

## THE EVOLUTION OF MECHANICAL CHARACTERISTICS FOR A THERMOMECHANICAL PROCESSED Ti-Ta-Zr ALLOY

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*În ultimii ani a existat o dezvoltare semnificativă a noilor sisteme de aliaje pentru implanturi, bazate pe Ti, de tipul Ti-Ta-Zr sau Ti-Ta-Nb. Titanul și aliajele sale au fost intens folosite în medicină încă din 1960, datorită biocompatibilității lor binecunoscute, proprietăților mecanice superioare, a densității scăzute și a stabilității chimice remarcabile în mediul corpului uman.*

*Studiul de față investighează microstructura și proprietățile mecanice pentru un aliaj Ti-Ta-Zr. Rezultatele experimentale au indicat faptul că acest aliaj posedă o gamă diferită de structuri și proprietăți mecanice în funcție de starea aliajului: turnat, laminat la rece și recristalizat.*

*In recent years has occurred a significant development of new implant alloys based on Ti such as Ti-Ta-Zr or Ti-Ta-Nb alloys systems. Titanium and its alloys have been widely used in medicine since the 1960s because of their known biocompatibility, superior mechanical properties, low density and remarkable chemical stability in the human body environment.*

*The present study investigates the microstructures and the mechanical properties of a Ti-Ta-Zr alloy. The experimental results indicated that this alloy possess a range of different structures and mechanical properties dependent upon the alloy condition: as-cast, cold-rolled, recrystallized.*

**Keywords:** titanium alloy, biocompatibility, thermo mechanical processing

### 1. Introduction

The ideal biomaterial for medical implant applications, especially for load-bearing joint replacements, is expected to exhibit excellent biocompatibility with no adverse cytotoxic reactions, excellent corrosion resistance, and a good

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combination of mechanical properties such as high strength and low elastic modulus [1]. In comparison with other metallic biomaterials, titanium and titanium alloys are more biocompatible, more corrosion resistant and can exhibit low elastic modulus. Considerable efforts are now made by material engineers to develop biomedical alloys with low modulus and non-toxic elements [2].

It has been reported that,  $\beta$  rich Ti-Ta-Zr alloys are better substitutes to conventional alloys, as these materials possess low modulus and consist of non-toxic elements [3]. Zr addition leads to a high level blood compatibility when used in cardiovascular implants and better corrosion resistance due to the formation of a stable oxide layer [4]. Another issue with the existing Ti-base medical alloy is that their elastic modulus is significantly higher comparing with bone tissue (30–40 GPa) elastic modulus, leading to stress-shielding that can potentially cause bone resorption and eventual failure of the implant.

Titanium can be alloyed with a variety of elements to alter its properties and enhance its strength, high-temperature performance, creep resistance, weldability and formability [5,6].

Thermo mechanical processing is often performed on titanium alloys to attain a desired combination of mechanical properties. In this paper, specimens in as-cast, cold rolled and recrystallized condition, were examined by scanning electron microscopy (SEM). These results suggested the presence of  $\beta$  phases.

The investigated Ti-Ta-Zr alloy was also subjected to mechanical tests, for measuring the following characteristics: ultimate tensile strength ( $\sigma_{UTS}$ ), yield strength ( $\sigma_{YS}$ ), elongation to fracture ( $\epsilon_f$ ) and elastic modulus (E).

## **2. Materials and methods**

### **2.1. Material synthesis**

The investigated alloys has been obtained using a vacuum induction melting in levitation furnace FIVES CELES with nominal power 25 kW and melting capacity 30 cm<sup>3</sup>, starting from elemental components. The resulted chemical composition in wt.% was: 50%Ti; 25%Ta; 5%Zr.

### **2.2. Cold roll-milling**

The cold-rolling process was carried out using a laboratory roll-milling machine Mario di Maio LQR120AS.

The strain obtained after a rolling pass can be calculated using the following formula:

$$\varepsilon_{pass i} = \frac{h_{i-1} - h_i}{h_{i-1}} \cdot 100 \quad [\%] \quad (1)$$

where:

$\varepsilon_{pass i}$  – represent the strain for rolling pass  $i$ ;  $h_{i-1}$  – represent the height before rolling pass  $i$ ;  $h_i$  – represent the height after rolling pass  $i$ ;

Total accumulated strain after the rolling process can be calculated using the following formula:

$$\varepsilon_t = \frac{h_i - h_f}{h_i} \cdot 100 \quad [\%] \quad (2)$$

where:

$\varepsilon_t$  – represents the total accumulated strain during rolling;  $h_i$  – represents the initial sample height before rolling process;  $h_f$  – represents the final sample height after rolling process;

The equation (2) can be rewritten as:

$$\frac{1}{\varepsilon_t} = \frac{h_i}{\varepsilon_{pass1} \times h_1 + \varepsilon_{pass2} \times h_2 + \varepsilon_{pass3} \times h_3 \dots + \varepsilon_{passn} \times h_n} \quad (3)$$

The above formulas will be used to calculate the geometric parameters of the total accumulated strain after the roll-milling process.

### 2.3. Recrystallization treatment

Samples were cut from the cold-rolled alloy in order to process them by recrystallization treatment. Recrystallization treatment was performed in a GERO SR 100X500/12 – high temperature furnace. Recrystallization parameters were as follows: recrystallization temperature: 850°C; recrystallization duration: 0.5 h; treatment media: argon; cooling media: air.

### 2.4. Mechanical testing

Samples in as-cast, cold-rolled and recrystallized state were subject to mechanical investigations. The tests were carried out using a tensile-compression testing module GATAN MicroTest 2000N. Samples with 0.35x1.65x40 mm dimensions were used. Main testing parameters were as follow: testing speed 0.4 mm/min; testing temperature 20°C. Ultimate tensile strength ( $\sigma_{UTS}$ ), yield strength ( $\sigma_{YS}$ ), elongation to fracture ( $\varepsilon_f$ ) and elastic modulus (E) were obtained by mechanical testing.

The fracture surfaces were investigated in order to observe the predominant fracture mechanisms presented in the as-cast, cold-rolled and recrystallized material, using a TESCAN VEGA II – XMU SEM microscope.

### 3. Results and Discussion

#### 3.1. Cold roll-milling

The roll-milling process was conducted in order to achieve on each rolling step a deformation degree of about 6.6%.

The total accumulated strain after the Ti-Ta-Zr alloy roll-milling process was about 82% for the (see fig. 1) [7-8].

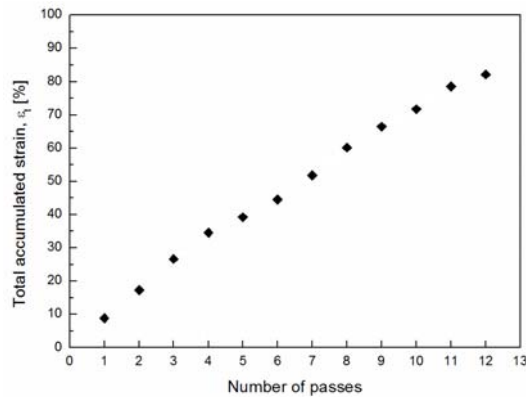


Fig. 1. Total accumulated strain and number of passes during Ti-Ta-Zr alloy cold roll-milling

#### 3.2. Recrystallization treatment

Recrystallization it is a thermo-mechanical process that applied after the cold-rolling process lead to obtain a new structure, with new, un-deformed grains.

Cold-rolling is accompanied by the strain hardening phenomenon that change all the properties of the material. The strain hardened materials have greater strength as the degree of deformation increases. The recrystallization process removes the strain hardened state.

Recrystallization process is influenced of :

- the size of the deformation degree;
- temperature;
- initial grain size;
- chemical composition.

### 3.3. Mechanical testing

Mechanical tests were performed on samples found in three conditions: as-cast, cold-rolled and recrystallized (see fig. 2 – 4).

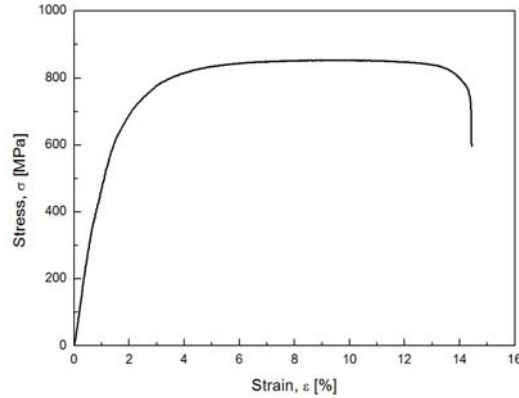


Fig. 2. Stress-strain curve for Ti-Ta-Zr alloy in as-cast condition

As presented in table 1, one can observe an increase in the ultimate tensile strength ( $\sigma_{UTS}$ ) for the cold-rolled material (1303.84 MPa) and for the recrystallized material (1125.33 MPa) in comparison with material in as-cast state (853.22 MPa), while the yield limits ( $\sigma_{YS}$ ) for both, as-cast and cold-rolled materials, have not too different values (450 – 500 MPa) in comparison with the recrystallized material (732.02 MPa). In case of as-cast material the elongation to fracture ( $\epsilon_f$ ) is double ( $\sim 14\%$ ) that in the case of cold-rolled and recrystallized materials ( $\sim 7\%$ ;  $\sim 5.74\%$ ) [7-8].

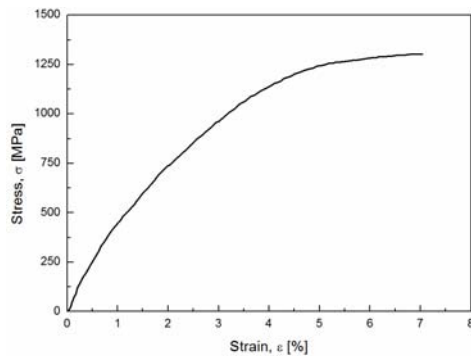


Fig.3. Strain-stress curve for Ti-Ta-Zr alloy cold-rolled condition

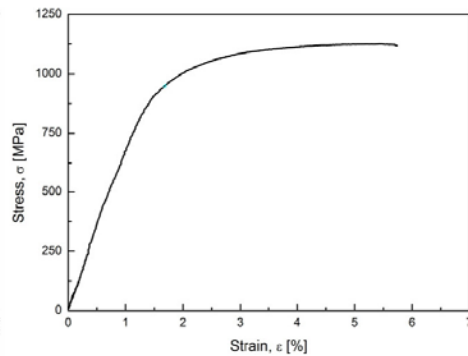


Fig.4. Strain-stress curve for Ti-Ta-Zr alloy recrystallized condition

Tablelul 1

**Comparative analysis of mechanical properties for Ti-Ta-Zr alloy.**

	As-cast	Cold-rolled	Recrystallized
Yield strength ( $\sigma_{YS}$ )	512.37 MPa	448.12 MPa	732.02 MPa
Ultimate tensile strength ( $\sigma_{UTS}$ )	853.22 MPa	1303.84 MPa	1125.33 MPa
Elongation to fracture ( $\epsilon_f$ )	14 %	7 %	5.74 %
Elastic modulus (E)	53.8 GPa	45.7 GPa	62.69 GPa

The most important characteristic, the elastic modulus, has a minimum value of about 45.70 GP in the case of cold-rolled condition, which can lead to the conclusion that in the case of orthopaedic implants the most suitable condition is the cold-rolled, due to the fact that the elastic modulus is very close to human bone (30 - 40 GPa) (see fig.5.).

The main phenomenon which accompanies cold-rolling process is strain hardening. A particular importance is the influence of strain hardening on material plasticity because to a certain degree of deformation plasticity decreases substantially, so that further processing by cold-rolling is no longer possible, taking into consideration the possibility to appear cracks.

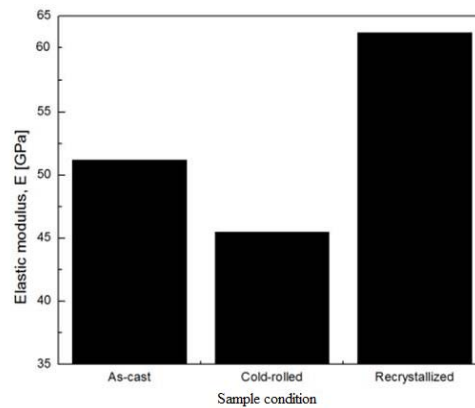


Fig. 5. Elastic modulus for the Ti-Ta-Zr alloy in as-cast, cold-rolled and recrystallized conditions

### 3.4. Fracture surfaces analysis

Based on SEM fractographys, presented in fig. 6, 7 and fig. 8, were observed the following fracture mechanisms.

The fracture predominant mechanism for as-cast samples is fracture by ductile void growth (fig. 6.);

The fracture predominant mechanisms for cold-rolled material are:

- Rupturing of atomic bonds by tensile stresses (cleavage fracture) (fig. 7.);
- Fracture by plastic void growth [4];

The fracture predominant mechanisms for recrystallized samples are:

- Mixing of mixing of plastic flow (fig.8.);
- Fracture by ductile void growth;

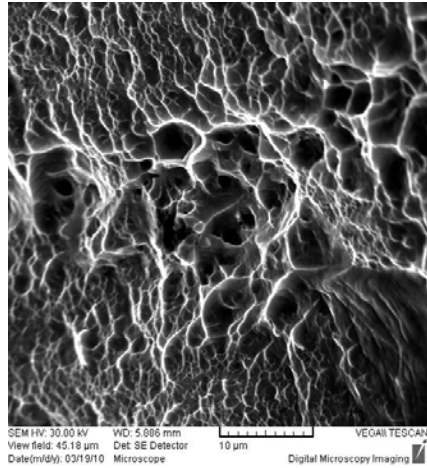


Fig. 6. SEM fractography of Ti-Ta-Zr alloy in as-cast condition

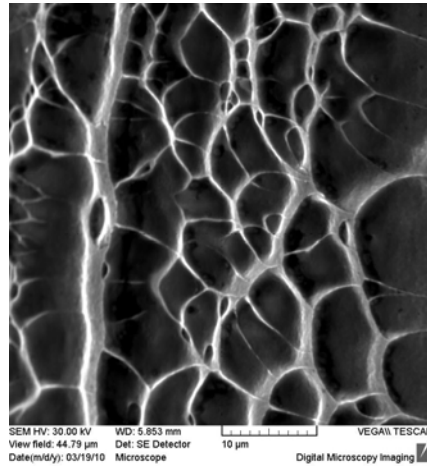


Fig. 7. SEM fractography of Ti-Ta-Zr alloy in cold-rolled condition

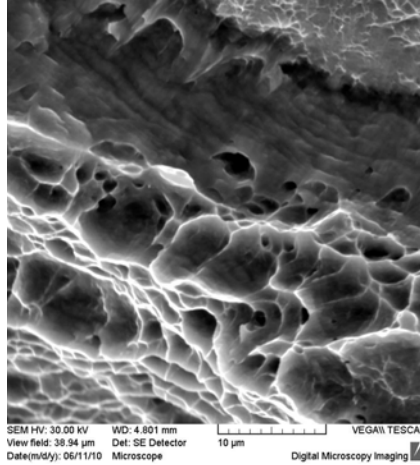


Fig. 8. SEM fractography of Ti-Ta-Zr alloy in recrystallized condition

#### 4. Conclusions

From all performed experiments data concerning changes in Ti-Ta-Zr alloy mechanical properties for as-cast, cold-rolled and recrystallized conditions were obtained.

The ultimate tensile strength ( $\sigma_{UTS}$ ) of alloy cold-rolled and recrystallized conditions have higher values then in the case of alloy as-cast condition. The cold-rolled alloy exhibits an increase in mechanical properties due to strain-hardening. The yield strength ( $\sigma_{YS}$ ) of as- cast and cold – rolled Ti-Ta-Zr alloy exhibits a decrease, while in recrystallized condition it increases.

Different fracture mechanisms were observed in all cases. The predominant fracture behaviour for alloy in as-cast and recrystallized conditions consists in a mixing of ductile type fracture mechanisms, while in the case of alloy in cold-rolled condition a brittle fracture mechanism can be observed.

As a general remark, one can say that from biological point of view (low elastic modulus requirement) the Ti-Ta-Zr alloy in cold-rolled condition exhibit a most suitable mechanical properties association (high mechanical characteristics and low elastic modulus).

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