

Ash Permeability Determination in the Diesel Particulate Filter from Ultra-High Resolution 3D X-Ray Imaging and Image-Based Direct Numerical Simulations

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ABSTRACT

Diesel engine exhaust aftertreatment components, especially the diesel particulate filter (DPF), are subject to various modes of degradation over their lifetimes. One particular adverse effect on the DPF is the significant rise in pressure drop due to the accumulation of engine lubricant-derived ash which coats the inlet channel walls effectively decreasing the permeability of the filter. The decreased permeability due to ash in the DPF can result in increased filter pressure drop and decreased fuel economy. A unique two-step approach, consisting of experimental measurements and direct numerical simulations using ultra-high resolution 3D imaging data, has been utilized in this study to better understand the effects of ash accumulation on engine aftertreatment component functionality.

In this study, ash permeability was directly measured on the surface of ceramic (cordierite) wafers as a function of ash type (field ash, lab-generated and with chemical/morphological variations) and packing density. Ultra-high resolution X-ray Computed Tomography (CT) with a transmission X-ray source (voxel size ~500nm) was utilized for direct and accurate 3D visualization of the catalyst substrate structure, the individual ash particles (which have an average size of 1-2 μ m) and the ash which penetrates into the substrate surface pores. In combination with CT data deep image analysis, a new generation of direct numerical simulation algorithms (solving Navier-Stokes equations efficiently for imaging data with billions of voxels) is used to compute permeability of the combined ash/washcoat/substrate system directly from the 3D CT images.

This study discusses the sample preparations necessary for high CT resolution, the combination of CT data and deep image processing for permeability calculations, the comparison between calculated and experimentally measured permeability values and the implications of the ability to calculate accurate permeability values in the combined ash-catalyst substrate system.

CITATION: Kamp, C., Zhang, S., Bagi, S., Wong, V. et al., "Ash Permeability Determination in the Diesel Particulate Filter from Ultra-High Resolution 3D X-ray Imaging and Image-Based Direct Numerical Simulations," *SAE Int. J. Fuels Lubr.* 10(2):2017, doi:10.4271/2017-01-0927.

INTRODUCTION

Increasingly stringent internal combustion engine emissions regulations combined with regulated fuel economy standards requires improvements in both engine and engine aftertreatment technologies. Engine aftertreatment systems utilize several different components and requires coordinated design with the engine in order to achieve the highest fuel economy and lowest emissions. Current light-duty gasoline and diesel particulate matter emissions standards are 3mg/mi (LEV III

2017-2021 phase-in) in the US and 6x10¹¹ particles/km in Europe (Euro 6). Heavy duty diesel particulate matter emissions standards are 0.01g/bhp-hr in the US and 6x10¹¹ particles/km in Europe (Euro 6) [1, 2, 3, 4, 5, 6, 7]. Decreasing the amount of soot which exits the tailpipe requires increased filtration efficiency in the particulate filter, generally resulting in increased filter pressure drop which negatively affects fuel economy [7, 8, 9, 10, 11, 12]. Fuel economy standards in the US are currently set in the range of 31-41 mi/gal for passenger cars of different sizes and 24.5-34 mi/gal for light-duty trucks of different sizes and will

be increased by up to 50% by 2025, according to the CAFE standards. Similarly, heavy duty vehicles in the US are set to reduce fuel consumption by 8-24% between 2017 and 2027 [13, 14, 15, 16, 17]. The simultaneous evolution of emissions limits and fuel efficiency places significant challenges on the industry which require novel approaches and solutions.

Typical diesel engine exhaust aftertreatment consists of the diesel oxidation catalyst (DOC), which oxidizes NO_x , unburned hydrocarbons and CO, and removes the soluble organic fraction (SOF) from the particulate matter (PM), the diesel particulate filter (DPF), which traps and oxidizes the carbonaceous PM, and Selective Catalytic Reduction (SCR), which removes the NO_x . General trends in the aftertreatment design process include reduction in component and system size, multi-functional components, such as the combined DPF-SCR (SCRf), and extended component lifetimes. In addition, direct injection gasoline engines (GDI) may soon require PM filtration, likely by way of a combined particulate filter and three-way catalyst (GPF), which can help to reduce GDI cold start soot emissions [18].

Of the typical diesel aftertreatment components, the DPF is unique due to the monolith wall-flow architecture. This design consists of a porous ceramic monolith filter (cordierite is discussed in this study), with or without a catalytic coating, where alternate channels are blocked, forcing exhaust gas through the porous filter wall in order to effectively capture the soot particles. The wall-flow design in the DPF significantly increases the pressure drop over the filter and negatively affects fuel economy, in some cases with a reduction of up to 5% over the filter lifetime [19,20]. The increased fuel consumption attributed to the DPF is due to filter back pressure and to the fuel required to heat the exhaust up to temperatures sufficient for soot oxidation (~650°C). The fuel economy losses due to the DPF are also related to the specific engine drive cycle [21, 22, 23]. Backpressure in the DPF is due to several factors including flow resistance in the porous filter media and by the accumulation of soot and lubricant-derived ash.

Ash is comprised of inorganic crystalline particles from degraded lubricant additives such as detergents and anti-oxidants/anti-wear compounds (comprised of Ca, Zn, Mg, S and P in the form of oxides, sulfates and phosphates). Lubricant-derived ash accumulates in the monolith DPF channels in several specific ways; within the filter surface pores of the inlet channels, as a layer on the inlet channel surfaces and as a plug in the rear of the filter [24]. Filter pressure drop due to ash has been shown to be due to many factors including amount [25, 26, 27], ash chemistry [8,12,21,28], interaction with substrate [12,25,29, 30, 31], ash morphology [8,9,29,21,22,24] and packing structure/density [9,32].

Filter pressure drop is due to multiple factors including flow expansion/contraction and friction along the inlet and outlet channel walls, however frictional losses as the flow passes through the porous wall, soot and ash layers accounts for the majority of flow resistance. Permeability is a key parameter for understanding the magnitude of pressure drop over the DPF. In addition, estimates for permeability values of the DPF substrate and for the layer of ash are often used in the engine and aftertreatment design and optimization processes. It is

an intensive property of the porous medium and thus does not depend on the sample size for a homogenous medium. Permeability values for the filter substrate, soot and ash layer are generally approximated by measurement of the filter pressure drop with a number of assumptions, and often utilizes a form of Darcy's law, such as Forchheimer-Extended Darcy equation for each layer:

$$\Delta P_{filter/ash/soot} = \left(\frac{\mu}{K_p} \right) \cdot v_w \cdot w + \beta \cdot \rho \cdot v_w^2 \quad (1)$$

where K_p is the layer permeability of the filter, ash or soot, μ is the dynamic viscosity of the exhaust gas, v_w is the exhaust gas wall velocity, w is the thickness of the filter wall, soot or ash layer, ρ is the exhaust gas density and β is a term describing inertial resistance, however inertial resistance is generally neglected as the Reynolds number for flows through the porous layers is less than one. Ash and soot accumulation in the DPF increases the material layer thickness (w) and introduces additional flow resistance through the porous layer which is reflected in the layer permeability, K_p [33,34]. The permeability of the filter substrate, soot and ash can be approximated by the following equation:

$$K_w = \frac{1}{5.6} \left(1 - \frac{\rho_{measured}}{\rho_{theoretical}} \right)^{5.5} d_p^2 = \frac{1}{5.6} \varepsilon^{5.5} d_p^2 \quad (2)$$

where ε is the porosity, $\rho_{measured}$ is the measured density and $\rho_{theoretical}$ is the true material bulk density and d_p is the pore diameter [33]. It should be noted that equation 2 uses several assumptions and is only valid for a specific range of porosities and particle sizes. In general, calculating permeability through the Darcy's equation makes a number of assumptions, including homogeneous properties such as porosity, and neglects effects on solid boundaries and will be discussed later in the results section. Also, the effect of material accumulated in the pores, as opposed to the cake layer, is more complicated; however, it can be accounted for in the above equation by modifying the filter's wall permeability to reflect the amount of material occupying the pores, i.e. reduction in pore diameter.

While measurements of substrate permeability, porosity, density, and mean pore size are fairly straightforward, the same measurements are significantly more challenging when applied to the ash and soot layers. Further, soot and ash also accumulate in the pores of the DPF during the initial filter loading phase, altering the properties of the substrate as well. Soot packing density and the resulting soot layer permeability is significantly affected by soot particle size, deposition angle, and velocity, among others.

In-situ investigations have shown that the material which accumulates in the DPF pores does not penetrate deep into the filter, but rather accumulates in the pores near the filter surface [35]. Due to the large effect of depth filtration on the overall filter pressure drop, composite permeability models have been developed previously to account for material accumulation in the filter pores [33]. The approach taken to estimate the composite permeability of the filter walls, accounting for soot and ash trapped in the pores, is analogous to the sum of resistances in series. The total wall permeability is, therefore, computed as:

$$\frac{1}{K_{Composite}} = \frac{1}{K_{Wall}} + (AWF) \frac{1}{K_{Ash}} + (SWF) \frac{1}{K_{Soot}} \quad (3)$$

where $K_{composite}$ is the composite permeability of the wall layer including material trapped in the DPF pores, K_{wall} is the permeability of the clean filter wall, K_{ash} is the permeability of the ash, and K_{soot} is the permeability of the soot. Further, a weight fraction term is defined to describe the amount of ash and/or soot accumulated in the pores relative to the available pore volume. The composite permeability is therefore, based on the relative weight fractions or amount of material deposited in the filter pores.

Permeability values in literature have been measured/calculated in the range of 0.95×10^{-14} – $8 \times 10^{-13} \text{ m}^2$ for cordierite filters [36, 37, 38, 39, 40], 1.8 – $9.6 \times 10^{-14} \text{ m}^2$ for the ash layer [34, 37, 41, 42] and 3.2 – $3.3 \times 10^{-14} \text{ m}^2$ for the soot layer [34]. Permeability is demonstrated to be a non-linear characteristic for the soot layer, the ash layer and the wall with respect to porosity as shown in [equation 2](#). These physical coefficients are required to solve the differential equations that correlate back-pressure with flow parameters. The soot layer thickness and the soot permeability depend on the morphology and density of soot, and its compaction. The engine combustion conditions and the fuel characteristics primarily influence the nature of soot, while the flow velocity induces the compaction and is well-correlated with the Peclet number. A large range of soot density values from 0.026 g/cm^3 [43] to 0.11 g/cm^3 [44] or 0.123 g/cm^3 [45] have been reported in the literature. Previous studies [46, 47, 48, 49] use empirical equations to define permeability coefficients, where permeability is also a function of particle porosity. Recent studies have also shown that the permeability of the ash layer depends on upstream lubricant additive chemistry [33, 50].

In a study by Fujii et al., it was seen that permeability of the ash layer is more than double of that of soot layer [41], however this study used synthetic ash which may not be realistic. The presence of an ash layer on the DPF wall prevents trapping of soot particles into the wall pores which would have resulted in a steep back pressure increase otherwise. In consequence, deep bed filtration becomes less dominant due to ash accumulation. On the other hand, ash deposits accumulated on the wall do not result in a steep back pressure increase because of the higher permeability. In some studies, the determination of permeability of a wall-flow filter sample is computed from careful experimental measurements of entire DPFs combined with continuum-level mathematical models [39]. As for the determination from first principles of the inertial losses coefficient, the use of three-dimensional CFD methods has been already demonstrated to be promising [49, 50, 51]. However, the computational effort required for an extensive parametric study of a multichannel configuration makes experimental testing the route of choice for the immediate future.

The aim of this study is to introduce a method capable of accurately measuring/calculating the permeability of filtration components in the aftertreatment system, such as the Diesel Particulate Filter (DPF), which can help to improve the design and optimization of the engine and other aftertreatment components. This method utilizes the combination of ultra-high resolution (sub-micron) 3D X-ray

computed tomography (CT) and image-based direct numerical calculations. Experimental wafer-based permeability measurements will be compared with the method introduced in this work.

EXPERIMENTAL

This study includes efforts in three areas which contribute to a method for improved resolution in obtaining accurate permeability values relevant to engine aftertreatment components with a focus on the wall-flow diesel particulate filter and lubricant-derived ash within the DPF. The three areas of focus include: (i) an experimental setup to measure ash permeability as a function of lubricant chemistry and packing density, (ii) efforts to improve X-ray CT capabilities specifically for aftertreatment filtration media, and (iii) utilization of a combined image analysis and direct numerical flow simulation toolset. The aforementioned focus areas introduce a unique approach for calculating permeability in porous media in the context of engine aftertreatment systems.

Experimental Permeability Measurement

The goal of the experimental permeability measurement was to decouple several of the key variables, including ash packing density and additive chemistry, which affect the permeability of the ash layer and ultimately the pressure drop of the filter. This was done in part by removing both the ash and filter material from the large honeycomb structure of the DPF in order to set up a 1D flow regime so that the permeability can be measured accurately. In addition, the pressure drop contributions from friction losses and contraction/expansion losses are removed. Another practical outcome of such an experiment which measures permeability over a range of ash chemistries and packing densities is that it allows for significant improvements in modelling accuracy.

A flow bench was set up with accurate air flow control and pressure measurement in a sealed system, as shown in [figure 1a](#), and was mounted vertically to reduce any potential effects of gravity on the ash layer which could redistribute the layer. The density of the ash is controlled by measuring a precise mass of ash and compressing it to a constant volume using a specially designed packing tool and using the known diameter of the tube, as shown in [figure 1b](#). This process of controlling the packing density through a controlled volume allows for a density accuracy of $\pm 0.005 \text{ g/cm}^3$.

A wafer holder, shown in [figure 1c](#), was designed in order to hold the brittle ceramic wafer and to prevent leakage for accurate pressure measurements, while retaining the ash cake layer in place. A cordierite wafer is prepared by cutting it from a monolith and then sanding with fine grit sandpaper to a diameter of 0.75" and two channels thick in order to provide structural support, as shown in [figure 1d](#). The flow through the wafer passes through 3 cordierite walls from inlet to outlet. The wafer edges are coated in epoxy, as shown in [figure 1e](#), and the wafer is held between two compliant gaskets. While the epoxy may penetrate into the ceramic edges to some extent, the ash cake layer diameter is restricted from the epoxy-coated edges by the gasket, thus the epoxy should not affect flow measurements through the ash.

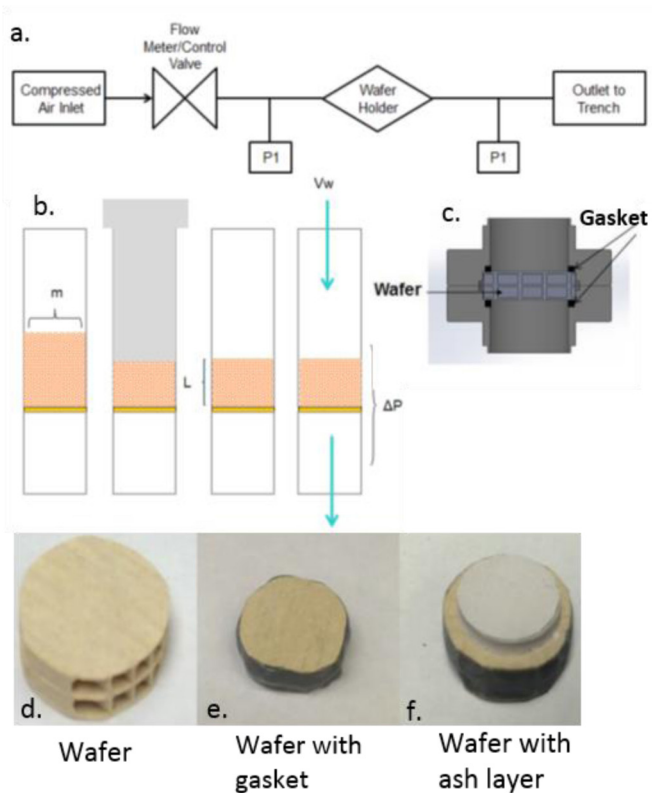


Figure 1. Images show a) the experimental setup as a flowchart, b) an explanation of the 1D flow regime in the wafer setup where the height of the ash layer is controlled by a cylindrical ash packing tool, c) the wafer holder design, d) a freshly cut cordierite wafer, e) a wafer with the gasket material and f) a wafer with an ash layer after measurement.

To accurately represent the flow conditions existing within a DPF in typical use, the wall velocity through the wafer must be equal to the average wall velocity through the channel walls of the DPF. Space velocity (SV) is used to describe the normalized volumetric flow rate through a given volume of filter, where typical values range from 0 – 100,000 hr⁻¹, representing conditions from off to idle to full throttle. Space velocity in a filter is converted to wafer wall velocity by the following equation,

$$VPA = 2 * (l - 2l_p) * \left(\left(\frac{1}{\sqrt{CPA}} \right) - t \right) * CPA * \pi \left(\frac{D^2}{4} \right) \quad (4)$$

where VPA is the volume per unit flow area, D is the filter diameter, l is the filter length, t is the filter thickness, CPA is the cells per unit area and l_p is the length of the ceramic channel plugs in the filter. For a typical filter (5.66" x 6", 300 cells per square inch (CPSI)), the approximate wall velocity ranges from 0 to 3.5 cm/s. An ash layer diameter 0.62" was chosen resulting in a flowrate of 0-400 cm³/min (SCCM) for the system volume flow rate. An Omega mass flow controller (model: FMA-2620A) was used to accurately control the flow rate through the system.

The lubricant additive chemistry/ash composition and ash packing density are varied in this experiment. Typical packing densities of ash cake layers found both in laboratory-aged and field-aged filters range

from typical values of 0.18 to 0.6 g/cm³ and over 1 g/cm³ for highly sintered ash. This experiment is primarily concerned with ash particles packed into a cake rather than a fully sintered region; hence, a range of packing densities of 0.15 - 0.75 g/cm³ was chosen to represent the full range of typical values and to see the trends near the extremes of packing densities encountered. The powder ash samples used in this experiment have been carefully removed from both laboratory and field-aged filters. The field ash samples are from on-road applications which use CJ4 oils that contain 1% ash by mass. The lab-generated ash samples are all derived from the loading experiments described in previous literature [52]. The ash types used in this experiment are described below.

Table 1. Ash types used for ash cake layer permeability experiment

Ash Sample Type	Regeneration type
Field Ash (Active)	Active
Field Ash w/Soot	Active
CJ4 (Active)	Active
CJ4 (Passive)	Passive
Base Oil + Ca	Active
Base Oil + Zn (ZDDP)	Active
Base Oil + Mg	Active
Base Oil + Zn (ZDDP)+Mg	Active

After mounting the clean wafer into the sample holder and assembled in the flow bench, the flow rate is swept from 0 to 400 SCCM at a rate of 100 SCCM/min while the pressure drop is measured. This allows for calculation of the wafer permeability as well as a normalization to find the pressure drop increase strictly due to the addition of the ash cake layer. Next, the sample section is removed and a precise mass of ash is added to the wafer giving an expected packing density of 0.15 g/cm³. The ash is then evenly distributed to avoid regions of varying density with an overall nominal packing density. A specially made slip-fit piston is used to compact the ash to the correct volume. The piston has a seat matching with the top of the tube to prevent additional unwanted travel. With the ash loaded, the holder is reattached to the flow bench where the flow sweep and pressure measurement procedures are repeated. For all additional ash loading steps, the preceding ash cake layer is loosened and disturbed before adding the new ash to prevent stratification of the cake layer or localized density variation. The new mass ash is then added to increase the packing density to the next step. The ash is then mixed until evenly distributed and packed again. This procedure is repeated for each value of packing density and the full test matrix of ash types. The pressure drop contribution from the wafer was then subtracted from the pressure drop of the composite (wafer+ash) system, leaving the pressure drop associated only to the ash layer. In addition, each packing density value for each ash type was repacked and measured several times to ensure reproducibility. The ash-related pressure drop varied by <5% for the repeated measurements.