Dual Layer Coated High Porous SiC for SCR Integration into DPF

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Outline

- Motivation for this development
- The high porous SiC substrate
- The first coating layer for improvement of mechanical strength
- The second functional layer: SCR catalyst
- Analysis of coated lab samples
- SCR efficiency in lab scale test
- Engine bench test data
- Summary
Need for future diesel exhaust emission systems: Euro VI/Tier 4 final and beyond

Reduction of space and costs

CO₂ reduction via the exhaust system

- Weight
- Back pressure
- Regeneration strategy – efficient control of temperature – optimal use of fuel – lower fuel consumption

Integration of DeNOₓ (SCR) functionality into DPF

Literature:
SAE 2011-01-1312 → reduced packaging by SCR-DPF / SCR
SAE 2011-01-1140 → Cu zeolite on Cordierite DPF
SAE 2013-01-0840 → high porous SiC
Design of a high porous DPF substrate for SCR integration

- High porosity level (> 60%)
- High spec. Surface area
- Good mechanical strength
- Reasonable soot load limit (> 5g/l)
- Specific weight high enough → sufficient heat capacity
- High filtration efficiency

SiC with 65% porosity
The high porous SiC substrate

<table>
<thead>
<tr>
<th>Property</th>
<th>Value</th>
</tr>
</thead>
<tbody>
<tr>
<td>CTE (RT – 800°C), 1/K</td>
<td>4.7</td>
</tr>
<tr>
<td>Therm. heat cond. 400°C, W/mK</td>
<td>2.2</td>
</tr>
<tr>
<td>Spec. heat capacity 400°C, J/gK</td>
<td>1.032</td>
</tr>
<tr>
<td>Bending strength, MPa</td>
<td>2.8</td>
</tr>
<tr>
<td>Maximum operating temperature, °C</td>
<td>1400</td>
</tr>
</tbody>
</table>
Performance of blank filters due to filtration and back pressure

<table>
<thead>
<tr>
<th>200 cpsi – 5.66”x8”</th>
<th>PN efficiency</th>
<th>Back pressure @ 200kg/h, 450°C</th>
</tr>
</thead>
<tbody>
<tr>
<td></td>
<td>fresh</td>
<td>3 ESC</td>
</tr>
<tr>
<td>58%</td>
<td>15-18µm</td>
<td>400µm</td>
</tr>
<tr>
<td>60%</td>
<td>15-18µm</td>
<td>400µm</td>
</tr>
<tr>
<td>65%</td>
<td>20-22µm</td>
<td>400µm</td>
</tr>
</tbody>
</table>

| 300 cpsi – 5.66”x8” | fresh | 3 ESC | fresh | 5g/l |
|---------------------|---------------|-------------------------------|
| 58%                 | 14-16µm | 300µm | >92% | >99.9% | 3.7kPa | 8.8kPa |
| 60%                 | 14-16µm | 300µm | >92% | >98% | 3.5kPa | 8.2kPa |
First coating layer for improvement of mechanical strength
Second coating layer for SCR functionality
SCR catalyst candidates

<table>
<thead>
<tr>
<th>Function of every single compound</th>
<th>Vanadia-based ($V_2O_5/WO_3-TiO_2$)</th>
<th>Cu-Zeolite (Cu-ZSM-5)</th>
<th>Fe-Zeolite (Fe-β)</th>
<th>Mixed metal oxide (CeO$_2$-ZrO$_2$ based)</th>
</tr>
</thead>
<tbody>
<tr>
<td>SCR activity</td>
<td>High (dependent on $V_2O_5$ content, best: ~ 3 wt%)</td>
<td>High at low temperatures steadily decreasing beyond 350°C</td>
<td>High (at high temperatures up to 600°C, above NH$_3$ over-consumption) Low (&lt; 300°C)</td>
<td>High (variable temperature window dependent on Ce/Zr ratio + dopants)</td>
</tr>
<tr>
<td>SCR temperature interval</td>
<td>$T_{50}$: 200°C ≥ 90%: 300°C – 500°C</td>
<td>$T_{50}$: 180°C ≥ 90%: 250°C – 400°C</td>
<td>$T_{50}$: 300°C ≥ 90%: 400°C – 650°C</td>
<td>$T_{50}$: ~ 250°C or lower ≥ 90%: ~300°C – 500/550°C dependent on Ce/Zr ratio + dopants</td>
</tr>
<tr>
<td>NH$_3$ storage</td>
<td>Low</td>
<td>High</td>
<td>High</td>
<td>Medium</td>
</tr>
<tr>
<td>N$_2$O formation</td>
<td>Increasing formation at &gt;400°C (at 10 ppm NH$_3$ slip)</td>
<td>High formation tendency even at low temperatures</td>
<td>No formation Reduces N$_2$O to N$_2$ above 400°C</td>
<td>Low formation tendency</td>
</tr>
<tr>
<td>Toxicity</td>
<td>$V_2O_5$ volatility (&gt;690°C)</td>
<td>Concerns due to CuSO$_4$ creation</td>
<td>No</td>
<td>No/low</td>
</tr>
</tbody>
</table>
### Lab sample specifications

<table>
<thead>
<tr>
<th>Sample ID</th>
<th>First layer</th>
<th>Second layer</th>
</tr>
</thead>
<tbody>
<tr>
<td>#FeZ01</td>
<td>—</td>
<td>Fe-β-zeolite, 120g/l</td>
</tr>
<tr>
<td>#FeZ02</td>
<td>—</td>
<td>Fe-β-zeolite, 60g/l</td>
</tr>
<tr>
<td>#FeZ03</td>
<td>SiO₂, 60g/l</td>
<td>Fe-β-zeolite, 65g/l</td>
</tr>
<tr>
<td>#CeZr01</td>
<td>CeO₂/ZrO₂/Nb₂O₅ nano slurry, 160g/l</td>
<td>—</td>
</tr>
<tr>
<td>#CeZr02</td>
<td>CeO₂/ZrO₂/Nb₂O₅, 130g/l</td>
<td>—</td>
</tr>
<tr>
<td>#CeZrFeZ01</td>
<td>—</td>
<td>CeO₂/ZrO₂/Nb₂O₅, 50g/l + Fe-β-zeolite, 50g/l</td>
</tr>
<tr>
<td>#CeZrFeZ02</td>
<td>SiO₂, 60g/l</td>
<td>CeO₂/ZrO₂/Nb₂O₅, 50g/l + Fe-β-zeolite, 40g/l</td>
</tr>
<tr>
<td>#CeZrFeZ03</td>
<td>CeO₂/ZrO₂/Nd₂O₅/Pr₆O₁₁ nano slurry, 55g/l</td>
<td>Fe-β-zeolite, 55g/l</td>
</tr>
</tbody>
</table>
Test of lab samples

- Main gas flow: pressurized air
- NO concentration: 250/500 ppm
- NO$_2$ concentration: 250/0 ppm
- NH$_3$ concentration: 500 ppm
- Water content: 10 %
- Space velocity: 31,000/h (normalized 20°C, 1013 hPa)
- (50,000/h @ 200 °C – 75,000/h @ 450 °C)
- Temperature range: 200 – 450 °C
Fe-β zeolite

#FeZ01
65% / 20µm → 56% / 18µm

#FeZ03
65% / 20µm → 55% / 18µm

NO₂/NOₓ ratio was adjusted to 50%
CeO$_2$-ZrO$_2$ based mixed metal oxides

#CeZr01
65% / 20µm → 60% / 18µm

#CeZr02
65% / 20µm → 61% / 19µm

NO$_2$/NO$_x$ ratio was adjusted to 50%
The combinations of Fe-β-zeolite with CeO₂-ZrO₂ mixed metal oxides

#CeZrFe01

65% / 20µm → 47% / 17µm

#CeZrFe03

65% / 20µm → 50% / 18µm

NO₂/NOₓ ratio was adjusted to 50%
NO$_X$ conversion under standard SCR conditions compared to fast SCR
Engine bench test setup

- OM 904 engine, 4.25L – 4 Cyl. - 129 kW
- Dynamometer: Horiba T250
- Gas analyzer: Horiba MEXA6000-FT
- AdBlue dosing: Emitec Airless urea doser
- An uncoated DPF was mounted upstream to take the soot out of exhaust mass flow

![Diagram of engine setup]

- Inlet DPF
- DOC
- urea injection
- Hyd Cat
- F-SCR
### Filters with SCR coatings for engine bench tests

<table>
<thead>
<tr>
<th></th>
<th>#FeZ01-DPF</th>
<th>#FeZ03-DPF</th>
<th>#CeZrFeZ01-DPF</th>
<th>#CeZrFeZ03-DPF - layered</th>
</tr>
</thead>
<tbody>
<tr>
<td><strong>PN efficiency, fresh</strong></td>
<td>&gt;99.8%</td>
<td>&gt;99.8%</td>
<td>&gt;99.8%</td>
<td>&gt;99.8%</td>
</tr>
<tr>
<td><strong>Back pressure cold flow @ 600m³/h</strong></td>
<td>6.8kPa</td>
<td>6.9kPa</td>
<td>6.2kPa</td>
<td>6.0kPa</td>
</tr>
<tr>
<td><strong>Back pressure @ 140kg/h 480°C</strong></td>
<td>3.4kPa</td>
<td>3.5kPa</td>
<td>3.3kPa</td>
<td>3.1kPa</td>
</tr>
</tbody>
</table>

- 60g/l SiO₂
- 60g/l Fe-β-Z
- 60g/l CeO₂/ZrO₂/Nb₂O₅
- 60g/l Fe-β-Z

Dinex - going the extra mile
SCR efficiency for all the test filters

![Graph showing SCR efficiency at different temperatures for different filters.](image-url)
Performance of #CeZrFeZ03-DPF_layered during ESC

- SCR performance over cycle: **70.0 %**
- Filtration performance over cycle: **99.6 %**
Performance of CeZrFeZ03-DPF_layered during ETC

- SCR performance over cycle: 76.3%
- Filtration performance over cycle: 99.7%
Summary and conclusions

- A high porous SiC with a dual layer coating was presented
  - Enhancing mechanical strength
  - High SCR performance at low catalyst loadings

- Catalyst solutions based on a Fe-β-zeolite and mixed metal oxides (doped ceria/zirconia) have been developed

- Lab scale and engine bench test show a high SCR efficiency between 80 % and 95 % for the zeolite and zeolite/mixed metal oxide solutions

- High porosity of 65 % combined with the initial layer is the optimum substrate for the used catalysts

- The developed SCR coated DPF is a very promising candidate for future Euro VI systems with reduced packaging size
Thank You for your Attention!

For more details see: SAE2014-01-1484