

Preparation of Silicon Carbide porous Ceramics

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Abstract. Preparation of Silicon Carbide (SiC) porous ceramics with different sizes of SiC powders as raw material. The effects of reaction temperature and particle size on porosity, pore size distribution and flexural strength of SiC porous ceramics were studied. The results show that: Using SiC labeled F600 as raw material, when the sintering temperature is 2200 °C, the SiC porous ceramics have the best properties, the average pore size is 2.48 μm, the pore size distribution is uniform, the porosity can reach 53.75 % and the flexural strength is 21.23 Mpa. The maximum pore size distribution of SiC porous ceramics is proportional to the particle size of SiC, and the average pore size is linearly related to the median diameter of the raw material.

1. Introduction

SiC materials have high strength, high temperature resistance, corrosion resistance and low coefficient of thermal expansion. SiC porous ceramics inherit the advantages of SiC, so they have a wide application prospect. In recent years, the production technology of SiC is more mature, its synthesis method is diversified, the price is low, and the quality of SiC raw material is getting higher and higher. In summary, SiC porous ceramics will have a broad market prospect. At present, Denmark, France and the United States have applied SiC porous ceramics to industrial production. However, although there are many studies on the preparation of porous ceramics and SiC ceramics in China [1-5], there are still some problems in the production of SiC porous ceramics with excellent performance.

In order to solve some problems existing in SiC porous ceramics, this paper presents the preparation of silicon carbide porous ceramics. According to the principle of the closest packing of single grain size particles, the ceramic green body of SiC is prepared by the ceramic extrusion molding method. The SiC porous ceramics were prepared by high temperature sintering, and the process of preparing the SiC porous ceramics with excellent performance is investigated. It provides a basis for the practical production of SiC porous ceramics.

2. Experimental

2.1. Preparation of SiC porous Ceramics

The preparation process of SiC porous ceramics is as follows:

(a) SiC powders were weighed according to certain proportion, carbon black(C) and solvent were placed in ceramic ball mill tank, alumina balls were added and mixed at 100rpm speed for 10 h, then silicon (Si) powders were added in proportion for 4 h to obtain mixed slurry.



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(b) The slurry was placed in a blast dryer and dried for 12 h at 100 degrees. The alumina balls were separated from the mixture and SiC-Si-C mixture was obtained.

(c) The mixture was mixed with HPMC and mixture in a certain proportion for 1h in a ceramic mixer, and then a certain amount of glycerol, oleic acid, PEG and water were mixed for 0.5 h. The mixture was decayed for 24 hours after three times of vacuum mud scouring. The green bodies were prepared by vacuum extrusion.

(d) After drying the green bodies in a 30 oC blast dryer for 12 h, heating up to 100 oC for 4 h and then drying at 120 oC for 2 h, the billet can be put into a tube furnace for degumming in nitrogen atmosphere.

(e) The SiC porous ceramics were obtained by sintering the samples in a SiC sintering furnace at a certain temperature.

2.2. Sample Characterization

Determination of porosity of material based on Archimedes principle, the pore size and distribution of ceramics were measured by foaming point method, and the flexural strength of porous ceramics was measured according to <GB/T 1965-1996 method of bending strength test>.

3. Results and Discussion

3.1. Effect of sintering temperature on properties of SiC porous ceramics

SiC porous ceramics were sintered at different temperatures using SiC labeled F600 (D50=13.81 μm) as raw material. The effect of sintering temperature on the properties of SiC porous ceramics was investigated.

3.1.1. Effect on Porosity. Figure 1 is a diagram of the relationship between porosity and sintering temperature. As can be seen from the figure, as the temperature increases, the ceramic porosity becomes progressively larger. From 2050 oC to 2250 oC, the porosity of SiC ceramics increased from 52.70 % to 54.30 %. It is concluded that when the sintering temperature increases, the small size SiC formed by the reaction of silicon and carbon in the raw material evaporates and condenses, and the porosity of SiC ceramics increases slightly due to the escape of some SiC atmosphere through the pores.

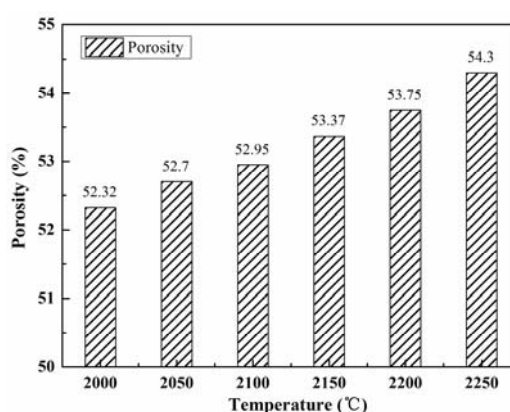


Figure 1. Effect of sintering temperature on porosity of SiC porous ceramics

3.1.2. Effect on Pore Size. Pore size and distribution are the most important indexes of porous ceramics. Figure 2 shows the pore size distribution of ceramics at different sintering temperatures. It can be seen from the figure that, when the temperature is between 2000 oC and 2150 oC, the average pore size of the ceramics has no obvious change, which is about 2 μm , and the pore size distribution of the ceramic is broadened in the direction of the large aperture, and the maximum aperture is increased

from 5.78 μm to 8.47 μm . The reason is that there is no reaction in the process of early sintering, so the average pore size of SiC does not change, while the small size SiC produced by the reaction is recrystallized and deposited on the surface of large particles by evaporation-condensation process. Leading to an increase in the maximum aperture. When the temperature reaches 2200 oC, the average pore size increases from 2.08 μm to 2.48 μm , and the maximum pore size reaches 9.25 μm , which indicates that the recrystallization reaction occurs at this time, and the average pore size increases when the temperature is 2250 oC. The average pore size changed to 2.48 μm , and the maximum pore size increased to 11.8 μm .

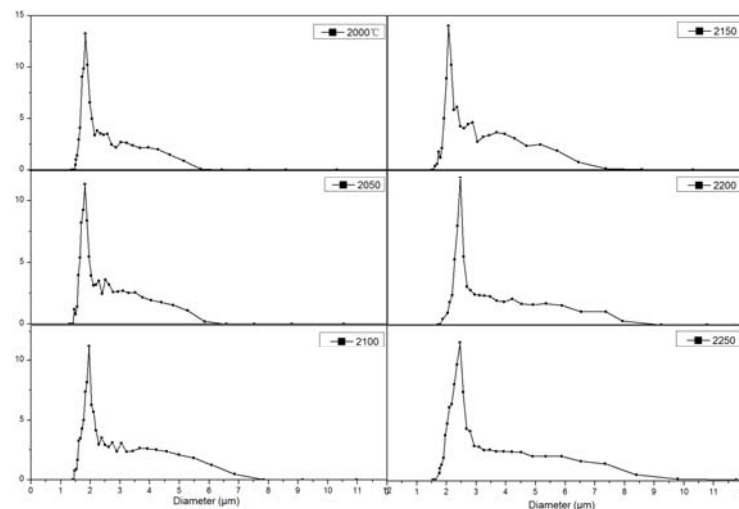


Figure 2. Effect of sintering temperature on pore size of SiC porous ceramics

3.1.3. Effect on flexural strength. Figure 3 shows the flexural strength of SiC porous ceramics prepared at different temperatures. It can be seen from the diagram that at 2200 oC, the highest flexural strength is 21.23 MPa. When the temperature rises to 2250 oC, the strength of ceramics decreases to 17.25 MPa. When sintering temperature increases, the development of sintering neck is more complete, and the flexural strength is also higher. When the temperature increases continuously, the vapor pressure in SiC atmosphere becomes larger, the gas escapes from the pores, and the porosity increases, which leads to the decrease of strength. At 2000 oC-2150 oC, the flexural strength did not change obviously, which indicated that in this temperature range, there was no reaction of SiC with large particle size, and the small particle SiC formed by reaction of C with Si powder participated in recrystallization reaction, which provided strength for ceramics.

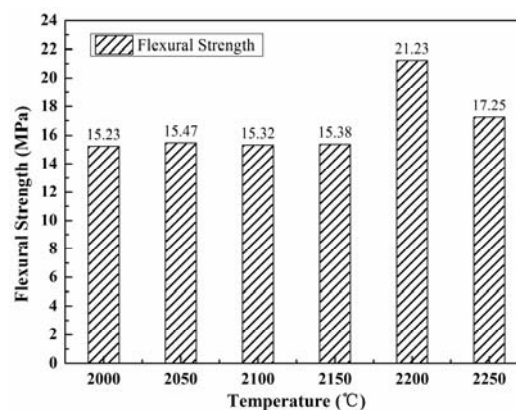


Figure 3. Effect of sintering temperature on flexural strength of SiC porous ceramics

3.2. Effect of raw material particle sizes on properties of SiC porous Ceramics

Nine kinds of SiC with different particle sizes were used as raw materials, labeled F1500(D50=3.42 μm), F1200(D50=4.79 μm), F1000(D50=6.06 μm), F800(D50=8.03 μm), F600(D50=13.81 μm), F400(D50=24.01 μm), F360(D50=25.98 μm), F320(D50=30.14 μm) and F280(D50=42.62 μm). The SiC porous ceramics were prepared by extrusion molding and sintering at 2200 oC for 1 h. The effect of particle sizes on the properties of SiC porous ceramics was investigated.

3.2.1. Effect on flexural strength. In order to study the effect of SiC particle sizes on ceramic properties, the pore formed between the larger size raw material particles is mainly analyzed, so the smaller particle size (F1500, F1200, F1000) should be removed during the discussion. Figure 4 is a fitting diagram of the relationship between SiC particle size and pore size after removing these unreliable points. It can be seen from the figure that when the particle size of SiC is in a certain range, the particle size is linearly related to the average pore size. The relationship between the particle size of SiC raw material and the average pore size of ceramics is as follows:

$$Y = 0.15538X + 3.42051 \quad (1)$$

Figure 5 shows the pore size distribution of SiC ceramics corresponding to different sizes of raw materials. It can be seen from the diagram that a small number of large pores are distributed in SiC porous ceramics. The formation of such pores is related to the particle size distribution of SiC raw materials, and the narrower the size distribution, the narrower the pore size distribution.

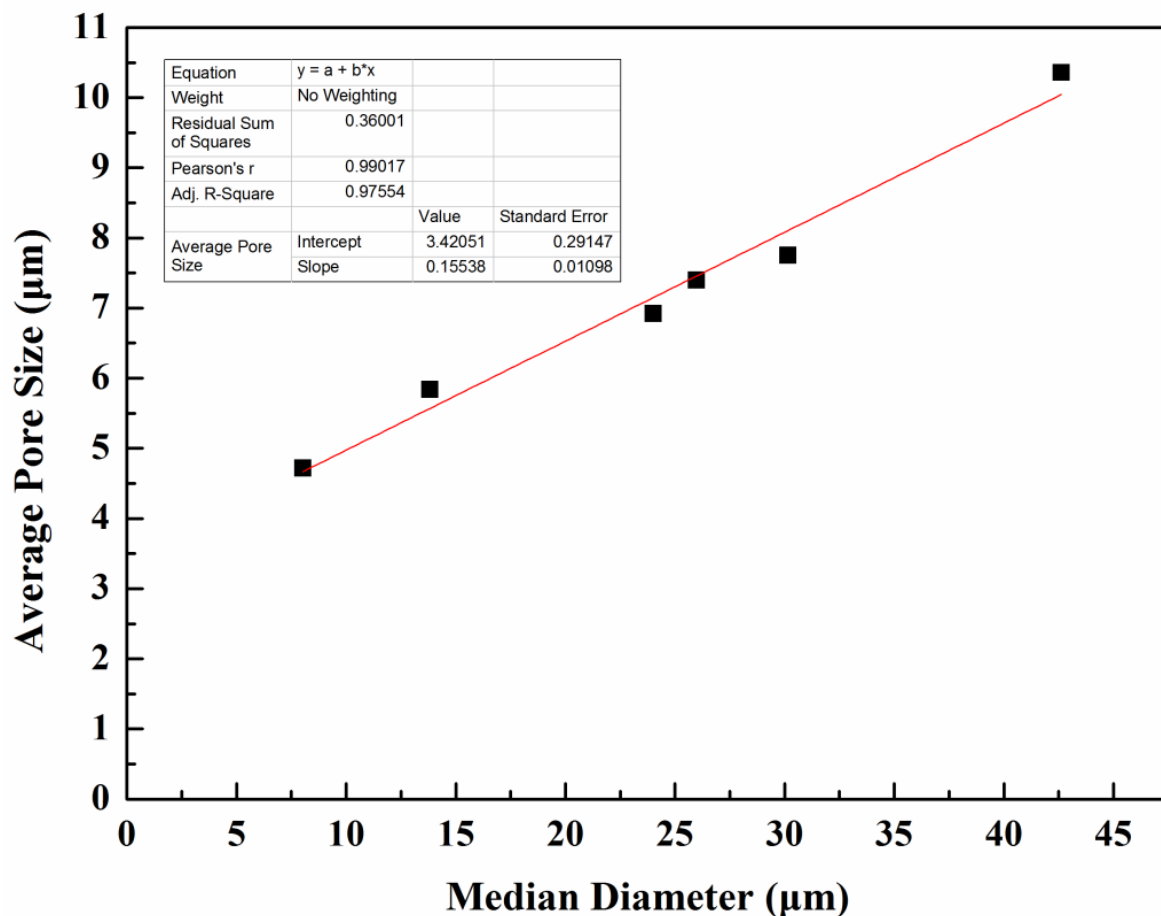


Figure 4. Fitting diagram of SiC particle size and ceramic average pore size

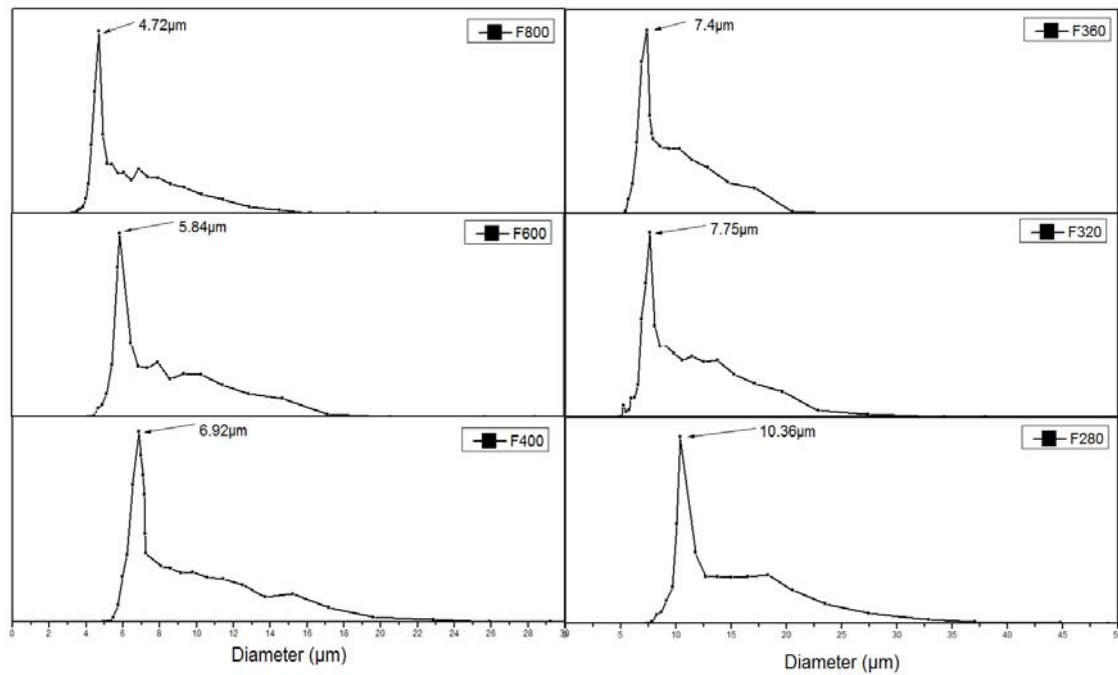


Figure 5. Distribution of pore size of SiC with different particle size

3.2.2. Effect on flexural strength and porosity. Figure 6 shows the effect of SiC particle size on flexural strength and porosity of SiC porous ceramics. As can be seen from the figure, when the particle size of the raw material increases from 13.81 μm to 42.62 μm , the ceramic strength decreases from 21.5 MPa to 10.3 MPa, whereas the porosity of the ceramic does not change significantly. When the particle size of raw material is lower than 13.81 μm , the change of strength corresponds to the change of porosity, the porosity increases and the strength decreases. It shows that when the particle size of raw material is larger than a certain value, the main factor affecting the strength of ceramics is the particle size of SiC. The sintering activity is low, the surface energy is small, and the reaction temperature is high. When the particle size is less than this value, it is the porosity that affects the strength of ceramics.

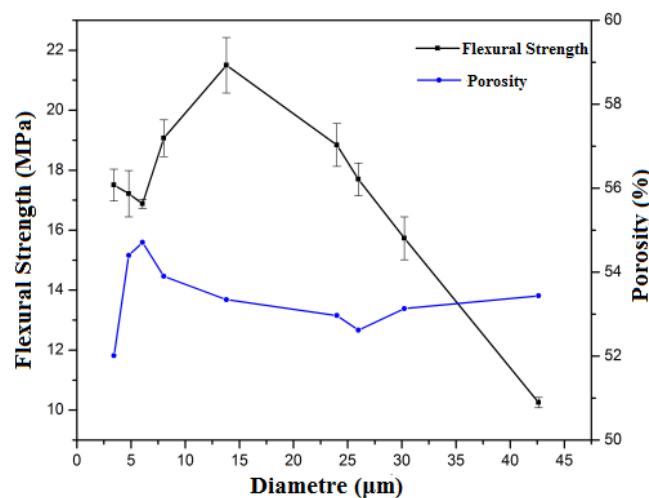


Figure 6. Effect of SiC particle size on flexural strength and porosity

4. Conclusion

(1) Using SiC labeled F600 as raw material, when the sintering temperature is 2200 °C, the SiC porous ceramics have the best properties, the average pore size is 2.48 μm, the pore size distribution is uniform, the porosity can reach 53.88% and the flexural strength is 21.14 MPa.

(2) The maximum pore size distribution of SiC porous ceramics is proportional to the particle size of SiC, and the average pore size is linearly related to the median diameter of the raw material.

Acknowledgments

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