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Original Article

# Selective laser sintering of porcelain

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## ABSTRACT

Selective Laser Sintering (SLS) technique is used to fabricate 3D porcelain products with complex shapes. Commercial powder has been studied and optimized in terms of morphology and particle size distribution in order to get a perfect powder layerwise which remains the critical step of such a technique. The influence of laser energy density (through the laser power and scan speed) and hatching space have been investigated to determine the optimized parameters that allow the greater densification of this complex multi-materials composed of kaolinite, quartz and potassium feldspar. The laser-sintered porcelain products which exhibit about 60% of porosity have been post-treated at 1350 °C under vacuum or air to further improve densification.

## 1. Introduction

Ceramics are very attractive materials for their outstanding physical and chemical properties which allow them to be used in a wide range of applications related to aeronautic, energy, optics or biomedical domains for example [1]. These materials are usually shaped by casting, forging or machining. However these techniques cannot compete with rapid prototyping that allows designing ceramics with very complex shapes. Additive manufacturing (AM) or 3D printing technology consists in building objects with complex shapes, layer by layer, from ink, slurry or powder. Stereolithography, inkjet printing, Layer-wise Slurry Deposition (LSD), Fused Deposition Modeling (FDM), micro-extrusion or Selective Laser Sintering/Melting (SLS/SLM) are such techniques that are emerging during the last decades [2–6]. The SLS/SLM technique is based on a thin layer (few tens of micrometers) that is spread by a roller from a powder container to the sintering platform. The layer is selectively sintered by a laser from a Computer-Aided Design (CAD) model. Depending on the laser energy density applied during the process as well as the composition of the powder, either the term of SLS or SLM is used [7–9]. Different strategies have been developed to increase the material density after laser sintering. This can be achieved by a higher density in the powder layer, for example using a slurry instead of a dry powder which can be deposited in a similar way as tape casting or using an electrophoretic assisted device [10–13]. Mühler et al. have obtained porcelain objects with 65% of relative density using Layer-wise Slurry Deposition [13]. The relative density can also be improved using an additional heating device or chamber when the laser consolidates the powder. This reduces stresses in the object during fabrication and allows the use of higher energy densities [14,15]. Deckers et al. have obtained alumina objects with 85% of the theoretical density with a pre-heating temperature of 800 °C. Wilkes et al. obtained almost fully dense zirconia-alumina samples with a pre-heating of 1600 °C which allows the laser to completely melt the powder.

Few studies report on the SLS sintering of porcelain powders, most of porcelain 3D products being built-up from slurry-based materials combined with SLS technique [13,16,17]. SLS technique starting from powder has been already applied to metals, glasses or ceramics like alumina, zirconia or hydroxyapatite [18–22]. The SLS technique is a pressureless sintering process in which a close packing of powder particles is essential to enhance sintering density and to avoid defects in the consolidated object [23].

In the present work, commercial porcelain powder without any additives has been used to investigate the laser sintering process. The powder characteristics *i.e.* the particle size distribution and morphology are critical parameters that play a major role on the fabrication process especially regarding the powder flowability during the layer building. The influence of a post-sintering treatment at high temperature has been studied in order to reduce the porosity of the final objects.

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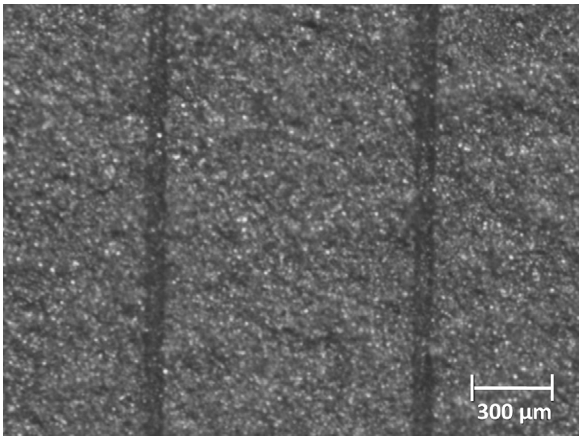

**Fig. 1.** Laser marks on a porcelain plateau coated with graphite.

## 2. Experimental procedure

### 2.1. Preparation of the porcelain powder

Commercial porcelain powder (Imerys PC804B) with the following composition (wt%): 60% kaolinite, 25% quartz, 15% potassium feldspar was used as the starting powder.

### 2.2. Selective laser sintering and post processing

3D porcelain objects were designed using a PM100T (Phenix Systems) SLS apparatus equipped with a $Nd^{3+}$-doped fiber laser (1.06 µm, 200 W) and a x-y scanner. The apparatus is composed of a powder alimentation container, a roller and a sintering platform which is, in our case, a homemade porcelain plateau. The layer thickness is fixed and equal to 60 µm. Using a porcelain plateau coated with a graphite spray, we have estimated the beam diameter to be approximately 70 µm (Fig. 1). Different scanning strategies can be applied during the laser sintering [23]. In our case, we used a strip hatch with unidirectional vectors and a difference of 90° between even and odd layer vectors. All the experiments were conducted with a laser scanning velocity of 0.5 m/s. We have varied the distance between laser vectors, or hatching space, in the range (20–40 µm) and the laser power from 65 W to 113 W. The conducted experiments are summarized in Fig. 2. In this representation, the total energy supplied to consolidate a layer increases when moving respectively to the right and to the bottom. The laser-sintered objects were then heat-treated at 1350 °C for 2 h under different conditions (under air and 0.2 mbar vacuum) in order to decrease the porosity of the final object.

### 2.3. Characterizations

The morphology and particle size of the initial powder as well as the chemical analysis of the laser-sintered samples were observed by Scanning Electron Microscopy (EDS-SEM) with a JEOL IT 300 LV equipment. The particle size distribution of the porcelain powder was determined using a Malvern Mastersizer apparatus. The microstructure of the laser-sintered objects was studied by a Nikon Eclipse optical microscope and by SEM. The structure of the initial powder, the 3D objects after laser sintered and after post-processing (heat-treatment) was checked by X-Ray Diffraction using a Bruker D8 Advance. The bulk density ($\rho_{bulk}$) and the open porosity ($v_{op}$) of the different porcelain objects were measured by a homemade Archimedean apparatus under water and vacuum using the following equations [24]:

$$\rho_{bulk} = \frac{\rho_{water} m_1}{m_3 - m_2} \quad (1)$$

$$v_{op} = \frac{m_3 - m_1}{m_3 - m_2} \quad (2)$$

where $m_1$, $m_2$ and $m_3$ are the weights of the dry sample, the water impregnated sample measured immersed in water, and the water impregnated sample measured in air, respectively. $\rho_{water}$ is the water density. The total porosity ($v_p$) was then calculated using the relation:

$$v_p = 1 - \frac{\rho_{bulk}}{\rho_{solid}} \quad (3)$$

Where $\rho_{solid}$ is the solid phase density measured with a helium pycnometer on crushed samples.

Reflectivity measurements of porcelain powder at the laser wavelength (1.06 µm) was performed with a UV-vis-IR Lambda 365 Perkin Elmer equipped with an integrated sphere.

Surface topography of porcelain layers before laser-sintering was determined using a profilometric microscope (ZoomSurf, Fogale Nanotech®, France) [25]. Mountains Map® software allows to build numerically the 3D surface topography. The main principle of 3D optical profilometry technique is based on the measurement of interference fringes (1) resulting from the recombination of both (i) reflected light at the surface of a specimen and (ii) reflected light by a reference

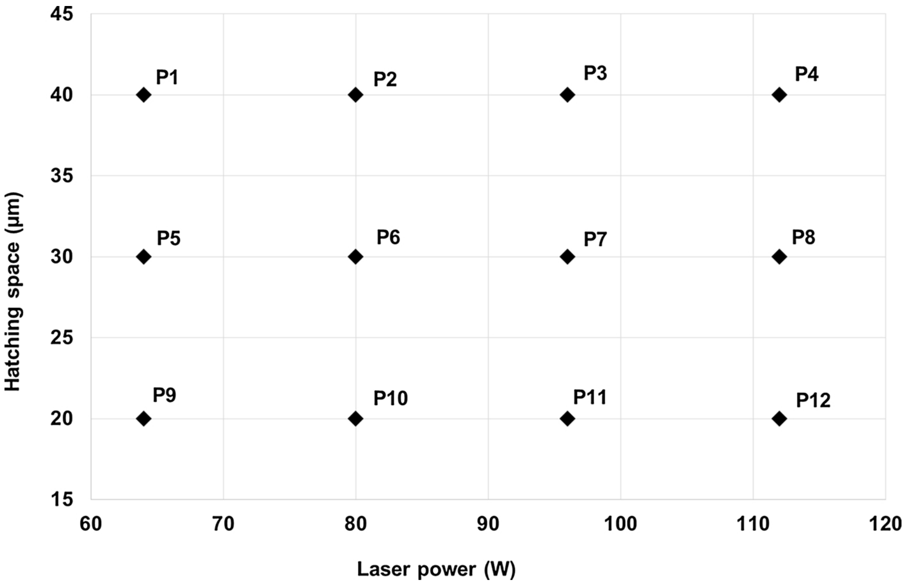

**Fig. 2.** Values of hatching space and laser power used in the different experiments.