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Regular Article

Nucleation driving force for ω-assisted formation of α and associated ω morphology in β-Ti alloys

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ABSTRACT

The structural and chemical changes at ω/β interfaces and the evolution of the morphology of ω in a near-β alloy during isothermal ageing at 573 K were investigated by atom probe tomography and aberration-corrected high-resolution transmission electron microscopy. Ledges and local O enrichment at semi-coherent isothermal ω interfaces are proposed to provide the key driving force for nucleation of ω-assisted α. Following nucleation of α, the morphology of ω evolves from ellipsoidal to rod-like, induced by rapid consumption of ω by α.

β titanium alloys have attracted considerable attention in aerospace and biomedical applications due to their superior mechanical properties and biocompatibility [1]. The mechanical properties can be improved by precipitation of uniform distributions of fine α phase, nucleated from the metastable ω phase [2]. The ω-assisted formation of α has been widely adopted to optimise mechanical properties via appropriately designed heat treatments [2]. Recently, athermal ω [3, 4], i.e. incommensurate (embryonic) ω [5], was demonstrated to not directly assist α formation; while isothermal ω, i.e. commensurate ω, actively assists α formation [6]. However, the key driving force for nucleation in the ω-to-α phase transformation remains elusive.

For large ω/β misfit systems in which cuboidal-shaped ω precipitates form (e.g. Ti-V [2, 4]), defects such as ledges at the ω/β interface provide nucleation sites for the α phase [4, 7]. In low misfit systems, such as Ti-Mo [2, 8], ellipsoidal ω precipitates form within β due to elastic strain energy considerations. The precise role of coherent ω/β interfaces in formation of the α phase remains unclear. A recent study asserted that elastic stresses associated with coherent ω/β interfaces, along with compositional variation, potentially trigger formation of the α phase [3]. In previous work [9], we observed ω phase surrounded by O-enriched regions (within α), which may assist nucleation of α. However, it is unclear if either compositional variation or elastic stress, or both are the dominant driving force for nucleation of α. Furthermore, ω can co-exist with α upon isothermal ageing [10, 11]. Changes to the morphology of the ω phase during co-growth with α have not been reported. Understanding the morphology evolution of the ω phase, and associated structural and compositional changes at or around the ω/β interface, may clarify the stage at which ω assists α formation. Therefore, further experimental evidence is required to determine the dominant nucleation driving force and preferred nucleation sites for ω-assisted α formation. Atom probe tomography (APT) is a powerful technique that provides quantitative three-dimensional information on elemental distributions at the atomic scale [12]. In this study, we combine APT with aberration-corrected TEM to investigate structure and chemistry at the ω/β interface, and the morphology and chemical evolution of the ω phase in an isothermally aged near-β Ti alloy, with the aim to i) ascertain the key nucleation driving force for ω-assisted α, and ii) understand growth and dissolution of ω during the ω-to-α transformation.

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A new near-β Ti alloy, Ti-6Cr-5Mo-5 V-4Al (wt%) (Ti-6554), solution treated under vacuum at 1103 K, was used for our study [13]. Ageing treatments to promote formation of ω were conducted at 573 K for 12, 16, 32 and 48 h [14]. TEM samples were prepared using twin-jet electro-polishing (Tenupol-3, struers) with a solution containing $HClO_4$ (6%) and methanol at 253 K and 20 V. TEM was conducted in a JEOL-3000F operating at 300 kV and STEM experiments were carried out in a probe-corrected JEOL-ARM200F with cold field emission gun. High-angle annular dark field (HAADF) images were acquired with 68 mrad and 280 mrad inner and outer collection angles, respectively, with dwell times of 38 μs. APT samples were electropolished in a solution containing $HClO_4$ (5%) and methanol at 38 V and 243 K, and subsequently sharpened in a Zeiss Auriga FIB. APT experiments were conducted in a Cameca LEAP 4000× SI instrument at 50 K, with a target evaporation rate of 5 ions per 1000 pulses and laser energy of 80pJ. The composition of the quenched Ti-6554 measured by APT is Ti-(5.5 ± 0.1) Cr-(2.3 ± 0.1)Mo-(4.9 ± 0.1)V-(7.4 ± 0.1)Al-(0.32 ± 0.01)O, in at.%.

To investigate the nucleation driving force for ω-assisted α formation, the transitional stage at which α commences to form must be identified. According to our previous study [6], incommensurate embryonic ω forms below 12 h. At 12 h, a bimodal size distribution of ω precipitates, 2–4 nm and 4–8 nm in size, are observed in Fig. 1a, a dark-field image formed from diffuse reflections between 1/3 and 2/3 $(21\bar{1})_\beta$ along $[110]_\beta$ highlighted in the selected area diffraction pattern, inset. The smaller ω are incommensurate embryonic ω and the larger commensurate isothermal ω [6]. Another dark-field TEM image from a different region of the 12 h sample recorded using the same selected area aperture position reveals a small number of ultrafine plate-like features likely to be α based on their morphology (Fig. 1b). However, the volume fraction of plates was too low to give discernible reflections at 1/2 $(21\bar{1})_\beta$ positions. Nevertheless, observation of embryonic ω, isothermal ω and α at 12 h, suggests that the initial stages of ω-assisted formation of α occur at around 12 h. Aberration-corrected high-resolution HAADF/STEM was employed to view the atomistic structure at 12 h. Only small embryonic ω was captured from thirty regions. The enlargement in Fig. 1c shows five bright $\{112\}_\beta$ planes, separated by planes locally collapsed along $\langle 111\rangle_\beta$, characteristic of the ω phase [10]. The interface between ω embryos and the β matrix is fully coherent. After 16 h, co-located ω and α is observed in Fig. 1d. The selected regions marked 1, 2 and 3 correspond to regions of β, ω and α, respectively, as confirmed by fast Fourier transform (FFT) images. An [0001]-orientated α plate forms at the edge of the $[11\bar{2}0]$-oriented ω phase (Fig. 1d), consistent with the reported orientation relationship, $(\bar{1}11)\beta//(0001)\omega//(11\bar{2}0)\alpha$ [15]. The ω/β interface at 16 h is semi-coherent and ledges are observed at the interfaces between ω and [110]-orientated β and α. A previous study [4] has questioned our experimental data regarding the formation of ω-assisted α in [6], because fast Fourier transform (FFT) images were not shown. Here, we provide further evidence showing that the fine α precipitates form at the ω/β interface.

Compositional variation around ω and the chemistry of ω-assisted α were analysed by APT in the initial stages of ω-assisted formation of α at 12 h. No significant compositional partitioning was detected in β (7 out of 8 APT samples) via conventional APT analysis techniques. However, application of a customised point-by-point binomial analysis [6, 16] revealed nano-scale chemical modulation of Mo (reported in [6]), which facilitates formation of embryonic ω [11]. Mo isoconcentration surfaces at 1.2 at.% in Fig. 2a reveal mainly ellipsoidal ω embryos with a size of 3–4 nm. Only one APT dataset contained low densities of large isothermal ω co-located with ultrafine α, indicated by 5.0 at.% Al and 83.4 at.% Ti isoconcentration surfaces [6], respectively, in Fig. 2b. The majority of β is occupied by embryonic ω (highlighted by 1.2 at.% Mo isoconcentration surfaces). To assess chemistry at their interfaces of

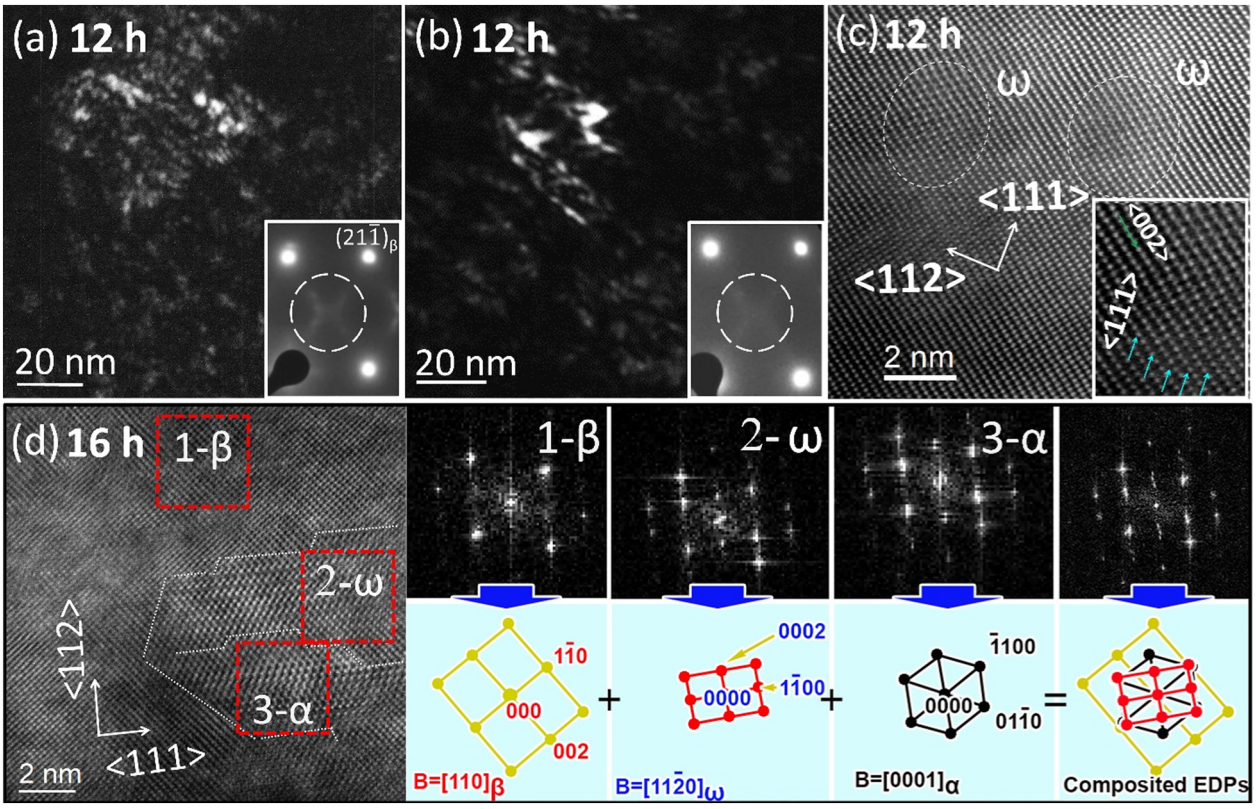

Fig. 1. (a–b) dark-field TEM images from two regions of Ti-6554 aged for 12 h, recorded using the reflections spots marked by the dashed lines in the enlargements of (a–b), and aberration-corrected HAADF-STEM images of Ti-6554 aged for (c) 12 h and (d) 16 h with associated FFT images of the red dashed regions in (d) marked 1, 2, 3 corresponding to β, ω, α respectively, and composited electron diffraction pattern (EDP) showing the orientation relationship $[110]_\beta//[0001]_\alpha//[11\bar{2}0]_\omega$. (For interpretation of the references to colour in this figure legend, the reader is referred to the web version of this article.)