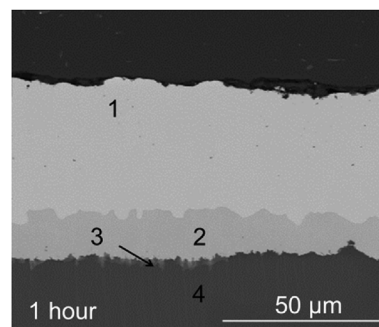


Fig. 1. Typical SEM micrograph of a 29  $\mu\text{m}$  Au-coated  $\gamma$ -TiAl alloy -Specimen A.

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