RESEARCH OF PROPERTIES OF CERAMIC COMPOSITE FIBER PRINTED USING FUSED DEPOSITION MODELING TECHNOLOGY

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This research article presents a comprehensive study on ceramic materials using Fused filament fabrication of ceramic (FFFC) for the production of test samples. The study investigates the effects of different production strategies on three types of ceramic materials: experimental ceramic composite fiber (e.c.c.f.)(composite material produced by STU FCHPT), Zirconium Silicate, and White Zirconia. The primary objective is to compare the production times of the different strategies and investigate their effects on the properties of the samples. This research is important because production time is a crucial factor in the manufacturing industry, and minimizing production time without compromising quality is essential. The study aims to explore the impact of various production strategies on the production time and properties of the test samples, such as hardness, porosity, and flexural strength. The experimental procedure involves printing test samples using a MakerBot METHOD X 3D Printer with FDM technology and varying the layer thickness, raster angle, infill density, and other production strategies. The study aims to identify the optimal production strategy for each ceramic material, which can significantly reduce production time while maintaining the desired quality of the parts. Overall, the research provides valuable insights into the optimal production strategies for different types of ceramic materials and contributes to the development of novel, cost-effective, and efficient manufacturing processes for ceramic composites.

KEYWORDS

COMPOSITE, ADDITIVE MANUFACTURING, FDM, CT, CERAMIC, STRATEGY

1 INTRODUCTION

Ceramic composites have gained significant attention in various industries due to their superior properties such as high strength, stiffness, and excellent resistance to extreme environments. However, the conventional manufacturing methods for ceramic composites are often complex and expensive, limiting their practical applications. As a result, additive manufacturing techniques such as Fused filament fabrication of ceramic (FFFC) have emerged as promising alternatives for the production of ceramic composites. The aim of this study was to investigate the influence of different production strategies on the quality of the printed parts, including the production time and selected properties. The investigated materials in this research include experimental ceramic composite fiber (e.c.c.f.), Zirconium Silicate, and White Zirconia. All produced samples were digitized by a Metrotom 1500 computed tomography in 2K resolution. This CT scanner is located in the Center of Excellence for 5 Axis Machining (CE5AM) at the STU MTF. These insights could contribute to the development of novel ceramic composite materials that are both cost-effective and efficient, revolutionizing the manufacturing processes of ceramic composites. FDM (FFFC) is defined by ISO/ASTM 52900 as the process of joining materials to produce parts from 3D model data, usually layer by layer, as opposed to subtractive or formative manufacturing methods. While various additive manufacturing processes, such as Selective Laser Melting (SLM), Direct Energy Deposition (DED), or Stereolithography (SLA), have been used for the production of composite parts, FDM has demonstrated significant potential in the production of high-performance ceramic composites. In summary, this research article aims to provide valuable insights into the production of ceramic composites using FDM technology. The outcomes of this research could contribute to the development of cost-effective and efficient manufacturing processes for ceramic composites, making them more accessible for a wide range of industries [Veteška 2021], [Yakout 2017].

2 COMPOSITE MATERIALS

Ceramic materials have emerged as a promising class of materials for use in 3D printing applications due to their unique properties, such as high strength, stiffness, and chemical stability. However, the brittle nature of ceramics presents challenges when used as standalone materials for 3D printing. To address this, ceramic composites have been developed, which combine ceramics with other materials to achieve superior mechanical properties. One approach to developing ceramic composites for 3D printing is to incorporate ceramic fibers into a polymer matrix, using a process known as Fused Deposition Modeling (FDM).

Previously, Fused Deposition Modeling (FDM) has utilized materials including plastics, metals, and ceramics. However, it is apparent that ceramics, which are inherently brittle, are unsuitable for being shaped into flexible and pliable wires as input material. Once the printing process is completed, which closely resembles the traditional FDM procedure, the printed ceramic component is treated to eliminate the binder and sintered to attain densification. The pioneer use of FDM for producing ceramics was documented by *Danforth* in 1995 at Rutgers University, where binder systems filled with Al_2O_3 and Si_3N_4 were utilized [Danforth 1995].

The potential uses of FDM lie in the production of functional electronic parts. *Allahverdi et al*. have effectively exhibited the creation of different shaped ceramic transducers utilizing FDM (as shown in Fig. 1), utilizing feedstocks consisting of polymers combined with piezoelectric ceramics such as lead-zirconate-titanate (PZT) and lead-magnesium niobate (PMN) composites [Allahverdi 2001].

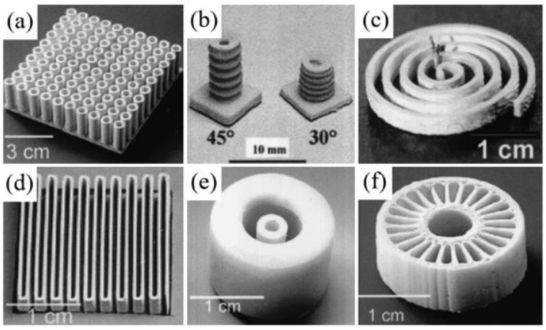

Figure 1. Different types of transducers made by FDC: (a) tube-array; (b) bellows; (c) spiral; (d) curved transducer; (e) telescoping; (f) radial actuators [Allahverdi 2001].

Accoriding to *Gasdaska* and *Jafari* new FDM techniques have been created for producing ceramics. *Jafari* et al. have effectively shown the use of four extruders in producing multilayer PZT sensor parts, which include a layer with two types of soft and hard PZT ceramics. By utilizing filaments loaded with 55-60 vol % of ceramic, comparable mechanical properties to other processing methods were achieved for creating dense structural Si_3N_4 parts via FDM [Iyer 2008].

The quality of ceramic parts produced using FDM depends on several process parameters, including rod width, layer thickness, building orientation, and raster angle, similar to conventional plastic materials. Surface roughness, homogeneity, dimensional accuracy, and mechanical properties are important factors that depend on these parameters. However, surface roughness is the main issue for FDM-produced ceramic parts, and the staircase effect is the primary problem associated with this technique. The staircase effect is a major disadvantage of FDM and is due to the size of the extruded filament, resulting in limited control in the z direction. This effect is commonly observed in printed ceramic parts [VL. Tsang 2004], [Chen et al. 2017], [Azlin 2021].

Several research studies have been conducted to explore the properties of ceramic composite fibers printed using FDM technology.

Zhant et al. in their research focused on advances in FDM 3D printing of polyamide-based composites and the properties of the printed parts as well as their practical or potential applications are highlighted. The particular emphasis is placed on the formation and the performance of polyamide/polymer blends, inorganic particle reinforced polyamide composites, and fiber reinforced polyamide composites [Zhang 2021].

Another study conducted by *Ryder* et al. This scientific study explores the fabrication of polymer-metal composites using fused deposition modeling (FDM) and evaluates the physical and mechanical properties of these novel materials. Specifically, the study focuses on the use of an acrylonitrile butadiene styrene (ABS) - 420 stainless steel (SS) composite system with varying weight percentages of SS powder (10, 15, and 23 wt%). The study develops a new methodology for creating the composites, extruding the resulting materials into composite filaments which were then used to print test specimens. To evaluate the effects of different print conditions, the study employed tensile testing, modulated differential scanning calorimetry, and scanning electron microscopy to characterize the composite materials. The study found that FDM is a feasible method to produce ABS-SS composites, which can maintain or even enhance the mechanical properties of the base polymer, while adding increased functionality [Ryder 2018].

This study explores the impact of different production strategies on the properties of the printed parts and analyzes the microstructure of the printed parts to ascertain the fiber distribution and orientation within the matrix. The investigation includes three different types of ceramic materials, including an experimental ceramic composite fiber abbreviated as e.c.c.f., Zirconium Silicate, and White Zirconia. The study is expected to provide valuable insights into the potential of FDM technology for the production of high-performance ceramic composites.

3 MATERIALS AND METHOLOGY

Additive manufacturing typically commences with either a designed 3D model or a scanned one. Slicing software is then utilized to convert the point cloud into an STL file. This file is then transmitted to the additive manufacturing machine, which uses successive layering of materials to produce the desired object. The process of additive manufacturing is illustrated in Figure 2.

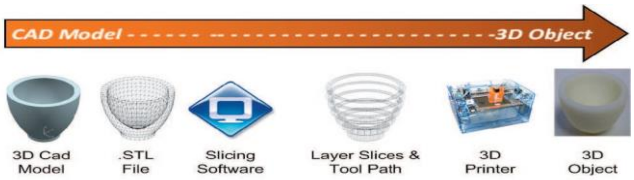

Figure 2. The steps of additive manufacturing [Taneva 2015]

Fused Filament Fabrication of Ceramics (FFFC) is a 3D printing process that involves the use of a ceramic-filled filament, which is melted and extruded layer by layer to create a ceramic object. FFFC has become an attractive option for producing complex ceramic shapes due to its relatively low cost and high degree of design flexibility.

In FFFC, the ceramic powder is typically mixed with a thermoplastic binder material to create a filament that can be fed through a conventional desktop 3D printer. The filament is heated to a temperature just below the melting point of the ceramic particles, allowing them to fuse together and form a solid object. The advantages of FFFC include its low cost, its ability to produce complex shapes with ease, and its potential for Rapid Prototyping. However, the process is not without its challenges. The high firing temperatures required to fully sinter the ceramic material can cause significant shrinkage and warping, and the surface finish of the final product may not be as smooth as with other ceramic manufacturing techniques. Despite these challenges, FFFC has shown promise as a viable technique for producing complex ceramic components for a range of applications, including biomedical implants, aerospace components, and electronics [Veteška 2021]. Scheme of Fused Filament Fabrication of Ceramics (FFFC) is shown on Fig. 3.

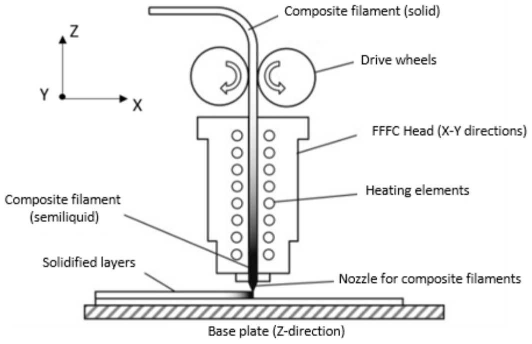

Figure 3. Fused Filament Fabrication of Ceramics (FFFC)

In this study on authors used two types of ceramic materials: experimental ceramic composite fiber (e.c.c.f.) and Zirconium Silicate

ECCF was manufactured by a wet route from precursors including mullite-based ceramic powder mixture, aliphatic acid - based surface adhesion modifier (AAK Poland Sp.z o. o., Poland), thermoplastic polymer Kuraray-POVAL (Kuraray Europe GmbH) and glycerol-based softener (Centralchem s.r.o., Slovakia) as the plasticizing agent. The ceramic powder mixture containing mullite-forming phases and a sintering agent (Table 2) was homogenized in a porcelain mortar prior to composite production. The polymer was mixed with deionized water and the ceramic powder, surface adhesion modifier and softener were added during vigorous mixing to ensure homogeneity of resulting material. The material was stirred for 60 min and spread on a glass plate using the Doctor blade method, dried and cut to smaller sections which were then extruder into the filament in single-screw extruder PL 2000 (Brabender, Germany) equipped with filament extrusion head at extrusion head temperature of 200 °C. The ceramic powder loading was set to 65 wt% (50 vol%). More details about components of the ceramic powder mix and processing parameters, are available in Patent Application nr. 50062-2019 (Industrial Property Office of The Slovak Republic). The filament diameter was checked using a digital caliper to be of nominal diameter of 1.75 mm ± 0.07 mm – very similar to the commercial grade filaments. The ovality/circularity of the filament was checked by the means of the optical microscopy and subsequent image analysis using the circularity tool in the software Fiji, version 1.53t [Schindelin 2012].

Zirconium silicate is a widely used material for 3D printing of ceramics due to its excellent mechanical and physical properties. It has a high melting point of approximately 2500°C, making it suitable for high-temperature applications. Zirconium silicate is also known for its high strength and toughness, as well as its resistance to wear and corrosion. These properties make it an ideal material for use in biomedical and dental applications, such as bone replacement and dental implants.

In conclusion, ceramic composites have emerged as a promising class of materials for use in 3D printing applications. By incorporating ceramic fibers into a polymer matrix via FDM, it is possible to achieve improved mechanical properties while maintaining the processability of the thermoplastic material. The use of other types of reinforcements, such as graphene oxide, has also been investigated. As the technology of 3D printing continues to advance, ceramic composites printed via FDM have the potential to revolutionize the manufacturing of complex ceramic parts, with applications in fields such as aerospace, biomedical, and electronics.

To ensure consistency in the initial printing conditions, a separate extruder was utilized for each filament examined. This was due to the microscopic image of the flow section of the nozzle, which is depicted in Figure 4. The images demonstrate a detailed view of the nozzle's flow section before and after fifteen meters of the experimental ceramic composite filament were extruded. As a result of this extrusion, the diameter of the nozzle's flow section increased by 0.09 mm, from the original 0.4 mm to 0.49 mm. This observation underscores the fact that if a single extruder were used to extrude all ceramic composite filaments, it would be impossible to maintain the same initial conditions

Three different infill manufacturing strategies were used to print samples with. Linear, Hexagonal and Morrocan star Fill. Manufactured samples were then scanned via Zeiss Metrotom 1500 CT Scanner. The precision with which the samples were produced and the deviations in samples made from various materials and with different infill strategies were evaluated by creating color maps of the deviations. Furthermore, to further examine the variability of the different fabrication strategies, a section through the middle of each strategy was generated using the VGStudio MAX software, since the surface of the samples was consistent in all cases. This highlighted the differences in the various fabrication strategies.

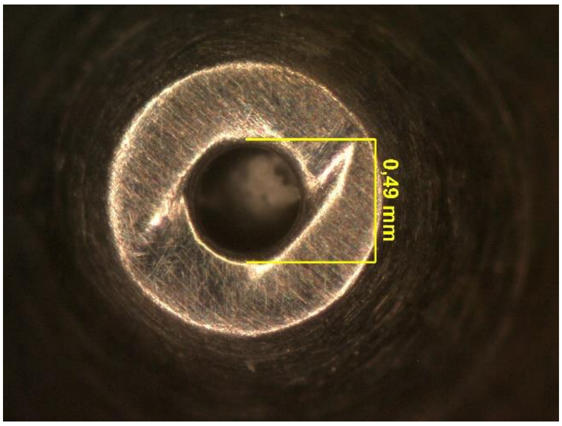

Figure 4. Flow portion of the nozzle

Upon inspection of the different fabrication strategies, it is evident that the experimental ceramic composite filament samples are of higher quality in terms of surface texture and porosity compared to the Zirconium Silicate samples, as observed visually. However, both materials exhibit significant surface imperfections compared to the nominal 3D model. To gain a better understanding of these imperfections, a color deviation map was generated using VGStudio MAX software to illustrate the dimensional deviations of each fabrication strategy from the 3D model. Figure 5 displays the deviation map of the linear manufacturing strategy of the experimental ceramic composite filament, while Figure 6 shows the deviation map of the Zirconium Silicate filament.

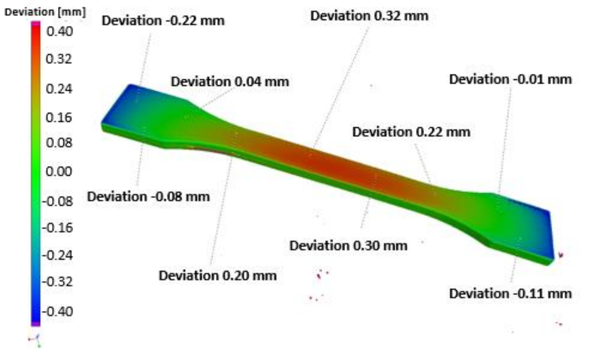

Figure 5. Deviation map of the linear manufacturing strategy (e.c.c.f.)

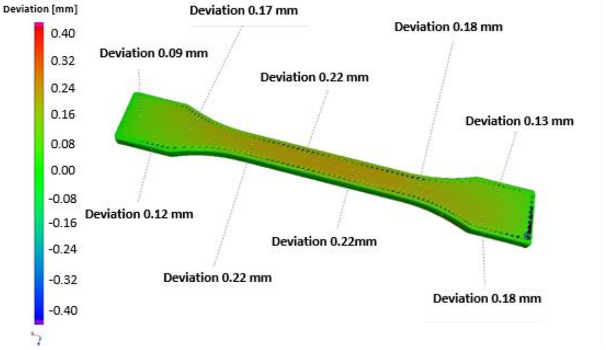

Figure 6. Deviation map of the linear manufacturing strategy of Zirconium Silicate fiber

The Zirconium Silicate filament samples exhibit greater accuracy compared to the 3D model in contrast to the experimental ceramic composite filament samples. Nonetheless, the accuracy of the samples made from the experimental ceramic composite filament could be greatly improved if the bending of the samples could be avoided after the printing process. This claim is supported by the Moroccan Star Fill manufacturing strategy, where the deviations in the experimental ceramic composite filament samples were primarily within the green values of the deviation column when compared to the 3D model. Additionally, the experimental ceramic composite filament yields a superior surface quality, as indicated by the following comparison table. Table 1 compares the deviations of individual points in identical parts of the investigated samples, with ESSF denoting the experimental composite ceramic filament and ZS representing the Zirconium Silicate filament.

Table 1. Deviations of Points

	ECCF	ZS	ECCF	ZS	ECCF	ZS
	Linear		Hexagonal		Maroccan Star Fill	
Deviation 1 [mm]	-0.22	0.09	0.20	0.18	0.15	0.05
Deviation 2 [mm]	0.04	0.17	0.02	0.27	0	0.20
Deviation 3 [mm]	0.20	0.22	-0.09	0.30	-0.04	0.25
Deviation 4 [mm]	0.32	0.22	-0.16	0.30	-0.02	0.31
Deviation 5 [mm]	0.30	0.22	-0.17	0.25	-0.03	0.23
Deviation 6 [mm]	0.22	0.18	-0.12	0.25	0.04	0.16
Deviation 7 [mm]	-0.01	0.13	0.08	0.16	-0.08	0.13
Deviation 8 [mm]	-0.11	0.18	0.09	0.18	0	0.11
AM|α| [mm]	**0.19**	**0.17**	**0.11**	**0.23**	**0.04**	**0.18**

Fig. 7 shows the homogeneity of the filament deposition.

Figure 7. Section through the middle of linear manufacturing strategy (ECCS top, ZS bottom)

Printing for both materials took the longest among the production strategies studied. A section through the centre of the hexagonal manufacturing strategy Fig. 8 shows the often regularly recurring porous regions.

Figure 8. Section through middle of hexagonal manufacturing strategy (ECCS top, ZS bottom)

In the case of the hexagonal production strategy, the printing time per sample was shorter compared to the linear strategy in all cases studied. However, when looking at the nozzle during the printing process, it appeared to have travelled a significantly longer path compared to the linear manufacturing strategy, however, the time to print one sample did not suggest this. Thus, the aforementioned porous regions were responsible for the reduction in the production time per sample of between two and five minutes. Similar to the hexagonal production strategy, the porous regions are also found in the case of the Moroccan Star Fill production strategy Fig. 9.

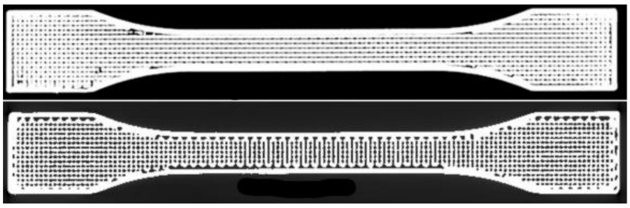

Figure 9. Section through middle of manufacturing strategy Maroccan Star Fill (ECCS top, ZS bottom)

The debinding of the composite materials and the subsequent sintering of the samples was performed using a two-stage thermal procedure using furnaces from Clasic a.s., Czech Republic – a simple resistance-heater furnace type Clasic 1015 and the superkanthal furnace Clasic 0518. For the experimental ceramic composite fibre samples, the debinding process was carried out according to a proprietary thermal programme developed at the DIM FCHPT STU with the final temperature of the process being 1200 ° C. The samples were initially placed in a sand ballast powder (see Fig. 10) and subjected to an debinding regime with the average heating rate of ca. 0.2 ° C per minute. Subsequently, the samples were removed from the debinding furnace and transferred to a ceramic plate in the high-temperature sintering furnace and subjected to the final sintering process using the heating rate of 10 ° C per minute up to the maximum sintering temperature of 1400 ° with a holding time of 1 hour.

Similarly, for the Zirconium Silicate fiber samples, the samples were placed in the sand ballast powder (see Fig. 10) following the same procedure as that for the experimental ceramic composite fiber. However, the drying process for this fiber did not exceed 1232 °C, as per the parameters specified by the manufacturer. After removing the binder, the samples were sintered at 1400 °C with a residence time of 1 hour.

At each stage of the process, the samples were weighed to determine the percentage weight loss relative to the printed sample. For the experimental ceramic composite fibre, the weight loss exceeded 40% in most cases. Similarly, for the Zirconium Silicate filament, the weight loss for each sample was more than 42% compared to the printed sample.

Figure 10. Samples placed in the sand ballast powder

Another noticeable change was in the case of the colour of the samples after extrusion compared to the samples after sintering for both filaments. While in the case of the experimental ceramic composite fibre the samples remained white after sintering, as declared by colleagues from the OAM FCHPT STU, in the case of the samples from Zirconium Silicate fibre there were spots of brown colour on the sintered samples (for unspecified reasons), although the manufacturer of the fibre does not declare anything similar. The intention was to conduct tensile strength testing on the samples, but the testing process encountered several issues with clamping. The primary issue was the slipping of samples on the contact surface during clamping, despite applying pressure with metal jaws. The sample slipped between the jaws, and when higher pressure was applied, the sample was crushed, as shown in Fig. 11. This problem was observed in samples of both materials.

Figure 11. Crushed specimens

Post-sintering delamination was the cause of specimen crushing when clamped in the metal jaws of the test apparatus as can be seen in Fig. 12.

Figure 12. Delamination of specimens

It seems that the inflation of the specimens in the clamping area caused them to deflect from the clamping axis when one of the jaws was tightened. This, in turn, led to specimen fracture before the actual tensile test could begin.

Figure 13. Inflation of specimens

The sintering process resulted in deformation of the samples in various directions, which made it difficult to perform the tensile strength testing. The deformation caused the samples to deflect from the clamping axis when placed in the testing apparatus. As a result, the jaws could not properly clamp the samples, and subsequent tightening of the jaws led to fracture of the specimens. Thus, it was not possible to perform tensile strength testing on the sintered specimens.

4 EVALUATION OF EXPERIMENTS

The experiments were carried out on samples made of ceramic composite fibers. The first part of the experiments shows how demanding printing using ceramic composite filaments is on the mechanical parts of the 3D printer. It can be seen that even in a short period of time, wear and tear of the components occurs, as in the case of increased nozzle flow, or their malfunctioning, as was the case with the extruder feed wheels. The following experiments show that the printed samples often do not achieve the required accuracy.

5 CONCLUSIONS

For the still developing field of additive manufacturing, composite materials are a significant asset. The use of composite materials makes the technology more competitive compared to other previously commercial technologies. When using an additive technology such as FDM to print ceramic components from ceramic composite filaments, several factors must be considered. The first factor is the material loss during the sintering process, which is essential to achieve the final ceramic part. Ceramic composite filament parts have a relatively large material loss, but the percentage variation is comparable in the case of repetitive printing. Material loss must therefore be taken into account during modelling, but the question remains how accurately it can be estimated without first testing the composite filament, and whether it is even possible to print dimensionally accurate parts using such filaments. If further testing proves that printing dimensionally accurate parts is not possible using ceramic composite filaments, the modelling process would need to take into account not only the material loss, but also the enlargement of the part for additional machining. Since printing using ceramic composite filaments has a demonstrable impact on the mechanical parts of the printer, the second factor is the economic impact on the resulting part. On the economic side, it is also necessary to consider whether the printer allows the use of filaments from alternative manufacturers or only allows the use of filaments recommended or directly produced by the printer manufacturer. It may happen that even though the printer is presented as open source and therefore any filament designed for the technology can be printed on it, the filament will not be compatible with the printer. However, such a thing is not foreseeable, unless the printer manufacturer excludes printing any filament, yet does not offer all types of filament, as was the case with ceramic composites. Printing a part can thus become significantly more expensive due to the need to change the filament because of incompatibility with the printer. The third factor is the manufacturing strategy. By choosing an appropriate manufacturing strategy, it is possible to increase the quality of the part, but also to reduce the production time. By choosing an appropriate manufacturing strategy, the impact on the part in the form of deformation after the sintering process will be reduced. In this case, however, the question remains whether and what impact a change in manufacturing strategy will have on the resulting already sintered specimen in terms of strength. As not all of the mechanical tests are suitable for testing ceramic components after the sintering process, precisely because of the deformation that occurs after the process.

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