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Microstructure and selected mechanical properties of aged Ti-15Zr-based alloys for biomedical applications

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ABSTRACT

In this study, Ti-15Zr-xMo (5, 10, 15, and 20 wt%) alloys were submitted to solution and aging treatments and their effects evaluated in terms of phase composition and selected mechanical properties (Vickers microhardness and Young's modulus) for use as biomedical implants. The solution treatment was performed at 1123 K for 2 h, while aging treatments were carried out at 698 K for 4, 8, and 12 h, followed by water quenching. Phase composition and microstructure were dependent of the heat treatments, with Ti-15Zr-5Mo ($\alpha + \beta$ type) and Ti-15Zr-10Mo (metastable β type) alloys exhibiting intense α phase precipitation. The α-phase precipitates were related to $\alpha'' \rightarrow \alpha$ and $\beta \rightarrow \alpha$ phase decompositions. The Ti-15Zr-10Mo alloy exhibited an intermediary isothermal ω-phase precipitation after aging for 4 h. Vickers microhardness and Young's modulus values changed gradually with the amount of α phase. Aged Ti-15Zr-15Mo and Ti-15Zr-20Mo alloys presented better combinations of hardness and Young's modulus than CP-Ti and Ti-64 ELI for biomedical applications.

1. Introduction

Titanium (Ti) and its alloys have been extensively employed as biomaterials specially in dental and orthopedical implants due to their well-suited properties, such as appropriate corrosion resistance, relatively low Young's modulus, high strength-to-density ratio, and recognized biocompatibility [1, 2]. In despite of the fact that Young's modulus values of novel Ti-based alloys (60–100 GPa) are closer to hard tissues (10–30 GPa), which makes it possible to avoid bone atrophy and the stress-shielding effect [3, 4], their mechanical strength should be adequate to support biomechanical loads along the life span of the implant [5, 6]. By alloying elements and heat treatments, novel biomedical Ti-based alloys are being developed that combine a low Young's modulus and high mechanical strength [5, 7].

Alloying elements and heat treatments plays an important role in the microstructure-properties relationship in Ti-based alloys, since phase precipitation mechanisms are intrinsically dependent on the composition and processing steps [8]. Zirconium (Zr) is considered a neutral element, although recent research has indicated that it has a β-stabilizer action when in a solid solution with another β-stabilizer element [9–11]. Molybdenum (Mo) is a strong β-stabilizer that could fully retain β phase at 10 wt% after water quenching. Moreover, the addition of Mo can decrease Young's modulus and improve the corrosion resistance of the material [12, 13]. In previous studies, we have found low Young's modulus (60–80 GPa), high tribocorrosion resistance, and good cell viability in Ti-15Zr-Mo alloys [9, 14–16]. In particular, the Ti-15Zr-15Mo alloy displayed the best potential for biomedical application.

Aging is an industrially applied heat treatment in Ti-based alloys which improves their mechanical strength through secondary phase precipitation [2, 17, 18]. β-type Ti-based alloys exhibited α-phase precipitation along the β-phase matrix when submitted to aging treatments, which results in high mechanical and fatigue strength [19, 20]. Tane et al. [20] obtained an enhancement in the elastic components (bulk and shear modulus) of metastable β-type Ti-Nb-based alloys after room temperature aging, which resulted in interesting properties for biomedical applications. Li et al. [21] studied the effect of aging treatment in Ti-Nb-Zr-based alloys, obtaining improvements in the superelastic and mechanical properties of the alloy for use as metallic implants. Similarly, Liang et al. [22] obtained a low Young's modulus

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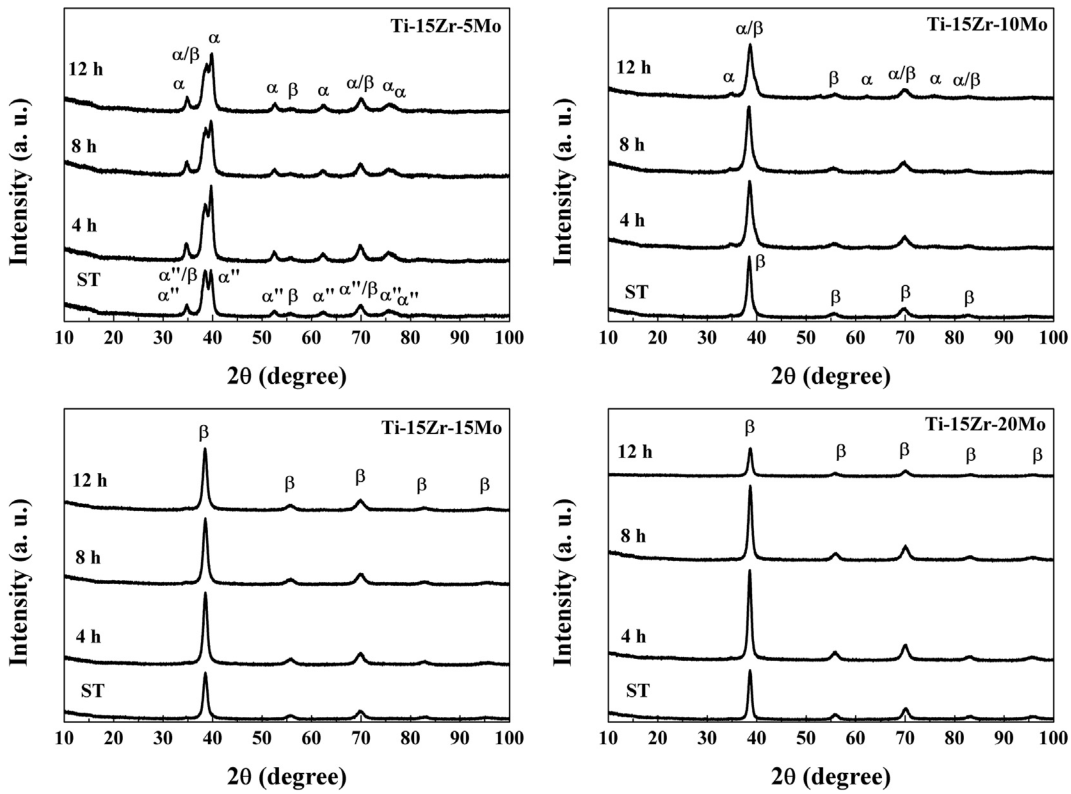

Fig. 1. XRD patterns for Ti-15Zr-Mo alloys.

Table 1
Rietveld parameters for Ti-15Zr-Mo alloy.

Alloy	Condition	Gof[a]	R_{exp} (%)[a]	R_{wp} (%)[a]	R_p (%)[a]
Ti-15Zr-5Mo	ST	1.743	9.78	8.42	6.51
	4 h	1.928	8.02	7.75	6.00
	8 h	1.754	8.80	7.29	5.64
	12 h	1.875	9.28	7.57	5.94
Ti-15Zr-10Mo	ST	1.668	9.08	8.33	6.49
	4 h	1.603	7.15	6.74	5.22
	8 h	1.561	9.93	6.63	5.15
	12 h	1.587	7.91	6.88	5.43
Ti-15Zr-15Mo	ST	1.869	7.07	8.83	6.80
	4 h	1.795	6.16	6.76	5.33
	8 h	1.677	5.77	6.70	5.27
	12 h	1.745	8.39	6.96	5.47
Ti-15Zr-20Mo	ST	1.356	7.05	7.91	6.13
	4 h	1.902	8.46	7.01	5.50
	8 h	1.756	8.51	6.83	5.42
	12 h	1.561	8.27	10.22	8.07

[a] Godness of fitness (GoF or χ^2); Expected residual factor (R_{exp}); Weighted-profile residual factor (R_{wp}); Parametric or Bragg residual factor (R_p).

and large plasticity in Ti-Nb-Zr-Mo alloy by combining theoretic compositional approach and aging treatment.

This study aims to analyze the effect of aging time in the structure, microstructure, and selected mechanical properties of Ti-15Zr-xMo alloys (at 5, 10, 15, and 20 wt%) for use as biomaterial. Aging treatments were performed to induce α-phase precipitation, increase mechanical strength, and maintain a low Young's modulus for use as biomedical implants.

2. Materials and methods

Ti-15Zr-xMo alloys (at 5, 10, 15 and 20 wt%) were produced by argon arc melting from commercially pure metals: CP-Ti (grade 2), pure Zr (99.8%), and pure Mo (99.9%). The ingots were molded in plate-type samples by air-cooled hot rolling at 1273 K, followed by annealing treatment at 1273 K in a vacuum of 10^{-5} Torr for 24 h with slow cooling. Afterwards, the samples were submitted to solution treatment (ST) at 1123 K in a vacuum of 10^{-6} Torr for 2 h with water quenching. Aging treatments were subsequently carried out at 698 K, with a heating rate of 10 K/min from room temperature, in a vacuum of 10^{-6} Torr for 4 h, 8 h, and 12 h with water quenching. All heat treatments were conducted in a quartz tube. The chemical composition of the samples can be found in previous reports, where it is shown the alloying elements remained within a deviation range of 1 wt% from the nominal composition, and the O and N interstitial gases presented values around 0.20–0.30 wt% and 0.02–0.04 wt%., respectively [12].

The phase composition was evaluated by X-ray diffraction measurements (XRD) (Rigaku D/Max 2100/PC diffractometer). XRD patterns were acquired by the powder method at 40 kV and 20 mA, between 10° and 100°, with a step size of 0.02° and a time per step of 3.2 s, using monochromatic CuK_α radiation (λ = 0.1544 nm). The results were analyzed by the Rietveld method throughout standard crystallographic sheets and the GSAS/EXPGUI program. A standard Y_2O_3 sample was used to obtain the instrumental parameters of the equipment [23].

Microstructural analysis was performed by optical microscopy (OM) (Olympus BX51M microscope), scanning electron microscopy (SEM) (EVO LS15 Carl Zeiss microscope) and transmission electron microscopy (TEM) (Titan FEI microscope). Chemical micro-analysis was performed by energy-dispersive X-ray spectroscopy (EDS) (Oxford detector) coupled in the SEM equipment. The samples were submitted to a metallographic process by grinding with silicon carbide (SiC) waterproof sandpapers; polishing with a colloidal solution of alumina and diamond; and etching with H_2O, HNO_3, and HF solution (80:15:5). For TEM analysis, the samples were previously prepared in thin foils by a dual beam Quanta 3D SEM instrument with Ga^+ source and organometallic gas of $PtCO_6$. The samples were analyzed by TEM in a C_s corrected FEI Titan 80–300 (300 kV operation voltage) equipped with a X-FEG filament and monochromator. Selected area electron diffraction