

# Scanning Electron Microscopic Visualization of Bridge Formation inside the Porous Channels of Diesel Particulate Filters

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## ABSTRACT

Time-lapse images of particulate matter (PM) deposition on diesel particulate filters (DPFs) at the PM-particle scale were obtained via field-emission scanning electron microscopy (FE-SEM). This particle scale time-series visualization showed the detailed processes of PM accumulation inside the DPF. First, PM introduced into a micro-pore of the DPF wall was deposited onto the surface of SiC grains composing the DPF, where it formed dendritic structures. The dendrite structures were locally grown at the contracted flow area between the SiC grains by accumulation of PM, ultimately constructing a bridge and closing the porous channel. To investigate the dominant parameters governing bridge formation, the filtration efficiency by Brownian diffusion and by interception obtained using theoretical filtration efficiency analysis of a spherical collector model were compared with the visualization results. The initial deposition of PM on the SiC grains showed good agreement with theoretical observations, where Brownian diffusion played a principle role in PM trapping; however, once the bridge formation commenced, another model was required to explain the locally and intensively growing dendritic structure inside the porous channel because the filtration efficiency by pre-deposited PM on SiC exceeded that of the SiC. Since the bridge formation was accelerated by the physical extent of particles due to size reduction of the flow channel via particle deposition, the flow conditions did not significantly affect the position of bridge formation; however, they did strongly affect the time duration of the bridge formation and also the PM accumulation within the DPF wall.

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## INTRODUCTION

CO<sub>2</sub> emission from the transportation sector is an important and serious problem contributing to global warming. Owing to the high thermal efficiency of diesel engines compared to that of gasoline engines, the use of diesel engines is one solution to reduce the fuel consumption rate and CO<sub>2</sub> emission. However, the exhaust gas emitted by diesel engines contains high concentrations of pollutants such as nitrogen oxides (NO<sub>x</sub>) and particulate matter (PM) that affect human health and the environment. Hence, the regulations regarding PM emission have become more stringent in the last decade. For example, the latest European emission legislation (i.e., Euro 6 or Euro VI) limits not only the mass, but also the number of PM particles emitted [1].

A diesel particulate filter (DPF) is an important technology used to meet emission legislation. A DPF is generally made of ceramic materials such as SiC or cordierite and consists of several square channels with alternating channels blocked at each end. This structure forces the exhaust gas to flow through the channel walls, which trap the PM in their numerous microscopic pores (i.e., the trapping process).

The PM collected in the filter must be removed regularly by oxidizing the PM in order to maintain the filter performance and to avoid an increase in the back pressure on DPF (i.e., the regeneration process).

The regeneration mechanism differs depending on the size and usage of various vehicles, which affect the temperature of the exhaust gas from the engine. The regeneration processes are roughly classified as passive regeneration and active regeneration. During passive regeneration, which occurs continuously, the PM is oxidized by employing the heat of the exhaust gas and the NO<sub>2</sub> content. The amount of NO<sub>2</sub> entering the DPF is increased from the oxidation of NO by a diesel oxidation catalyst (DOC) placed upstream of the DPF. The passive regeneration system is more beneficial for a heavy load-driving mode, such as that of construction machinery and highway driving. By contrast, during active regeneration, which is performed periodically and intentionally, when the pressure drop through the DPF reaches a critical level, the temperature of the DPF is maintained at approximately 600°C to oxidize the PM via post injection of fuel. The active regeneration system is necessary for low-speed city driving because the exhaust temperature is not sufficiently high to oxidize the PM.

The timing of active regeneration is actually determined by the PM mass accumulated on the DPF, which should be estimated by the measured pressure drop. A precise prediction model is necessary to reduce the number of regeneration cycles and thus the fuel consumption of an active regeneration system, without causing any damage by the overheating that results from PM oxidation. However, the unknown relationship between the engine operation mode and the PM accumulation state, defined by parameters such as packing density and penetration depth, makes it difficult to estimate the PM mass accumulated and determine the regeneration timing correctly. Furthermore, the regeneration timing is important from the viewpoint of ash accumulation because the amount of PM accumulated prior to regeneration and the packing density of a cake play key roles in determining the ash accumulation behavior, which in turn determines the DPF life [2,3]. In order to build a good prediction model of the PM accumulation state based on the pressure drop measured across the DPF, it is necessary to comprehensively understand the PM trapping process and determine which parameters have the greatest influence on the pressure drop.

In the PM trapping process, PM is first trapped inside the porous wall of the DPF; this process is called “deep-bed filtration” [4]. Recently, “surface pore filtration” has also been used to describe this PM trapping process because the trapped PM is mainly observed in pores opening into the inlet channel in close proximity to the surface of the wall [5,6]. Subsequently, the PM trapping transitions to cake filtration, where the PM covers the wall surface uniformly. In cake filtration, the pressure drop increases linearly because the PM piles up on the uniform plane surface and the filtration efficiency shows nearly 100% in actual DPFs because the PM cake layer has high filtration efficiency of the PM [7, 8, 9, 10]. In contrast with the simple process of cake filtration, the process of surface pore filtration on the substrate wall involves a nonlinear increase in pressure drop, and is more difficult to understand. Therefore, little information on the detailed mechanism of PM trapping for surface pore filtration is available and the reason for PM trapping being observed only near the surface is unknown. Furthermore, in view of the strict regulations, which may become even stricter, a detailed understanding of the phenomena of PM trapping inside the wall is required to improve the filtration efficiency of the DPF.

In a previous study, we visualized the PM trapping process using scanning electron microscope (SEM) and found that a bridge structure of PM, which is built at a certain depth inside the porous channel at the beginning of the PM trapping process, plays an important role in determining the pressure drop and the filtration efficiency of the DPF [11]. Once the bridge, acting as a filter made of PM, obstructed the porous channel, all the PM introduced into the pore was trapped on the bridge, thereby filling the upstream pore (referred to as a surface pore). The images of PM accumulation process and the changing pressure drop are shown in Figure 1.

While PM accumulated in the surface pores, the pressure drop increased drastically before it increased linearly due to the uniform cake filtration on the DPF wall top surface. The pressure drop through the DPF with accumulated PM is determined by two significant parameters: the PM permeability and the PM penetration depth, because the flow velocity through the PM is different for inside the

pore and the wall surface. By correctly predicting these two factors, a more accurate prediction of accumulated PM mass from the measured pressure drop can be obtained. The permeability of the PM layer is thought to be determined by the carrying gas flow velocity and PM size and some models have been proposed [12,13]. However, a model for systematically estimating the penetration depth for different flow velocities or PM size has not been established because it has not been studied on a submicron scale. The final aim of this study is to develop a model to systematically estimate PM mass accumulated inside pores, determined by the position of bridge formation. Similar studies have been conducted using single fiber filters in which the different morphology of particle deposition on the single fiber for different filtration conditions such as flow velocity, particle size, and the collector size was observed and represented using Péclet and Stokes numbers [14,15].

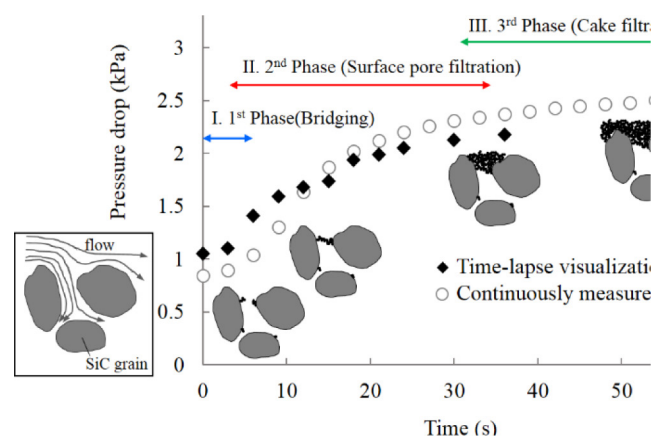


Figure 1. PM accumulation images of the trapping process and increasing pressure drop over time [11].

In this study, in the pre-stage of developing the model, the detailed process of PM bridging was visualized through a particle-scale time-lapse observation using a field emission scanning electron microscope (FE-SEM). Additionally, the dominant mechanism of bridge formation was investigated by comparing the visualization results with the corresponding theory. To achieve this, the theoretical filtration efficiency was calculated for different particle trapping mechanisms, namely, Brownian diffusion and interception.

## VISUALIZATION

### Method

In a previous study, the three steps of PM trapping on a DPF were observed through time-lapse visualization using SEM imagery where we alternated between short-term PM trapping and SEM observation [11]. In this study, we used the same visualization method but with FE-SEM, which provides better resolution and magnification for observing the location and manner of formation of the PM bridge structures than SEM. Figure 2 shows the conceptual schematic images of the visualization experiment of alternatively repeating the PM trapping process and FE-SEM observations. During the FE-SEM observation process, magnified views of the same locations were obtained for each observation. Using these images, it was verified that the PM was deposited with sufficient strength so that it was not detached and lost in the vacuum chamber.

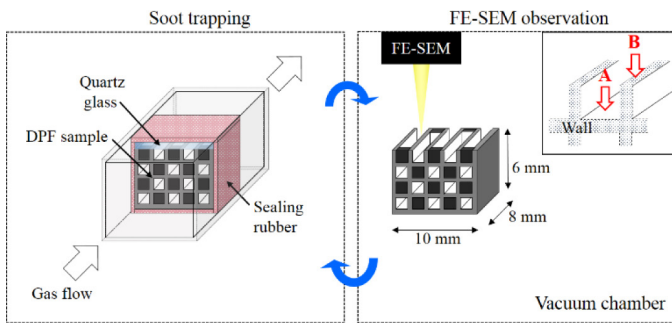


Figure 2. Conceptual image of experiment: alternatively repeating PM trapping process and FE-SEM observation with a one-wall removed sample.

In this experiment, a small DPF with 20 channels ( $4 \times 5$  matrix of channels, as shown in Figure 2) was used as a sample. This was cut into 8 mm segments having cross sections of  $6 \times 10$  mm from an actual DPF. One wall was removed to form open channels for cross-sectional observation. The top surface of each vertical wall was then polished to create a mirror-like cross-sectional surface and to make them flush. These opened channels were covered and sealed with a quartz glass plate having a smooth surface before introducing the PM into the DPF. This allowed visualization of the phenomenon of PM trapping along the cross section of the wall. The space between the DPF and sample holder was sealed with rubber to ensure that the flow carrying the PM only passed through the DPF channels. The pressure drop was measured with the same flow velocity before and after the PM was introduced into the DPF. Table 1 lists the specifications of the DPF used, for which the porosity and mean pore size were measured by a mercury penetration method and the grain size is the nominal value.

Table 1. DPF Specifications

| Filter Material  | SiC (non-catalytic) |
|------------------|---------------------|
| Cell Density     | 200 cpsi            |
| Wall Thickness   | 400 $\mu\text{m}$   |
| Porosity         | 39 %                |
| Median Pore Size | 6.5 $\mu\text{m}$   |
| Grain size       | 10 $\mu\text{m}$    |

For the visualization experiments, a small DPF sample was first placed into a flowing duct; then, diesel fuel lamp PM was introduced into the duct using an air vacuum pump. The flow volume was maintained at a constant value to ensure a superficial velocity through the wall of approximately 0.05 m/s, where the gas including PM was diluted thrice by air to observe the PM deposition process with high resolution. After 3 s of PM trapping, the pressure drop through the DPF was measured, and the DPF was carefully removed from the flowing duct into the vacuum chamber of the FE-SEM system for particle-scale visualization. The DPF sample was then placed in the flowing duct again, and PM was reintroduced. The PM deposition and observation steps were performed on the same sample in an alternating manner. Because the same microscopic field was used, particle-scale observations of the PM deposition process including the bridge formation could be compiled into “time-lapse” images corresponding to the increase in pressure drop at each time step for the surface view of wall (A) and the cross section of wall (B), respectively, as pointed in Figure 2.

In this study, PM produced by a diesel fuel lamp was used to simulate the PM emitted from practical diesel engines. In each step of PM trapping, PM was suctioned from the point at which the ratio of the flame height from the bottom of the flame to total flame height was 1:7. Figure 3 shows the PM size distribution exhausted from the diesel fuel lamp as measured using an Engine Exhaust Particle Sizer (EEPS), where the size distribution of ② was considered for the visualization. The PM size distribution shifts because the level of agglomeration changes depending on the PM-suctioning height in the flame [16].

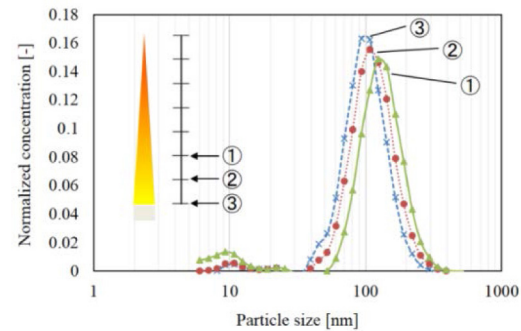


Figure 3. PM size distribution suctioned from different heights in the flame as measured by an EEPS.

### Visualized Bridge Formation and Pressure Drop

Figures 4 and 5 present a series of time-lapse images of bridge formation from the cross-sectional and surface views obtained in 3 s increments. As seen from the time-lapse images shown in Figure 4, the PM first deposited on the surface of SiC grains until 6 s elapsed. From 9 to 18 s, the PM was locally deposited atop the previously deposited PM, and dendritic structures grew perpendicular to the SiC surface, obstructing the porous flow channel. The dendritic structure of the PM finally bridged the porous channel by 21 s. This bridge formation was often observed in the contracted flow area between the SiC grains. The contribution of each filtration mechanism of large growth rate of the dendritic structure at such constricted gaps, namely, interception, Brownian motion, and inertial, were examined by comparing the experimental results with each filtration efficiency obtained theoretically; this comparison is described in the discussion section.

Similar dendritic structures were observed in the surface view of the pores depicted in Figure 5; however, the bridge was not completed until 27 s because the pore size was large. In the case of this observation, the bridge formation might have occurred in the deeper area of the porous channel behind the top SiC grains at the surface where the smaller pore is expected to exist.

In Figure 6, the red squares show the measured pressure drop during the visualization, and the black circles show the continuously measured pressure drop in another experiment without visualization. The horizontal axis represents time, beginning when the PM was introduced in the visualization, and the vertical axis represents the pressure drop measured for each PM trapping process. Since the PM number density differs between the two experiments, the horizontal axis for the two plots also differs. During bridge formation, the pressure drop through the DPF gradually increased showing a downwardly convex shape. This increase was smaller than one-third