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Extrusion-based 3D printing of ceramic components

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Abstract

Engineering ceramics are becoming increasingly important in the nowadays-industrial landscape, thanks to the exceptional combination of good mechanical, thermal and chemical properties. Nevertheless, traditional ceramic manufacturing technologies lack the ability to compete in a market of customized complex components. Additive Manufacturing therefore provides an important contribution, given the nearly unlimited design freedom. This research aims at developing an extrusion-based AM technology using UV-curable dispersions. The homogeneity, rheology and printability of these dispersions, containing 22,5%vol to 55%vol ZrO₂ in different commercially available resins were investigated. A sintered density of 92% was obtained, proving the potential of the technology in development.

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1. Introduction

Engineering ceramics, such as oxides, borides and carbides, receive increasing industrial interest. Due to their unique combination of mechanical, thermal and chemical properties, these materials find applications in different industries. The Freedonia Group expects the engineering ceramics market to reach 15.5 billion dollars in 2017 [1]. Especially Zirconium-dioxide (ZrO₂, Zirconia) is an interesting material: due to its high toughness, thermal insulation, biocompatibility and ionic conductivity, it is nowadays used as material for e.g. body-implants, dental crowns, stamping dies, oxygen sensors and several micro components [2].

The manufacturing of ceramic components consists nowadays of a series of discrete production-steps, as can be seen in Fig. 1. Among them, green- and final machining (e.g. grinding and polishing) are the most cost-, labor- and tool-intensive. Moreover, conventional shaping techniques such as Ceramic Injection Molding (CIM) and Gel Casting, which involve the use of a tailored mold, are only economically competitive for large-size batches and the production of simple and medium-complex ceramic components. Furthermore, the

production of highly complex 3D shapes, micro features, or structures with tailored porosity, such as scaffolds, is still seen as a major limit [2–4].

Additive manufacturing (AM) offers new opportunities in the gamma of shaping techniques for ceramics. Thanks to its (almost) unlimited freedom in design and flexibility, AM enables the production of customized and 3D complex shaped forms, even in small-size batches and with a limited time-to-market [5].

The feasibility of different AM processes, such as Stereolithography (SLA) [6], Lithography-based Ceramic Manufacturing (LCM) [7], Freeze-Form Extrusion (FFE) [8], Selective Laser Sintering (SLS) [9], Fused Deposition of Ceramics (FDC) [10] and Robocasting [11] has recently have been investigated for the production of ceramic components.

Most of these techniques make use of a composite ceramic-binder material, of which the binder is solidified, for the shaping of the green product. This latter is subsequently subjected to firing, to remove the binder and sintering in order to achieve a dens ceramic component.

SLA adopts UV-curable resin as a binder, mixed with ceramic powder. The resin is selectively cured by means of an UV-laser, providing the necessary consistence to the green product, and is subsequently removed by firing.

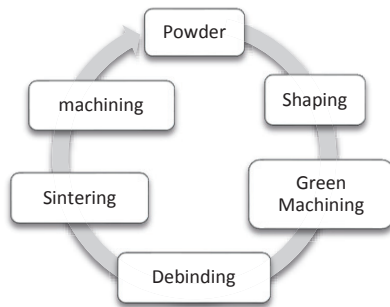


Fig. 1 – Conventional production cycle

After sintering of this fired product, a density of nearly 98% is achievable [6,12]. However, a full vat of particle-filled UV-resin is needed, which is both cost- and material-inefficient.

Indirect SLS uses alumina-polypropylene (PP) composite particles. The PP-phase functions as binder and it is selectively solidified by means of a laser. Recent research indicates that a density of up to 93% after Warm Isostatic Pressing is achievable [9]. However, further research is needed to augment this density after sintering. Besides, coating the particles with the PP-phase induces an extra step.

FDC employs a thermoplastic filament, filled in with ceramic particles. This filament is heated up to make the binder viscous, and subsequently extruded through a nozzle. Upon deposition, the binder cools and solidifies, trapping the ceramic particles and giving the green product a high strength. Using this technique, sintered densities up to 97% have been achieved [10]. However, a filled in thermoplastic filaments, made within close tolerances is needed.

Finally, Robocasting extrudes aqueous low-organic binder dispersion, containing up to 60 %vol of ceramic particles. Because of the high content of ceramic particles, the dispersion becomes dilatant upon minimal evaporation of the binder. Research indicates the possibility of producing parts with a final density of more than 95% [11]. However, a rigorous control of the drying conditions and rheological behavior of the dispersion is required to achieve adequate dimensional stability. Another important disadvantage is distinct stair casing, due to the low strength of the green product [13].

The present research specifically aims at developing a novel Additive Manufacturing process that combines the strengths of the aforementioned techniques through the combination of the economic usage of material of the extrusion process, as used in Robocasting and FDC, with the high green strength of UV-curing techniques such as LCM and SLA. This is achieved through the preparation of a dispersion, based on ceramic powders and UV-resin. This dispersion is subsequently deposited using a syringe-

based 3D printing equipment, while being cured using a power-LED source.

2. Experimental set-up

This section provides a description of materials, methods and tools adopted for the preparation and characterization of the dispersions. The 3D printing method is also described.

2.1. Materials

Commercial yttrium-stabilized Zirconia (TZ-3YE, Tosoh Corp, Tokyo) nanoparticles ($D_{50} = 0.6 \mu\text{m}$) of high purity (>99,9%) were chosen as ceramic compound for the sake of lowering the sintering temperature. A measure of the maximal particle size ($D_{\text{max}} = 4 \mu\text{m}$) is needed in order to determine the homogeneity of the dispersion. Two commercially available UV (Ultra-Violet) curable resins were used as binder: XC11122 (DSM) and UV-A 2137 (Sadecraf). The properties of the resins are listed in Table 1.

Table 1 – Properties of the UV-resins

Property	XC 11122	UV-A 2137
Viscosity (20°C)	260 mPa.s	750 mPa.s
Density	1.13 g/cm ³	1.05 g/cm ³
$E_{\text{threshold}}$	11.15 mJ/cm ²	1000 mJ/cm ²
T_{max}	46°C	30°C
Spectrum	±355 nm	320–355nm

2.2. Preparation of the dispersion

In order to achieve a homogenous dispersion, the zirconia particles were dispersed into the UV-resins for 24 hours using the “solution-mixing” (SM) principle, by means of a Turbula T2-F shaker-mixer. The mixture was then placed in an opaque container, along with zirconia mixing balls (diameter = 10mm) and 15 ml of ethanol; this latter to decrease the viscosity, so that formed agglomerates can be broken by the mixing balls. The ethanol was subsequently evaporated in a darkroom, at 45°C for 48 hours. The low temperature was specifically chosen to avoid thermal polymerization, which renders the dispersion waste. Samples were also prepared using mechanical mixing (M) of the dispersion in order to determine whether a less time-consuming method of homogenization could be feasible.

Dispersions of different compositions (Φ), containing 22.5; 25; 27.5; 30; 45 and 55 %vol zirconia particles mixed in the two UV-resins, were prepared using both mixing techniques.

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