

Analysis of High Mileage Gasoline Exhaust Particle Filters

Christine K. Lambert, Mira Bumbaroska, Douglas Dobson,
Jon Hargas, James Pakko, and Paul Tennison
Ford Motor Company

ABSTRACT

The purpose of this work was to examine gasoline particle filters (GPFs) at high mileages. Soot levels for gasoline direct injection (GDI) engines are much lower than diesel engines; however, noncombustible material (ash) can cause increased backpressure, reduced power, and lower fuel economy. In this study, a post mortem was completed of two GPFs, one at 130,000 mi and the other at 150,000 mi, from two production 3.5L turbocharged GDI vehicles. The GPFs were ceramic wall-flow filters containing three-way catalytic washcoat and located downstream of conventional three-way catalysts. The oil consumption was measured to be approaching 23,000 mpqt for one vehicle and 30,000 mpqt for the other. The ash contained Ca, P, Zn, S, Fe, and catalytic washcoat. Approximately 50 wt% of the collected ash was non-lubricant derived. The filter capture efficiency of lubricant-derived ash was about 50% and the non-lubricant metal (mostly Fe) deposition rate was 0.9 to 1.2 g per 10,000 mi. Between 58 and 61 g of ash in total was collected, or 23 to 24 g/L. About 60-70% of the ash was on the filter walls and the rest was in the plug region. Average wall ash thicknesses were 12.4 μm . Plug ash density was 0.7 g/cm³. Wall ash density was over twice as high at 1.6 g/cm³ and reduced the filter wall permeability by about 75%. Although the exhaust gas backpressure did increase, there was no significant decrease in fuel economy as a result of the ash accumulation.

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INTRODUCTION

Direct injected gasoline (GDI) engines can create significant amounts of soot under certain operating conditions such as cold start and high load. California LEVIII and U.S. Tier 3 will require a tailpipe particulate matter (PM) maximum of 3 mg/mi, down from the current 10 mg/mi. Phase-in for the lower PM will occur in the 2017-2021 model years. In Europe, Stage 6 particle number (PN) standards for DI vehicles are currently at 6×10^{12} particles/km and become 90% lower at 6×10^{11} particles/km in September 2017. New tests like real world driving emissions (RDE) will cover more driving conditions than on the older NEDC (New European Driving Cycle). Wall flow ceramic filters are one well known way to remove exhaust PM and PN. The filter may be used with or without a catalytic washcoat and is typically located downstream of a conventional three-way catalyst. In a relatively clean condition relative to DPFs, the GPF can usually offer filtration efficiencies on the order of 70-80% [1]. This is due to the much lower soot output of GDI engines compared to diesel engines. GDI soot emissions primarily occur during cold start [2].

While carbonaceous soot may be collected and later oxidized via passive or active processes, non-combustible material, known commonly as ash, will be collected in any type of exhaust filter. It is important to understand what ash is and where it comes from, since ash collection in the filter will contribute to increased exhaust backpressure. During the last fifteen years, there was a great deal of research and development pertaining to ash deposition, origination and the operating conditions under which

ash is created on diesel engines [3]. It is well known that ash can originate from additives in the lubrication oil and to some degree the fuel, and when these elements or compounds go through the combustion process, ash components are left behind on the emission control components. The degree of ash deposition in an exhaust gas filter depends on several physical parameters such as porosity, soot accumulation, and temperature. Final ash location appears to be closely related to soot accumulation and oxidation cycles [4]. Sizing for full useful life (FUL) ash deposition is important for gas and diesel applications to minimize negative impacts and optimize the system. This study aims to understand ash accumulation in gasoline particle filters (GPFs) over significant mileage.

EXPERIMENTAL

Vehicles

The two vehicles used in this study, a 2010 Ford Taurus SHO (Vehicle 1) and a 2010 Ford Flex (Vehicle 2), were equipped with 3.5L turbocharged GDI engines. Both vehicles used production engine calibrations. No special calibration or controls were implemented for the GPFs. It was thought that fuel cuts during decelerations would provide enough oxygen for soot oxidation. Additionally there was catalyst washcoat containing oxygen storage material in the filters. Vehicle 1 mileage was 130,000 miles at the end of the testing while Vehicle 2 completed 150,000 miles.

System Layout

Both vehicles used a similar production exhaust system consisting of two close-coupled production three way catalysts (TWCs) followed by an underbody TWC. The close-coupled, round TWCs were 4.66 x 2.93" (50 in³) and contained Pd and Rh. The oval underbody TWC was removed and replaced with a round GPF that contained TWC washcoat. The filter substrate was a cordierite wall-flow substrate with 1.0 g/in³ of three-way washcoat containing Pd and Rh. The porosity of the filter was approximately 65% with a mean pore size of 22 μ m. The GPF had a diameter of 5.66", a length of 6", 2.5 L total geometric volume, cell density of 300 cells per in², and 305 μ m (12 mil) walls. The general exhaust layout for both vehicles was as shown in Figure 1. Emission probes were located at the midbed position between the TWCs and the GPF, and tailpipe (post GPF). Pressure was measured upstream of the GPF.

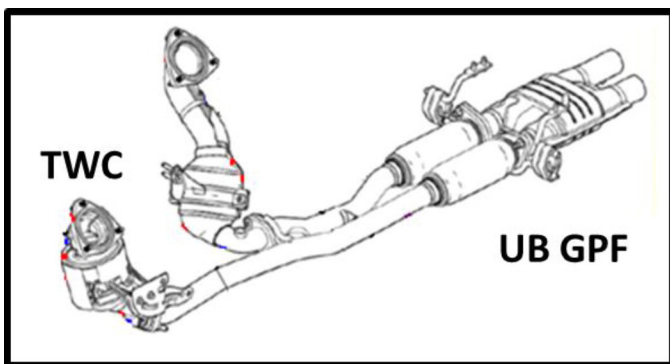


Figure 1. System layout with GPF inserted into the underbody (UB) position.

Aging Cycle

The U.S. EPA Standard Road Cycle (SRC) was used for mileage accumulation (Figure 2) using automated chassis roll dynamometers. The peak speed was 80 mph, average speed was 46.3 mph, and maximum cruise speed was 75 mph with light to hard accelerations. There were no wide-open-throttle accelerations. The SRC contained 24 decelerations. The deceleration rates range from coast-down (no brake force applied) to moderate.

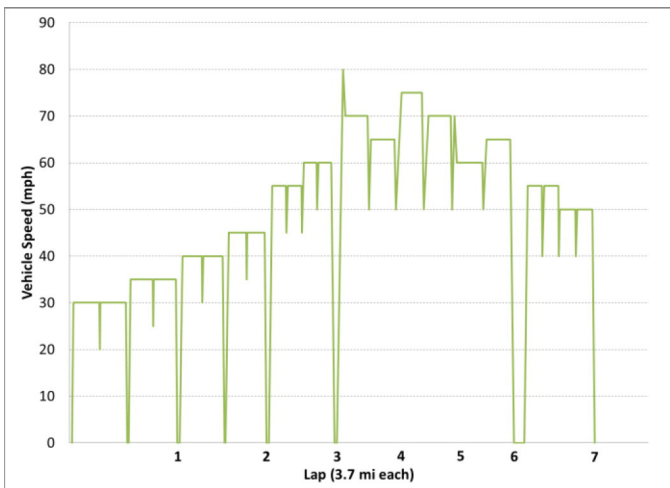


Figure 2. Vehicle speed on the EPA SRC (Standard Road Cycle).

Oil Consumption and Oil Quality

The oil consumption differed between the two vehicles used in this study. Oil changes were done per Ford standards every 10,000 mi. The measured oil consumption, as shown in Figure 3, between the two vehicles was within 20% of each other at the end of life. Vehicle 1 consumed oil at a higher rate of approximately 23,000 miles per quart (mpqt) vs nearly 30,000 mpqt for Vehicle 2.

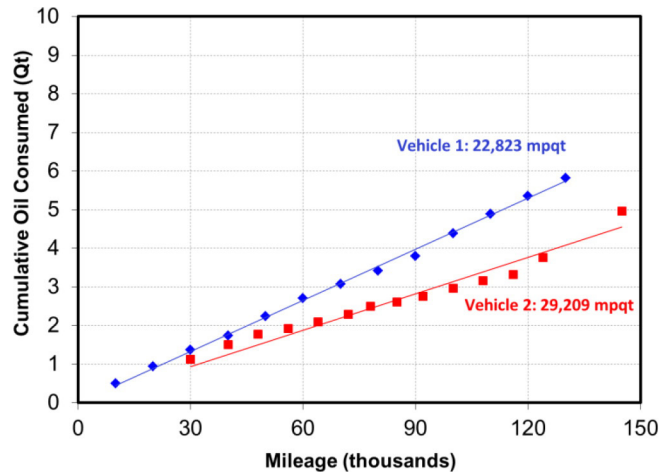


Figure 3. Summary of oil consumption for the two vehicles equipped with 3.5L GTDI engines and GPFs.

The oil type was general purpose 5W30 Motorcraft, with GF-5 oil used in Vehicle 1 and GF-4 used in Vehicle 2. The standards for P and S in these oils are the same (Phosphorus Content, ASTM D 4951 = 0.08% (mass) maximum; Sulfur Content, ASTM D 4951 or D 2622 for SAE 5W multigrades = 0.5% (mass) maximum). Ash content in the fuel (E10 gasoline) was negligible. The elemental content assumed for GF-4 and GF-5 oils and the aged GF-5 oil are shown in Table 1.

Table 1. Elemental analysis of 5W30 GF-4 and GF-5 engine oils. Elements were analyzed by ICP-AES per ASTM D5185.

Element	Fresh GF-4 (mg/kg)	Fresh GF-5 (mg/kg)	GF-5 at 10,000 mi (mg/kg)
P	778	756	683
S	3400	2300	Not provided
B	345	10	10
Ca	2320	1760	1991
Mo	42	10	73
Zn	939	835	765
Na	0	421	4
Mg	9	6	9
K	4	0	0
Si	0	0	15

Sample Analysis

High mileage GPFs were scanned on a computed tomography (CT) scanning tool at the Non-Destructive Evaluation Laboratory (NDEL) at Ford Motor Company. Interim scans, not included here, at lower mileages were taken of the filters to fully document the aging process. Five full length one inch diameter cores were extracted from

the substrate. Pressure drop across each core was measured at room temperature over a range of flow rates so that estimations could be made for wall and ash layer permeability. Sample nomenclature was based on filter location (Figure 4).

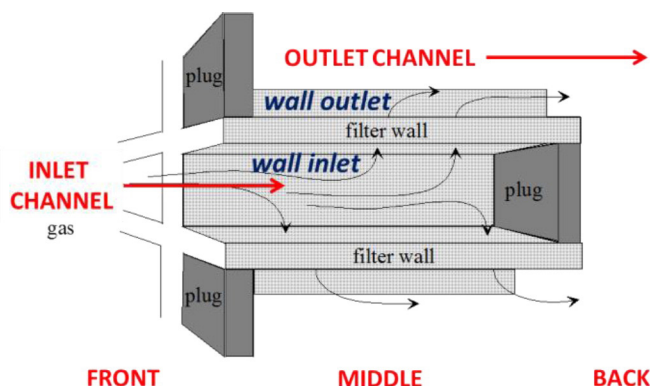


Figure 4. Nomenclature used for sample locations within the GPF.

X-ray fluorescence (XRF) analysis was done to understand the elemental composition of the collected ash. Specimens were prepared by first grinding each core sample in a ceramic-lined grinding dish to obtain a fine, homogenous powder. A 5-gram sample of each powder was then mixed with 0.5 grams of Chemplex® X-Ray Mix and compacted into a pellet using a hydraulic press. X-ray intensity data were collected using a Panalytical® PW2400 sequential wavelength-dispersive XRF spectrometer with a chromium target x-ray tube. Element concentrations were calculated from the x-ray data using UniQuant® (version 5), a fundamental parameters quantitative analysis program.

Electron probe micro analysis (EPMA) was performed to determine ash composition and location. Selected pieces of cores were vacuum impregnated in Epofix epoxy. The mounted samples were ground and polished down to a final grit size of 1 μm , and then carbon coated. Wavelength dispersive x-ray mapping was obtained in a JEOL 8200 electron probe analyzer. Semi-quantitative maps displayed in log wt% have the advantage of displaying elemental concentrations from trace (<1%), to minor (<10%), through to major (>10%) as proposed by Newbury and Bright [5].

Conditions used for mapping were an accelerating voltage of 15keV, a beam current of 30 nA, and total dwell time of 300 ms from multiple accumulations. ImageJ [6] was used to process the maps and one background image for each map. Each background image was "blurred" using a maximum filter with a radius of 3 pixels before subtracting it from the elemental map. The subtracted maps were transformed from x-ray counts to log wt% using the relation $C_{\text{unknown}}/I_{\text{unknown}} = C_{\text{std}}/I_{\text{std}}$. C is the concentration in weight percent of the element in either the unknown or an analytical standard, I is the net x-ray intensity in counts/sec/ μA for either the unknown or the standard.

An estimation of the ash layer thickness on the inlet wall surfaces was made from microscopy images taken of samples from every 1/2 inch along a 1" diameter filter core using a Nikon microphot-FXA

with SPOT 5.1 software. As a check, SEM images were used to compare the measurements against another sample/core from the same brick.

To estimate the amount of ash in the brick, cores from each filter were cut into a series of 1" in diameter by 1/2" long sections. Ash was removed using multiple steps. First, samples were cut and loose ash particles were removed using compressed air. Next, samples were placed into an ultra-sonication bath for one hour to remove any soluble components. Then samples were dried to remove any excess water and weighed. To remove ash particles on the filter walls, a combination of Tween® 80 (Croda International PLC) and acetic acid was used. Final sample weights were taken to determine total weight of ash in each section.

RESULTS AND DISCUSSION

Backpressure and Fuel Economy

There was no significant decrease in fuel economy as a result of the mileage; however, the exhaust backpressure did steadily increase. A point at 80 mph was used to assess exhaust backpressure in front of the GPF at mileage intervals; the backpressure increased from 3 kPa to almost 7 kPa after Vehicle 1 accumulated 130,000 mi (Figure 5). There was an event at approximately 80,000 mi that caused a brief increase in backpressure. The increase was due to an accumulation of soot in addition to ash, due to a sensor failure that stopped fuel cuts during decelerations, depriving the filter from oxygen. The soot subsequently burned off when fuel cuts were re-enabled, and backpressure dropped. No increase in soot emissions were noted afterwards, and the backpressure returned to the same increasing slope as before.

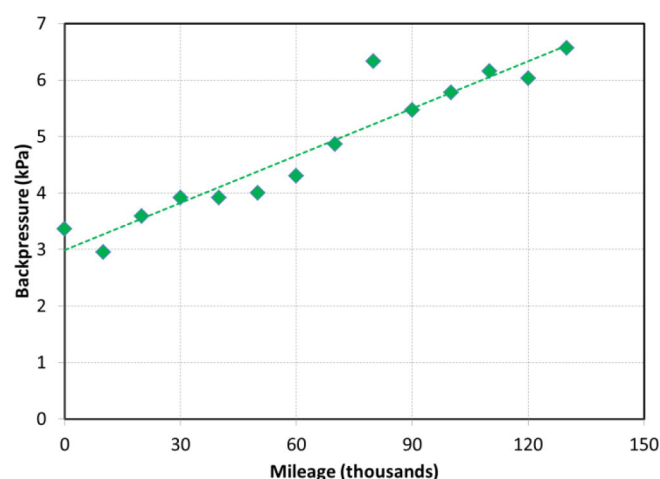


Figure 5. Increase in steady-state exhaust backpressure with mileage on Vehicle 1 at 80 mph.

Nondestructive Analysis

A few of the full mileage CT scans for the Vehicle 1 GPF at 130,000 mi are presented in Figure 6, and the Vehicle 2 GPF scans at 150,000 mi are in Figure 7. For the Vehicle 1 filter, some ash was noticeable at the back of the inlet channels, and the distribution was not uniform. A small void was located at the back