

Sensitivity Analysis of Ash Packing and Distribution in Diesel Particulate Filters to Transient Changes in Exhaust Conditions

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ABSTRACT

Current CJ-4 lubricant specifications place chemical limits on diesel engine oil formulations to minimize the accumulation of lubricant-derived ash in diesel particulate filters (DPF). While lubricant additive chemistry plays a strong role in determining the amount and type of ash accumulated in the DPF, a number of additional factors play important roles as well. Relative to soot particles, whose residence time in the DPF is short-lived, ash particles remain in the filter for a significant fraction of the filter's useful life. While it is well-known that the properties (packing density, porosity, permeability) of soot deposits are primarily controlled by the local exhaust conditions at the time of particle deposition in the DPF, the cumulative operating history of the filter plays a much stronger role in controlling the properties and distribution of the accumulated ash. Results of this work indicate that short-duration, transient, high temperature events can have a profound impact on ash packing and DPF pressure drop, while exposure to transient high flow rate conditions produce only marginal changes.

The tests conducted in this work utilized core samples, removed from DPFs containing known ash levels, to study the effect of transient changes in exhaust conditions on ash properties using a flow bench. Use of the core samples enabled more precise control of DPF exposure to transient variations in exhaust flow and temperature conditions. In addition to the filter performance characterization, application of advanced diagnostics including scanning electron microscopy (SEM), energy dispersive x-ray analysis (EDX), and x-ray diffraction (XRD) provide insights into the changes in ash properties induced by the variations in exhaust conditions, useful to explain the observed results. Enhanced understanding of the fundamental mechanisms controlling ash properties and their impact on DPF performance is useful to not only extend the filter's service life, but more importantly to improve both DPF control and on-board diagnostic capabilities.

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INTRODUCTION

Diesel particulate filters (DPF) currently provide the most effective means for reducing particulate matter (PM) emissions from diesel engines to below mandated levels. While extremely effective at trapping and oxidizing the soot particles, incombustible material, or ash, also accumulates in these filters over time. Ash accumulation increases exhaust flow restriction through the DPF, negatively impacts fuel consumption, and further decreases the filter's soot storage capacity. In order to minimize the impact of ash accumulation on DPF performance, filters are often oversized to provide additional ash storage capacity, periodic maintenance and filter cleaning or replacement is required, and lubricant formulators have reduced the amount of ash-related additives in diesel engine oils, among other strategies. It is the latter

topic, namely ash originating from the lubricant additives, which is the focus of this study.

Ash accumulated in the DPF is generally a complex mixture of various metal sulfates, phosphates, and oxides. While engine wear metals, exhaust system corrosion products, and trace metals in diesel fuels may contribute to some extent, the majority of the ash originates from lubricant additives. Chief among the additive components regularly found in DPF ash are detergents, either calcium- or magnesium- based additives, and anti-wear/anti-oxidant additives, primarily zinc dialkyl-dithiophosphates (ZDDP). In order to reduce the accumulation of lubricant-derived ash in diesel particulate filters, the CJ-4 oil specification was developed and limits the sulfated ash content of the lubricant to 1.0%, while placing additional specific chemical limits on the oil [1].

Despite reducing the lubricant's ash-related additive content, lubricant-derived components still make up the majority of the ash accumulated in the DPF. Unlike the soot accumulation and oxidation processes (regeneration) which occur on relatively short time-scales, ash deposit build-up in the DPF occurs much more slowly, over thousands of hours of filter operation. Currently the EPA minimum ash cleaning interval for heavy-duty diesel applications is 150,000 miles [2]. Over the course of these long residence times in the filter, the ash deposits are subjected to a wide range of exhaust conditions, which range from near ambient conditions with the engine off, to high temperature operation during active DPF regeneration, and a range of exhaust flow and temperature conditions in between. While much work has been devoted to understanding the manner in which lubricant ash level, and more recently specific additives, impact ash properties and DPF performance, much less is known about the manner in which exhaust conditions influence ash properties such as packing density and permeability, as well as the ash mobility and distribution in the filter channels. The aim of this work is, thus, to identify the extent to which ash properties, and ultimately DPF performance, may be affected by transient variations in exhaust conditions.

EXHAUST CONDITIONS

Before embarking on a detailed discussion of exhaust conditions, temperature and flow histories, and various operational scenarios experienced by the DPF, it is first useful to understand the time scales of these processes in relation to the ash accumulation and buildup process. For purposes of this paper, two general time-scales are defined: the first being long time-scales on the order of thousands of hours and tens of thousands of miles during which the ash deposits accumulate in the DPF, and the second being relatively short time scales on the order of seconds or minutes to describe transient fluctuations in exhaust conditions.

Ash accumulation and deposit formation in the DPF occurs over the course of the long time scales described above. During this time, nanometer-sized ash precursors, originating from the lubricant additives, are transported through the exhaust intimately bound to the engine-out soot particles [3]. These ash precursors initially accumulate in the DPF, well mixed throughout the soot cake layer along the filter walls. Following repeated regenerations, where the soot is oxidized, the ash precursors begin to agglomerate and form the micron-sized ash particles generally observed in the DPF ash deposits [4, 5]. Over these long time-scales, the ash accumulates either in a layer along the channel walls or moves from the walls to form an ash plug at the back of the DPF channels [6, 7].

There is a growing body of circumstantial evidence indicating the DPF operating history, as defined by a specific regeneration strategy, may directly impact the mobility and location of the ash deposits over the long time-scales during which the ash accumulates. In particular, several studies have shown that DPFs subjected to continuous or passive

regeneration, where little soot is accumulated prior to oxidation, exhibit thicker ash deposits along the channel walls, and less ash accumulation in the channel end-plugs. On the other hand, the same studies have shown that filters subjected to periodic, and higher temperature, regenerations exhibit less ash accumulation along the channel walls and more ash build-up in the channel end-plugs [5, 8, 9, 10].

Generally the average exhaust conditions defining the DPF operating history over the duration of the ash accumulation processes are fixed; that is they are determined by the aftertreatment system design, regeneration strategy, vehicle drive cycle, and other related and relatively inflexible parameters. Thus for a particular aftertreatment system architecture installed on a specific application, the exhaust flow and temperature conditions can be expected to fall within a fairly well-defined range for "typical" system operation.

In contrast to the average exhaust conditions experienced by the DPF and its accumulated ash over the long time-scales, described above, transient changes in exhaust conditions occur over much smaller time-scales. Whether intentional or unintentional (such as an uncontrolled regeneration), the ash accumulated in the DPF may also experience short periods of time under fairly extreme exhaust conditions. In these cases, ash deposits, whether in the wall layer or end-plug, have already accumulated under a given set of exhaust conditions, but may occasionally experience transient variations in the exhaust conditions outside the "normal" range under which the ash deposits were accumulated. The question this work aims to address is, how do short-duration, transient exhaust temperature or high flow excursions affect the properties (packing, distribution, permeability, etc.) of the preexisting ash layer?

In order to address this question, it is useful to understand the range of exhaust conditions, specifically exhaust temperature and flow rates, which the filter may experience over the course of its useful life. Figure 1 presents the measured temperature distribution for two different DPFs operated for 1,720 and 2,640 hours to simulate 300,000 and 420,000 miles of on-road use, respectively [6]. Although generally designed to avoid temperature excursions much above 600 °C or 700 °C, the data clearly indicates a small amount of time spent at temperatures in excess of 800 °C over the course of the DPF's total service life.

Exposure of ash to elevated temperatures has been shown to not only induce sintering and significant changes in ash morphology, but also to result in potential interactions with the filter substrate. Previous work investigating ash sintering and DPF interactions observed pinholes and the formation of glassy layers, due to ash wetting, on the DPF surface at temperature above 1,250 °C for cordierite and silicon carbide DPFs. Certain ash components, the presence of iron in particular, were also identified to promote ash-substrate interactions. The same study further defined an upper temperature limit of 1,050 °C for DPF operation, which was determined to be the temperature at which the investigated

ash compounds began to sinter and adhere to the filter walls [11].

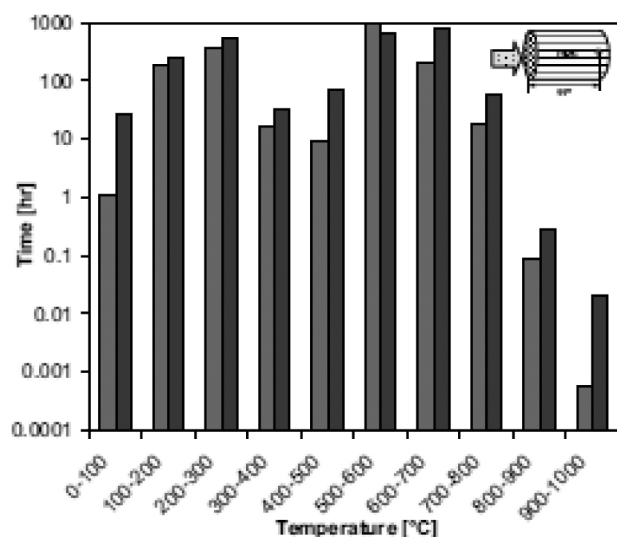


Figure 1. Cumulative temperature distributions measured for two DPFs over 1,720 and 2,640 hours. Adapted from [6].

More recent studies investigating the effect of less extreme temperatures on ash morphology and DPF performance reported larger ash particles formed in the DPF during high temperature regeneration (630 °C) and smaller ash particles formed during lower temperature (430 °C) regeneration. Furthermore, the authors suggested ash particle size to be affected not only by the regeneration temperature at which the initial ash particles were formed in the DPF, but also to be a function of the filter's subsequent thermal history [5]. Additional work investigating thermal aging effects on the integrity and performance of various diesel particulate filter materials at similar temperatures, between 550 °C and 650 °C, showed no significant effect on DPF performance, filtration efficiency, or regeneration characteristics [12]. In contrast to the extreme changes in ash properties at excessively high temperatures, these studies suggest some degree of influence of exhaust temperature on ash properties and particles size well within the “normal” operating temperature range of most diesel particulate filters.

Aside from high temperature excursions, rapid, transient fluctuations in exhaust flow rate are also possible. Figure 2 presents a figure from a 2009 Corning study showing measured exhaust flow and temperature variations over a typical highway drive cycle [13]. While generally considered a fairly steady drive cycle, the figure shows several large variations in exhaust flow conditions associated with hard accelerations and changing engine load over various road grades.

The data presented in Figure 2 indicates the potential for transient high temperature and high flow rate excursions during the DPF's useful life. Understanding how these events impact ash properties and their resulting effects on DPF

pressure drop and performance will provide useful information to improve DPF control strategies and on-board diagnostics capabilities. More importantly, if specific means of positively influencing ash properties can be identified, such as preferentially packing the ash at the back of the channels for example, or inducing other beneficial changes in the ash deposits, then intelligent control of engine and aftertreatment system operation may provide additional means for mitigating ash impacts on DPF performance.

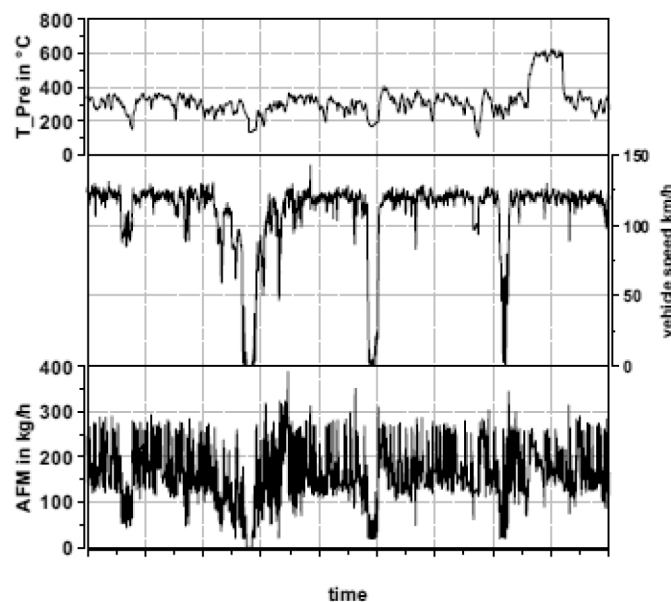


Figure 2. Measured exhaust flow and temperature profiles for a typical highway drive cycle. Adapted from [13].

EXPERIMENTAL SET-UP AND PROCEDURES

The experiments described in this study utilized ash-loaded DPF samples originating from particulate filters either loaded with ash in the laboratory or installed on vehicles operating in the field. The filter core samples were evaluated on a specially-designed flow bench under carefully-controlled conditions in order to study the effects of exhaust temperature and flow rate on ash properties and DPF performance. A number of advanced analytical techniques were also used to measure ash properties and explain the observed differences in filter performance.

SAMPLE HISTORY

All of the filters previously loaded with ash in the laboratory were subjected to an accelerated ash loading procedure developed in-house at MIT. Ash accumulation in the laboratory, under specific, controlled conditions, ensures the filter operating history is well-known and enables correlations between exhaust conditions and changes in filter performance to be readily ascertained. In contrast, while

field-aged DPFs provide arguably the most realistic specimens for analysis, data analysis and interpretation is oftentimes complicated due to lack of information related to the filter's operating history and conditions. The majority of the detailed sample analysis in this study is concentrated on the laboratory ash-loaded DPFs, however several field samples are also included to demonstrate similar phenomena observed in field ash.

The laboratory ash loading process has been described in detail previously [8, 14, 15], and is briefly summarized here. The accelerated ash loading and aftertreatment aging system was designed to load a conventional D5.66" (14.38 cm) × 6" (15.24 cm) DPF to 40 g/l of ash in slightly under 100 hours. This system is used in conjunction with a Cummins ISB 300 engine to enable the feed-gas particulate and ash characteristics to be carefully controlled and monitored. In its most basic form, the accelerated ash loading system produces soot with high ash content via the integration of a secondary diesel burner and oil injection system into the exhaust of the Cummins ISB. This system further allows for control and acceleration of specific oil consumption mechanisms, enabling the simulation of engine-out ash and PM as closely as possible. Previous studies showed similar exhaust ash emissions characteristics between the accelerated ash loading system and Cummins engine [14]. More recently, a comparison of DPFs aged in the laboratory using the accelerated ash loading system with filters subjected to 180,000 miles of on-road fleet testing using CJ-4 oils showed ash properties, distribution, and composition to be quite similar for both the laboratory and field-aged filters [8].

Three different lubricant formulations were used with the laboratory ash-loaded filters. The predominant lubricant, a commercial CJ-4 oil, served to define the baseline. Two other lubricants, each consisting of the same base oil, but containing only one additive component were also employed to control the composition of the ash in the DPF. All of the oils were formulated to the same nominal 1.0% sulfated ash level. A calcium-based detergent and a zinc-based anti-wear/anti-oxidant additive were selected for the two individual lubricant formulations, as calcium and zinc are among the most prevalent elements found in DPF ash [2, 7, 16, 17]. Table 1 provides additional details related to the lubricant formulations.

Table 1. Lubricant elemental composition. *Values supplied by manufacturer. All other values determined via ASTM D5185.

Lubricant	Ca	Mg	Zn	P	S	B	Mo
	ppm	ppm	ppm	ppm	ppm	ppm	ppm
Base Oil + Ca	2,928	5	<1	2	609	3	<1
Base Oil + ZDDP	<1	<1	2612	2,530	6,901	1	<1
Commercial CJ-4	1,388	355	1,226	985	3,200*	586	77

All of the cordierite diesel particulate filters loaded with ash in the laboratory measured D5.66" (14.38 cm) × 6" (15.24 cm) giving a total DPF volume of 2.47 liters with a

cell geometry of 200 cells/in.² (31 cells/cm²) and wall thickness of 0.012 in. (0.30mm). All of the filters were coated with a platinum catalyst and washcoat. The filters were loaded with ash using the accelerated ash loading system to simulate either active (periodic) or continuous regeneration. Additional details for each of the DPFs, including operating conditions and lubricants are listed in Table 2.

Table 2. DPF core sample ash levels and operating history for filters loaded in the laboratory.

DPF	Ash Level	Lubricant	Regeneration
Cordierite Catalyzed 200/12	12.5 g/L	CJ-4	Periodic
	42.0 g/L	CJ-4	Periodic
	33.0 g/L	CJ-4	Continuous
	28.0 g/L	ZDDP	Periodic
	29.0 g/L	Ca	Periodic

The ash levels listed in Table 2 were determined gravimetrically, from the hot filter, and correspond to the final filter ash levels prior to flow bench evaluations and post-mortem analysis. The specifics of the ash loading procedure used in this study were previously described in [7, 15], where additional DPF performance data is also presented. For the DPFs regenerated periodically, typical soot loading conditions were at a filter inlet temperature of 250 °C for one hour followed by filter regeneration at an inlet temperature of 620 °C for 15 minutes. The loading and regeneration cycles were repeated over the course of the ash loading protocol. On the other hand, the DPF subjected to continuous regeneration was operated with a filter inlet temperature slightly above 600 °C for the duration of the loading process.

The maximum specific DPF ash load achieved in this study was 42 g/l. This level of ash accumulation is estimated to be equivalent to roughly 240,000 miles (386,000 km) or 5,960 hours of DPF operation. This estimate is based on the following assumptions for a typical medium heavy-duty diesel engine: 15 g/hr average oil consumption [18], 40 mi/hr (64 km/hr) average speed, and a full sized particulate filter volume of 12 liters. In some cases different levels of ash loading were desired, with correspondingly different on-road equivalent distance/hours.

In addition to the DPFs loaded with ash in the laboratory, several cordierite field-aged DPFs from fleet test vehicles were investigated in this study as well. All of the field-aged filters were operated with engines using commercial CJ-4 oils for at least 150,000 miles, on OEM applications (no retrofit filters). While the operating history, lubricant formulations, and ash properties of the DPF samples loaded with ash in the laboratory are well-known, much less information was available for the field-aged filter samples. Unless specifically indicated as field samples, all of the references to DPF samples in the experimental results of this work are to laboratory ash-loaded DPFs.

TEST PROCEDURES

The DPF samples used in this study consisted of core samples removed from the field- and laboratory-aged DPFs described in the previous sections. Flow bench testing utilized cylindrical cores 0.875" in diameter (2.22 cm) $\times$ 6" (15.24 cm) long. The small cylindrical cores were wrapped, using an intumescent mat mount, and canned in stainless steel sample holders. Use of the sample holder facilitates installation of the core samples on the flow bench for pressure drop measurements and performance characterization. Figure 3 presents a schematic of the flow bench used in this work, which was based on an existing catalyst flow reactor. All of the flow bench testing in this study utilized dry, laboratory air, at ambient conditions.

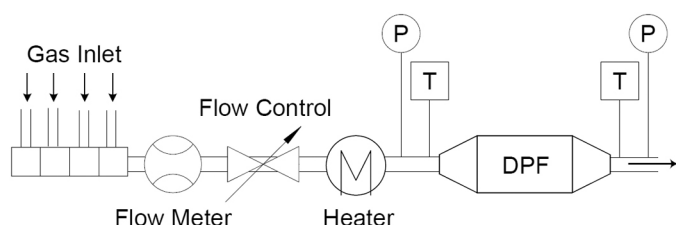


Figure 3. DPF core sample flow bench setup and test configuration.

Prior to evaluation on the flow bench, all of the DPF core samples were heated, using a laboratory furnace, to 650 °C for 1 hour to ensure that only ash, and no soot, remained in the filter samples. In order to evaluate the impact of short-duration high temperature and flow exposure on ash properties and DPF pressure drop, the following basic test procedure was utilized:

1. Heat filter core in furnace to specified temperature and hold for 30 minutes.
2. Weigh filter core hot; then cool to room temperature.
3. Measure pressure drop on flow bench over range of flow conditions.
4. Repeat steps 1-3 over specified temperature range.

The baseline condition in this study was defined as the DPF pressure drop and flow characteristics following heating to 650 °C. The process, outlined above, was then repeated, with each iteration exposing the sample to higher temperatures in increments of 50 °C or 100 °C until a maximum temperature of 1,100 °C was reached. In addition to the ash-loaded samples, several control filters consisting of the same cordierite substrate, but no ash, were also evaluated. Select samples, specifically the control and CJ-4 laboratory samples were also tested in triplicate to confirm the results.

A similar test procedure was repeated for the field-aged DPF core samples. Unlike the laboratory DPFs, the field DPFs were generally much larger, with a length of 10" (25.4 cm) to 12" (30.5 cm) making evaluation of cylindrical core

samples on the flow bench impractical. As such, rectangular core samples, generally 1" (2.54 cm) cubes were removed from these filters and subjected only to the heating studies (no flow bench testing).

All filter samples, both lab and field, were subjected to a detailed analysis to characterize ash distribution, packing, morphology, and composition, following heating and/or flow testing at each temperature interval. A number of advanced analytical techniques including scanning electron microscopy (SEM) to characterize ash particle size and morphology, energy dispersive x-ray (EDX) for ash compositional analysis, and x-ray diffraction (XRD) for quantitative ash compound identification were the predominant techniques applied in this work. Ash sintering temperatures and associated volume reduction was also quantified via dilatometry, and SEM image analysis utilized ImageJ for digital image processing, available from the National Institute of Health (NIH), for ash particle size and porosity measurements. Additional details describing the basic sample preparation and analysis procedures are available in [7].

EXPERIMENTAL RESULTS

The cumulative results of this work indicate transient (short-duration) exposure of existing ash deposits to high flow rate excursions have only a small impact on ash packing or distribution, as measured by a change in filter pressure drop. On the other hand, similar transient high temperature events can significantly affect the ash packing characteristics, resulting in an equally significant change in filter pressure drop. The following sections present the measurements illustrating the observed changes in filter performance, as well as the characterization of the ash properties necessary to provide a fundamental basis for interpreting these results.

EXHAUST FLOW

The impact of transient high-flow exposure on ash packing and distribution was evaluated on the flow bench following the test procedure outlined in Figure 4. Initially, DPF core sample pressure drop was measured as a function of flow rate over a space velocity range from 0 - 40,000 hr⁻¹, to establish the baseline DPF pressure drop (curve 1 in Figure 4). Following the baseline case, flow was gradually increased to a maximum of 200,000 hr⁻¹, much greater than would normally be achieved on the vehicle. A second set of flow data was then collected, corresponding to curve 2 in Figure 4. Comparison of the change in pressure drop between measurement points 1 and 2 in Figure 4 provides an indication of how DPF exposure to gradually higher flow rates would be expected to impact ash packing and/or redistribution, as manifested by a difference in pressure drop. Following the repeat of the baseline measurement, the flow through the DPF was pulsed at a maximum rate of 200,000 hr⁻¹, and a third baseline measurement conducted, denoted curve 3 in Figure 4. This simulated pulsed high flow test provides an extreme case of transient spikes in exhaust flow.

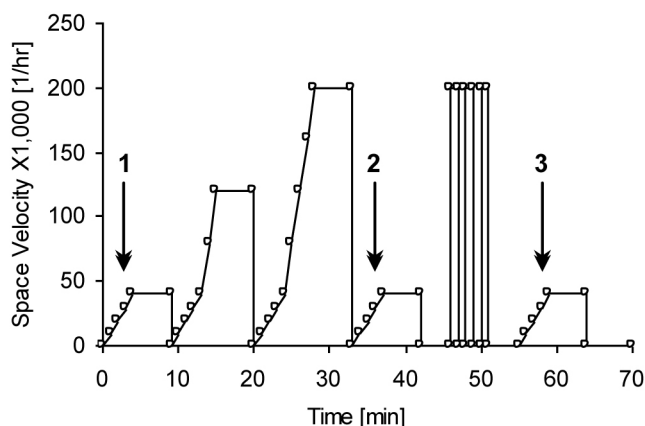


Figure 4. DFP core sample flow bench test procedure.

Figure 5 presents the relative change in pressure drop for a number of laboratory ash-loaded samples following exposure to both gradual and pulsed high flow conditions (i.e. the difference in pressure drop between curves 1 and 3 in Figure 4). All of the filter samples evaluated on the flow bench in Figure 5 were previously heated to 650 °C for 1 hour to ensure complete oxidation of any residual soot. The results indicate that even exposure to extreme, transient high flow conditions exerts little impact on ash packing or distribution, as measured by a change in filter pressure drop. For nearly all of the cases tested, pressure drop variation was within $\pm 5\%$, with most of the samples exhibiting a change in pressure drop of less than 2.5%, close to the variability of the measurement. It thus appears that short-term exposure to flow rates up to 200,000 hr^{-1} , several times higher than typical values achieved on the vehicle, does little to redistribute or pack the ash, once an existing ash layer and/or channel end-plug has been formed. These results were further confirmed in subsequent flow bench tests following elevated temperature exposure (see Test Procedures), which showed little change in DPF pressure drop due to high flow excursions.

While most of the observed pressure drop variation in Figure 5 is considered insignificant, it is interesting to note an apparent correlation between the initial ash layer distribution and measured change in pressure drop. Figures 6 and 7 present the measured ash distribution profiles for the samples evaluated in Figure 5. The sample exhibiting the largest reduction in pressure drop (-5.3% for the continuously regenerated CJ-4 sample with 33 g/L ash) also exhibited the thickest ash layer along the channel walls, nearly twice that of the other samples. Similarly, both the Ca- and Zn- ash loaded samples exhibited a somewhat thicker ash layer than the other periodically regenerated CJ-4 samples, and both the Ca- and Zn- samples also showed a slight reduction in pressure drop as well. On the other hand, the periodically regenerated CJ-4 samples with relatively thin wall ash layers showed a slight increase in pressure drop following high flow exposure.

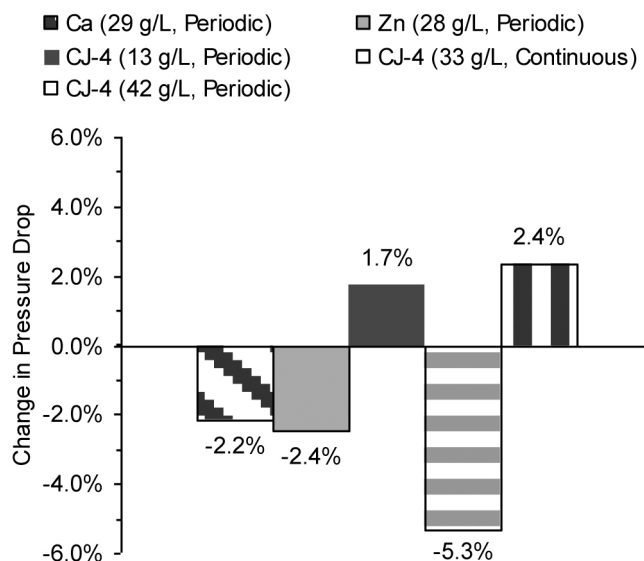


Figure 5. Change in ash-loaded DPF pressure drop before and after high flow exposure.

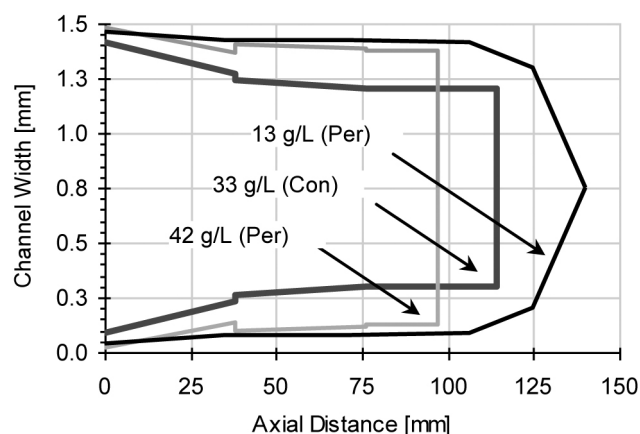


Figure 6. Measured ash distribution profiles for DPFs loaded with ash from commercial CJ-4 oil and subjected to periodic (Per) or Continuous (Con) regeneration, prior to high flow exposure. Adapted from [19].

Comparison of the measured pressure drop response in Figure 5 with the initial ash distribution profiles in Figures 6 and 7, provides some indication that a small amount of ash may be sheared from the wall layer and transported to the plug at the back of the DPF following high flow exposure, for filters with relatively thick ash layers along the channel walls. While consistent with the prevalent theories regarding ash critical shear stress, the effect due to short-duration transient spikes in exhaust flow rate appears small. It should be noted, however, that these observations pertain specifically to short-duration events, and not processes occurring over much longer timescales such as the tens or even hundreds of thousand of miles during which the ash deposits build up in the DPF.