Physico-Mechanical Properties Characterization of the Parts from PA 2200 Manufactured by Selective Laser Sintering Technology

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Abstract: This paper presents researches in the field of biocompatible materials manufactured by Selective Laser Sintering method (SLS). In the last years, the field of biocompatible materials has developed increasingly more because of the advantages they offers. It was made processability tests and physical-mechanical properties of the parts manufactured by Selective Laser Sintering from biocompatible powder PA 2200.

Key words: SLS, biocompatible material, PA 2200, processability tests.

1. INTRODUCTION

Biomaterials represents "any substance or combination of substances of natural or synthetic origin which may be used in a well-defined period of time, as a whole or as a part of a system that treats hurries, or replace a tissue, organ or function of the human body". (Williams 1992).

Performance of biomaterials depends on the physicochemical properties, design, biocompatibility, surgical technique and patient health.

Rapid prototyping (RP) is a key group of prototyping technologies with the ability to rapidly fabricate complex three-dimensional (3-D) physical structures, (Chua, 1997).

Rapid Prototyping technologies are based on the use of a model data created in a computer aided design programme (CAD) to generate 3D physical objects. The virtual model is exported in the computer of the RP machine in *.STL format file, as a mathematical slicing model.

The RP systems then utilise the slice data to replicate or reconstruct a physical object through layer by layer manufacturing whereby solid material layers are fabricated, stacked vertically and bonded to the previous layer to give rise to the physical object, (Tan, 2003).

There are many Rapid Prototyping technologies, but the use of these techniques depends on which kind of material will be use for the manufacture of the final part.

The working principle of Selective Laser Sintering machine is the same as 3d printing technologies. This powder is brought layer by layer on a carrier mobile platform, which moves down with an equal distance to the thickness of the following sintered layer. Once pulverised on the platform surface, the powder is heated to near the melting point with the help of the laser beam, which drawn the shape of the virtual model. Thus, the component particles are bonded one to others, solidifying into the shaped form drawn by laser beam. A solid layer is obtained, on which powder is spread again and is sintered in the form of a new layer. The previous layer sintered or not, represented an support for the current layer of material (Berce, 2000).

2. MATERIALS AND METHODS

The material use for this research was a polyamide powder, PA 2200, produced by Electro Optical Systems - EOS GmbH, Munich, Germany.

The white powder PA 2200 on the basis of Polyamide 12 serves a wide variety of applications with its very well-balanced property profile.

The PA 12 powder used for the purpose of this research has a specified average particle size 50 µm and a density of the powder 930 g/cm³.

Laser-sintered parts made from PA 2200 possess excellent material properties, such as good chemical resistance, excellent long-term constant behaviour, high selectivity and detail resolution, various finishing possibilities (e.g. metallisation, stove enamelling, vibratory grinding, tub colouring, bonding, powder coating, flocking),
bio compatible according to EN ISO 10993-1 and USP/level VI/121, (Electro Optical Systems & Solutions).

Typical applications of the material are fully functional plastic parts of highest quality. Due to the excellent mechanical properties the material is often used to substitute typical injection moulding plastics. The biocompatibility allows its use e.g. for prostheses, the high abrasion resistance allows the realisation of movable part connections, (Electro Optical Systems & Solutions).

The thermal proprieties of polyamide PA 2200 used in this research are presented in Table 1.

Table 1. Thermal proprieties of PA 2200, (Electro Optical Systems & Solutions).

<table>
<thead>
<tr>
<th>Thermal properties</th>
<th>Value</th>
<th>Unit</th>
<th>Test Standard</th>
</tr>
</thead>
<tbody>
<tr>
<td>Melting temperature (10°C/min)</td>
<td>176</td>
<td>°C</td>
<td>ISO 11357-1/-3</td>
</tr>
<tr>
<td>Vicat softening temperature A</td>
<td>181</td>
<td>°C</td>
<td>ISO 306</td>
</tr>
<tr>
<td>Vicat softening temperature (50°C/h 50N)</td>
<td>183</td>
<td>°C</td>
<td>ISO 306</td>
</tr>
</tbody>
</table>

The PA 2200 powder was processed on a commercial SLS system, Sinterstation 2000 (DTM Products Inc., USA), to produce test specimens. Except for changing the process parameters on the operating software of the SLS system, no modifications were made on the SLS system for processing the new materials.

2.1 Processability tests

Given that a new material was processed by selective laser sintering method, for which the working parameters are unknown, the first step was to determine these parameters.

Working temperature is obtained by decreasing the Glazing temperature with 10-12°C. Therefore, in order to determine the working temperature have been performed experimental tests for determining the Glazing temperature.

For the beginning the working powder bed was heated at 183°C temperature, but at this temperature it was observed that the powder melts and after conveyer roll remains a "trace" (Fig. 1).

Fig.1. PA 2200 powder at 183°C temperature

The temperature was decreased at 181°C. In this case, the powder bed has a normal aspect, uniform, without trace. Therefore, it was determined that the Glazing temperature at 181°C and on this basis it was agreed that the working temperature to be 170°C.

In order to verify the working parameters were chosen two test parts with the shape and dimensions suggested in the technical documentation of the machine. The laser power used in the test parts was 4.5 W.

The total time of manufacturing for this work package was 3 hours 47 minutes, including pre-heating time (Warm-up stage) and cooling time (Cool-Down Stage).

A first conclusion that could be observed in the manufacture of the first work package with the chosen parameters is that the temperature was too high because the powder located in the working area is more solidified than the powder located on the supply tanks, fig 2.

Fig.2. Picture inside the machine Sinterstation 2000 after processing the first test pieces from PA 2200 powder

The package has been removed from the machine with the help of a cylinder and was transported on the vibrating table, where has been cleaned the parts.
The resulting pieces were compact, and their geometry was in large part respected, pieces have sharp edges and the thin wall of the piece number 2 is fairly rigid (fig 3, fig 4). It was noted, however, a relatively small deformation of both test parts, a curve form, on their base.

Fig.3. First test part

Fig.4. Second test part

Because curved of the parts could be generated by overheating of the working powder bed to 183°C (when was determined the Glazing temperature) the manufacturing was repeated, with the same working parameters (laser power 4.5 W, the working temperature 170°C).

The pieces produced in the second manufacturing process were good in terms of the geometry, the bending phenomenon has been removed, the measured dimensions matched with the projected dimensions of the parts, fact which means that the working parameters were correctly chosen.

In order to determine the influence of laser power on the geometry and characteristics of the part, were loaded five models of parts which have been processed with different laser powers, from 3.5 W to 5.5 W, with a power increment 0.5 W.

The first part (fig 5) was achieved at 3.5 W. It can be seen a good geometry of the part with straight edges (very sharp) and the thin wall is rigid, without cracks.

At a power of 4W it was observed that the piece has also a good geometry with straight edges and the thin wall is not broken or cracked, but is slightly more rigid than it was in the first part made at 3.5 W laser power.

The part number 3, obtained at 4.5 W, has chamfered edges and the thin wall is more rigid than in the case of part number 2 (fig. 6).

Fig.5. Part from PA 2200 powder produced at 3.5 W

Fig.6. Part from PA 2200 powder produced at 4.5 W

At 5W, it can be seen that the part geometry is not as good, there are no fine details, edges are fillet, but the thin wall is more rigid than in the case of others manufactured parts.

The part made at 5.5 W laser power, has very rigid thin wall, but the details of part (the written) are not clear and the edges are fillet fig 7.

Fig.7. Part from PA 2200 powder produced at 5.5 W

After the manufacture of test parts with different laser powers were observed the following: Once the laser power is higher, the thin walls of the parts become more rigid, but it is losing the clearness of the details.

The use of higher laser power, are suitable for the manufacture of parts with simple geometry with thin walls, which does not require any details.
Because the Rapid Prototyping manufacturing process is recommended in achieving complex parts, for which may not be used a conventional manufacturing technology, it was chosen for manufacturing specimens for testing the physico-mechanical properties at 3.5 W, 4 W, and 4.5 W power, where the detail quality of the tested parts obtained was acceptable. To determine the physical and mechanical properties of the material PA 2200, were made 5 lots of test specimens, according to STAS SR EN ISO 527-4, for each established laser power. In Figure 8 are presented the dimensions and shape of the tensile test specimens.

To determine the elastic strain field of these materials (PA 2200 obtained with laser powers between 3.5 ... 4.5 W), initially for each one, was drawn characteristic curves. From these curves result the elastic zone and can be calculated the elastic modulus in the longitudinal direction (E-modulus).

3. EXPERIMENTAL RESULTS FOR PHYSICO-MECHANICAL PROPERTIES

To obtain the maximum strains values for the elastic zone, a set of specimens made with the three powers of the laser beam was puted to tensile tests, until breaking, for each laser power, was performing five tests.

The tests were performed in the Strength of Materials Laboratory of the Faculty of Mechanical Engineering from Technical University of Cluj-Napoca.

![Graph](image)

### Fig.9. Variation of tensile stress with tensile strain for specimens made at 3.5 W laser power

The details of working parameters, geometrical and mechanical properties of specimens made with different laser power are presented in tables 3, 4 and 5.

### Table 3. The characteristics of specimens made at 3.5 W laser power

<table>
<thead>
<tr>
<th>Characteristics</th>
<th>Specimen</th>
</tr>
</thead>
<tbody>
<tr>
<td>Rate, [mm/min]</td>
<td>1 2 3 4 5</td>
</tr>
<tr>
<td>Width, [mm]</td>
<td>10 10 10 10 10</td>
</tr>
<tr>
<td>Thickness, [mm]</td>
<td>6 6 6 6 6</td>
</tr>
<tr>
<td>Load at break, [N]</td>
<td>1809.40 1629.99 1575.71 1750.42 1748.54</td>
</tr>
<tr>
<td>Tensile stress at break, [MPa]</td>
<td>30.16 27.17 26.26 29.17 29.14</td>
</tr>
<tr>
<td>Tensile strain, [%]</td>
<td>6.21 6.25 4.40 5.90 6.69</td>
</tr>
</tbody>
</table>

In order to determine precise directional deformation was used the INSTRON 3366 biaxial extensometer, whose characteristics are presented in Table 2.

### Table 2. Characteristics of INSTRON 3366 biaxial extensometer

<table>
<thead>
<tr>
<th>Characteristics</th>
<th>Values</th>
</tr>
</thead>
<tbody>
<tr>
<td>Electrical resistance, [Ω]</td>
<td>350</td>
</tr>
<tr>
<td>Sensitivity, [mV/V]</td>
<td>2.5±20%</td>
</tr>
<tr>
<td>Measuring range, [mm]</td>
<td>12.5; 25; 50</td>
</tr>
<tr>
<td>Working Course, [mm]</td>
<td>±2.5</td>
</tr>
<tr>
<td>Specific deformation, A [%]</td>
<td>20;10;5</td>
</tr>
<tr>
<td>Working frequency, [Hz]</td>
<td>75</td>
</tr>
<tr>
<td>Temperature limits, [°C]</td>
<td>-80…+200</td>
</tr>
</tbody>
</table>

![Diagram](image)
For load stress to remain in the elastic range of the material, it was necessary to establish the maximum extension. According to experimental graphics obtained, in the case of specimens made at 3.5 W laser power, this value has been determined for about 0.5 mm.

Table 5. The characteristics of specimens made at 4.5 W laser power

<table>
<thead>
<tr>
<th>Characteristics</th>
<th>Specimen</th>
</tr>
</thead>
<tbody>
<tr>
<td>Rate, [mm/min]</td>
<td>1</td>
</tr>
<tr>
<td>Width, [mm]</td>
<td>10</td>
</tr>
<tr>
<td>Thickness, [mm]</td>
<td>6</td>
</tr>
<tr>
<td>Load at break, [N]</td>
<td>2118.72</td>
</tr>
<tr>
<td>Tensile stress at break, Rm [MPa]</td>
<td>35.31</td>
</tr>
<tr>
<td>Tensile strain, [%]</td>
<td>6.89</td>
</tr>
</tbody>
</table>

The figure 11 shows the linearity variation of tensions on the interval chosen. On this graph were drawn the linear interpolation functions for each curve thus obtaining the values of longitudinal elasticity modulus (E-modulus) in MPa.

It can be observed a small variation of E-modulus from 12.1107 to 14.149 MPa. In the cases of specimens made at 4 W and 4.5 W, the variation of E-modulus can be observed in figures 13 and 14.

Analyzing all 15 samples, it can be observed that the highest value of E-modulus (16.863 MPa) is for the specimen number 5 made at 4 W laser power, and the minimum value (11.121 MPa) is for the specimen number 2 made at 3.5 W laser power.
4. CONCLUSIONS

A biomaterial, polyamide PA 2200 was experimentally processed on a commercial selective laser sintering (SLS) RP system. The SLS technique is highly advantageous as it provides a good control over the microstructure of the parts by adjusting the SLS process parameters.

In order to obtain a good quality of the surfaces, the working parameters were determined for the PA 2200 powder.

Once the laser power is higher, the thin walls of the parts become more rigid, but it is losing the clearness of the details.

The use of higher laser power, are suitable for the manufacture of parts with simple geometry with thin walls, which does not require any details.

To determine the physical and mechanical properties of the material PA 2200, were made 5 lots of test specimens, according to STAS SR EN ISO 527-4.

Was used the INSTRON 3366 biaxial extensometer, in order to determine precise directional deformation.

The maximum extension in the case of specimens made at 3.5 W laser power, has been determined for about 0.5 mm.

The highest value of E-modulus (16,863 MPa) is for the specimen number 5 made at 4 W laser power, and the minimum value (11,121 MPa) is for the specimen number 2 made at 3.5 W laser power.

It may be noted the fact that as the laser power used in manufacturing process of parts is higher; the best mechanical properties can be obtained.

5. ACKNOWLEDGEMENTS

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6. REFERENCES

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