

## Ash Accumulation and Impact on Sintered Metal Fiber Diesel Particulate Filters

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### ABSTRACT

While metal fiber filters have successfully shown a high degree of particle retention functionality for various sizes of diesel engines with a low pressure drop and a relatively high filtration efficiency, little is known about the effects of lubricant-derived ash on the fiber filter systems.

Sintered metal fiber filters (SMF-DPF), when used downstream from a diesel engine, effectively trap and oxidize diesel particulate matter via an electrically heated regeneration process where a specific voltage and current are applied to the sintered alloy fibers. In this manner the filter media essentially acts as a resistive heater to generate temperatures high enough to oxidize the carbonaceous particulate matter, which is typically in excess of 600°C. The regeneration process does not require additional fuel, such as in the case of active regeneration in conventional/ceramic wall-flow diesel particulate filters (DPF), since the energy required for regeneration typically comes from another source like an auxiliary battery. In addition, the SMF-DPF is not sensitive to fuel quality (especially fuel sulfur levels), engine load and exhaust temperature.

Just as in the case of the wall-flow DPF, lubricant derived ash (typically comprised of ionic crystalline solids including the following elements: Ca, Zn and Mg in the form of solid phosphates, sulfates and oxides) is observed to accumulate in the fiber filter systems over its lifetime.

This study examines the behavior of lubricant-derived ash accumulation in SMF filters which were used to remove particulate emissions from diesel engine exhaust in various field applications. High resolution visualization and quantification of both inherent ash properties and ash accumulation characteristics are presented. Specifically, the regeneration strategy (active vs. passive) utilized in each filter is shown to significantly affect the inherent ash properties, such as ash particle size and interaction with the metal fibers within the filter. A number of analytical tools have been used in this study including focused ion beam (FIB) milling, Ar<sup>+</sup> ion milling for cross sectional analysis, environmental scanning electron microscopy (ESEM), energy dispersive X-ray spectroscopy (EDX) and X-ray Computed Tomography (CT). The primary objective of this study is to show, for the first time, some of the general characteristics of lubricant-derived ash within SMF particulate filters, and to provide some insight into the role ash plays over the useful lifetime of the fiber filters.

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## INTRODUCTION

**METAL FIBER FILTERS** - Sintered metal fiber filters (SMF) have recently been used for both retrofit and OEM applications as diesel particulate filters for a number of reasons. These include applications which typically operate at low exhaust temperatures, and/or high sulfur levels in fuel. The SMF filter media provides low back pressure and high soot trapping efficiency (>85%) with very low fuel penalty (<1%) [1]. In comparison, the wall-flow DPF typically has a filtration efficiency of >95% but with higher fuel penalty. The SMF-DPF traps diesel PM and oxidizes it by passing current through the metal fibers, turning the filter into a resistive heater. SMF filters have been utilized successfully in diesel stationary power generators and are currently being applied to other applications including mobile diesel generators, locomotive generators, trucks (TRUs and APUs), buses, off-road construction vehicles and marine applications (power generation and propulsion) [1].

The SMF-DPFs investigated in this study are provided by Rypos, Inc. and the fibers are made of FeCrAlloy, an alloy of iron, chromium, aluminum and yttrium, with a thin aluminum oxide protective layer surrounding the fibers (held on the fiber surface by the yttrium) which protects the fibers against corrosion from high temperature [1]. Fibers of three different nominal sizes (35  $\mu\text{m}$ , 22  $\mu\text{m}$  and 17  $\mu\text{m}$ ) are pressed (in random X-Y orientation) into a layered 1mm (nominally) thick mesh filter, as schematically shown in [figure 1](#), with a high porosity of about 85%.

For comparison, ceramic wall-flow DPFs are generally between 50 and 60% porous. The SMF filter is made into a corrugated, cylindrical cartridge, a number of which are combined in stacks with multiple cartridges depending on the specific application (size of engine, exhaust flowrate, etc.), and is accompanied by an electrical control circuit, flow control hardware and a filter housing.

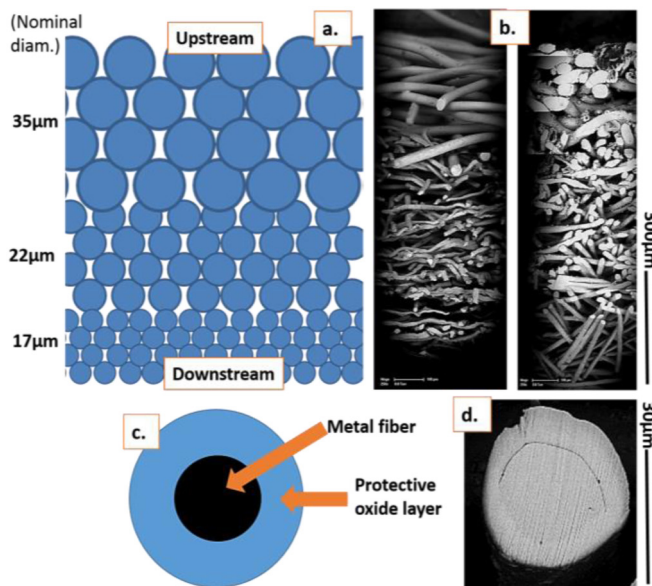


Figure 1. a) Schematic of the SMF filter where the filter substrate consists of 3 layers, each layer with a different nominal fiber thickness, shown via SEM images prepared by an  $\text{Ar}^+$  ion mill in b). c) Schematic of an individual fiber showing the fiber with the surrounding protective oxide layer, which is shown via SEM and prepared again by an  $\text{Ar}^+$  ion mill in d).

The SMF filter unit can be used with or without a diesel oxidation catalyst (DOC) and is controlled by a microprocessor which regulates the automatic regeneration cycles, the timing sequence/duty cycle, the flow control and filter unit selection, pressure and temperature sensing, and remote display. The filter material is heated quickly due to its low thermal mass and multiple filter cartridges are used in stacks, but heated separately to optimize energy usage. In some applications, the stacks include a combination of electrically regenerated filter cartridges and non-electrically regenerated cartridges. The electrically regenerated cartridges are termed 'active' in this study and the non-electrically regenerated cartridges are termed 'passive' in this study. Exhaust flow through a stack is reduced while a cartridge in the stack is regenerating.

## Particulate Matter Filtration

The basic function of the particulate filter is to trap and burn all PM exiting the engine via oxidation. Catalyzed particulate filters rely on platinum-based and/or other catalysts to convert engine-exhaust NO to  $\text{NO}_2$ , in order to oxidize carbon with a significantly lower activation energy. Non-catalyzed filters increase the exhaust temperature up to approximately 600°C for a short period of time in order to thermally oxidize carbon with  $\text{O}_2$ . SMF filters used as particulate traps utilize the latter oxidation mechanism, as they do not rely on catalyst coatings, and provide the necessary thermal energy to the reaction by an applied voltage since the metal fibers act as a resistive heater.

Regardless of which filter regeneration method is used, the filter retains a small amount of inorganic, incombustible material which comes from the engine lubricant additives (generally from anti-wear and detergent additives). Typically, this inorganic material, known as ash, accounts for less than 1% of the total engine-exhaust PM and consists of elements Ca, Zn, Mg, S and P in the form of ionic crystalline sulfates, phosphates and oxides. Ash precursors, or ash bound to the soot leaving the engine, are on the nanometer scale (approx. 5-80nm particles) while the ash agglomerates can be hundreds of microns in dimension built up of primary particles in the range from approx. 100nm to several microns [2].

As trapped soot is oxidized it shrinks in size, concentrating ash precursors and supplying energy for ash crystalline growth through the exothermic carbon oxidation reaction. [Figure 2](#) shows TEM images of primary ash units (primary units are small, micron sized agglomerate of ash) which have been collected from field-aged wall-flow DPFs (fully formulated CJ4 oil) and from DPFs aged in an accelerated aging system (base oil with single additives including Ca, Mg, Zn) as described in [3]. The different ash types shown in [figure 2](#) indicate a relationship between lubricant chemical composition and ash morphology where CJ4 oil contains additives including Ca, Zn and Mg, yet each additive produces unique ash composition and morphology.

Ash in the wall-flow DPF has been discussed heavily in literature due to the fact that it leads to significant filter pressure drop increase, but also increases filtration efficiency [4,5]. Most notably ash increases

the filter pressure drop by clogging pores in the filter substrate and by forming a cake layer of ash on the filter inlet channel walls, as shown in the SEM image in [figure 3](#) and schematically in [figure 4](#).

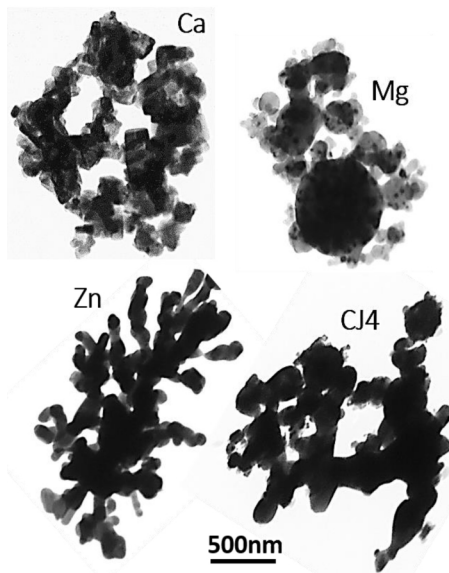


Figure 2. TEM images of lubricant-derived ash agglomerates showing that specific inorganic lubricant additives result in specific ash morphologies.

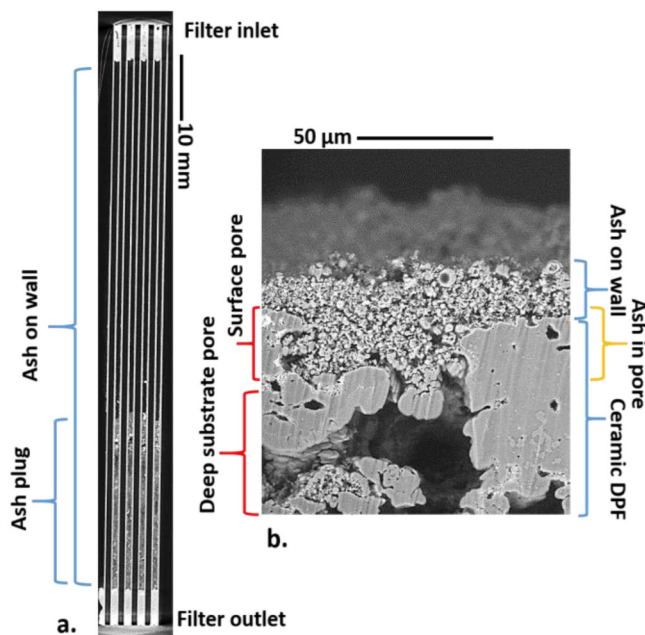


Figure 3. X-ray CT image of ash distribution within a ceramic wall-flow DPF shown in a) and an SEM image of a sample prepared by Argon ion milling showing ash deposition on the surface of the ceramic DPF and within a surface pore in b).

The specific filtration mechanism of fiber-type filters is inherently different than wall-flow filters and shows a unique dependence on trapped solid material. For example, trapped material initially interacts with wall-flow DPFs and fiber filters differently, as described by the schematics in [figure 4](#). Solid material gets trapped initially in surface pores of the wall-flow DPF, in what is known as deep bed filtration, and has a very strong impact on the pressure drop. Solid material initially accumulates randomly in the internal void spaces within the

SMF filter and has a less severe impact on pressure drop, as described schematically and by a relative pressure drop plot in [figure 4](#). Solid particulate matter filtration has been described in literature for High Efficiency Particulate Air filters (HEPA) both experimentally and theoretically, showing this pressure drop trend [6].

After trapped solid material fills the filter substrate void spaces, being surface pores for the wall-flow DPF and internal porosity for the fiber filter, the pressure drop response transitions to a cake-layer deposition mechanism where a layer of solid material forms on the upstream side of the filter surface. In this cake layer filtration mechanism, the pressure drop response is based on the thickness and permeability of the accumulated solid material and should be similar for both filter types for accumulated ash, as shown by [figure 4](#).

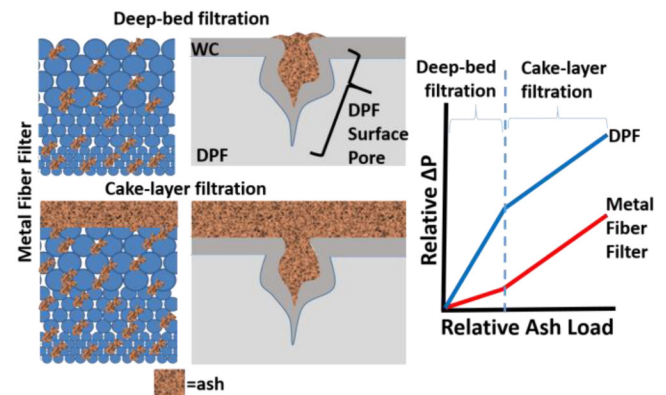


Figure 4. Schematic diagrams (left) explaining deep-bed and cake-layer filtration types in the wall-flow DPF and metal fiber filter, and an explanatory relative pressure drop vs. relative ash load plot (right).

The filter pressure drop evolution as a function of trapped material, soot and/or ash, is fairly well understood for the case of the wall-flow DPF [4,5], but is less understood in the case of the SMF filters being used to trap diesel PM. For this reason, this study investigates the ash accumulation characteristics from several field-aged SMF-DPFs, including general ash distribution and specific ash-fiber interactions. The results of the present study provide useful insight for modeling, design and optimized use of sintered metal fiber filters for diesel exhaust PM filtration.

## EXPERIMENTAL

Several experimental tools are used for both destructive and non-destructive analysis of clean and field-aged SMF filters in order to examine ash properties and distribution characteristics. General imaging is done with an FEI/Phillips XL30 Field Emission Gun (FEG) Environmental Scanning Electron Microscope (ESEM) with EDAX Energy Dispersive X-ray Spectroscopy (EDX). Filter cross sections are prepared on the mm scale with a JEOL Ar+ ion mill cross section tool and on the nm-μm scale with an FEI Helios Nanolab 600 Ga+ dual beam Focused Ion Beam (FIB). The FIB technique used in this study is described in [7,8]. Three dimensional data is collected with an XRA-002 X-Tek MicroCT X-ray Computed Tomography (CT), with a general resolution reflection X-ray source and a high resolution NanoTech transmission X-ray source and processed in Visual Graphics StudioMax.