Investigation of microstructural and micro-hardness properties of Ti-15.05Mo alloy for biomedical applications

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Abstract: This study presents the analysis of the microstructure and Vickers micro-hardness of β-Ti type Ti-15.05Mo alloy obtained by different cooling rates. The alloy was fabricated using an arc-melting furnace, solution treated in a muffle furnace and cooled using different cooling medium such as ice water, furnace cool and air cooling. The X-ray analysis of the as-cast showed β phases peaks only and water quenched samples showed peaks belonging to α′′ and β phases. The (β, α) dual phases were detected in the furnace and air-cooled samples, respectively. The Optical Microscope (OM) micrographs of the as-cast Ti-15.05Mo sample showed large equiaxed β grains and sub-structures belonging to orthorhombic martensitic α" phase. The water quenched sample showed β equiaxed grains, however their grains were much smaller You should leave 8 mm of space above the abstract and 10 mm after the abstract. The heading Abstract should be typed in bold 9-point Arial. The body of the abstract should be typed in normal 9-point Times in a single paragraph, immediately following the heading. The text should be set to 1 line spacing. The abstract should be centred across the page, indented 17 mm from the left and right page margins and justified. It should not normally exceed 200 words.

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1 Introduction

Titanium (Ti) and its alloys are gradually being utilised in biomedical engineering applications because of their properties such as high strength, low modulus, and better corrosion resistance [1], [2]. Ti alloys are divided into 3 categories based on the phases present in their microstructures such as α, β, β + α Ti alloys. Alpha (α)-Ti alloys are known for their high strength; however, they exhibit very low ductility and anisotropic properties due to the single phase hexagonal-close-packed (hcp) crystal structure [3]. β+α Ti alloys have combined properties such as high strength, suitable ductility, excellent corrosion resistance, and low Young's modulus compared to Cr-Co alloys and stainless steel, attributed to dual phase comprised of hcp and body-centred-cubic (bcc) crystal structures [1]. The β -Ti alloys consist of a single-phase bcc structure yielding a good combination of high ductility and low Young's modulus [4]. Most of the Ti alloys are highly alloyed to stabilize the prerequisite phase such as α, β, β + α, hence existence of a wide range of compositions of Ti alloys. According to Weiss et al the type, size, shape, and distribution of various phases in the microstructure of Ti alloys are normally influenced by the thermomechanical processing history and have a direct impact on their mechanical properties [5]. For instance, in a β + α Ti alloys, this is achieved by heating the alloy above the β phase field or below the β transus and cooling it at a desired rate [4,10,14]. The α phase precipitates which results from heating and cooling of the samples under nearly equilibrium conditions, classified as primary α, widmanstatten α, globular α, lamellar α and secondary α, depending on the shape and size [6]. Faster cooling rate by either oil or water quenching promotes the diffusionless transformation from the β to α′ or α′′ phase, which is harder than α phase, with some level of brittleness in the overall structure [7], [8]. The α′ phase retains the hcp crystal structure with lattice parameters comparable to those of α phase, while α′′ is orthorhombic in nature [1], [9]. According to several authors [1], [3], [10] the evolution of the β phase primarily depends on the alloying elements and the processing temperature. For instance, if strong β stabilizing elements are added and the alloy is heated above the β transus followed by quenching, the β phase can be completely retained.

In literature, several studies have been and are still being published on the relationship between cooling rates, microstructure, and properties of Ti alloys. For examples, Shao et al reviewed the relationship between cooling rate and microstructure in the TC21 titanium alloy and discovered that diffusionless transformation β → α′′ takes place after water quenching and β → β + α phase on air cooling (AC) and furnace cooling (FC) [11]. The cooling rate is also vital in deciding the shape and size of the α phase in the microstructure [12]. Another publication by Gil et al reported the effect of cooling rate on the microstructure and mechanical properties of Ti6Al4V alloy during heat treatment in the β phase field. They reported that the size of the α phase increased with decrease in the cooling rates [13]. In the Ti-6Cr-5Mo-5V-4Al alloy, it was illustrated that the tensile strength and ductility were higher when the alloy was heated in the β /α phase field followed by aging as compared to heating in the β phase field [14]. Jianwei et al showed the influence of cooling rate on the microstructure after heat treatment in the β phase field. They observered lamellar structure for slow cooling rates (15 °C/s) and combined lamellar-martensitic structure for 15-90°C/s, whereas above 90°C/s, the microstructure was wholly martensitic [8]. Slow cooling rate from the β phase field also supported the formation of the α phase. The above literature motivated the investigation of heat treatment and cooling rates on binary β-Ti alloys that are most promising for biomedical applications due to low Young's modulus [15]. Since heat treatment forms a key component of the manufacturing process of these alloys, this current study systematically investigates the effect of cooling rate on the microstructural and phase evolution as well as the Vickers micro-hardness of binary Ti-15.05Mo alloy.

2. Materials and Methods

2.1 Materials preparations

The commercially pure Ti (Cp-Ti) and Molybdenum (Mo) powders with a composition of 85g and 15g, respectively, were compacted into a 100g green bodies. The four compacted Ti-15.05Mo green bodies were prepared using a button arc furnace (BAF), in which melting was performed under argon atmosphere in à water-cooled copper hearth. The 4 ingots were re-melted three times in-order to ensure chemical homogeneity. From the 4 samples, 1 sample was characterized in the as-cast condition whereas the other three cast ingots were subjected to solution treatment using a muffle furnace at 1200 ºC for 1hr and followed by different cooling rates as follows : (i) quenched in ice water referred as : (WQ), (ii) furnace cooled (FC) and (iii) air cooled (AC).

2.2 Microstructural and phase evolution

Phase and microstructural analysis for the 4 samples were investigated using an X-ray diffractometer (XRD) and the Optical microscope (OM) respectively. XRD analysis were carried out over a 2θ range from 10º to 100º at a scanning rate of 0.5º/s with a step size of 0.02º using a Cu Kα radiation source. The OM samples were prepared by following the metallographic preparation methods and the samples were etched with Kroll etchant for 30 seconds.

2.3 Hardness testing

The Vickers micro-hardness tests were performed using the Zwick Roell Vickers hardness tester. A small indentation was made with a load of 500gf for 10s, two diagonals were made after 10 indentation per sample, they were measured microscopically, and their averages were recorded.

3. Experimental Results

3.1 Phase and microstructural characterization

3.1.1 X-ray diffraction

X-ray diffraction (XRD) was carried out to identify the constituent phases of Ti-15.05Mo after various cooling rates. Figure 1 shows the XRD patterns of Ti-15.05Mo alloy cooled at different medium. The as-cast sample illustrated β peaks only without the presence of the orthorhombic α″ martensitic phase. The water quenched (WQ) samples exhibited peaks corresponding to β and orthorhombic α″ martensitic phases. The presence of the α″ phase peaks in these alloys indicate that the composition and the quenching medium were not sufficient enough to completely stabilize the β phase. From the current XRD analysis, the presence of other phases such as the omega phase was not detected.

The XRD patterns of the air-cooled (AC) sample revealed the presence of α and β phases. Based on the peak intensities, it follows that the samples subjected to WQ and AC heat-treatment conditions had high crystallinity, most common in materials with larger grain sizes. However, the cooling rate of this sample was exposed proved to be inadequate for full stabilization of the β phase. Different from other heat-treatment processes used, the analysis of the furnace cooled (FC) sample showed presence of α and β phases. In comparison, this sample contained high volume fraction of the α phase. This may be attributed to the diffusion process, in which the bcc Mo had adequate time to diffuse from the β solid solution to form α phase with less Mo content as well as Mo-rich β phase. Due to lack of published literature work on the Ti-15Mo binary after different cooling rates, it was challenging for the authors to compare their findings with those found in literature.

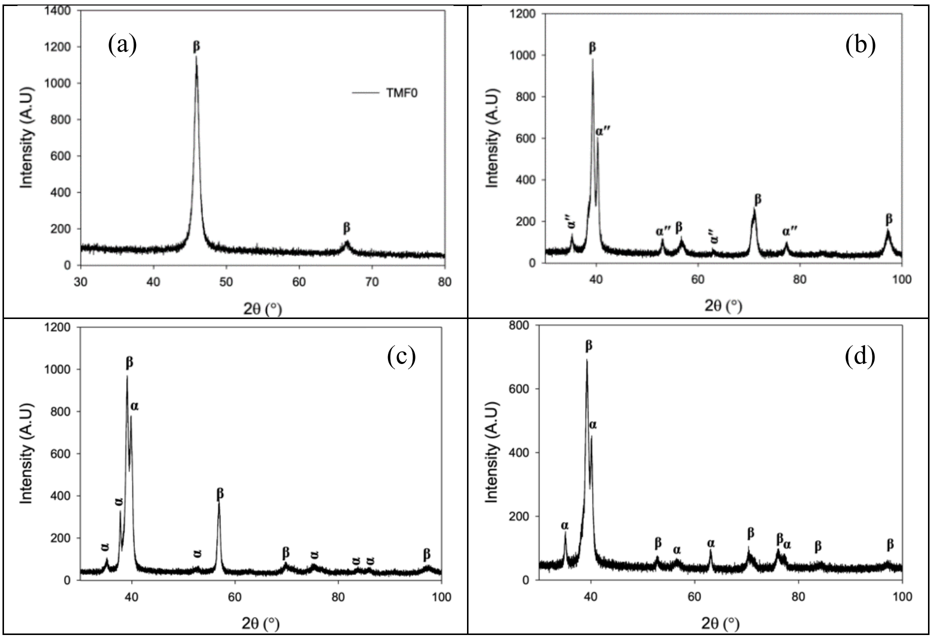

Fig. 1 : X-ray diffraction of Ti-15.05Mo at different cooling medium : (a) As-Cast, (b) Water Quenched (WQ), (c) Air cooled (AC) and (d) Furnace cooled (FC).

3.1.2 Optical Microscope

The microstructural evolution of Ti-15.05Mo alloy subjected to different cooling rates traced using the optical microscope (OM) is depicted in Figure 2. The OM micrograph of as-cast sample in figure 2(a) was comprised of large equiaxed grains of β structure and acicular sub-grains structures precipitated inside the grains. The morphology of the sub-grain structure was established to be corresponding to the orthorhombic martensitic structure (α″). Similar results were reported by Davis et al in 6wt%. Mo alloys. The water quenched (WQ) sample in figure 2(b) consisted of equiaxed grains of β phase only. It was observered that the grains after WQ were significantly smaller in comparison to those in as-cast sample. These results indicate that rapid cooling from very high temperature is only effective to induce the martensitic phases as opposed to yielding a completely martensite-free β single-phase. Furthermore, based on the peak intensity, the water-cooled Cu hearth subjects the as-cast sample to more rapid cooling than in the WQ process.

Decreasing the cooling rate from WQ to FC, the micrographs illustrated larger equiaxed grains of β, within the grains and along the grain boundaries were precipitates of clustered needle like structure that could be associated with the α phase. However, this possibility requires further microstructural investigation using the scanning electron microscopy (SEM) coupled with EDS. The micrographs of the FC cooled sample are in agreement with the XRD peaks presented in figure 1. Upon air cooling (AC), the micrographs were comprised of larger equiaxed grains of β and sub-structures inside the grains, however the substructures were observed not to be acicular but fine. The fine sub-structure could be those of α phase as observered in the XRD patterns. Large equiaxed grains of α phase and fine clustered needle like structure precipitated inside the grains and along the grain boundaries. These fine needle structures could be associated with Mo-rich β phase. However, this possibility requires further microstructural investigation using scanning electron microscopy (SEM).

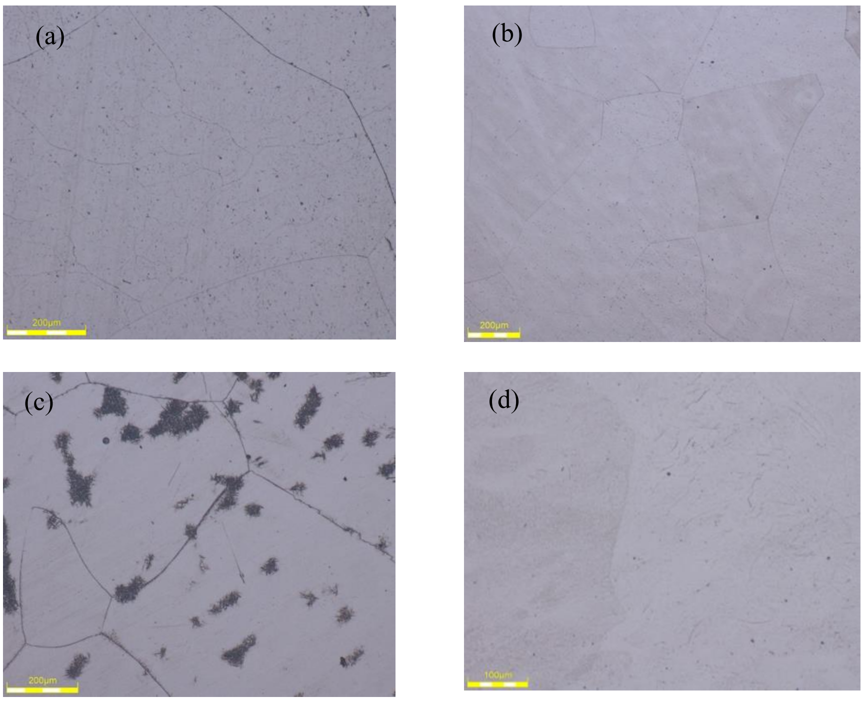

Fig. 2 : Optical Micrographs of Ti-15Mo alloy in different cooling types (a) As-cast, (b) water quenched (WQ), (c) Furnace cooled (FC), and (d) Air Cooled (AC)**.**

3.2.2 Vickers Micro-Hardness

The Vickers Micro-hardness results of Ti-15.05Mo alloy heat-treated at various cooling rates are presented in figure 3. The as-cast sample showed the 2nd highest hardness value of 440.17 $HV_{0.5}$, while the WQ sample resulted in the highest hardness of 444,69 $HV_{0.5}$ which is slightly higher compared to AC sample. The FC samples yielded the lowest hardness of all samples, at a value of 329,95 $HV_{0.5.}$ It was reported that the phase constituent affects the Vickers micro-hardness value of beta Ti alloys [Lee, (2002)], which in theory decreases in the following trend : $H_\omega > H_{\alpha''} > H_{\alpha'} > H_\alpha > H_\beta$ [17]. As a result, the hardness of the alloy containing a high-volume fraction of omega (ω) phase will have the highest hardness due to its brittle character. In addition to the presence of α″ phase, the high micro-hardness in the WQ sample compared to the as-cast and AC samples could be attributed to the smaller grains as observed in the OM micrographs. The high hardness in the as-cast sample as compared to AC sample may be due to the presence of both α′ and α″ which are reported to have higher hardness compared to β and α phases. The low hardness in the FC sample was because of the presence of only β and α phases.

The micro-hardness value of WQ Ti-15.05Mo alloy (444,69 $HV_{0.5}$) was higher than that reported in Ref. [18] in Ti-15Mo (307 $HV_{0.2}$) alloy, and also higher than the hardness of Cp-

Ti (156 $HV_{0.2}$) reported in Ref. [19]. Similar findings of lower hardness in Ti-15Mo alloy and Cp-Ti (330 $HV_{0.5}$ and 210 $HV_{0.5}$, respectively) compared to the studied alloy were also reported by authors in Ref. [20] . Furthermore, a low micro-hardness value of Ti6Al4V was 337 $HV_{0.5}$ compared to WQ and AC Ti-15.05Mo alloy reported by Song et al [21].

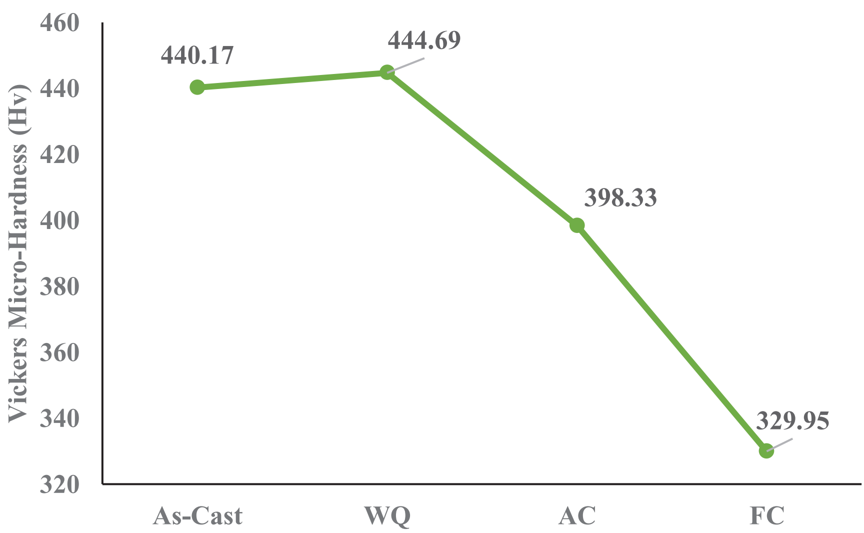

Fig. 3 : Micro-Vickers hardness of Ti-15.05Mo alloy after various cooling rates.

4. Conclusion

Based on the effect of cooling rate on the microstructure and micro-Vickers hardness of binary Ti-15.05Mo alloy the following conclusions can be drawn :

- The samples in the as-cast condition and after ice-water quenching (WQ) resulted in 3 phase material comprised of β, α′ and α″ phases as detected by XRD analysis. On the other hand, the samples subjected to AC and FC resulted in dual phase (β, α′) and (β, α), respectively.
- The OM micrographs of the as-cast Ti-15.05Mo sample showed large equiaxed β grains and sub-structures belonging to orthorhombic martensitic α" phase. The WQ sample showed β equiaxed grains, however their grains were much smaller as compared to the ones in as-cast. The FC sample showed large equiaxed grains of α phase and fine clustered needle like structure precipitated inside the grains and along the grain boundaries, whereas the AC sample showed bigger grains with fine sub structures inside the grains.
- Due to presence of orthorhombic α" phase and smaller grains, the micro-hardness test of the fast cooled (WQ) sample showed the highest hardness value compared to all the other samples. Similarly, the as-cast sample showed the second highest hardness. The FC and AC showed low hardness values compared to both as-cast and WQ samples, but higher HV compared to Ti6Al4V alloy and Cp-Ti.

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