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High-temperature strength of gel casting silica-based ceramic core

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Abstract: Considering the fracture problem of the silica-based ceramic core in the integrated casting of hollow turbine blades during directional solidification, the influence of various whiskers, including silicon carbide whiskers, silicon nitride whiskers, and mullite whiskers, on the high-temperature strength of the silica-based ceramic core was investigated. Additionally, the formation of microstructure morphology and phase structure was analyzed. Research results show that silicon carbide whiskers can reduce the microcracks caused by the shrinkage of cristobalite. During the sintering process, some of the silicon carbide whiskers oxidize and react with aluminum powder to form mullite, which can improve the high-temperature strength of the ceramic cores. When the content of silicon carbide whiskers is 3wt.%, the high-temperature bending strength of the cores reaches the maximum value of 21 MPa. Silicon nitride whiskers decompose in a high-temperature environment and react with aluminum powder in the matrix material to form mullite whiskers. When the content of silicon nitride whiskers is 5wt.%, the high-temperature bending strength of the cores reaches 20 MPa. By adding mullite whiskers, a structure of cristobalite wrapped mullite whiskers can be formed to achieve toughening. When the content of mullite whiskers is 4wt.%, the high-temperature bending strength can reach 17.2 MPa. By comparing the performance of silicon carbide whiskers, silicon nitride whiskers, and mullite whiskers, along with conducting slurry viscosity tests and casting experiments, it is determined that a ceramic slurry containing 4wt.% mullite whiskers is the most suitable for making the cores used in the integrated casting of hollow turbine blades.

Keywords: hollow turbine blades; ceramic core; gel-casting; high-temperature strength; whisker

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1 Introduction

Turbine blades are important components of aircraft engines and gas turbines. The structure and manufacturing quality of the blades have a direct impact on the overall performance of engines and gas turbines [1, 2]. In the process of forming hollow blades through investment casting, ceramic cores must withstand various complex and harsh conditions, as well as the impact of high-temperature alloy liquids. During the sintering process, it is imperative to minimize the shrinkage rate of the ceramic core as much as possible to prevent significant deformation or shifting of the mold.

With the increasing complexity of the internal structure of turbine blades, efficiently removing ceramic cores after casting is also a major challenge in manufacturing turbine blades with complex cavities and small channels. Therefore, ceramic core materials must exhibit a low sintering shrinkage rate, sufficient high-temperature strength, good chemical compatibility with molten high-temperature alloys, and high thermal shock resistance [3, 4].

At present, there are two main materials for preparation of cores used for hollow turbine blade: alumina and silicon dioxide. The alumina-based ceramic core is made of Al_2O_3 as the matrix material, which does not react with acid or alkali corrosive solution at room temperature. This makes it difficult to remove the cores, especially for the blades which have complex internal cavities. Compared to alumina-based ceramics, silica-based ceramics have a faster core removal rate, establishing them as the current mainstream ceramic core material [5-7]. However, the low high-temperature strength of silica-based ceramics makes them prone

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to creep and other problems after long-term immersion in high-temperature liquid metals. Therefore, improving the high-temperature strength of silica-based ceramics has become the most critical issue nowadays.

The predominant focus of current research lies in optimizing the injection molding process to enhance the strength of silica-based ceramics. This is achieved through the incorporation of materials such as alumina and zirconia, aiming to generate a high-temperature strengthening phase characterized by cristobalite, mullite, and zirconium (IV) silicate. Additionally, micro-crack toughening is employed as another avenue to augment the material's strength [8-10]. This is realized by generating dispersed micro-cracks to guide the deflection and branching of the main crack, and by increasing the surface energy during the expansion process. However, both methods have many drawbacks. Although phase transition toughened ceramics are effective, they exhibit high sensitivity to temperature changes. Meanwhile, although micro-crack toughening can help improve strength, it may also damage the structural integrity of the material and reduce its thermal stability. Therefore, the effectiveness of both methods is significantly limited [11, 12].

Whiskers have ultra-high strength and modulus due to their extremely small size and almost defect-free microstructure. Consequently, whiskers emerge as an ideal material for reinforcing silica-based ceramic matrix. Silicon carbide whiskers are widely used to reinforce metal-based materials, ceramic-based materials, and other composites due to their excellent tensile strength, elastic modulus, thermal conductivity and thermal expansion coefficient. Garnier et al. [13] significantly improved the thermomechanical properties of alumina ceramics by adding SiC whiskers. Kargin [14] observed that using Al_2O_3-CaO as a sintering aid and SiC whiskers to reinforce Si_3N_4-based ceramics resulted in the formation of uniformly distributed dendritic SiC crystals, which created a high-temperature skeleton. Silicon nitride whiskers have good physical compatibility with silica, and possess better high-temperature strength, expansion performance, and chemical stability than silicon carbide whiskers. It is considered to be an ideal component for reinforcing ceramic materials [15, 16]. Moreover, the volume expansion of silicon nitride whiskers is high after decomposition, which can effectively compensate the shrinkage of the core. Mullite whiskers have excellent high-temperature resistance, with a melting point above 2,000 °C and a maximum operating temperature of 1,500 °C to 1,700 °C. In addition, the thermal shock resistance and chemical stability of mullite whiskers are excellent. It is also an excellent material for improving the mechanical properties of ceramic matrix [17].

The gel casting process can be utilized to achieve the solidification and molding of whisker-reinforced slurry. This technique can be integrated with additive manufacturing technology to accomplish near-net-shape production, which significantly reduces the preparation cycle, thereby enhancing efficiency and yield [18, 19]. Since the main component of the acrylamide system commonly used in gel injection molding is a neurotoxin, which can easily cause harm to staff, current research on gel injection molding primarily focuses on the low-toxic and non-toxic slurry systems [20-22]. However, there are few reports on how whiskers can improve the high-temperature strength of gel injection slurry.

In this study, a silica-based ceramic slurry suitable for the gel casting process was designed. Different quantities of silicon carbide whiskers, silicon nitride whiskers, and mullite whiskers were introduced into the ceramic slurry. Subsequently, standard samples were sintered for comprehensive phase analysis and microstructure characterization. The experiments elucidated the effects imparted by whisker types and content on the key properties of silica-based ceramic cores. Moreover, to verify the findings, computed tomography (CT) analysis was employed on ceramic cores prepared using the mullite whisker reinforced slurry. The outcomes provide valuable insights into optimizing the composition of ceramic slurries for enhanced core properties.

2 Materials and methods

2.1 Raw materials

The matrix material consisted of silica powder with particle sizes of 100 μm, 40 μm, 5 μm, and 2 μm. Aluminum powder and nano-zirconia were used as the sintering additives, deionized water was used as the solvent, with acrylamide used as the organic monomer. N, N'-methylenebisacrylamide and sodium polyacrylate were used as the crosslinking agent and dispersant, respectively. Ammonium persulfate was used as the initiator and N, N, N', N'-tetramethylethylenediamine was used as the catalyst. Silica sol was used as the impregnation solution. Silicon carbide whiskers of industrial grade, 99.9% purity, from Brofos Nanotechnology (Ningbo) Co., Ltd., silicon nitride whiskers from Juyuan Plastic Agent Chemical, and mullite whiskers were used as the reinforcement phase.

2.2 Mullite whiskers preparation

Mullite whiskers were prepared by sintering a mixture of alumina and silica, as shown in Fig. 1(a). The materials used for preparing mullite were γ-alumina (γ-Al_2O_3, chemical pure, Shaanxi Hammer Biological Technology), spherical silica (SiO_2, chemical pure, Jiangsu Hui Mai Powder Technology Co., Ltd.), and aluminum fluoride ($AlF_3{\cdot}3H_2O$, chemical pure). The microscopic morphology of the prepared mullite whiskers is shown in Fig. 1(b). It can be seen that the aspect ratio of the mullite whiskers is as high as 32:1. Mullite whiskers with a high aspect ratio significantly enhance the strength of the ceramic matrix [20], but a higher aspect ratio also makes the whiskers susceptible to damage during high-speed ball milling, as shown in Fig. 1(c). In order to maintain the integrity of mullite whiskers, silicon powder, aluminum powder, and zirconia were added to a ball mill at first. The grinding balls were then added according to the feed ball ratio (in volume) of 1:1 and ball milled at a speed of 360 $r{\cdot}min^{-1}$ for 40 min to prepare a premix. Then, mullite whiskers were added to the premix, smaller

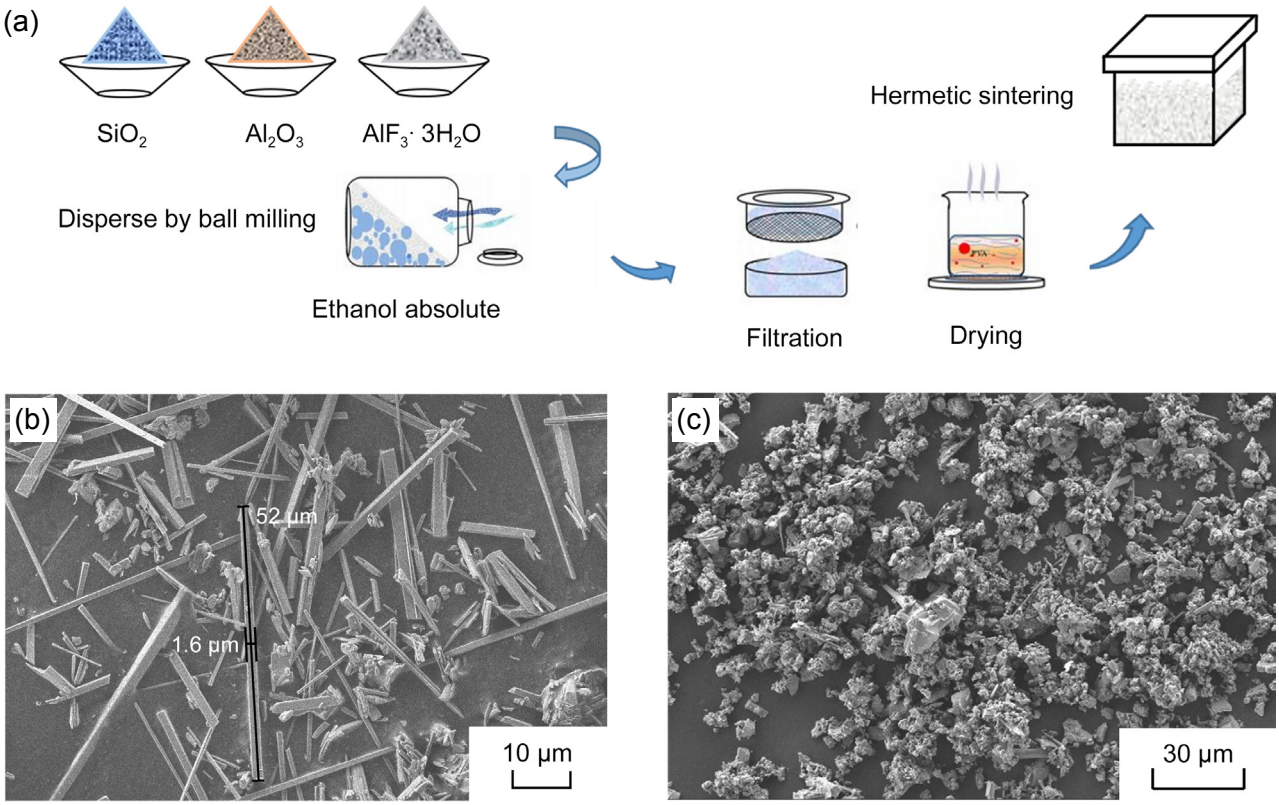

Fig. 1: Preparation process of mullite whiskers (a), and morphology of mullite whiskers before (b) and after (c) ball milling

grinding balls in the feed ball ratio (in volume) of 10:1 were used, and ball milled at a low speed (100 r·min^{-1}) for 20 min to reduce damage to the whiskers during the ball milling process. The components of mullite whisker reinforced silica-based ceramic are shown in Table 1.

2.3 Slurry preparation

Based on the previous research results, the mass ratio of 100 μm, 40 μm, 5 μm, 2 μm particles in ceramic slurry powder was determined to be 21.1:40.6:21.6:14.56, according to the Funk Dinger (F-D) distribution function [21] and the theory of closest packing of particles. The remaining composition comprised sintering agents and other components.

The length of silicon carbide (SiC) whiskers used in this experiment is 5 μm, and the length of silicon nitride (Si_3N_4) whiskers is approximately 40 μm. The micromorphologies of silicon carbide whiskers and silicon nitride whiskers observed under electron microscopy are shown in Figs. 2(a) and (b), respectively. Silicon nitride whiskers have a small aspect ratio, and some of them are granular. To ensure the consistency of the mass ratio of various particle sizes in the slurry, the amount of 5 μm and 40 μm silica powder was correspondingly reduced in both reinforced slurries. The components of silicon carbide whisker reinforced and silicon nitride whisker reinforced silica-based ceramics are shown in Tables 2 and 3.

A premixed solution was prepared by uniformly mixing deionized water, acrylamide, N, N'-methylenebisacrylamide, and sodium polyacrylate. The silica powder, aluminum powder, zirconia powder, and whiskers were added to the pre-mixed solution, and grinding balls were then added to the premix material in a volume ratio of 1:1, and finally, ceramic slurry with a high solid phase and low viscosity was obtained by ball milling at 360 r·min^{-1} for 40 min.

Table 1: Mullite whisker reinforced silica-based ceramics

Sample	Mass fraction (wt.%)							
	SiO_2 (100 μm)	SiO_2 (40 μm)	SiO_2 (5 μm)	SiO_2 (2 μm)	Al (40 μm)	ZrO_2 (2 μm)	Mullite whisker	Organosilicon
M2	22.1	28.9	21.6	12.4	9.7	2.16	2	1.14
M4		26.9					4	
M6		24.9					6	
M8		22.9					8	

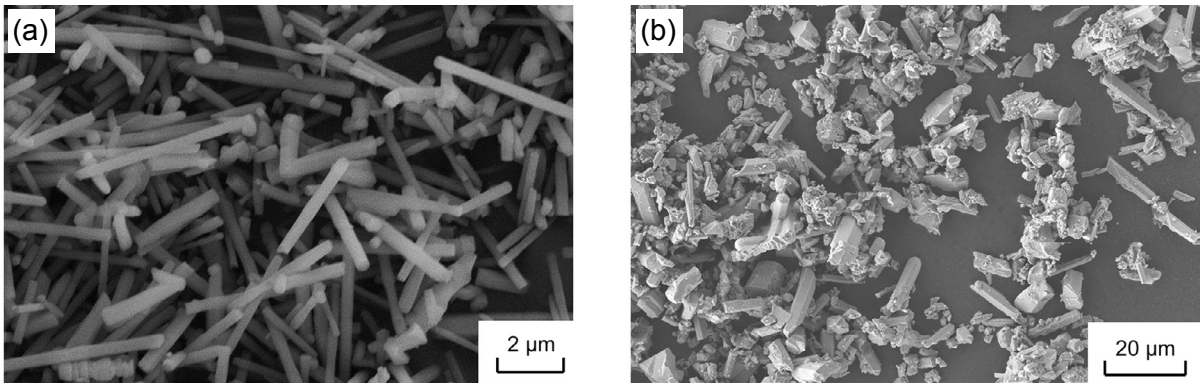

Fig. 2: Morphology of silicon carbide whiskers (a) and silicon nitride whiskers (b)

Table 2: Silicon carbide whisker reinforced silica-based ceramics

Sample	Mass fraction (wt.%)							
	SiO_2 (100 μm)	SiO_2 (40 μm)	SiO_2 (5 μm)	SiO_2 (2 μm)	Al (40 μm)	ZrO_2 (2 μm)	SiC whisker	Organosilicon
C0			21.6				0	
C1			20.6				1	
C2	22.1	30.9	19.6	12.4	9.7	2.16	2	1.14
C3			18.6				3	
C4			17.6				4	

Table 3: Silicon nitride whisker reinforced silica-based ceramics

Sample	Mass fraction (wt.%)							
	SiO_2 (100 μm)	SiO_2 (40 μm)	SiO_2 (5 μm)	SiO_2 (2 μm)	Al (40 μm)	ZrO_2 (2 μm)	Si_3N_4 whisker	Organosilicon
S0		30.9					0	
S5	22.1	25.9	21.6	12.4	9.7	2.16	5	1.14
S10		20.9					10	

2.4 Sample preparation

The samples were prepared by gel casting molding. The N, N, N', N'-tetramethylethylenediamine and ammonium persulfate were added to the slurry in sequence, and the slurry was injected into the standard molds at 20 °C. After solidified, the slurry sample underwent a series of processes including freeze-drying, pre-sintering, vacuum impregnation strengthening, and final-sintering to obtain the final samples. The pre-sintering and final-sintering processes parameters are shown in Table 4.

Table 4: Pre-sintering process

Temperature (°C)	Heating rate/holding temperature/holding time
0–200	30 °C·h^{-1}/200 °C/1 h
200–300	40 °C·h^{-1}/300 °C/1 h
300–400	40 °C·h^{-1}/400 °C/1 h
400–600	100 °C·h^{-1}/600 °C/1 h
600–900	100 °C·h^{-1}/900 °C/1 h

2.5 Measurement and characterization procedures

Sample strips of 60 mm×10 mm×4 mm were prepared, and the high-temperature bending strength of the samples was obtained by testing 6 samples as a group. The high-temperature bending strength was tested according to the Industrial Standard HB5353: Performance test method of investment casting ceramic core. A vernier caliper was used to measure

Table 5: Final sintering process

Temperature (°C)	Heating rate/holding temperature/holding time
0–300	60 °C·h^{-1}/300 °C/30 min
300–700	100 °C·h^{-1}/700 °C/2 h
700–1,200	100 °C·h^{-1}/1,200 °C/3.8 h

the size change of the sample in the length direction and the shrinkage rate was calculated. The high-temperature deflection was tested also according to the Industrial Standard HB5353. The viscosity of ceramic slurry was measured using a rotary rheometer MCR302 and the reaction temperature was analyzed using a thermogravimetric analyzer. The microstructure was characterized using a field emission scanning electron microscope (SEM) and the phase constitution was analyzed using X-ray detection (XRD). The high-temperature strength of the sample was tested using a high-temperature stress-strain machine at 1,500 °C.

3 Results and discussion

3.1 Effect of silicon carbide whiskers on high-temperature strength of ceramics

The XRD phase analysis of the sample with a silicon carbide whisker content of 4wt.% is shown in Fig. 3. The main component is silicon dioxide (SiO_2), with diffraction peaks at 21.962° and 26.692° being more prominent, while the diffraction peak at 20.923° is lower, indicating that quartz and cristobalite occupy the majority of the sample, and the proportion of tridymite is relatively small. The content of cristobalite is the highest at a diffraction peak of 26.692°. This is because silicon carbide whiskers can serve as seed-crystals for cristobalite under atmospheric heating conditions, forming amorphous silicon dioxide through surface oxidation, as shown in Eqs. (1) and (2), which ultimately transforms into cristobalite when crystallized at 1,200 °C.

$$\mathrm{SiC(s)}+\frac{3}{2}\mathrm{O_2(g)}\rightarrow\mathrm{SiO_2(s)}+\mathrm{CO(g)} \quad (1)$$

$$\mathrm{SiC(s)}+2\mathrm{O_2(g)}\rightarrow\mathrm{SiO_2(s)}+\mathrm{CO_2(g)} \quad (2)$$

Figure 4 illustrates the variations in high-temperature strength and shrinkage rate of silica-based ceramics at 1,500 °C with varying silicon carbide whisker contents. Figure 4 clearly depicts that as the silicon carbide whisker content increases, both high-temperature strength and shrinkage rate follow an initially upward trend, reaching a peak before subsequently decline. At a silicon carbide whisker content of 3wt.%, the high-temperature strength reaches its maximum value of 21 MPa. However, with a further increase in whisker content to 4wt.%, a decline in high-temperature strength to 17 MPa is observed. It is speculated that the decrease in high-temperature strength may be due to that the increase in whisker content causing additional microcracks.

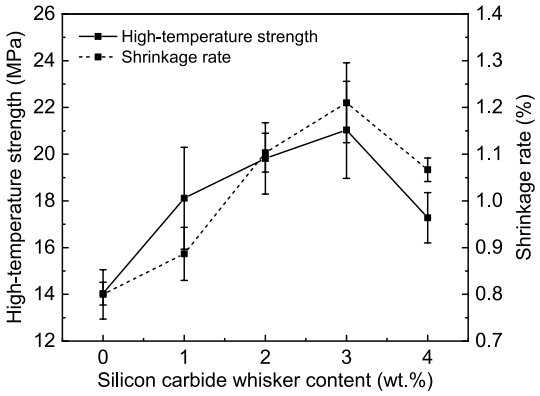

Fig. 4: Effect of SiC whisker content on high-temperature strength and shrinkage

When the silicon carbide whisker content is no more than 3wt.%, the whiskers are closely combined with the matrix material, and the generated cross structure (Fig. 5) can effectively improve the fracture toughness of the material by restraining the interface fracture through the whisker pulling out. At the same time, amorphous silicon dioxide, generated by the oxidation of silicon carbide whiskers, reacts with aluminum powder to form mullite whiskers, which accelerates the viscous flow sintering of silicon dioxide. This process promotes the viscous flow, makes the sintering neck thicker, and ultimately makes the sintered sample denser. Therefore, both the high-temperature strength and shrinkage rate increase with the increase of whisker content.

When the content of whiskers exceeds 3wt.%, excessive whiskers severely reduce the sintering performance of the ceramic and significantly increase the porosity. In addition, silicon carbide whiskers can affect the generation amount and location of cristobalite, thereby affecting the size and location of microcracks. Figure 6 shows the influence of silicon carbide whiskers content on microcracks. With the increase of silicon carbide whisker content, the gaps between microcracks become wider and the crack extension distance lengthens.

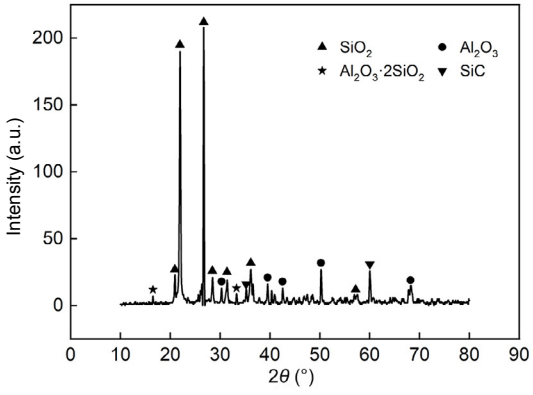

Fig. 3: XRD pattern of 4wt.% SiC whisker reinforced samples

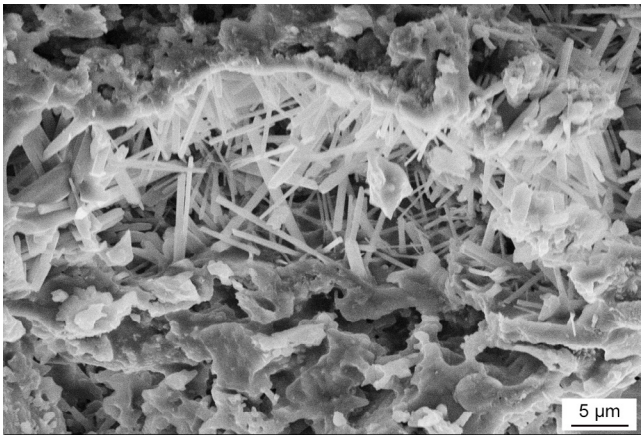

Fig. 5: Cross structure generated by silicon carbide whisker

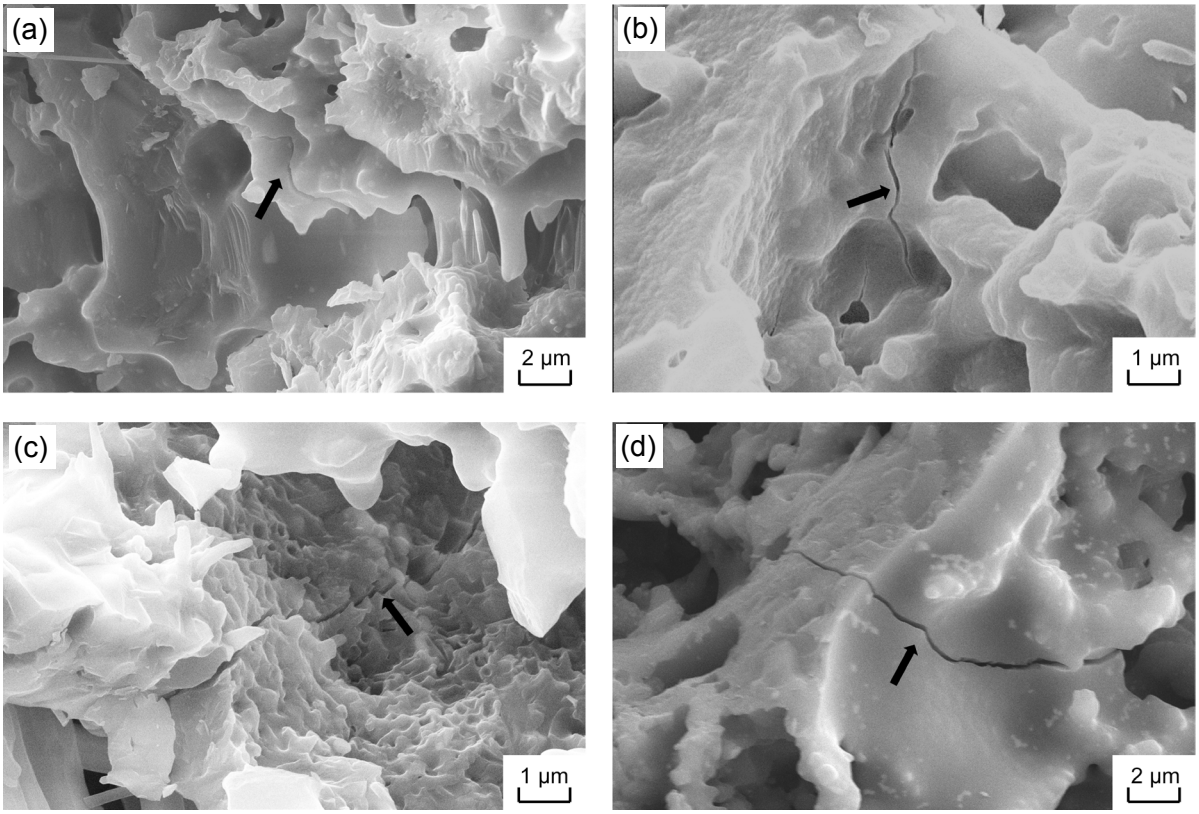

Fig. 6: Effect of silicon carbide whisker content on microcracks: (a) 1wt.%; (b) 2wt.%; (c) 3wt.%; (d) 4wt.%

At this point, whisker toughening no longer dominates, and the increase in porosity and the propagation of microcracks significantly degrade the high-temperature strength of ceramics and reduce the shrinkage rate.

3.2 Effect of silicon nitride whiskers on high-temperature strength of ceramics

Figure 7 shows the phase constitution of the samples with silicon nitride whiskers. The silicon nitride whiskers generate mullite phase when being heated in an atmospheric environment, as shown in Eqs. (3–5). Figures 8(a) and (d) show the morphology of Samples S5 and S10 after sintering, respectively. It can be seen that due to the different amounts of silicon nitride whiskers addition, Sample S5 is fully sintered, while in Sample S10, there are still large particles that have not been fully sintered. This is because too many silicon nitride whiskers hinder the viscous flow of silica and have an inhibitory effect on the sintering process. Therefore, in practical application, it is necessary to limit the amount of silicon nitride whiskers added to ensure the sintering effect. Figures 8(c) and (f) show the morphology of whiskers, with needle shaped mullite whisker clusters around the long and thick columnar mullite and silicon nitride whiskers, with a aspect ratio of approximately 10:1.

$$Si_3N_4 + 3O_2 = 3SiO_2 + 2N_2 \quad (3)$$

$$4Al + 3O_2 = 2Al_2O_3 \quad (4)$$

$$3Al_2O_3 + 2SiO_2 = 3Al_2O_3 \cdot 2SiO_2 \quad (5)$$

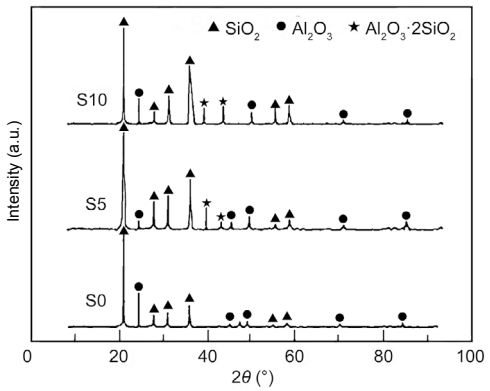

Fig. 7: Phase analysis of samples with different silicon nitride whisker contents

To prevent oxidation, the silicon nitride whiskers are completely decomposed or coated with a dense layer of silicon dioxide on the surface, making their morphology difficult observed directly. Therefore, energy spectrum analysis was performed on Sample S5. The results in Fig. 9 show that the percentage of N atoms of the whisker at Position I is 8.49at.%, and the mass fraction is about 5.71wt.%, indicating that these are silicon nitride whiskers coated with silica. The whiskers at Position II do not contain N, indicating that these whiskers are mullite whiskers. The whisker group here exhibits a whisker cross structure formed by mullite whisker clusters around Si_3N_4 whiskers.

As shown in Fig. 10, with the increase of silicon nitride whisker content, the sintering shrinkage rate of the sample continuously decreases. There are two main reasons: On the one hand, the volume expansion of silica generated by the

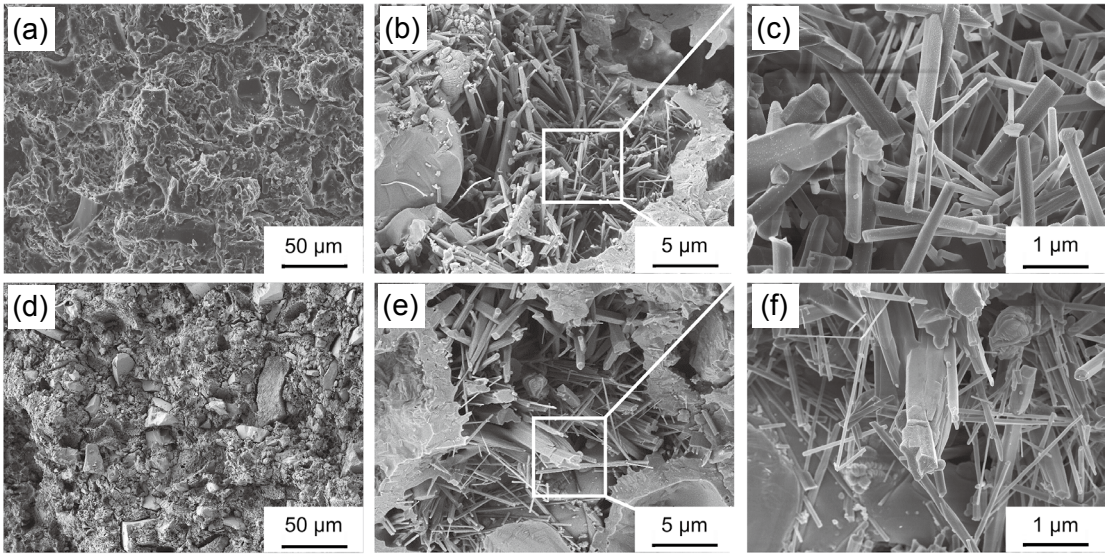

Fig. 8: Morphology of Samples S5 and S10 after sintering: (a–c) Sample S5; (d–f) Sample S10

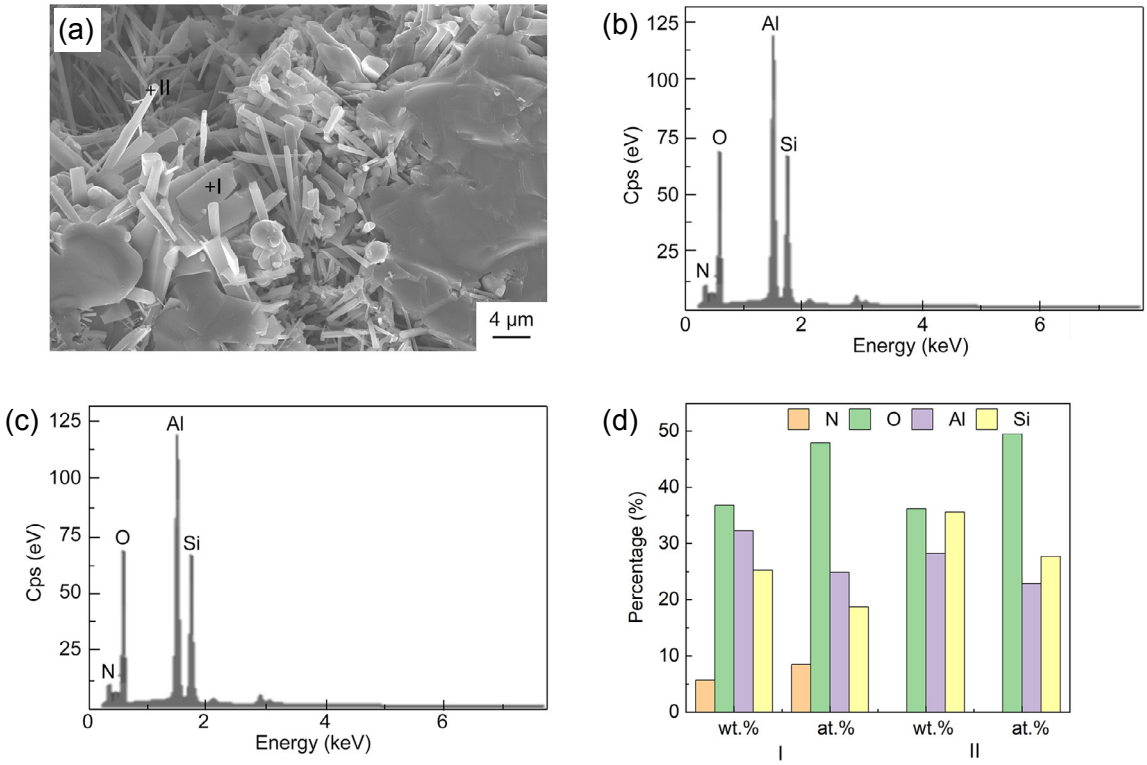

Fig. 9: Energy spectrum analysis of Sample S5: (a) whisker electron microscope diagram; energy spectrum analysis at Position I (b), Position II (c), and element content at Positions I and II (d)

decomposition of silicon nitride whiskers compensates for the shrinkage generated during sintering. On the other hand, the thermal expansion coefficients of mullite whiskers and silicon nitride whiskers are relatively small. The staggered grid structure formed in the ceramic matrix serves as a skeleton, inhibiting the viscous flow of silica at high temperatures and reducing shrinkage and bridging cracks at low temperatures.

The trend of high-temperature strength is similar to that of silicon carbide whisker samples: when the content of whiskers is below 5wt.%, the toughening effect of whiskers plays a dominant role, and the interlaced structure formed by mullite whiskers and dense silica layer coated silicon nitride whiskers play a bridging role [Fig. 9(a)]. When subjected to external forces, cracks will deflect under the action of whiskers, and the energy absorbed by whisker debonding, pulling out, and fracture also improves the toughness of ceramics to a certain extent. Compared to the silica-based ceramic Sample S0 without the addition of silicon nitride whiskers, the high-temperature strength of Sample S5 with the addition of silicon nitride whiskers is significantly increased from 14 MPa to 20 MPa. However, when the content of whiskers exceeds 5wt.%, the rearrangement ability of the whiskers is decreased. This weakens the bond between the whiskers and the matrix (Fig. 11), resulting in a weakened toughening ability of the whiskers on the sample.

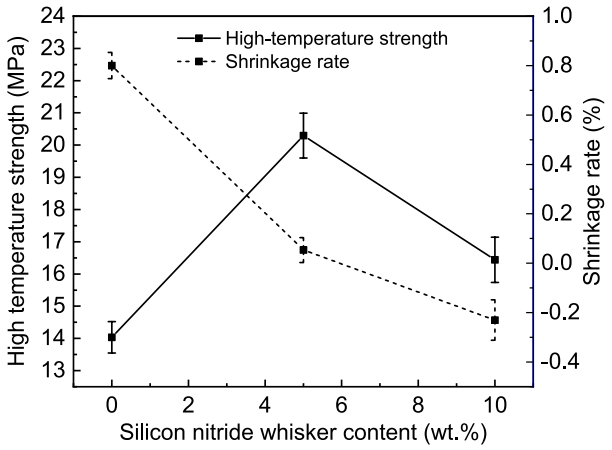

Fig. 10: Effect of silicon nitride whisker content on high-temperature strength and shrinkage

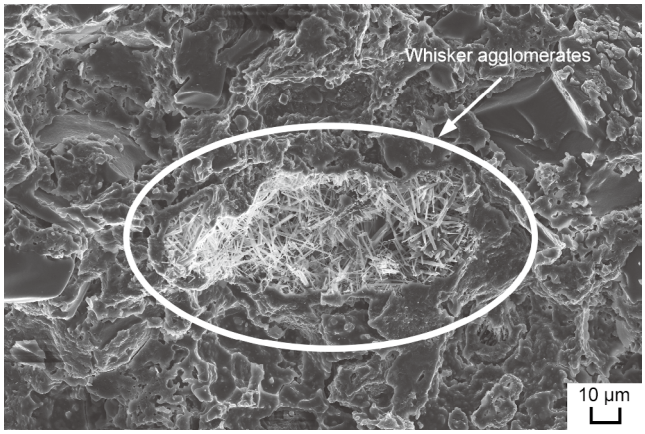

Fig. 11: Whisker agglomerates observed in S10 sample

3.3 Effect of mullite whiskers on high-temperature strength of ceramics

Mullite whiskers have a significant impact on the high-temperature strength and shrinkage rate of the core. The first reason is that mullite whiskers can act as the second phase during the sintering process of ceramic cores, hindering atomic diffusion and mass transfer. Due to the large aspect ratio and low surface energy of whiskers, the viscosity of the slurry becomes higher, resulting in a lower shrinkage rate and higher porosity of the ceramic cores [22]. The elongated mullite whiskers in Figs. 12(a) and (c) bridge the matrix material, exerting a pulling effect on the silica-based ceramic material and transferring part of the stress to the whiskers. The second reason is that the extraction and fracture of whiskers consume energy. As shown in Fig. 12(b), the extraction or fracture of mullite whiskers wrapped in cristobalite from the matrix effectively improves the high-temperature strength of the material. The third reason is that mullite whiskers are uniformly dispersed in the matrix of the ceramic cores, forming a high-temperature skeleton that can effectively suppress the viscous flow of silica and suppress microcracks caused by the shrinkage of square quartz. From the serrated cracks in Fig. 12(d), it can be found that mullite whiskers promote crack deflection and improve the toughness of the ceramic core.

Energy spectrum analysis was conducted on the samples reinforced with mullite whiskers at 1,200 °C, as shown in Fig. 13. The energy spectrum analysis shows that the surface of the sample is mainly composed of Si and O elements, and its surface morphology is mainly irregular aggregates of equigranular crystals with polygonal morphology. Therefore,

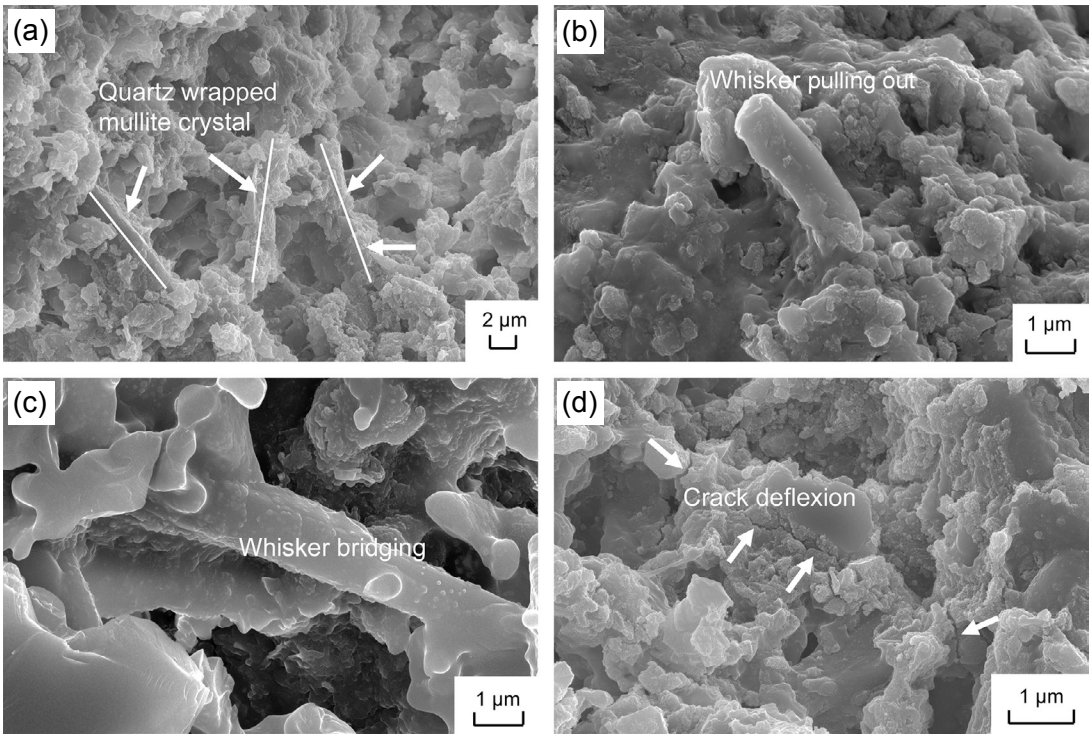

Fig. 12: Microtopography of mullite reinforced ceramics: (a) quartz wrapped mullite whisker; (b) whisker pulling out; (c) whisker bridge; (d) crack deflection

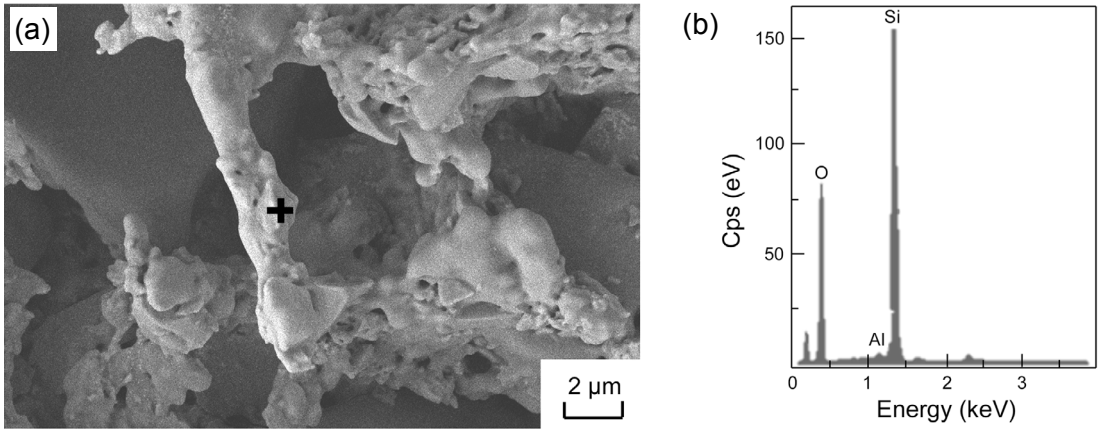

Fig. 13: Energy spectrum analysis of mullite whisker wrapped by cristobalite: (a) electron microscope diagram of mullite whisker wrapped by cristobalite; (b) distribution of element

it can be inferred that the aggregates on the surface of mullite whiskers are cristobalite. It indicates that during the sintering process, the mullite whiskers are stable and do not react with the quartz matrix and Al powder. The mullite whiskers act as heterogeneous phases, and the silica in the surrounding quartz and silica sol forms cristobalite crystals attached to the surface of the mullite whiskers. With the extension of the holding time, the generated cristobalite crystals eventually wrap around mullite whiskers, as shown in Figs. 12 and 13. The columnar structure formed by wrapping cristobalite with whiskers can form a high-temperature skeleton, and the presence of whiskers reduces the detachment of cristobalite from the matrix, achieving a toughening effect similar to that of ceramic fiber composites.

Figure 14 shows the high-temperature strength and shrinkage rate of the sample with the addition of mullite whiskers at 1,500 °C. The high-temperature strength of the sample with 4wt.% mullite whiskers reaches 17.2 MPa, and the shrinkage rate is only about 0.6%. Compared with the sample without mullite whiskers, the strength is increased by about 20%, and the shrinkage rate is decreased by about 30%. Excessive crystal whiskers can affect the viscous flow of silica, leading to insufficient sintering and reducing the precipitation of cristobalite on the surface of silica. This weakens the inhibitory effect of cristobalite on the viscous flow of silica at 1,500 °C,

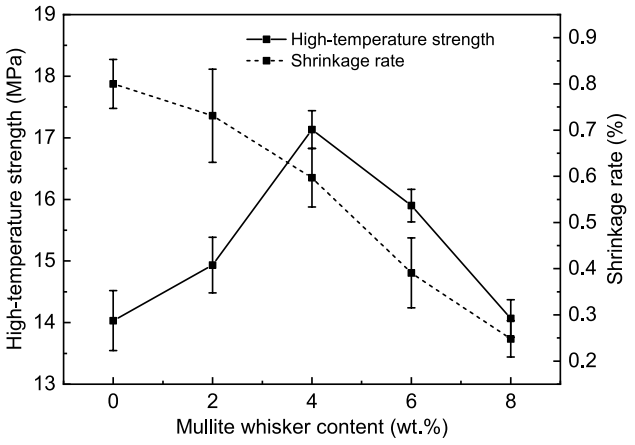

Fig. 14: Effect of mullite whisker content on high-temperature strength and shrinkage rate at 1,500 °C

ultimately resulting in a decrease in high-temperature strength with the increase of whisker content. There are two main reasons: first, the increased content of mullite whiskers inhibits sintering, leading to insufficient densification; second, the high-temperature skeleton effect of mullite whiskers restricts sintering shrinkage.

4 Gel casting experiment of ceramic core

In the actual production process, the ceramic core is produced by gel casting, and its production process mainly includes six steps: preparation of photocurable resin mold, preparation of ceramic green body, freeze drying, pre-sintering, impregnation strengthening, and final sintering.

Three kinds of slurries with the best comprehensive performance were selected for ceramic core gel casting testing. They are the 3wt.% silicon carbide whisker slurry, 5wt.% silicon nitride whisker slurry, and the 4wt.% mullite whisker slurry. The high-temperature deflection and rheological properties were compared with those of silica ceramic slurry without addition of whiskers.

Figure 15(a) shows the high-temperature deflection of silica-based ceramics with different types of whiskers at 1,500 °C. The deflection of the ceramic core without the addition of whiskers is 1.26 mm, while, the deflection is reduced to 0.64 mm for the sample with 3wt.% silicon carbide whisker, and further reduced to 0.58 mm for the sample with 5wt.% silicon nitride whisker. The sample with 4wt.% mullite whisker has a high-temperature deflection of 0.57 mm. These three types of whiskers effectively reduce the deformation of the ceramic core at high temperatures. On the one hand, as a high-temperature phase, the whiskers inhibit the viscous flow of fused silica, thereby reducing high-temperature creep. On the other hand, the core's strength is enhanced through microcrack bridging and whisker extraction, preventing sample deformation at high temperatures.

Figure 15(b) shows the effect of different whiskers on the rheological properties of ceramic cores. The viscosities of the four ceramic slurries at a shear rate of 100 s^{-1} are 0.72 Pa·s,

0.464 Pa·s, 0.803 Pa·s, and 0.458 Pa·s, respectively. The viscosity meets the pouring conditions of less than 1 Pa·s at a shear rate of 100 s^{-1} [23]. Among them, the viscosity of the slurry with silicon nitride whiskers is slightly higher than the ceramic slurry without whiskers, while the addition of silicon carbide whiskers and mullite whiskers decreases the viscosity of slurries to a certain extent. Improved fluidity is beneficial for enhancing the success rate of core forming and preventing the defect associated with insufficient filling of core details.

Among the three types of whiskers: silicon carbide whiskers, silicon nitride whiskers, and mullite whiskers, the high cost of silicon nitride whiskers is not conducive to practical production. Although the preparation process of silicon nitride whiskers is relatively mature at present, the N element in them can react with elements such as Al, Ti, and Nb in high temperature, generating brittle phases, deteriorating the high-temperature mechanical properties and corrosion resistance of castings [24].

To summarize, considering the effects of whiskers on the high-temperature strength, high-temperature deflection, and shrinkage rate of the ceramic cores, as well as cost issues in practical applications, the optimal formulation selected for the core pouring verification experiment is a ceramic slurry with 4wt.% mullite whiskers. The experimental results are shown in Fig. 16. It can be seen that the ceramic core sample possesses a complete shape with high surface quality, and no defects are found at key complex structures such as the exhaust edge and U-shaped tube. This confirms the feasibility of using mullite whisker slurry for the rapid preparation of silica-based ceramic cores.

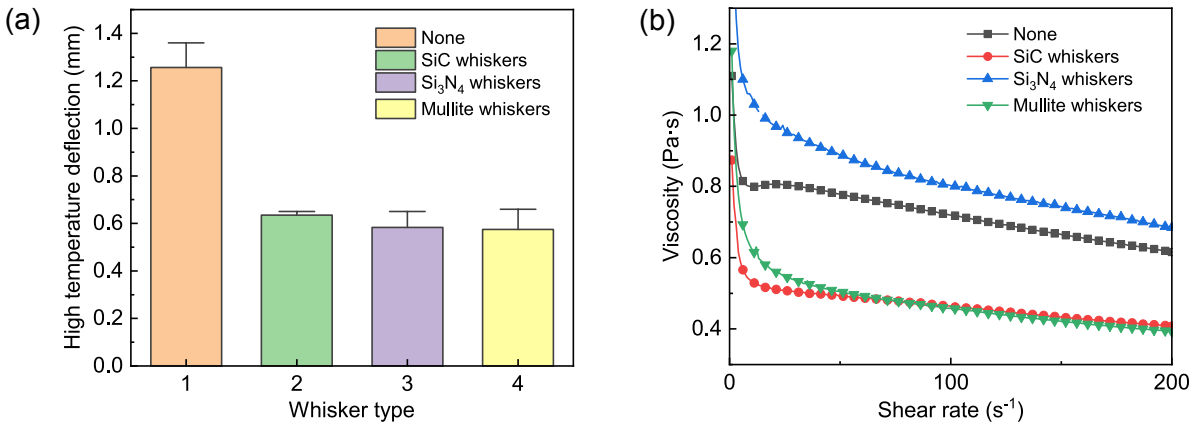

Fig. 15: Comparative testing of slurry performance: (a) high-temperature deflection test; (b) rheological test

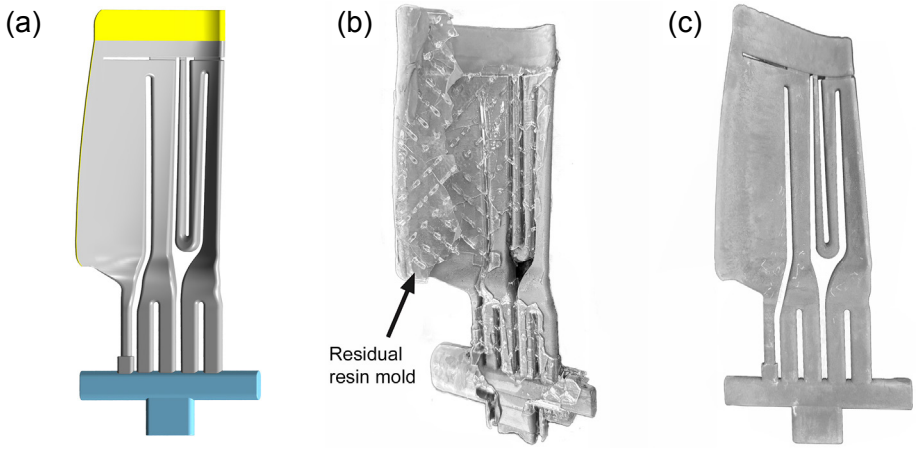

Fig. 16: A certain type of hollow turbine blade ceramic core: (a) digital model; (b) green body by gel casting; and (c) ceramic core after sintering

5 Conclusions

This study involved the design and utilization of a silica-based ceramic slurry in the gel casting process, where different types and contents of whiskers were incorporated to assess their influence on high-temperature mechanical properties and shrinkage. The key findings and analysis are as follows:

(1) Microscopic characterization shows that silicon carbide whiskers can reduce microcracks caused by shrinkage of calcite. Some of the silica generated after the decomposition of silicon carbide whiskers can react with aluminum powder to form mullite whiskers, both of which have an enhancing effect on high-temperature strength. When the content of silicon carbide whiskers is 3wt.%, the high-temperature bending strength reaches the maximum value of 21 MPa.

(2) Silicon nitride whiskers are easy to decompose in high-temperature environments and react with aluminum powder in the material to synthesize mullite whiskers in situ. When the

content of silicon nitride whiskers is 5wt.%, the high-temperature strength reaches 20 MPa and nearly zero shrinkage is achieved.

(3) By heating a mixture of alumina and silica, mullite whiskers with a aspect ratio of 20:1 and a length of 50 μm were prepared. A structure of cristobalite wrapped mullite whiskers is formed in the matrix material, and the high-temperature strength can reach 17.2 MPa when the content of mullite whiskers is 4wt.%.

(4) Considering both performance indicators and cost factors, a ceramic slurry containing 4wt.% mullite whiskers was selected. Ceramic core gel casting experiment using this slurry formulation results in complete, well-shaped ceramic cores with high surface quality. Key complex structures, such as exhaust edges and U-shaped pipes, display no defects, confirming the feasibility of the mullite whisker slurry for the rapid preparation of silica-based ceramic cores.

Conflict of interest

Prof. Zhong-liang Lu is an EBM of *CHINA FOUNDRY*. He was not involved in the peer-review or handling of the manuscript. The authors have no other competing interests to disclose.

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