Microstructure and tensile properties of heat-treated Ti-Mo alloys

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Abstract. Current research is focused on development of β-type titanium alloys for biomedical applications as substitutes of the undesirable Ti6Al4V alloy. Ti6Al4V alloy has a higher elastic modulus (110 GPa) than that of the human bone (10-30 GPa) and this mismatch in elastic moduli can cause stress shielding effect, which can cause bone resorption and implant failure. Moreover, the dissociation of vanadium and aluminium can cause long term diseases including Alzheimer, neuropathy. β-type titanium alloys are potential substitute materials due to their good biocompatibility and the β phase has a lower elastic modulus. The aim was to study the microstructure and tensile properties of heat-treated Ti-xMo alloys (x= 8 & 10wt%). Phase analysis was conducted using X-ray diffractometer, while the microstructure was observed using an optical microscope. The tensile properties were examined using a tensile test machine. Acicular structures of α" phase precipitated in the β matrix in Ti-8Mo alloy, while Ti-10Mo alloy showed predominant β phase. The theoretically predicted phase constituents were not consistent with the experimental findings. Ti-10Mo alloy possessed superior yield and tensile strengths, larger elongation, and lower elastic moduli than that of Ti6Al4V alloy. Based on the obtained findings, the Ti-10Mo alloy can be a potential candidate for orthopaedic application. acicular structures of α" phase.

1 Introduction

Metallic materials are preferable in orthopaedic applications as they can withstand high load applied on human bones. They are primarily employed in the human body as replacing materials that substitute or repair different damaged tissues such as bone, cartilage or ligaments and tendons, and even by guiding repair if it is essential [1]. They are in high demands due to the rapid increase in the average age of the human population [2]. A potential orthopaedic material possesses excellent biocompatibility, superior corrosion resistance, the exceptional combination of high strength and low modulus, high fatigue and wear resistance, high ductility [3]. Its tensile properties, which include tensile and yield strengths and ductility should be sufficiently high, while the elastic modulus must match that of the human bone to

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mitigate the stress shielding effect that can result in bone resorption and eventually implant failure [4][5][6].

Over the past decades, titanium (Ti) alloys have exhibited a remarkable impact as metallic biomaterials for orthopaedic applications. In particular, Ti6AL4V alloy is the most widely used alloy and it possesses an elastic modulus (110 GPa) that is higher than that of the human bone (30GPa) [7][8][9]. A mismatch in elastic moduli between an implant and a human bone can cause stress shielding effect, which can result in resorption of the bone and failure of the implant [10][5][11]. Moreover, the dissociation of vanadium and aluminium can cause long term health problems including Alzheimer disease, neuropathy, etc [12][3][[14]. These drawbacks have motivated for the development of non-cytotoxic Ti biomaterials with low elastic moduli [15][16]. Thus, metastable β-type Ti alloys, mainly alloyed with non-toxic β stabilizing elements such as molybdenum (Mo), niobium (Nb), tantalum (Ta), etc. are considered potential alternatives [17][18].

The tensile properties of an alloy are strongly dependent on its phase constituents. The formation/ precipitation of these phases can be controlled by alloying elements (e.g., beta stabilizers) and heat treatment procedures, e.g., solution treatment (ST). Moshokoa *et al*. [19] micro-alloyed Ti with Mo to study the effect of Mo on the microstructure and mechanical properties. The observed microstructures comprised secondary precipitates of the martensitic orthorhombic α'' phase and the omega (ω) phase. Although the presence of these phases in the alloys contributed to high values of tensile strengths, all studied alloys the elastic moduli that were significantly higher than that of the human bone. The alloys also failed in a brittle manner. The phase constituents of an alloy can also be controlled by heat treatment, dependent on the content of the β stabilizers [20]. The β phase can be fully retained in metastable β-type alloys with a higher β stabilizers after solution treatment (above β transus temperature) and subsequent quenching. While the secondary phases tend to precipitate in alloys containing low contents of β stabilizers upon quenching [21]. 100% β phase was observed in Ti–12Moalloy [22], Ti-17Mo and Ti-18Mo alloys [23], Ti–40Ta–22Hf–11.7Zr alloy [24] and Ti–12Mo–5Zr [25]. Therefore, in this study, the effect of solution treatment (ST) and subsequent quenching on the microstructure and tensile properties of Ti-Mo alloys was investigated.

2 Methodology

2.1 Phase Prediction and Alloy Fabrication

The Mo equivalence, which quantifies the stability contribution of each alloying element in comparison to that of the major β-Ti stabilizer (Mo), was calculated using the MO formula ((Mo_{eq}= 1.00Mo+ 0.67V+0.44W+0.28 Nb+0.22 Ta+2.90Fe+ 1.60Cr+ 0.77 Cu+1.11 Ni+1.43 Co+1.54 Mn+0 Sn+0 Zr-1.00Al (wt%)) proposed by Bania [26]. As listed in Table 1, the Moeq value of the Ti-8Mo binary alloy is 8wt%, which indicates that the alloy would precipitate the martensitic phase(s). The Mo equivalence of Ti-10Mo is 10wt%, which is the same as the critical limit for the retention of 100% β phase. This suggests that only Ti-10Mo alloy will form a 100% β phase, with no precipitation of martensitic and/ or the ω phase upon rapid cooling.

The average electron concentration (e/a) approach is defined as the average number of valence electrons in each atom of an alloy. The stability of the β phase is guaranteed at e/a ratio of 4.20 or more [27][28]. The e/a ratio ($\frac{e}{a}\, ratio = (v_1\, m_{1+}\, v_2\, m_2\, + v_3\, m_3\, + \cdots + v_n\, m_n\,)/100$) of the Ti-Mo binary alloys are also displayed in Table 1. Ti-8Mo and Ti-

10Mo binary alloys with e/a ratio of 4.16 and 4.20, which suggests that only the Ti-10Mo alloy would precipitate the β phase.

Table 1. Mo equivalence and E/a ratio of the Ti-Mo alloys.

Alloy	***Moeq* (wt.%)**	***e/a* ratio**
Ti-8Mo	8	4.16
Ti-10Mo	10	4.20

100 g ingots of Ti-8Mo and Ti-10Mo were fabricated by casting CP Ti (99.9%) and molybdenum (99.5%) elemental powders. A cold-press machine was employed to produce green compacts, which were melted in a water-cooled copper crucible with a tungsten electrode using a commercial arc melting vacuum pressure casting system. Prior to melting, evacuation and purging of the melting chamber with argon was done. To promote chemical homogeneity, the ingot was turned over and remelted three times. The Ti-8Mo and Ti-10Mo alloy ingots were solution-treated in a muffle furnace at 894°C and 891°C respectively, for 1 hr at a heating rate of 20 °C/min and subsequently quenched in ice water.

2.2 Microstructural and Phase Analysis

Specimens for microstructural and phase analysis were precision cut, ground on a silicon carbide papers up to 2400 mesh, polished 3 µm diamond suspension and colloidal silica (final polishing) and subsequently etched using Kroll's reagent (85 ml of distilled water, 15 ml of Nitric acid and 5 ml of hydrofluoric acid). Phase constituents were identified using X-ray diffractometry (XRD) with Co Kα radiation operated at 45 kV and 40 mA at room temperature. Microstructure was observed using an optical microscope (Leica CTR4000).

2.3 Mechanical Tests

Following the ASTM E8 standard test method, tensile test specimens with gauge dimensions of 3 × 4 × 10 mm shown in Figure 1 were prepared using electrical discharge machining (EDM) and tested at room temperature. The Instron^{TM} 1342 model apparatus was utilized to measure the properties. Strain was measured via an extensometer, which was removed around 1.3% of strain to avoid breakage, whereas the yield stress was derived via the 0.2% offset method.

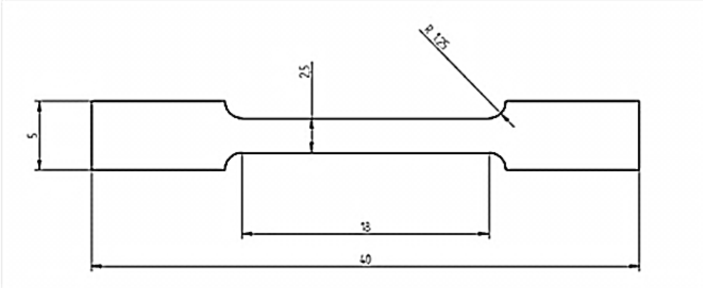

Figure 1: Schematic drawing showing dimensions of the tensile specimen.

3 Results and Discussion

3.1 Microstructural and Phase Analysis

Figure 2 exhibits the XRD patterns of the solution-treated Ti-Mo alloys. The XRD pattern of the Ti-8Mo alloy exhibited primarily BCC β peaks and peaks corresponding to the secondary orthorhombic α" martensitic phase. When the Mo content was increased to 10 wt%, the intensities of the β phase peaks were slightly increased, demonstrating increased β phase stability. A small ω phase peak was also detected at $2\theta \approx 34°$. The existence of the orthorhombic α" martensitic phase in Ti-10Mo is confirmed by the splitting of the XRD peaks and has also been observed in the experimental findings of Oliveira et al. and Almeida et al. [29][30]. These observations demonstrate the dependence of phase transformation on Mo content, as shown by the diffractograms.

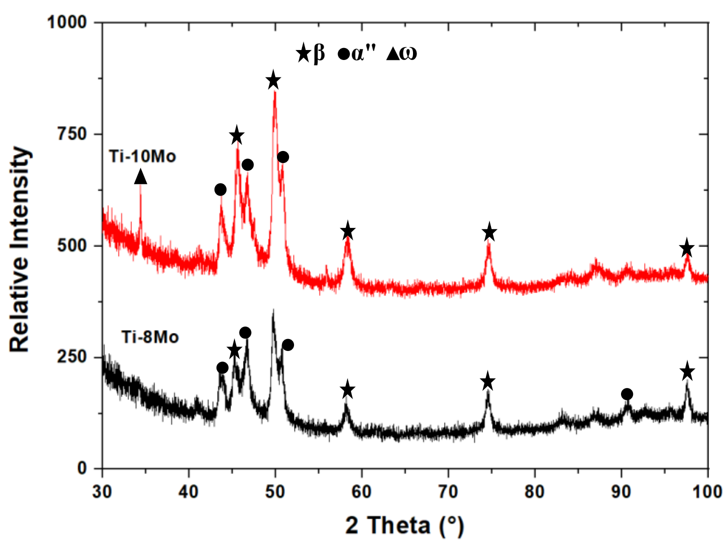

Figure 2: XRD patterns of the Ti-Mo binary alloys.

The optical micrographs of the Ti-8Mo and Ti-10Mo alloys are shown in Figure 3 a) and b), respectively. The micrograph of Ti-8Mo alloy reveals acicular structures corresponding to the orthorhombic α" martensitic phase precipitated in the BCC β phase matrix. Grain boundaries (GB) are clear and visible. When the Mo content was increased to 10 wt%, precipitation of the acicular α" martensitic phase was substantially suppressed, and the β phase was significantly retained as shown in Figure 3 (b). Davis et al. [31] reported that the orthorhombic martensitic α" phase in Ti-Mo binary alloy series was characterized by acicular structures while the hexagonal α' phase was made up of lamellar structures. These observations are not in agreement with Davies et al. [31] findings, in which the solution-treated Ti-10Mo alloy consisted of untransformed equiaxed β phase grains, with no traces of the martensitic phase(s). They indicated that the martensite start transformation temperature was reduced to below room temperature, thus hindering the transformation of the high-temperature β phase to the martensitic α" phase. The ω phase detected by the XRD could not be observed in the micrographs, probably due to its existence in the alloys as nano-particles or small volume fraction, which could have been below the detection limit of the two analytical techniques.

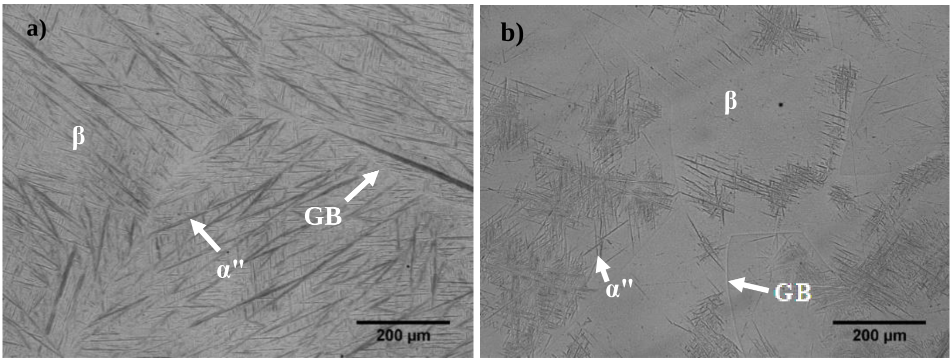

Figure 3: Optical micrographs of a) Ti-8Mo and b) Ti-10Mo alloys.

It was proposed that a binary alloy requires a Moeq of 10.0 wt% or more to form a single BCC β phase upon rapid cooling [32]. This suggests that the Moeq of 10.0 wt% would be sufficient to suppress the formation of the martensitic phases (α' and α"). According to the Moeq values calculated in this study, Ti-8Mo binary alloy was expected to precipitate the martensitic α" phase in the β phase matrix, whereas Ti-10Mo alloy would retain 100% β phase upon rapid cooling. However, the experimental results revealed martensitic α" phase and ω phase precipitates in the Ti-10Mo alloy. This indicates that the Moeq approach overestimated the stabilization effect of molybdenum in the alloy. This discrepancy could be ascribed to chemical inhomogeneity in the alloy, which could have led to Mo-lean and Mo-rich areas in the specimens, thus resulting in the precipitation of the secondary phases. Furthermore, this prediction approach does not take into consideration the precipitation of the ω phase, but the stability of the β phase and the precipitation of martensitic phases in the alloy.

3.2 Mechanical Properties

The tensile properties of an alloy are determined by the phases (crystal structure) it is composed of. The formation/ precipitation of these phases can be controlled by alloying elements (e.g., b stabilizers) and heat treatment procedures (e.g. solution treatment) [19][20][21][24]. High tensile and yield strengths are a prerequisite in the design of orthopaedic materials because they should sustain load-bearing function of human bones and reconstruction. Table 2 lists the tensile properties of the alloys. Both the ultimate tensile and yield strengths of Ti-10Mo alloy were significantly higher than those of Ti-8Mo alloy. This could be attributed to the presence of the ω phase in Ti-10Mo alloy, which possesses the highest strength amongst the phases in the metastable β-type Ti alloy [33]. The elastic modulus of a biomaterial must match that of the human bone (10 – 30 GPa) to mitigate the stress-shielding effect during implantation [34]. When the Mo content was increased from 8wt% to 10wt%Mo, a significant increase in the elastic modulus was observed. This could be attributed to the presence of a higher proportion of the ω phase in Ti-10wt%Mo than in Ti-8Mo alloy. These observations show a strong dependence of the elastic modulus values on the phase constituents. Although the obtained elastic moduli of the Ti-Mo alloys are higher than that of the human bone, they can be considered for use in biomedical applications, because they have the potential to prevent the stress shielding effect compared to the conventional Ti6Al4V alloy. Large elongation is desirable in orthopaedic applications to prevent breakage in case an alloy is accidentally stressed beyond its proportional limit [35]. When the Mo content was increased from 8wt% to 10wt%Mo, the elongation was significantly increased. This suggest that both alloys failed in a ductile fracture manner. Therefore, both alloys in this study would have good fracture resistance due to their ductile properties.

In orthopaedic applications, it is a requirement for ideal biomedical implant materials to have greater elastic admissible strain. The elastic admissible strain is defined as the ratio of the yield strength over the elastic modulus of a material, and it is a useful parameter in orthopaedic applications. The greater the elastic admissible strain, the more desirable the material is for such applications [36][37][38][39]. Thus, a good biomaterial should have a higher permissible strain than that of human cortical bone, which is reported as (0.67). The admissible elastic strain of Ti-10M0 alloy was higher than that of Ti-8Mo alloy. Ti-10Mo alloys exhibited a higher admissible strain than the human bone.

Table 2: Tensile properties of Ti-Mo alloys in this study and the alloys considered for comparison.

Alloy	Ultimate tensile strength (MPa)	Yield Strength (MPa)	Elastic Modulus (GPA)	Admissible elastic strain (%)	Elongation (%)
Femur bone (longitudinal)[40]	135	71.6	17.9	0.4	-
Ti-8Mo (this study)	659.23 ± 11.0	450.48 ± 13.83	78 ± 8.4	0.58	12.48
Ti-10Mo (this study)	704.77 ± 28.5	612.30 ± 25.4	95 ± 1.7	0.74	20.71± 13.

Both the ultimate tensile and yield strengths of Ti-10Mo alloy were significantly higher than those of Ti-8Mo alloy. This could be attributed to the presence of the ω phase in Ti-10Mo alloy, which possesses the highest strength amongst the phases in the metastable β-type Ti alloy [33].

4 Conclusion

The microstructure and tensile properties of solution-treated Ti-8Mo and Ti-10Mo alloys were investigated using the tensile test machine. Based on the observed findings, the following conclusions were drawn:

1. Both alloys had equiaxed β grains with acicular precipitates of the secondary orthorhombic α" phase and the athermal ω phase.
2. The theoretically predicted phase constituents were not consistent with the experimental findings.
3. Ti-10Mo alloy possessed superior yield and tensile strengths, larger elongation, and lower elastic moduli than that of Ti6Al4V alloy.
4. Based on the obtained findings and reported admissible elastic strain, the Ti-10Mo alloy is a potential candidate for orthopaedic application.

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