

## MECHANICAL, STRUCTURAL AND CORROSION ANALYSIS OF A Ti-Nb-Zr-Fe ALLOY DESIGNATED TO ORAL IMPLANTOLOGY

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*The present study presents the mechanical and structural evaluation, as well as the corrosion resistance of a Ti-31.7Nb-6.21Zr-1.4Fe-0.16O (wt%) alloy, designated to oral implantology. The as studied alloy was mechanically and structurally tested, in condition obtained after melting in a levitation induction furnace type FIVE CELES - MP25. Regarding alloy mechanical properties, the microhardness was studied; the alloy structural evaluation comprises the microstructure and fracture surfaces analysis. The corrosion resistance was determined through the linear polarization technique, with a Potentiostat/Galvanostat/EIS Analyzer (model PARSTAT 4000, producer Princeton Applied Research); the potentiodynamic curves were obtained with the VersaStudio software.*

**Keywords:** biomedical titanium alloy, mechanical properties, structural characterization, titanium alloy corrosion resistance

### 1. Introduction

Titanium alloys have an extended use as dental implant materials, due to their biocompatibility, corrosion resistance and high specific strength. As alloying base, Titanium is considered to be totally inert and immune in the human body, and thus wholly biocompatible [1].

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Many  $\alpha$ - $\beta$  titanium alloys were developed, but their toxicity to a certain extent and their elastic modulus, still higher than that of human bone, are characteristics which have to be improved.

The necessity of a new biomedical titanium alloy, with a high property-to-cost ratio, leads to the development of new alloys.

The research is focused on  $\beta$ -type titanium alloys, with non-toxic alloying elements, in order to obtain improved tissue response, lower elastic modulus and higher corrosion resistance [2]. Newly developed metastable  $\beta$ -titanium alloys, containing high density metals such as: Mo, Nb and Ta, have a high cost of raw materials and alloy preparation.

$\beta$  type alloys with alloying elements such as Nb, Zr, are non-toxic materials and provide a lower elastic modulus, which is the main target for medical materials [3], [4]. Also Fe can effectively decrease titanium alloys melting point, being a solution strengthening element and a strong  $\beta$ -stabilizer [5].

A high corrosion resistance of biomedical alloys is important because, if corrosion occurs, metallic ions are released into the body, interfere with the process of life.

The aim of this study was to investigate the mechanical and structural properties, as well as the corrosion resistance of a Ti-Nb-Zr-Fe alloy, designated to oral implantology.

## 2. Materials and methods

The as studied alloy was obtained after melting in a levitation induction furnace type FIVE CELES - MP25, under argon protective atmosphere, with a nominal power of 25 kW and a melting capacity of 30 cm<sup>3</sup>. The samples for the structural and mechanical testing, as well as for the corrosion analysis, were obtained from the ingots of the as studied alloy. The chemical alloy composition is presented in Table 1.

Table 1

**Elemental composition of the as studied alloy**

Element (wt%)			
Nb	Zr	Fe	Ti
31.7	6.21	1.4	Residue

Regarding the mechanical evaluation of the Ti-31.7Nb-6.21Zr-1.4Fe-0.16%O (wt%) alloy, the micro-hardness was determined with a Wilson-Wolpert 401MVA micro-hardness indenter and the tensile strength was measured with a GATAN MicroTest 2000 N traction-compression module.

A Tescan Vega II-XMU electronic microscope, with two integrated detectors, (in order to) for obtaining SEM images - Back-Scattered Electron

detector (BSE) and Secondary Electron detector (SE), was used to investigate the microstructure and the fracture surfaces of the as studied alloy.

The corrosion resistance was determined through the linear polarization technique, which consists of plotting the linear polarization curves with a Potentiostat/Galvanostat/EIS Analyser (PARASTAT 4000, produced by Princeton Applied Research). Two steps were applied: the measurement of the open circuit potential ( $E_{oc}$ ) for 20 hours, and the plotting of the potentiodynamic curves, with a scanning rate of 1mV/s, from -1V (vs OC) to +1V (vs SCE). The potentiodynamic curves were obtained with the VeraStudio v.2.4.2 software. In order to carry out the tests a standard corrosion cell was used, having a saturated calomel electrode (SCE) – as reference electrode, a platinum electrode – as recording electrode and a working electrode - consisting of a sample obtained from the studied alloy. A Faraday cage was used, so that the electromagnetic interferences were eliminated during the tests. Artificial saliva (A.S.) and Hank's solution were used as simulated physiological environments, at a temperature of  $37 \pm 0.5^\circ\text{C}$ .

### 3. Results and discussions

#### 3.1. Hardness evaluation of the Ti-31.7Nb-5.21Zr-1.4Fe-0.16O (wt%) alloy

Vickers micro-hardness (HV) was determined with a 30 seconds maximum force contact, by using a force of 0.2 kgf. A number of 10 determinations were performed and after eliminating the minimum and the maximum, the average of the 8 remaining values was computed. For the acicular phase, the micro-hardness showed a value of 222 HV<sub>0.2</sub> and for the homogenous phase the value was 238 HV<sub>0.2</sub> (Fig.1.).

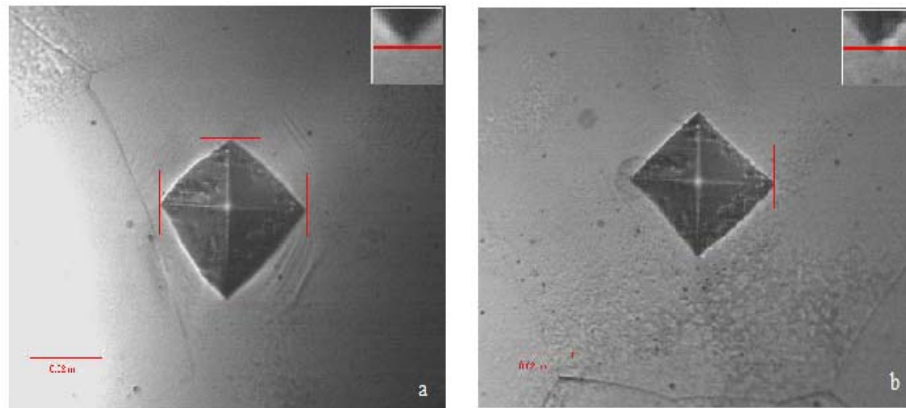


Fig. 1. Images of micro-hardness impression (a and b) in case of Ti-31.7Nb-6.21Zr-1.4Fe-0.16%O (wt%) alloy.

### 3.2 Fracture surfaces of the Ti-31.7Nb-5.21Zr-1.4Fe-0.16O (wt%) alloy

The fracture surfaces of the as studied alloy were analyzed by using Scanning Electron Microscopy (SEM). SEM images reveal a ductile fracture of the Ti-31.7Nb-5.21Zr-1.4Fe-0.16O (wt%) alloy, through void nucleation, growth and coalescence (Fig.2.).

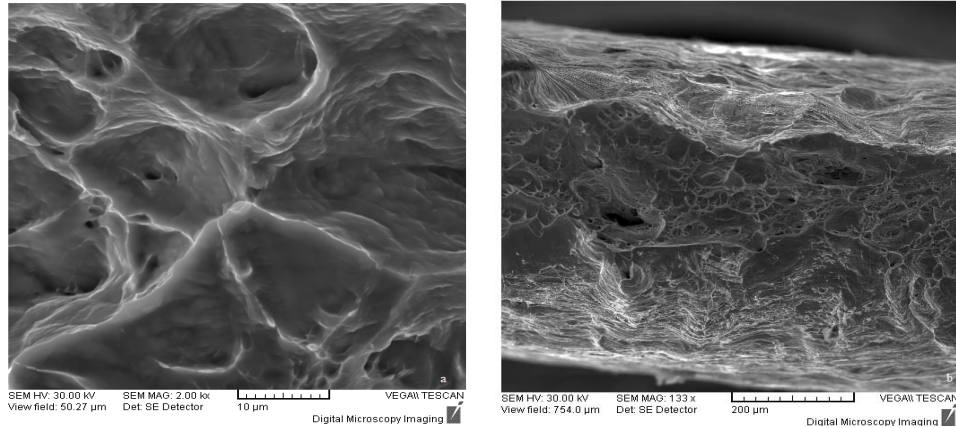


Fig. 2. SEM images of the fracture surface of Ti-31.7Nb-5.21Zr-1.4Fe-0.16O (wt%) alloy (a,b), with different magnifications, using SE detector

### 3.3. Structural characteristics of the Ti-31.7Nb-5.21Zr-1.4Fe-0.16O (wt%) alloy

The Scanning Electron Microscopy (SEM) micrographs, obtained using BSE detector and with no metallographic attack of the investigated samples, show areas rich in Nb, represented in white, and areas with a lower content of Nb, which are darker represented (Fig. 3a). When metallographic attack was used (10ml HNO<sub>3</sub>, 5 ml HF, rest H<sub>2</sub>O), the crystallographic structure reveals an unique  $\beta$ -Ti phase, with equiaxed polyedric grains, having an average dimension of 100-120  $\mu$ m. Areas with different concentrations of Nb can be observed inside the  $\beta$ -Ti crystal grains (Fig.3b).

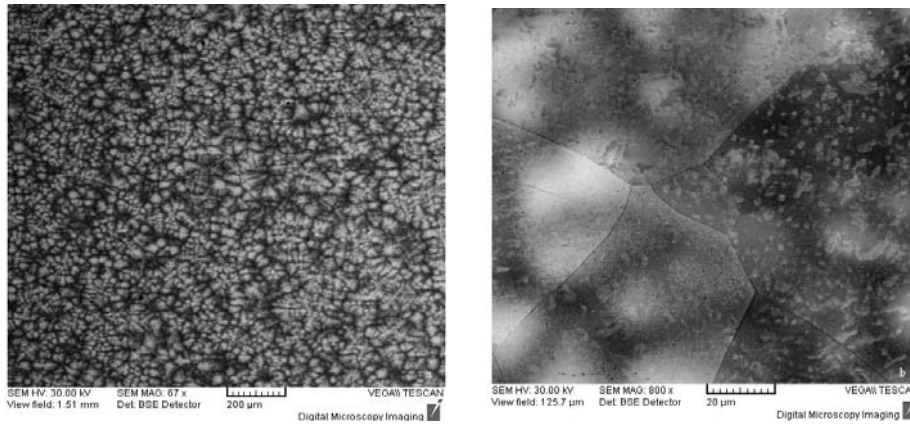


Fig. 3. SEM micrographs showing the microstructure of the Ti-31.7Nb-6.21Zr-1.4Fe-0.16%O (wt%) alloy in as-cast state (a) and SEM micrographs showing the microstructure when metallographic attack was used (b).

### 3.4. Corrosion behavior evaluation of the Ti-31.7Nb-5.21Zr-1.4Fe-0.16O (wt%) alloy

The parameters determined from the polarization curves, characterizing the corrosion resistance of the Ti-Nb-Zr-Fe alloy, are as follows: the open circuit potential ( $E_{oc}$ ), the corrosion potential ( $E_i=0$ ), the corrosion current density ( $i_{cor}$ ), the corrosion rate (CR). The following formula, according to the ASTM G102-89 (2004), was used to calculate the corrosion rate:

$$CR = K_i \frac{i_{cor}}{\rho} EW \quad (1)$$

where:

CR- corrosion rate  
 $K_i$  –  $3.27 \times 10^{-3}$   
 EW – equivalent weight  
 $i_{cor}$  – corrosion current density  
 $P$  – material density

Table 2

The main parameters of the corrosion process of the Ti-31.7Nb-5.21Zr-1.4Fe-0.16O (wt%)

$E_{oc}$ [mV]		$E_{cor}$ [mV]		$i_{cor}$ [nA/cm <sup>2</sup> ]		CR [μm/an]	
A.S.	Hank	A.S.	Hank	A.S.	Hank	A.S.	Hank
-70,158	-117,544	-222,602	-324,159	111,688	88,324	0,917	0,725

The evolution of the open circuit potential can be seen in Fig. 4. and the potentiodynamic curves in Fig.5.

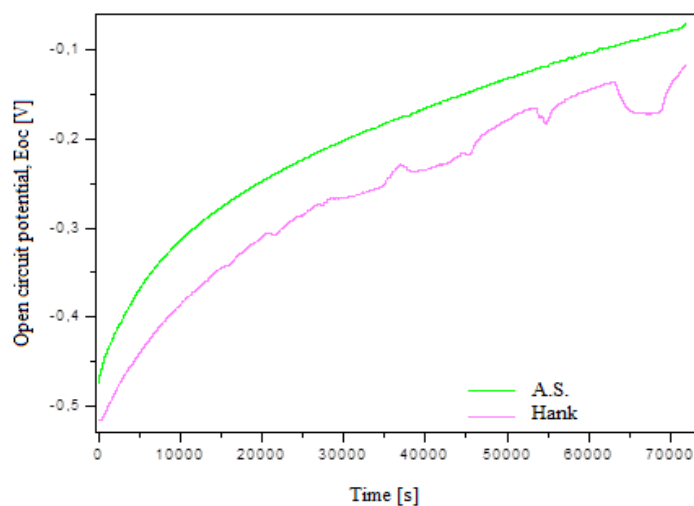


Fig.4. Evolution of the open circuit potential ( $E_{oc}$ ) of the Ti-31.7Nb-5.21Zr-1.4Fe-0.16O (wt%) alloy in the solutions used as an electrolyte

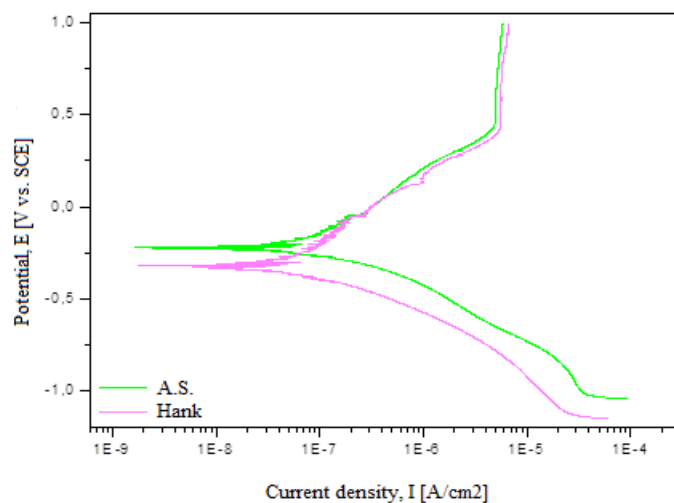


Fig.5. The potentiodynamic curves of the Ti-31.7Nb-5.21Zr-1.4Fe-0.16O (wt%) alloy in the solutions used as an electrolyte.

#### 4. Conclusions

The Ti-31.7Nb-5.21Zr-1.4Fe-0.16O (wt%) alloy was developed and manufactured and then being examined, in order to evaluate its mechanical and structural characteristics, as well as its corrosion behavior. In as-cast condition the alloy shows an unique  $\beta$ -Ti phase, with equiaxed polyedric grains and areas with different concentration of Nb. Regarding the mechanical behavior of the as studied alloy, the Vickers microhardness was evaluated and found to be good (222 HV<sub>0.2</sub> for the acicular phase and 238 HV<sub>0.2</sub> for the homogenous phase) compared with other standard titanium alloys. The fracture surfaces revealed a ductile fracture of the as studied alloy, by void nucleation, growth and coalescence.

From the shape of the potentio-dynamic curve and especially from the anodic curve, it can be seen that after the value of aprox. 400 mV, regardless of the electrolyte used in all experiments, the as studied alloy has a passivation trend, which demonstrates that the alloy forms an adhesive and protective oxide layer on its surface.

The resulted mechanical, structural and corrosion combination of features indicate the suitability of the Ti-31.7Nb-5.21Zr-1.4Fe-0.16O (wt%) alloy as candidate for a dental implant alloy.

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