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### Regular Article

# Change in the deformation mode resulting from beta-omega compositional partitioning in a Ti—Mo alloy: Room versus elevated temperature

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#### 1. Main text

#### Over the last few decades, body-centered-cubic (bcc) beta $(\beta)$ titanium alloys have largely been exploited as structural alloys owing to the richness in their microstructural features [1,2]. These features which lead to a unique combination of high specific strength and ductility. excellent hardenability, good fatigue performance, and corrosion resistance make these alloys viable candidates for many applications, including aerospace, automobile, and orthopedic implants [1-5]. The mechanical properties of these alloys strongly depend on the various phases present; which can be controlled by thermomechanical treatments and/or alloy design. Recently a lot of such alloy design efforts have been based on the "d-electron method"- originally proposed by Morinaga [6–8]. This approach aims at providing a physical background to the phase stability and phase transformations in titanium alloys by connecting the values of two electronic parameters, Bo (the covalent bond strength between Ti and alloying elements) and Md (the mean d-orbital energy level concerning electronegativity and elements radius) to the stability of the $\beta$ phase. While the Bo-Md plots obtained from this approach can also evaluate the "stability" of the $\beta$ phase, i.e.,

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ABSTRACT

The effect of beta-omega compositional partitioning, introduced via isothermal annealing, on the deformation behavior of a Ti—Mo alloy was investigated via tensile testing at room and elevated temperatures. While beta with compositionally congruent athermal omega precipitates primarily exhibits twinning, after beta-omega compositional partitioning the same alloy deforms via slip. This change in deformation mode can be rationalized based on the solute content within the beta matrix, with reference to the Bo-Md plot. Tensile testing at elevated temperatures revealed that both conditions deformed via slip while exhibiting serrated flow.

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for a given concentration of  $\beta$ -stabilizers in the alloy, on quenching from the single  $\beta$  phase field, if the  $\beta$  will precipitate secondary phases like omega, martensite etc., or will retain its single phase *bcc* structure, it can also give insights into the deformation mode of these alloys.

Understanding  $\beta$  phase stability is critical since it is intricately associated with the deformation behavior of  $\beta$ -Ti alloys. Slip is the most common deformation mechanism observed in these alloys [9,10]. However, few alloy systems like Ti-1023 (Ti—10V—2Fe—3Al) are susceptible to deformation induced  $\alpha''$  martensite formation [11], while, on the other hand Ti—15Mo—5Zr deforms via {332} (113) twinning [12]. Recently, however, Sun et al. in their work on Ti—12Mo and Ti—Mo—W have shown that, using Bo-Md plot, both these deformation modes could be concurrently activated which would yield better mechanical properties [13,14]. Interestingly, such deformation-Bo-Md correlations are typically limited only to  $\beta$ -solutionized alloys where the microstructure exhibits no compositional partitioning between the coexisting phases [7,8,13]. Therefore, it is of scientific interest to test the applicability of Bo-Md plots on multi-phase systems in  $\beta$ -Ti alloys, involving compositional partitioning between the matrix and precipitate phases.

Towards this goal, the present investigation focuses on the deformation behavior of a model Ti-12 wt.% Mo alloy. Our choice of Ti-12 wt.% Mo was guided by three considerations: (i) it forms well developed athermal omega ( $\omega$ ) precipitates with fully collapsed {111} beta planes

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[15], (ii) annealing at temperatures below the  $\omega$  solvus results in pronounced compositional partitioning between the  $\beta$  and  $\omega$  phases, and (iii) prior detailed investigation of deformation behavior in  $\beta$  solutionized and quenched condition of the same alloy, reported in the literature [13]. The overarching goal of the present study is to compare and contrast the principal deformation mechanisms operating in this model Ti-12 wt.% Mo alloy, in the absence as well as presence of compositional partitioning between the  $\beta$  and  $\omega$  phases, at room and elevated temperatures.

The Ti–12 wt.% Mo alloy specimens were first  $\beta$  -solutionized at 900 °C for 30 min followed by water quenching. Subsequently, the  $\beta$ -solutionized specimens were annealed at 475 °C/48H to form well-developed  $\omega$  precipitates and promote compositional partitioning. Microstructural characterization was performed using scanning electron microscopy (SEM, FEI NovaNano SEM230, and transmission electron microscopy (TEM, FEI Tecnai F20-FEG TEM operating at 200 kV). Nanometer-scale compositional analysis was carried out using 3D atom probe tomography (APT) in a local electrode atom probe (LEAP) 3000× HR system from Cameca Inc. TEM foils and APT tips were prepared via a FEI Nova NanoLab 200<sup>™</sup> focused ion beam (FIB). Dogbone shaped tensile specimens of gage length ~3 mm, width ~1 mm and thickness ~0.4-0.6 mm were used for the mechanical testing, which was done under uniaxial tension at a strain rate of  $10^{-3}$  s<sup>-1</sup> at 25 °C (RT) and 300 °C (inside an oven). Details of the setup for the mechanical testing are presented elsewhere [16,17].

The microstructures obtained after  $\beta$  -solutionizing and subsequent annealing at 475 °C/48H are shown as dark-field TEM (DFTEM) images in Fig. 1(a) and (b) respectively. The dark-field images were recorded by orienting the specimens along the [110] $\beta$  zone axis, and their corresponding selected area diffraction patters (SADPs) are shown as insets. The  $\beta$ -solutionized ( $\beta$  -sol<sup>n</sup>) microstructure consisted of a homogeneous distribution of very fine scale  $\omega$  precipitates, while subsequent annealing at 475 °C/48H coarsened the  $\omega$  precipitates (~70–90 nm) into well-developed ellipsoidal particles. The presence of  $\omega$  was further confirmed by the presence of intensity maximas at 1/3 and  $2/3[\overline{2}1\overline{1}]\beta$  locations in the [110] $\beta$  SADP. The position of such distinct  $\omega$  intensities indicate that they have an ideal hexagonal structure (symmetry *P*6/mmm) and an  $\omega/\beta$  orientation relationship [18,27]: {011}<sub>B</sub>//{2110}<sub>w</sub> and  $<\overline{1}1\overline{1}$  $>_{\beta}/(0001)_{\omega}$ . Furthermore, the full collapse of the (111) planes of bcc ( $\beta$ ) to form the ideal  $\omega$  structure has been clearly demonstrated by Zheng et al. in a previous report on the Ti—12Mo alloy [15]. Atom probe tomography of the  $\beta$ -solutionized condition is presented in Fig. 1(c) via binomial distribution plots obtained by measuring every 100 at. blocks within the 3D atom probe reconstruction [19]. This distribution plots shows that the observed Mo ion distribution (blue line) closely follows the distribution expected from a random solid solution (red line). In other words, in the  $\beta$ -solutionized condition, Mo partitioning between the  $\omega$  and  $\beta$  phases was not discernable, within the limitations of binomial distribution analysis routine, implying that the athermal  $\omega$  precipitates had formed congruently within  $\beta$  -matrix (  $\omega$  and  $\beta$  have the same composition). In comparison, the annealed specimens exhibited substantial partitioning between  $\omega/\beta$ , indicated in Fig. 1(d). The coarse  $\omega$  precipitates are depleted in Mo (~2 at.% Mo), while the surrounding  $\beta$ -matrix is Morich with ~16 at.% or 27 wt.%Mo. These compositions were measured by delineating the  $\omega$  precipitates with an 85 at.% Ti isoconcentration surfaces (inset in Fig. 1(d)) [19] and then measuring a 1D composition profile across the  $\omega/\beta$  interface using a cylinder of diameter ~10 nm. Thus,  $\beta$ solutionizing followed by subsequent annealing results in well-developed coarse  $\omega$  precipitates with substantial compositional partitioning between these precipitates and the surrounding  $\beta$ -matrix.

Both, only  $\beta$  solutionized, as well as,  $\beta$  solutionized and annealed samples were tensile tested to failure both at room temperature as well as at 300 °C. Fig. 2(a) shows the tensile test results in the form of engineering stress vs. engineering plastic strain curves for the different conditions. The  $\beta$ -solutionized sample tested at room temperature exhibits a pronounced strain hardening, leading to an ultimate tensile strength (UTS) of ~550 MPa, and a ductility of 35%. In contrast, the



**Fig. 1.** Dark-field TEM (DFTEM) images showing  $\omega$  size-scale and distribution after (a)  $\beta$ -solutionizing and (b) subsequently annealing at 475 °C/48H. Insets show the [011] $\beta$  electron diffraction patterns used for recording DFTEMs. Atom probe tomography (APT) results showing Mo concentrations: (c) by comparing binominal distribution in  $\beta$ -solutionized specimen and (d) plot of 1D composition profile indicating Mo partitioning across  $\omega/\beta$  interfaces (shown with isoconcentration surfaces in inset).

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