Selective Laser Sintering of Ti Alloy Powders for Hip Implants

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Abstract. Selective Laser Sintering, SLS, is a rapid prototyping technique, based on additive manufacturing. It enables fast, flexible, and efficient manufacturing of complex parts, customized according to the requirements of users. Research results on SLS additive manufacturing technique and its specific materials, like Ti alloy powders (Ti-6Al-4V) for hip implants are presented in this paper. The research is on various parameters values for the SLS process (laser power, focal diameter of the laser beam, number and direction of laser beam passes) and their influence on mechanical characteristics (strength resistance and fracture surface) of the sintered samples. Some metallographic tests and the electron microscopy evidence the influence of laser spot direction on the sintering process of Ti alloy powders. The scope of this paper is to study the various mechanical and morphological characteristics of Ti-6Al-4V selective laser sintered material used for hip implant. There is the need for this research to avoid accidentally break of implants and mixing the powders with human tissues.

Keywords: selective laser sintering · Ti powders · strength · hip implants

1 Introduction

Selective Laser Sintering (SLS) is already known as a technique that provides the fastest way from product idea to market launch. Innovative companies around the world use this additive manufacturing technique for fast, flexible, and minimal production costs when obtaining medical implants, directly from an electronic format, at any stage of the manufacturing cycle. Implants must have properties close to the bones and/or tissues they replace. One of the most used materials in implants are Ti alloys, such as Ti-6Al-4V (Ti64) [1, 2].

As well known, widespread and important use of titanium and its alloys is that of medical nature, in implant surgery – as material of prostheses and implantable elements. This is due to the biocompatibility properties of Ti-6Al-4V (Ti64) type alloys, raw material as powder that can be selectively sintered by laser.

Laser selective sintering techniques allow the generation of multilayered materials of 3D complex parts by consolidating successive layers of material in the initial form of powder, using the thermal energy provided by a computer-controlled and concentrated

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Fig. 1. The SLS technique sequences

laser beam. The main mechanisms underlying powder consolidation are: solid phase sintering, liquid phase sintering, partial or complete melting [3].

The quality of part produced by this technology depends a lot on the quality of each layer. Identifying the optimal parameters values of power and speed of the scanning laser beam is a challenge, as their values di greatly influence the porosity, hardness, and mechanical properties of the sintered part.

The process of rapid manufacturing and/or prototyping by selective laser sintering is based on the materialization of a virtual 3D model (built in a CAD system) by adding successively molten layers (additive technology). A focused laser beam ballads point by point the entire area of the surface, sintering the fine layer of powder deposited on a work platform. For each part, it is necessary to build supports (support structures) that allow the part to be detached from the machine building table (Fig. 1).

Since titanium parts have a high production cost and because titanium (with a high affinity for oxygen) is difficult to pour and process, research on SLS additive manufacturing technique and its specific materials, like Ti alloy powders is worth to be done [4]. Some relevant results of our research are further presented by this paper.

2 Materials and Methods

Lately, special attention has been paid to the assimilation of titanium and titanium alloys that are usually used to obtain parts with special requirements.

Sintering is a heating operation during which complete powder consolidation occurs by diffusion welding processes [5, 6]. The main technological parameters of the sintering operation are: temperature and time. The heating for sintering is carried out at the temperature of 2/3–4/5 of the melting temperature of the heaviest fusible component in the powder mixture. To prevent internal oxidation, the sintering operation is performed in controlled atmosphere (reducing or neutral) or in vacuum [7].

While sintering, there are three distinct stages, as follows (see Fig. 2):

A - initial stage (point bridges, unmodified porosity, distinct particles);
B - intermediate stage (bridges on large contact surfaces, reduction of pore, new recrystallized grains);
C - final stage (loss of individuality of powder particles, consolidated material, increase in recrystallized grains).

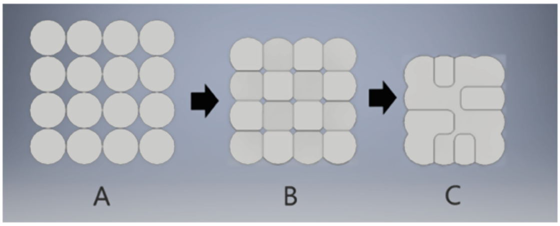
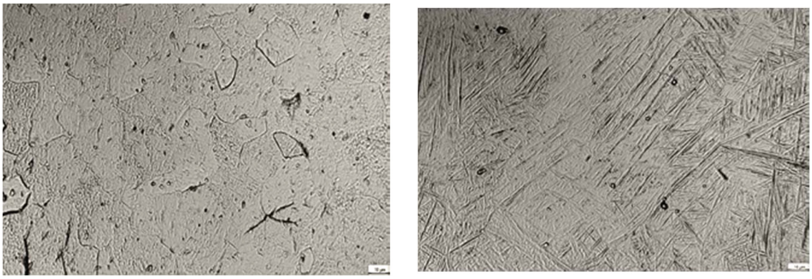


Fig. 2. Stages of particle melting during sintering



a. probe obtained by classical processes (casting, lamination)

b. the sample made of titanium alloy EOS Ti64, obtained by the SLS process

Fig. 3. Crystallographic structure of Ti-6Al-4V alloy (800x)

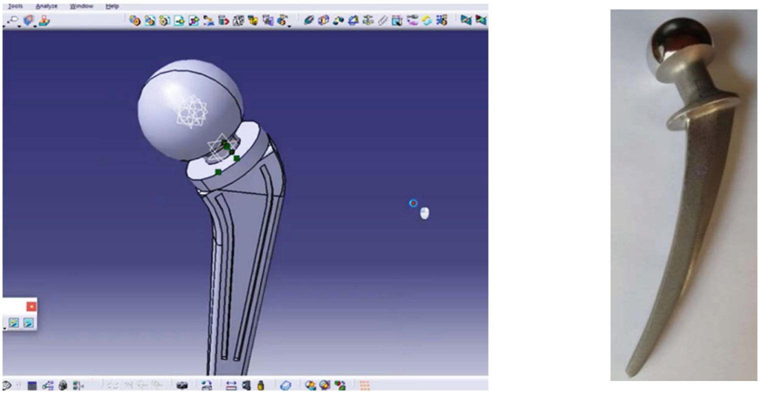
Comparative research on the structure of Ti alloys, obtained from metal powders, has been carried out. The study of the crystallographic structure led to the obtaining of the images as shown in Fig. 3.

- Ti-6Al-4V cast bar (Fig. 3a), material used by manufacturers of medical devices and hip prostheses - there are more pores than in parts obtained by SLS process;
- Ti-6Al-4V sintered sample (Fig. 3b) obtained by the SLS process - the sintered structure has crystals oriented along preferential directions that coincide with the direction of laser beam passes so that the structure thus obtained is a denser one.

This metallographic investigation was done on the optical microscope Carl Zeiss AX10. As there were determined only the crystallographic structure (no distances and geometries) there was no need for software. There was only the need for well known specific procedures to obtain the metallographic samples.

The high-tech EOSINT M 270 Titanium Version SLS equipment has a high-performance laser system and optics with excellent variable focus, enabling to obtain high-resolution parts. The variable focusing diameter leads to increased productivity by decreasing the processing time. Controlled atmosphere of the working enceinte enables to use a wide range of materials. The equipment works with STL files.

The example of modeling and prototyping of a hip implant is shown in Fig. 4. The nominal thickness of the powder layer was 30 μm.



a. 3D model of hip implant [8] b.hip implant prototype

Fig. 4. Hip implant made of Ti-6Al-4V

Fig. 5. Products obtained by TEST-type SLS for mechanical tests

3 Experiments and Results

To carry out the tests, there were chosen samples (obtained by SLS technique) with different geometries like: tubes, cylindrical bars, cubes, these being configured with internal structures of porous type. In obtaining them, the same process conditions were used so that to enable the most accurate comparisons of the obtained results [9] (Fig. 5).

Mechanical tests were performed on the test equipment HOUNSFIELD (600 Series Servo-hydraulic Test Machines). As reference for the cylindrical samples it was used the ASTM E8/E8M standard [10]. In Fig. 6 it is shown the capture of the sample mounted for the tensile test process. Figure 7 evidence the fracture surface of the test bar, the highlighted zone of the fracture surface points out a fragile breakage.

The aspect of tensile breaking surface and its sintered material the choice of an inappropriate SLS processing program, with a reduced number of laser beam passes being incapable of achieving uniform melting of the entire mass of the powdered material.

This is a primary disadvantage in the case of making implantable products for orthopedic surgery, because if an accident resulting in breaking of the implant occurs, danger of leakage and mixing of non-sintered or partially sintered powders with the human

a. test-piece during the tensile test   b. test-piece after fracture

Fig. 6. Samples on tensile strength test equipment

Fig. 7. Fracture surface aspect

tissue with which they come into contact exists. Although the powders are made of biocompatible materials this event should be avoided.

The tensile force – strain diagram, for one of the tested samples is shown in Fig. 8. The Ti-6Al-4V sample had a tensile strength of 4870 N, which corresponded to a deformation of 1.85 mm (at fracture).

Hardness tests on SLS samples were further perfumed. In the case of the analyzed metal powder, the initial values in bulletins of the powder manufacturer were compared to the values determined by our own tests with Rockwell 600A bench durometer (EN-ISO 6508 and ASTM E-18). The obtained values, of 34–35 HRC, are according to the medical requirements.

The determination of the mechanical characteristics of the test-pieces was performed considering the state of stress (and deformation) to which the hip implant is subjected, in “exploitation” - see Fig. 9. It was considered a safety coefficient of 3.2 for a person of 80 kg weight. The simulation was done in SimScale software.

For a more complete characterization of the parts SLS from Ti-6Al-4V (metal powders used in medicine), the electron microscopy process was also used. Also, by

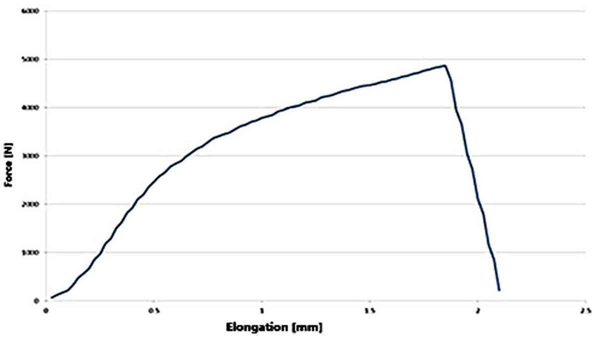


Fig. 8. Tensile force – strain diagram for Ti-6Al-4V sample

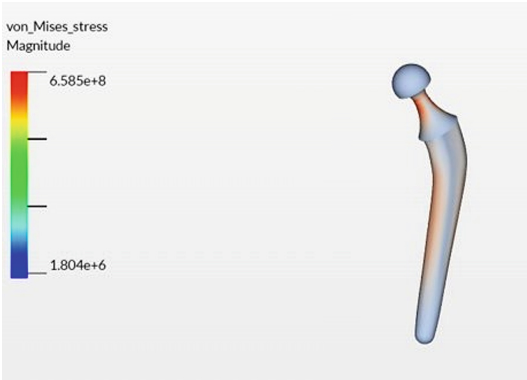


Fig. 9. Stress simulation for hip implant

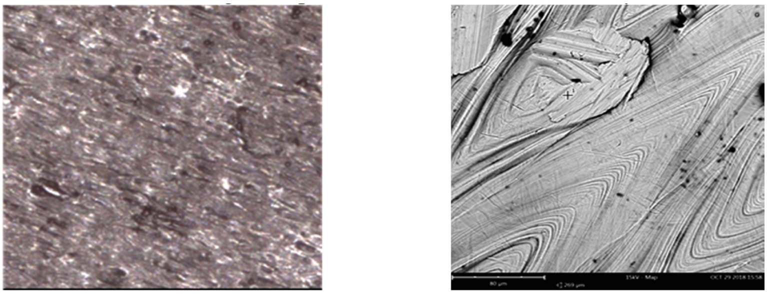
using the microprobe, a qualitative type of investigation was carried out aimed at better understanding the complex phenomena generated through selective laser sintering process.

When making the investigated sample, the process parameters were modified to obtain different structures of material specific to hip prostheses. By comparing the obtained structures correlated with their mechanical properties, it is aimed at obtaining optimal properties of the implant, specific to the human bone.

The optical images of the sample in Fig. 10 (obtained by microscope Carl Zeiss AX10 (a.) and Schottky Field Emission Scanning Electron Microscope, SU5000 FE-SEM (b.)), show Ti-6al-4V structure obtained by the laser sintering process. Thus, the directions of passes of the laser spot that generates traces/waves in the material, according to the direction of laser beam motion, are identifiable.

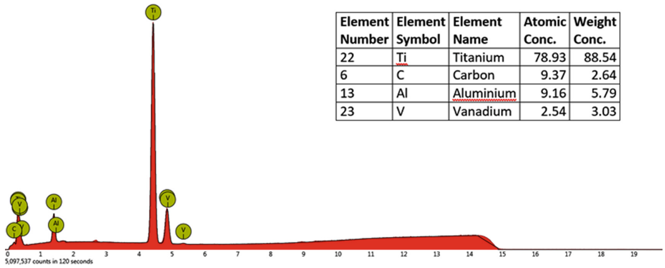
In the case of obtaining close information from the investigation of different spots, the conclusion was that of good repeatability and thus of uniformity of the material.

Further investigation with SU5000 FE-SEM, using microprobe was done in order to determine chemical composition of the SLS sample's material (by dedicated equipment software). The investigated zone is that of a particle with a degree of spherical degree



(a) (b)

Fig. 10. **a** Optical microscopy sample magnification X50. **b** Electronic microscopy sample magnification according to FOV standard: 269 μm, Mode: 15kV - Map.



Element Number	Element Symbol	Element Name	Atomic Conc.	Weight Conc.
22	Ti	Titanium	78.93	88.54
6	C	Carbon	9.37	2.64
13	Al	Aluminium	9.16	5.79
23	V	Vanadium	2.54	3.03

Fig. 11. Chemical distribution of elements obtained using the micro-probe

close to 1. The presence of these particles (Ti, C, Al, V) is evenly distributed, both dimensionally and structurally – see Fig. 11. The average size is around 30 μm, that falls within the manufacturer's prescriptions.

More of it, the spectrometry analysis evidences that chemical elements were similar to those investigated in various zones of the sample, thus highlighting a constancy of material and process.

There was also noticed the existence of particles completely sintered with splashes of material resulting during the laser sintering process. The directions of passes of the laser beam spot and the uniformly sintered material are identifiable.

4 Conclusion

Research has been carried out on the selective laser sintering technique of Ti-based powders (Ti-6Al-4V), commonly used to obtain (hip) implants.

Analysis of the properties of the samples in relation to the laser power (input energy) showed that the properties of samples processed by SLS additive technology increased with increased exposure energy. In addition, experiments have shown that the tensile properties of sintered samples are dependent on the direction of part build-up.

The rapid prototyping of powders from biocompatible metal biomaterials opens new paths in prosthetics and orthopedics, allowing to obtain conceptual models and functional prototypes.

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