Intermetallics 34 (2013) 112–121

Contents lists available at SciVerse ScienceDirect

Intermetallics

journal homepage: www.elsevier.com/locate/intermet

Oxidation of Ti–46Al–8Ta in air at 700 °C and 800 °C under thermal cycling conditions

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ARTICLE INFO

Article history:
Received 28 July 2012
Received in revised form
29 October 2012
Accepted 30 October 2012
Available online 11 December 2012

Keywords:
A. Titanium aluminides, based on TiAl
B. Oxidation

ABSTRACT

The Ti–46Al–8Ta alloy was tested for oxidation resistance in laboratory air under thermal cycling conditions (1-h cycles) at 700 °C and 800 °C. Reaction progress was followed gravimetrically. After exposure, the specimens were subjected to systematic analyses comprising X-ray diffraction (XRD), scanning electron microscopy (SEM), transmission electron microscopy (TEM), and energy dispersive X-ray spectroscopy (EDS). Marker method was used to assess the mechanism of scale growth. Nano-indentation and scratch test were performed to evaluate scale adhesion and micromechanical properties.

1. Introduction

Titanium aluminides have been intensively studied for more than 20 years, as prospective structural materials [1,2]. The advanced third and fourth generation two-phase ($\gamma + \alpha_2$) alloys with a fully lamellar microstructure [3–5], are recognized as the most promising for applications in the automotive [6,7] and aerospace industries [8,9]. They have low densities 3.8–4.0 g/cm^3, good high-temperature specific strength, excellent creep properties and reasonable oxidation resistance up to 700 °C [10,11]. The following properties are regarded as major disadvantages: low room-temperature ductility and fracture toughness [12], insufficient oxidation resistance at temperatures exceeding 800 °C [13], difficult and high-cost production [14]. Moreover, at elevated temperatures titanium aluminides are prone to embrittlement caused primarily by dissolved oxygen and hydrogen [15,16].

Further improvement of properties is seen in alloying and/or surface engineering. A great variety of alloying additions and coating systems have been investigated, so far [17–22]. However, only a few papers deal with the oxidation behaviour of the TiAlTa alloys [23,24]. As reported recently [25], titanium aluminide alloys with Ta as a ternary addition have a number of advantages over those with Nb: easier processing, reproducible microstructure and mechanical properties being among the most important. It is not clear, whether and how tantalum affects oxidation resistance. It has been reported lately [26] that compared with niobium, tantalum has a more pronounced effect on oxidation rate, particularly at 1000 °C.

The aim of this work was to assess the oxidation resistance of a Ti–46Al–8Ta alloy in laboratory air at temperatures not exceeding those mostly used in mechanical tests, i.e. 700 °C and 800 °C.

2. Experimental

The experimental material was Ti–46Al–8Ta with a fully lamellar microstructure consisting of γ-TiAl and α_2-Ti_3Al (Fig. 1). An ingot, 13 mm in diameter, produced by horizontal centrifugal casting was cut into pellets (Fig. 2), 0.8–1.0 mm thick, using a diamond saw. For the oxidation studies, surface of the specimens was ground with emery paper to 1200 grit number, washed with distilled water and acetone. The weighed specimens were placed in alumina crucibles to collect any spalled material during the heating and cooling cycles. Each thermal cycle consisted of heating to the desired temperature, i.e. 700 °C or 800 °C, exposure at constant temperature for 1 h (hot dwell time) and cooling in static air. Heating rate was 50 °C/min. Overall cooling time was about 25 min. The fastest temperature drop of 100 °C/min was registered during the first 3 min. The cold dwell time of 15 min was measured from the moment when the specimens attained the temperature of 100 °C. An automatic laboratory setup was used for thermal cycling experiments. Specimens were usually weighed once a day on

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http://dx.doi.org/10.1016/j.intermet.2012.10.014

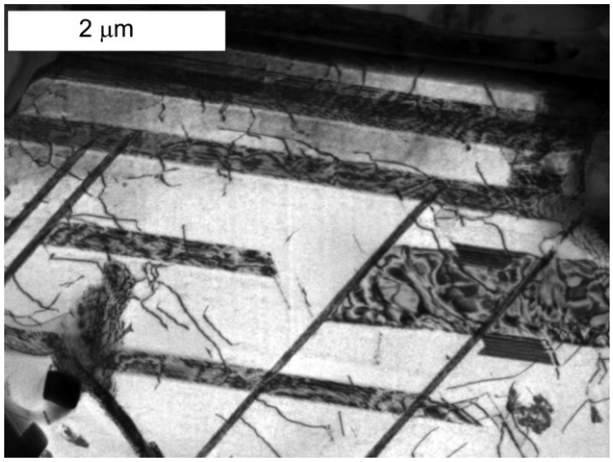

Fig. 1. TEM image of Ti–46Al–8Ta microstructure with visible dislocations.

working days using a laboratory balance with an accuracy of 10^{-4} g. For weighing, the specimens were transferred to a desiccator as soon as their temperature dropped below 100 °C. The overall time spent for weighing was 15–25 min, depending on the number of samples, so the weighing procedure did not largely affect the number of cycles per 24 h, which was 13–14 on average. The scales were examined by means of light microscopy (EPIPHOT 300, Nikon) and scanning electron microscopy (Nova Nano SEM 200). Phase and chemical compositions of the oxidation products were analysed by X-ray diffraction (Seifert XRD7) and energy dispersive X-ray spectroscopy (EDS). Cross-sections of selected specimens were examined by SEM using secondary and backscattered electron images (SEI and BEI) as well as by transmission electron microscopy (TEM-PHILIPS CM 20–200 kV TWIN). The oxidation mechanism was assessed by marker experiments. Gold markers in the form of regularly distributed islands were deposited onto the alloy surface by magnetron sputtering. The specimens with the gold markers were isothermally oxidized in air for 80 h at 800 °C. Cross-sections of the specimens were subsequently examined by SEM and the location of gold markers was detected by EDS. Some specimens were prepared for hardness measurements in the near-surface region and scale adhesion after exposure, using Micro-CombiTester (CSEM). These specimens were ground with emery papers to 2000 grit number and isothermally oxidized at 700 °C for 120 h. Indentation hardness and Young's modulus were measured using a Vickers tip. Six indentation cycles were done on each specimen. The applied load gradually increased to 10 mN at a speed of 20 mN/min. Maximum loading time was 5 s. All calculations were done using the Oliver & Pharr method. The scratch test was performed with a Rockwell C diamond tip. Three measurements were done on each specimen with a load increasing gradually from 0.03 N to 30 N at a speed of 3 mm/min. The maximum scratch length was 3 mm.

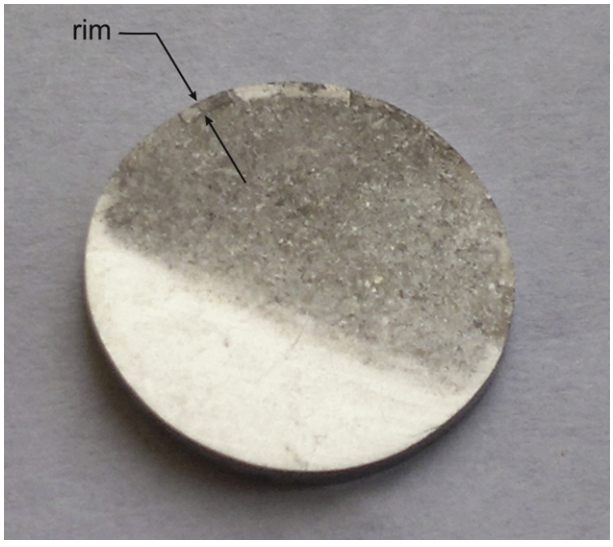

Fig. 2. Digital picture of a Ti–46Al–8Ta pellet half-etched with Kroll's reagent to reveal a rim along the circumference.

3. Results

3.1. Oxidation kinetics

Fig. 3 shows the results of mass change measurements during cyclic oxidation in air at 700 °C and 800 °C. As can be seen, the overall mass changes after a 300-h exposure at 800 °C did not exceed 0.4 mg/cm^2. The fluctuations were probably due to specimen handling since no scale debris were collected in alumina crucibles and the surface of specimens after oxidation was smooth without visible cracks or exfoliation.

Digital pictures of Ti–46Al–8Ta specimens after 20 and 300 oxidation cycles at 700 °C and 800 °C are presented in Fig. 4. The specimens have different colours: after a short time these are shades of golden and green, and after a longer time shades of grey. A rim, about 1 mm thick, can be seen along the circumference of the specimen in Fig. 4a, similar to the one visible on the etched part of the specimen in Fig. 2.

3.2. Scale morphology and composition

SEM images of specimen surfaces after 20 and 160 h of oxidation at 700 °C and 800 °C with the average composition determined by EDS are shown in Fig. 5. The surface layer is extremely fine grained, with submicrometric grain sizes. Grinding scratches are still visible on the surface of specimens oxidized at 700 °C, which is indicative of the presence of a very thin and most likely discontinuous oxide layer. After oxidation at 800 °C for 160 h, larger crystalline protrusions become visible. The average atomic ratio Al:Ti on the surface increases with exposure time, both at 700 °C and 800 °C. The Al:Ti atomic ratio is higher for specimens oxidized at 800 °C after the same exposure time.

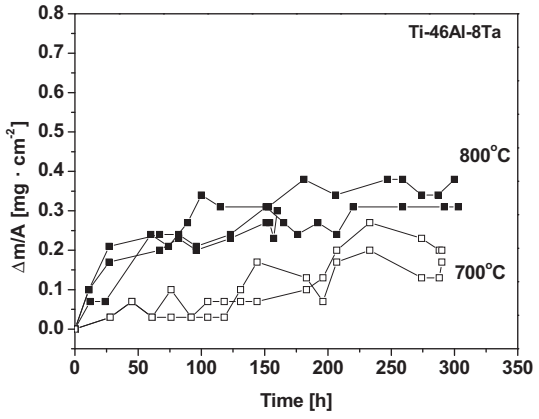

Fig. 3. Mass change vs. time data for Ti–46Al–8Ta during cyclic oxidation (1-h cycles) to 700 °C and 800 °C in air.