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MICROSTRUCTURE AND MECHANICAL PROPERTIES OF MEDICAL IMPLANT MADE FROM TITANIUM ALLOYS

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***Abstract:** This paper presents a new method that has been proposed and developed at the Technical University of Cluj-Napoca (Romania) for estimating the durability of the customized implants made by Selective Laser Melting (SLM) process from titanium alloys. Commercially pure (CP) titanium and titanium alloys have wide range of applications in aerospace, energy, chemical and automotive industries. Some of the titanium alloys are excellent materials for biomedical use, especially as orthopedic and dental alloys. The replacement of the human bones is a complex and dynamic field of application. Engineers and orthopedics combine to make a person's life as normal and painless. The most important characteristic features of the biomedical titanium and its alloys are high strength, low density, excellent corrosion resistance and the best biocompatibility among metallic materials.*

***Key words:** implants, titanium alloys, biomedical materials, microstructure, mechanical properties.*

1. INTRODUCTION

The aim of Rapid Prototyping (RP) technologies is the production of parts directly out of CAD data within a short time and without the use of any tools or cutting devices. These generic processes allow the manufacturing of extremely complex structures with almost no restriction in freedom of design. The material properties, however, cannot be compared with the manufacturing accuracy, mechanical properties and surface structure achieved by conventional cutting processes [1]. SLM is a layer-wise material addition technique that allows generating complex 3D parts by selectively melting successive layers of metal powder on top of each other, using the thermal energy supplied by a focused and computer controlled laser beam. Medical and dental applications are very suitable to be produced by SLM due to their complex geometry, strong individualization and high aggregate price. Moreover, the manufacturing of multiple unique parts in a single production run enables mass customization [2]. Therefore, the study of the microstructure and mechanical properties of such new materials produced by a new technique is crucial. Furthermore, the

biocompatibility of implants strongly depends on the surface chemistry and topography [1].

2. SLM FOR BIOMEDICAL USE

Biocompatibility may be defined as the acceptance of the implant material by the surrounding tissues without any adverse response from the body and vice versa. Therefore, a biocompatible implant material should be nontoxic, no carcinogenic, with little or no foreign body reaction and be chemically stable or corrosion resistant.

In light of this, some important applications of biomaterials include (1) orthopedics, (2) cardiovascular systems, (3) ophthalmic, (4) dental applications, (5) wound healing and (6) drug delivery systems [3].

One determining factor for the success of new alloys as biomaterials is their corrosion behavior. Therefore, the in vitro evaluation of their corrosion parameters is one of the first steps in the development of new biomaterials.

However, the corrosion of the metallic implants is critical because it could affect negatively the biocompatibility and the mechanical integrity. Titanium and its alloys are widely used as implant materials for failed

hard tissue because of excellent corrosion resistance and good compatibility with bone[4].

Titanium alloys have been clinically applied since the 1970s when surgical implants were made with the high-strength Ti-6Al-4V alloy. The Ti-6Al-7Nb alloy was introduced into clinical use in the mid 1980s as a substitute for Ti-6Al-4V, since niobium is more biocompatible and cheaper than vanadium. The vanadium oxide VO_2 , generated by passivation of the metal surface, is thermodynamically unstable and some vanadium could be released in human body and cause toxic effects [5]. The corrosion resistance of the three electrodes was in the order of $Ti > Ti-6Al-7Nb > Ti-6Al-4V$ therefore Ti-6Al-7Nb was developed [4]. However, critical problems caused by the mismatch of Young's modulus between the implant (110 GPa for Ti) and bone (12-23Gpa) are still unsolved [6]. The low elastic Young's modulus of porous titanium alloys are expected to reduce the amount of stress-shielding at the bone where the metallic part is implanted [7].

Biocomposite materials have been developed in order to combine bioactivity of ceramics and mechanical properties of metals. Hydroxyapatite (HA) is known for its weakness and brittleness but has an excellent biocompatibility and is a bioactive material. When HA is added to titanium, an improvement of the biomaterial chemical properties occurs [8].

3. MAIN CHARACTERISTICS OF THE PART PRODUCED BY SLM

The main characteristics of the part produced by SLM are (1) density issue, (2) surface quality, (3) mechanical properties, (4) microstructure, (5) residual stresses [9].

The objective in SLM is often to obtain 100% dense parts. This goal, however, is difficult to achieve since there is no mechanical pressure, as in moulding processes, SLM being only characterized by temperature effects, gravity and capillary forces. Moreover, gas bubbles can be entrapped in the material during the solidification due to various causes such as decrease in the solubility of the dissolved elements in the melt pool.

During solidification, an insufficient surface quality can cause low density as well. High

roughness peaks and valleys that are formed after each layer can avoid the coater to deposit a homogenous powder layer. Moreover, the laser energy may be not enough to melt the new layer completely since the depth of the powder in some regions will be thicker.

At sufficiently low scan speeds, the relative density is almost independent of the layer thickness, and a maximum of 99% relative density is achievable.

At higher scan speed values, a higher layer thickness results in less density. However, the layer thickness can be increased if the scan speed is sufficiently lowered to achieve the same density values [9].

Four main process parameters are selected for experimentation laser power, layer thickness, scan speed and hatching space. These factors determine the energy supplied by the laser beam to a volumetric unit of powder material. The relatively high surface roughness of SLM parts could be important for medical applications. The surface roughness depends on many factors: material, powder particle size, layer thickness, laser and scan parameters, scan strategy and surface post-treatment [2].

The resulting heat transfer and fluid flow affect the size and shape of the melt pool, the cooling rate, and the transformation reactions in the melt pool and heat-affected zone. Since the material properties such as yield strength, elongation, ductility and hardness are highly affected by the microstructure features, the mechanical properties obtained with SLM might be different from the properties of bulk materials produced by conventional production techniques [9].

Due to the high thermal energy which is required to promote all steps of material re-arrangement and densification during sintering within an extremely short period of time, thermal gradients exist in the densified structure. Thermal stresses and corresponding residual stresses induce warping and/or cracking of the object. Warping normally leads to tolerance losses but cracking results in a more detrimental loss of quality [10].

The process is carried out in a closed chamber continuously filled with argon because of the high reactivity of titanium to interstitial

elements such as oxygen, nitrogen, carbon and hydrogen [11].

4. SAMPLES MANUFACTURED BY SLM AND PARAMETER ANALYSIS

The experiment consisted in manufacturing samples with different holes on the MCP Realizer SLM 250 equipment installed in the RP laboratory of the Technical University of Cluj-Napoca. The samples were produced from pure Ti and Ti-6Al-7Nb alloy, as well as the following new biocompatible materials: Ti-6Al-7Nb+2%HA and Ti-6Al-7Nb+5%HA.

The 3D models of the samples elaborated with SolidWorks have 22mm length and 3.5mm diameter. The samples are penetrated by circular holes with the diameter of 1 mm or square holes having the edge length of 1mm and 0.5 mm. In the case of medical implants, such holes provide the space where the bone will grow and will serve to make the connection with the healthy bone. Holes are rotated at different angles with respect to the vertical direction, in order to examine the accuracy of their manufacturing. This distribution of the holes is also useful when evaluating the strength of the aggregate subjected to loads acting along different directions as shown in Figure (1).

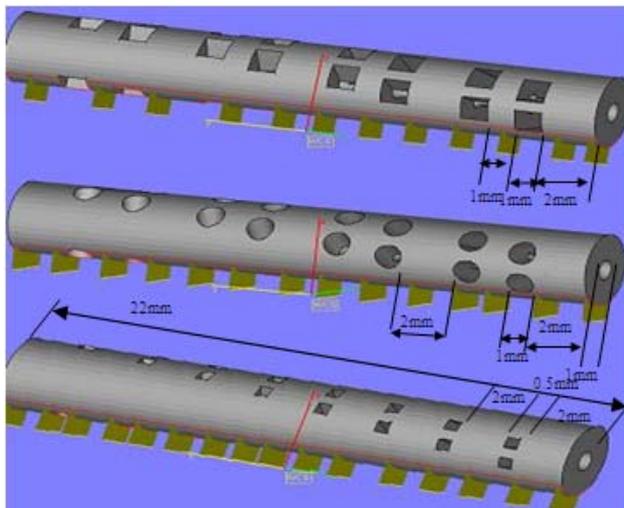


Fig. 1. Medical samples

The technological parameters that have an important influence on the porosity issue of the samples made by SLM were varied as follows:

- Laser power (70 – 160) W

- Scanning speed 0.4 m/s
- Hatching along the X and Y axis directions 0.12 mm
- Spot size 0.15 mm
- Platform pre-heated at 248°C.

These parameters affect the specifications and accuracy of samples. The chart in Figure (2) presents statistical data referring to the average lengths for different types of materials and levels of the laser energy used to produce the samples.

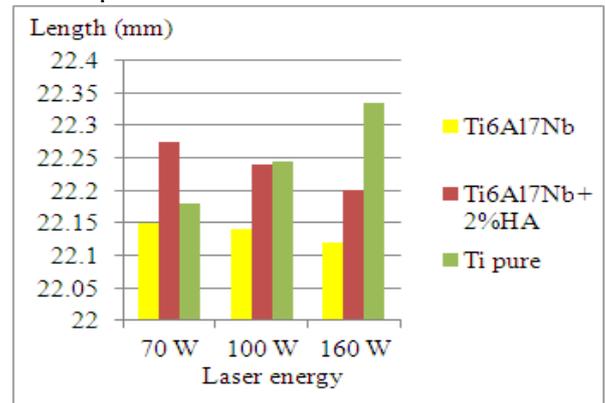


Fig. 2. Mean length of the sample

From the chart above we observe that in the case of pure Ti increasing energy leads to the increase of the length from the design specification. In contrast, when using the Ti alloy, we notice the opposite trend: increasing the energy level leads to the decrease of the length, but this dimension remains closer to the design specification.

Therefore, we can calculate the amount of elongation that must be taken into consideration when designing products of the same type of material and energy as shown in Figure (3).

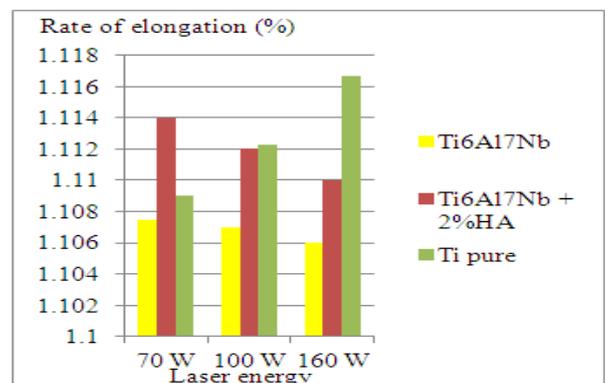


Fig. 3. Rate of elongation

The surface roughness is one of the most important features required in the

manufacturing of medical application parts. So, we made tests in the laboratory of the Technical College in Cluj-Napoca to determine the average roughness depending on the type of material and energy level used in the production of samples using the measuring device shown in Figure (4).



Fig. 4. Device for measuring the surface roughness

The results of the roughness measurements are shown in Figure (5). We notice that increasing the energy level when processing pure Ti leads to the increase of the roughness. In the case of the Ti-6Al-7Nb alloy, the maximum roughness corresponds to the energy level of 100W. The same energy level produces a minimum value of the roughness in the case of the Ti-6Al-7Nb +2% HA.

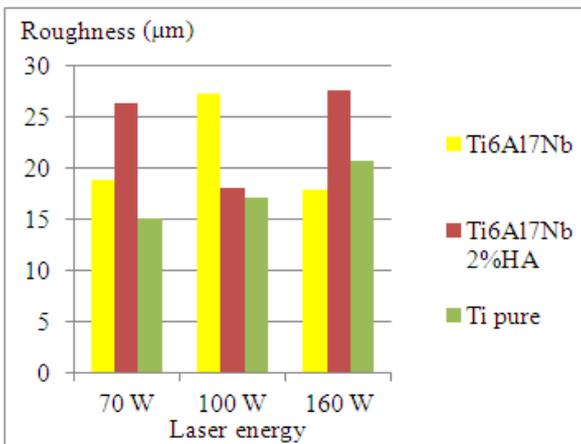


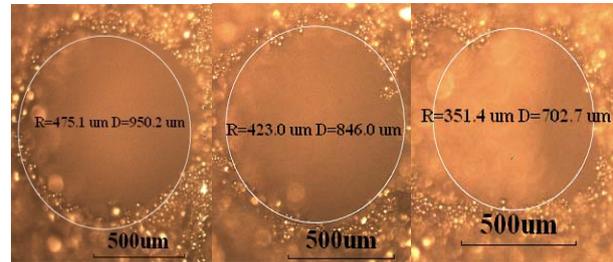
Fig. 5. Roughness chart

5. OPTICAL MICROSCOPY ANALYSIS

After conducting the tests of surface roughness and length measurement, we examined the samples by optical microscopy using the equipment available in the laboratory of the Technical College in Cluj-Napoca.

The best accuracy was obtained for the circular holes of the samples made from pure titanium when using 70W laser power. We may

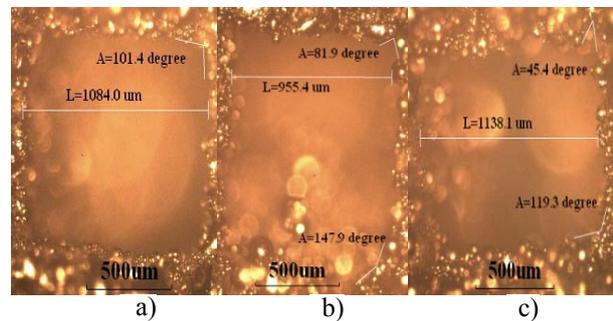
notice from Figure (6) that the accuracy of the circular holes decreases for higher levels of the laser power in the case of the pure titanium.



a) pure Ti 1mm diameter section at 70W
 b) pure Ti 1mm diameter section at 100W
 c) pure Ti 1mm diameter section at 160W

Fig. 6. Pure Ti 1mm diameter section different energy

Figure (7) shows the influence of the compound complexity on the accuracy of the square holes processed at the same level of the laser power (70W). We notice that the best accuracy is obtained in the case of pure titanium and the accuracy decreases when the complexity of the compound increases.



a) Ti pure 1mm side 70W
 b) Ti-6Al-7Nb 1mm side 70W
 c) Ti-6Al-7Nb 1mm side & 2% HA 70W

Fig. 7. Different Ti alloy at 70W

6. FINITE ELEMENT ANALYSIS FOR TESTING THE DURABILITY OF AN IMPLANT

This research was focused on studying the durability of the samples manufactured on the MCP machine from titanium, titanium alloy structures Ti6Al7Nb and Ti6Al7Nb with 2% and 5% hydroxyapatite (HA). These alloys are specially designed for medical applications such as prosthesis and implants.

Several simulations were performed using the Pro MECHANICA program of the PTC software company.

We designed several 3D models in the SolidWorks CAD program, models that have rectangular section holes of 1mm and 0.5mm edge size distributed along all directions.

The mechanical and physical characteristics of the materials (density, tensile strength, Young's modulus and Poisson's ratio) were obtained from the Rapid Prototyping laboratory of the Technical University of Cluj-Napoca – see Table (1).

Table 1
Mechanical and physical characteristics of Ti6Al7Nb alloy

Ti6Al7Nb	Density (g/cm^3)	Tensile ultimate stress (MPa)	Young's modulus (GPa)	Poisson's ratio
70 w	3.8	130	35	0.26
100 w	4.35	400	82	0.35
160 w	4.4	500	90	0.37

For FEA, the 3D model was considered fixed up to middle and the next half of the sample was subjected to a load of 40N, as show in Figure (9).

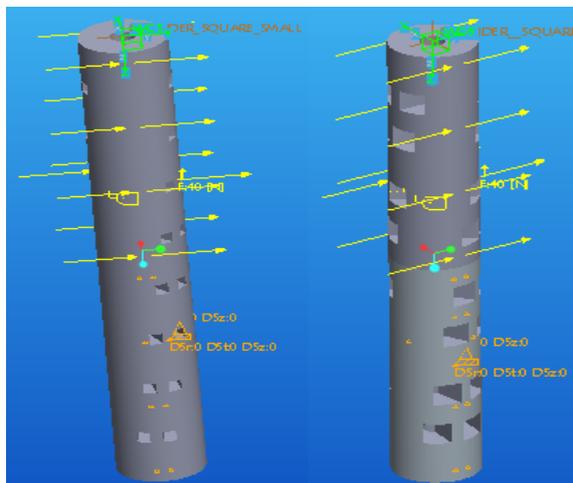
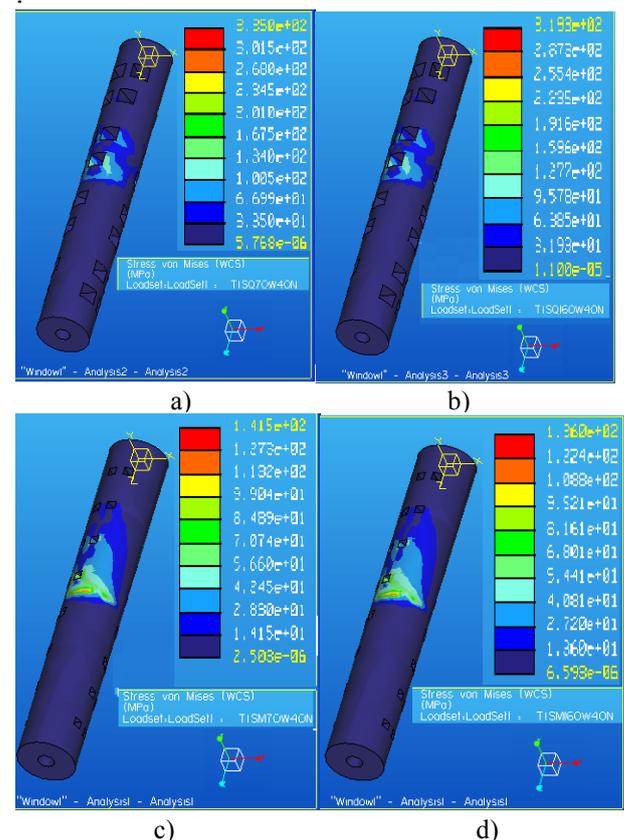


Fig. 9. Constraints and loads applied to the samples.

Figure 10 shows the distribution of the von Mises equivalent stress corresponding to the Ti6Al7Nb samples with different hole sizes manufactured at different levels of the laser power

In the case of the samples manufactured at 70W the FE analysis shows stresses which are over and close to 130MPa (tensile ultimate

stress – see Table 1). More precisely, the samples with holes having 1mm and 0.5mm edge size have 301.5MPa and 127.3MPa, respectively. In the case of the samples manufactured at 160W, the maximum von Mises stresses are under the ultimate stress 500MPa – see also Table 1 (287.3MPa and 122.4MPa for the holes with the edge size of 1mm and 0.5mm, respectively).



a) Ti-6Al-7Nb 1mm side 70W;
b) Ti-6Al-7Nb 1mm side 160W;
c) Ti-6Al-7Nb 0.5mm side 70W;
d) Ti-6Al-7Nb 0.5mm side 160W

Fig. 10. Stress von Mises

It can be observed that the size and shape of holes influences the stress levels. In general, the samples having 0.5mm holes have a superior strength and their use is recommended for producing implants.

7. ACKNOWLEDGEMENTS

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8. CONCLUSIONS

The addition of hydroxyapatite (HA) increases the difficulty of getting a homogeneous structure of the composite, making it difficult to remove the sample from the working plate.

The increased complexity of compounds also increases the difficulty of manufacturing and decreases the quality of the parts.

For the same constraints and external loads, an increased size of the holes will lead to an increased level of the stresses for samples manufactured using the same laser power.

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MICROSTRUCTURA ȘI PROPRIETĂȚILE MECANICE DE IMPLANT MEDICAL EFECTUAT DIN ALIAJE DE TITAN

Rezumat: Această lucrare prezintă o nouă metodă care a fost propusă și dezvoltată la Universitatea Tehnică din Cluj-Napoca (România), pentru estimarea durabilității implanturilor personalizate, fabricate prin procedeul de Topire Selectivă cu Laser (SLM), prelucrate din aliaje de titaniu. Materialele comerciale ca și titanul pur și aliajele din titaniu, au o gamă largă de aplicații de la industria aerospațială, în domeniul energetic, în domeniul chimic și industria constructoare de mașini. Unele aliaje din titaniu sunt excelente materiale pentru utilizarea biomedicală, în special ca aliaje folosite în domeniul ortopedic și dentar. Înlocuirea oaselor umane este un domeniu de aplicație complex și dinamic. Împreună atât inginerii cât și medicii ortopezi fac posibil faptul că viața persoanei să fie normală și nedureroasă. Cea mai importantă trăsătură caracteristică a titaniului biomedical și a aliajelor este duritatea, densitatea mică, excelentă rezistență la coroziune și o bună compatibilitate printre materialele metalice.

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