From Prototyping to Serial Production – 3D-Printing in Ceramic Manufacturing

F. Raether, J. Vogt

For two decades, Additive Manufacturing (AM) was used mainly for the rapid prototyping of visual or functional models. However, with growing awareness of its potential and increasing performance of 3D-printers, it becomes more and more relevant for small- and medium-scale production. Quality Management (QM) was no major issue in the period of prototyping but – besides production cost, process integration and automation – it becomes a key enabling factor for the implementation of AM methods in industrial production. It turns out that several challenges are to be met especially by ceramic AM to comply with customary manufacturing methods.

1 Introduction

There are five main reasons for the strong growth of AM in component production:

- It enables extremely lightweight constructions, saving energy in transportation and heating technology (Fig. 1 a).
- It can substitute complex constructions customarily fabricated from many individual components by integration in a single piece, saving assembly costs (Fig. 1 b).
- It can be used for the manufacturing of individual components and is of particular interest for the replacement of bones, teeth or organ parts in medical engineering.
- It allows for the production on demand, enabling new routes in logistics and spare parts supply.
- It eliminates the manufacture of expensive moulds, which is extremely important in the small scale production of ceramic parts.

The growing variety of AM methods has been classified in seven main categories: Powder bed fusion, directed energy deposition, material jetting, material extrusion, sheet lamination, binder jetting and vat photopolymerisation (ISO 17296-1). The first two of these categories comprise single stage processes, where forming and densification is done simultaneously. The other five categories require two stages in ceramic 3D-printing. In the first stage, green parts are formed, and in a second stage, the green parts are processed in a subsequent heat treatment to obtain the final products. Recent reviews on AM of ceramics can be found in [1, 2].

In one stage AM processes, high temperature gradients are applied, creating high thermal stress and damage in ceramic parts. For that reason, powder bed fusion and directed energy deposition have only minor importance in the AM-based production of ceramics. The remaining five categories still include a multitude of methods and variations, reflecting the ingenuity of engineers all over the world. However, this diversity can drive quality managers to despair.

2 Quality management with AM of ceramics

When ceramic parts are fabricated by AM, several challenges are to be met to achieve high strength and reliability as well as net shape performance. As usual, strength and reliability are controlled by microstructure homogeneity and surface roughness. Both are harder to achieve with AM than with standard production. Deformation during

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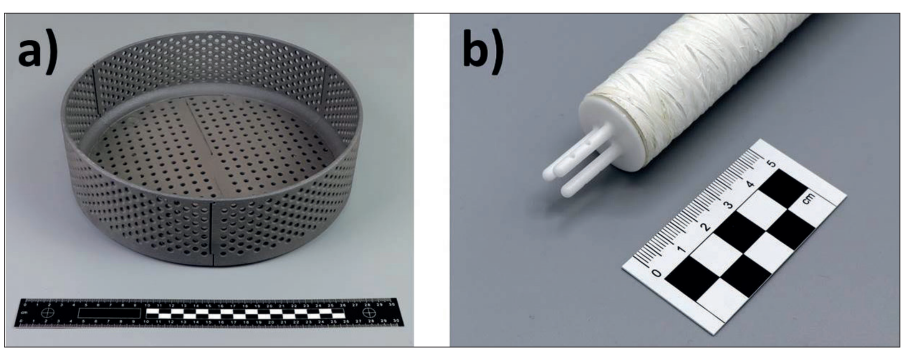

Fig. 1 a–b Ceramic parts produced by 3D-printing: a) lightweight container for heat treatment of ceramic fibres; b) high temperature sensor for simultaneous measurement of gas flow and temperature

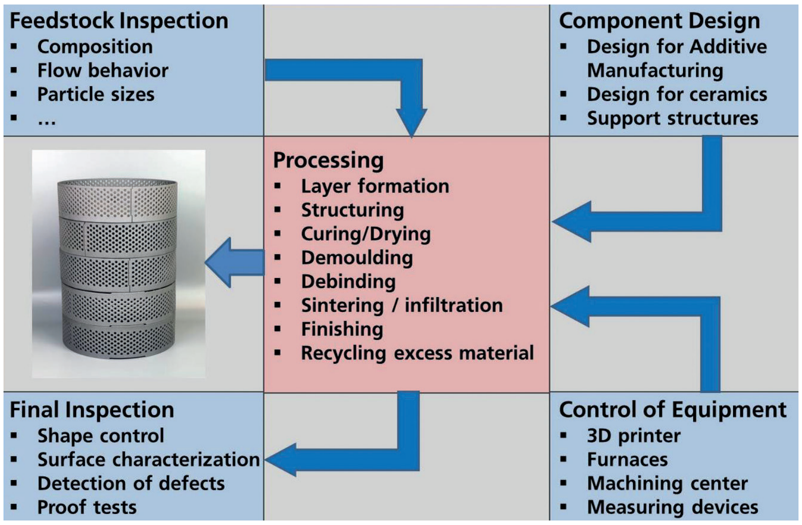

Fig. 2 Survey on quality issues related to 3D-printing of ceramics

sintering impairs net shape performance. In addition, with direct printing techniques, i.e. material jetting and material extrusion, net shape performance often is deteriorated by deviations during the printing process itself. An in-line dimensional control of printed parts, which is used in a closed loop, is rarely used but would be strongly recommended. Since the feedstock has to fulfil additional requirements compared to standard production, feedstock inspection is a critical issue in quality management of AM processes (Fig. 2). Happily, numerous well suited methods, e.g. rheological measurements, are available for feedstock control.

AM in particular offers completely new design possibilities, e.g. freeform surfaces, grid structures, undercuts and cavities. On the other hand, special restrictions exist, e.g. overhanging parts requiring support structures during printing and sintering. Design for Additive Manufacture (DFAM) becomes a separate discipline among the traditional Design for Manufacturing and Assembly (DFMA).

Specific software is available to support construction engineers [3]. However, specialists in the field of DFAM are rare, and the majority of engineers need professional development to utilise the full potential of AM. Other specific issues affect the processing, plant monitoring and final inspection (compare Fig. 2).

Depending on its field of application, AM has to fulfil the same standards as customarily production processes, e.g. EN/AS 9100 in aerospace, IATF 16949 in the automotive industry and ISO 13485 in medical technology. Due to its technical complexity, the implementation of AM in production started with polymers, followed by metals and is in an early stage in the case of ceramics. This chronology is reflected by the number of AM standards available for AM with different materials.

So far, no specific standard for ceramic 3D-printing exists. However, a number of general standards can be used, e.g.: ISO 52910-17 for DFAM, ISO 52915-16 for file formats, ISO 52921-13 for coordinate systems and test methodologies. Furthermore, a new VDI guideline on the DFAM of ceramics (VDI 3405 Sheet 3.6) is currently in preparation [4].

In the following sections, we address some general aspects of quality control in 3D-printing of ceramics which are relevant beyond individual AM methods. First of all, the control of green compact quality is outlined, followed by the heat treatment and final component inspection.

3 Control of green compact quality

Since nearly all ceramic AM techniques are two-stage processes, first of all green compacts are formed, which are available for quality inspection. Yet, AM forming processes are more complex than customary forming processes.

Considering this, it is very helpful to optimise 3D-printing parameters and feedstock properties on the basis of key figures derived from the green compacts. In doing so, the subsequent heat treatment can be developed separately (compare next section) – reducing the complexity of the entire process development considerably. The criteria for green compact quality are the same as for customary forming: close match to set shape, homogenous and dense packing of ceramic particles, homogenous distribution of the binder, smooth surfaces and absence of flaws like voids or delaminations.

Therefore, in principle the same methods can be used for characterisation of AM green compacts as those with standard processes [5]. However, due to the complex geometry of AM green parts, measuring shape usually is more complicated than with green parts from standard forming processes.

This suggests using Computed Tomography (CT) as a non-contact method, which can measure any shape, e.g. very fine struts or cavities. In addition, micro- or nano-CT

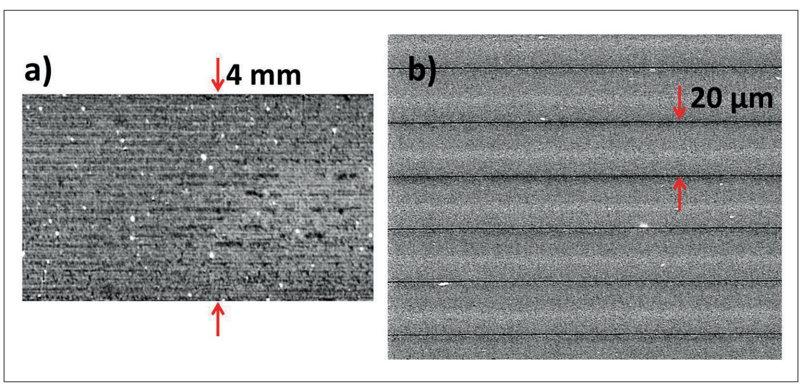

Fig. 3 a–b Measurement of homogeneity and layer structure of 3D-printed alumina green compacts: a) cross section of a part produced by binder jetting measured by computed tomography; b) cross section of a part produced by vat photopolymerisation and measured by scanning electron microscopy after cross section polishing

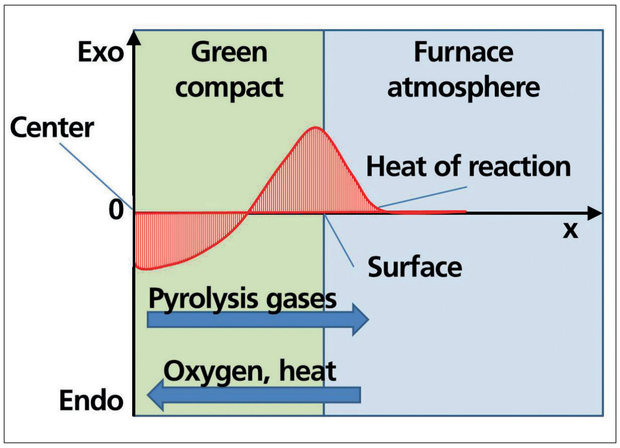

Fig. 4 Heat and mass flow during debinding

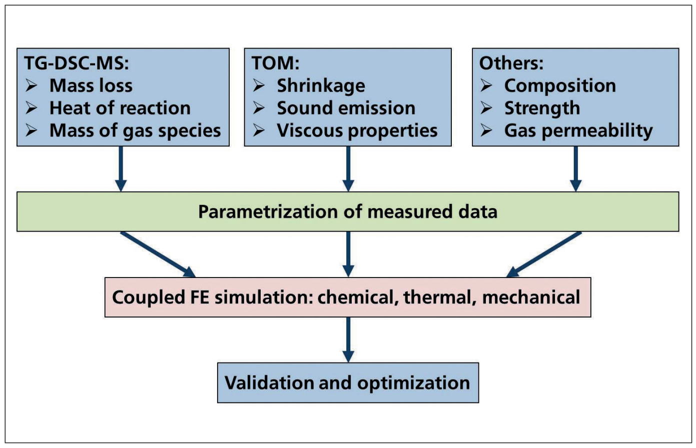

Fig. 5 Work flow during systematic optimisation of debinding cycles

detects flaws and rough structures at the parts' surfaces.

Moreover, CT can be used to analyse special structural features occurring with AM. There are three two-stage AM techniques using a layerwise building of the 3D-structure: sheet lamination, binder jetting and vat photopolymerisation. Structuring is done within the layers, after the entire component has been divided into slices. In any case, the interface between the layers deserves special attention.

Fig. 3 a shows a vertical cross section through an alumina green part from a CT measurement. The part was fabricated via binder jetting, using a dry alumina powder which was spread in a building chamber layer by layer and selectively impregnated with a binder [6]. In this process, only flowable powders can be used, limiting the particle size to coarse powders with a diameter above 10 µm.

It can be seen that the interface between the layers has a lower density, which is attributed to insufficient filling of pores in the top layer when the next layer is set up on top. The variation in X-ray density in the vertical direction is a measure for the interlayer homogeneity, while the variation of X-ray density in the horizontal direction is a key figure for the overall microstructure homogeneity. Equations and interpretation for this quantity are given in [7].

Fine ceramic particles with diameters below 1 µm can be used in slurry based processes like vat photopolymerisation [6]. In this case, slurry layers are applied in the vat and selectively cured by light. The resolution of most CT devices however is not sufficient to resolve details of microstructure. By means of cross section polishing, a clean cut through the green compact can be obtained. Scanning Electron Microscopy (SEM) is used to investigate the structure with high resolution (Fig. 3 b). It can be observed that the particle density between the layers is lower. The overall homogeneity is much higher than in case of binder jetting, being typical for slurry based AM processes. The same key figures as discussed with CT in the previous paragraph are extracted from the SEM images. These can be used for an optimisation of rheology of the feedstock and the printing parameters. Basically, the mentioned principles for green compact inspection can also be applied to the other two-stage AM processes for ceramics.

4 Quality issues during heat treatment

In principle, the issues occurring during debinding and sintering of 3D-printed parts are similar to customary green compacts. Nevertheless, the optimisation of heat treatment is often more difficult.

Depending on the AM process, debinding requires special care due to high binder concentration and/or low adhesion between layers, which frequently causes delamination or cracking.

Sintering is often complicated in AM processes with a lower green density such as binder jetting when compared to customary forming processes. This leads to enhanced shrinkage and warping, mostly superimposed by an anisotropic shrinkage. Distortions of shape are especially serious in case of complex and filigree structures. Yet, these structures are one of the most powerful characteristics of AM components. In order to overcome difficulties with heat treatment, a systematic approach is required which is outlined in the following paragraphs separately for debinding and sintering.

The thermal decomposition and combustion of binder and other organic constituents during debinding is accompanied by endothermic and exothermic processes, combined with enhanced gas pressure within the pore channels of the green compact during outward flow of gaseous pyrolysis products (Fig. 4). Temperature gradients and overpressure lead to local stress concentration and failure if the corresponding stresses exceed the strength of the compacts.

Debinding can be simulated in a Finite Element (FE) model, in which pyrolysis and combustion reactions, gas flow within the pore channels and at the surfaces, and heat flow and heat production/consumption due to these reactions are combined (Fig. 5). Based on the temperature and pressure distribution, mechanical stresses are calculated during the entire debinding cycle. Based thereon, debinding cycles are adapted to ensure that stresses are always well below the strength limit.

In order to achieve a sufficient accuracy of the simulation, a certain amount of experimental data is required (compare Fig. 5). Standard thermal analysis, simultaneously using thermogravimetry, differential scanning calorimetry and mass spectrometry (TG-DSC-MS) delivers input for very small samples, which correspond to individual elements in the later FE simulations.

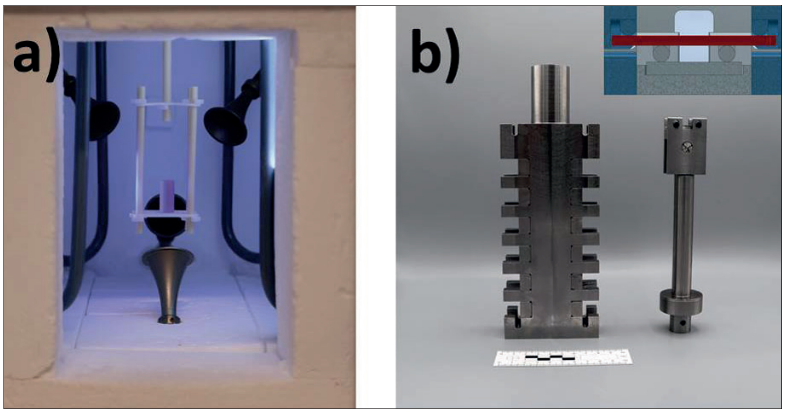

Fig. 6 a–b Non-standard equipment required for data acquisition of debinding optimisation: a) thermooptical measuring furnace equipped with weight sensor, bells for sound emission measurement and an optical beam path for dimension measurement; b) multi-sample-holder for 4-point bending strength tests on six partially debinded specimens

Specific thermooptical measuring (TOM) furnaces are used to measure larger samples and to obtain other quantities (Fig. 6 a). TOM methods are also relevant for the validation of simulations by sound emission measurements, sensitively detecting cracks in the samples during the thermal cycle. Additional material properties have to be measured to obtain a sufficient data base for a realistic simulation of debinding (compare Fig. 5).
A delicate task is the mechanical testing of partially debinded samples, since they are very fragile. A multi-sample-holder (Fig. 6 b) and highly sensitive load cell are used to measure these samples at different temperatures.

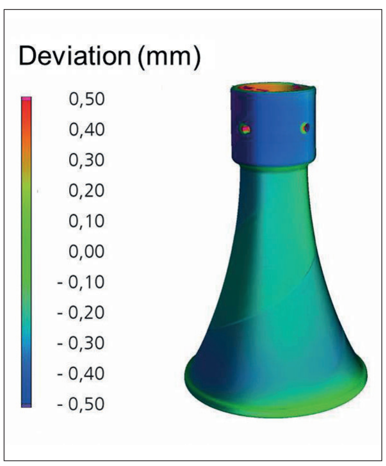

Fig. 7 Difference between set geometry and final shape of an alumina bell (height 54 mm) produced by vat photopolymerisation

A validation of the methodology as stated above was already published previously [8], and examples for 3D-printing will be published soon. The methodology is considered powerful, since it can be applied to any shape, once the model with the parametrized material data has been set up. If the shape is simple and component dimensions are small, simplified methods are available. They are based on a formal kinetic approach for debinding and need much less experimental and numerical effort [9, 10]. However, for more complicated parts these simple methods cannot identify optimum debinding conditions. The effort in obtaining additional data for the accurate model rapidly pays off, if different components are manufactured from the same feedstock.
A similar approach, i.e. an experiment-based continuum FE model, has also been developed for the simulation of sintering. Input data for sintering are shrinkage, thermal diffusivity and viscous material properties. Again, special TOM furnaces were developed to obtain these data [11]. The FE model combines temperature field, sintering kinetics and mechanical stresses. It also considers gravity and friction, which is important for sintering of 3D-printed parts. A detailed description of the model and its validation was published recently [12].
Sometimes, in order to obtain dense parts, the infiltration of the porous compacts with a melt is an interesting alternative to sintering. Thereby, the melt is soaked into the pore channels by capillary forces. The infiltration of SiC parts manufactured by binder jetting with a silicon melt, e.g., has been used for some years to produce high temperature components [13]. The containers shown in Fig. 1 a are fabricated in the same way. The advantage of melt infiltration is the avoidance of shrinkage and sintering distortions. It is especially useful for green parts with low sintering activity, as they are typical for binder jetting. Melt infiltration processes can be optimised by in situ measurement and FE simulations as well [14].

5 Final inspection of AM components

Dimensional inspection is one of the most important tasks in quality control of AM parts. However, compared to customary measuring, it becomes more difficult when dealing with complex structures with cavities etc. CT can be used as a universal tool for dimensional control, independently of structural constraints. In using special algorithms, the deviation between actual geometry and target geometry can be quantitatively measured.
Fig. 7 shows as an example a sintered acoustic bell out of alumina for sound emission measurements produced by vat photopolymerisation (compare section 4). Sintering deformations were calculated according to section 4. Deviations are indicated by a colour code. From this, key figures for quality control and process optimisation can be derived. Likewise, internal defects can be detected. The measured structures – including large defects – are transferred to FE models where loads are applied to check for significant impairments of application properties.
Frequently, attractive sculptures are printed as demonstrators for the capability of AM (Fig. 8 a). However, they are rarely suited for a quantitative comparison of dimensional tolerances. Instead, test geometries are proposed, for which CAD files can be downloaded on the internet [15]. These test structures are designed for the evaluation of thin walls, stairs, angles, needles, holes etc. within 3D-printed structures. As an example, Fig. 8 b shows an alumina test structure printed by vat photopolymerisation simultaneously checking several structural features.
The surface roughness of AM parts can be critical due to stress concentration and also for aesthetic reasons. It can be measured

by laser scanning microscopy nearly independently of the surface shape. At the HTL, in-house software is used to extract several key figures to estimate local stress concentration. As with CT data, the surface scans can be transferred to FE models, in which they are evaluated in terms of allowed stresses. If surfaces are not smooth enough or dimensional tolerances are very strict, machining is unavoidable. For that purpose, automated machining centres with five axes are available which can adjust freeform surfaces to set shapes.

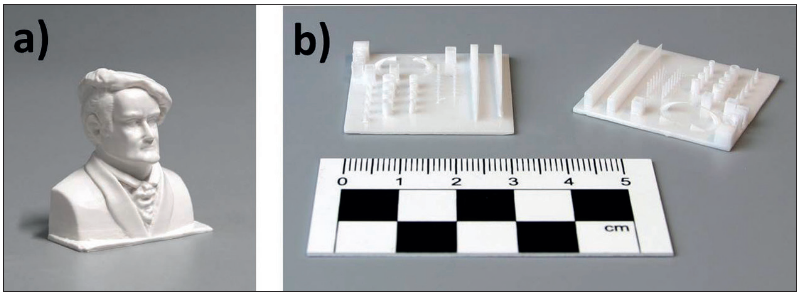

Fig. 8 a–b Alumina components made by vat photopolymerization: a) sculpture of Richard Wagner, a famous composer from the city of Bayreuth; b) standard test geometry

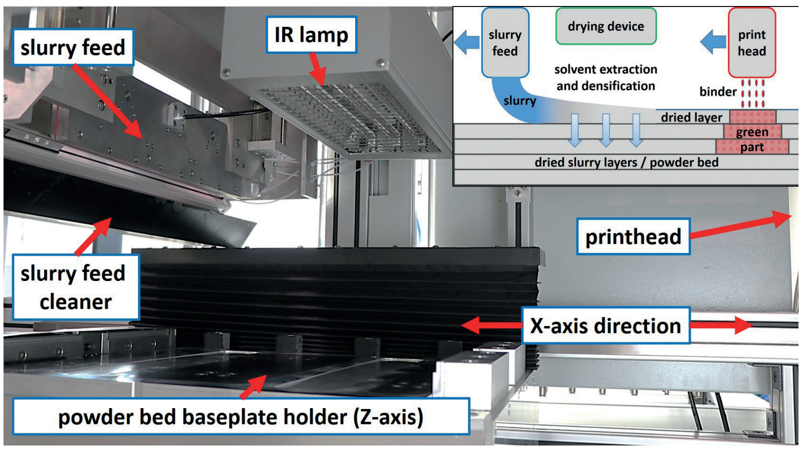

Fig. 9 Setup of a FFS printer at the HTL and schema illustrating the FFS printing process

6 Future trends

In addition to product quality, costs are a key performance indicator for use of AM in ceramic production. They are affected by throughput and production time. The bottle neck for throughput usually are the preparation of the printing process, the 3D-printing process itself and the post-processing of the parts. Since the subsequent heat treatment can be performed in large furnaces handling many parts in parallel, it usually doesn't affect throughput.

On the other hand, the limiting factor for production time often is the heat treatment, especially when long debinding steps are required. Production time is an important issue for production on demand, allowing new business models with higher added value.

Optimisation of debinding as outlined in chapter 4 is essential for utilising this potential. Avoiding costly finishing processes by net shape printing and sintering processes is a cost-relevant topic as well. The sintering optimisation methods indicated in chapter 4 can be used to minimise shape deviations. Other important cost factors are raw materials.

Today, feedstocks for 3D-printing are much more expensive than standard raw materials. Recycling of unused feedstock is an important cost factor in all powder bed based AM methods. Depreciation is another important cost factor in ceramic AM.

Typically, 3D-printers cost several hundreds of thousand euros. Nevertheless, there is a strong trend to smaller and cheaper devices allowing more flexibility. Many printers are already available in a price range of some thousands of euros. Both the costs and reliability of the printing process, as well as the pre- and post-processing can be significantly reduced by process automation and the integration of the respective equipment.

New AM methods arise with an increasing frequency. Among them, two step methods based on ceramic slurries are most relevant for ceramic production. Slurries enable small particles and particle mixtures and lead to relatively high green densities. Up to now, the highest resolution is obtained using two-photon polymerization [16] and highest homogeneity was measured with local electrophoretic deposition [17].

The latter requires a lot of work for practical use, yet. Layerwise Slurry Deposition (LSD) [2] and Free Flow Structuring (FFS) [18] are promising slurry based processes, which can be used for larger components. Fig. 9 shows an FFS-printer for free-flow-deposition of a ceramic slurry on a dried powder bed. LSD and FFS can be used for multi-material printing, e.g. by adding other materials via an additional print head.

Multi-material printing is also an advantage of ink jet printing processes, which showed rapid progress in terms of resolution [19]. However, AM of multi-material parts is limited by sintering, where a careful adaption of shrinkage properties is required. Otherwise problems occur, which are well known from the co-firing of multilayer structures. It is estimated that the initial enthusiasm and the current disillusion with respect to introduction of AM in ceramic production will be replaced by selective use of AM in customized production of small series. AM methods and printers can be carefully selected according to the specific production task. The growth rate of AM in ceramic production will be controlled by quality and cost of AM processes.

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