Bulletin of the *Transilvania* University of Braşov
Series I: Engineering Sciences • Vol. 6 (55) No. 1 - 2013

# INVESTIGATION OF A NEW Ti-Nb-Zr-Ta ALLOY BY ELECTRON MICROSCOPY, X-RAY DIFFRACTION AND NANOINDENTATION

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***Abstract:*** *This paper presents a new titanium alloy, corresponding to the Ti-Nb-Zr-Ta system, whose chemical composition is Ti-21Nb-6Zr-15Ta. Microstructure and phase characteristics were analyzed by X-ray diffraction and scanning electron microscopy. Mechanical properties such as Young's modulus and hardness were determined by nanoindentation. Ti-21Nb-6Zr-15Ta is a near-β titanium alloy, containing β grains with a fine lamellar structure of α phase. The investigated alloy presents a low Young's modulus, an important condition for preventing stress shielding phenomenon, which is one of the main factors that causes implant failure.*

***Key words:*** *Ti-21Nb-6Zr-15Ta, scanning electron microscopy, X-ray diffraction, nanoindentation.*

## 1. Introduction

Ti-Nb-Zr-Ta alloys are considered to be very promising for biomedical applications, due to their good corrosion resistance, excellent biocompatibility and good mechanical properties [3].

The alloys corresponding to the Ti-Nb-Zr-Ta system were developed in order to eliminate the negative aspects of the currently used metallic materials in the orthopedic applications, like Ti-6Al-4V, stainless steels and Co-Cr alloys, which present the following disadvantages: low corrosion and wear resistance, high values of modulus of elasticity and poor biocompatibility with human tissues [2].

The Young's modulus of the actual metallic materials is much higher than that of bone, causing bone atrophy as a result of the stress shielding phenomenon that occurs between the bone and the implant [6]. According to Wolff's law, the bone tissue is continuously remodeled depending on the mechanical stress and its decrease or absence may cause bone resorption. Also in fracture healing, mechanical stress has an important role on callus formation and, therefore, on the remodelling [6].

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The Ti-Nb-Zr-Ta alloys are composed mainly of β-phase, which has the lowest Young's modulus among all the titanium phases, reason for which there have been developed many β-type titanium alloys composed of biocompatible elements, the most studied being the following: Ti-29Nb-13Ta-4,6Zr and Ti-35Nb-5Ta-7Zr [3]. Theoretical studies realized by Song et al. [5] have shown that Nb, Zr, Ta and Mo represent the best suited alloying elements for lowering the modulus of elasticity, without compromising the strength of the alloy.

The advantages of Ti-Nb-Zr-Ta alloys as orthopedic implants with low values of modulus of elasticity have been confirmed by experimental studies performed on rabbits [1], [6]. In the case of the implants that are integrated into bone tissue, fixation and bone remodeling are two major concerns, closely related to the local response of bone tissue and osteointegration [1].

The experimental studies conducted by Niinomi and his collaborators have demonstrated that Ti-29Nb-13Ta-4,6Zr alloy with a low value of modulus of elasticity improves acceleration of bone healing and remodelling in comparison with the metallic biomaterials that have higher values of Young's modulus [1]. Similar studies were realized by Sumitomo et al. [6].

## 2. Materials and Methods

### 2.1. Fabrication of the alloy

The titanium alloy investigated in this study was produced from pure Ti, Nb, Zr and Ta using vacuum induction melting method in a levitation furnace Five Celes under pure argon atmosphere, which is shown in Figure 1.

The ingot obtained in as-cast form with a 19 mm diameter was cut into disks of 3 mm height. The chemical composition of the alloy investigated in this study in weight percentages is the following: 21% Nb, 6% Zr, 15% Ta and balance Ti.

Fig. 1. *Levitation furnace Five Celes*

### 2.2. Scanning electron microscopy and X-ray diffraction

For metallographic characterization, the sample was prepared using conventional techniques of grinding and mechanical polishing. The microstructure was revealed by etching with Kroll's reagent, containing a mixture of $H_2O$, HF and $HNO_3$.

Backscattered and secondary electron images of the surface alloy and EDS analyses were made using a Quanta 200 3D scanning electron microscope (Figure 2), working in high vacuum mode at a 20 kV accelerating voltage.

Fig. 2. *Scanning electron microscope Quanta 200 3D*

X-ray diffraction analysis was performed with a PANalytical X'Pert PRO MRD diffractometer, presented in Figure 3, with CuKα radiation, over a scan range of 2θ = 30º to 2θ = 100º angle. The current and the voltage of the X-ray tube during the analysis were 40 mA and 45 kV, respectively. The results were analyzed with X'pert HighScore Plus software.

Fig. 3. *X-ray diffractometer PANalytical X'Pert PRO MRD*

### 2.3. Nanoindentation method

The nanoindentation tests were made using a CETR-UMT-2 microtribometer, presented in Figure 4.


Fig. 4. *Microtribometer CETR-UMT-2*

A Rockwell diamond indenter with an included angle of 120 ºC and a spherical tip with 200 µm radius were used for all the measurements.

The nanoindentation test was repeated five times under the same condition. The load-displacement data resulted from the nanoindentation tests were used to calculate the hardness and elastic modulus.

## 3. Results and Discussion

### 3.1. As-cast microstructure and phase analysis

Figure 5 represents a backscattered electron image (BSE) of the Ti-21Nb-6Zr-15Ta alloy that shows the as-cast microstructure of the alloy.

In the as-cast microstructure of the alloy can be observed dendritic and interdendritic regions, that appear light grey and dark colored, due to the atomic number contrast.

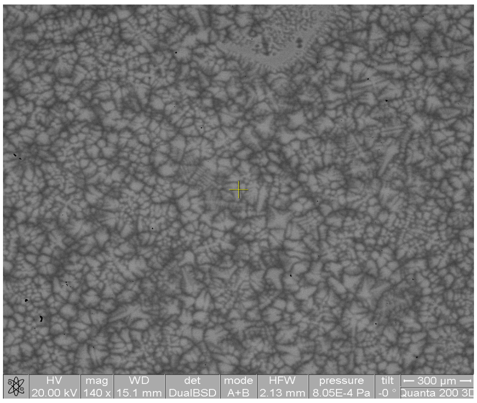


Fig. 5. *BSE image showing the as-cast microstructure of Ti-21Nb-6Zr-15Ta (140X)*

EDS line scan analysis, presented in Figure 6, has revealed that dendritic regions are rich in niobium and tantalum, while the interdendritic ones have a higher content of zirconium and titanium. The chemical inhomogenity is due to microsegregation, which appears during solidification process.

Xu et al. [7] considers that the extent of the microsegregation depends on several factors, like solidification path, cooling rate and diffusion rate of the component elements.

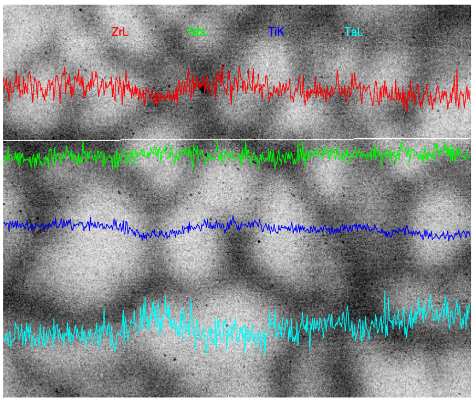

Fig. 6. *EDS line scan showing a non-uniform distribution of the Ti, Nb, Zr and Ta along microsegregation (1500X)*

Secondary electron images of Ti-21Nb-6Zr-15Ta alloy are shown in Figures 7 and 8.

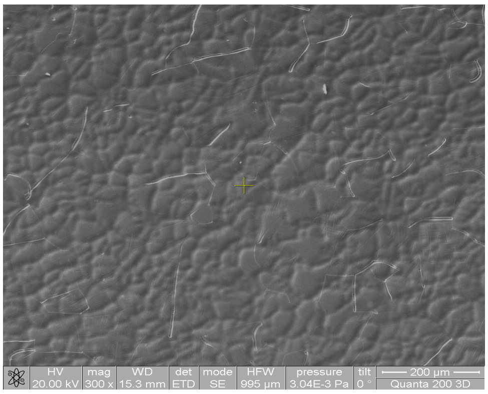

Fig. 7. *SE image revealing the β matrix with grains containing lamellar structures of α phase (300X)*

The microstructure of Ti-21Nb-6Zr-15Ta is characterized by the presence β large grains having a 26.81 μm average grain size. Also in Figure 8, where is presented in detail a β-grain, it can be observed intra-granular lamellar structures specific to α-phase.

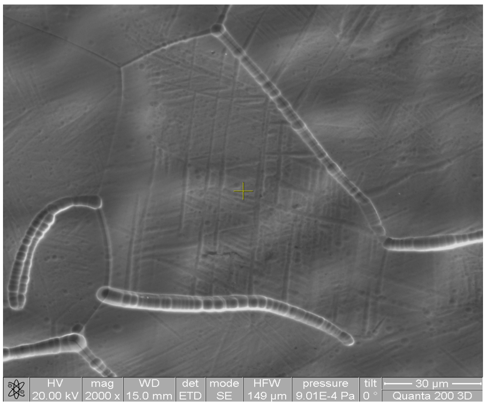

Fig. 8. *SE image showing in detail a β grain (2000X)*

The XRD pattern of Ti-21Nb-6Zr-15Ta alloy is presented in Figure 9. According to XRD results, the as-cast alloy consists of beta and alpha phases, confirming the results obtained by scanning electron microscopy analysis.

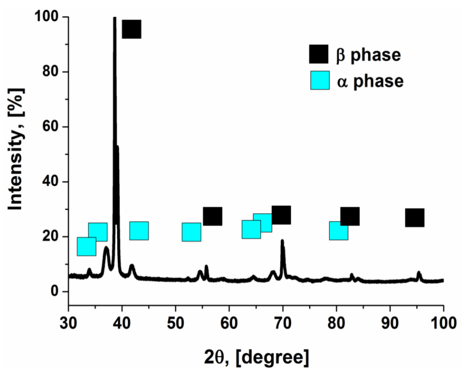


Fig. 9. *XRD pattern of Ti-21Nb-6Zr-15Ta*

### 3.2. Elastic modulus and hardness of Ti-21Nb-6Zr-15Ta

The values of elastic modulus and hardness of Ti-21Nb-6Zr-15Ta alloy resulted from nanoindentation tests are listed in Table 1.

According to the results obtained, the modulus of elasticity of Ti-21Nb-6Zr-15Ta is approximately 58 GPa, a value much lower compared to that of the current metallic materials used in the orthopedic field: 110 GPa for Ti-6Al-4V, 180 GPa for stainless steel 316L and 220 GPa for Co-Cr alloys [4].

*Nanoindentation tests results* Table 1

| Test number | Elastic modulus [GPa] | Hardness [GPa] |
|---|---|---|
| 1 | 54.8 | 1.227 |
| 2 | 58 | 1.393 |
| 3 | 60 | 1.440 |
| 4 | 58.7 | 1.398 |
| 5 | 57 | 1.431 |

In Figure 10 is presented the load-displacement curve resulted after nanoindentation test with number 3. As can be seen, for a maximum load of approximately 14 N the maximum displacement is about 9.077 μm.

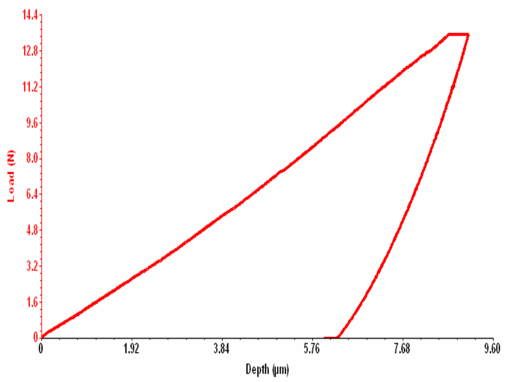


Fig. 10. *Load-displacement curve obtained during nanoindentation test*

Scanning electron microscopy was used for detecting the imprints obtained after nanoindentation. In Figure 11 can be identified the marks of the indenter left on the alloy surface. The contact depth is 7.605 μm.

## 4. Conclusions

The Ti-21Nb-6Zr-15Ta alloy, produced by vacuum induction method, is a near β titanium alloy with large β grains that contain lamellar structure of α phase.

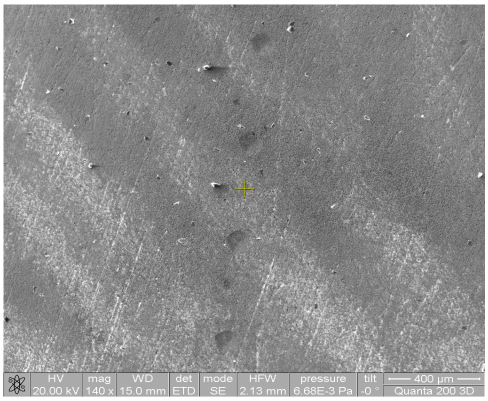


Fig. 11. *SE image showing nanoindentations imprints on Ti-21Nb-6Zr-15Ta surface*

Elastic modulus determined by nanoindentation method is approximately 58 GPa, a lower value compared to that of the other metallic biomaterials, which is an important factor for favouring acceleration of bone healing and remodeling.

A lower value of the hardness alloy can accelerate the wear process, which will influence the durability of the orthopedic implant, reason for which it is desired to use a surface modification method in order to eliminate this disadvantage.

## Acknowledgments

The authors thank to the S.C. R&D Consulting and Services S.R.L. from Bucharest for providing the titanium alloy needed for realizing this study.

This paper was realised with the support of POSDRU CUANTUMDOC "Doctoral Studies for European Performances in Research and Inovation" ID79407, project funded by the European Social Found and Romanian Government.

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