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5. Porous Silicon Nitride for Low Pressure Loss DPF

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Low pressure loss is a fundamental requisite for Diesel Particulate Filter (DPF) material. A conventional method to reduce the pressure loss is to increase the porosity of its base material. However, mechanical strength and heat capacity decrease seriously as the porosity gets closer to 70%. Therefore improvement of gas permeability with minimum porosity increase has become a mandate issue. In this study, samples with the gas permeability over 3.5μm^2 which is twice as much as that of conventional one, with the porosity less than 70%, was obtained by partial substitution of the pore former with smaller particles.

1. Introduction

Reaction-bonded silicon nitride (RBSN) is a candidate material for Diesel Particulate Filter (DPF). As the market demand for low pressure loss material with sufficient durability has been increasing, we set the target values at 65～70% for porosity and over 3μm^2 for gas permeability. Meanwhile, porosity increases as the amount of pore former increases. In our conventional process, pore former with unimodal size distribution has been used to obtain porous RBSN. However, the gas permeability 3μm^2 could not be obtained even at the porosity 70% when we used acrylic resin particles as pore former. **Figure 1** illustrates gas permeability depends not on the diameter of pore former particles but on the orifice sizes at their contact points. Therefore, the gas permeability has not been improved so efficiently as the porosity increases. In this study, we tried to improve the gas permeability without increasing the porosity by substituting a part of pore former with smaller particles. **Figure 2** illustrates the total number of contact points increases by blending different sizes of particles. Thus the gas permeability should be improved with minimum porosity increase.

2. Experimental Procedure

RBSN[(1)-(4)] is fabricated from commercial silicon powder whose average particle size is 40μm and the particle size ranges from 25 to 100μm. In addition

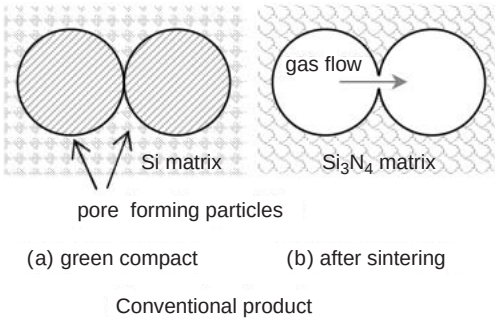

Fig. 1 Contact of same size particles.

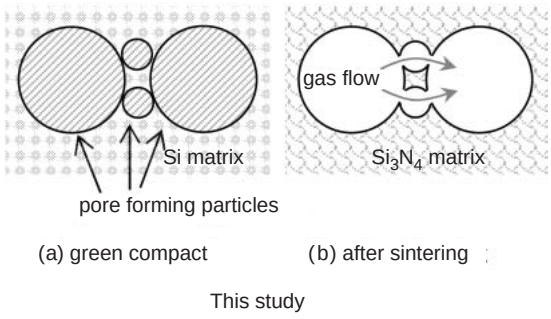

Fig. 2 Contacts of different size particles.

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to pore former, $Mg(OH)_2$ and $CaCO_3$ were added as sintering aid. The amounts of these additives were 2 mass % equivalent of MgO and 1 mass % equivalent of CaO to Si_3N_4, on the assumption of the complete nitridation of silicon. The average diameters of pore formers chosen for this study were 70μm (conventional), 40μm and 20μm. In each case, the amount of pore former was 70 % in volume to the sum of silicon and pore former. The ratios of pore former particles are shown in **Table 1**. In Sample 1 through Sample 3, pore formers with unimodal size distribution have been used, i.e. 70μm for Sample 1 (Reference), 40μm for Sample 2 and 20μm for Sample 3. On the other hand, particles of 70μm and 20μm were mixed in Sample 4 and Sample 5 at the ratio of 70:30 and 85:15 respectively.

Raw powders described above were mixed with methylcellulose and water prior to the intrusion process. The green body has honeycomb structure with approximate dimension of 22mm×22mm×100mm. Theses samples were drained with microwave and dried in an oven at 110℃, then heated to the temperature at 500℃ under nitrogen flow to decompose resin component. Sintering[5)-6)] was carried out under nitrogen atmosphere at the heating rate of 400℃/h up to 1000℃, 120℃/h to 1250℃, 30℃/h to 1350℃ then 120℃/h to 1750℃. The temperature was kept constant for 1 hour both at 1350℃ and 1300℃, for 2 hours at 1350℃ and for 3hours at 1750℃. Pore size distribution and porosity were measured by mercury immersion method. Permeability was evaluated with Perm-Porometer (Porous Material Inc. U.S.A.). Fracture strength was determined by compressing the sample along the extrusion direction.

3. Results and Discussion

Details about sintered bodies are shown in **Table 1**. Pore size distributions are shown in **Fig. 3**. In general, fracture strength decreases as the porosity increases. However, **Table 1** shows fracture strength depends on the structure as well. That is, samples consist of smaller pores have higher fracture strength and lower permeability (Samples 1～3). Meanwhile samples with open pores (samples 4 and 5) have lower fracture strength and higher permeability in comparison to that with closed pores (sample 1). In samples with unimodal size dis-

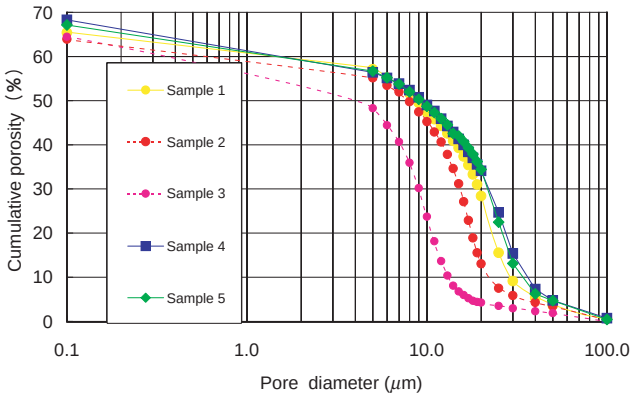

Fig. 3 Pore size distribution

Table 1 Characteristics of Sintered Samples.

	Sample 1	Sample 2	Sample 3	Sample 4	Sample 5
Pore former content	(vol%)				
70μm	70.0			49.0	59.5
40μm		70.0			
20μm			70.0	21.0	10.5
Pore size distribution	(vol%)				
50 < (μm)	5.5	5.4	2.9	7.0	7.0
50−40	2.6	1.3	0.6	3.8	2.3
40−30	5.8	2.4	1.0	11.8	10.3
30−25	9.8	2.6	0.9	13.6	13.9
25−20	19.6	8.7	1.1	13.8	17.8
20−15	16.7	28.4	3.9	10.6	11.3
15−10	12.4	22.0	26.3	11.3	10.0
10−5	14.8	15.5	38.1	10.8	11.9
< 5	12.7	13.7	25.1	17.4	15.5
Porosity (%)	65.4	64.0	64.5	68.3	67.1
Average pore size(μm)	18.3	14.7	8.7	20.0	20.2
Permeability(μm^2)	1.9	1.2	0.6	3.8	3.7
Fracture strength(MPa)	2.7	3.4	6.0	1.8	2.3

tribution pore formers, the average pore size and permeability increase as the size of pore former increases. Meanwhile, fracture strength shows the opposite tendency and porosities are almost constant. In contrast, permeability is improved by twice through blending different size pore formers, nevertheless the average pore size does not increase significantly compared to the reference (Sample 1). Thus we conclude that the remarkable increase of permeability which accompanies no significant increase of porosity is due to the increase of contact points among pore formers through particle blend. In particular, pore distribution shows that pores between 20 and 40μm, which is said to be most effective for soot capture, increase by particle-blend, whereas pores below 20μm decrease.

4. Conclusion

Gas permeability has been improved without significant increase of porosity by partial substitution of the pore former with smaller particles. It is due to the increase of contact points among pore former particles. In this study we used acrylic resin particles as pore former. Permeability and porosity through conventional process used to be 1.9μm^2 and 65.4% respectively. In this study, we have obtained 3.7μm^2 for permeability with the porosity at 67.1 % by blending 15 vol% of 20μm particles with 85vol% of 70μm particles as pore former. This method is applicable to non-resin pore formers such as glass particles and can be extended to the mixture of three or more different size particles.

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