

# Influence of the selective laser sintering process parameters on the physical properties of spatial model of the ceramic implant

Wpływ parametrów procesu selektywnego spiekania laserowego na właściwości fizyczne modelu przestrzennego implantu ceramicznego

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Selective laser sintering (SLS) is a promising technique for modern materials preparation of complex shapes in one operation without any additional equipment. This technique allows production of ceramic implants at a preset porosity. Performed researches aimed at describing the effect of operating parameters of EOS EOSINT 250Xt machine on microstructure (XRD) and physical properties (apparent density, open porosity) of ceramic preliminary spatial models of implants built from submicrocrystalline sintered corundum and 99A noble electrocorundum

**KEYWORDS:** SLS technique, ceramic implant, submicrocrystalline sintered corundum, 99A noble corundum, open porosity, apparent density

*Selektywne spiekanie laserowe (SLS) jest obiecującą, nowoczesną techniką otrzymywania materiałów o skomplikowanych kształtach w jednej operacji technologicznej, bez wprowadzania dodatkowego oprzyrządowania. Technika ta pozwala na wytwarzanie implantów ceramicznych o zaprogramowanej dużej porowatości. Badano wpływ parametrów pracy urządzenia EOS-EOSINT 250Xt na mikrostrukturę (XRD) i właściwości fizyczne (gęstość pozorną, porowatość otwartą) kształtek ceramicznych wstępnych modeli przestrzennych implantów z submikrokryształicznego korundu spiekanego i elektrokorundu szlachetnego 99A*

**SŁOWA KLUCZOWE:** technika SLS, implant ceramiczny, submikrokryształiczny korund spiekany, elektrokorund szlachetny 99A, porowatość otwarta, gęstość pozorną

Selective laser sintering (SLS) is one of the methods of additive manufacturing of components, prototypes and tools. The process involves melting and the merging the layers of powder with a laser beam. Majority of commercially available SLS systems use the radiation source in the infrared range: the CO<sub>2</sub> laser (10.6 μm) or Nd: YAG, fiber lasers (1,06 μm). The laser beam melts the powder surface in accordance with the previously programmed and properly configured information on the successive layers of the cross-sectional image of the spatial object.

The selection of appropriate parameters of the laser beam (beam power, the scanning speed, distance between lines thickness of layers) allows for the sintering (melting) at specific areas of the powder particles [1, 2].

The SLS technology provides a flexible and inexpensive production of small series and individual components. The technique of selective laser sintering of ceramic powders are rarely used due to the low availability of laser equipment for sintering ceramics. The Institute of Advanced Manufacturing Technology has EOS EOSINT 250Xt machine, which can be used for sintering of free-form ceramic components (Fig. 1).

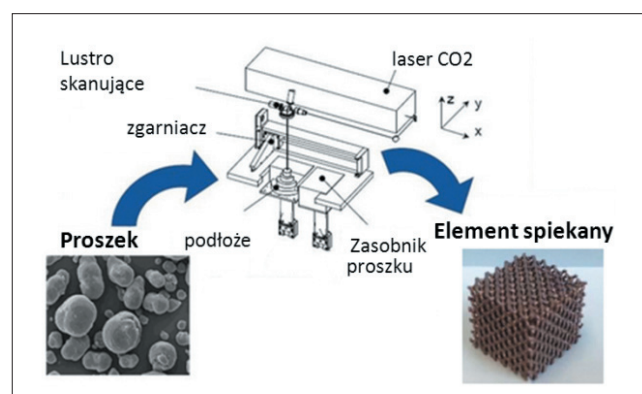


Fig. 1. Scheme of the SLS process

Selective laser sintering is an example of technology appreciated after 30 years of its invention. The first patent for a system of forming a layered object was reported by Ross Hausholder in 1979 (Patent US4247508A). However, it wasn't commercialized due to lack of funds. Regardless of Ross Hausholder's in 1981 by Carl Deckard developed the concept of building a similar machine parts using selective laser sintering. The first commercial unit was developed in the company at Desk Top Manufacturing (DTM) in 1989. Currently, the market leaders of the SLS systems are: 3D Systems, EOS, Phoenix Systems [3].

## Methodology of research and discussion of results

The initial spatial models (shapes 10×10 mm) were sintered of submicrocrystalline sintered corundum of F80 granulation (180÷212 μm) long milled in a ball mill Pullverisette 6 devices, later on sieved through a mesh 40 μm and non-milled submicrocrystalline sintered corundum of F80 granulation and 99A noble electrocorundum of F120 granulation (95÷126 μm).

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For the needs of SLS process using EOS EOSINT 250Xt following parameters were used: scanning speed of 100 mm/sec, layer thickness 0.1 mm, hatch space of 0.1 mm and laser power was changed in the range of 25÷45% of maximum power.

Diffractometry (XRD) analysis of sinters (Fig. 2) allowed to determine the phase composition, crystallite size and unit cell parameters of the materials were obtained. Also determined apparent density, open porosity and density of the skeleton for a noble electrocorundum 99A were measured. Initially, at the maximum parameters of operation, sintering of fine submicrocrystalline sintered corundum gave no positive results. Due to the fineness of the grains of sintered alumina, when too high laser power was used, powder seeds were ejected from the powder bed surface out of the laser influence zone.

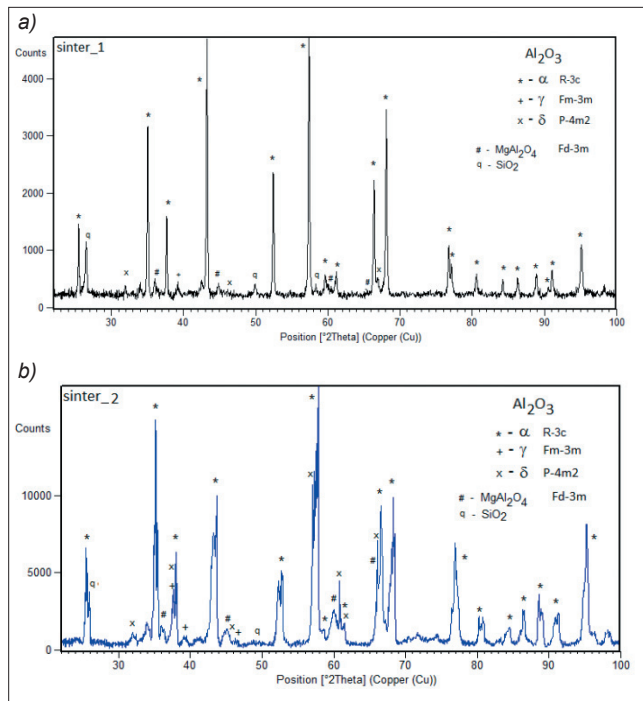


Fig. 2. Diffraction patterns of samples obtained from unmilled submicrocrystalline sintered corundum (a), milled (b)

There was no adhesion to the substrate (titanium plate) for particles of submicrocrystalline sintered corundum, lowering the laser power to a range of 25÷45% of maximum power allowed to obtain sintered moldings, the greater the grain size (F80, F120), the better they formed the fittings. In case of samples obtained from milled grains of the sintered corundum surface of sintered sample had deformed shapes.

In the X-ray diffraction spectra (Fig. 2) in all three sinter: unmilled and milled submicrocrystalline sintered corundum and noble electrocorundum presence of  $\alpha, \gamma, \delta - \text{Al}_2\text{O}_3$  and  $\text{Mg}_{0.4}\text{Al}_{2.4}\text{O}_4$  non-equilibrium phase (spinel  $\text{MgAl}_2\text{O}_4$ ) were identified. The phase composition of the samples are shown in Table I. The crystallite size were varied from 15.8 to 100 nm.

Samples of noble electrocorundum were sintered at different laser power (25, 30, 35, 40 and 45% of maximum power). The open porosity, the density of the skeleton and apparent density were measured using hydrostatic measurement method (EN 623-2). The open porosity decreased with increasing laser power from value of 38%, reaching minimum (20%) at 40% of maximum power, then increased to 25% at 45% power (Table II).

TABLE I. Phase analysis of sinter

Sample	Phase	Cell unit/parameters a/b/c, nm	Amount, %	Crystallite size D, nm
sinter_1	$\text{Al}_2\text{O}_3 \alpha$	0,47590/ 1,29926	84	51,9
	$\text{Al}_2\text{O}_3 \gamma$		3	
	$\text{Al}_2\text{O}_3 \delta$		3	
	$\text{MgAl}_2\text{O}_4$	0,39415	3	
	$\text{SiO}_2$	0,59543/ 2,35889	7	
sinter_2	$\text{Al}_2\text{O}_3 \alpha$	0,47630/ 1,30019	84	15,8
	$\text{Al}_2\text{O}_3 \gamma$		3	
	$\text{Al}_2\text{O}_3 \delta$	0,45695/ 0,54466/ 2,55181	3	
	$\text{MgAl}_2\text{O}_4$	0,80069	4	
	$\text{SiO}_2$		6	
sinter_3	$\text{Al}_2\text{O}_3 \alpha$	0,47589/ 1,29979	91	> 100
	$\text{Al}_2\text{O}_3 \delta$		6	
	$\text{MgAl}_2\text{O}_4$	0,55936/ 2,36479 0,79220	3	

TABLE II. Effects of laser power on the open porosity and apparent density

% max power	Open porosity, %	Apparent density, g/cm <sup>3</sup>
25	37,982	2,43
30	35,729	2,53
35	32,577	2,64
40	20,111	3,17
45	24,977	2,96

## Resume

Selective laser sintering is a promising technique that allows to build components of corundum ceramics at preset high porosity. Fragmentation of grains of submicrocrystalline sintered corundum did not allow to obtain samples of the assumed shape, only increasing the grain size (>100  $\mu\text{m}$ ) enabled to receive samples of the desired shape. The open porosity of the prepared samples, reached a minimum for 40% of the maximum power of the laser beam.

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