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METALLOGRAPHIC CHARACTERIZATION OF A NEW BIOMEDICAL TITANIUM-BASED ALLOY FOR ORTHOPEDIC APPLICATIONS

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Abstract: *Orthopedic biomaterials play an important role for the patients, improving the quality and expectancy of life. Titanium alloys corresponding to the Ti-Nb-Zr-Ta system, composed of non-toxic elements, has attracted the attention of the researchers, because of their unique combination of properties, which make them potential candidates for medical applications in the orthopedic field. In this paper is presented a new biocompatible titanium alloy, Ti-25Nb-10Zr-8Ta, which was produced from pure metals (Ti, Nb, Zr and Ta) using the vacuum induction melting method in a levitation furnace Five Celes. Microstructure and phase composition of the titanium alloy were investigated by scanning electron microscopy (SEM) and X-Ray diffraction (XRD).*

Key words: *Ti-25Nb-10Zr-8Ta, scanning electron microscopy, X-ray diffraction.*

1. Introduction

In recent years, the increase in the average age of the population has lead to the growth in the number of surgical procedures, including hip and knee replacement (total or partial), whereas as the human body ages, the joints become more prone to illness [5]. Also, the trend of biomaterials research and development is also justified by the estimates that by the end of 2030 the number of hip replacements will reach up to 572.000, while the number of knee arthroplasties will rise to around 3.48 million [4].

Not only the replacement surgeries have increased, but also the revision surgeries of the orthopedic implants [4]. As a consequence, researchers have become more interested to improve biomaterials properties and manufacturing technologies of the orthopedic implants [4], [8].

Metallic biomaterials are considered to be the best choice for the medical applications, which require replacement of hard biological tissue, due to their excellent mechanical properties, good corrosion resistance and biocompatibility.

The current metallic materials used in the orthopedic field are: cobalt-chromium alloys,

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stainless steels and titanium alloys, especially Ti-6Al-4V.

These biomaterials have demonstrated to be inefficient, due to their lack of biocompatibility, low corrosion and wear resistance and high values of modulus of elasticity. Studies have shown that both vanadium and aluminium ions released from Ti-6Al-4V alloy are toxic for human body, causing severe health problems, such as osteomalacia, Alzheimer disease [4].

Therefore, the researchers have developed a new generation of titanium alloys without aluminium and vanadium, corresponding to the Ti-Nb-Zr-Ta system, which are composed of non-toxic and non-allergenic elements. These alloys were intensively investigated in the last decade, being published many studies, regarding their biocompatibility, mechanical properties, corrosion resistance and methods of improving their properties, in order to eliminate the negative aspects, so that these biomaterials could successfully become future orthopedic implants [4], [8].

Ti-Nb-Zr-Ta alloys are the result of the studies made by Rack and Silvestri and their collaborators from Clemson University and from Teledyne Allvac, respectively [1]. The selection of the alloying elements for titanium was realized, taking into consideration the studies on cell viability for pure metals, corrosion resistance, biocompatibility and also the allergic properties of the pure metals [9]. As a result, niobium, tantalum and zirconium have been selected as the best suited alloying elements for titanium.

Beta titanium alloys have lower values of modulus of elasticity than α or α+β alloys, an important condition for preventing the stress shielding phenomenon, which is one of the main factors that leads to the failure of the implant [7]. Reduction of the implant stiffness improves the stress redistribution to the adjacent bone tissues, minimizing the stress-shielding phenomenon and prolonging the life of the implanted device, [8].

Niobium and tantalum are known as β-stabilizers and reduce the modulus of elasticity of the alloy if they are added in certain quantities, without compromising its strength. Also, zirconium is isomorphous with both the alpha and beta phases of titanium and plays an important role as β-stabilizer, when is combined with niobium or tantalum, improving the strength of the alloy, because of the solution strengthening effect [2], [3].

A considerable attention have received Ti-29Nb-13Ta-4,6Zr and Ti-35Nb-5Ta-7Zr, which were developed by Niinomi from University of Tohoku of Japan and by Rack from Clemson University of USA, respectively. These alloys are considered to be excellent candidates for medical applications, due to their low values of Young's modulus: 65 and 55 GPa. These values are much lower compared to that of the current biomaterials used as orthopedic implants: 110 GPa for Ti-6Al-4V alloy, 180 GPa for stainless steel 316L and 220 GPa for Co-Cr alloys [9], [10].

This paper presents the metallographic characterization of a new titanium alloy corresponding to the Ti-Nb-Zr-Ta system using modern methods of analysis, such as scanning electron microscopy and X-Ray diffraction.

2. Materials and Methods

2.1. Fabrication of the alloy

The experimental titanium alloy was produced from pure metals (Ti, Nb, Zr and Ta) using the vacuum induction melting method in a levitation furnace Five Celes, which has the possibility to combine elements with very different melting points, achieving high temperatures in just a few seconds. The temperature in the furnace can reach over 2000 ºC and this is sufficient to melt all the component elements of the alloy, without contamination and obtaining a uniform chemical composition, [11].

The cylindrical ingot obtained had a diameter of about 19 mm and was cut with a diamond disc into sections with a thickness of approximately 3 mm.

2.2. Chemical composition and microstructure

The chemical composition and microstructure of the alloy were determined using a Quanta 200 3D scanning electron microscope coupled with energy dispersive X-ray spectrometer, which both form a high performance instrument with three imaging modes: high vacuum, low vacuum and ESEM. The analyses were made in high vacuum mode at a 20 kV accelerating voltage and at a working distance of 15 mm.

For metallographic characterization, the sample was prepared by grinding with silicon carbide abrasive papers of 600, 1200 and 2000 grit and finally by polishing with suspension of alumina. For metallographic etching was used Kroll's reagent, a mixture of H_2O, HF and HNO_3.

2.3. X-Ray diffraction analysis

The phases of the Ti-25Nb-10Zr-8Ta alloy were revealed by X-ray diffraction (XRD) using a PANalytical X'Pert PRO MRD system with CuKα radiation. During the analysis, the X-ray tube operated at a voltage of 45 kV and at a 40 mA current.

3. Results and Discussion

3.1. Chemical composition of the alloy

The chemical composition of the alloy was determined by energy dispersive X-ray spectroscopy.

The EDX spectrum and chemical composition in atomic (at %) and weight (wt %) percentages of each of the element identified in the material are presented in Figure 1.

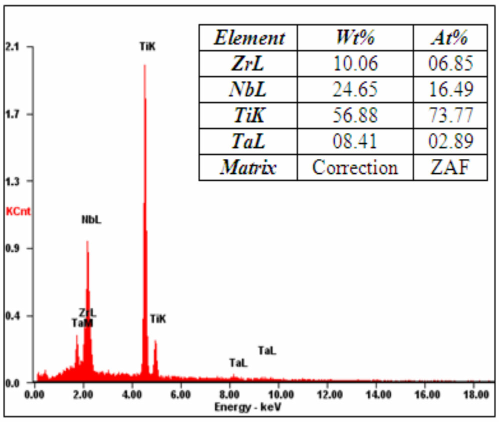

Fig. 1. *EDX spectrum and chemical composition in atomic (at %) and weight (wt %) percentages of each of the element identified in the alloy*

3.2. Microstructure of the alloy

Backscattered and secondary electrons images were used for analyzing the microstructure of the Ti-25Nb-10Zr-8Ta alloy. According to the SEM micrographs, the microstructure of the as-cast titanium alloy seems to be inhomogeneous, due to microsegregation, which occurs during solidification process and phase transformation of the alloy, due to high cooling rate and low atom diffusion rate [12]. Also, the addition of large amounts of refractory metals, such as niobium and tantalum results in segregation problems during solidification [6].

Backscattered electron image (BSE) of the as-cast titanium alloy is presented in Figure 2. Due to different average atomic numbers present in the composition of the alloy, the microsegregation area appears as dark and light grey regions. The grey regions represent dendrite morphology and the dark ones are the interdendritic junctions of the primary solidified dendrites.

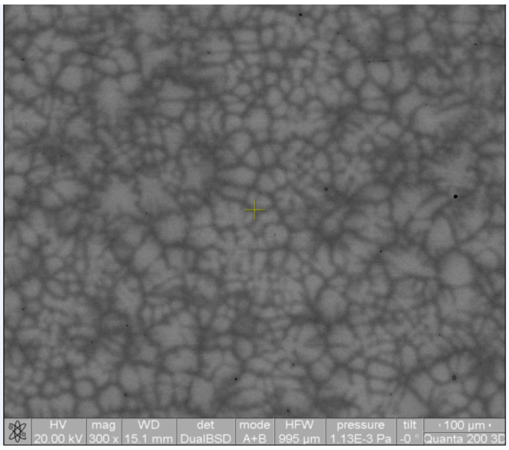

Fig. 2. *BSE image of the as-cast alloy showing typical dendritic structure (300X)*

As can be seen in Figure 3, EDS compositional analyses revealed that the dark areas are rich in zirconium and titanium, while the grey matrix presents a higher content of niobium and tantalum.

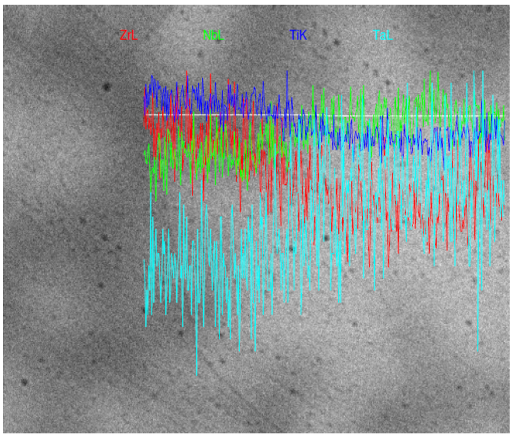

Fig. 3. *EDX line scan showing overlapping profiles for the component elements of the alloy (3000X)*

According to the EDX line scan analysis presented in Figure 4, there is a non-uniform distribution of the component elements of the titanium alloy along microsegregation.

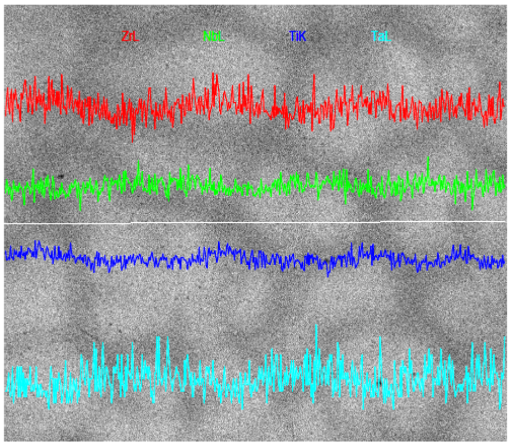

Fig. 4. *EDX line scan showing a non-uniform distribution of the elements across microsegregation (1500X)*

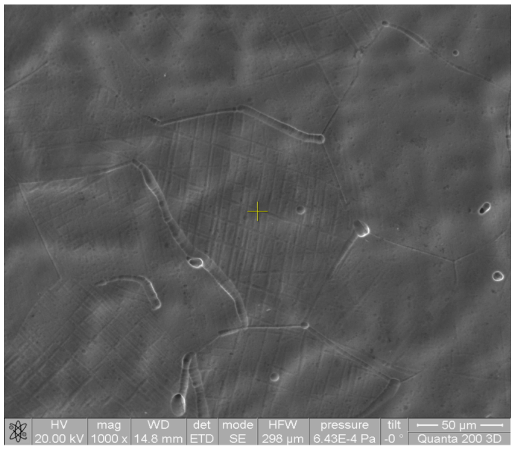

Fig. 5. *Secondary electron image (SE) showing the microstructure of Ti-25Nb-10Zr-8Ta alloy (1000X)*

Although Ti-25Nb-10Zr-8Ta alloy has a high concentration of β-stabilizers, such as niobium and tantalum, it presents a biphasic structure, consisting mostly of β phase with some amounts of intra-granular α phase too (Figure 5). Figure 6 presents a detailed view of a β grain, in which can be identified the lamellar structures specific to α-phase.

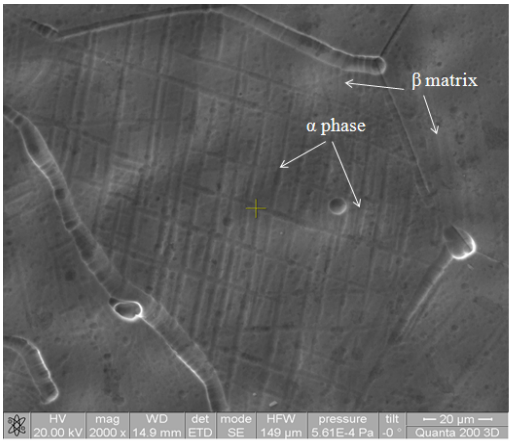

Fig. 6. *SE image revealing the main phases of the as-cast titanium alloy (2000X)*

3.3. X-ray diffraction results

The diffractogram of the as-cast titanium alloy studied in this paper is presented in Figure 7. According to XRD profile, Ti-25Nb-10Zr-8Ta alloy consists mainly of β-phase with a body-centered cubic (bcc) crystal structure and a minor content of α-phase too.

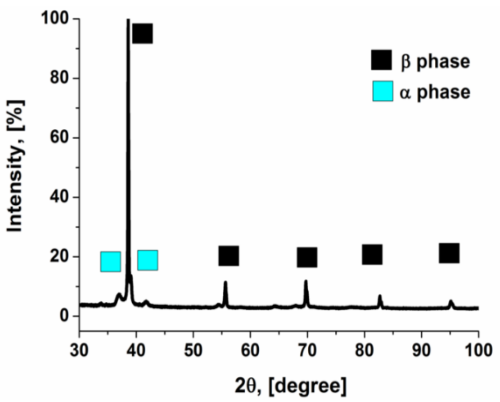

Fig. 7. *XRD diffraction pattern of the as-cast Ti-25Nb-10Zr-8Ta alloy*

4. Conclusions

Scanning electron microscopy with backscattered and secondary electrons coupled with EDS analyses and X-ray diffraction provide a complete metallographic characterization of the alloy, being modern tools for determining chemical composition, identifying defects appeared during solidification process or revealing the main phases of the alloy.

The novel Ti-25Nb-10Zr-8Ta alloy presents a biphasic structure, consisting mostly of β phase with some amounts of intra-granular and lamellar α phase too.

The Ti-25Nb-10Zr-8Ta can be classified as a near-β titanium alloy.

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