Available online at www.sciencedirect.com
SCIENCE DIRECT
Scripta Materialia 54 (2006) 645–648
Scripta MATERIALIA
www.actamat-journals.com

Evidence of α-nanophase heterogeneous nucleation from ω particles in a β-metastable Ti-based alloy by high-resolution electron microscopy

F. Prima [a,1], P. Vermaut [b], G. Texier [a], D. Ansel [a], T. Gloriant [a,*]

[a] *Institut National des Sciences Appliquées de Rennes (INSA), Groupe de Recherche en Chimie-Métallurgie, 20 Avenue des Buttes de Coesmes, CS 14315, F-35043 Rennes, France*

[b] *Ecole Nationale Supérieure de Chimie de Paris (ENSCP), LPCS, Groupe de Métallurgie Structurale, 11 rue Pierre et Marie Curie, F-75231 Paris, France*

Received 21 July 2005; received in revised form 23 September 2005; accepted 13 October 2005
Available online 2 November 2005

Abstract

The precipitation of nanostructured ω and α phases by heating a metastable Ti-based alloy was observed by high-resolution transmission electron microscopy. The ω_{iso} precipitates formed are shown to behave as nucleation sites for an extremely fine and dispersed α phase leading to a final stable nanostructured (β + α) duplex alloy.

Keywords: Titanium alloy; Phase transformations; Nanostructure; High-resolution electron microscopy (HREM)

1. Introduction

Over the last few years, beta Ti alloys have become an ever more important class of material because of their unique combination of high strength, excellent hardenability, low density and good corrosion resistance [1]. However, cost is becoming an increasingly important parameter. Recently, a metastable low cost beta (LCB) Ti-based alloy has been developed by TIMET to be used as a high performance substitute for more classical materials in non-aerospace new technologies [2].

It has been shown that in most titanium alloys, the homogeneous nucleation and subsequent growth of finely dispersed intragranular α phase was accompanied by considerable strengthening and a general improvement in mechanical properties (strength and toughness are enhanced while maintaining ductility to an acceptable level) [3]. The persistent problem is that the α phase generally appears preferentially at grain boundaries, on intragranular defects and along dislocation lines, making it rather difficult to obtain a uniform and dispersed α phase distribution. Nevertheless, in beta metastable Ti alloys (obtained by quenching the alloy from the beta phase field) the precipitation of nanoscale ω phase particles is observed upon heating. This additional phase has been widely observed to act as a heterogeneous nucleation site for the α phase. In high misfit systems where isothermal ω forms as cuboidal precipitates, the α phase was shown to nucleate at ledges of dislocations which develop at the β/ω interface [4]. In low misfit systems where isothermal ω forms as ellipsoidal precipitates, a two-step aging process was recently proposed: ω precipitation at 250 °C and subsequent α precipitation at temperature higher than 300 °C. A uniform and finer distribution of α precipitates was obtained in the Ti–15V–3Cr–3Sn–3Al alloy by this method [5]. However, the ellipsoidal ω precipitates' role in low misfit systems (such as LCB titanium) has not been clarified and the aim of this paper is to study, using high-resolution transmission electron microscopy (HREM),

* Corresponding author. Tel.: +33 2 2323 8519; fax: +33 2 2323 8540.
E-mail address: thierry.gloriant@insa-rennes.fr (T. Gloriant).
[1] Present address: LPCS, Groupe de Métallurgie Structurale, ENSCP, F-75231 Paris, France.

1359-6462/$ - see front matter
doi:10.1016/j.scriptamat.2005.10.024

the mechanism of phase transition between ω and α in the medium temperature range (350–450 °C) for Ti–6.8Mo–4.5Fe–1.5Al (a low misfit system).

2. Experimental

The composition of the LCB Ti rod provided by TIMET is 87.2wt.%Ti–6.8wt.%Mo–4.5wt.%Fe–1.5wt.% Al. From the rod, 3 mm thick and 10 mm width plaque-like samples (30 mm long) were machined, solution-treated at 850 °C for 30 min (beta phase field) in a high vacuum furnace (10^{-7} mbar) and were quenched in water in order to retain the beta phase at room temperature in a metastable state.

Heat treatments of the beta metastable alloy were carried out in a tubular furnace under high vacuum (10^{-7} mbar) from room temperature to the desired temperature with a heating rate of 5 °C/min.

Thin foil specimens to be used for transmission electron microscopy (TEM) are obtained by mechanical grinding to 200 μm and twin jet electropolishing (6% perchloric acid in acetic acid; 22 V). HREM observations were carried out on a Topcon 002B high-resolution electron microscope operating at 200 kV with a point to point resolution of 0.18 nm. The dark-field images were recorded on at least two spots of two different variants of the omega phase.

3. Results and discussion

The formation of athermal omega phase (ω_{ath}) during quenching has already been reported. A $(1\,1\,1)_\beta$ plane collapse model has been proposed to explain the $\beta \rightarrow \omega_{ath}$ displacive transformation: the ω lattice can be obtained by collapsing one pair of (1 1 1) planes to the intermediate position leaving the adjacent (1 1 1) planes unaltered. The usual orientation relationships between ω and β resulting from this displacement controlled transition were reported to be $(0\,0\,0\,1)_\omega \| (1\,1\,1)_\beta$ and $[1\,1\,\bar{2}\,0]_\omega \| [1\,1\,0]_\beta$ [6,7].

Upon heating, ω_{iso} phase, also hexagonal, can precipitate irreversibly during ageing, implying diffusional phenomena. An example of the $(\beta + \omega_{iso})$ structure obtained after heating the beta metastable sample up to 300 °C is presented in Fig. 1. On the dark-field TEM image (Fig. 1a), ellipsoidal ω phase precipitates (in bright) dispersed through the β matrix are observed and their sizes are nanometer in scale (<10 nm in diameter). Fig. 1b shows the corresponding diffraction pattern and the positions of the reflections detected are consistent with those expected from the hexagonal structure of the ω phase. On this diffraction pattern, and according to the zone axis, two ω variants are detected (ω_1 and ω_2), although four are normally possible in the crystallographic system studied here.

In order to investigate the precipitation mechanism of the α-phase occurring in the medium temperature range (350–450 °C), the beta metastable LCB sample previously heated to 300 °C was re-heated from room temperature to 400 °C (5 °C/min). Examples of TEM micrographs are presented in Fig. 2.

A direct relationship between ω and α is evident in the TEM dark-field image (presented in Fig. 2a). Homogeneous dispersion of α-phase platelets as large as the ω precursor ellipsoidal particle is easily visible on this micrograph.

From the corresponding diffraction pattern presented in Fig. 2b, an additional set of spots can be observed in comparison with the one presented in Fig. 1b (same $[1\,1\,0]_\beta$ zone axis). These spots are localized in the middle of the four spots detected for ω and are clearly those related to the presence of the α-phase. Consequently, the ω_{iso} particles are clearly observed as the precursor phase for α heterogeneous nucleation.

The ability of ω to act as a heterogeneous nucleation site for the α phase has been widely observed in high misfit systems where isothermal ω forms as cuboidal precipitates and where α phase nucleates at ledges or dislocations on the β/ω interfaces [8]. However, the role played by ellipsoidal ω precipitates in low misfit systems (such as LCB Ti) in the nucleation of the α phase has not been clearly defined.

There is an important controversy about the formation mechanism of this α phase in low misfit systems. According to some authors [9], it appears at some distance from the β/ω interface. It was shown [10] that a local enrichment in the

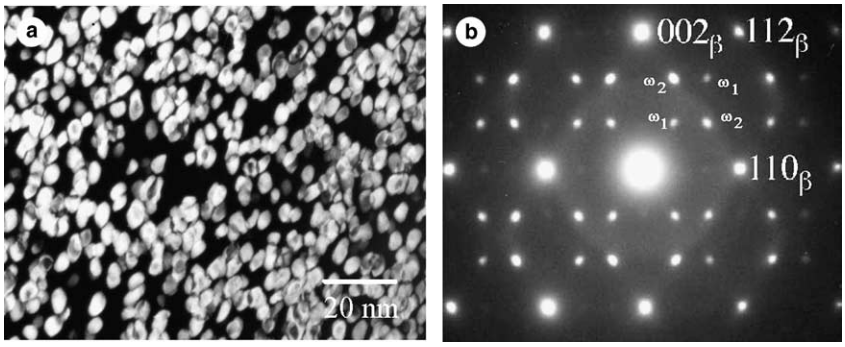

Fig. 1. Dark-field TEM image of ω_{iso} phase dispersed in a β matrix (a). Corresponding TEM diffraction pattern with a $[1\,1\,0]_\beta$ zone axis (b) (heat treatment to 300 °C, heating rate of 5 °C/min).