Fundamental Processes Controlling Ash Accumulation in Diesel Particulate Filters and Impacts on DPF Performance

2012 DOE Crosscut Workshop on Lean Emissions Reduction Simulation

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Ash Accumulation Reduces DPF Life and Engine Efficiency

Ash Function Of:
- Incoming ash/soot particles
- Lubricant composition
- Exhaust conditions/Regeneration
- DPF design parameters
- DPF operating history

More Ash than Soot in DPF!
Ash Accumulation and Deposit Formation Differs from PM!

\[ \tau_{cr} \]

~ 100 nm

~ 1 \( \mu \)m
Program Approach & Consortium Activities

“Holistic approach considering lubricant chemistry, engine operation, and aftertreatment design through combination of focused experiments and theoretical models.”

Enhance fundamental understanding of key parameters controlling ash properties and impact on aftertreatment performance.
Experimental Facilities

DPF Bench Reactors

Cummins ISB 300
- Variable geometry turbocharger
- Cooled EGR
- Common rail fuel injection
- Fully electronically controlled
- Gaseous and PM emissions measurement systems

Accelerated Ash Loading
Center for Materials Science (CMSE)

• Materials Analysis
  • FTIR, Raman, XPS, Optical Microscopes

• Thermal Analysis
  • TGA, ICP-OES

• Electron Microscopy
  • SEM – EDX, TEM, FIB

• X-Ray Diffraction

Ash Exposed to Elevated Temperatures

Extensive and increasing use as part of DPF post-mortem analysis and ash characterization
Key Parameters Controlling Ash Deposits and DPF Impacts

**Engine-Out Ash Emissions and Transport**
- *Form of ash in exhaust/feed gas entering DPF; ash trapping efficiency*

**Ash Deposit Accumulation and Build-Up in the DPF**
- *Agglomerate formation and ash mobility/distribution in DPF*

**Ash Impact on DPF Pressure Drop Response**
- *Ash composition and properties relevant to DPF performance*

**Sensitivity of DPF Design Parameters to Ash Accumulation**
- *Substrate materials, pore size & distribution, porosity*

**Role of Engine Control Strategies and Exhaust Conditions**
- *Temperature, flow, and feed gas conditions affecting ash deposits*

**Regeneration Processes**
- *Real-time optical studies of ash formation and mobility*

**Ash – Catalyst Interactions**
- *Chemical and physical interactions of ash and catalyst/washcoat*
Lubricant-Derived Ash Precursors Bound to PM

Lubricant-derived ash transported to DPF bound to carbonaceous PM
- Size of ash precursors of same order or smaller than PM agglomerates
- No lubricant-derived ash particles found separate from PM
- Cu peaks due to background from copper TEM grid
Nearly All Metallic Ash Components Trapped in DPF

1682 rpm, 25% load (Steady-State)

Measured elemental trapping efficiency in DPF

<table>
<thead>
<tr>
<th></th>
<th>Ca</th>
<th>Fe</th>
<th>Mg</th>
<th>P</th>
<th>S</th>
<th>Zn</th>
</tr>
</thead>
<tbody>
<tr>
<td>Trapping Efficiency</td>
<td>99.87%</td>
<td>92.43%</td>
<td>98.01%</td>
<td>85.09%</td>
<td>64.89%</td>
<td>99.67%</td>
</tr>
</tbody>
</table>

- Elemental emission rates determined from ICP analysis
- Post-DPF PM sampling 20 hours for sample size of 2-3 mg
Particle Mass Spectrometer for Ash Measurements

ASME ICEF2011-60100
Real-Time Measurement of Exhaust Ash Emissions (I)

ASME ICEF2011-60100

Isotopic Abundances

<table>
<thead>
<tr>
<th></th>
<th>Meas.</th>
<th>Known*</th>
</tr>
</thead>
<tbody>
<tr>
<td>B11</td>
<td>0.811</td>
<td>0.802</td>
</tr>
<tr>
<td>B10</td>
<td>0.188</td>
<td>0.198</td>
</tr>
</tbody>
</table>

Date and Time

5:30 PM 6:00 PM 6:30 PM 7:00 PM 7:30 PM 8:00 PM
10/11/10

Total SMPS Mass (ug/m³)

Oil Injection Rate (ml/min)

Inject_rate
SMPS Mass
Boron
Real-Time Measurement of Exhaust Ash Emissions (II)

ASME ICEF2011-60100

Date and Time

5:30 PM 10/11/10

6:00 PM

6:30 PM

7:00 PM

7:30 PM

8:00 PM

diameter (nm)

signal, Hz

Oil Injection Rate (ml/min)

Total SMPS Mass (ug/m3)

Hz [Zn64, Zn66, Zn67, Zn68]

Hz [Σ Zinc Signals]

Isotopic Abundances

Meas. Known

Zn64 0.458 0.486
Zn66 0.304 0.279
Zn67 0.042 0.041
Zn68 0.196 0.188


14x10^3

6000

5000

4000

3000

2000

1000

0

6

5

4

3

2

100

7

6

5

4

3

2

10

20

30

40

50

60

70

80

90

100

0

10

20

30

40

50

60

70

80

90

100

0

2

4

6

8

10

12

14x10^3
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Ash First Accumulates Along DPF Channel Walls

- Ash accumulation reduces DPF volume and affects pressure drop.

Diagram showing:
- 12.5 g/l Ash (70K mile equiv.)
- 42 g/l Ash (240K mile equiv.)

Images of channel walls at 57 mm and 133 mm show the effect of ash accumulation.
Initial Ash Deposition and Layer Formation

Ash layer build-up evident by changing $\Delta P$ profiles.

Benefit from ash layer formation

Ash layer not fully established until 10 g/L or ~50,000 miles
Application of Tracer Produces Stratified Ash Layers

ASH Thickness and Distribution

Channel Width [µm]

Axial Distance [mm]

Ash Plug

Centerline

Periphery

Ca Ash

Zn Ash

Mg Ash

DPF Substrate
Voids in Ash Plug – Opportunities to Improve Packing

Voids may explain low ash packing density...

Large voids throughout ash layer

Start of plug and ash layer

Ca

Zn

Mg
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Additive Chemistry Impact on Ash Properties

Lubricant matrix all formulated to 1% sulfated ash, except base oil.

<table>
<thead>
<tr>
<th>Lubricant</th>
<th>Ca (ppm)</th>
<th>Mg (ppm)</th>
<th>Zn (ppm)</th>
<th>P (ppm)</th>
<th>S (ppm)</th>
<th>B (ppm)</th>
<th>Mo (ppm)</th>
</tr>
</thead>
<tbody>
<tr>
<td>Base</td>
<td>&lt;1</td>
<td>&lt;1</td>
<td>&lt;1</td>
<td>8</td>
<td>60</td>
<td>1</td>
<td>&lt;1</td>
</tr>
<tr>
<td>Base + Ca</td>
<td>2,928</td>
<td>5</td>
<td>&lt;1</td>
<td>2</td>
<td>609</td>
<td>3</td>
<td>&lt;1</td>
</tr>
<tr>
<td>Base + Mg*</td>
<td>&lt;1</td>
<td>2,070</td>
<td>&lt;1</td>
<td>&lt;1</td>
<td>460</td>
<td>&lt;1</td>
<td>&lt;1</td>
</tr>
<tr>
<td>Base + ZDDP</td>
<td>&lt;1</td>
<td>&lt;1</td>
<td>2612</td>
<td>2,530</td>
<td>6,901</td>
<td>1</td>
<td>&lt;1</td>
</tr>
<tr>
<td>Base, Ca+ZDDP*</td>
<td>2480</td>
<td>&lt;1</td>
<td>1280</td>
<td>1,180</td>
<td>2,700</td>
<td>&lt;1</td>
<td>&lt;1</td>
</tr>
<tr>
<td>Base, Mg+ZDDP*</td>
<td>&lt;1</td>
<td>1730</td>
<td>1280</td>
<td>1,180</td>
<td>2,840</td>
<td>&lt;1</td>
<td>&lt;1</td>
</tr>
<tr>
<td>Commercial CJ-4</td>
<td>1,388</td>
<td>355</td>
<td>1,226</td>
<td>985</td>
<td>3,200*</td>
<td>586</td>
<td>77</td>
</tr>
</tbody>
</table>

Composition of ash directly related to lubricant additive chemistry.

<table>
<thead>
<tr>
<th>Major Ash Components</th>
<th>Density (g/cm³)</th>
<th>Melting Point (°C)</th>
<th>Description</th>
</tr>
</thead>
<tbody>
<tr>
<td>CaSO₄</td>
<td>2.96</td>
<td>1,460</td>
<td>Sinters/Decomposes ~1,250 °C</td>
</tr>
<tr>
<td>CaZn₂(PO₄)₂</td>
<td>3.65</td>
<td></td>
<td></td>
</tr>
<tr>
<td>Zn₃(P₂O₇)</td>
<td>3.75</td>
<td></td>
<td></td>
</tr>
<tr>
<td>Zn₅(PO₄)₂</td>
<td>4.00</td>
<td>900</td>
<td>Sintering begins ~ 800 °C</td>
</tr>
<tr>
<td>Zn₂Mg(PO₄)₂</td>
<td>3.60</td>
<td></td>
<td></td>
</tr>
<tr>
<td>MgO</td>
<td>3.58</td>
<td>2,832</td>
<td></td>
</tr>
<tr>
<td>MgSO₄</td>
<td>2.66</td>
<td>1,124</td>
<td>Decomposition 900-1,100 °C</td>
</tr>
</tbody>
</table>
Ca Ash Shows 2X Increase in $\Delta P$ Over Zn & Mg Ash

Flow Bench @ 25 °C, Space Velocity: 20,000 hr$^{-1}$

- Lubricant additive chemistry affects ash properties and pressure drop
- Ca-based ash shows much larger effect on pressure drop than Zn ash

* Assumes: 15 g/hr avg. oil consumption, avg. speed of 40 mph, and full size DPF of 12 L volume
Ash Chemistry Impacts Ash Properties and DPF ΔP

Ash Distribution Profiles

Pressure Drop [kPa] vs. Cumulative PM Load [g/l]

Ca - Ash

Cordierite 200/12

Ca + ZDDP 25 g/L
Ca 29 g/L
No Ash
ZDDP 28 g/L
Mg 28 g/L
Mg + ZDDP 23 g/L
CJ-4 33 g/L
Ca 29 g/L
Ca + ZDDP 25 g/L

Differences in DPF ΔP due to ash properties (ρ, ε, D_p)

ASME ICES2012-81237
Detailed Understanding of Ash Properties Required

\[ \Delta P_{\text{Wall / Ash / Soot}} = \left( \frac{\mu}{K_p} \right) \cdot V_w \cdot W \]

\[ K = f(\varepsilon, \bar{D}_p) \]

\[ \varepsilon = 1 - \frac{\rho_{\text{Packing}}}{\rho_{\text{Theoretical}}} \]

Ash Properties
- Provide critical information to explain fundamental differences in \( \Delta P \)
- Complex mixture of metal oxides, sulfates, phosphates
- Characterization of particle morphology, physical, chemical properties challenge

New Techniques and Diagnostics
- C. Kamp Presentation (12/2010)
CJ-4 Ash Composition and Porosity

<table>
<thead>
<tr>
<th>Ash</th>
<th>Wall Density [g/l]</th>
<th>Theoretical Density [g/cm³]</th>
<th>Ash Porosity [%]</th>
</tr>
</thead>
<tbody>
<tr>
<td>CaSO₄</td>
<td>42.0</td>
<td>0.30</td>
<td>91.1</td>
</tr>
<tr>
<td>Zn₂Mg(PO₄)₂</td>
<td>12.5</td>
<td>0.18</td>
<td>94.6</td>
</tr>
</tbody>
</table>

\[ \varepsilon = 1 - \frac{\rho_{\text{Packing}}}{\rho_{\text{Theoretical}}} \]
Ash – PM Layer Interface Clearly Defined

SAE 2012-01-0836

Focused Ion Beam (FIB) Milling Coupled with SEM

Unlike PM depth filtration in DPF surface pores, very little soot penetrates into ash layer.
Focused Ion Beam (FIB) Milling Coupled with SEM

Particle most likely formed from sintering/agglomeration of ash precursors.
Ash Particles and Agglomerates Porous Shells!

- Ash agglomerates consist of porous particles
- Consistent with low packing density and high porosity measurements

Images: C. Kamp
Ash Accumulation Also Influences Soot Properties

- **RPS** equation:
  \[ RPS = \left( \frac{\partial \Delta P}{\partial \text{PM}} \right)_{\text{Ash,i}} \div \left( \frac{\partial \Delta P}{\partial \text{PM}} \right)_{\text{Clean,i}} \]

- **Adjust specific soot load to account for actual available trap volume**

- **Ash deposits displace soot in DPF – higher local soot loads**
Variation in PM Layer Properties with DPF Flow Well-Known

$$K_{soot} x 10^{-14} \text{ [m]}$$

$$\text{Source: SAE 2002-01-1015}$$

$$Pe = \frac{U_w \cdot d_{primary}}{D}$$

$$Pe = \frac{\text{Inertia}}{\text{Diffusion}}$$

Variation of Soot Properties Due to Ash Deposits

Soot Properties
- Estimated from empirical Pe number correlations for constant flow rate
- Low space velocity conditions most strongly affected by ash loading

Soot deposited at flows > 75K 1/hr insensitive to ash loading
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Sensitivity of DPF Design Parameters to Ash

<table>
<thead>
<tr>
<th>Matrix</th>
<th>Porosity</th>
<th>Mean Pore Size</th>
</tr>
</thead>
<tbody>
<tr>
<td>1\textsuperscript{st} Trial</td>
<td>Low High</td>
<td>Low</td>
</tr>
<tr>
<td>2\textsuperscript{nd} Trial</td>
<td>High</td>
<td>Low High</td>
</tr>
<tr>
<td>3\textsuperscript{rd} Trial</td>
<td>Moderate</td>
<td>Low</td>
</tr>
</tbody>
</table>

Additional DPF Parameters
- Filter/substrate materials
- DPF coatings and catalysts
- Filter geometry and cell configuration

Multi-Cartridge Filter Holder

12 DPF Segments

Ash Loading
DPFs Experience Even Ash Loading and Temperatures

Average Ash Deposition after 60 hrs of operation: 20 g/L

- Ash evenly distributed in core samples
- Little ∆P variation between duplicate samples

Pressure Drop Variation

Inlet Temperatures

Inlet Temperature (before the cone)

610 °C (±25)

(Space Velocity range: 6,500 – 55,000 [1/hr])
Similar ΔP Response to PM Accumulation for All DPFs

* Indicates pore and porosity size class

DPF Pressure Drop Response 0 g/L Ash

- SiC (40%, 15*µm) with 0 g/L ash
- Cd (50%, 15*µm) with 0 g/L ash
- SiC (50%, 15*µm) with 0 g/L ash
- SiC (60%, 15*µm) with 0 g/L ash
- SiC (60%, 25*µm) with 0 g/L ash
Sensitivity of DPF Porosity to Ash Accumulation Varies

- Sensitivity of $\Delta P$ to ash accumulation increases with decreasing DPF porosity at low filter ash levels.
- At high ash loads, ash dominates $\Delta P$, which is insensitive to initial DPF porosity of filter, over range tested.
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Exhaust Conditions Are Continually Changing

DPF Temperature Distribution
(300-420K Miles, HD Diesel)

- Potential for short excursions above 700 °C over DPF operating history
- Exhaust flow rates also vary considerably, even over highway drive cycle
Exhaust Temperature Significantly Affects Ash Volume

■ Large decrease in ash volume for temperatures over 700 °C
  □ Reduction in ash weight over temperature ranges less than 10%
  □ Typical ash porosities 85% - 95% means large potential to reduce volume
Elevated Temperatures Exert Large Effect on Ash Packing

High Flow Exposure to 200,000 hr⁻¹

- Ca (28 g/L, Per) 1.7%
- Zn (28 g/L, Per) 2.4%
- CJ-4 (13 g/L, Per) -2.2% -2.4%
- CJ-4 (33 g/L, Con) -5.3%
- CJ-4 (42 g/L, Per) 3.8%

High Temperature Exposure

- Control (No Ash)
- 28 g/L Ca Ash
- 28 g/L ZDDP Ash
- 13 g/L CJ-4 Ash
- 42 g/L CJ-4 Ash

Ash Volume Reduction Fast

DPF core heated to 880 °C in 5 min, then quenched.
Large Reduction in Ash Volume at Elevated Temperatures

SAE 2012-01-1093

- 650 °C
- 700 °C
- 800 °C
- 900 °C
- 1,000 °C
- 1,100 °C

ZDDP
Calcium
CJ-4 (P)
CJ-4 (C)

28 g/L
28 g/L
42 g/L
33 g/L
High Temperatures Cause Ash Layer Cracking/Shrinking

- 42 g/L CJ-4 Ash (Periodic)
- 33 g/L CJ-4 Ash (Continuous)

650 °C
650 °C
650 °C

700 °C
700 °C
700 °C

800 °C
800 °C
800 °C

900 °C
900 °C
900 °C

1,000 °C
1,000 °C
1,000 °C

Despite large volume reduction, ash weigh change < 7%
### Similar Behavior in Lab/Field Ash May Be Due to Chemistry

<table>
<thead>
<tr>
<th></th>
<th>650 °C</th>
<th>700 °C</th>
<th>800 °C</th>
<th>900 °C</th>
<th>1,000 °C</th>
<th>1,100 °C</th>
</tr>
</thead>
<tbody>
<tr>
<td>Lab Calcium</td>
<td><img src="calcium_650%C2%B0C.png" alt="Image" /></td>
<td><img src="calcium_700%C2%B0C.png" alt="Image" /></td>
<td><img src="calcium_800%C2%B0C.png" alt="Image" /></td>
<td><img src="calcium_900%C2%B0C.png" alt="Image" /></td>
<td><img src="calcium_1000%C2%B0C.png" alt="Image" /></td>
<td><img src="calcium_1100%C2%B0C.png" alt="Image" /></td>
</tr>
<tr>
<td>Field D</td>
<td><img src="field_d_650%C2%B0C.png" alt="Image" /></td>
<td><img src="field_d_700%C2%B0C.png" alt="Image" /></td>
<td><img src="field_d_800%C2%B0C.png" alt="Image" /></td>
<td><img src="field_d_900%C2%B0C.png" alt="Image" /></td>
<td><img src="field_d_1000%C2%B0C.png" alt="Image" /></td>
<td><img src="field_d_1100%C2%B0C.png" alt="Image" /></td>
</tr>
<tr>
<td>Lab CJ-4 (P)</td>
<td><img src="cj-4_p_650%C2%B0C.png" alt="Image" /></td>
<td><img src="cj-4_p_700%C2%B0C.png" alt="Image" /></td>
<td><img src="cj-4_p_800%C2%B0C.png" alt="Image" /></td>
<td><img src="cj-4_p_900%C2%B0C.png" alt="Image" /></td>
<td><img src="cj-4_p_1000%C2%B0C.png" alt="Image" /></td>
<td><img src="cj-4_p_1100%C2%B0C.png" alt="Image" /></td>
</tr>
<tr>
<td>Field A</td>
<td><img src="field_a_650%C2%B0C.png" alt="Image" /></td>
<td><img src="field_a_700%C2%B0C.png" alt="Image" /></td>
<td><img src="field_a_800%C2%B0C.png" alt="Image" /></td>
<td><img src="field_a_900%C2%B0C.png" alt="Image" /></td>
<td><img src="field_a_1000%C2%B0C.png" alt="Image" /></td>
<td><img src="field_a_1100%C2%B0C.png" alt="Image" /></td>
</tr>
</tbody>
</table>
Equilibrium Crystal Structures formed at High Temperatures

\[ \min(\int \gamma_S dA_S) \]

Chemical and Physical Changes in Ash at High Temps.

Ash Composition Changes Irreversibly
Applications to Understand Field DPF History

“Normal” Field Ash

Field Ash Exposed to Thermal Event

Prior High Temperature Exposure

Ash Necking & Agglomeration

Wetting on Substrate

SAE 2012-01-1093
Conceptual Description of Temperature Effects on Ash

Possible Effect of Temperature on Ash Deposits

Ash Deposits in Typical DPF

DPF Pores Exposed!

Ash Deposits After High Temp.

Competing Processes Require Detailed Understanding

- Elevated temperatures result in significant ash volume reduction
- Location of ash deposits (channel vs. wall) plays a large role in impact on $\Delta P$
- Deterioration of ash cake layer could result in increased $\Delta P$ with soot
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Regeneration Processes
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Optical Access System for DPF Regeneration Studies

- Understand Influence of Regeneration on Ash Properties
  - Active/Passive strategies may impact ash agglomeration and mobility
  - Role of soot interactions with ash during regeneration important

Regeneration Parameters
- Thickness of PM Layer
- Role of NO\(_2\) from DOC vs. CDPF
- Temperature and flow conditions
- Catalysts interactions
Video: PM Oxidation on Clean DPF Surface (Heavy PM)
Video: PM Oxidation with Ash (DPF Cross Section)
Current Optical Setup for Flow Reactor Testing

- Gasket
- Quartz Glass

27mm by 1.44mm Slit in Matting and One Channel of the DPF

Channel View
Video: PM Oxidation on Clean DPF Surface (Heavy PM)
Ash Deposition on Top of PM Layer: Regeneration

Coat surface of soot cake with thin layer of ash

- Allows for visualization of ash/PM mobility during regeneration
- Enables visualization of ash agglomerate formation

- Thin ash layer not fully-established
- Ash applied via accelerated loading system
- Thick, fully-established PM layer
- PM loading accomplished using genset
Ash Deposition on Top of PM Layer: Regeneration

Regeneration with thin ash layer covering PM surface

1 2 3 4
Video: Ash Deposited on Top of PM Layer

Substrate
Ash Agglomeration Process During Soot Oxidation

Ash residence time in DPF is long ~ 100,000 + Miles

Internal void shows walls composed of ~nm scale particles
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  • Ash composition and properties relevant to DPF performance

Sensitivity of DPF Design Parameters to Ash Accumulation
  • Substrate materials, pore size & distribution, porosity

Role of Engine Control Strategies and Exhaust Conditions
  • Temperature, flow, and feed gas conditions affecting ash deposits

Regeneration Processes
  • Real-time optical studies of ash formation and mobility

Ash – Catalyst Interactions
  • Chemical and physical interactions of ash and catalyst/washcoat
Summary and Conclusions

**Detailed understanding of all system parameters important to reduce impact of ash on DPF degradation and fuel efficiency.**

I. Ash Build-Up: Ash loading of ~ 10 g/L or around 50,000 miles required to form fully-established ash layer.

II. Ash Morphology: Two porosity scales identified in ash layer and ash primary particles, which are themselves hollow.

III. Lube Chemistry: Ash properties and DPF pressure drop strong function of additive composition.

IV. Exhaust Conditions: Transient changes in temperature induce much larger variations in ash packing than high flow rates.

V. DPF Parameters: DPF pressure drop relatively insensitive to original substrate porosity following ash layer build-up.

VI. Regeneration Effects: Preliminary optical studies highlight importance of regeneration parameters but requires further study.
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