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Preparation of silica ceramic cores by the preceramic pyrolysis technology using silicone resin as precursor and binder

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HIGHLIGHTS

- Silica ceramic cores were prepared by a preceramic pyrolysis technology.
- Silicone resin had the good cohesiveness in shaping silica ceramic cores.
- Silicone resin transformed into some small amorphous silica particles after pyrolysis.
- The properties of ceramic cores were affected by silicone resin contents.

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ABSTRACT

A facile and effective preceramic pyrolysis technology was used to prepare the fused silica ceramic cores, among which fused silica powders were used as the raw materials and the silicone resin with -Si-O-Si- as the main chains was used as the precursor. Effects of the silicone resin content and sintering temperature on the properties of fused silica ceramic cores were researched. Results showed that the silicone resin powders had the good cohesiveness, which was beneficial for directly shaping the silica ceramic cores. The organic to inorganic transformation of silicone resin occurred with the increase of temperature, forming the amorphous SiO_2 at the temperature less than 1250 °C and the cristobalite by the crystallization process of amorphous SiO_2 at the temperature more than 1250 °C. Based on TG analysis, it was found that the ceramic yield (SiO_2) of silicone resin had 83.55wt%. In green bodies, the silicone resin was mainly coated on the surface of fused silica particles via the function of pressing force. After the pyrolysis process was completed, the silicone resin formed some small amorphous silica particles, which was coated on the surface of raw fused silica and filled into the gaps between fused silica particles. With the increase of silicone resin content, the apparent porosity gradually increased, and bending strength showed a decreasing trend. The increasing temperature from 1200 °C to 1300 °C promoted the densification of silica ceramic cores when the silicone resin content was the same. However, the highest bending strength was obtained at 1250 °C. Further increasing temperature was detrimental to the bending strength of silica ceramic cores owing to the microcracks caused by the surface crystallization of fused silica to cristobalite.

1. Introduction

Silica ceramic cores have been widely used in the precise casting of hollow gas turbine parts including the superalloy turbine blades and vanes because of their low thermal expansion coefficient ($0.55 \times 10^{-6} K^{-1}$ between 25 °C and 1000 °C), good chemical stability with molten metals and good leachability [1–3]. As the complex internal cooling passages in hollow gas turbine parts are offered by ceramic cores with special shape, the ceramic cores with excellent comprehensive properties including good mechanical properties and dimensional stability are extremely important. In the casting of hollow parts, the ceramic cores will bear the thermal shock from the wax casting, mould shell sintering and the casting of molten metal, which require the ceramic cores have enough bending strength at room and high temperature [4]. The dimensional stability of ceramic cores at low and high temperature is also important as it affects the dimensional

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accuracy of hollow parts. In addition, high shrinkage makes the porosity decrease in the ceramic cores, which is not beneficial for leaching of ceramic cores in the final hollow superalloy parts. Furthermore, high porosity of ceramic cores also decreases their bending strength [5,6]. Therefore, the balance between the bending strength, dimensional stability and porosity needs to be considered.

At present, the commercial ceramic cores are mainly fabricated by injection molding using ceramic powders and thermoplastic binders, where the thermoplastic binders are mainly some kinds of wax such as the paraffin wax and microcrystalline wax [7,8]. As we known, it needs a long time to complete the sintering process of the ceramic cores consisted of ceramic powders and wax. Among, the eliminating of wax as binder in the ceramic cores at low temperature is very slow process, which needs to be controlled precisely in order to obtain the good properties and dimensional stability of the ceramic cores. This method of preparing ceramic cores has the high preparation cost and low production yield. Therefore, new preparation technology is still needed to prepare the ceramic cores with low cost and excellent properties.

The preceramic pyrolysis technology is a simple and novel technology, and it has been widely applied in the preparation of porous ceramic materials because of its excellent advantages such as the processing versatility, good mechanical properties and low preparation temperature [9,10]. In this technology, the used polymer precursors can convert into ceramic phases, which is beneficial for promoting the properties of sintered bodies. For example, porous SiC, SiOC, Al_2O_3-based ceramics have been successfully prepared by this technology, and these porous ceramics both show good properties [10–12]. Meanwhile, the porous ceramic cores with complex shape may be produced by some plastic forming methods. As a result, the technology has been one of hotspots in preparing porous ceramics. It is believed that the preceramic pyrolysis technology has main two merits in preparing ceramic cores [9–11]. Firstly, a large number of gases are released in the sintered bodies during the cross-linking process of precursor at low temperature, which is beneficial for the formation of porous ceramic cores with open pore channels. And the existence of the open channels in the ceramic cores will make them be easily leached out from the hollow superalloy parts. Meanwhile, the escape process of gases from the ceramic cores may inhibit their shrinkage and ensure their dimensional accuracy. Secondary, the ceramic products formed by the pyrolysis of precursor in the ceramic cores may improve their mechanical properties, which will be beneficial for the service stability and quality of hollow parts. Therefore, it is reasonable and feasible to fabricate the silica ceramic cores by the preceramic pyrolysis technology.

Herein, silicone resin with -Si-O-Si- as the main chains is used as the precursor to prepare the silica ceramic cores. Except for these universal merits of the precursor mentioned above, the silicone resin firstly converts into the amorphous silica at the low temperature and gradually forms the cristobalite by the crystallization process at the high temperature [13,14]. Consequently, a high number of silica products may be obtained after heating treatment, which is beneficial for the enhancement of densification and properties of silica ceramic cores. Additionally, as the silica products formed by the pyrolysis of silicone resin are consistent with fused silica as the matrix, the chemical and physical compatibility between the matrix and products may be not considered. In our early researches, the solvent silicone resin was used to prepare the porous ceramics by injection molding [12,14]. However, a high temperature ($> 250\,^{\circ}C$) is needed to cure the silicone resin directly during the preparation of ceramics. Owing to the volatilization of solvent and curing of silicone resin, the pressure must be also applied to avoid the formation of pores and cracks on the surface of the ceramic bodies. Undoubtedly, the cost of preparing the porous ceramics by this method is high and the good properties of the ceramics are instability. Therefore, in order to solve these problems, the silicone resin powders are used to replace the solvent silicone resin. Meanwhile, the silicone resin powders have well cohesiveness so that they can be directly used as the binder to prepare the silica ceramic cores by pressing method. This research will be beneficial for promoting the application of polymer technology in ceramic core fields.

In this paper, porous silica ceramic cores were prepared via the preceramic pyrolysis technology, among which fused silica powders were used as the matrix and the silicone resin powders were used as the precursor. The effects of silicone resin content and sintering temperature on the properties of silica ceramic cores were investigated.

2. Materials and methods

Commercially available fused silica powders with the average diameter of 45 μm (Xuzhou Sensor Quartz Co. Ltd. China) were used as the matrix. The silicone resin powders with a trade mark of MK (Shanghai Bohuai Chemical Co. Ltd. China) were chosen as the precursor. The mixtures of fused silica powders and silicone resin powders at various mass ratios were ball-milled for 24 h in a plastic jar with ZrO_2 balls as the balling medium. The added silicone resin content was 5 wt%, 10 wt % and 15 wt% (relative to the total mass of powders), respectively. After being sieved through a 100-mesh screen, the powders were pressed into the rectangular bars with $5 \times 6 \times 45$ mm under 20 MPa pressure for 3 min using a stainless steel die. Then the rectangular bars were cured at 250 °C for 4 h with a heating rate of 1 °C/min to make the silicone resin form the network molecular structure, heat treated at 600 °C for 2 h with a heating rate of 2 °C/min to pyrolyze the silicone resin, and subsequently further sintered at the targeted temperature (1200, 1250 and 1300 °C for 2 h) with a heating rate of 5 °C/min in a muffle furnace in air atmosphere.

The pyrolysis process or thermal behavior of silicone resin powders (MK) was investigated by TG with a heating rate of 5 °C/min in air atmosphere. The pyrolysis products of the silicone resin powders (MK) and the phase compositions of ceramic cores at different temperatures (1200, 1250 and 1300 °C) were characterized by XRD. The microstructures of fused silica powders and silica ceramic cores under different conditions were scanned by SEM. The bulk-density and the apparent porosity of silica ceramic cores were measured by the Archimedes method in the distilled water. The rectangular bars with $3 \times 4 \times 40$ mm were used for the bending strength test by three point bending method with a span of 30 mm and a cross speed of 0.5 mm/min. The above each value was the average of five samples.

3. Results and discussion

The thermal gravity behavior of the silicone resin powders (MK) with increasing temperature in air atmosphere has been studied in our early research [15]. The silicone resin contains a total weight loss of ~16.45 wt% from room temperature to 900 °C. During this stage, the cross-linking reaction of the silicone resin happens accompanied by the releasing of water caused by the condensation reaction between Si-OH, which contributes to the weight loss. As the silicone resin is a kind of preceramic polymer with -Si-O-Si- as the main chains and $–CH_3$, –OH and –Ph as the side chains, its pyrolysis include the degradation of main chains and oxidation removal of organic groups at side chains in air atmosphere, leading to the formation of a weight loss.

To further analyze the compositions of products formed by the pyrolysis of the silicone resin, the products prepared at different temperatures are characterized by XRD, and the results are shown in Fig. 1. It is obtained that when the temperature is lower than 1250 °C, the obtained products show the amorphous peaks in the XRD pattern. With the further increase of temperature, the crystal peak appears in the XRD pattern, meaning that the increasing temperature promotes the transformation of amorphous to crystallized phases. It is confirmed that the amorphous SiO_2 forms at low temperature and the cristobalite gradually forms by the crystallization of amorphous silica at high temperature. The crystallization law of the products formed by the silicone resin was consistent with that of raw fused silica.

Herein, the used silicone resin is a kind of organic polymer, among

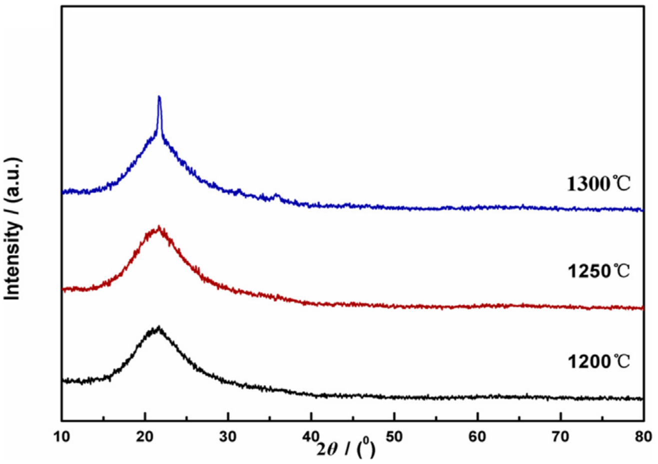


Fig. 1. XRD patterns of pyrolysis products heated at different temperatures.

which the Si-O-Si- as the main chains shows the inorganic structures and –CH_3, –OH and –Ph as the side chains shows the organic structures. As a result, the materials not only have the characteristics of inorganic compound but also organic polymer. When the silicone resin powders are heated in air atmosphere, they cross-linking reaction happens quickly at about 250 °C, leading to the formation of polymer with big network structures. With the further increase of temperature, the pyrolysis process of silicone resin occurs at about 600 °C. Owing to the existence of oxygen in the pyrolysis process in air atmosphere, oxygen will participate into the reaction of the degradation of main chains and removal of organic groups at side chains, where –Si-O-Si- transforms into the amorphous silica ceramic by reacting with oxygen or self-degradation and –CH_3, –OH and –Ph as the side chains produces CO, CO_2, H_2O, H_2 et al. by the thermal oxidation degradation. These released gases escape from the sintered sample, only leaving amorphous silica ceramic phase with inorganic structure in sintered samples. With further increase of sintering temperature, the amorphous silica starts to crystallize and new cristobalites phase gradually forms, as shown in Fig. 1.

Additionally, in our research process, it is found that the used silicone resin powders have enough cohesiveness property to make the fused silica powders shape under relative low pressure. Among, the silicone resin powders are mainly coated on the surface of fused silica particles, which plays a connection role between silica particles. During the cross-linking process at 250 °C, the silicone resin forms the network structure by the polymerization reaction, making the strength of ceramic core increase by the connecting force. During the pyrolysis process, the decomposition of the silicone resin happens and the big network structure is destroyed by the degradation of main chains and oxidation removal of organic groups at side chains [12,14]. Some amorphous silica products formed by the main chains continue to adhere on the surface of fused SiO_2 particles, and other are located in gaps between the fused SiO_2 particles. Meanwhile, a large number of pores in the ceramic cores also form. During the subsequent sintering at high temperature, the amorphous SiO_2 formed by the silicone resin and fused SiO_2 as the matrix both experience the crystallization process. Finally, the silica ceramic cores are prepared by this technology.

Fig. 2(a) shows the XRD patterns of silica ceramic cores with 5wt% silicone resin at different temperature. It is seen that the core mainly shows the amorphous SiO_2 peak when the sintering temperature is 1200 °C. As the increase of the temperature from 1250 °C to 1300 °C, the cristobalite peaks gradually appear and become sharp, meaning more and more cristobalite phases form. By the Rietveld analysis of the XRD peaks [16], it is obtained that the degree of crystallinity is about 10.5wt % at 1200 °C. When the temperature is 1250 °C and 1300 °C, the degree of crystallinity reaches 27.6% and 54.9%, respectively. These results show that the increasing sintering temperature can promote the crystallization of fused silica to cristabalite by offering enough activation energy. Fig. 2(b) shows the XRD patterns of silica ceramic cores with different silicone resin contents at 1250 °C. It is seen that the XRD peaks of cores have no evident difference with the increase of silicone resin contents. It is calculated that the degree of crystallinity is 27.6%, 28.8% and 30.1%, respectively. Based on Fig. 1, as the silicone resin mainly shows the amorphous state at 1250 °C, it is believed that the slight increase of the degree of crystallinity with silicone resin contents is mainly caused by silica particles with smaller size formed by the pyrolysis of silicone resin.

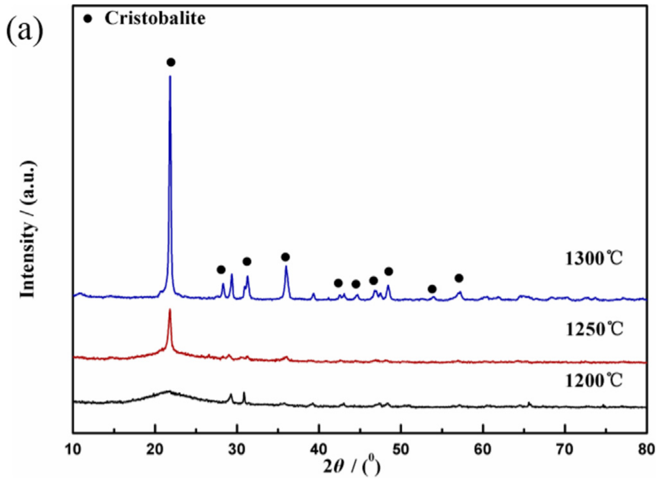


Fig. 2. XRD patterns of silica ceramic cores (a) with 5wt% silicone resin content at different temperatures; (b) with different silicone resin content prepared at 1250 °C.

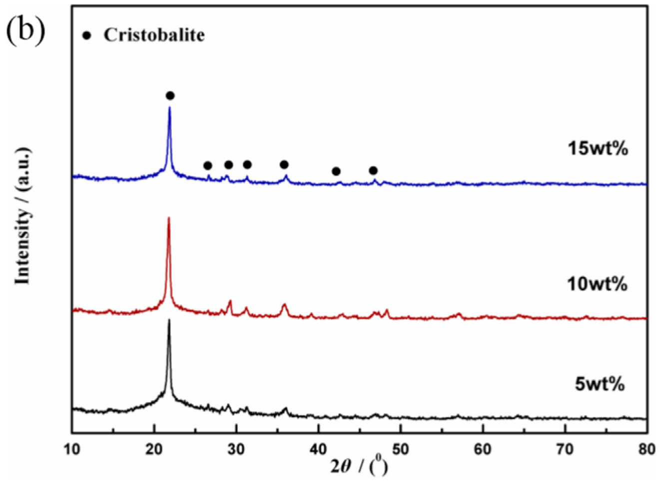


Fig. 2. (*continued*)

Fig. 3(a) shows the weight loss of silica ceramic cores prepared by the preceramic pyrolysis technology. It is obvious seen that the weight loss of the silica ceramic cores gradually increases with the increasing silicone resin content at the same temperature. The increasing temperature has no evident effect on the weight loss of the samples. It is obtained that the silicone resin exists a weight loss process, mainly contributing to the weight loss of the whole cores. Consequently, with the increase of the silicone resin content in the silica ceramic cores, the weight loss of the cores increases. Fig. 3 (b) shows the shrinkage rate of the silica ceramic cores prepared by the preceramic pyrolysis technology. There exists a decreasing trend in the shrinkage rate with the increasing silicone resin content at the same temperature. The increasing sintering temperature promotes the shrinkage rate of the cores.

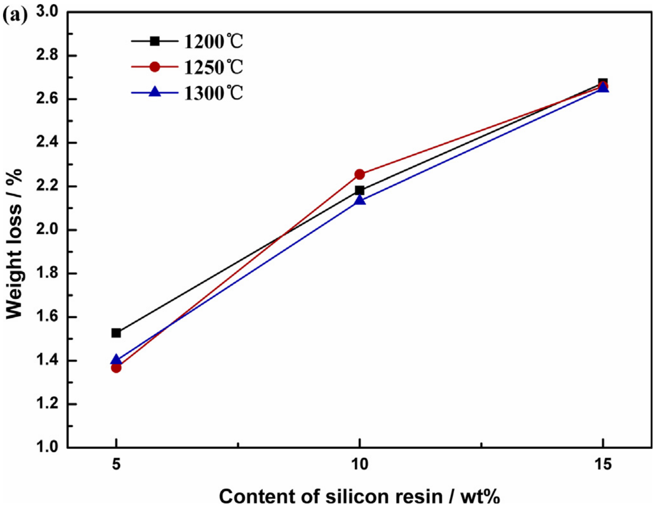
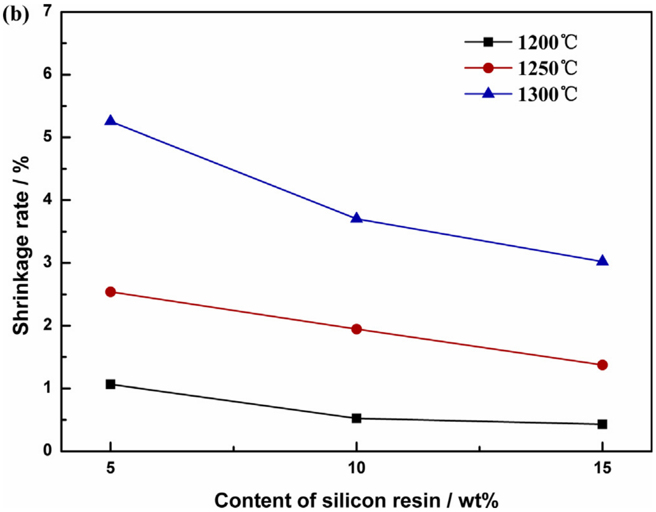


Fig. 3. (a) Weight loss and (b) shrinkage rate of silica ceramic cores prepared by the preceramic pyrolysis technology.



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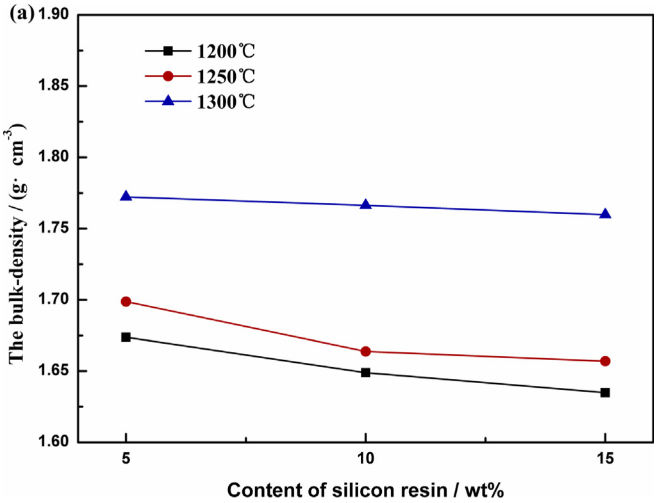


Fig. 4. (a) Bulk-density and (b) apparent porosity of silica ceramic cores prepared by the preceramic pyrolysis technology.

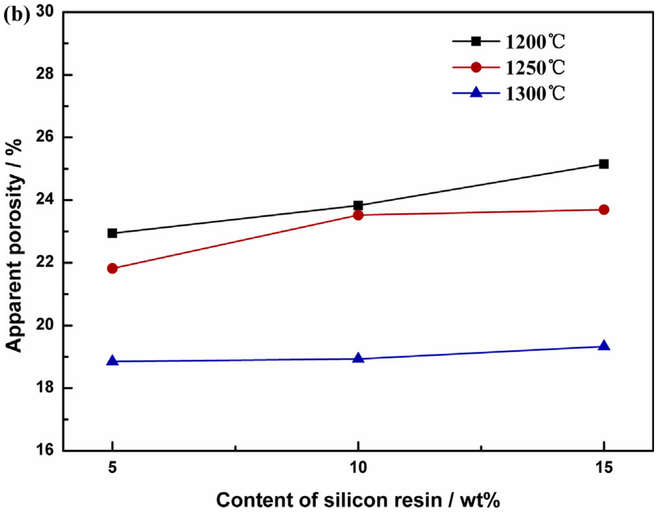


Fig. 4. (*continued*)

The added silicone resin precursor experiences the cross-linking reaction to form the network structure at about 200–300 °C, during which the slight expansion occurs in the ceramic cores. The increase of silicone resin in the ceramic cores easily causes the increase of the expansion. In the sintering process at the temperature more than 1000 °C, the sintering necks between silica particles form gradually to promote the densification by viscous fluid, leading to the shrinkage of the silica cores. It is seen that the shrinkage rate of the cores are positive, meaning that shrinkage behavior caused by densification is bigger than the expansion behavior caused by the cross-linking. Therefore, at the same temperature, the increasing silicone resin content causes the slight increase of expansion, consequently leading to the decrease of the shrinkage rate in the ceramic cores. When silicone resin added is constant, the increasing sintering temperature promotes the formation of a large number of sintering necks by increasing the viscous fluid, making the densification of the ceramic cores [3,17]. If so, the shrinkage rate of ceramic cores gradually increases with the sintering temperature. Herein, the formation of sintering necks includes two aspects. One is from fine amorphous silica particles formed by the pyrolysis of silicone resin between big fused silica particles. The other is from cristobalite phases formed by the crystallization process between adjacent fused silica particles.

Bulk-density and apparent porosity of silica ceramic cores prepared by the preceramic pyrolysis technology are shown in Fig. 4(a) and (b). It is obtained that when the silicone resin content added is constant, the increase of sintering temperature leads to the decrease of open porosity and increase of bulk-density due to the process of sintering densification [12]. As analyzed above, the sintering of fused silica ceramic cores is promoted by viscous fluid [3,17]. The increasing temperature promotes the viscous fluid by decreasing the hindrance and resistance force. As a result, the distance between silica particles gradually decreases, leading to the increase of the densification of the ceramic cores gradually. When the sintering temperature is constant, the increase of silicone resin content results in the slight increase of open porosity and decrease of bulk-density due to the increase of weight loss and decrease of shrinkage rate.

Fig. 5 shows the bending strength of silica ceramic cores prepared by the preceramic pyrolysis technology. It is found that the ceramic cores with different silicone resin content have the highest bending strength after sintering at 1250 °C. Meanwhile, the bending strength shows a decreasing trend with the increase of silicone resin content. Under the whole conditions, the highest bending strength (31.15 ± 2.89Mpa) is obtained in the ceramic cores with 5wt% silicone content prepared at 1250 °C. As we known, in fused silica ceramic cores, the cristobalite content has an important role in the bending strength [8,18]. It is obtained from Fig. 2(a) that the cristobalite content gradually increases with the sintering temperatures in the ceramic

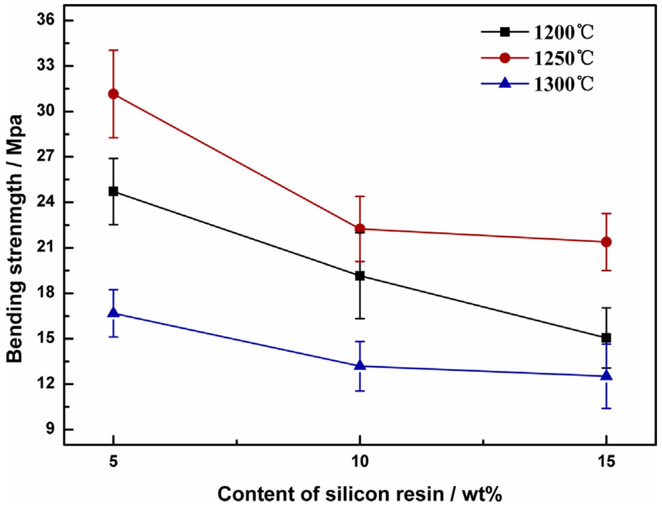


Fig. 5. Bending strength of silica ceramic cores prepared by the preceramic pyrolysis technology.

cores. In this system of fused silica ceramic cores, the fused silica undergoes the crystallization process during the heating process, leading to the formation of the β-cristobalite. The existence of cristobalite as the crystallized phases can promote the improvement of bending strength of fused silica ceramic cores. The negative side is that during the cooling, the phase transformation of β to α-cristobalite accompanies with the volume contraction with 2.8%, leading to the formation of microcracks in ceramic cores. These microcracks on the surface of fused silica particles may cause the evident decrease of bending strength at room temperature [18,19]. In our research, fused silica particles with amorphous state are used as the starting materials and the silica formed by the pyrolysis of silicone resin also shows the amorphous state at the temperature below 1250 °C. In generally, fused silica exists in the form of stable α-quartz at room temperature. When the temperature is about 573 °C, the α-quartz will transform into the β-quartz at a fast speed. And the β-quartz will form β-tridymite at 870 °C. With the further increase of the temperature, the β-cristobalite will form by phase transformation process. The β-cristobalite with crystal structure can improve the mechanical properties of ceramic cores. However, during cooling process, at about 180 °C–270 °C, the β-cristobalite can transform into α-cristobalite. Unfortunately, the transformation process causes the volume contraction with 2.8%, causing the microcracks on the surface of grains. It should be noted that the amorphous silica grains by the pyrolysis of silicone resin have smaller size compared with the raw silica grains as the starting materials. Thus, it is believed that during transformation of β to α-cristobalite, the preferential formed α-cristobalite with samller size can be acted as the crystal nucleus, promoting the transformation of β to α-cristobalite. As a result, the increasing silicone resin will lead to the formation of more microcrack in the ceramic cores, leading to the decrease of the strength. It is reported that the bending strength may have the highest value when there exists a suitable number of cristobalite in the fused silica ceramic cores [19–21]. In our study, it is concluded that about 27.6 wt% cristobalite at 1250 °C is beneficial for the improvement of bending strength and an excess of cristobalite decreases the bending strength. The increasing silicone resin contents also make the porosity decrease, which makes a contribution to the decrease of bending strength.

Fig. 6(a) shows the SEM images of fused silica powders. It is seen that the edges of the fused silica powders are clear and smooth and the average particle size is about 35 μm by the statistics of about 200 grains. Fig. 6(b) shows that SEM images of silica ceramic cores with 5 wt% silicone resin prepared at 1200 °C. Many small particles existed between big fused silica particles are identified to be silica by XRD and EDS. Combined with the XRD from Figs. 1(b) and Fig. 2(a), it is concluded that the silica products formed by the pyrolysis of silicone resin and fused silica mainly show the amorphous state, and there contains a few cristobalite which easily falls off the surface of amorphous silica particles in the silica ceramic cores obtained by sintering at 1200 °C. Therefore, it is believed that small silica particles are mainly formed by the pyrolysis of silicone resin.

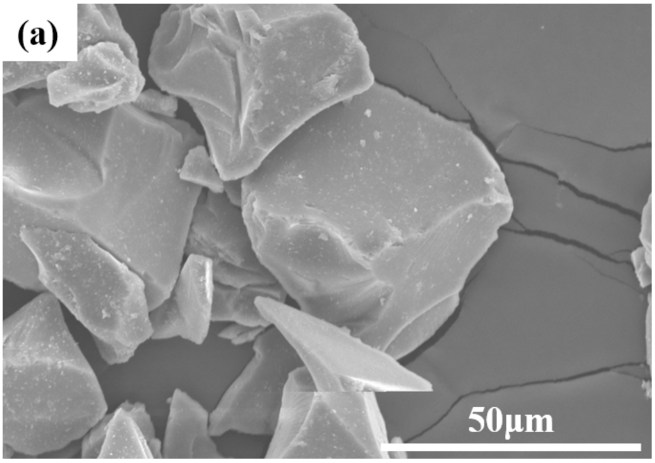


Fig. 6. SEM images of (a) raw silica powders and (b) silica ceramic core with 5wt% silicone resin prepared at 1200 °C.

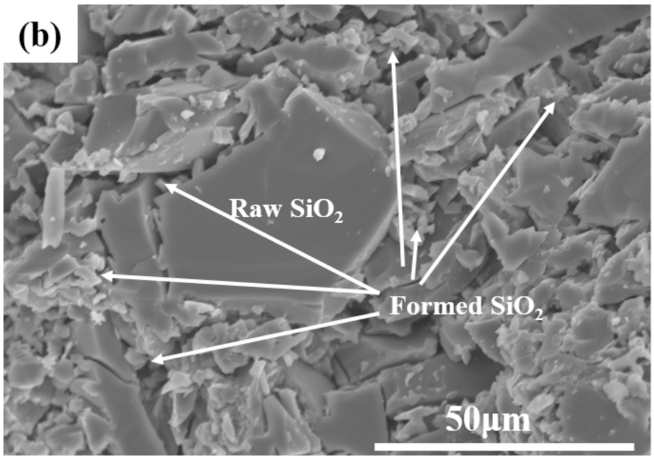


Fig. 6. (*continued*)

Fig. 7(a) and (b) shows the fracture microstructures of silica ceramic cores with 5 wt% silicone resin at 1250 °C and 1300 °C, respectively. Similarly, it is seen that the microstructure of silica ceramic cores includes the big particles as the framework and small particles filled in the gaps between the big particles. Herein, small particles mainly consist of silica by the pyrolysis of silicone resin and cristobalite falling off from the surface of amorphous silica particles. Furthermore, it is obtained that the fracture behavior consists of the transcrystalline (existed flat and smooth plane) and intergranular fracture (relatively complete particles) in all the cores. Fig. 7(c) shows the magnified SEM image of silica ceramic cores with 5 wt% silicone resin at 1300 °C. As pointed by the double white lines and arrows, there exists many concave on the surface of silica particles, which is caused by shedding of cristobalite. When amorphous silica powders are used to fabricate porous silica ceramics, the cristobalite crystallization occurs at high temperature and its phase transformation of β to α also occurs during the cooling at the temperature of 200–300 °C.The phase transformation could produce microcracks by the volume contraction, leading to the shedding of some cristobalite from the surface of silica particles [17,18]. Meanwhile, a large number of formed microcracks lead to the decrease of bending strength. Fig. 7(d) and (e) shows the SEM images of

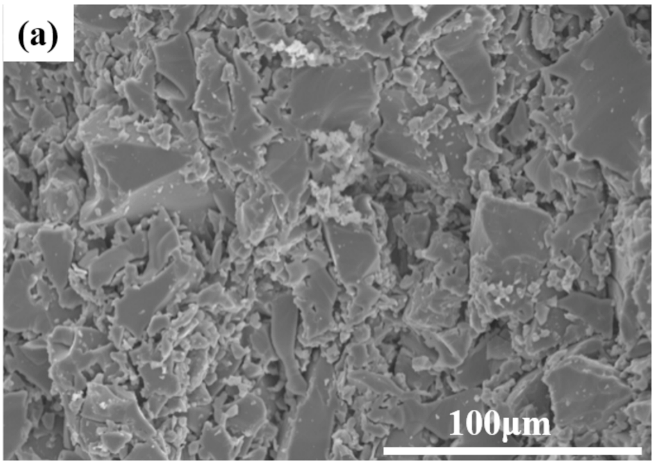
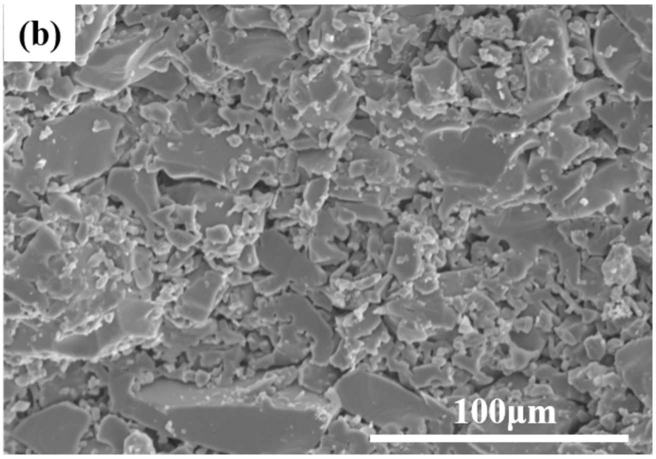


Fig. 7. SEM images of silica ceramic cores. (a) with 5 wt% silicone resin at 1250 °C; (b) with 5 wt% silicone resin at 1300 °C; (c) magnified image with 5 wt % silicone resin at 1300 °C; (d) with 10wt% silicone resin at 1250 °C; (e) with 15wt% silicone resin at 1250 °C.



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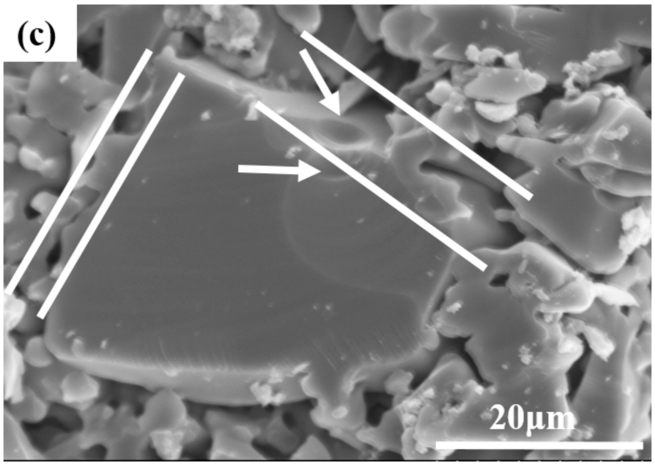

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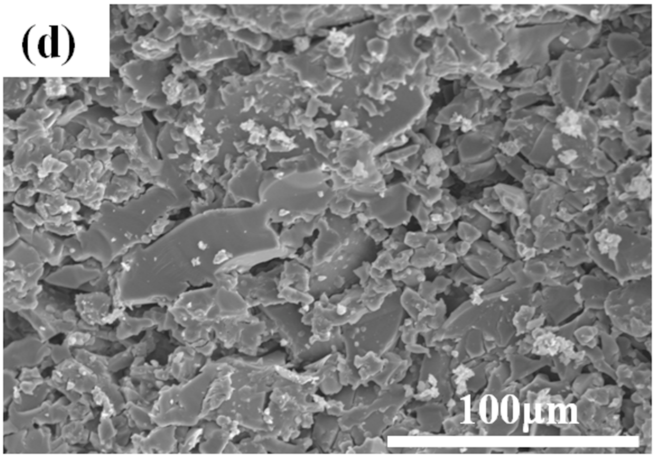

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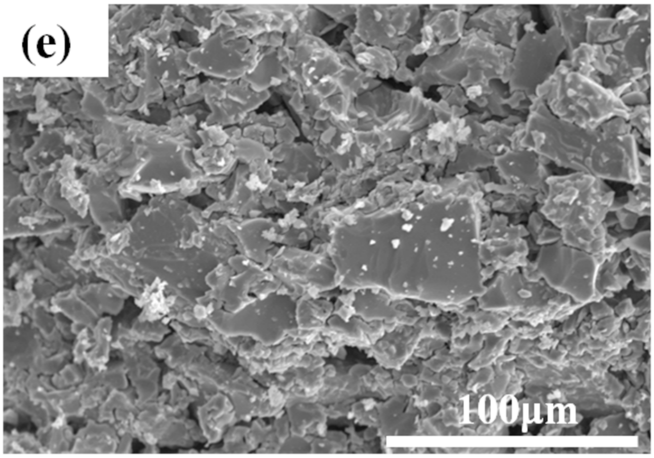

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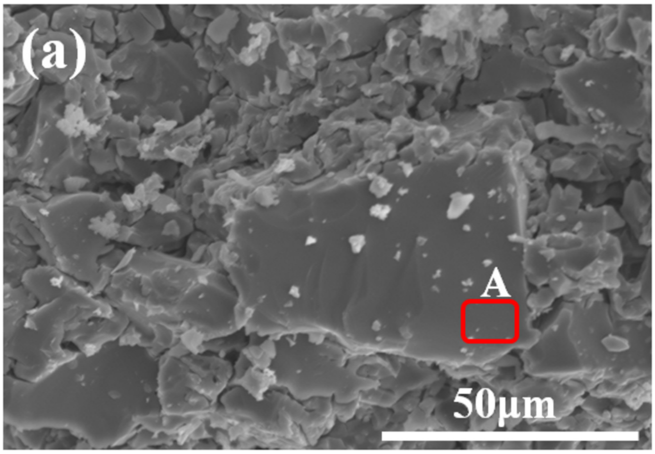

Fig. 8. SEM images (a) with EDS (b) of silica ceramic cores with 15 wt% silicone resin prepared at 1250 °C for 2 h.

silica ceramic cores with 10 wt% and 15 wt% at 1250 °C, respectively. By comparing with SEM images from Fig. 7(a), it is seen that the number and size of pores gradually increases with the increase of the silicone resin content. Meanwhile, the number of small particles adhered on the surface of big silica particles and located in the gaps between the big fused silica particles also gradually increases. More the silicone resin content is, more the number of small silica particles formed by the pyrolysis process is. The increasing silicone resin also releases more gases, leading to the increase of the number of pores in microstructure of ceramic cores. Fig. 8 shows the SEM images with EDS of silica ceramic cores prepared with 15wt% silicone resin prepared at 1250 °C for 2 h. It is evidently seen that there mainly exists Si and O elements on the surface of silica particles. The percentage of Si and O elements is 54.42% and 45.58%, respectively. Because the products

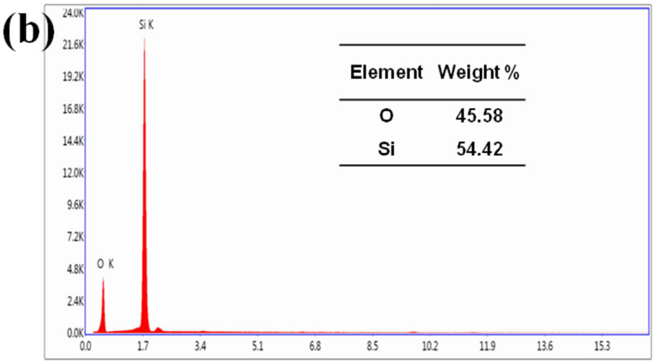

Fig. 8. (*continued*)

obtained by the pyrolysis of silicone resin with –Si-O-Si- as main chains are silica, the Si and O elements are mainly distributed on the surface of raw silica particles.

4. Conclusions

Fused silica ceramic cores were successfully prepared via the pre-ceramic pyrolysis technology in air atmosphere. It was found that the silicone resin powders had well cohesiveness to shape the fused silica particles. With the increase of sintering temperature, the silicone resin with -Si-O-Si- as the main chains experienced the cross-linking process to form the network structure at 250 °C. At about 600 °C, the pyrolysis of silicone resin happened in air atmosphere, leading to the formation of the amorphous silica in the ceramic cores. The silicone resin had a ceramic yield of 83.55 wt% by TG analysis. The increasing temperatures promoted the crystallization of amorphous silica to cristobalite. With the increase of sintering temperature from 1200 °C to 1300 °C, the porosity gradually decreased and the bulk-density gradually increased by the densification process. However, the highest bending strength was obtained at 1250 °C. At 1300 °C, the microcracks formed by the phase transformation of β to α-cristobalite decreased the bending strength. With the increase of silicone resin content, the apparent porosity gradually increased owing to the increase of weight loss, consequently resulting in the decrease of bending strength.

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