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Citation: AIP Conference Proceedings **1772**, 030003 (2016); doi: 10.1063/1.4964541 View online: http://dx.doi.org/10.1063/1.4964541 View Table of Contents: http://scitation.aip.org/content/aip/proceeding/aipcp/1772?ver=pdfcov Published by the AIP Publishing

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On the Possibility to Fabricate Ceramics Using Fused Deposition Modeling

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Abstract. The paper presents a uniquely designed device that enables controlled manufacturing of semi-fabricated products from thermoplastic ceramic suspensions by fused deposition modeling. Sintering of the products yields ceramics with high strength and hardness. We use ceramic aluminum oxide (Al_2O_3) as an example to prove that additive ceramic structures can be produced without noticeable boundaries between layers of the material.

INTRODUCTION

Recent breakthroughs in digital technologies and equipment have revolutionized the world engineering science in general and additive layer manufacturing of structural elements in particular. What differs additive technologies from traditional manufacturing methods is that a model (part or item) is produced by depositing material on the growing workpiece with accurate three-dimensional (3D) reproduction of the shape irrespective of its complexity. Additive technologies are a powerful tool that can speed up pre-production and manufacturing operations as well as improve the quality of items fabricated, if used for manufacturing new products in aviation as well as mechanical and instrument engineering.

When evaluating the possibility of manufacturing engineering ceramics by fused deposition modeling (FDM), we need to first analyze the current approaches to choose the optimal method and then study the parameters of the resulting additive ceramic structures.

Research has shown that there are 3D printing methods of manufacturing products from refractory powders, but the approaches of sticking the particles together do not allow producing high-density and high-strength articles [1-3]. This substantially limits the scope of their application in modern engineering. A method combining that of die casting using solid material powders and FDM would make it possible to expand the list of materials used for printing and create cost-efficient technological solutions for molding complex shapes from oxide, nitride, and carbide ceramics as well as other refractory compounds.

It is also known [4, 5] that ceramic materials have such properties that make them competitive against some structural materials including high alloy steels, nonferrous metals, and hard alloys. An example to illustrate this can be the experience of applying ceramic materials to manufacture tools and machine components. However, the development of modern science and technology puts forward new demands to ceramics in terms of increasing their physical and mechanical properties, accuracy of dimensions and geometrical complexity of the products. This encouraged extensive research to create new ceramic materials with special properties (high dielectric permeability, high mechanical strength, low/high thermal conductivity, crack resistance, heat

Prospects of Fundamental Sciences Development (PFSD-2016) AIP Conf. Proc. 1772, 030003-1–030003-6; doi: 10.1063/1.4964541 Published by AIP Publishing. 978-0-7354-1430-3/\$30.00

030003-1

resistance, etc.) [6-9]. As a result, researchers have accumulated a significant amount of fundamental data on patterns of forming the structure and properties of ceramic materials. At the same time, the key methods of manufacturing ceramic products are still compression molding (with further mechanical treatment) and diecasting.

Therefore, the development of advanced high-precision devices tailored to work with thermoplastic ceramic systems is an urgent task, since it will make it possible to produce complex shapes without using expensive technological equipment and with minimum labor costs. For example, it is reported in [10-12], that there is possibility of producing ceramic/metal products from powders and polymeric binders. In particular, the fusing deposition method using an extruder fusing has original design [11, 12].

The main idea of this work is the integration of the scientific basis behind synthesizing thermoplastic suspensions with controlled rheological properties and engineering developments of the uniquely designed 3D printer in order to produce functional and structural items from ceramics.

MATERIALS AND METHODS

For 3D printing of complex ceramic shapes, we have designed a novel construction of printer (see Fig. 1) and extruder (see Fig. 1, items 7-9), which provides the extrusion of thermoplastic ceramic suspensions through an exchangeable die. A thermoplastic suspension was heated up to a temperature within the range of $(70 \div 90)$ °C and then fed through a nozzle under a pressure derived from the following formula:

$$\Delta p = \frac{1}{\rho} \left(\frac{G}{\varphi S_{\kappa \rho}}\right)^2 \tag{1}$$

where Δp is the feed pressure, Pa; G is the required consumption of thermoplastic suspension, kg/s; ρ is the suspension density, kg/m³; S_{kp} is the nozzle throat area, m²; φ (T) is a nondimensional factor determining the dependence of thermoplastic suspension viscosity on temperature.

We used a mixture of refractory powders of aluminum oxide with a thermoplastic binder for thermoplastic suspension: ceresin/paraffin and wax. The commercial name of the mixture - thermoplastic slip VK 95-1 produced by HC Open Joint-Stock Company NEVZ-Soyuz, the content of basic components: 95% alumina, 5% - additives powders SiO₂, MgO; the binder content is typically less than 12%. Layers of the hardening material were rapidly cooled by pumping cold air to the point of extrusion.

After analyzing literary references and experimental studies to obtain the dependence of viscosity on temperature for various thermoplastic suspensions [13], we chose the working temperature range $(70 \div 90)$ °C for the extruder. We found it impossible to provide controlled consumption of the suspension at a temperature t<70 °C, since even small fluctuations of temperature cause the viscosity of suspension to change rapidly, which leads to a change in φ and, at a given Δp , there is a significant dispersion of flow rate. The top limit was 90 °C, because at this temperature, the suspension viscosity did not depend much on the temperature $\mu(t)$, and, therefore, on $\varphi(t)$ as well, which provides stable consumption G. Further increase in the temperature provides even higher stability, while at the same time requiring more power for heating. It may also trigger a spontaneous ignition of the binder, which disturbs the technological regime.

See Fig. 1 for a schematic representation of our 3D printer design. The device consists of an electronic control unit with CAD/CAM software installed (10) controlling the movement of the platform (7) along the required path as well as the work of the gearbox (2) to regulate the pressure in an air supply system from the compressor (1). Thus, the gearbox (2) and valve (8) control pressure in the system feeding the thermoplastic suspension over the pipeline and thus determine the material consumption at each point. The insulated vessel (3) destined for heating the thermoplastic suspension up to the required temperature has a pressure gage, heating elements and a stirrer driven by the electric motor (4). Stirring the thermoplastic suspension melted in the tank (3) is necessary to avoid delamination.



FIGURE 1. Scheme of 3D printer developed for printing ceramic objects. 1 – compressor, 2 – gearbox, 3 – vessel, 4 – electric motor, 5 – pipeline, 6 – table, 7 – platform, 8 – valve, 9 – diffuser nozzle, 10 – software controller

The printing process was as follows. We first designed the necessary shape of a part. In this case, we chose a thin-walled cube with an internal diagonal (Fig. 2 (a)). The thermoplastic suspension was heated up to the working temperature and pressure-fed over the heated flexible pipeline (5) to the printing unit. It is important to control the temperature of the flexible pipeline. Under predetermined pressure (0,15 MPa) and temperature, the thermoplastic suspension was fed to the printing unit with a nozzle (diameter of 600 μ m). The required shape was formed from the thermoplastic suspension by fused deposition modeling. Each consecutive layer formed by the feeder tip is higher than the previous one by a predefined (in this case 400 μ m) and controlled value equal to the distance covered by the feeder head. It is essential to cool the suspension layer by supplying compressed air to the injection point. Figure 2 (b) shows a model of the sample produced after breaking it down to layers for SD printing.



FIGURE 2. Model of the part to be produced. 3D model with dimensions in mm (a) Image of the part after slicing (b)

Figure 3 depicts a fragment of the 3D printing process for a ceramic sample. The process clearly follows the predetermined scheme. There is no layer delamination. Striations occurred due to the structural peculiarities of the printing device, which requires further modernization.



FIGURE 3. Fragment of 3D printing process of an object from engineering ceramics

To remove the linking components, the printed workpiece was fired at a temperature of 1100 °C and then sintered at 1700 °C. The structure of the resulting ceramic samples was surveyed on a scanning electron microscope Philips SEM 515.

RESULTS AND DISCUSSION

After high-temperature sintering of the samples, the width and height of a layer was approx. 400 μ m (Fig. 4), which complied with the predetermined layer height as the extruder moved along its path.



FIGURE 4. Additive structure of ceramics after high-temperature sintering

Detailed research into the structure of the samples produced has shown that there were isolated agglomerates of sintered particles (Fig. 5 (a)) on free surfaces. These seem to have agglomerated due to the aluminum oxide powder adhesion, in which we performed initial firing to remove the thermoplastic binder. Images with a greater zoom showed grain structure of ceramics. The mean size of an Al₂O₃ grain made up 3 μ m with the maximum size not exceeding 8 μ m, see Fig. 5 (b). The research into the element composition of the sample surface using the analysis of variance has shown that the material consists of aluminum oxide without any impurities. This testifies to full removal of the thermoplastic binder after sintering.



FIGURE 5. Additive structure of ceramics (a) and size distribution of aluminum oxide grains (b)

We studied the internal structure of ceramic samples. Figure 6 depicts the structure of additive ceramic fracture Boundaries between layers of material are clearly noticeable (and marked by a full line). An important result obtained during the research and development is the conclusion that the internal ceramic frame is solid, without any boundaries between layers formed during the printing process. Thus, the proposed method shows an advantage over other additive technologies for ceramic materials. For example, in the paper [14] it has been shown that there is formation of boundaries between the printed layers, and as a consequence of the high porosity of the material. The character of the fracture testifies to the intercrystalline cracking, which in turn proves high strength of the grain boundaries.



FIGURE 6. Structure of additive ceramic fracture

The research into mechanical properties of additive ceramic structures based on aluminum oxide has shown that the average flexural strength of the samples under three-point bending test was 270 MPa. The average Vickers microhardness amounted to 22 GPa, while Young's modulus was approx. 300 GPa. These parameters are not inferior to the hardness properties of the samples produced traditionally by die casting [13].

CONCLUSION

We have shown that additive ceramic structures can be produced from thermoplastic suspensions with controlled rheological properties. After sintering the ceramic structures produced by the additive layer method, the height of a layer was approximately 400 μ m with the average size of an aluminum oxide grain not exceeding 3-4 μ m. According to the authors of this work, it is of crucial importance that the inner structure of ceramics is a solid frame without any apparent boundaries between horizontal and vertical (walls) layers of the material in

samples. The strength and hardness of additive ceramic structures are not inferior to those of the samples produced conventionally by pressure molding.

ACKNOWLEDGMENTS

The work was financially supported from the Ministry of Education and Science of the Russian Federation within the framework of the Federal Target Program. Agreement No. 14.578.21.0034 (RFMEFI57814X0034).

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