The indirect influence of the ω phase on the degree of refinement of distributions of the α phase in metastable β-Titanium alloys

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ABSTRACT

Non-conventional transformation pathways have been exploited to determine the degree of refinement of the microstructure of a metastable β-Ti alloy. This has involved a set of heat-treatments where samples, initially quenched to room temperature (RT) from above the β transus are slowly heated to 350 °C, then isothermal annealed at 350 °C for various times prior to up-quenching to 600 °C and again isothermally annealed at that temperature, followed by quenching to RT. Interrupted treatments have also been performed so that the factors influencing the development of the final microstructure could be determined. Microstructures have been characterized by scanning and transmission electron microscopy, and atom probe tomography. It has been found that a distribution of the α phase may be formed whose scale is intermediate between the "super-refined" and "refined" versions observed in previous research. The results are consistent with precursory isothermal ω precipitates playing an indirect role in the formation of the refined distribution of the α phase via a non-conventional transformation pathway.

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1. Introduction

Metastable β Ti alloys, such as Ti–5Al–5Mo–5V–3Cr (Ti-5553), compositions in wt.%, have been the subject of considerable interest in the recent past. One of the characteristics that provokes this interest is the refinement in microstructure compared with that exhibited typically by the more commonly used α/β Ti alloys, for example, the alloy TIMETAL 6-4 (Ti–6Al–4V in wt.%). Here refinement refers specifically to the fine scale of the distribution of the α phase precipitated from β, that can be developed by varying heat-treatments following solution treatment above the β transus. There have been a number of assertions in the literature concerning factors that can influence the scale of the precipitation of α from β, generally invoking heterogeneous nucleation involving structural (e.g., the formation of the ω phase [1–9]) and compositional (e.g., phase separation of the β phase [10–12]) instabilities in these alloys. In general, these various proposed influences of instabilities involve phase transitions that follow non-conventional transformation pathways.

In recent work, the results of experiments performed on the metastable β alloy Ti-5553 show no phase separation in β, but that the ω phase does form on quenching to room temperature from β initially held at temperatures above the β transus. The first set of experiments involved step-quenching samples from above the β transus to temperatures above the stability range of the ω phase, i.e., where α forms from β in the absence of ω [13]. In these experiments, it was found that refined distributions of the α phase formed in samples quenched to temperatures ≤650 °C, and it was concluded that the pseudo-spinodal mechanism, i.e., one involving a non-conventional transformation pathway, was responsible for the refined distributions of the α phase observed [13–16]. The second set of experiments involved precipitating α from β in samples that initially contained a distribution of the ω phase [17]. In these experiments, samples were solution treated above the β transus, and quenched to room temperature to effect a distribution of the ω phase (i.e., athermal ω). Subsequently, these samples were heated fairly rapidly (at rates ≥50 °C/min) to 600 °C, and it was found that the scale and distribution of α which resulted were similar to that formed by the pseudo-spinodal mechanism [13]. It was shown experimentally that the ω transformed back to β during the heating of samples to 600 °C, and so the ω phase played no direct role in the nucleation of the α phase [17]. The third set of experiments followed the same experimental scheme as in Ref. [17], but heating rates in the range 20 °C/min to 1 °C/min, i.e., relatively slow heating rates, were employed [18]. Under these conditions, extremely refined distributions of α were formed, termed "super-refined", and based on results of both interrupted tests and phase field modeling, it was

concluded that the ω phase did indeed influence markedly the precipitation of the α phase.

It has been noted that ω particles may influence precipitation of the α phase by providing additional driving forces for nucleation through either compositional and/or stress variations associated with the presence of the particles. In the case of the slow heating experiments discussed above [18], the compositional variations were measured directly, which were used subsequently with CALPHAD modeling to deduce contributions to the driving force from composition. The stresses associated with the ω particles were assessed using Khachaturyan's microelasticity theory [19], and these have been used with the Burgers lattice correspondence [20] to determine an extra contribution to the driving force for nucleation from stress. It was not possible in that work to ascertain whether both of these factors made effective contributions to the increased driving force for nucleation, or whether one or the other is more dominant. Hence, the first aim of the present study was to determine the relative contributions of these two factors. The results of previous work [17,18] are consistent with the ω phase either playing no role in, or in marked contrast having a direct influence on, the nucleation of the α phase. To understand the role of ω fully, it is important to determine whether ω forming during heat-treatment of these metastable β alloys can play an *indirect* role in effecting refined distributions of the α phase. Hence, the second aim of the present research was to determine under what conditions an indirect role might be played by this metastable phase.

2. Experimental procedure

The alloy studied in this research was Ti-5553 which was kindly supplied by the TIMET Company. The composition of this material has been measured using wet chemistry, and reported previously as Ti–5.67Al–5.00Mo–4.92V–3.04Cr–0.48Fe–0.18O–0.002N [18]. Bars were cut using electrical discharge machining from the Ti-5553 (subscale: 1.5 mm × 3 mm × 40 mm) for heat-treatment studies. The heat-treatments were performed in Electrical–Thermal–Mechanical-Tester (ETMT) in vacuum. All samples were β solution heat-treated at 1000 °C for 30 min and subsequently fast cooled to room temperature.

After the β-solution treatment, two sets of experiments were performed. In the first set, samples were heated to 350 °C at a rate of 5 °C/min, and subsequently up-quenched to 600 °C and isothermally held for various lengths of time at that temperature before being fast cooled to room temperature. The second set of experiments involved samples being heated to 350 °C at a rate of 5 °C/min, and subsequently held isothermally at 350 °C for various lengths of time before being up-quenched to 600 °C. In both sets of experiments, interrupted tests were performed to permit the evolution of microstructure as a function of heat-treatment to be determined.

The microstructures of the various samples were first characterized using scanning electron microscopy (SEM), mainly using an FEI Sirion SEM operating at an accelerating voltage of 12 kV and mostly recording backscattered electron (BSE) images. Backscattered SEM images were analyzed quantitatively with a custom-developed image processing package, MIPAR™ [21]. The aerial number density of particles in given images was determined using a watershed routine for feature; the accuracy of these determinations is about ±10%. For finer scale microstructural characterization, site-specific TEM foils were prepared using the Focused Ion Beam (FIB) technique, employing an FEI Helios Nanolab 600 system. Foils were "polished" subsequently at low voltage using a Fischione Model 1040 Nanomill. TEM dark-field images and diffraction patterns were obtained on a Philips CM200 microscope at 200 kV and energy dispersive X-ray spectroscopy (XEDS) analyses were performed in an aberration-corrected (S)TEM, equipped with a SuperX (FEI) four-quadrant silicon drift detector, FEI Titan3™ G2 60-300 S/TEM at 300 kV. Atom probe needles were prepared using the FEI Nova 200 NanoLab. These needles were used in a local electrode atom probe (LEAP™) system from Imago Scientific Instruments, using the voltage mode at a temperature of 60 K, with an evaporation rate 0.2–1.0% and a voltage pulse fraction at 20% of steady-state applied voltage, for 3D atom probe (3DAP) tomography.

3. Results and discussion

Two sets of experiments have been performed. In both sets, samples were first solution treated above the β transus, and then quenched to room temperature. In this way, all the various experiments described below began with samples that contained a fine homogeneous distribution of athermal ω precipitates (as shown previously in Ref. [18]).

3.1. Experiment set #1: slow-heating to 350 °C, immediate up-quenching to 600 °C

The first set of experiments involved heating samples slowly, at 5 °C/min, to 350 °C in order to promote the transformation from athermal to isothermal ω, and, because of solute redistribution accompanying the development of this latter phase, effect compositional variations in the samples. Upon reaching 350 °C, samples were up-quenched to 600 °C, the temperature where previously refined distributions of the α phase in this particular alloy have been effected, and held isothermally for various times, as depicted in the schematic diagram in Fig. 1(a). In this way, it was hoped that the α phase would form in the presence of the compositional variations resulting from the isothermal ω phase, but not in the presence of the phase itself. The resulting distributions of the α precipitates
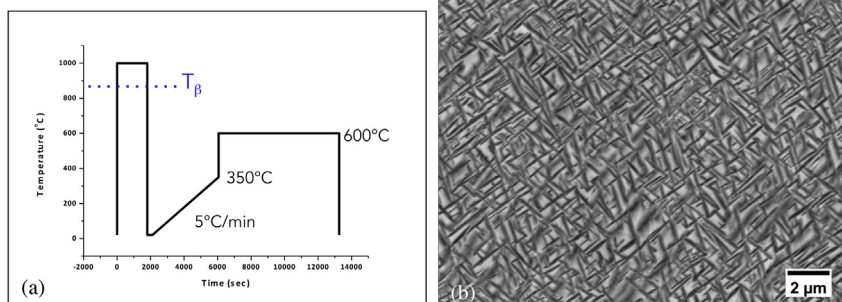

Fig. 1. (a) Heat-treatment scheme for experiment set #1. (b) Microstructure of Ti-5553 following the heat-treatment shown schematically in (a), i.e., slow heating to 350 °C, up-quenching to 600 °C, and isothermally held at 600 °C for 2 h.