

MICROSTRUCTURAL CHARACTERIZATION OF THE TiMoZrTa ALLOY

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ABSTRACT

In recent years, different types of titanium alloys have been investigated with the aim of using materials in biomaterials field, and Ti-Mo system alloys are very promising materials. Alloying titanium with biocompatible elements like Mo, Zr and Ta make then possible for these alloys to be used in medical applications. Microstructures of two TiMoZrTa alloys were investigated. Obtaining of this original recipes alloys was prepared using vacuum arc re-melting method afterwards composition of this alloys were verified by quantitative qualitative analysis EDX. Aim of this study is investigating aspects of microstructures TiMoZrTa alloys using optical and scanning electron microscopes, verifying the type of grains, that will show us the most important properties.

KEYWORDS: Ti-based alloy, biomaterials, microstructure, XRD analysis

1. Introduction

Metallic biomaterials, like stainless steel or Co-based alloys, titanium and its alloys continue to be used extensively in different medical applications, therefore these are researched and improved [1, 2].

Titanium and its alloys are still widely used for implant materials, because they present an acceptable biocompatibility, relatively low elastic modulus, have an excellent corrosion resistance due superficial oxide layer and the density / resistance rapport is very good [3, 4].

Representative titanium alloys used for implant materials are Ti-6Al-4V and commercial pure titanium. These alloys of the type of $\alpha+\beta$, in time, has been shown that V and Al are toxic for the human body. Therefore, researchers replaced V and Al with non-allergic elements like Mo, Nb, Ta, Sn and Zr. In recent years, the β -type titanium alloys composed of nontoxic elements was extensively investigated because processing variables can be controlled to produce selected results (e.g. Ti-15Mo, Ti-35.5Nb-5.1Ta-7.1Zr, Ti-24Nb-4Zr-8Sn and Ti-12Mo-6Zr-2Fe alloys) [5-8]. The β type alloys compared to α and ($\alpha+\beta$) type Ti have advantages like lower elastic

modulus, improved fatigue resistance and excellent resistance to wear and abrasion [9, 10].

Titanium has an allotropic transformation from a hexagonal close-packed (hcp) which is referred to "alpha" phase into a body-centered cubic (bcc), called "beta" phase at 882 °C.

Depending on phase microstructure of the alloyed, titanium alloys can be classified into three main structural types: alpha alloys, alpha + beta alloys and beta alloys, but these metallographic transformations can be improved by additions of selected α or β alloying stabilizers [11-13].

The aim of this study is to characterize microstructures of a new alloy with medical applications.

2. Experimental procedure

The alloy was obtained from the melting of pure elements like Ti, Mo, Zr, Ta in an arc melting furnace, MRF vacuum ABJ 900.

Due to the big difference in the melting point of elements (Ti: 1.668 °C, Mo: 2.623 °C, Zr: 1.855 °C, Ta: 3.020 °C) and density (Ti: 4.51 g/cm³, Mo: 10.2 g/cm³, Zr: 6.5 g/cm³, Ta: 16.65 g/cm³) between the

four pure metals, the alloy was followed by re-melting of the alloy for 6 times in the same installation for refining and homogenizing its structure [11, 14].

The alloy used in this study, after melting, was characterized by chemical composition, X-ray diffraction, optical microscopy. Chemical composition of the alloy was analyzed with the EM VEGA II LSH scanning electron microscope manufactured by the TESCAN Co., the Czech Republic, coupled with an EDX QUANTAX QX2 detector manufactured by the BRUKER/ROENTEC Co., Germany.

The phases were analyzed using the X-ray diffraction, made on the diffractometer X'Pert PRO MPD PANalytical X-ray with the following parameters: continuous scan, 2θ - (10° - 90°), Step size: 0.0131303, Time per step: 61.20, Scan speed: 0,05471,45 KV and 40 mA using a copper anode X-ray tube.

After the X-ray diffraction, the alloys were prepared by a standard metallographic process, by polishing alloys in SiC waterproof papers until #2000 grit and colloidal alumina suspension.

The metallographic structure of alloys was shown by the attack surface with a chemical solution having the following composition: 10 ml HF, 5 ml HNO₃, 85 ml H₂O immersed in 5-30s [12].

The device used in the microstructure analysis for Ti alloy was the metallographic microscope Leica 5000DMI.

3. Results and discussions

3.1. Chemical composition

The chemical analyses (EDX or XRF) were performed in many different areas. The results and spectrum are shown in Fig. 1.

The chemical composition of the alloy (Table 1) was homogeneous and it showed no differences between surface and core.

Figure 1 shows the spectrum and distribution map of the chemical elements of the studied alloy.

The distribution map shows that studied alloy is homogeneous all over the surface.

Table 1. Chemical composition of alloy studied

Element (wt.%)	
Ti	67.65
Mo	12.84
Zr	8.8
Ta	10.71

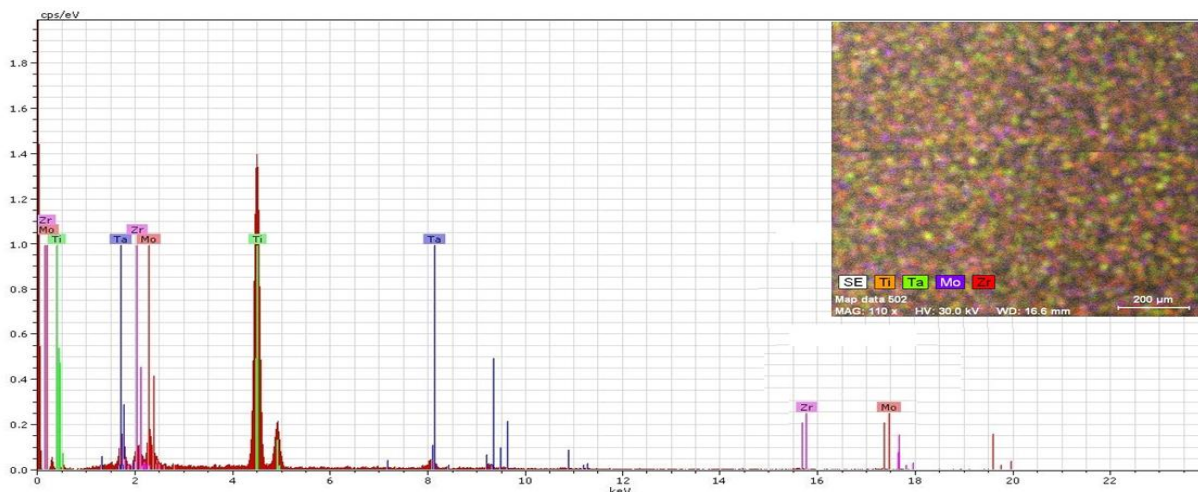


Fig. 1. Spectrum and distribution map of the main chemical elements of the alloy studied

3.2. X-ray diffraction

Figure 2 shows the X-ray diffraction patterns for Ti-based alloy. The patterns of alloy could be indexed in the cubic β type structure, the space group of Im-3m [10, 14].

According to Table 2, the increased amount of β stabilizing elements (Mo, Ta) alloys present in the composition was not enough to form a single phase β .

In the structure of the alloy there have been identified two main phases: β with a body-centered cubic structure and α'' with orthorhombic structure.

The adding of pure tantalum leads to the appearance emergence phase α'' as β phase is confirmed and to Y. L. Zhou [8]. According X.H. Min, by adding zirconium in conjunction with a higher percentage of 10% of Mo leads predominantly to the β phase appearance [15].

In the alloy sample β phase was found with the major peak at the angle $2\theta = 38.8485$ and α'' with the major peak at the angle $2\theta = 37.0213$.

Table 2 gives the network unit cell parameters of the alloy.

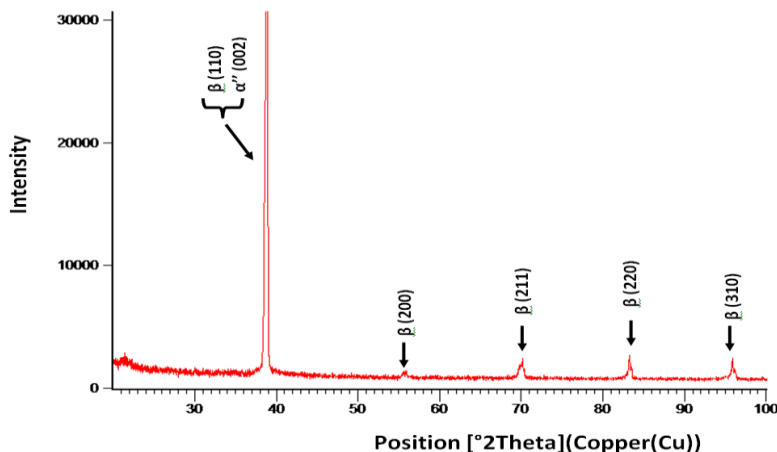


Fig. 2. X-ray diffractograms for Ti-based alloy

Table 2. Compound parameters

Elements	Ti, Mo, Zr, Ta
Space Group	Im-3m
Crystal system	Cubic
a(Å)	3.31
b(Å)	3.31
c(Å)	3.31
α (°)	90
β (°)	90
γ (°)	90
Cell volume (10 ⁶ pm ³)	36.30

Its very important to know the microstructure of a metallic biomaterial, like Ti and its alloys, because of a coexistence of hexagonal α Ti and bcc β Ti.

Knowing these important aspects can control the mechanical properties and corrosion resistance, having a direct effect on their biocompatibility.

3.3. Microstructures

The microstructure of the studied alloy, as shown in the Fig. 3-4, was consistent with the XRD results.

The optical microstructure of the Ti-based studied alloy present acicular (dendritic) structure with irregular grain boundaries (Fig. 3). The microstructure of the TiMoZrTa alloy is a specific beta [16, 17].

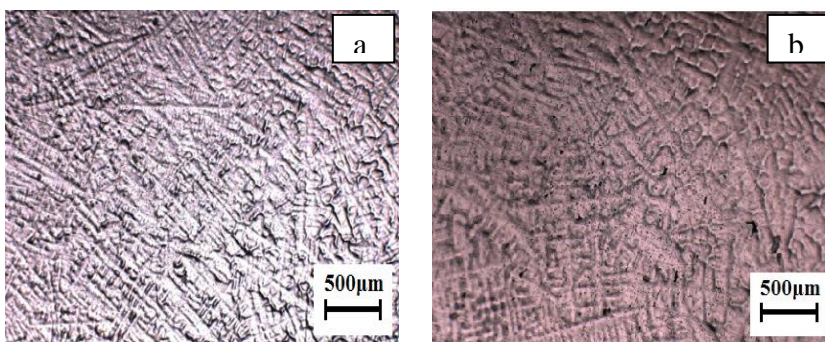


Fig. 3. Optical microstructure of Ti-based alloy: a) 50 X, b) 100 X

For the microstructure characterization of the studied alloy were acquired images secondary electron (SE) and backscatter (BSE) using detectors BSE (Backscattered Detector). Because metal

samples were analyzed, the High Vacuum mode using a voltage of 20 kV was used.

The SEM images of the studied alloy confirm the acicular (dendritic) structure.

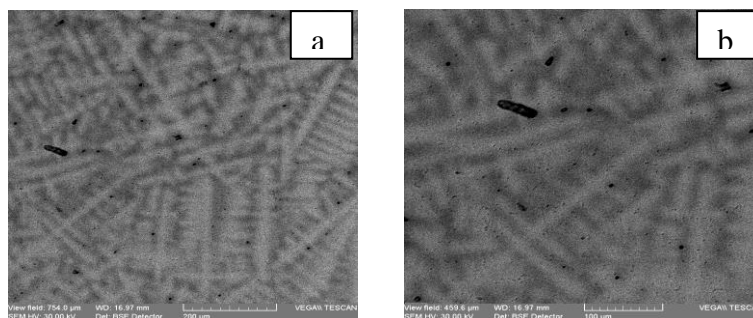


Fig. 4. SEM image of Ti-based alloy: a) 300 X, b) 500 X

The electron micrographs (Fig. 4) of the alloy show a biphasic structure, consisting of a high proportion of β solid solution, occurring a lamellar structures intragranulation peculiar to orthorhombic martensite α'' , as shown in Fig. 3. The orthorhombic martensite α'' occurs frequently in the case of titanium base alloys which are found in β -stabilized transition metal category, in which Ta and Zr is included [15, 18].

4. Conclusions

An alloy was developed from the melting of the pure elements like Ti, Mo, Zr, Ta in an arc melting furnace.

The SEM analysis revealed surfaces without defects and the elements mapping showed a homogeneous distribution for all alloys.

Molybdenum, zirconium and tantalum alloying elements present an influence on the structure and microstructure of alloy, through the β - stabilizer action. The microstructure of the studied alloy presents an acicular structure with irregular grain boundaries, with a β phase. The XRD results present a predominant β phase and an α'' secondary phase shown in the Figure 2. Finally, we conclude that the presented alloy can be used for medical application.

References

- [1]. D. M. Bombac, *et al.*, Review of materials in medical applications, Materials and Geoenvironment, 54, (4), p. 471-499, 2007.
- [2]. M. G. Minciuna, P. Vizureanu, D. C. Achitei, N. Ghiban, A. V. Sandu, N. C. Forna, Structural Characterization of Some CoCrMo Alloys with Medical Applications, 65, (3), p. 335-338, 2014.
- [3]. M. Niinomi, M. Nakai, J. Hieda, Development of new metallic alloys for biomedical applications, Acta Biomater, 8, p. 3888-3903, 2012.

- [4]. C. Leyens, P. Manfred, Titanium and titanium alloys: Fundamentals and Applications, John Wiley & Sons, 2003.

- [5]. N. T. C. Oliveira, G. Aleixo, R. Caram, A. C. Guastaldi, Development of Ti-Mo alloys for biomedical applications: Microstructure and electrochemical characterization, Materials Science and Engineering A, 452-453, p. 727-731, 2007.

- [6]. L. C. Zhang, D. Klemm, J. Eckert, Y. L. Hao, T. B. Sercombe, Manufacture by selective laser melting and mechanical behavior of a biomedical Ti-24Nb-4Zr-8Sn alloy, Scr. Mater, 65, p. 21-24, 2011.

- [7]. M. G. Minciuna, P. Vizureanu, V. Geanta, I. Voiculescu, A. V. Sandu, D. C. Achitei, A. M. Vitalariu, Effect of Si on the Microstructure and Mechanical Properties of Biomedical CoCrMo Alloy, Revista de chimie, 66(6), p. 891-894, 2015.

- [8]. Y. L. Zhou, M. Niinomi, T. Akahori, Effects of Ta content on Young's modulus and tensile properties of binary Ti-Ta alloys for biomedical applications, Materials Science and Engineering A, 371, p. 283-290, 2004.

- [9]. S. B. Gabriel, *et al.*, Characterization of a new beta titanium alloy, Ti-12Mo-3Nb, for biomedical applications, J. Alloys Compd., 536 (Suppl.1), p. S208-S210, 2012.

- [10]. A. C. Bărbintă, R. Chelariu, M. Benchea, C. I. Crimu, S. Iacob Strugaru, C. Munteanu, A comparative analysis of new Ti-Nb-Zr-Ta orthopedic alloys, Advanced Materials Research, 837, p. 259-264, 2014.

- [11]. ***, ASM Handbook, Alloy Phase Diagrams, vol. 3, p. 254.

- [12]. ***, ASM Handbook, Metallography and Microstructure, vol. 9, p. 2157-2208.

- [13]. I. C. Lupu, D. Agop-Forna, I. G. Sandu, C. Mocanu, Microscopic Assessment of the Corrosion Resistance of some Superficially Enhanced Ti-Based Dental Alloys with Hydroxyapatite, Revista de Chimie, 66 (6), p. 808-812, 2015.

- [14]. Lutjering G., Williams J. C., Titanium, Springer-Verlag, Berlin, p. 289, 2003.

- [15]. X. H. Min, S. Emura, L. Zhang, K. Tsuzaki, Effect of Fe and Zr additions on ω phase formation in β -type Ti-Mo alloys, Materials Science and Engineering A, 497, p. 74-78, 2008.

- [16]. Oliveira N. T. C., Aleixo G., Caram R., Guastaldi A., Development of Ti-Mo alloys for biomedical applications: Microstructure and electrochemical characterization, Materials Science and Engineering A, 452-453, p. 727-731, 2007.

- [17]. D. R. N. Correa, *et al.*, Effect of substitution elements on the microstructure of the Ti-15Mo-Zr and Ti-15Zr-Mo system alloys, J Mater Res Technol., 4(2), p. 180-185, 2015.

- [18]. S. Ehteman Haghighi, H. B. Lu, C. Y. Jian, G. H. Cao, D. Habibi, L. C. Zhang, Effect of α'' martensite on the microstructure and mechanical properties of beta-type Ti-Fe-Ta alloys, Materials and Design, 76, p. 47-54, 2015.