

## The role of the $\omega$ phase on the non-classical precipitation of the $\alpha$ phase in metastable $\beta$ -titanium alloys



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### ABSTRACT

The metastable  $\beta$ -Ti alloy, Ti-5553, has been heat-treated to permit a determination of the role of the  $\omega$  phase on non-classical precipitation of the  $\alpha$  phase. Thus, samples containing the athermal  $\omega$  phase have been up-quenched to 600°C. The observations made are consistent with the activation of the non-classical, pseudo-spinodal, nucleation mechanism, with the  $\omega$  phase playing essentially no direct role. The  $\alpha/\beta$  interfacial structures have been shown to exhibit significant degrees of coherency compared with those in conventional  $\alpha/\beta$  alloys, and implications of this on lowering the interfacial energy, and so the activation for nucleation, are discussed.

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The microstructures of near- $\alpha$  and  $\alpha/\beta$  Ti alloys in the solution treated and aged (STA) condition, are characterized typically by an allotriomorphic distribution of the  $\alpha$  phase along prior  $\beta$  grain boundaries, and a fairly coarse distribution of Widmanstätten plates of the  $\alpha$  phase, separated by ribs of the  $\beta$  phase. It is generally considered that the allotriomorphs form by heterogeneous nucleation at the grain boundaries [1–3], and it has been claimed that the Widmanstätten plates nucleate from the allotriomorph [1,4] and also from plates so nucleated [5]. In marked contrast, the distribution of the  $\alpha$  phase in metastable  $\beta$  alloys, such as in the alloy Timetal-5553, especially following a  $\beta$ -annealed, slow cooled and aged (BASCA) heat-treatment, is rather refined [6]. This refined size scale of the uniformly distributed  $\alpha$  precipitates results in some interesting balances of properties [6], and recent research has focused on novel heat-treatments to determine both the mechanisms and pathways for such direct intragranular precipitation of  $\alpha$  [e.g., [7–10]]. It has often been suggested that the nucleation of the  $\alpha$  phase may involve some influence of the  $\omega$  phase, or some other instability such as  $\beta$  phase separation. For example, the hexagonal nature of both  $\omega$  and  $\alpha$  phases has been claimed to play a role [11], as has compositional variations resulting from the development of isothermal  $\omega$  [12,13] or  $\beta$  phase separation.

In an attempt to determine unambiguously the transformation pathways of intragranular precipitation in metastable  $\beta$ -Ti alloys, experiments were recently performed in which the influences of instabilities, such as the precipitation of the  $\omega$  phase and  $\beta$  phase separation, were deliberately avoided [7]. Thus, samples of the alloy Timetal-5553 were solution treated in the  $\beta$  phase field and then step-quenched to 600°C; at this temperature, the  $\omega$  phase is unstable and in this alloy there is no evidence of phase separation. It was determined that the precipitation of a refined distribution of the  $\alpha$  phase involved activation of the pseudo-spinodal mechanism [14,15], i.e., as a result of compositional fluctuations in the solid state, where such fluctuations cause the compositions of pockets of the solid solution to be smaller than that of the  $C_\alpha(T)$  composition such that a congruent  $\beta \rightarrow \alpha$  transformation may occur in the regions defined by the compositional pockets. In this way, a refined distribution of the  $\alpha$  phase may be produced, in the absence of influence from instabilities such as the  $\omega$  phase. The activation of the pseudo-spinodal mechanism is a *non-classical* transformation pathway for homogeneous precipitation of the  $\alpha$  phase in metastable  $\beta$ -Ti alloys.

In the present study, samples of the alloy Timetal-5553 have been employed that contain a distribution of athermal  $\omega$ , produced by  $\beta$  solution heat-treatment followed by quenching to room temperature. In this way, the role of the  $\omega$  phase, when deliberately made to be present in samples rather than being avoided as in the previous study [7], on the nucleation of the  $\alpha$  phase may be assessed. In the experiments described below, samples have been

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rapidly up-quenched to 600°C, i.e., the temperature where the pseudo-spinodal mechanism was found previously to be activated; results of experiments involving slow heating rates, rather than up-quenching, are described elsewhere [16].

Ti-5553 raw material was provided by TIMET Company; the chemical composition of the supplied material, measured by wet chemical analysis using Thermo Elemental IRIS Advantage ICP, was Ti-5.7Al-4.9V-5.0Mo-3.0Cr (compositions in wt%). Small blocks of the as-received Ti-5553 were  $\beta$  solution heat-treated at 1000°C for 30 min (the  $\beta$  transus for this alloy is  $\approx$ 855°C) in a tube furnace, and subsequently quenched to room temperature. Coupons were machined from these heat-treated blocks, measuring 1.5 mm  $\times$  3 mm  $\times$  40 mm, by electrical discharge machining. These coupons were then heat-treated in an Electro-Thermal-Mechanical-Tester (ETMT). The coupons were up-quenched to either 500°C or 600°C at a controlled rate of 100°C/min in the ETMT, under vacuum ( $\approx$ 10<sup>-3</sup> torr). Following heat-treatment, the samples were prepared for microstructural characterization using scanning electron microscopy (SEM), transmission electron microscopy (TEM), and aberration-corrected scanning/transmission electron microscopy (S/TEM). For SEM observations, observations were made in an FEI Sirion SEM at an accelerating voltage of 12 kV. TEM and (S)TEM samples were prepared in a Dual-Beam Focused Ion Beam (DB-FIB), and observations were made in an FEI Tecnai F20, operating at 200 kV, and an FEI Probe Corrected Titan3™ 80–300 S/TEM operating at 300 kV. High angle, annular dark-field (HAADF) imaging was performed on the latter instrument using a convergence semi-angle of 12 mrad at a camera length of 91 mm with an incident beam dwell time of 4  $\mu$ s/px and a current of  $\sim$ 60 pA; inner and outer detection angles were 34.8 mrad and 230.0 mrad, respectively.

Fig. 1(a) shows a TEM image, recorded in dark-field using weak reflections exhibited in the accompanying selected area diffraction pattern, of the microstructure typical of that in samples of Timetal-5553 that have been  $\beta$  solution heat-treated and quenched to room temperature. The weak reflections are due to the presence of athermal  $\omega$ , the size and distribution of which are revealed in the micrograph. Fig. 1(b and c) exhibit SEM backscattered electron images showing the results of up-quenching experiments, i.e., heating rapidly at a rate of 100°C/min, of samples, that had been initially  $\beta$  solution heat-treated and quenched to room temperature, to 600°C and then isothermally aged; Fig. 1(b and c) show the results of aging for 5 and 30 min, respectively. As can be seen, the up-quenching and aging treatments have resulted in a fine distribution of the  $\alpha$  phase. The microstructures in samples aged for 5 min and 30 min are fairly similar.

The microstructures shown in Fig. 1(b and c) appear to be very similar to those formed by step-quenching from above the  $\beta$

transus to 600°C, i.e., those that have formed via the pseudo-spinodal mechanism [7]. A comparison between the microstructures resulting from rapid heating (100°C/min), or step-quenching, to 600°C followed in both cases with isothermal annealing for 30 min at temperature, are shown in Fig. 2(a and b), respectively. These images have been processed using the MIPAR™ software package [17] to permit a determination of the areal number density of  $\alpha$  precipitates, with typical image segmentation being shown in Fig. 2(c and d). In these figures, discrete  $\alpha$  laths have been assigned random colors simply to aid in identifying them as separate features from their neighbors. It should be noted that the numbers obtained from these analyses have an accuracy of  $\approx$ ±10%, which is usually much smaller than the variation of microstructure from different areas of a given sample. To obtain statistically significant data, for each condition, five areas were chosen randomly and processed individually, from which average values were obtained. The numerical results obtained are as follows. For the case of rapid heating to 600°C the aerial number density of  $\alpha$  precipitates is  $\approx$ 1.5/sq  $\mu$ m, and for the case of step quenching, the aerial number density of  $\alpha$  precipitates is  $\approx$ 2.1/sq  $\mu$ m. Clearly, the microstructures are quantitatively fairly similar, and it is tempting to speculate that the same mechanism of precipitation occurs in the two heat-treatments, an assertion that would imply that the  $\omega$  phase plays essentially no role in the nucleation process.

It has been established (Fig. 1(a)) that the  $\omega$  phase is present at the beginning of rapid heating, while being absent in the step-quenching experiments, and it is important to establish whether, or not, this metastable  $\omega$  phase plays a role in  $\alpha$  nucleation. This has been done using the following two heat-treatments. In the first, a sample that had been  $\beta$  solution treated and quenched to room temperature, i.e., containing athermal  $\omega$ , was then rapidly heated (100°C/min) to 500°C, and subsequently quenched to room temperature. The microstructure of this sample is represented by the dark-field TEM image shown in Fig. 3(a). This dark-field image has been recorded from a set of  $\omega$  reflections, close to the 1/3 and 2/3 {112}  $\beta$  locations, marked by the red ellipse on the [011]  $\beta$  zone axis diffraction pattern shown as an inset in Fig. 3(a). As can be seen, there is a distribution of athermal  $\omega$ , similar in size scale and morphology, to that shown in Fig. 1(a). It appears that at 500°C, the athermal  $\omega$  phase present prior to rapid heating has become unstable and transformed to the  $\beta$  phase. Upon quenching to room temperature, athermal  $\omega$  has formed again. The second heat-treatment involved the rapid heating of a sample, that had first been  $\beta$  solution treated and quenched to room temperature, to 600°C and subsequently quenched to room temperature (with no aging). The microstructure of this sample is represented by the dark-field image shown in Fig. 3(b) and consists of a

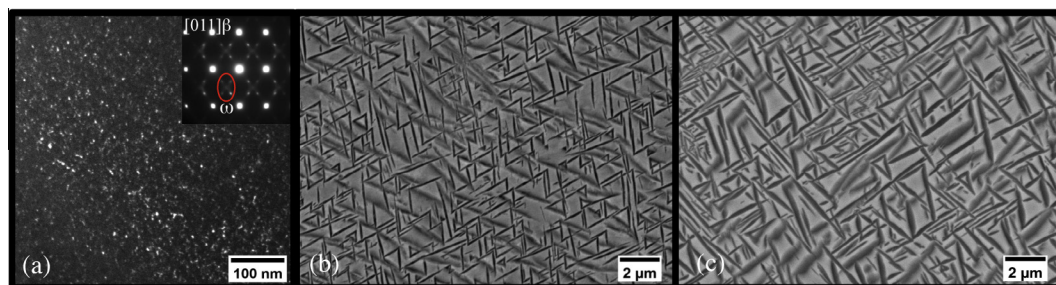


Fig. 1. (a) TEM image and SAD pattern showing the microstructure of Ti-5553 following solution heat-treatment, and quenching to room temperature; reflections in the red ellipse are from the  $\omega$  phase (b and c) Backscattered electron SEM images showing the microstructure of Ti-5553 heat-treated as in (a), followed by rapid heating to 600°C at a rate of 100°C/min and then isothermally aged for 5 min (b), and 30 min (c). (For interpretation of the references to color in this figure legend, the reader is referred to the web version of this article.)

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