



## Regular article

## The influence of aluminum and oxygen additions on intrinsic structural instabilities in titanium-molybdenum alloys

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## ABSTRACT

The influences of aluminum and oxygen additions on structural instabilities in the  $\beta$  phase of binary Ti-Mo alloys have been investigated, using conventional and aberration-corrected scanning/transmission electron microscopy and atom-probe tomography. An addition of 5 wt% Al to the alloy Ti-18 wt% Mo tends to decrease the stability of the  $2/3\langle 111 \rangle$  longitudinal phonon, compared with that of the phase O' formed by the  $\{110\}\langle 1\bar{1}0 \rangle$  transverse phonon. Experiments have been performed to assess whether interstitial oxygen influences these phase instabilities by adding yttrium to getter the matrix oxygen. It has been found that oxygen is not a necessary alloying addition to form the O' phase.

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The investigation of instabilities in the  $\beta$  phase of metastable  $\beta$  titanium alloys has attracted considerable attention in recent years because of their influence on various aspects of microstructural evolution and mechanical behavior in this alloy class [1]. For example a transformation pathway to the equilibrium  $\alpha$  phase using  $\omega$  as a precursor can produce extremely refined dispersions of  $\alpha$  in the alloy Ti-5553 [2] and has been exploited to produce very high strength levels in the alloy  $\beta$ -21S [3]. An alternative distinct nanodomain structure has been observed in metastable  $\beta$  alloys containing deliberate oxygen additions such as gum metal, by Yano et al. [4] and Tahara et al. [5]. The latter authors concluded that these nanodomains originated from a  $\{110\}\langle 1\bar{1}0 \rangle$  type transverse lattice modulation in the  $\beta$  phase [5] and that these nanodomains acted as barriers to transformation to orthorhombic martensite ( $\alpha''$ ). In a different study, Wang et al. [6] suggest that a Ti-Nb-Ta-Zr-O alloy (TNTZ-1.2O), alloy undergoes a 'strain glass transition' to 'martensitic nanodomains' [7,8]. The 'martensitic nanodomains' in this study appear identical to those reported in [4,5] based on similarities in diffraction. In all these studies, it has been suggested that the nanodomains form as a result of strain centers associated with oxygen atoms, with the implication [6–8] that oxygen additions are essential in the development of the unique properties of this class of alloys.

An alternative viewpoint for the formation of the  $\{110\}\langle 1\bar{1}0 \rangle$  nanodomains has been proposed in which the  $\{110\}\langle 1\bar{1}0 \rangle$  transverse wave is recognized to be a component of the martensitic transformation from  $\beta$  to the hexagonal  $\alpha'$  or the orthorhombic  $\alpha''$  martensite [9–11]. The martensite transformation occurs by the  $\{112\}\langle 111 \rangle$  shear that transforms the  $\{110\}_\beta$  planes into the (0001) or (001) planes of  $\alpha'$  or  $\alpha''$ , while a  $\{110\}\langle 1\bar{1}0 \rangle$  displacement wave leading to a shuffle on every alternate plane changes the stacking sequence of these planes, as proposed by Burgers in 1934 [12]. Thus it has been shown in [9] that when the  $\{110\}\langle 1\bar{1}0 \rangle$  shuffle occurs after the  $\{112\}\langle 111 \rangle$  shear (the Bain distortion) that transforms  $\beta$  to martensite, a domain structure related to these shuffles occurs within the martensite plates. However, if the shuffle precedes the shear, 12 variants of nanodomains can form completely coherently with the matrix since the parent cubic motif is retained by the shuffle. These domains have been imaged by ultra-high resolution high angle annular dark field imaging more recently in [10,11] and designated as O' (due to the orthorhombic symmetry, Cmc<sub>m</sub>, of the nanodomains and to distinguish them from the equilibrium, ordered, orthorhombic O phase observed in Ti-Al-Nb alloys [13] and the ordered, orthorhombic phase, O'', in aged metastable  $\beta$  alloys [14]).

The objective of this paper is therefore two-fold. Firstly, the role of an  $\alpha$  stabilizer on the relative stability of the O' and  $\omega$  phases is examined in two alloys, Ti-18Mo and Ti-18Mo-5Al (wt%). Secondly, a series of experiments is aimed at determining whether oxygen is a necessary alloying addition for the formation of the O' phase. It is well known

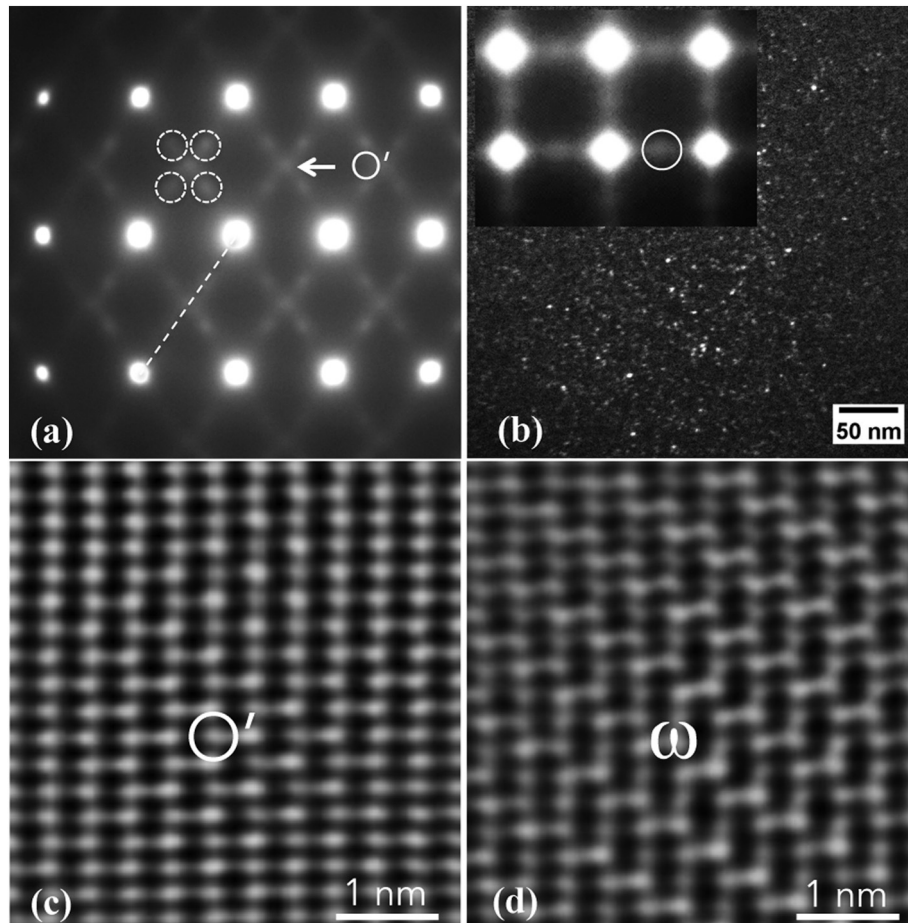
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that oxygen will be present in essentially all alloys prepared from commercially available sponge. To produce an alloy where it would be known with certainty that oxygen is not contained in the alloy matrix, additions of yttrium have been made so that in the heat-treated condition, particles of  $Y_2O_3$  would be formed because of the strong affinity of the rare earth element for oxygen (the thermodynamics of such Ti-rare earth alloys has been described previously [15]). Provided the concentration of the rare earth is such that there is an excess of yttrium in the matrix following formation of the oxide, the residual concentration of oxygen in the matrix will be at a vanishingly low value. Hence, yttrium was added into Ti-18Mo-5Al to essentially remove oxygen from the  $\beta$  matrix.

The Ti-18Mo alloy was the same as that used in a previous study [11]. The Ti-18Mo-5Al alloy used in current work was provided by the TIMET Corporation (Henderson, NV), and its composition measured by conventional wet chemistry, being Ti-17.29Mo-4.7Al-0.26O (wt%), of which the oxygen content is comparable to that in the Ti-18Mo. The as-received alloy (Ti-18Mo-5Al) was then annealed in a vacuum furnace at 1100 °C for 96 h and then furnace-cooled to room temperature, in order to promote its homogeneity. The bulk material was cut into 20 mm × 10 mm × 10 mm pieces using electrical discharge machining (EDM) for further heat treatment in a tube furnace. Sectioned Ti-18Mo-5Al samples were  $\beta$  solutionized at 910 °C, approximately 100 °C higher than the calculated  $\beta$  transus temperature (assessed using ThermoCalc), for 1 h and quenched into water at room temperature. In order to further reduce the oxygen level in the  $\beta$  phase matrix, 0.9 g pure yttrium (99.9%) was added to 22.8 g the binary Ti-18Mo button

(approximately 3.80 wt%) and 31.1 g the ternary Ti-18Mo-5Al button (approximately 2.81 wt%) using an arc-melter; in both of these yttrium containing alloys, there will be excess yttrium over that required to form  $Y_2O_3$ . These alloys, with the yttrium additions, were then  $\beta$  solutionized at 1000 °C for 30 min and subsequently quenched into water. Thin foils for transmission electron microscopy (TEM) and high angle annular dark field-scanning transmission electron microscopy (HAADF-STEM) analysis were prepared using the Dual Beam Focused Ion Beam (DB-FIB) technique, using an FEI Helios Nanolab 600 system, and the resulting ion beam damage on the foil surfaces was minimized using low voltage  $Ar^+$  ion milling in a Fischione Nanomill™ Model 1040. TEM dark-field images and diffraction patterns were recorded on a Philips CM200 transmission electron microscope operating at an accelerating voltage of 200 kV. Line profiles from a number of TEM diffraction patterns were analyzed using ImageJ, and for each alloy a representative set of data for each diffraction pattern is presented. HAADF-STEM images were recorded using a probe-corrected FEI Titan3™ 80-300 S/TEM operated at 300 kV, using a convergence semi-angle of 12 mrad and a collection semi-angle of 44 mrad, with an incident beam dwell-time of 4  $\mu s/px$  and a beam current of  $\approx 90$  pA. X-ray energy dispersive spectrometry (XEDS) analyses were performed in an aberration-corrected (S)TEM (FEI Titan3™ G2 60-300 S/TEM at 300 kV), equipped with a SuperX (FEI) four-quadrant silicon drift detector. Needle shape samples were prepared using the FEI Nova 200 NanoLab and analyzed in Cameca 3000X HR local electrode atom probe (LEAP) system, using the voltage mode at a temperature of 60 K, with an evaporation rate 0.5–0.7% and a voltage pulse fraction of



**Fig. 1.** The  $O'$  phase in Ti-18Mo-5Al. (a) SAD pattern with the electron beam parallel to  $\langle 110 \rangle_{\beta}$ , showing reflections consistent with the  $\omega$  and  $O'$  phases. (b) Dark-field TEM micrograph formed using the diffracted intensity from the  $O'$  phase (white circle) shown in the  $\langle 110 \rangle_{\beta}$  SAD pattern (inset). (c) and (d) HAADF images showing the  $O'$  and  $\omega$  phases, respectively, in as-quenched Ti-18Mo-5Al.

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