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Journal of the European Ceramic Society xxx (2017) xxx-xxx



Contents lists available at www.sciencedirect.com

Journal of the European Ceramic Society



journal homepage: www.elsevier.com/locate/jeurceramsoc

Additive manufacturing of green ceramic by selective laser gasifying of frozen slurry

Geng Zhang^a, Hua Chen^{a,*}, Hongwei Zhou^b

^a School of Mechatronic Engineering, XI'AN Technological University, China

^b School of Materials Science and Chemical Engineering, XI'AN Technological University, China

ARTICLE INFO

Article history: Received 26 January 2017 Received in revised form 20 February 2017 Accepted 20 February 2017 Available online xxx

Keywords: Additive manufacturing Ceramic Slurry Laser gasifying Freezing

ABSTRACT

The slurry-based additive manufacturing (AM) of ceramics involves a drying process to form solid support; however, the drying process is time-consuming, and the support is not easily removed. We propose a new AM process for green ceramic that includes freezing a layer of aqueous ceramic slurry, laser gasifying of the frozen-layer ice to process 2D green ware, and removing the support in water to release the 3D ceramic part. With a suitable laser power and scanning speed, this approach can yield a layer that has a thickness of 90 μ m, a cantilever structure with a wall thickness of 115 μ m and a span of 30 mm without deflection. The casting layer cannot be damaged by using a cryopanel to rapidly freeze the slurry, and redundant frozen materials can be melted in water without swelling. Therefore, this new process can rapidly form a solid support and has a high removal efficiency.

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1. Introduction

Additive manufacturing (AM) can be used to process ceramic parts that have complicated shapes [1]. The direct process of ceramic AM includes selective laser sintering (SLS) [2-4] and selective laser melting (SLM) [5,6]. The former may produce a relatively good surface and uniform organization with low density, whereas the latter may form completely dense parts with a high surface roughness. Another feasible method is to process green ceramic in a certain shape with AM technology and then sinter such green ware in the furnace. The materials used in green ceramic AM may be classified as powder, paste or slurry according to the solid content. The powder-based AM includes indirect SLS/SLM [7-11] and threedimensional printing (3DP) [12]. The relatively low packing density of the powder makes the products not dense [11], and the larger sized particles indicate a relatively thicker packing layer, which affects the precision of the products [2]. Paste-based AM includes fused deposition of ceramics (FDC) [13] and freeze form extrusion fabrication (FEF) [14–16]. FEF extrudes the paste layer by layer with a 3D extrusion device to obtain the frozen part at a temperature that is lower than the freezing point of the paste, and the ceramic paste is prepared with ceramic powder, a binder and water. To satisfy the extrusion and accumulation process, the paste must have

* Corresponding author. *E-mail address:* xatu.ch@outlook.com (H. Chen).

http://dx.doi.org/10.1016/j.jeurceramsoc.2017.02.040 0955-2219/© 2017 Elsevier Ltd. All rights reserved. a high solid content; thus, the outlet should have a large diameter, which causes the formed parts to have a severe stairstepping effect.

The slurry-based AM involves mixing water, ceramic particles and the water-soluble binder together. In addition, the good fluidity and dispersion of the slurry make the casting layer more homogeneous. Ceramic particles of a smaller particle size are acceptable in the preparation of the slurry so that an ultra-thin layer may be casted, which may improve the stairstepping effect and enhance the surface quality of the products [3]. A slurry of a high solid content can be used to produce high density parts [17]. Ceramic laser gelling (CLG) [18,19] involves the gelling effect of sol and irradiates the slurry with a laser directly to form a three-dimensional network structure, forming porous parts without any impurities. However, it is necessary to design a support specifically when processing the cantilever structure. There are also slurry-based AM methods that do not require designing a support, such as ceramic laser fusion (CLF) [20], ceramic laser sintering (CLS) [21], layer-wise slurry deposition (LSD) [22–24], selective laser scanning the gelled layer [25] (hereinafter referred to as SLSG), and selective laser burnout (SLB) [26]. In these processes, the current slurry layer should be dried before laser scanning, and the ceramic particles are combined together due to the dehydration effect to consolidate them into a solid support, thus improving the strength of the deposited layer and reducing deformation. It is time-consuming to dry the entire layer. However, the solid support is not easily removed.

This study aimed to develop an AM process for green ceramic via selective laser gasifying, which retains the advantage of the slurry

Please cite this article in press as: G. Zhang, et al., Additive manufacturing of green ceramic by selective laser gasifying of frozen slurry, *J Eur Ceram Soc* (2017), http://dx.doi.org/10.1016/j.jeurceramsoc.2017.02.040

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in the casting process and provides a solid support for the building part. In addition, by using this new process, the formation and removal efficiency of the solid support should be improved.

2. Experiment

2.1. Experimental apparatus

To verify the feasibility of the new process, an experiment platform was constructed in this paper. The experimental platform is composed of a casting apparatus, a freezing apparatus and a laser scanning apparatus. As shown in Fig. 1, the casting apparatus is composed of a scraper, which was used to evenly pave the slurry and lift the platform to control the casting thickness and the accumulation of layers in the direction of the Z axis. The freezing apparatus consists of a cryopanel and a low-temperature building cylinder, which was used to quickly freeze the slurry and maintain the frozen state of the deposited layers. The laser scanning apparatus includes a CO_2 laser that has an adjustable power (60 W maximum) and an X-Y stage, which was used for selective gasification.

2.2. Materials

The slurry was composed of silicon dioxide powder (10–20 nm in size; Dingshengxin Chemical Industry, China) and sodium silicate (silicon dioxide content \geq 26 wt%, sodium oxide content \geq 8.2 wt%, modules 3.1–3.4; Huixin Chemical Industry, China). A slurry with a good liquidity and dispersibility and a silicon dioxide content of 40 wt% was prepared.

2.3. Experimental procedure

The new process includes the following steps: a. prepare the aqueous ceramic slurry; b. use the scraper to pave a layer on the lifting platform; c. contact the current layer surface with the cryopanel to freeze the slurry; d. according to the path of the 2D pattern planned by the computer, selective scanning is carried out with lasers to locally gasify the ice crystals; e. lower the lifting platform; repeat steps b to e until the 3D parts are constructed; f. immerse the entire ceramic workpiece in water to remove the material not yet being scanned to obtain the green ceramic. Steps b to e are shown in Fig. 2, which explains the manufacturing processes of a single layer.

To quickly obtain a smooth frozen casting layer, a cryopanel with a temperature of -50 °C and an internal circulation that is equipped with a refrigerating fluid was used to contact the surface of the current layer for 1 s. A layer of hydrophobic film was coated at the bottom of the cryopanel to avoid adhesion of the frozen slurry. To keep the deposited layers in the frozen state, the temperature of the building cylinder should be always lower than the freezing point of the slurry. To explore the relationship between the gasified thickness, the laser power and the scanning speed, a series of monolayer samples were processed at different laser powers and scanning speeds. Then, their support materials were removed by melting in water, and they were allowed to dry naturally at room temperature. Their thickness was measured using biological microscopes (Model: OLYMPUS CX31, UIS2 optical system, Japan). Finally, a cantilever structure part was processed to show the characteristics of the solid support that was formed by the new process.

3. Results and discussion

3.1. Process of laser gasifying

The principle of selective laser gasifying is shown in Fig. 3. When freezing the aqueous ceramic slurry, liquid water in the slurry turns into solid ice as the volume expands. The ice crystals grow in a columnar manner and are pushed by ice crystals to make the ceramic particles come closer to each other. The absorption coefficient of ice for CO₂ lasers (with a wavelength of 10.6 μ m) reaches 158,000/m [27]. When frozen slurry is irradiated using a CO₂ laser, most of the laser energy is absorbed by the ice at the surface, the temperature instantly exceeds the boiling point of water; thus, the ice crystals gasify directly. During the gasification process of ice, no liquid water is produced; thus, the location of the ceramic particles will not be damaged by the surface tension of the solid-liquid interface. Therefore, the ceramic particles remain in situ, thus forming a porous structure that is exactly the same as the structure of an ice crystal. After laser gasifying, the sodium silicate loses moisture and



Fig. 1. Schematic diagram of the experiment platform.

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