Fabrication and Static Bending Properties of 3D Printed Zirconia Part via Digital Light Processing Method

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Fabrication and static bending properties of 3D printed zirconia part via digital light processing method

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Abstract

Zirconia is widely applied as an implant due to its excellent biocompatibility and mechanical properties such as high hardness and extraordinary resistance to wear and corrosion. However, these outstanding mechanical properties make it challenging to fabricate Zirconia into complex shapes using conventional manufacturing techniques. In the current study, the digital light processing method was used to manufacture the Zirconia part. Its mechanical property was evaluated via a three-point bending test with digital image correlation and fractography analysis. The 3D-printed Zirconia sample had a relative density of approximately 98.8% and a Vickers hardness value of 1128 HV. The flexural strength under parallel and vertical bending loads (with respect to the printing direction) were 56.63±3.97MPa and 70.98±6.62MPa, respectively. Surrounding by a few dense layers, the interior of the sintered sample was interlaced with needle-like and winding cracks. Under the three-point bending, the cracks initiated at the bottom surface due to the tension effect and propagated faster along the width direction than the thickness direction. There was a large area of cleavage morphology in the dense boundary layers, whereas the plastic fracture mode also appeared in the interior of the sintered samples. The digital light processing method is expected to be comparable to other advanced ceramic processing techniques for fabricating spatial lattice structural products.
Keywords: Zirconia; Digital Light Processing; Microstructure; Flexural Strength; Digital Image Correlation; Fractography
1. Introduction

Ceramic materials are widely used in biomedical engineering, chemical industry and aerospace because of their excellent biocompatibility, wear resistance, heat resistance and corrosion resistance [1]. However, traditional manufacturing technology (gel casting, slip casting, dry pressing, injection molding, tape casting, and so on) is challenging to make ceramic materials into sophisticated shapes. As a result, it cannot take full advantage of their outstanding characteristics. In addition, there are particular issues in the conventional processes [2]: (1) The shaping process of complex parts highly depends on the mold; (2) Sintered bodies often need additional mechanical post-processing; (3) Certain unique shapes (e.g., inner cavities/pores/groove) are difficult to generate using traditional ways. Recently, it has been of great interest to use the Additive Manufacturing (AM, also known as 3D printing) [3] technique in the ceramic industry to sidestep the issues mentioned above and could save up 80% of the fabrication cost of the ceramic parts [4].

Usually, AM is classified according to the material statement that is used (powder materials, solid materials, and liquid materials) [5] and the material accumulating (indirect, direct) [3]. However, the implementation of AM in the ceramic part fabrication has been much slower than in the polymer and metal parts fabrication due to the low surface quality and mechanical properties of ceramic parts fabricated by the available AM method compared to conventional methods [6–8]. Among the various AM techniques that be used for ceramic parts fabrication (three-dimensional printing (3DP) [9], stereolithography (SLA) [10], 3D gel-printing [11], a robocasting technique [12], direct inkjet printing (DIP) [13] and fused deposition modeling (FDM) [14], etc.), digital light processing (DLP) [4,15–23] is one of the
most promising technique. These parts fabricated by this technique exhibit fine surface finishing and very high spatial resolution. In this technique, a ceramic slurry is solidified layer by layer via UV-light exposure, and then the printed part is sintered to produce a fully dense sample.

Zirconia (ZrO₂) is a typical structural ceramic (known as the “ceramic steel” [24]) that is used as cutting tools and sensors. It is also used as an implant due to its excellent biocompatibility, mechanical properties, and satisfying aesthetics. Many researchers tried to use different AM techniques to fabricate the fully dense ZrO₂ part. He et al. [2] used the DLP technique to manufacture the complex dense ZrO₂ part. D. Komissarenko et al. [4] successfully printed Scandia-stabilized ZrO₂ parts by a DLP 3D printer, and no visible cracks or defects were observed in printed samples. They used 33 vol.% Scandia-stabilized ZrO₂ slurries for printing. This slurry is suitable to print the part by printing a thickness layer of 50 μm. Xia et al. [25] fabricated the fully dense ZrO₂ part by the direct ink writing technique. Yu et al. [26] used extrusion-based additive manufacturing to manufacture the ZrO₂ sample. The flexural strength of the samples fabricated by this method was much lower than the sample prepared conventionally. Recently, Sun et al. [27] studied the effect of the slurry and debinding process on the printing of the ZrO₂.

The studies mentioned above mainly emphasize the preparation of slurry, the DLP printing parameters, the sintering process and the physic properties of the printed specimens, and only a limited investigation on the fractography analysis as well as the deformation mechanism. Therefore, the presented work prepared a high solid loading ZrO₂ slurry (55 wt.%) for DLP printing. The flexural strength of printed samples was measured via a three-point bending test
with the digital image correlation (DIC) method. Combining with the SEM and fractography analysis, the deformation mechanism was discussed before the conclusion section.

2. Methodology

2.1. Materials and slurry preparation

One of the critical steps in the DLP method is to prepare a photo-curable slurry with characteristics that meet the printing requirements (such as not having a high viscosity and appropriate curing characteristics). The following raw materials were used in this experiment:

3 mol% yttria-stabilized ZrO$_2$ (3Y-TZP), $D_{30} = 500\text{nm}$ (FREDS, JIANGSU FREDS POWDER TECHNOLOGY Co. Ltd, Yixing, China); 1,6-hexanediol diacrylate, HDDA, two shrink three propylene glycol acrylic ester, TPGDA, and the photoinitiator TPO (Diphenyl(2,4,6-trimethylbenzoyl) phosphine oxide, three of them from Yinchang Xin Cailiao Ltd, Shanghai, China). The dispersant was used DISPERBYK (a lower molecular weight polycarboxylic acid polymer (BYKGardner GmbH, Geretsried, Germany)).

The process of powder modification and preparation of slurry is presented in Fig 1. The original ZrO$_2$ powder was humid, so it should be in an oven for drying thoroughly. After that, using 3 wt.\% of DISPERBYK, based on the mass of ZrO$_2$ powder, was dissolved in ethanol solution by magnetic stirring at 60 °C. Then added a suitable amount of the dried ZrO$_2$ powder into the DISPERBYK solution grandly. Next, the resulting ZrO$_2$ slurry was magnetically stirred at water bath 60 °C for 1 h to promote the adsorption of the dispersant on the surface of the ZrO$_2$ particles adequately. Lastly, the modified ZrO$_2$ powder was dried at 70 °C for 24 h and then screened through a sieve.
First, HDDA (70g), TPGDA (33g) and TPO (1g) were mixed to obtain a homogeneous photosensitive resin. Next, the prepared photosensitive resin was added an appropriate amount (55 wt.%) of modified ZrO$_2$ powder, and stirred with a mixer for 15 minutes. Then the stable ZrO$_2$ slurry was achieved using a planetary ball mill for 4 hours (KE4, Ruiru Tec, Guangzhou, China) with a mass ratio of ZrO$_2$ grinding ball to ZrO$_2$ powder of 2:1.

2.2. **DLP procedures**

ZrO$_2$ samples were printed using a DLP 3D printer (Beijing TenDimensions Technology Co., Ltd. Beijing, China). First, the prepared ceramic slurry was poured into the scraper slot and spread slurry on the Teflon film. Then, UV light was exposed to the Teflon film surfaced, and the slurry was polymerized and cross-linked to form a single layer. After that, the Z - stage moved upwards, and the slurry was recoated on the Teflon film surface by scraper moving in the X-direction. Finally, this procedure was repeated to print the whole part. Fig. 2 schematically shows the DLP process.
The printed samples were cleaned by ultrasonic cleaning in deionized water with resin cleaner in order to remove the remaining slurry on the printed samples, and then dried at 60°C for 24 hours.

**Fig. 2.** The schematic diagrams of DLP 3D printing procedures.

2.3. *Debinding and sintering*

A one-step sintering method was adopted after trial and experiment since the samples became exceptionally fragile after debinding. The debinding and sintering process uses only a traditional muffle furnace (KSL-1700X, Hefei Kejing Material Technology Co., Ltd., China).

After printing the green parts, a debinding process must be performed to remove resin from the green body. The debinding process should be designed carefully to avoid the formation of cracks during it. The debinding behavior depends on several factors, such as resin composition, component geometry, and solid loading. For these reasons, the thermogravimetric analysis (TGA) and Derivative Thermogravimetry (DTG) of the cured slurry were performed and carried out on Thermo Gravimetric Analyzer type (METTLER TG/DSC3+, METTLER TOLEDO, USA).
Printed specimens must be sintered as fast as possible to avoid the potential effects of environmental conditions on the mechanical properties of the ceramics. The sintering process was described in detail in the results and discussion section.

2.4. Flexural strength tests

The dimensions of the specimens were 45 mm × 4 mm × 3 mm (length, width, thickness). Ceramics' mechanical behavior is largely dependent on the applied fabrication process, strain rate, and material type. According to ASTM C 1161-02c (2008), the three-point bending test (Fig. 3) was carried out on a universal testing machine (ETM104B, WANCE, China). The three-point bending span was 20 mm, and the loading rate was 0.3 mm/min. Before testing, the samples were visually inspected to exclude artificial defects, the support structures were removed, and no further surface finishing was implemented after printing and cleaning. The mechanical test was terminated when the sample failed, and the maximum load obtained was used to calculate the flexural strength ($\sigma_f$).

$$\sigma_f = \frac{3PL}{2bd^2}$$ (1)

where $P$, $L$, $b$, and $d$ are the maximum load (N), support span (mm), the width of the tested beam (mm), and the thickness of the tested beam (mm), respectively.

DIC technology was used to analyze the cracking process of the sintered ZrO$_2$ samples under the three-point bending test. The surface of the samples was photographed at a speed of 3 frames per second by a CDD camera (GRAS-50S5M/C) with a pixel resolution of 5 million pixels. To obtain high-quality images measured by digital image correlation (DIC), two 95 W high-power LED lamps were used as light sources and provided adequate illumination intensity.
2.5. Characterization

A rheometer (Physica MCR301, Anton Paar, Austria) was used to study the rheological behavior of the ceramic slurry. The functional relationship between viscosity and shear rate was measured in the range of 0-200 s\(^{-1}\) under isothermal conditions (25.0 °C).

The shrinkage of sintered samples was measured via a Vernier caliper. The apparent density and open porosity of the sintered sample were determined by True Density Analyzer (AccuPyc II 1345, Micromeritics instrument Ltd. U.S.A). Vickers hardness was determined using the Vickers indentation method at an indentation load of 9.8 N for 15 seconds (HMV-2TADW, Shimadzu Corporation, Japan). For phase identification, an X-ray diffractometer (XRD) (Bruker AXSD2 Advance, Billerica, MA, USA) was utilized. The microstructures and
the fracture surfaces of samples were examined using the scanning electron microscope (SEM) (SU100, HITACHI, Japan).

3. Results and Discussion

3.1. Rheological properties

The slurry used in the DLP printing process should have high fluidity to ensure the printing of ceramic parts with complex geometry within a reasonable processing time, meaning the slurry viscosity should not be too high. The viscosity of 3 Pa·s at a shear rate of 10 s⁻¹ is generally considered to be the upper limit of processable slurry [28]. The rheology of slurry is affected by the dispersant property. Sun et al. [29] prepared the slurry with five various dispersants (oleic acid, coupling agent KH560, stearic acid, Disperbyk, and variquat CC42 NS). It was found that 3 wt.% Disperbyk is the best option, that’s why the same type and amount of dispersant was used in this research. Fig.4 shows the rheological properties of the ZrO₂ slurry. The viscosity at 10 s⁻¹ and 100 s⁻¹ are 0.9 Pa·s and 0.13 Pa·s, respectively. The prepared slurry revealed shear-thinning fluid behavior, indicating the slurry was appropriate for printing processing.
**Fig. 4.** Shear viscosity ($\eta$) of ZrO$_2$ slurry with respect to the shear rate in the range of [0, 200] (s$^{-1}$).

### 3.2. Sintering profile

Fig. 5(a) illustrates the TG-DTG curves of the green body obtained after DLP printing. The cured resin began to decompose at 80 °C, and finished at 540 °C. There were four prominent decomposition peaks around 180 °C, 360 °C, 410 °C, and 480 °C. The resin decomposed quickly between 180 °C and 410 °C during the debinding process.

Fig. 5(b) shows the temperature scheme used in the debinding and sintering. In the debinding sintering process, the pores among the powders were squeezed and eliminated, and a highly dense component was formed. In the low-temperature stage, the rapid expansion of gas would accelerate the initiation and growth of cracks [30]. Thus, the heating rate between 80 °C and 540 °C should be delicately controlled: various heating rates of 1 °C / min and 0.2 °C / min were implemented for the temperature range of RT - 80 °C and 80 - 540 °C. Meanwhile, in order to assure the fully chemical decomposition, one-hour dwelling time was applied at temperatures of 180 °C, 360 °C, 410 °C, 480 °C, and 540 °C, respectively. Moreover, a heating rate of 5 °C/min was selected for the entire sintering process up to 1500 °C, and kept for another 200 mins.

![Fig. 5.](image-url) (a) The TG/DTG curves and (b) the debinding and sintering profiles of the green body.
3.3. **Physical properties of the printed sample**

In DLP printing, curing thickness is a significant parameter determining the thickness of available layers and printing quality. Therefore, appropriate exposure times and light intensity must be found to ensure good bonding between cured layers during printing. After the preliminary test, the exposure time was determined as 2 s with a light intensity of 18 mW/cm², and the average curing thickness was measured as 35 μm.

The insets in Fig. 3 show the sintered sample's front surface (along the printing direction) and the top surface (perpendicular to the printing direction). Good flatness can be observed on the top surface, and this uniform pattern shows the precision of the printing process.

The density of the sintered ZrO₂ sample was around 5.98 g/cm³, which was approximately 98.8% relative density (the theoretical density was taken as 6.05 g/cm³). This relative density was slightly lower than that of other samples (99%) prepared by the DLP method [31], which resulted in lower Vickers hardness values (1128 HV) of the sample compared to the sample with higher relative density (~1190 HV) [31].

The variation of the anisotropic linear shrinkage of the sintered specimen is listed in Table 1. Due to the friction between the specimen and crucible during sintering, the linear shrinkage in the width (b) direction was slightly lower than that in the thickness (d) direction. Reducing cracks and improving repeatability and dimensional precision was profit from lower linear shrinkage and shrinkage deviation.

**Table 1**

| Shrinkages of the samples. | Shrinkage (%) | Mean+SD. | Mean+SEM. |
|---------------------------|--------------|----------|-----------|
| Thickness, d              | 41.90        | 0.7659   | 0.2124    |
| Width, b                  | 41.58        | 0.8199   | 0.2274    |
| Average                   | 41.74        | 0.4991   | 0.1384    |
3.4. **XRD**

Cubic ZrO$_2$ (c-ZrO$_2$) exists at temperatures higher than 2370 °C. Tetragonal ZrO$_2$ (t-ZrO$_2$) exists between 1170 and 2370°C. When the temperatures are lower than 1170 °C, the phase transition from t-ZrO$_2$ to monoclinic ZrO$_2$ (m-ZrO$_2$) occurs [32]. The phase of t-ZrO$_2$, however, can be retained at RT by adding metal oxides, such as yttria (Y$_2$O$_3$). The crystalline phase of m-ZrO$_2$ and t-ZrO$_2$ were both detected in the original ZrO$_2$ powder because the 3 mol% Y$_2$O$_3$-stabilized ZrO$_2$ powder was used in the present study (Fig. 6). However, after sintering up to 1500°C, the amount of m-ZrO$_2$ was below the detection limit in the specimen due to the m→t phase transition. As a result, the prominent peaks of 2θ at 30.2°, 34.7°, and 35.2° became more significant (Fig. 6). These three peaks corresponded to the characteristic crystal planes (101)$_t$, (002)$_t$, and (110)$_t$ of t-ZrO$_2$, respectively.

![XRD pattern](image)

**Fig. 6.** The XRD pattern of the original ZrO$_2$ powder and the sintered ZrO$_2$ sample.

3.5. **Mechanical property**
The force-displacement curves of the samples under parallel and vertical bending loads (with respect to the printing direction) are shown in Fig. 7. The flexural strength under parallel and vertical bending loads were 56.63±3.97MPa and 70.98±6.62MPa, respectively.

There is still a certain gap between the results in this study and better levels achieved in other literature [16]. But this lower strength may be able to provide better biocompatibility by avoiding the stress shielding phenomenon. The values of elastic modulus and strength of cancellous bone are 10 to 1570 MPa and 1.5 to 38 MPa, respectively [33]. The researchers have been designed and successfully printed various ceramic bone scaffold [15,21,23]. The DLP-printed ZrO$_2$ with different lattice structures would show great potential in the implant field.

![Fig. 7. The flexural strength curves.](image)

The measured horizontal ($e_{xx}$) and vertical strain ($e_{yy}$) on the surface of the specimen are given in Fig. 8 for the parallel loading and Fig. 9 for the vertical loading. The crack initiated at the sample bottom surface due to the tension effect and developed upward until the failure of the samples. The material under tension during testing influenced the flexural strength and the failure mode of the experimental groups [34]. Clearly, the crack propagated through the width direction (under vertical loading) was
faster than that propagated through the thickness direction (under parallel loading). One of the key reasons that affect the flexural strength of the specimen is the existence of cavities in the specimen. During the three-point bending test, if a cavity is present at any point, the specimen tends to break quicker than the solid portion, and the strength decreases. A detailed fractography analysis will be given in the next section, combined with the SEM images.

Surface treatment has an important influence on flexural strength. The flexural strength of ceramics can be improved by some surface modification methods, such as sandblasting and wet-grinding [29,35]. For ZrO$_2$, grinding has been recommended to create a surface region of compressive stresses, which increases the mean flexural strength of ZrO$_2$. Noted that no surface treatment was used in the present study in order to keep the as-fabricated property, so the surface defects were unavoidable and resulted in the crack initiation points. It would be worthy of investigation the surface treatment on the fracture toughness of 3D printed ZrO$_2$ in the future.
Fig. 8. The DIC images of the horizontal strain ($e_{xx}$) and the vertical strain ($e_{yy}$) of ZrO$_2$ samples under a three-point parallel bending test.

Fig. 9. The DIC images of the horizontal strain ($e_{xx}$) and the vertical strain ($e_{yy}$) of ZrO$_2$ samples under a three-point vertical bending test.

3.6. SEM
The SEM analysis can provide additional evidence for the microstructure of printed ZrO₂ samples (Fig. 10) and the fracture modes (Fig. 11-12).

Before sintering (green body), the top surface was flat and smooth without any visible cracks (Fig. 10a, 10b); however, after sintering, the top surface became coarse due to the shrinkage effect, and the cracks were observed (Fig. 10d, 10e). Moreover, the front surface exhibited a clear lamination along the printing direction (blue arrow) before and after sintering (Fig. 10c, 10f), which the interlayer was thinner after sintering.

The fracture morphology and its partially enlarged views for paralleling and vertical bending tests are displayed in Fig. 11 and 12, respectively. Although the samples appeared to be stacked layer by layer from the outside, they were actually interlaced with irregular and winding cracks, indicating the debinding and sintering process has a prominent influence on the interior microstructure.

The sintered ZrO₂ specimen was quite dense at depths of only a few hundred microns (as pointed with yellow rectangular in Fig. 11b and 12b), and this dense layer along the width direction was thicker than the printing direction. Moreover, the interior of the ZrO₂ specimen
was filled with needle-like cracks, meaning that the thermal diffusion in the debinding-sintering process and the mismatch of thermal expansion coefficient between resin (organic matter) and solid phase material determine the final microstructure. Meanwhile, such differences in internal and external microstructures also reflected that 3D printing technology might be more suitable for preparing spatial lattice structures with limited thickness to give full play of their strength rather than continuum bulk materials.

It can be seen that in the case of parallel loading, mainly the bottom printing layer was stretched to both lateral sides. Because the tensile strength of ceramic is typically much lower than the compressive strength, the failure started at the bottom first. However, under vertical loading, this bottom was a dense solidified layer, whose strength was significantly higher than that of the bottom layer with surface cracks under parallel loading, that’s why the vertical bending strength was higher than that obtained in the parallel bending test.

In addition, the crack shape was different inside and outside. The dense outer layer was mainly composed of large and straight cracks between printing layers, whereas the interior contained numerous small winding cracks. When the big crack propagates and meets the small crack, the crack tip would be weakened, and the big crack may change propagate direction and decomposed into several small cracks (Fig. 13). As a result, the crack propagation would slow down, and some cracks may even be compressed and closed. Therefore, there was a large cleavage fracture morphology in the boundary layer (yellow rectangular in Fig. 11 and 12). Still, the plastic fracture mode (stepped morphology with blue ellipses) appeared in the interior (Fig. 12).
Fig. 11. The SEM images of three-point parallel loading. (a): Schematic diagram of the experiment; (b-d): Fracture morphology and its partial enlarged views.

Fig. 12. The SEM images of three-point vertical loading. (a): Schematic diagram of the experiment; (b-d): Fracture morphology and its partial enlarged views.
Similar to other 3D printing technologies, there are still some challenges in the lamination process and sintering process of printing parts with DLP preparation technology [36]. It is expected that a more detailed study of the curing process itself is necessary for future development. For example, suppose the interaction between UV light and materials can be understood better. In that case, the DLP method can be comparable to other advanced ceramic processing techniques by fabricating structural products (i.e., bone scaffold).

4. Conclusion

ZrO$_2$ parts have been fabricated using a DLP 3D printing technology in which an attempt has been made to investigate its three-point bending deformation mechanism via DIC and fractography. The main conclusions are summarized as follows.

- The viscosity of the prepared slurry at 10 s$^{-1}$ and 100 s$^{-1}$ were 0.9 Pa·s and 0.13 Pa·s, respectively, which was desirable for processing.
- The relative density of the sintered ZrO$_2$ sample was approximately 98.8%, resulting in a Vickers hardness value of 1128 HV. The linear shrinkage was 41.74% on average.
- The crystalline phase of m-ZrO$_2$ and t-ZrO$_2$ were both detected in the original ZrO$_2$ powder; however, only the t-ZrO$_2$ phase remained in the sample after sintering up to 1500°C.
- The flexural strength under parallel and vertical bending loads were 56.63±3.97MPa and
70.98±6.62MPa, respectively. The crack propagates along the width direction (under vertical loading) was faster than that propagates along the thickness direction (under parallel loading).

- There was a large area of cleavage morphology in the dense boundary layers; still, the plastic fracture mode (stepped morphology with winding needle-like cracks) appeared in the interior of the sintered samples.

**Declaration of competing interest**

The authors declare that they have no known competing financial interests or personal relationships that could have influenced the work reported in this paper.

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