The technique of selective laser sintering (SLS) in the design high-porous ceramic implants

Technika selektywnego spiekania laserowego w projektowaniu implantów z ceramiki wysokoporowej

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Most of the metal and ceramic implants (eg. dental implants, ENT implants) can be performed by SLS technique with a proper initial materials and technological process parameters. Attention should be drawn to the process prerequisites like chemical composition of powder, particle size distribution and shape, the optical properties of the material, its thermal conductivity and the type of protective atmosphere. Spatial model of the implant with a porosity of 80% based on the MatLab program was developed.
KEYWORDS: SLS technique, 3D implant model, ceramic implant, submicrocrystalline sintered corundum

Większość implantów metalowych i ceramicznych (np. implanty stomatologiczne, wszczepy otolaryngologiczne) można wykonać techniką SLS przy odpowiednim doborze materiałów i parametrów technologicznych procesu. Należy zwrócić uwagę na warunki wstępne procesu, takie jak: skład chemiczny proszku, rozkład wielkości ziaren i ich kształt, właściwości optyczne materiału i jego przewodnictwo cieplne oraz rodzaj zastosowanej atmosfery ochronnej. Opracowano model przestrzenny implantu o porowatości 80% w oparciu o program MatLab.
SŁOWA KLUCZOWE: technika SLS, model przestrzenny implantu, implant ceramiczny, submikrokrystaliczny korund spiekany

Due to the aging of the European population, increased number of traffic accidents and lifestyle diseases (including cancer) and limited transplantation possibilities, more and more attention is paid to the possibility of using bioactive ceramic implants received by using new techniques (SLS, SLM, EBM). The Engineering Ceramics in Europe and USA report shows that the market for the implants using the ceramic-ceramic connection has increased rapidly from 2009 to 2012 (approx. 20%). In Lesser Poland (Małopolska) about 7,500 implants (including hip replacements, knee prostheses, spinal stabilizers, plates, screws, bone implants) were used annually. The technique of selective laser sintering is one of the latest techniques for the production of models, prototypes and series of products consisting of merging layers of powder using laser light [1]. To date, high-porous materials were derived by the techniques like chemical foaming, frothing mechanically or by mapping porous matrix, etc.

The technique of selective laser sintering by the preliminary sintered powder in suitable granulation is the most stable method for obtaining high-porous materials at predetermined, stable physical and mechanical properties, not yet used in Poland in relation to the bioceramic materials.

Literature analysis

Selective laser sintering process of different materials is the most commonly used due to their ability to produce complex parts with complex geometry without having to use additional equipment. This technique allows to obtain a product with the physical, chemical and mechanical properties different from the properties of the initial material components in a fast way, with fixed repetition and greater accuracy. From a historical point of view at the beginning of the development of this technology metals and their alloys, then plastics were used. Last decade brought much interest in much more difficult material like ceramics. Analysis of literature leads to the conclusion that the most of the ceramic implants (eg. on the prosthetic eye, dental implants, implants transdermal stabilizers of the spine, hip replacements and knee joint prostheses) [1], can be manufactured by SLS technique with the proper selection of materials and technological parameters of the process.

However, attention should be paid to the prerequisites of the process like: the chemical composition of the powder grain size distribution (below 40 μm) and shape (preferably spheroidal), the optical properties of the material and its thermal conductivity, the type of laser (CO_2, Nd : YAG, Nd : YAG fiber) and the type of protective atmosphere (nitrogen, argon, oxygen, vacuum). Laser sintering techniques can be classified into three categories defining the connection mechanism: solid-state sintering, sintering of the liquid phase and melting-solidification process.

Solid state sintering relates to a thermally active transport of materials by reducing the surface energy of the aggregated particles. This process is free running at 50% of the melting point. In the second case, the sintering temperature is higher, because under the melting temperature a local liquid phase that affects capillary action to the solid particles by combining them together is formed.

Unfavorable phenomena in the process of SLS include increased residual stress of ceramic pieces because of the

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speed of the processes. This is due to scanning speed and distance between the laser spots. The consequence of obtaining extremely high temperatures of SLS process in a very short time is formation of SLS phases being far away from thermodynamic equilibrium. It causes a rapid transition of the high-temperature phase to transition phases which can be also present at room temperature. Atmosphere used in the sintering process (nitrogen, argon, oxygen, vacuum) may affect the chemical reactions with the sintered material. This is particularly important when introducing oxygen or nitrogen, as the partial pressure of oxygen affects the thermodynamic equilibrium of oxidation processes and reduction of sintered material. Tab. I lists types of SLS devices, work condition of devices and types of sintered ceramics published in literature in 2015.

TABLE I. Types of devices SLS, the parameters of work and the types of sintered ceramic materials

Article / Equipment	Working conditions	Material
Rapid Prototyping Journal 21 (2015): pp. 201÷206 [2] LENS	Laser YAG: Nd3+, Power 350 W Laser spot 220 µm Laser scaning 5 µm/sec	Al_2O_3
Applied Surface Science 336 (2015): pp. 59÷66 [3] Solar Laser SystemLQ929	Laser YAG: Nd3+, Laser spot 200 µm Laser scaning 110, 400, 550 µm/sec	MgO MgF_2O_4 $MgAl_2O_4$
Mat.Sci.Eng. A 628 (2015): pp. 188÷197 [4] No data	Laser YAG: Nd3+, Power 100÷400 W Laser scaning 1000–7000 µm/s	$AlSi_{10}Mg$

Spatial model of the implant

Chosen model belongs to the group of triply periodic minimal surface and is described by the equation:

$$k_1 \cdot [(\sin(x) \cdot \sin(y) \cdot \sin(z) + \sin(x) \cdot \cos(y) \cdot \cos(z) + \cos(x) \cdot \sin(y) \cdot \cos(z) + \cos(x) \cdot \cos(y) \cdot \sin(z)] + k_2 \cdot [\cos(4x) + \cos(4y) + \cos(4z)] + k_3 = 0$$

where: x, y, z are versors and k_1, k_2, k_3 are parameters determining the porosity of the unit structure.

Elementary cell of the structure was shown in Fig. 1*a*. The parameters k_1, k_2, k_3 are chosen in such way that the obtained porosity of the structure was Vv = 80%. The percentage was defined as the ratio of the material volume to the total volume of cuboid of the surrounding modeling area (assuming that the area of the modeling will provide a cube with dimensions of 10 x 10 x 10 mm). The modeling process was carried out in the original script MatLab. The three-dimensional mesh as a set of coordinates of the vertices and the set of elements that define the topology of the grid has been exported to *.obj* format. The final model is shown in Fig. 1*b*.

a) *b*)

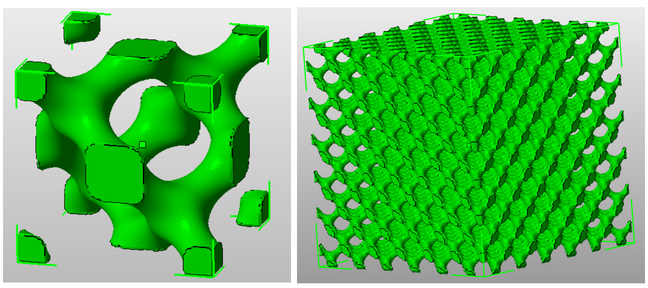

Fig. 1. 3D model of the implant: *a*) unit cell, *b*) model of Vv = 80% porosity

Research material

The research material for receiving the implant was submicrocrystalline sintered corundum (cubitron). It was pretested for physical, chemical, mechanical and biological properties at earlier work [5÷7]. Selected properties of cubitron are summarized in Tab. II.

TABLE II. Selected properties of submicrocrystaline sintered corundum grains (cubitron)

Properties	Results
Density, g/cm^3	3.884
Microhardness, GPa	21.5
Flexural strength, MPa	140
Young modulus, GPa	350
Wettability by FB1 glass	< 35°
Grain granulation, µm	D_{50}40 µm
Specific area S_{BET}, m^2/g	12.8

Conclusions

Selective laser sintering is a modern technique that creates the possibility of obtaining a stable ceramic with a very high porosity, the programmed architecture of pores and interporous connections [8]. On the basis of literature discernment it can be said that the technological parameters of the process of SLS have changed in the following areas: type CO_2 laser, Nd : YAG, Nd : YAG fiber, laser power 3–600 W, scan speed 6–1257 µm/s, the distance between the lines 2–300 µm, the thickness of 20–60 µm.

On grounds of performed analysis the following operating parameters for EOS EOSINT 250 XT to obtain the initial implant model have been suggested: power CO_2 laser 20, 60, 100 W, scanning speed from 1000 to 1250 µm/s, the distance between lines 150 µm, the thickness 50 µm, spot diameter 100 µm.

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