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Research Article

Novel Prospects and Possibilities in Additive Manufacturing of Ceramics by means of Direct Inkjet Printing

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Direct inkjet printing is a versatile additive manufacturing technology to produce complex three-dimensional components from ceramic suspensions. By successive printing of cross-sections, the sample is built up layer by layer. The aim of this paper is to show the different possibilities of direct inkjet printing of ceramic suspensions, like printing of oxide (3Y-TZP, Al_2O_3, and ZTA) or nonoxide (Si_3N_4, $MoSi_2$) ceramics, featuring microstructures, laminates, three-dimensional specimens, and dispersion ceramics. A modified thermal inkjet printer was used and the ink replaced by aqueous ceramic suspensions of high solids content. The suspensions were processed in an attrition mill or agitator bead mill to reduce the grain size $<1\,\mu$m to avoid clogging of printhead nozzles. Further significant parameters are rheological properties (viscosity and surface tension) and solids content which were adjusted to the requirements of the printheads. The printed and sintered samples were analysed by SEM. Mechanical properties of 3Y-TZP samples were examined as well by use of the ball-on-three-balls test. The biaxial flexural strength of 3Y-TZP specimens was up to 1393 MPa with a Weibull modulus of 10.4 for small specimens ($3 \times 4 \times 0.3\,\text{mm}^3$).

1. Introduction

Additive manufacturing (also known as freeform fabrication or generative manufacturing) comprises a group of technologies that feature construction of objects from 3D model data by assembly of materials, typically layer by layer [1]. By these means complex three-dimensional ceramic components can be produced directly without the need for moulds or part-specific tools. Depending on the technology the shape of the sample is realized by consolidating a powder bed either by addition of a binder (three-dimensional printing [2–4]), or by selective heat treatment (e.g., selective laser sintering or selective laser melting [5–8]), or by selective curing of a photosensitive resin containing ceramic particles (stereolithography [9–12]), or by direct deposition of material (e.g., fused deposition modelling [13, 14], 3D printing in filamentary form [15, 16], aerosol jet printing [17], or direct inkjet printing [15]).

Following ASTM F2792-12a, direct inkjet printing (DIP) is considered a form of 3D printing as the construction of a specimen is realized by material deposition through a nozzle of a printhead [1]. In contrast to classical 3D printing where a bonding agent is deposited in a powder bed (binder jetting), the emitted droplets in DIP contain the building material which is selectively deposited on a substrate (material jetting) [1]. The latter enables additive manufacturing of layers and (micro) structures as well as complex three-dimensional geometries from ceramic suspensions with high solids content. The selective deposition of single particle-loaded droplets permits high precision whose level of detail even allows accurate placement of different materials next to each other. Furthermore, the resulting structures printed by DIP feature high density and mechanical properties similar to conventionally fabricated ceramic components. Due to direct deposition of materials from suspensions, it is possible to produce multimaterial parts or even cavities which are not

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feasible in such form through other fabrication technologies. Additionally, the drop-wise use of varying materials makes it possible to specifically introduce internal stresses even in complex geometries.

In drop-on-demand inkjet printing a distinction is made between piezoelectric and thermal printheads. While both employ a pressure pulse to form drops, the method varies: the former ensures displacement of ink within the printhead chamber and drop ejection through deformation of a piezoelectric crystal thus mechanically creating a pressure pulse, while thermal printheads (also called bubble-jet) force ink through the nozzle by generating a bubble caused in the ink by a rapidly heated resistor [18, 19]. Thermal printheads were used for the studies shown in this paper.

The ceramic suspension used as ink in DIP consists of a suspension comprised of ceramic particles, dispersant, and functional additives, that is, surfactant, binder, and humectant. Depending on the specific requirements of the utilized printhead these ensure the necessary adjustment of rheological suspension properties like viscosity and surface tension. A further prerequisite for the ink development results from the nozzle diameter. As picolitre sized droplets are ejected through the fine nozzles of the printhead, the particle size needs to be controlled to prevent clogging of nozzles. Lejeune et al. [20] and Magdassi [18] have recommended the ratio of nozzle diameter to particle size to be larger than 50 or 100, respectively. Furthermore, for efficient and defect-free build-up on the one side and stabilization of dispersed particles against agglomeration, flocculation, and subsequent sedimentation on the other side high solids content (>20 vol%) is recommended [21].

Oxidic high-performance ceramics, in particular alumina (Al_2O_3) and yttrium stabilized zirconia (3Y-TZP), are distinguished by high flexural strength, wear resistance, and fracture toughness as well as confirmed biocompatibility. Based on these characteristics they have been used for many years in the field of endoprosthetics, for example, as hip joint ball or as matrix material for dental prosthesis like crowns and frameworks [22]. In recent years, zirconia has even been used for dental implants [23, 24]. The steadily rising demand for ceramic materials is due to aesthetic aspects and frequently occurring biological reactions, inflicted by application of metallic implants. Metallic hip joint parts (ball and acetabulum made of titan- or cobalt-chrome-alloys) may cause friction at the metal/metal pairing, generating undesirable biological reactions, for example, local and systemic metal intoxications.

Alumina and zirconia are bioinert; that is, after the implantation, no chemical or biological interactions between implant and surrounding tissue take place. Based on clinical studies it becomes more and more apparent that loosening of zirconia dental implants consistently takes place, like it has been observed in the early 1990s on monolithic alumina hip joint acetabula [25, 26]. The occurring loosening can be explained by the inert behaviour of the ceramic surface and the insufficient stimulation of the bordering bone tissue [25, 26]. For an enhancement of the cell adhesion and differentiation strategies for bioactivation have to be located. As the microstructure of a material significantly influences the biologic reaction at the interface [27–29], it is reasonable to change the microstructure of a surface in such a way that the cells could respond favourably with a change into osteoblasts (bone-building cells) to enhance the bone engraftment. Since no literature has been found to confirm response of cells to surface modification alone of alumina or zirconia further research has to be done to verify this thesis. With a high resolution printhead potential microstructures for this field of research could be produced by DIP.

Functionally graded materials (FGMs) are combinations of two or more different materials with a continuously varying distribution across the geometry of a part. In contrast to conventional composites FGMs are characterized by very smooth transitions between different materials, which substantially improve material properties or open up entirely new applications [32]. However, conventional forming technologies as slip casting or pressing are limited concerning their possibility to define and control such gradient material transitions [33]. In contrast DIP permits precise, accurate placement of single picolitre sized droplets and is thus eminently suitable to realize complex shaped geometries with functional material gradation.

The aim of this paper is to show the different possibilities of direct inkjet printing of ceramic suspensions, like printing of oxide or nonoxide ceramics, featuring microstructures, laminates, three-dimensional specimens, and dispersion ceramics. Ceramic suspensions are characterized regarding their particle size distribution, rheological properties (viscosity and dynamic surface tension), and solids content. The printed and sintered samples are analysed by SEM.

This paper provides an overview on work concerning direct inkjet printing originated at the Institute of Mineral Engineering of RWTH Aachen University. This concerns recapitulation of previously published work (on Si_3N_4, $MoSi_2$, complex shaped structures of zirconia, and three-dimensional zirconia specimens for ball-on-three-balls test) as well as innovative novel experimental work (regarding microstructures, laminates, and dispersion ceramics).

2. Materials and Methods

A modified office type drop-on-demand thermal inkjet printer (HP DeskJet 930c, Hewlett Packard, Palo Alto, USA) was used for all samples unless stated otherwise. The modifications allowed layerwise build-up of parts from cross-sections. The printed layers were subsequently dried and the substrate lowered owing to the installation of a z-axis thus ensuring a consistent distance between the printhead and the substrate or the uppermost layer. Figure 1 shows a schematic of the printing system. This printer uses thermal printheads, of which only the black ink cartridge (HP51645A) was used to eject the ceramic suspensions. After draining and rinsing the cartridges, they were completely dried to prevent dilution with residual water. Subsequently they were refilled with a minimum of 25 mL of ceramic suspension and the remaining air was evacuated. The printhead provides 600 dpi spatial resolution by use of 304 nozzles with ~30 μm nozzle diameter each.

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