

# High-temperature performance of silica ceramic cores with additives prepared by stereolithography 3D printing

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**Abstract:** Ceramic cores are key to forming a cooling structure within the hollow blade cavities. The use of stereolithography (SL) 3D printing technology eliminates the need for moulds, facilitating the preparation of complex-shaped ceramic cores. In this study, silica-based ceramic cores incorporating nano-3YSZ (3mol.% yttria stabilised zirconia) and micron-sized  $Y_2O_3$  were prepared via SL 3D printing ceramic technology to promote the formation of cristobalite and  $ZrSiO_4$ , thereby improving the high-temperature properties. The flexural strength at 25 °C and 1,500 °C, deflection at 1,500 °C, shrinkage rate, and porosity of the core samples sintered at different temperatures (1,170 °C, 1,185 °C, 1,200 °C, 1,215 °C, and 1,230 °C) were tested and investigated. The mechanism underlying the high temperature performance of the cores was elucidated through analysis of cross-sectional morphology, element distribution, and phase constitution of the samples. As the sintering temperature increases, the shrinkage and flexural strength at 25 °C of the core rise, while the open porosity and deflection at 1,500 °C decrease. When the sintering temperature reaches 1,200 °C or higher, the 1,500 °C flexural strength can be measured, which increases as the sintering temperature rises. The core exhibits excellent creep resistance when sintered at temperatures of 1,200 °C and above. Considering the comprehensive performance requirements for the core, the sintering temperature of 1,200 °C was selected. At the sintering temperature of 1,200 °C, the core exhibits shrinkage rates of 3.76% (X), 3.38% (Y), and 3.95% (Z), alongside a flexural strength of 9.01 MPa at 25 °C and 32.15 MPa at 1,500 °C, and an open porosity of 26.39%. The deflection of the core at 1,500 °C is 0.15 mm, which helps to maintain the dimensional stability of the ceramic core during casting. XRD results indicate that samples fractured after 25 °C flexural strength test still contain amorphous quartz glass, alongside substantial quantities of yttria stabilized zirconia and  $Y_2O_3$ . Samples fractured after 1,500 °C flexural strength test exhibit significant crystallisation of amorphous quartz glass into cristobalite, with silica and 3YSZ combining to form  $ZrSiO_4$ .  $Y_2O_3$  as a network modifier of the glass network destroys the bridging oxygen in the silica-oxygen bond, thereby reducing the energy required for glass crystallisation and promoting the crystallisation reaction of quartz glass to form cristobalite. In addition, nano-3YSZ combines with  $SiO_2$  at high temperatures to form  $ZrSiO_4$ . Since cristobalite and  $ZrSiO_4$  are crystals, both of them have strong creep resistance, thus improving the high temperature flexural strength and deformation resistance of the ceramic cores.

**Keywords:** 3D printing; ceramic cores; fused silica; high temperature performance; deflection

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## 1 Introduction

Aero-engines are the core components that provide power source for aerospace, and their future focuses

on improving the thrust-to-weight ratio<sup>[1]</sup>. Therefore, hollow turbine blades that determine the important performance of engines have become a top priority in research and development<sup>[2]</sup>. The ceramic core is the key to forming a complex cooling structure within the hollow turbine blade cavity<sup>[3]</sup>. It is usually required that the ceramic core has a low and stable shrinkage<sup>[4]</sup>, a room temperature flexural strength of over 8 MPa<sup>[5]</sup>, and an open porosity of over 20%

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to remove the core<sup>[6]</sup>. Since the ceramic core needs to be kept in molten metal at approximately 1,500 °C for 30 min during casting<sup>[7]</sup>, the ceramic core needs to have excellent high-temperature properties, including the smallest possible deflection and appropriate high-temperature flexural strength<sup>[8]</sup>. At present, the internal cooling structure of hollow blades has developed from single counter flow air cooling to double wall ultra air cooling<sup>[9]</sup>. For the development of this new type of blades with complex structures, traditional core preparation methods such as gel injection molding, thermal injection molding require to prepare multiple sets of molds in advance<sup>[10]</sup>, resulting in a long development and production cycle<sup>[11]</sup>. Therefore, an efficient ceramic core preparation method is urgently needed.

In recent years, additive manufacturing technology<sup>[12]</sup> (also known as 3D printing technology) has developed rapidly and has been applied to the production of various complex shaped parts<sup>[13]</sup>. 3D printing technology has the advantages of rapid forming, unrestricted moulding shapes, less material waste, and high dimensional accuracy of finished products<sup>[14]</sup>. At present, the most widely used 3D printing technology for ceramic materials is photopolymerization 3D printing ceramic technology<sup>[15]</sup>, mainly including stereolithography (SL) and digital light processing (DLP)<sup>[16]</sup>. The principle of SL forming ceramic cores is that the light source scans point by point to solidify the ceramic slurry<sup>[17]</sup>. The slurry solidifies layer by layer to obtain a green body, which is then subjected to debinding and sintering processes to obtain the final ceramic core<sup>[18]</sup>. By eliminating the time required for mold manufacturing and enabling flexible model modifications, the product iteration cycle is significantly shortened<sup>[19]</sup>. Digital light processing has been improved based on stereolithography<sup>[20]</sup>. The slurry is cured by surface light source to form the entire layer simultaneously<sup>[21]</sup>, so DLP is also known as surface exposure 3D printing technology<sup>[22]</sup>.

Silica-based ceramic cores are widely used in the casting of hollow turbine blades<sup>[23]</sup> because of their excellent properties such as low sintering temperature, low coefficient of thermal expansion, and good chemical stability<sup>[24]</sup>. Many researchers have applied stereolithography 3D printing technology to the preparation of silica-based ceramic cores. Wang et al.<sup>[25]</sup> studied the effect of nano silica content on the properties and microstructure of 3D printed silica-based cores, and found that as the nano silica content increased, the open porosity and shrinkage of ceramic cores decreased, while the flexural strength increased due to enhanced sintering densification. The bonding between adjacent ceramic particles became tighter, and the interlayer bonding grew stronger. Li et al.<sup>[26]</sup> investigated the anisotropy of 3D-printed ceramics and found that ceramic specimens had higher flexural strength in the direction parallel to the stack of printed layers and that the mechanical properties were modulated by controlling the printing and sintering parameters. Li et al.<sup>[27]</sup> employed a combined simulation and experimental approach to investigate the anisotropic behavior of ceramic cores fabricated via

photopolymerization 3D printing technology. It was concluded that as sintering temperatures increase, pores migrate towards the interlayer interfaces. However, most previous studies have focused on the room temperature performance of ceramic cores, with less research on the high temperature performance of 3D printed silica-based ceramic cores. High temperature performance determines whether the core can maintain its shape stability during casting, which affects the dimensional accuracy of the casting. The main reason for the deformation of silica-based ceramic cores at high temperatures is that the matrix material of the core, fused silica, is amorphous which undergoes viscous flow at high temperatures. The anti creep ability of crystals is much higher than that of amorphous materials<sup>[28]</sup>. Therefore, the anti deformation performance of silica-based cores can be improved by generating crystals. For example, the cristobalite produced by the crystal transformation of fused silica at high temperatures can suppress the deformation of the cores<sup>[29]</sup>.

In this study, ceramic cores with low high-temperature deformation were prepared via SL 3D printing by formulating its raw material composition and post-treatment process. Coarse fused silica was selected as the core matrix material. Nano-sized 3YSZ (3mol.% yttria stabilised zirconia) and micron-sized  $Y_2O_3$  were added, and silica sol was used for strengthening. 3YSZ was selected to stabilize zirconia in tetragonal phase as it does at high temperature, and no crystal transformation occurs during sintering and high-temperature testing that could cause volume changes and lead to core cracking. Furthermore, addition of  $Y_2O_3$  improves the high-temperature creep resistance of the core by promoting crystallisation, thereby ensuring the dimensional stability of the core during casting.

## 2 Experimental procedure

The preparation process of ceramic cores is shown in Fig. 1. The raw material was composed of 78wt.% fused silica ( $D_{50}=25\text{ }\mu\text{m}$ ), 20wt.% 3YSZ (3mol.% yttria stabilised zirconia,  $D_{50}=20\text{ nm}$ ), and 2wt.%  $Y_2O_3$  ( $D_{50}=4\text{ }\mu\text{m}$ ) (All from Hebei Chuancheng Metal Materials Co., Ltd.). The addition of 3YSZ and  $Y_2O_3$  was based on the related studies<sup>[30]</sup> and previous experimental results. The monomer and crosslinking agent were 1,6-Hexanedioldiacrylate (HDDA) (DSM-AGI) and Trimethylolpropane triacrylate (TMPTA) (DSM-AGI), respectively. A BYK commercial dispersant was used to disperse ceramic powder. The photoinitiator TPO (BASF) was used to initiate photopolymerization reactions. BYK410 was added to improve the stability of the slurry. A planetary centrifugal mixer (ZYMC-580HV, ZYE, China) was used to mix and prepare a ceramic slurry with a solid content of 60vol.%. The composition of the ceramic slurry is shown in Table 1. The printing dimension of the ceramic core sample is shown in Fig. 2(a). CeraBuilder160Pro (iLaser, China) was used to print the ceramic paste for the fabrication of ceramic green bodies. The laser power was 1.2 W, the scanning speed was  $7,000\text{ mm}\cdot\text{s}^{-1}$ , and the layer thickness was  $50\text{ }\mu\text{m}$ . The

ceramic green body was degreased and sintered in air, with five sintering temperatures selected, as shown in Fig. 2(b). The sintered core samples were strengthened by immersing them in 20wt.% silica sol for 30 min.

The open porosity, shrinkage, flexural strength, and deflection of the sintered ceramic core samples were tested according to the method of HB 5353-2004 (China aeronautical department standard). All the tests were conducted on more than 5 samples in each group and the average value was taken.

The calculation equation for open porosity is as follows:

$$B = \frac{m_2 - m_1}{m_2 - m_3} \times 100\% \quad (1)$$

where,  $B$  – open porosity of the sample (%);  $m_1$  – dry weight of the sample (g);  $m_2$  – wet weight of the sample (g);  $m_3$  – floating weight of the sample (g).

The calculation equation for the shrinkage is as follows:

$$\delta = \frac{L - L_1}{L} \times 100\% \quad (2)$$

where,  $\delta$  – the shrinkage of the sample (%);  $L$  – length of the sample before sintering (mm);  $L_1$  – length of the sample after sintering (mm).

The flexural strength of the ceramic core at room temperature (25 °C) and high temperature (hold at 1,500 °C for 30 min) under different sintering temperatures was tested, and the equation is as follows:

$$\sigma_w = \frac{3Pl}{2bh^2} \quad (3)$$

where,  $\sigma_w$  – flexural strength (MPa);  $P$  – maximum load (N);  $l$  – length between the fulcrums (mm);  $b$  – width of the sample (mm);  $h$  – thickness of the sample (mm).

The deflection of the ceramic core was tested (hold at 1,500 °C for 30 min). The equation is as follows:

$$\Delta H = H_1 - H_2 \quad (4)$$

where,  $\Delta H$  – deflection (mm);  $H_1$  – height of the cantilever end of the sample measured from the bottom of the sample holder (mm);  $H_2$  represents the height of the same point after holding at 1,500 °C for 30 min and cooling in furnace to room temperature.

Field emission scanning electron microscopy (SEM, Thermo Fisher Scientific, Apreo 2C, America) and energy dispersive X-ray spectroscopy (EDS, Bruker QUANTAX XFlash730) were used to characterize the cross-sectional microstructure

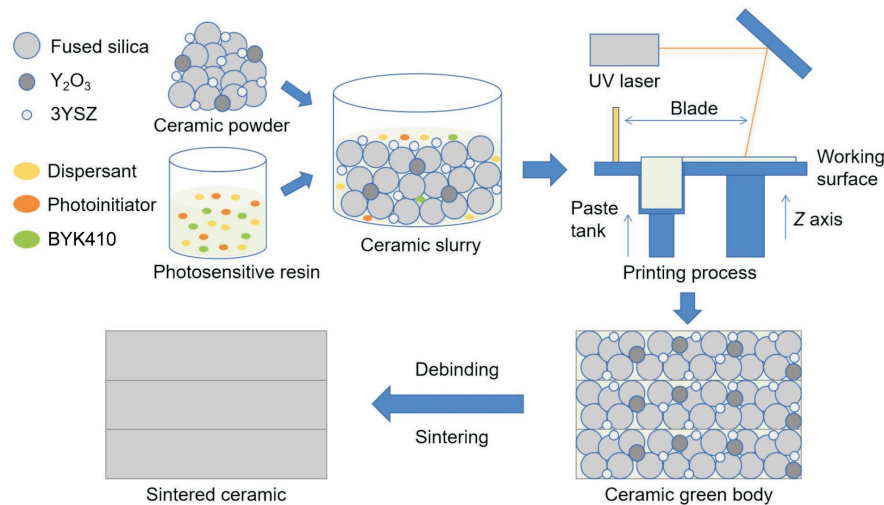


Fig. 1: Ceramic core preparation process

Table 1: Composition of ceramic slurry

| Ceramic powder | Photosensitive resin             | Dispersant                                                  | Photoinitiator                       | Auxiliary                                  |
|----------------|----------------------------------|-------------------------------------------------------------|--------------------------------------|--------------------------------------------|
| 60vol.%        | TMPTA:HDDA=4:6<br>(weight ratio) | BYK commercial dispersant<br>2wt.% (based on powder weight) | TPO 1wt.% (based on<br>resin weight) | BYK410 0.5wt.% (based<br>on slurry weight) |

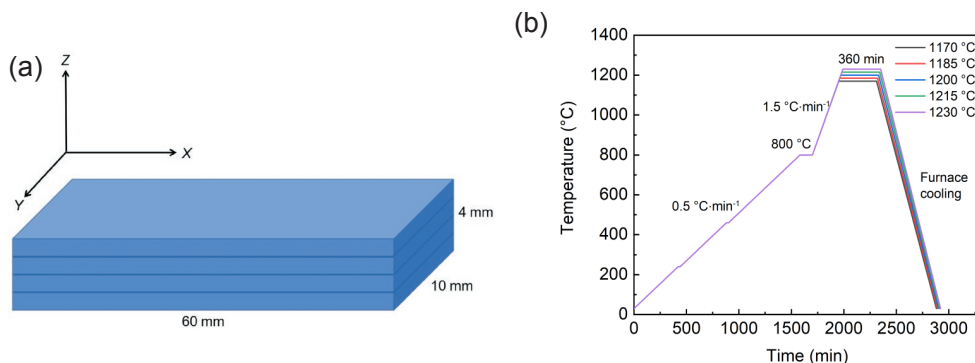


Fig. 2: Ceramic core sample printing model (a) and debinding-sintering process (b)

and elemental distribution, respectively, of the ceramic core samples (unsintered, 25 °C and 1,500 °C flexural strength testing). X-ray diffraction (XRD, Rigaku SmartLab, Japan) was used to identify the phases present in the ceramic core samples, including 25 °C and 1,500 °C flexural strength testing samples. The microstructure and elemental analysis of ceramic particles in the core samples after 1,500 °C flexural strength testing were analyzed by transmission electron microscopy (TEM, Thermo Fisher Scientific-Tecna TalosF2, America). TEM samples were prepared using precision ion polish system (PIPS, PIPS II Model 695, America).

## 3 Results

### 3.1 Performance of ceramic cores

The shrinkage, open porosity, flexural strength at 25 °C and 1,500 °C, and 1,500 °C deflection of the ceramic core samples at different sintering temperatures are shown in Fig. 3. The shrinkage of the samples is shown in Fig. 3(a). The shrinkage in the Z direction is higher than that in the X/Y direction. The anisotropic of shrinkage is caused by the layer-by-layer forming of 3D printing. During the forming process, the deposition of ceramic particles results in a higher resin content in the Z direction. After debinding and sintering, the resin is expelled, thereby causing a greater shrinkage rate in the Z direction. As the sintering temperature increases, the sintering of the core becomes more complete, resulting in an increase in shrinkage. The open porosity is shown in Fig. 3(b), and the reason for the overall low open porosity is that the silica sol reinforcement results in a portion of the pores being blocked by amorphous silica particles<sup>[28]</sup>. As the sintering temperature increases, the ceramic particles are sintered more densely

and the inter-particle pores are further expelled, which leads to a decrease in the open porosity. Figure 3(c) shows the flexural strength of the ceramic core at 25 °C and 1,500 °C. It can be seen that as the sintering temperature increases, the room temperature flexural strength of the ceramic core increases, which is also due to the densification of the ceramic core during sintering. When the sintering temperature reaches 1,200 °C and above, the room temperature flexural strength of the ceramic core is greater than 8 MPa<sup>[5]</sup>. When the sintering temperature is 1,170 °C and 1,185 °C, the core softens and deforms during the high-temperature flexural strength test, and no effective flexural strength data are obtained. This demonstrates that cores sintered at these temperatures exhibit poor creep resistance. When the sintering temperature increases from 1,200 to 1,230 °C, the flexural strength of the core at 1,500 °C increases from 32.15 MPa to 35.11 MPa. Figure 3(d) shows the deflection of the core held at 1,500 °C for 30 min. It can be seen that when the sintering temperature reaches 1,200 °C and above, the core has a very small deflection of 0.12 to 0.15 mm, which represents that the core is almost undeformed. This helps to maintain the stability of the shape of the ceramic core during casting. According to the comprehensive performance requirements for the core, it is expected that the core will have lower shrinkage rate, higher porosity, flexural strength above 8 MPa, and as low deflection as possible<sup>[4-6]</sup>. Therefore, the optimal sintering temperature was selected as 1,200 °C. At this sintering temperature, the shrinkage of the core is 3.76% (X), 3.38% (Y), and 3.95% (Z), the flexural strength at 25 °C is 9.01 MPa and at 1,500 °C is 32.15 MPa, the open porosity is 26.39%, and the deflection at 1,500 °C is 0.15 mm. Further testing and analysis were conducted on the samples obtained at this sintering temperature.

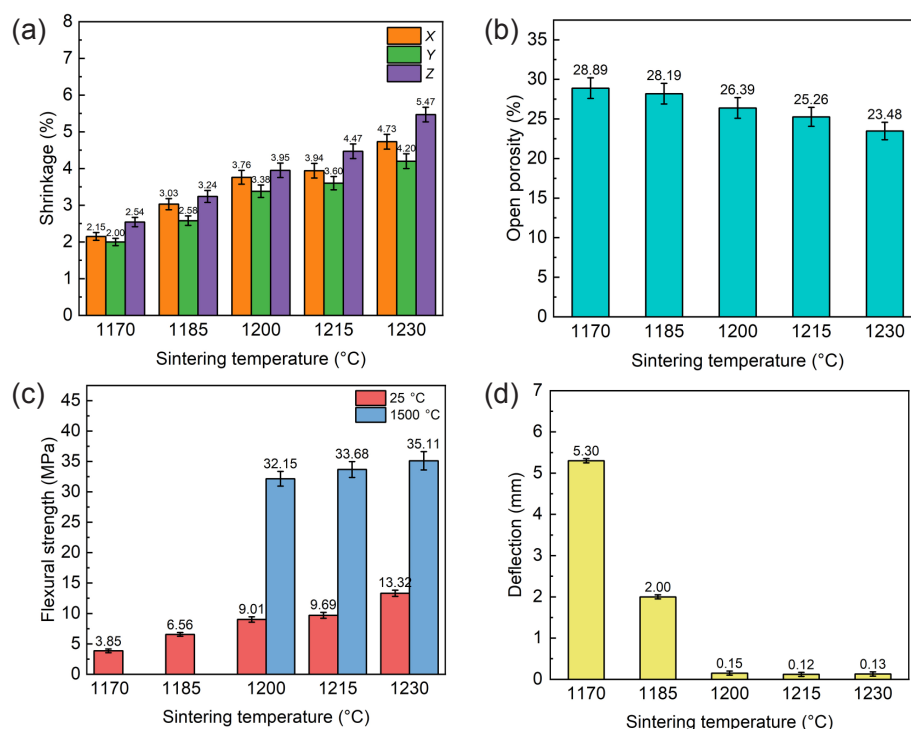


Fig. 3: Performance of ceramic core samples at different sintering temperatures: (a) shrinkage; (b) open porosity; (c) flexural strength at 25 °C and 1,500 °C; (d) 1,500 °C deflection

### 3.2 Microstructure and elemental distribution

The fracture cross-sectional morphology of the unsintered green body sample and the room-temperature flexural strength test sample (sintered at 1,200 °C) are shown in Fig. 4. The fracture cross-section of the unsintered sample in Fig. 4(a) shows a layered structure, which is caused by the 3D printing method of layer by layer stacking. The disappearance of the layered structure in the fracture cross-section of the sintered sample in Fig. 4(b) is due to the photosensitive resin decomposition in the ceramic core sample after debinding and sintering. The ceramic particles between layers diffuse and bond under thermodynamic action, resulting in the disappearance of the interface between layers. Microcracks and pores can be observed inside the sintered sample, and the reason for the formation of microcracks is speculated to be the transformation of quartz glass into cristobalite during sintering. During the cooling process, cristobalite undergoes a volume shrinkage of 2.8%<sup>[29]</sup>, resulting in microcracks inside the core.

The fracture cross-sectional morphology and elemental distribution of the ceramic core samples after 25 °C and 1,500 °C flexural strength test (both sintered at 1,200 °C) are shown in Fig. 5. Figure 5(a) shows that the fracture mode of the core sample experienced 25 °C flexural strength test is mainly intergranular fracture, and a large number of cracks can be observed. The presence of many unbound small particles around larger grains indicates that the sintering of the core is not complete, and the low strength of the grain boundaries leads to the fracture path of the sample mainly extending along the grain boundary. The fracture mode dominated by intergranular fracture results in lower flexural strength of the core. Figure 5(f) shows that a large number of transgranular fractures are found in the core sample after 1,500 °C flexural strength test. The sintering of fine particles at high temperature flexural strength test results in densification and the formation of sintered necks, which enhances the strength of grain boundaries and changes the fracture mode of the sample. Therefore, the high-temperature flexural strength of the core is significantly higher than the room temperature flexural strength. Figures 5(b–e) show the element distribution of the sintered core sample. It can be seen that Si elements form multiple large enrichment zones, surrounded by a large amount of dispersed Zr elements. The distribution of Y elements is basically consistent with that of Zr elements, with small areas

of concentration. This is consistent with the incorporation of nano-sized 3YSZ and micron-sized  $Y_2O_3$ . As shown in Figs. 5(g–j), after 1,500 °C flexural strength test, the binding degree of Si element in the sample increases, and the distribution state of Zr element changes from large-scale dispersion to small areas where it is tightly wrapped around Si element. The distribution of Y element is still basically consistent with that of Zr element, and the concentration degree of small areas decreases. Further analysis in conjunction with XRD results is required to determine the phase constitution.

### 3.3 Phase analysis

The XRD analysis of the ceramic core samples after 25 °C and 1,500 °C flexural strength test (both sintered at 1,200 °C) is shown in Fig. 6. The wide envelope peak near 20° in the 25 °C flexural strength test sample indicates the presence of amorphous fused silica inside, with a small portion transforming into quartz and cristobalite phases, and the presence of a large amount of 3YSZ and a small amount of  $Y_2O_3$ . After 1,500 °C flexural strength test, the sample exhibits a strong diffraction peak of cristobalite, due to the transformation of a large amount of amorphous fused silica into cristobalite.  $ZrSiO_4$  diffraction peaks can also be observed, due to the formation of  $ZrSiO_4$  by the combination of silica and 3YSZ at high temperatures, which has been verified in the research of other scholars<sup>[31]</sup>. However, the EDS image does not show the overlap of Si and Zr elements, which requires further analysis to identify its generation location. Due to the participation of some 3YSZ in the reaction, the remaining 3YSZ combines with  $Y_2O_3$  to form a new zirconium yttrium oxide compound with weakened peak intensity, which also leads to a weakening of the  $Y_2O_3$  peak. This is consistent with the changes in the distribution of Zr and Y elements shown in Figs. 5(c–d) and (h–i).

To further verify the formation of  $ZrSiO_4$ , TEM and element distribution tests were conducted on the ceramic core sample after 1,500 °C flexural strength test (sintered at 1,200 °C). The results are shown in Fig. 7. The results of elemental distribution in Figs. 7(b–e) show that there is a relatively uniform distribution of Si-rich area, and the Zr-rich area overlaps with part of the Si-rich area, which further proves the formation of  $ZrSiO_4$ ; the Y-rich area overlaps with part of the Zr-rich area, which proves the existence of zirconium-yttrium

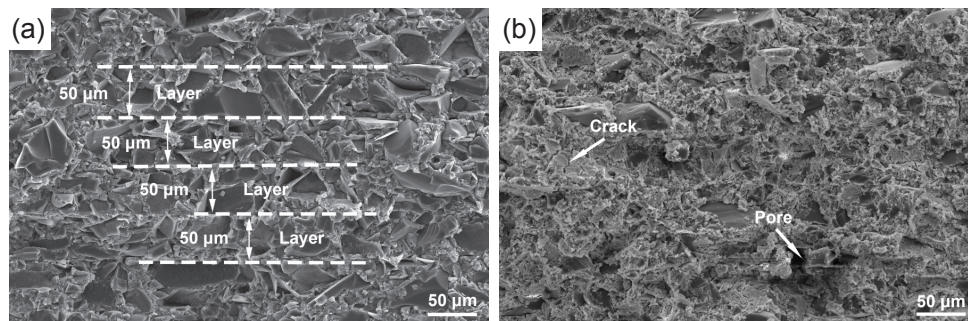


Fig. 4: Fracture cross-sectional morphology of ceramic core samples: (a) unsintered green body sample; (b) sintered sample after room-temperature flexural strength test

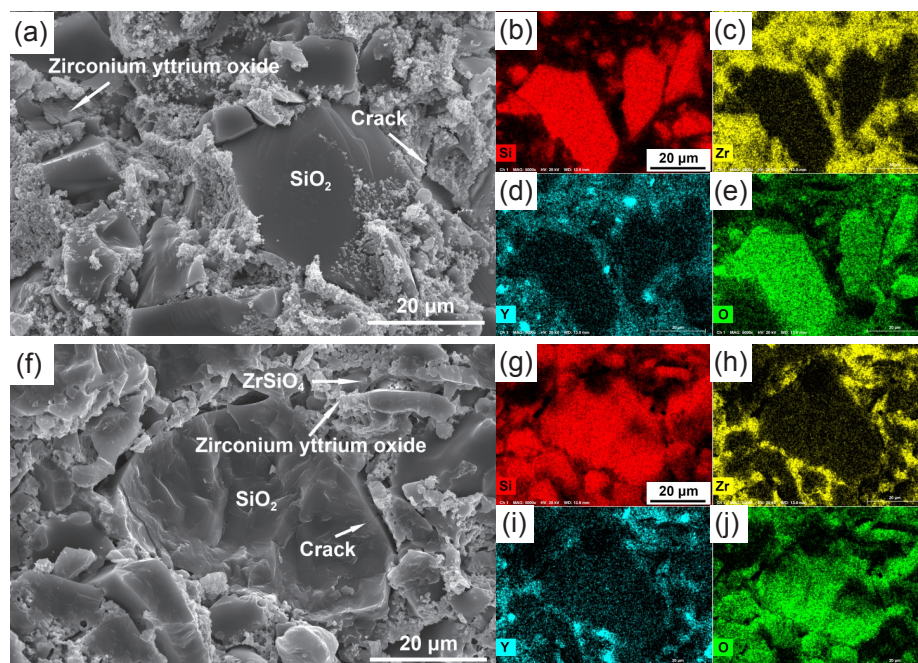


Fig. 5: Fracture cross-sectional morphology and elemental distribution of ceramic core samples after 25 °C and 1,500 °C flexural strength test (both sintered at 1,200 °C): (a) SEM fracture cross-sectional morphology of 25 °C flexural strength test sample; (b-e) Si, Zr, Y, O element distribution of 25 °C flexural strength test sample; (f) SEM cross-sectional morphology of 1,500 °C flexural strength test sample; (g-j) Si, Zr, Y, O element distribution of 1,500 °C flexural strength test sample

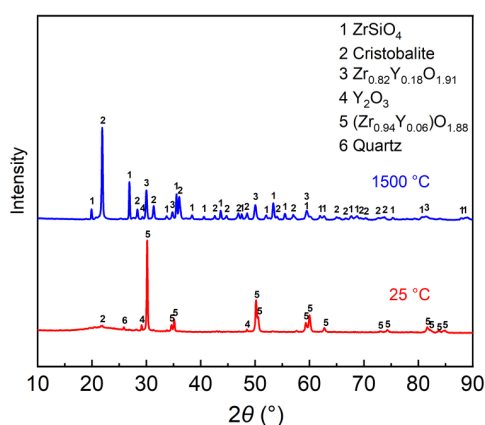


Fig. 6: XRD analysis of ceramic core samples after 25 °C and 1,500 °C flexural strength test (both sintered at 1,200 °C)

oxides. This is in agreement with the XRD results, and the large-sized  $\text{SiO}_2$  particles are wrapped by  $\text{ZrSiO}_4$ , as shown in Fig. 7(a).

## 4 Discussion

The following provides a further mechanistic analysis of the phenomenon where ceramic cores exhibit low deflection and high flexural strength at 1,500 °C. XRD results show that fused quartz undergoes crystallisation after 1,500 °C flexural strength test, producing a large amount of cristobalite. There are two main factors that promote this reaction. On the one hand, the silica sol particles, which act as reinforcing agents, are small amorphous  $\text{SiO}_2$  with great activity, it can effectively promote the crystallization of quartz glass at high temperatures<sup>[28]</sup>.

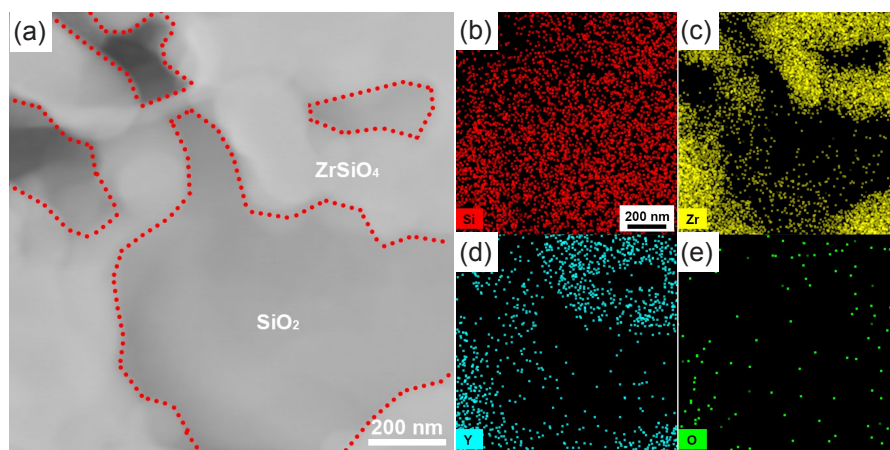
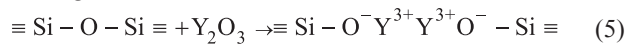


Fig. 7: TEM image and elemental distribution of ceramic core sample after 1,500 °C flexural strength test (sintered at 1,200 °C): (a) TEM images; (b-e) elemental distribution of Si, Zr, Y, and O

On the other hand, the addition of yttrium oxide promotes the formation of cristobalite, which can be microscopically explained by the fact that the charge of the added ions disrupts the bridging oxygen in the Si-O-Si bonds, thereby facilitating crystallisation. According to the hypothesis of the random network of glass structure proposed by Zachariasen<sup>[32]</sup>, when the bond energy formed between the ions and oxygen atoms of the additive was greater than 335 kJ·mol<sup>-1</sup>, it did not promote the crystallization reaction, as it acts as a glass network promoter. When the bond energy was less than 251 kJ·mol<sup>-1</sup>, it would destroy the bridging oxygen in Si-O-Si, reduced the viscosity of the liquid phase, and promoted the crystallization reaction. The Y-O bond formed by the addition of Y<sub>2</sub>O<sub>3</sub> in this experiment has an energy of 209 kJ·mol<sup>-1</sup><sup>[32]</sup> < 251 kJ·mol<sup>-1</sup>, which is a glass network modifier. Therefore, it will destroy the bridging oxygen in the silica oxygen bond, leading to the following reaction:



Bridging oxygen is cut off to generate non bridging oxygen, activating the network structure and reducing the energy required for glass crystallization, thus facilitating the transformation of fused silica into crystals which undergoes a crystallization reaction to form cristobalite.

The main reason for the deformation of silica-based ceramic cores at high temperatures is the high-temperature creep caused by the viscous flow of amorphous fused silica. Combining the aforementioned analysis with TEM results, it can be seen that the quartz glass crystallizes into cristobalite at high temperatures and generates ZrSiO<sub>4</sub> crystals with nano 3YSZ. Since the creep resistance of crystals is stronger than that of non-crystals, the generated cristobalite can hinder the viscous flow of fused silica, thus greatly improving the creep resistance of the ceramic core. Moreover, the generated ZrSiO<sub>4</sub>, which has a melting point of 2,500 °C, is distributed in the matrix and acts as a pinning agent on the surface of the fused silica. This also effectively hinders the viscous flow of the liquid phase. Thus, the formation of cristobalite and ZrSiO<sub>4</sub> results in the core having extremely low deflection at 1,500 °C.

According to the sintering theory and the SEM and TEM results mentioned above, the schematic diagram of crack extension inside the ceramic core samples after 25 °C and 1,500 °C flexural strength test (both sintered at 1,200 °C) was established, as shown in Fig. 8. The premise assumption is that the ceramic powder is spherical. At 1,200 °C, the ceramic core is insufficiently sintered, with loosely arranged fine particles filling the gaps between larger SiO<sub>2</sub> particles. This results in low strength of its grain boundaries, and the core's fracture paths mainly propagate along these boundaries during fracture. After the ceramic core sample underwent a bending strength test at a temperature of 1,500 °C for 30 min, the ZrSiO<sub>4</sub> attached to the surface of the SiO<sub>2</sub> particles combines to form sintered necks, and the bonding degree of ceramic particles is enhanced significantly, improving the strength of the grain boundaries inside the ceramic core. The fracture mode of the ceramic core changes from intergranular fracture

to transgranular fracture, resulting in the high-temperature flexural strength of the core being much higher than its room-temperature flexural strength.

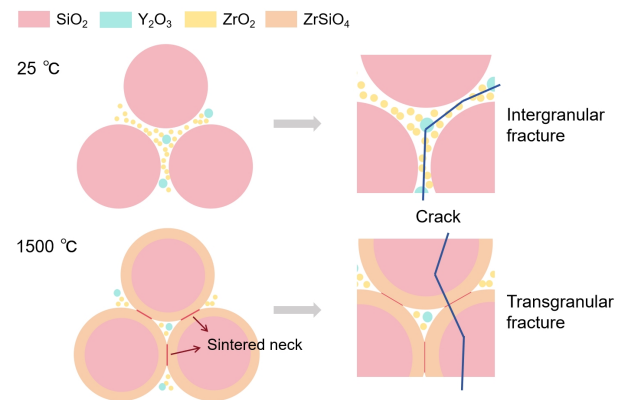


Fig. 8: Schematic diagram of internal crack extension in ceramic core samples after 25 °C and 1,500 °C flexural strength tests (both sintered at 1,200 °C)

## 5 Conclusions

Silica-based ceramic cores were prepared using SL 3D printing technology, and the raw material composition and debinding-sintering process of the cores were designed. The room temperature and high temperature properties of the samples were tested, the fracture morphology of samples was observed, and the phase constitution of the samples after sintering and high temperature flexural strength testing was analyzed. The main research results are as follows:

(1) As the sintering temperature is increased from 1,170 °C to 1,230 °C, the shrinkage and 25 °C and 1,500 °C flexural strength of the cores increase, while its open porosity and 1,500 °C deflection decrease. At sintering temperatures of 1,200 °C and above, the core exhibits excellent creep resistance. The sintering temperature of 1,200 °C is selected in conjunction with the comprehensive performance requirements for the ceramic cores. At this temperature, the sintered cores exhibit shrinkage rates of 3.76% (X), 3.38% (Y), and 3.95% (Z), with a flexural strength of 9.01 MPa at 25 °C and 32.15 MPa at 1,500 °C. The open porosity is 26.39%, and the deflection at 1,500 °C is 0.15 mm.

(2) The microstructure of the unsintered core specimen is laminar, and the laminar structure of the cross-section of the specimen sintered at 1,200 °C disappears, which is attributed to the decomposition of the photosensitive resin in the ceramic specimen, and the ceramic particles between the layers are diffused and bonded under thermodynamic action.

(3) The fracture mode of the ceramic core sample sintered at 1,200 °C at room temperature is mainly intergranular fracture, which dominates the low flexural strength of 9.01 MPa. During the 1,500 °C high-temperature flexural strength test, the main fracture mode of the sample is transgranular fracture, resulting in a significant increase in the flexural strength of the ceramic core to 32.15 MPa due to the generation of a large amount of cristobalite and ZrSiO<sub>4</sub> crystals.

(4)  $Y_2O_3$  and nanometer 3YSZ were added into coarse fused silica.  $Y_2O_3$  destroys the bridging oxygen in the Si-O bond, and promotes the crystallization reaction of fused silica to generate cristobalite. 3YSZ combines with fused silica at high temperatures to form  $ZrSiO_4$ . As both cristobalite and  $ZrSiO_4$  are crystals, their creep resistance is much higher than that of amorphous fused silica, thus greatly improving the deformation resistance of the core. The extremely low deflection helps maintain the dimensional stability of the ceramic core during casting.

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## Conflict of interest

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

## References

- [1] Backman D G, Williams J C. Advanced materials for aircraft engine applications. *Science*, 1992, 255(5048): 1082.
- [2] Wei Q, Zhong J, Xu Z, et al. Microstructure evolution and mechanical properties of ceramic shell moulds for investment casting of turbine blades by selective laser sintering. *Ceramics International*, 2018, 44: 12088–12097.
- [3] Wereszczak A, Breder K, Ferber M, et al. Dimensional changes and creep of silica core ceramics used in investment casting of superalloys. *Journal of Materials Science*, 2002, 37(19): 4235–4245.
- [4] Li Q L, Hou W Q, Liang J J, et al. Controlling the anisotropy behaviour of 3D printed ceramic cores: From intralayer particle distribution to interlayer pore evolution. *Additive Manufacturing*, 2022, 58: 103055.
- [5] Zhao X Z. Preparation and use of ceramic cores. Beijing: Science Press, 2013. (In Chinese)
- [6] Qin Y X, Pan W. Effect of silica sol on the properties of alumina-based ceramic core composites. *Materials Science & Engineering: A*, 2009, 508(1–2): 71–75.
- [7] Shabani S, Naghizadeh R, Golestanifard F, et al. Effect of  $TiO_2$  addition on microstructure and mechanical properties of alumina-based cores prepared by sol-gel method. *International Journal of Applied Ceramic Technology*, 2019, 16(6): 2409–2418.
- [8] Liang J J, Lin Q H, Zhang X, et al. Effects of alumina on cristobalite crystallization and properties of silica-based ceramic cores. *Journal of Materials Science & Technology*, 2017, 33(2): 204–209.
- [9] Guo W, Wang P F. Analysis of cooling configurations for turbine blade. *Aerospace Power*, 2020, 6: 55–58. (In Chinese)
- [10] Jiang J, Liu X Y. Dimensional variations of castings and moulds in the ceramic mould casting process. *Journal of Materials Processing Technology*, 2007, 189(1–3): 247–255.
- [11] Dong W J, Li Q L, Chen T C, et al. Effect of sintering temperature on microstructure and properties of 3D printing polysilazane reinforced  $Al_2O_3$  core. *China Foundry*, 2023, 20(5): 387–394.
- [12] Tang S Y, Yang L, Fan Z T, et al. A review of additive manufacturing technology and its application to foundry in China. *China Foundry*, 2021, 18(4): 249–264.
- [13] Sun J, Yu S, Wade-Zhu J, et al. 3D printing of layered ceramic/carbon fiber composite with improved toughness. *Additive Manufacturing*, 2022, 50: 102543.
- [14] Pham D T, Dimov S S. Rapid manufacturing: the technologies and applications of rapid prototyping and rapid tooling. London: Springer-Verlag, 2001.
- [15] Hu K H, Wang H Y, Lu K, et al. Fabrication of silica-based ceramic cores with internal lattice structures by stereolithography. *China Foundry*, 2022, 19(5): 369–379.
- [16] Chen Z W, Li Z Y, Li J J, et al. 3D printing of ceramics: A review. *Journal of the European Ceramic Society*, 2019, 39(4): 661–687.
- [17] Zakeri S, Vippola M, Levnen E. A comprehensive review of the photopolymerization of ceramic resins used in stereolithography. *Additive Manufacturing*, 2020, 35: 101177.
- [18] Li H, Liu Y S, Liu Y S, et al. Influence of debinding holding time on mechanical properties of 3D-printed alumina ceramic cores. *Ceramics International*, 2021, 47(4): 4884–4894.
- [19] Li H, Liu Y S, Liu Y S, et al. Evolution of the microstructure and mechanical properties of stereolithography formed alumina cores sintered in vacuum. *Journal of the European Ceramic Society*, 2020, 40(14): 4825–4836.
- [20] Park H Y, Kim E H, Choi H H, et al. New conversion process for fabricating a ceramic core by a 3D printing technique. *Surface & Coatings Technology*, 2017, 332: 527–532.
- [21] Zhang A P, Qu X, Soman P, et al. Rapid fabrication of complex 3D extracellular microenvironments by dynamic optical projection stereolithography. *Advanced Materials*, 2012, 24(31): 4266–4270.
- [22] Kim E H, Choi H H, Jung Y G. Fabrication of a ceramic core for an impeller blade using a 3D printing technique and inorganic binder. *Journal of Manufacturing Processes*, 2020, 53: 43–47.
- [23] Chao C H, Lu H Y. Optimal composition of zircon-fused silica ceramic cores for casting superalloys. *Journal of the American Ceramic Society*, 2010, 85(4): 773–779.
- [24] Li H, Liu Y S, Liu Y S, et al. Silica strengthened alumina ceramic cores prepared by 3D printing. *Journal of the European Ceramic Society*, 2020, 41(4): 2938–2947.
- [25] Wang X G, Zhou Y L, Zhou L, et al. Microstructure and properties evolution of silicon-based ceramic cores fabricated by 3D printing with stair-stepping effect control. *Journal of the European Ceramic Society*, 2021, 41(8): 4650–4657.
- [26] Li H, Hu K H, Liu Y S, et al. Improved mechanical properties of silica ceramic cores prepared by 3D printing and sintering processes. *Scripta Materialia*, 2021, 194: 1–4.
- [27] Li Q L, Hou W Q, Liang J J, et al. Controlling the anisotropy behaviour of 3D printed ceramic cores: From intralayer particle distribution to interlayer pore evolution. *Additive Manufacturing*, 2022, 58: 103055.
- [28] Zhan H X, Lu G, Yan Q S, et al. Research status of high temperature performance enhancement of  $SiO_2$  based ceramic cores. *Special Casting & Nonferrous Alloys*, 2020, 40(7): 749–752. (In Chinese)
- [29] Xu C M. Crystallization and amorphization of cristobalite. *Journal of Inorganic Materials*, 2007, 22(4): 577–582. (In Chinese)
- [30] Zeng Y. Study on the effects of  $Y_2O_3$  and cristobalite on the properties of silica oxide ceramic cores. Master's Dissertation, Harbin: Harbin Institute of Technology, 2014. (In Chinese)
- [31] Li Q L, Liang J J, Zhang Y L, et al. Fused silica ceramic core based on network-structured zircon design via 3D printing. *Scripta Materialia*, 2022, 208: 114342.
- [32] Zhou Y D. Physical chemistry of inorganic materials. Wuhan: Wuhan University of Technology Press, 1986. (In Chinese)