Published in final edited form as:

Ceram Int. 2017 October 15; 43(15): 11766–11772. doi:10.1016/j.ceramint.2017.06.012.

Short fiber reinforced 3d printed ceramic composite with shear induced alignment

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Abstract

This paper accounts for utilization of shear induced alignment method during ceramic stereolithography. Lateral oscillation mechanism, combined with 3d printed wall pattern, was employed to generate necessary shear to align fiber in desired direction. First, semicircular channel pattern was printed to assess the effect of difference between wall direction and oscillation direction on the fiber alignment. Then, flexural strength of ceramic matrix was tested with nickel coated carbon fiber and ceramic fiber reinforcements. The results demonstrated that the shear induced alignment further improves the flexural strength compare to randomly distributed samples. Flexural strength of aligned samples with 1.0 wt% carbon fiber loading was improved by ~90% compared to randomly orientated samples and by ~333% compared to unreinforced samples. Finally, fracture surface morphology of the flexural strength test specimens was evaluated. The main fracture mechanism was observed as fiber pull-out.

Keywords

Stereolithography; Composites (B); Shaping (A); Sintering (A)

1 Introduction

Traditional ceramic forming techniques, such as slip casting, shell casting, dry pressing; are not capable of producing complex parts with high accuracy without expensive mold or fixtures, or time consuming post-sinter machining [1]. Therefore, there is a demand and interest for alternative methods to produce complex ceramic pieces without the use of expensive tooling. Three dimensional printing (3DP) methods of ceramics and ceramic composites can directly produce complex parts from 3D computer-aided design (CAD) files without the use of any tooling. Fused deposition modeling (FDM) [2], selective laser sintering (SLS) [3], ceramic jet printing (CJP) [4], and stereolithography (SLA) [5] are some

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of the common 3DP methods which are used for additive manufacturing of ceramic and ceramic composite materials. During these processes, 3D complex objects are fabricated by rendering each layer according to CAD file and stacking them on each other.

SLS method involves the use of high power laser beam to bond ceramic particles to each other locally [6, 7]. Localized temperature gradient during sintering process can generate high internal stresses which might cause lots of defects and micro cracks. FDM, CJP, and SLA ceramic processing methods use a mixture of a ceramic powder and a polymeric binder to fabricate green parts without high energy input. Fabricated green part must be sintered to remove polymeric binder and to fuse the particles. However, extrusion of precursor filament is often disturbed by buckling failures and powder volume fraction is limited by extrusion pressure at the nozzle for FDM and CJP processes [2]. On the other hand, SLA of ceramics utilizes photosensitive resin as a binder to produce the green parts without any nozzle and high energy laser beam. Without any nozzle and high energy input, internal stresses, buckling failures, and extrusion pressure are avoided. Therefore, SLA method was used for this study.

The use of pure ceramic materials as a structural material has been limited, due to its overall low toughness and thermal shock resistance [8, 9]. As a result, additive of reinforcement such as whiskers [10, 11], short fibers [12, 13], particles [1, 14], and nanotubes [15, 16] into ceramic matrix attracted a lot of research interest in order to enhance desired properties. These enhancements are influenced by the material, dimensions, and orientation of the reinforcement [12, 17]. Therefore, fiber alignment can be used to tailor the material properties of 3D printed composite pieces, such as mechanical [18] and electrical [19]. We demonstrated shear induced alignment during SLA 3DP for reinforced polymer composites (RPC) at our previous study [20]. In this work, same approach was utilized for 3DP of ceramic composites. Micro scale alumina, silica, and nickel coated carbon fibers (NCCF) were used as reinforcement. Respond of reinforcement to patterned wall direction and enhancement of flexural strength of the composite material with fiber orientation were discussed.

2. Materials and Methods

2.1. Slurry preparation

The ceramic photocurable resin used in this paper is a mixture of both ceramic powders and liquid photocurable resin. Powder part contains silica powder, Feldspar powder, Kaolin clay powder and Ball clay powder (all from Axner Co.), and liquid resin part is a mixture of Laromer 8765 (BASF Co.), Laromer HDDA (Hexane-1,6-diol diacrylate, BASF Co.), and lauryl acrylate (Esstech Inc.). Irgacure 754 (a mixture of Oxy-phenyl-acetic acid 2-[2-oxo-2-phenyl-acetoxy-ethoxy]-ethyl ester and Oxy-phenyl-acetic acid 2-[2-hydroxy-ethoxy]-ethyl ester) and Irgacure 4265 (a mixture of Diphenyl (2,4,6-trimethylbenzoyl)-phosphine oxide and 2-Hydroxy-2-methyl-1-phenyl- propan-1-one)) from BASF Co. was used as photoinitiators. 0.2 wt%. Tinopal OB (BASF Co.) is used as a UV absorber. 60 wt% of silica content was added within the resin as the ceramic filler and mixed with shear mixer for 24 hours to provide homogeneity. During the mixing process, the viscosity of the slurry was reduced as result of shear thinning behavior of the fluid. After the mixing, if the slurry was

not used for 8–10 hours, ceramic fillers would settle to the bottom of the slurry as sediment and then slurry would require remixing.

While silica particles were used as the matrix, NCCF, alumina, and silica fibers were used as fiber reinforcement for the ceramic matrix composite. Carbon fiber is inclined to oxidation at temperature 500 °C and higher, as a result it requires protection from atmosphere to preserve its superior properties [21]. Nickel coating was used as the protection in our study. NCCF with 7 µm diameter and 1 mm mean length were obtained from Tfpglobal. Alumina and silica fibers with 2.5–3 µm diameter and 2.7–3.2 mm mean length have been obtained from Zircar Ceramics. NCCF were added in UV-curable slurry with the ratios of 0.25, 0.5, 0.75, and 1 wt% and ceramic fibers were added with the ratios of 2.5, 5, 7.5, and 10 wt% right before 24 hour shear mixing.

2.2. SLA apparatus and linear oscillatory mechanism

Production of ceramic samples and fiber alignment rely on SLA based curing system and linear sliding mechanism. As the systems shown in the schematic (See Fig. 1), the SLA curing system consists of a platform, a slurry tank, and a projection unit, while linear sliding mechanism consists of a linear stage which is connected to the slurry tank. In the projection unit, light is emitted through the optical lenses onto DMD chip to be patterned and reflected towards the projection lens and then refocused onto bottom of the slurry tank. Refocused image on the bottom of the slurry tank has the pixel size of 25 µm. Micro wall patterns are produced to generate shear induced alignment of short fiber reinforcement into the desired orientation with similar manner with our previous study for short nanofiber reinforced polymer [20]. Then linear sliding mechanism, which has a resolution of 10 µm, is actuated to generate oscillatory simple shear flow in the produced channels between the micro walls. Under simple shear flow condition, wide channels result with skin-core morphology which is observed as fiber alignment in the flow direction around the wall and perpendicular alignment at the center [22, 23]. Disappearance of such morphology was documented by the reduction of the channel thickness with adequate shear rate. In micro injection molding applications, core-free morphology was achieved with channel thicknesses lower than 200 µm [24–26]. In order to attain similar results, layer thickness of SLA curing system was set as 100 µm. Linear oscillation with 100 µm layer thickness was used to generate simple shear flow and to achieve alignment of fibers onto x–y plane. Fabrication of wall pattern provides different flow direction, and so it was used further alignment of fibers along the channel direction of the wall pattern (See Fig. 2).

2.3. Debinding and sintering process of green body

3D printed green bodies were debinded at 315 °C for 1 h with the heating rate of 1 °C/min and polymeric binder was removed. After the removal of the binder, samples were heated at 1035 °C with the heating rate of 1.85 °C/min, then at 1250 °C with the heating rate of 2.5 °C/min for a continuous sintering process. Finally sintering process was employed at 1250 °C for 1 h.

2.4. Characterization

In the study, silica particles were used as matrix of the samples and they were reinforced with various fractions of NCCF (0.0, 0.25, 0.5, 0.75, and 1.0 wt%) and short ceramic fibers (0.0, 2.5, 5.0, 7.5, and 10.0 wt%). Standard bar shaped samples ($2 \times 1.5 \times 12$ mm) were fabricated by the method previously described. 3-point bending tests were performed for flexural strength analyzes by using Ernest Fullam load frame (EFLF) with a micro bend fixture at room temperature with a speed of 0.5 mm/minute. Flexural strength of the samples was calculated according to equation below:

$$\sigma_F = 3FL/2bd^2 \quad (1)$$

where, σ_F is the flexural strength, F is the maximum load, L is span length which is distance two lower anvils, b is sample width, d is sample depth. 15 samples were tested for every fraction and material options. Additionally the microstructure and fracture surface were observed by scanning electron microscopy (SEM).

3. Results and Discussion

3.1. Fiber alignment

To investigate the influence of wall direction on fiber alignment, ceramic slurry was subjected to shear rate profile with maximum shear rate of 250 s^{-1} (see Fig. 3). Before the shear alignment, the distance between the platform and slurry tank was set for 100 μm and a wall pattern was printed to guide the fibers in the wall direction. Later linear oscillator was actuated to generate the time-dependent shear rate.

Linear oscillation generates simple shear flow and the flow is redirected by the walls of pattern to obtain fiber orientation different than oscillation direction. The direction of the flow in the channel influences the orientation of the short fibers exist in the slurry. Therefore, it can be used to reposition the fibers and stimulate alignment in the different direction of applied external force. Fig. 4 shows the distribution and alignment of the fibers in the green parts as microscope images. Effect of the oscillation is clearly demonstrated to align the fibers. Also, Fig. 5 shows the fiber orientation in different regions of the semicircular channel pattern. Fig. 5(a–f) are the microscope images of solidified pattern after linear oscillation. The orientations of the fibers were redirected by the wall pattern to align parallel to wall direction. Fig 5(a) and (b) show that the wall pattern aligns the fibers along the wall direction even when the angle between the wall and oscillation direction is about 45° .

3.2. Sintering and microstructure

The green part and some sintered complex 3d printed ceramic samples are shown in Fig. 6. The dimensions of the green parts were reduced after the sintering process. The shrinkage of the parts was around 5–7% in all directions. Such dimensional change corresponds with 15–20% total volumetric shrinkage of the sintered parts.

Fig. 7 shows The SEM images of the sintered 3d printed silica and silica composite samples. The microstructure of the sintered unreinforced silica sample surface is shown in Fig. 7a, while Fig. 7b, c, and d show the microstructure of sintered reinforced silica composite sample surfaces. Fig. 7b demonstrates that fibers in randomly orientated samples are not well dispersed. In Fig. 7c and d, it can be seen that carbon and ceramic fibers were aligned in the direction of the wall with the oscillation in similar results to each other. In contrast to randomly aligned samples, aligned fibers were found to be well dispersed. However, there was no visible difference for any influence of sintering process on the fiber orientation. The reinforced ceramic samples display anisotropy in the microstructure as a result of the fiber distribution in X–Y plane. Similar results can be obtained with other ceramic composite fabrication methods which cannot be utilized during 3d printing [27–29].

3.3. Flexural strength of the samples

Fig. 8 shows the flexural strength of the 3d printed ceramic composites by different weight content of carbon and ceramic fibers. Main factors which effect on the flexural strength are sintering temperature and devitrification process of the fused silica. The flexural strength of 3d printed silica sample without any reinforcement was obtained as 12.6 MPa. This result is in agreement with some other silica studies [30, 31]. The flexural strength of silica matrix composite increased with the carbon fiber and ceramic fiber content (See Fig. 8). The addition of 10.0 wt% silica fiber reinforcement enhanced the flexural strength of the samples by 19.8% to 15.1 MPa, while the addition of 10.0 wt% alumina fiber reinforcement improved the flexural strength by 70.7% to 21.5 MPa. The addition of 10.0 wt% half and half mixture of silica and alumina fiber reinforcement increased the flexural strength by 47.6% to 18.6 MPa. On the other hand, 1.0 wt% NCCF reinforcement into silica matrix provided 127.8% enhancement of the flexural strength to 28.7 MPa. The contribution of alignment on to the flexural strength can be understood by the comparison of the aligned and non-aligned samples in Fig. 8. The fiber alignment along the length of the test samples improved the flexural strength for all reinforcement types. Improvements were by 26.2% to 18.4 MPa with 10.0 wt% silica fiber, by 95.2% to 33.5 MPa with 10.0 wt% alumina fiber, by 78.6% to 28.5 MPa with 10.0 wt% half and half mixture of silica and alumina fiber, and by 204.8% to 54.5 MPa with 1.0 wt% carbon fiber. These improvements on flexural strength with increasing fiber alignment and content and better results with carbon fibers than ceramic fibers were expected based on general understanding of composite.

3.4. Morphologies of fracture surface

The fracture morphology of the investigated test samples with NCCF is shown in Fig. 9 by SEM images of the fractured surface. Fiber reinforced composite materials mainly involves matrix failure, the fiber/matrix (F/M) interface debonding, fiber failure, fiber pull-out, and delamination. When a crack grows and spreads in the matrix, process can trigger a fiber failure or F/M interface debonding. The F/M interface debonding can also result with fiber pull-out [32, 33]. In the case of our study, the layers of 3d printing process were not visible or detectable on the fracture surface. There was also no evident delamination, layering, or any crack propagation between 3d printed layers. The fracture surface consisted of both fiber pull-out and fiber failure as shown in Fig. 9. However, fiber pull-out was more prevalent than fiber failure. Fiber pull-out points out that the F/M interfacial bond in the samples were not

lasting enough to sustain to result until fiber failure takes place. Improvement of the interfacial bond can further improve the strength of the samples.

4. Conclusions

A DLP based SLA device, incorporated with the shear-induced fiber alignment apparatus was utilized to fabricate fiber reinforced silica matrix composites. 0.0–1.0 wt% NCCF and 0.0–10.0 wt% alumina, silica, half and half mix of alumina and silica were used as reinforcement for the silica matrix. The shear-induced fiber alignment as a result of oscillation was demonstrated. Additionally, printed wall pattern, such as channels with circular path, was shown to reorient the fibers into different directions from the oscillation direction. Up to 45° difference between alignment and oscillation direction was successfully achieved during the study. Both ceramic fibers and NCCF responded to the alignment process similarly and got aligned along the wall direction.

The EFLF was employed for the flexural strength analyzes of the samples. Flexural strength test indicated the expected increase of the strength along the alignment direction with the shear induced alignment of the reinforcement. Alignment improved the flexural strength further up by 26.2%, 95.2%, 78.6%, and 204.8% with silica, alumina, alumina-silica mix, and NCCF with used maximum weight fractions, respectively. Both fiber failure and fiber pull-out were existed in the fracture surface, but fiber pull-out was the main fracture mechanism.

In summary, shear induced alignment with oscillation and wall pattern was shown to be also viable for ceramic applications of SLA. The method can be utilized for production of anisotropic ceramic pieces with different fiber orientation and pattern on each layer.

Supplementary Material

Refer to Web version on PubMed Central for supplementary material.

Acknowledgments

This research was partially supported by National Science Foundation (NSF) grants CBET 1264808, CBET-1067502, DMS 1516236, IIP 1611718, National Institutes of Health (NIH) grants EB015105, R01HL131750, The Pennsylvania Infrastructure Technology Alliance (PITA) program and Alternatives Research & Development Foundation (ARDF).

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Figure 1.
System setup of SLA Ceramic 3DP with sliding mechanism.

Figure 2. Fiber alignment process: (a) Fiber alignment in a simple shear flow as a result of linear oscillation; (b) fiber alignment in a 3d printed wall pattern.

Figure 3.
The time dependent-shear rate profile which was used during fiber alignment process.

Figure 4. Microscope images of fiber alignment and dispersion within 3D printed green part (scale bars are 500 μm): (a) Microscope image of randomly orientated nickel coated carbon fiber; (b) microscope image of aligned nickel coated carbon fiber.

Figure 5.

Microscope images of fiber orientation distribution along the semicircular channel with different wall angles with respect to oscillation direction: (a) 0° ; (b) 45° ; (c) 5° right before a quarter curve; (g) Semicircular channel design with location of microscope images of (a–f). Red letters with dashed rectangles indicates the location microscope images are taken. Red arrows on microscope images show the direction of the wall at the local region.

Figure 6.
3d printed complex green and sintered parts: (a) 3d printed green turbine blade; (b) sintered ceramic turbine blade; (c) Sintered complex mesh structure; (d) Sintered complex shell structure.

Figure 7. SEM images of sintered 3d printed ceramic and ceramic composite samples: (a) SEM image of unreinforced silica sample; (b) SEM image of randomly orientated nickel coated carbon fiber reinforced silica composite; (c) SEM image of aligned nickel coated carbon fiber reinforced silica composite; (d) SEM image of aligned ceramic fiber reinforced silica composite.

Figure 8.

The Flexural strength of random and aligned reinforced silica matrix composites with different concentrations and materials; (a) Flexural strength values with different ceramic fiber reinforcement ; (b) Flexural strength values with nickel coated carbon fiber reinforcement.

Figure 9. Fracture surfaces of the nickel coated short carbon fiber reinforced ceramic composite samples; (a) 180 $\times$ magnification; (b) 250 $\times$ magnification.