Study on the Reactive Sintering SiC Materials

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ABSTRACT: SiC ceramic is a new ceramic material. For its excellent performance, it is widely used in the field of mechanical seal. It is used in the pump, blender and other equipment during the petrochemical, aerospace and other industries. In order to further improve the sealing properties of SiC ceramics, this paper study on the SiC ceramic material preparation of various process parameters influence on its main mechanical properties. During the study, we were investigated in the binder, Si powder size, the effect of carbon density and molding pressure influence on the properties of sintering SiC ceramic material. We find the optimum route finally.
KEYWORD: Sintering SiC ceramic material; Process parameters; Performance

1 BACKGROUND

SiC ceramics is a new ceramic material which developed in recent decades. Due to its special excellent high strength, high hardness, corrosion resistance, high temperature resistance, it has been large-scale development and application. It has been widely used in petroleum, chemical, metallurgy, machinery, aerospace, microelectronics, automotive, steel and other industries of machinery sealing areas. It shows advantages of unmatched by other special ceramics increasingly (Chen Yixin ET AL, 2015). Because the pump, mixer and other equipment working conditions are generally poor, such as high temperature, high pressure, corrosion, wear and so on. There are very high requirements on the performance of sealing materials (Zhang Wei ET AL, 2015). In order to further improve the performance of ceramic sealing material, which is more consistent with the requirements of sealing material. It need to depth study the relationship of preparation process, structure and properties. This paper studies the effect of parameters on the sintering of SiC ceramic material microstructure and mechanical properties. It gets the best preparation process finally.

2 EXPERIMENTAL MATERIALS AND EXPERIMENTAL PROCEDURES

This paper intends to use Sintering SiC ceramic technology. The main raw materials are SiC powder, binder, carbon black, Si particles and so on. The SiC powder is selected particle diameter of 7 m, 28 m and 56 m the three raw powders. Binder selected the polyvinyl alcohol and phenol resin. Si particle is diameters 5 ~ 8mm particles.

The development of the process is shown in Figure 1.

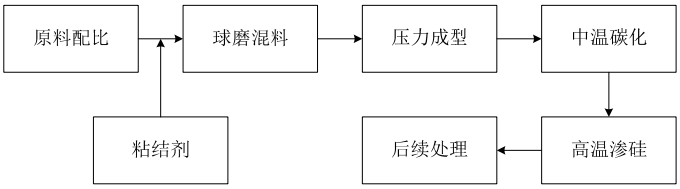


Figure 1 Flow chart of reactive sintering SiC ceramics

We can see from Figure 1, the sintering process of SiC ceramic has the following process:

(1). The ratio of raw materials: select three different particle sizes of SiC powder, using two kinds of polyvinyl alcohol and two kinds of binder phenol-formaldehydes. Design several corresponding biscuit formula, and determine raw materials, solvents, binder, curing agent and lubricant formulation proportion.

(2). Mixing: using the wet mixing process, in accordance with the formula in the proportion of weighing the raw materials, solvents, adhesives, curing agent and lubricant, and using grinding ball mill.

(3). Blank shaping: weigh a certain amount of the mixture with the mold and press molding, low temperature drying in an oven after molding.

(4)Temperature carbonization: SiC biscuit carbonization in the moderate tube oven which protected by argon, carbonized in 800 degree for 2hours. It ensures that the organic matter is fully cracked.

(5). Permeability and the high temperature ceramic Si: SiC prime billet after the middle temperature carbonization furnace for melting infiltration sintering in the vacuum graphite tube. The Si particles are filled in the BN coated surface of the graphite crucible. Ceramic biscuit is placed above in the Si particles, and together with graphite in the crucible and graphite carbon tube furnace for melting infiltration sintering.

(6). Subsequent processing: Diamond millstone will be attached for removal of the sintering sample surface adhesion of Si, and the sample grinding to reduce the surface roughness and improving surface quality.

3 SAMPLE CHARACTERIZATION METHOD

In order to evaluate the mechanical properties of the sample, the samples were measured following methods.

(1). Bending strength

According to the testing method of bending strength of GB/T4741-1999 ceramic materials, flexural strength test of sintering SiC ceramics samples. The principle is shown in Figure 2.

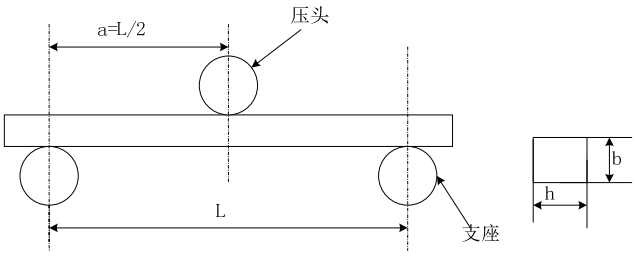


Figure 2 Bending strength test principle diagram

The bending strength calculation formula is as follows:

$$\sigma = \frac{\varepsilon PL}{2bh^2} \quad (1)$$

In this formula: σ--three-point bending strength (MPa)

P--the maximum load of fracture (N)

L--span (mm)

b--specimen width (mm)

h--specimen height (mm)

(2). Rockwell hardness

According to the general hardness of sintering SiC ceramics, selected Rockwell HRA to characterize the reaction sintering of SiC ceramics hardness. Rockwell hardness test can be read directly by Rockwell hardness tester.

(3). The microstructure and morphology

Reaction-bonded SiC ceramics after preparation of metallographic specimens, it need to fine diamond grinding wheel grinding, and then size w2.5 diamond grinding polishing paste. It can use the metallographic microscope and scanning electron microscope (SEM) to observe the microstructure of SiC ceramics.

4 RESULTS AND DISCUSSION

(1). Binder effect on the sintering of SiC ceramic structure and properties

This paper evaluates the effect of binder by SiC the flexural strength of the slab level. According to previous research experience, SiC strength of the biscuit must be bigger than 10 MPa to meet the requirement performance. Through the experiments of fixed binder content, compared with the bending strength of polyvinyl alcohol and two kinds of phenol resin binder preparing biscuit. The results are shown in Table 1.

Table 1 flexural strength of different binder SiC biscuit

Binder type	Content wt%	Molding pressure Mpa	Molding pressure Mpa
Polyvinyl alcohol	12	120	7
Phenol resin	12	120	18

Comparison of the data can be found, the bending strength of phenol resin as a binder for preparing SiC biscuit was better than polyvinyl alcohol. The biscuit strength fully meets the requirements of machining strength.

(2). SiC particle size effect on the reaction sintering SiC ceramic structure and properties

Experiment selected the three particle sizes of SiC powder, W7 (d50=7 m), W28 (d50=28 m) and f280 (d50=56 m). It produced different particle size of reaction sintering SiC ceramic material. Then I analyzed the SiC effect of particle size on the sintering of SiC ceramics and observed its microstructure characteristics. The density and bending strength were tested. The results are shown in Figure 3 and Figure 4.

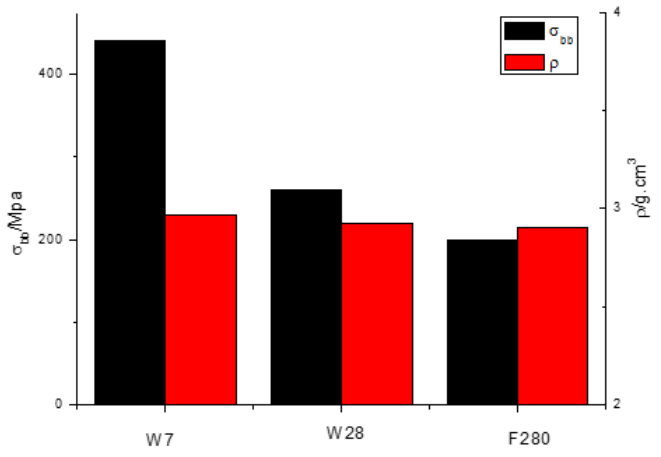
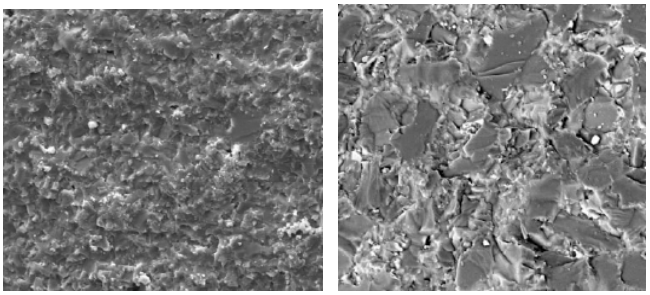


Figure 3 The anti bending strength and density contrast of different particle size of SiC powder reaction sintering of SiC ceramics

From Figure 3, we can see that strength flexural strength of reaction bonded SiC ceramics by SiC powder particle size effects is very obvious. When the powder particle size increases, the flexural strength of the samples decreased significantly. And sample density pollination particle size has little effect.



a. W7 b. W28

Figure 4 SEM fracture photograph of reactive sintering SiC

In Figure 4, the fracture is reactive sintering SiC material's 5000 times magnification pictures. It can be seen from the figure, the reaction sintering of SiC ceramics failure mainly transgranular fracture, and accompanied by a small percentage of intergranular fracture. When the particle size of SiC powder is W7, Free Si SiC particle size is small (Figure 4 in white or gray area), and uniform distribution. Because of the strength of Si is much lower than SiC ceramics. In the figure 4 b, the local segregation of Si resulted in coarse particles sintering of SiC ceramic strength greatly reduced (Zeng Zeng ET AL, 2015).

(3). Effect of carbon density of sintering SiC ceramics structure and properties.

The study found that the carbon content in the biscuit SiC effect of the performance of sintering of SiC ceramic materials. People used to characterize SiC carbon density carbon content in the biscuit. According to the definition of carbon density, it is the form of element C's content in SiC compacts per unit volume. We can find that the residual SiC in the biscuit element C mainly comes from the mixing when adding carbon black and binder dissociation C. The calculation method is shown as the following formula:

$$\rho_c = (\omega_c + \omega_n \chi_c)\rho_p \quad (2)$$

In this formula: ρc is carbon density of SiC biscuit. ωc and ωn respectively the biscuit quality percentage of C and binder. χc is carbon residue cracking rate of carbide binder.

We can adjust the content of black carbon and binder to control the carbon density. Specific formulations are shown in Table 2.

Table 2 Different carbon density SiC biscuit preparation conditions and parameters.

Sample	The content of carbon black wt%	Phenol resin containing wt%	Biscuit density g.cm^{-3}	Carbon densityg.cm^{-3}
A1	13	12	1.706	0.324
A2	18	5	1.745	0.292
A3	8	8	1.723	0.186
A4	0	14	1.782	0.127

As shown in Figure 5 is the different carbon density of SiC compacts prepared by reaction sintering SiC material density and hardness.

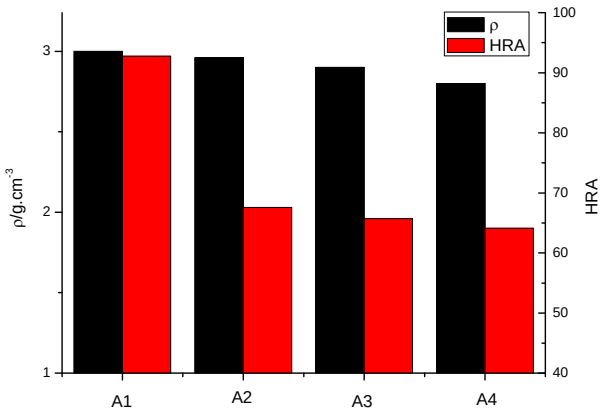


Figure 5 carbon density of biscuit influence on the performance of sintered SiC ceramics

Comprehensive table 2 and figure 5, it can be seen that the reduction of carbon density, reaction sintering SiC material density and hardness decreased. Because the preparation of sintering SiC materials mainly rely on liquid Si to SiC biscuit in the water, and the biscuit C formation of two SiC. The gap was filled material. It obtained the compact of materials. The higher carbon density decided to generate two times the content of SiC in the sintering process. Usually carbon density is small, the sintering materials two times the content of SiC is small, the reac-

tion sintering SiC material density and hardness decreased (Lu Youjun, 2014).

(4). The effect of molding pressure on the reaction sintering SiC ceramic structure and properties

Reaction sintering SiC ceramic material in the sintering process, the biscuit in the permeability is a slow process of liquid phase Si. After the sample was infiltrated density and uniformity of Si and SiC biscuit compacting degree of filling, i.e. with a relative density of SiC biscuit. It means that the relative density of the SiC pressing pressure depends on the size.

Figure 6 and Figure 7 shows the density of compacts with different molding pressures, the changes of bending strength and hardness.

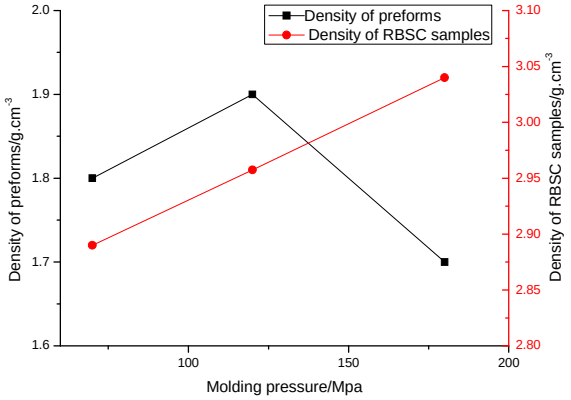


Fig. 6 molding pressure effect on the blank and the density of sintering SiC ceramics

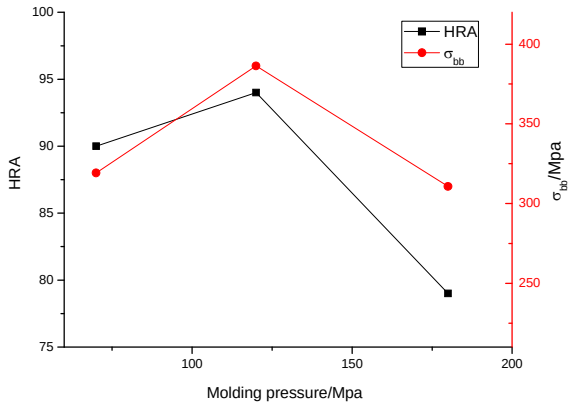


Figure 7 molding pressure influence the bending strength and hardness of sintering SiC ceramics

From Figure 6 and Figure 7 can be seen, molding pressure of 70 MPa biscuit with low density, pore in the biscuit high and Unicom. It can guarantee the biscuit complete penetration, but after infiltration reaction sintering SiC ceramic free residual Si is very high. The mechanical properties of the samples were low. Forming pressure 170Mpa biscuit density is too high, resulting SiC prime billet for Si liquid penetration into the internal pore closed. Si liquid is difficult to penetrate internal biscuit, leave many pores and carbon residue, density and strength. It also influences the reaction sintering SiC ceramic material, hardness decreased significantly.

5 CONCLUSION

In this paper, through the research on the preparation technique of sintering SiC ceramic material, obtained the following conclusions: to use the average particle size of 7um SiC powder, add 12% phenol resin as a binder, add 13% black carbon, and use of 120Mpa pressure molding biscuit has the best mechanical properties after sintering. Study on Preparation Technology for sintering SiC ceramic material not only has a great guiding significance to improve the performance of sintering SiC ceramics, also has certain reference value for the performance optimization of other ceramic materials. The improvement of sintering of SiC ceramics performance to has a significant role in promoting the development of aerospace and petroleum chemical industry.

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