SiC-based Diesel Particulate Filter Developed by New Sintering Process

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This paper explains the characteristics and the performance results of evaluation test on the SiC-based Diesel Particulate Filter (DPF) developed by a new sintering process (Partial reaction sintering in nitrogen atmosphere). The new process enables a control of porosity and pore size distribution of the filter material with comparatively high flexibility, in addition to the cost reduction compared with the normal sintering process. The developed filter indicated sufficient thermal durability and higher filtration efficiency on the initial stage which can respond to the severe regulation demanded in the future by controlling the pore size on the wall surface and the microstructure in the wall.

1. INTRODUCTION

In the present exhaust gas purification filter, SiC-DPF is widely used especially in European passenger car owing to excellent robustness and high filtration efficiency. In recent years, environmental regulation tends to become severe rapidly by introduction of particle number restrictions, and the further highly performance is demanded for DPF.[1)] Especially, higher soot filtration efficiency is required in the initial stage after regeneration. Normally, highest filtration efficiency is produced by the soot accumulation (soot cake) on the filter wall. [2), 3)] A considerable amount of soot leakage before the soot cake formation should be decreased quickly.

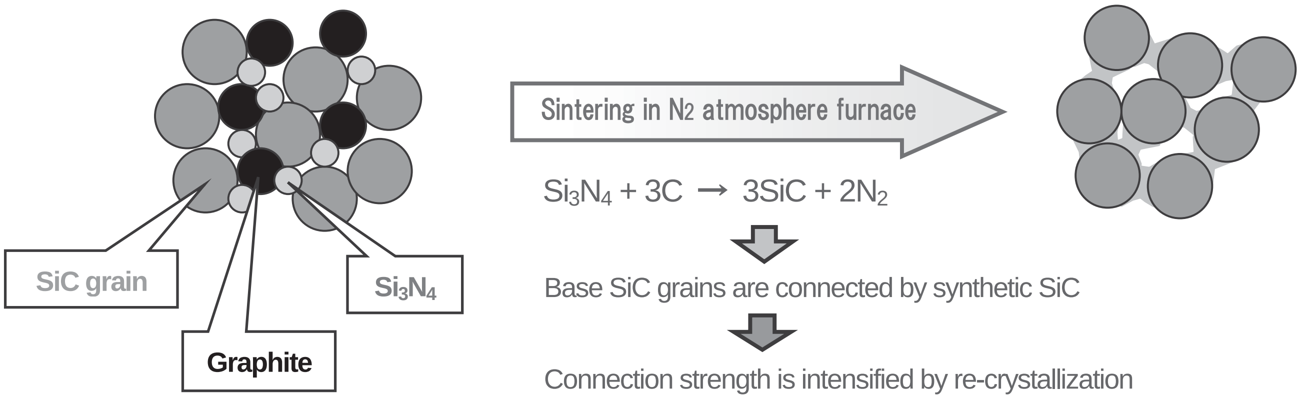
The performance of the SiC-DPF is mainly influenced by the microstructure of filter materials described in terms of the porosity and pore size distribution substantially.[4)] Generally, in order to increase the filtration efficiency, methods of decreasing the pore size in the filter material or increasing the wall thickness are used. On the other hand, we succeeded in development of SiC-DPF having a high soot mass limit (SML) performance and filtration efficiency by introducing a new sintering process (Partial reaction sintering in nitrogen atmosphere) which can control the porosity and the pore size of SiC material comparatively easily.[5)]

Material Development

As materials constituting the filter material in the new process, the SiC powder of high purity with uniform particle size, proper amount of carbon powder and silicon nitride powder as sintering aid material, and the organic binder were used. After the mixing of those mixture with proper water, a normal extrusion was applied to form a honeycomb body. After the drying and plugging, the filter elements were debindered and sintered in the high temperature sintering furnace with nitrogen atmosphere.

On this sintering process, the bonding between SiC grains was accelerated by the next reaction.

$$Si_3N_4 \quad + \quad 3C \quad \rightarrow \quad 3SiC \quad + \quad 2N_2$$



Therefore, since this sintering process is completed in shorter time and lower temperature as compared with the usual high temperature sintering, abnormal grain growth is restricted. Moreover, the control of porosity and pore size is easy, and lead to energy saving by this sintering process. The sintered filter element body shows a characteristic dark green color.

2. EXPERIMENTS

DPF test samples were prepared as shown in Table 1. Filter-A is the developed SiC filter which was produced by the new sintering process (Partial reaction sintering in nitrogen atmosphere). Filter-B is the commercial product which is a typical filter currently used in Europe. Usually, this filter is produced by higher temperature sintering in argon atmosphere. Although the specifications of these samples differ a little, they hardly influenced comparison testing.

The pore size distributions measured by mercury porosimetry are given in Fig. 2. Both samples show the sharp distribution peak which is peculiar to the SiC filter.

X-ray analysis was performed in order to check the crystaliyne phase in them, and the X-ray diffraction chart is shown in Fig. 3. They are different in the sintering process, but diffraction chart shows almost the same pattern. Since no compounds other than SiC are identified in each specimen, it turns out that they are high purity SiC re-crystallized material.

The engine bench testing and the measurement of initial filtration efficiency were carried out in order to compare the performance of these testing samples.

2-1. Engine bench testing

Engine tests were conducted using a facility in Fig. 4 with the sample of size D5.66" xL6".

2-1-1. Soot loading : A set point of 1400rpm/200Nm was used for soot loading. A theoretical soot loading charge of 10 g/liter was also used.

2-1-2. Drop to idle test (DTIT) : As shown in Fig. 5, as soon as the DPF temperature reach 680℃ by 3000rpm/190Nm, engine rotation is changed to idling condition immediately.

Table 1. Properties of DPF Test Samples

	Filter-A (Developed)	Filter-B (Commercial)
Cell Density / cpsi	169	178
Wall thickness / mm	0.38	0.36
Porosity / Vol%	42	42
Mean Pore Size / μm		
Bending Strength / MPa *	9.8	7.6
Initial Pressure Drop / KPa**	4.7	4.6

* Size: 2cell×3 cell×40mm, Span: 30mm, Load speed: 0.5mm/min

** Size: D5.66inch×L6inch, Air flow rate: 9.5Nm3/min

Fig. 1 Appearance of the test sample

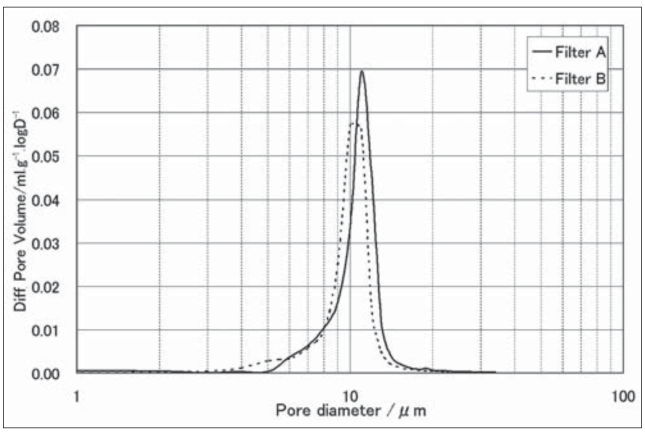


Fig. 2 Pore size distribution of DPF test sample.

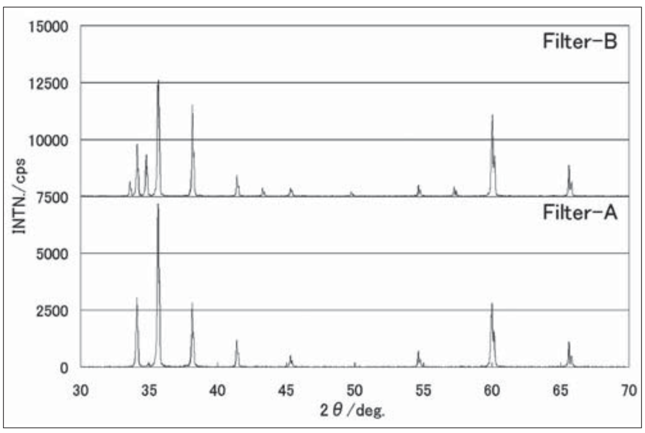


Fig. 3 X-ray diffraction patterns.

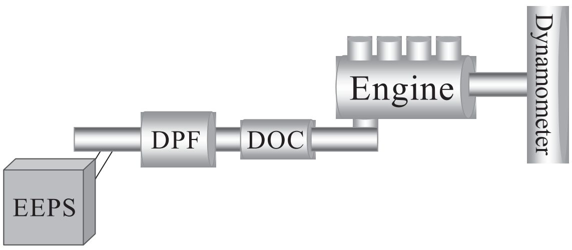


Fig. 4 Schematic of equipments.

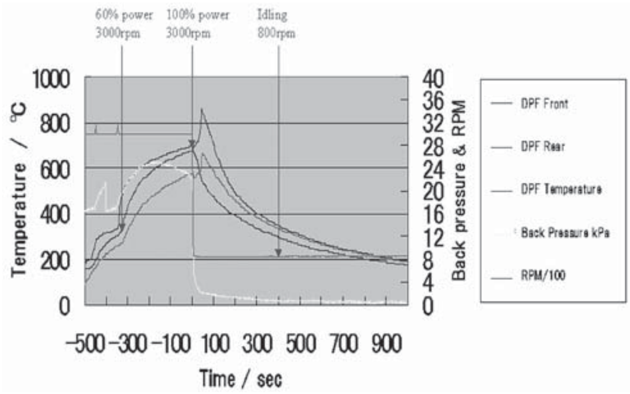


Fig. 5 Drop to idle test

2-1-3. Crack check : After cooling, DPF was cut and the existence of crack was investigated.

2-2. Initial filtration efficiency

In the same conditions as the soot loading, the concentration of particle which leaks from the outlet side of DPF during initial 2 minutes was measured by EEPS (Engine Exhaust Particle Sizer : TSI Model 3090).

3. RESULTS AND DISCUSSION

3-1. Engine bench testing

The data of exhaust gas temperature of inlet/outlet on DPF and engine r.p.m. are shown in Fig. 6 and Fig. 7. Summary of DTIT result is shown in Table 2. The maximum temperature of Filter-A is slightly lower than Filter-B although combustion efficiency is higher than the latter. Crack was not found in both samples. Filter-A shows preferable tendency of properties for regeneration and robustness.

3-2. Initial filtration efficiency

The concentration of particle which leaked from the outlet side of DPF is shown in Fig. 8, and the distributions of particle size are shown in Fig. 9 and Fig. 10. for Filter-A and B, respectively. The trapped soot amount was 0.1～0.2g/L in the initial 2 minutes. Filter-A showed less amount of soot leakage, and lapsed time until particle concentration decreases to $5\times10^6/cm^3$ was halved as compared with Filter-B.

In order to investigate the difference in filtration behavior, pieces of each filter wall after the initial 2 minutes filtration were cut out carefully, and the SEM observation was performed. SEM images of the wall surface are shown in Fig. 11. The SEM images of the wall sections are shown in Fig. 12. These SEM observation parts in the test samples are shown in Fig. 13.

On the SEM observation, the formation of soot cake was hardly found on the wall surface of each sample. This fact means that almost full filtration ability should function before the visible soot cake was formed on the wall surface. Therefore it is expected also that initial filtration efficiency is strongly affected by the microstructure of the filter material.

Generally, soot filtration on ceramic wall-flow type is performed by a combination of surface and depth filtration mechanisms. [6] On the surface of Filter-A, since the surface pores are distributing uniformly with small size, it is expected that particulates deposit efficiently on the grains which construct the pore channels and divide gas flow-lines homogeneously.

Table 2. Summary of DTIT result

	Max temperature (℃)	Combustion efficiency (%)	Weight (g)	Crack
Filter-A (Developed)	880	78	3040	no
Filter-B (Commercial)	900	69	2818	no

※Weight: Initial DPF includes canning weight

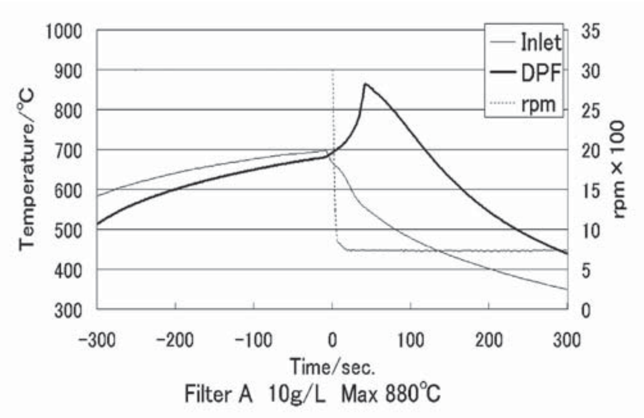


Fig. 6 DTIT Chart of Filter-A

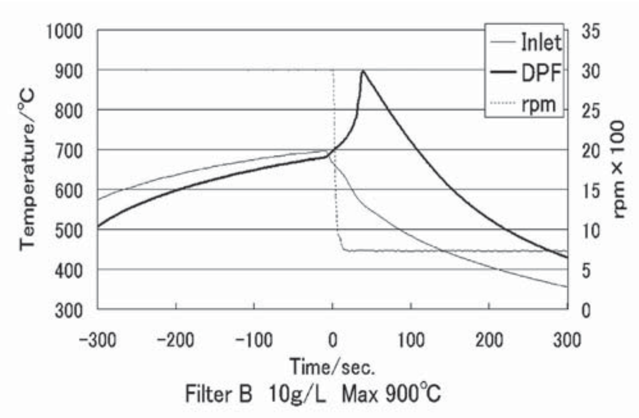


Fig. 7 DTIT Chart of Filter-B

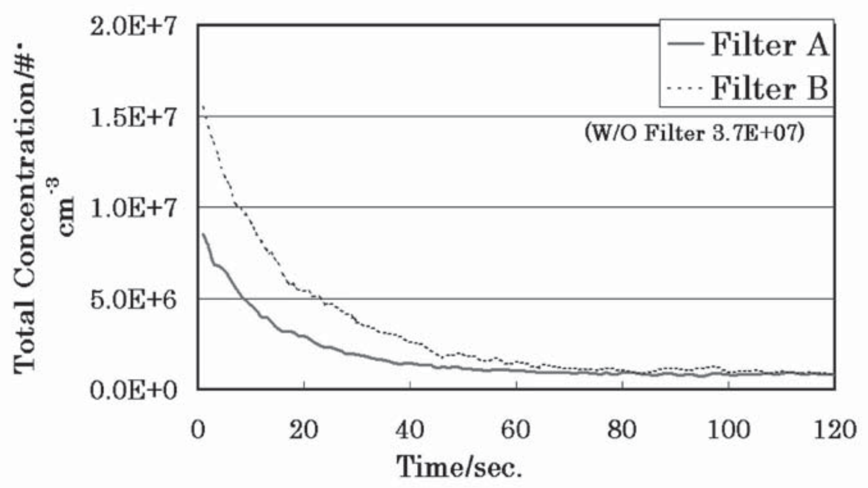


Fig. 8 Total concentration of particle which leaked from the outlet side of DPF in the initial 2 minutes.

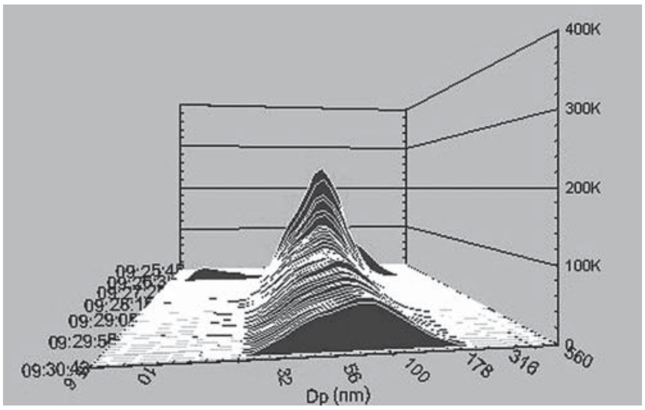


Fig. 9 Particle concentration and size distribution（Filter-A）

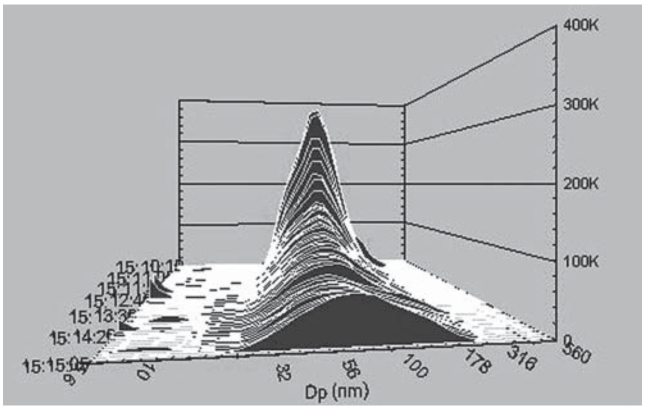


Fig. 10 Particle concentration and size distribution（Filter-B）

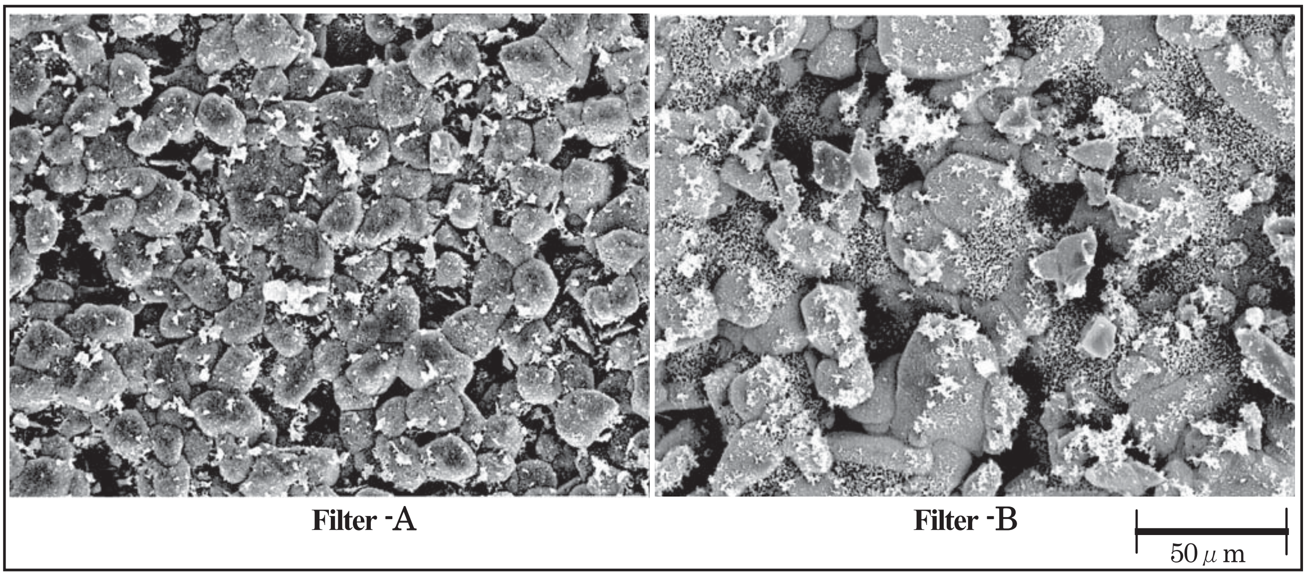


Fig. 11 SEM image of wall surface

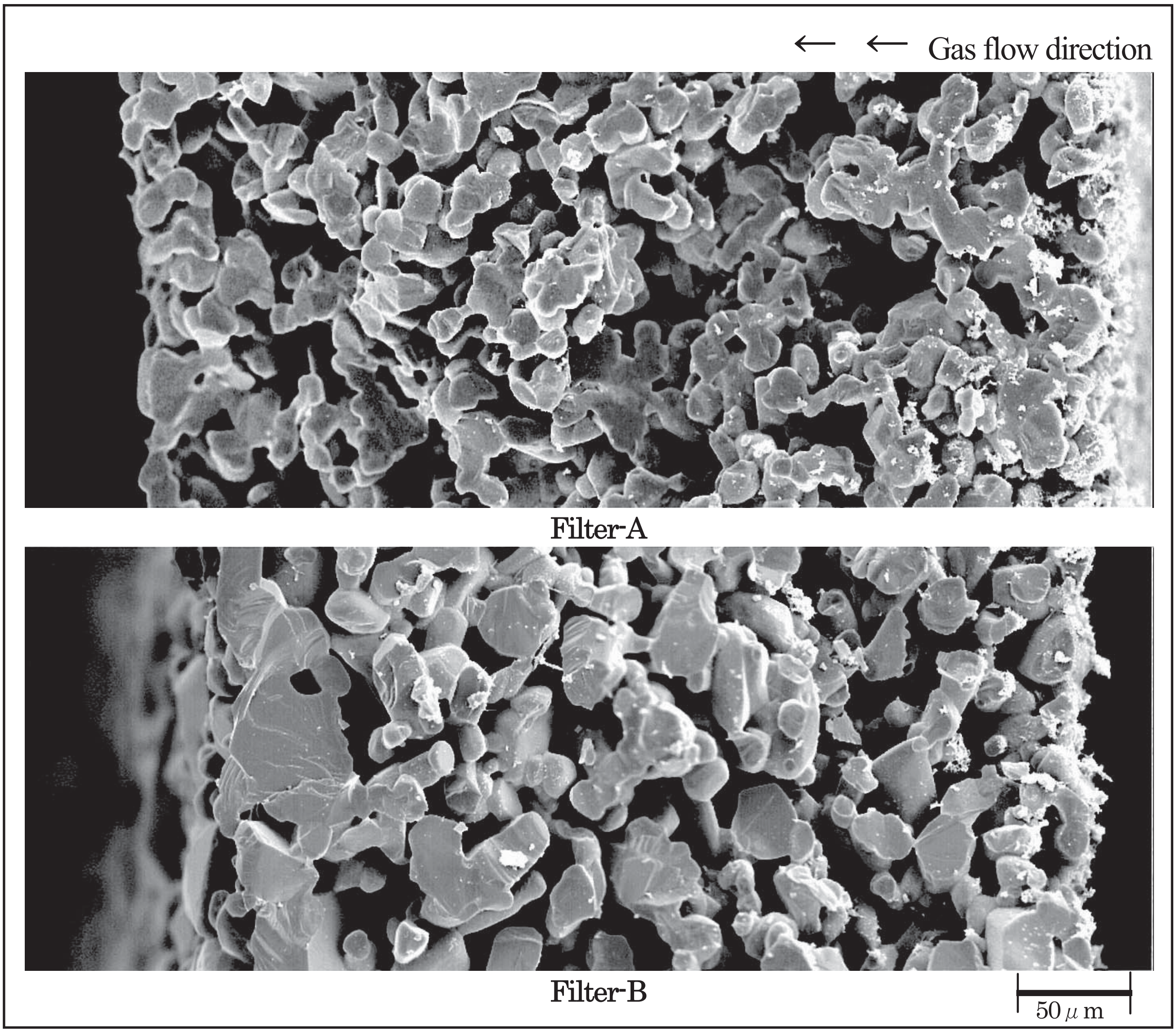


Fig. 12 SEM image of wall cross section

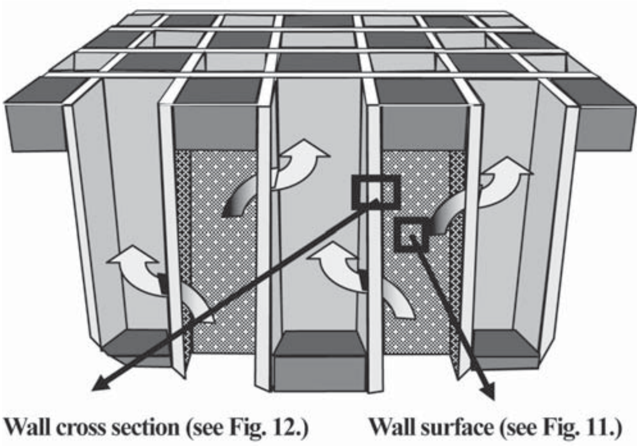


Fig. 13 SEM observation parts in the test samples.

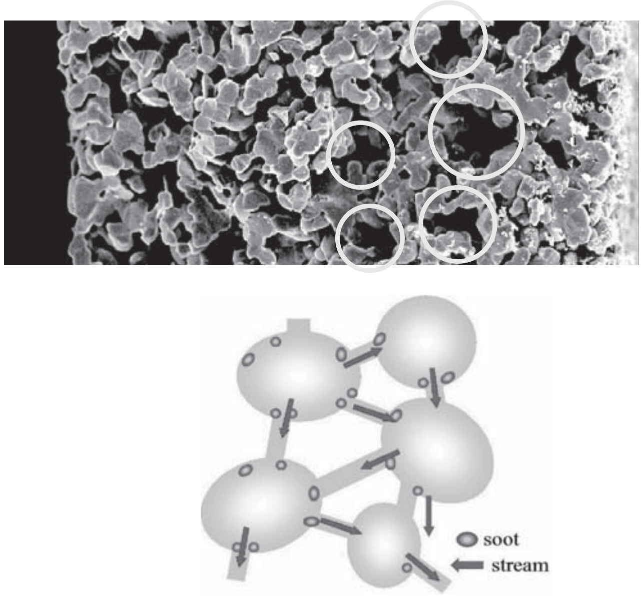


Fig. 14 Concept of microstructure in the wall.

On the section of Filter-A, the sizes of bounded SiC grains are uniform and small. Comparatively large pore are connected with complicated flow-pass distributing in the wall. This microstructure is brought about from the sintering process. It is thought that such structure is preferable for the diffusive deposition and flow-line interception of particulates[7]. (Fig. 14) Therefore, the superior initial filtration behavior on the Filter-A would be realized by the combination of the uniform pore distribution on the wall surface and complicated flow-pass structure inside the wall. On the other hand, the back pressures of Filter-A and Filter-B were almost the same level through the all range of soot loading.

4. CONCLUSION

The newly developed SiC-based diesel particulate filter which are brought about from a new sintering process (Partial reaction sintering in nitrogen atmosphere) has sharp peak of pore size distribution with high purity crystalline phase. Developed filter shows preferable tendency of properties for regeneration and robustness having lower maximum temperature and higher combustion efficiency. Developed filter shows superior initial filtration efficiency with normal back pressure. It was thought that this performance was derived from its uniform pore distribution on the wall surface and the characteristic flow-pass structure inside the wall.

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