

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



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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 DeNO_x (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



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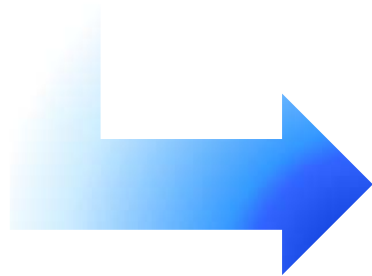
Design of a high porous DPF substrate for SCR integration

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

High wash coat
loading for high
 NO_x conversion

Robustness
and protection
of catalyst

PN efficiency
requirements

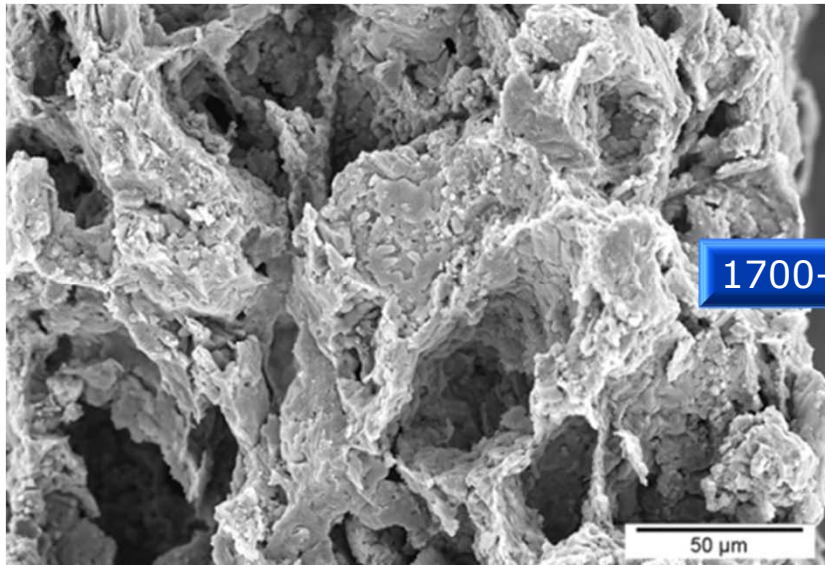


SiC with 65% porosity



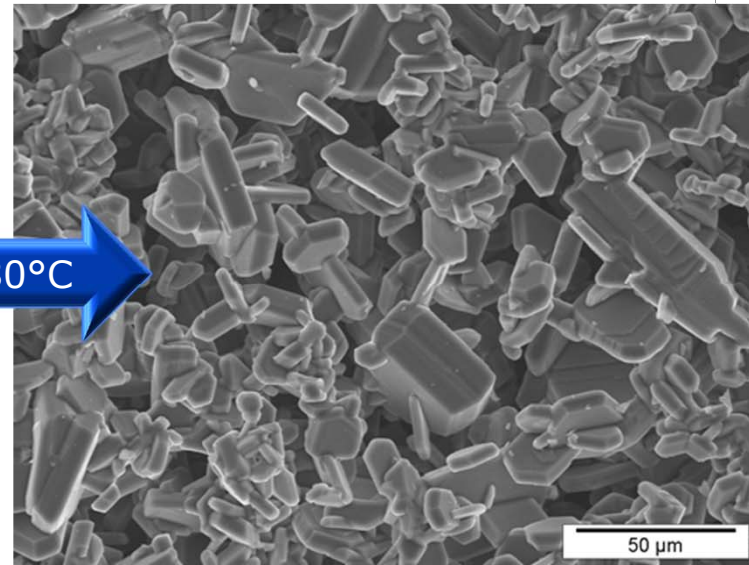
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The high porous SiC substrate



SAE 2010-01-0539

1700-1980°C



WO 2013/076045A1

CTE (RT – 800°C), 1/K	4.7
Therm. heat cond. 400°C, W/mK	2.2
Spec. heat capacity 400°C, J/gK	1.032
Bending strength, MPa	2.8
Maximum operating temperature, °C	1400



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Performance of blank filters due to filtration and back pressure

PN efficiency

Back pressure
@ 200kg/h, 450°C

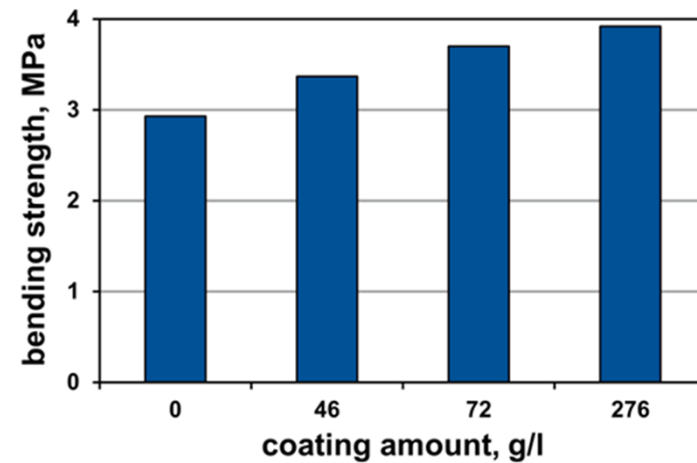
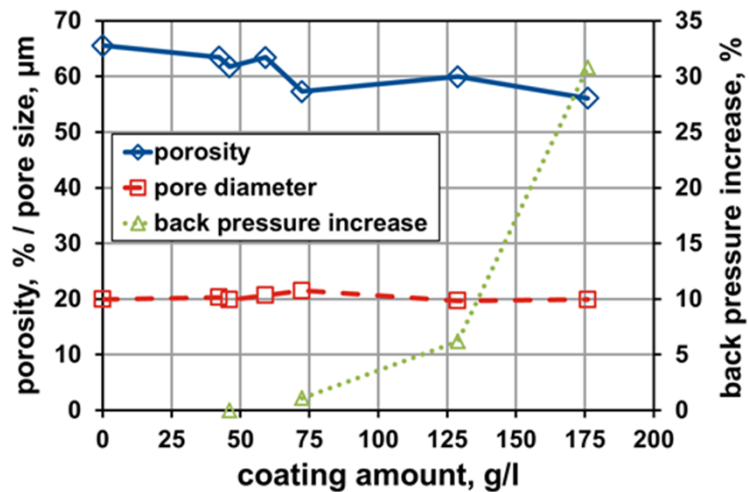
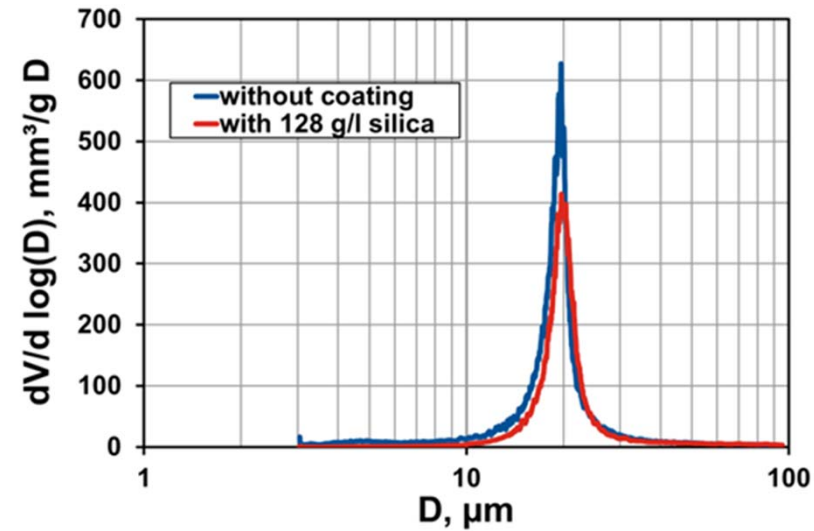
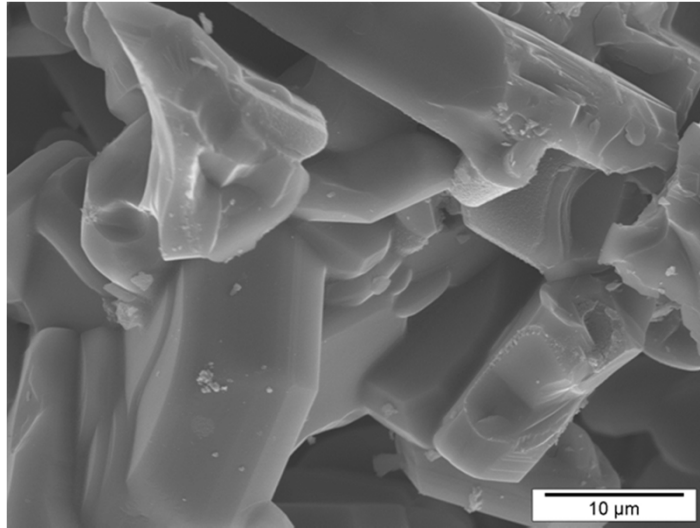
200 cpsi – 5.66''x8''			fresh	3 ESC	fresh	5g/l
58%	15-18µm	400µm	>98%	>99.9%	3.3kPa	10.1kPa
60%	15-18µm	400µm	>93%	>98%	3.1kPa	9.5kPa
65%	20-22µm	400µm	>80%	>95%	2.9kPa	9.0kPa

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



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First coating layer for improvement of mechanical strength



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Second coating layer for SCR functionality

SCR catalyst candidates

	Vanadia-based (V ₂ O ₅ /WO ₃ -TiO ₂)	Cu-Zeolite (Cu-ZSM-5)	Fe-Zeolite (Fe-β)	Mixed metal oxide (CeO ₂ -ZrO ₂ based)
Function of every single compound	V ₂ O ₅ : SCR active center WO ₃ : promoter TiO ₂ : support	Cu: NO oxidation to NO ₂ ZSM-5: host, SCR reaction, NH ₃ storage	Fe: NO oxidation to NO ₂ Beta: host, SCR reaction, NH ₃ storage	CeO ₂ : SCR active center, O ₂ storage ZrO ₂ : thermal stabilizer, NH ₃ storage
SCR activity	High (dependent on V ₂ O ₅ content, best: ~ 3 wt%)	High at low temperatures steadily decreasing beyond 350°C	High (at high temperatures up to 600°C, above NH ₃ over-consumption) Low (< 300°C)	High (variable temperature window dependent on Ce/Zr ratio, dopants, surface area + water content)
SCR temperature interval	T ₅₀ : 200°C ≥ 90%: 300°C – 500°C	T ₅₀ : 180°C ≥ 90%: 250°C – 400°C	T ₅₀ : 300°C ≥ 90%: 400°C – 650°C	T ₅₀ : ~ 250°C or lower ≥ 90%: ~300°C – 500/550°C dependent on Ce/Zr ratio + dopants
NH ₃ storage	Low	High	High	Medium
N ₂ O formation	Increasing formation at >400°C (at 10 ppm NH ₃ slip)	High formation tendency even at low temperatures	No formation Reduces N ₂ O to N ₂ above 400°C	Low formation tendency
Toxicity	V ₂ O ₅ volatility (>690°C)	Concerns due to CuSO ₄ creation	No	No/low

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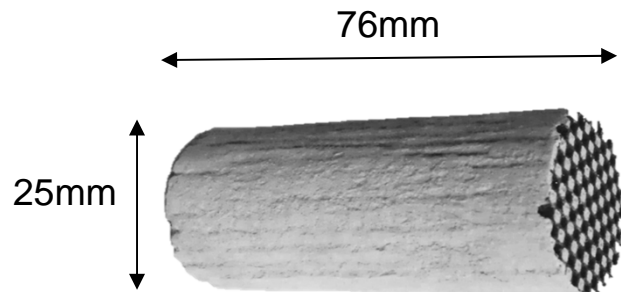
Lab sample specifications

Sample ID	First layer	Second layer
#FeZ01	—	Fe-β-zeolite, 120g/l
#FeZ02	—	Fe-β-zeolite, 60g/l
#FeZ03	SiO ₂ , 60g/l	Fe-β-zeolite, 65g/l
#CeZr01	CeO ₂ /ZrO ₂ /Nb ₂ O ₅ nano slurry, 160g/l	—
#CeZr02	CeO ₂ /ZrO ₂ /Nb ₂ O ₅ , 130g/l	—
#CeZrFeZ01	—	CeO ₂ /ZrO ₂ /Nb ₂ O ₅ , 50g/l + Fe-β-zeolite, 50g/l
#CeZrFeZ02	SiO ₂ , 60g/l	CeO ₂ /ZrO ₂ /Nb ₂ O ₅ , 50g/l + Fe-β-zeolite, 40g/l
#CeZrFeZ03	CeO ₂ /ZrO ₂ /Nd ₂ O ₃ /Pr ₆ O ₁₁ nano slurry, 55g/l	Fe-β-zeolite, 55g/l



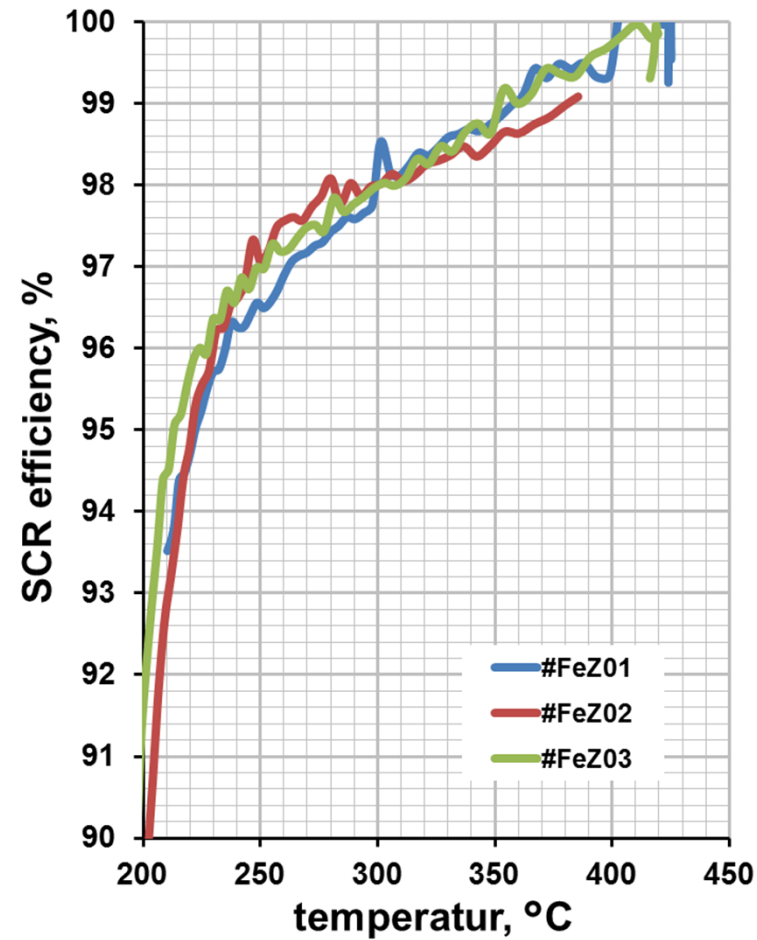
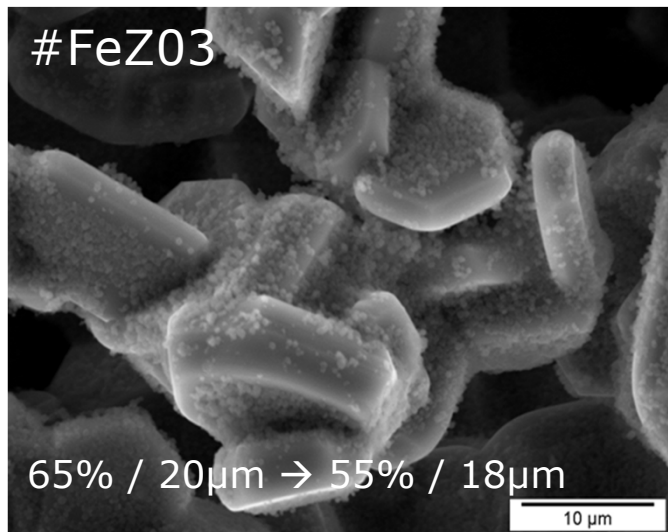
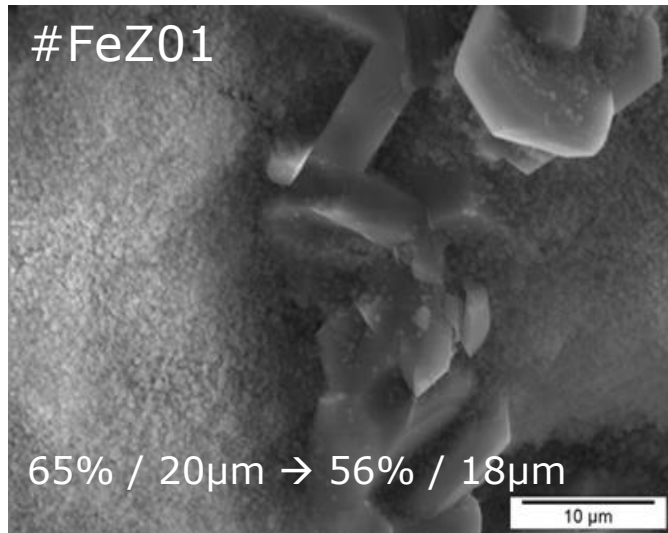
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Test of lab samples



- ▶ Main gas flow: pressurized air
- ▶ NO concentration: 250/500 ppm
- ▶ NO₂ concentration: 250/0 ppm
- ▶ NH₃ 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

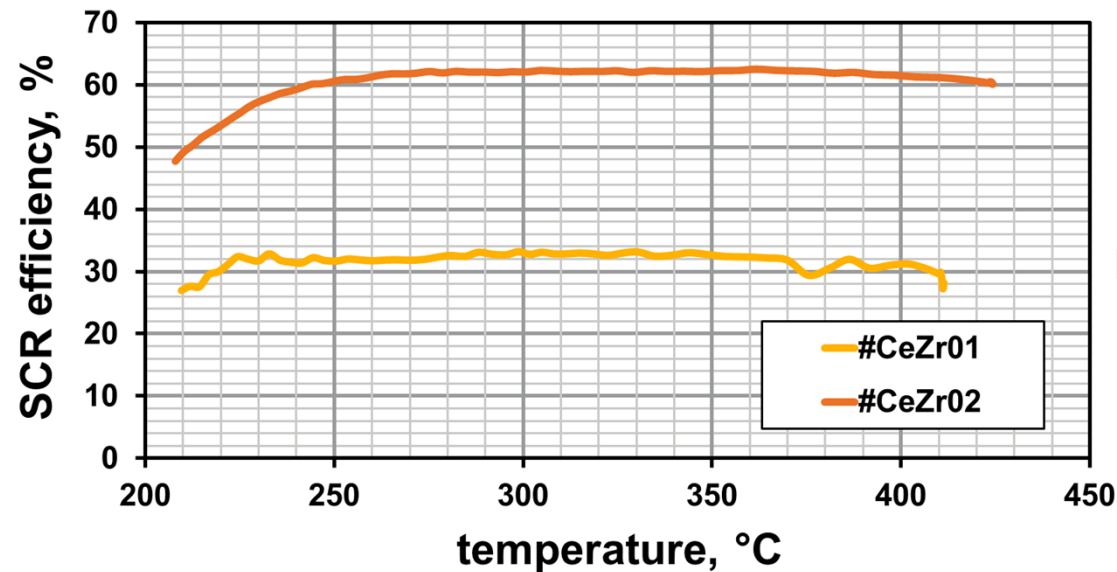
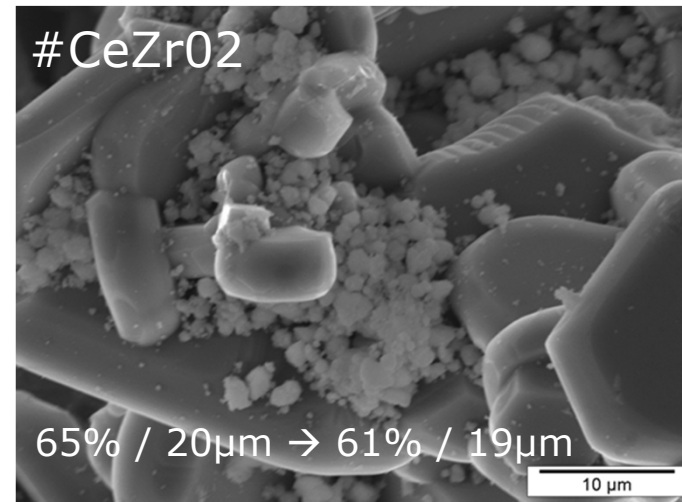
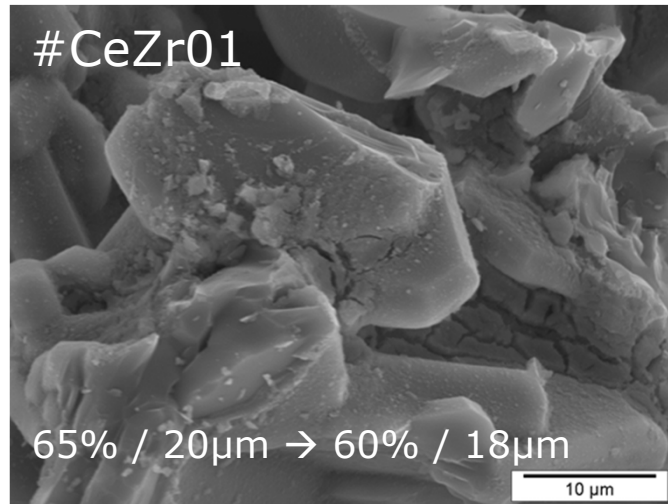


NO₂/NO_x ratio was
adjusted to 50%



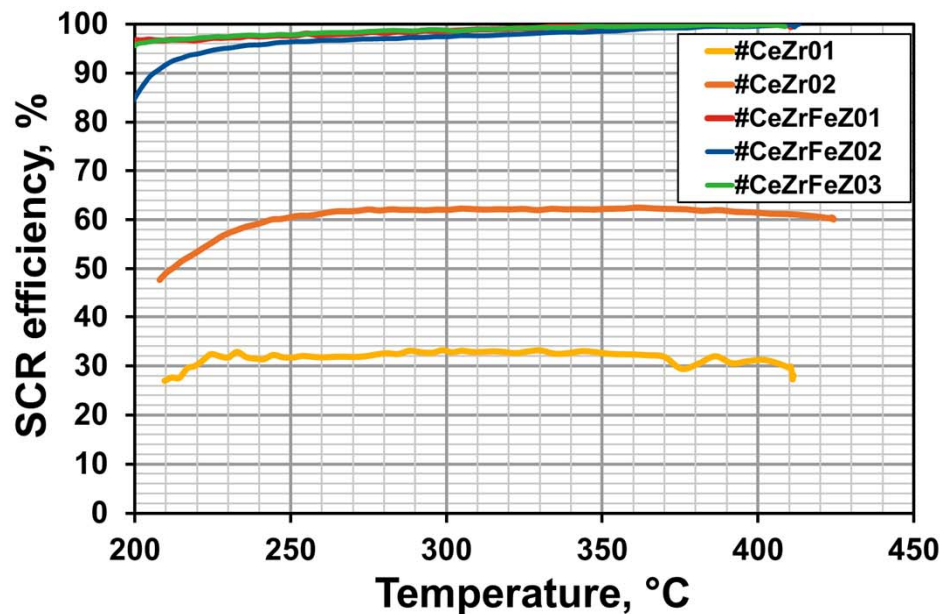
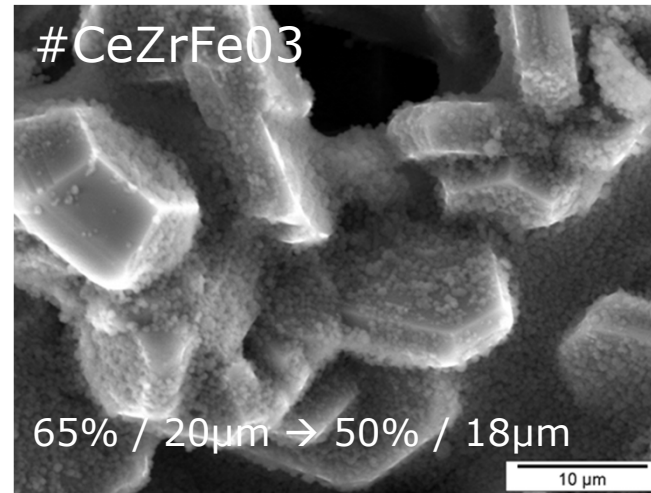
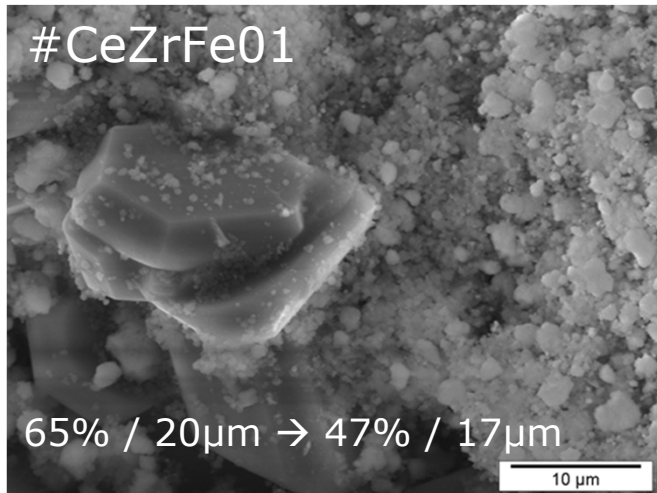
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CeO₂-ZrO₂ based mixed metal oxides

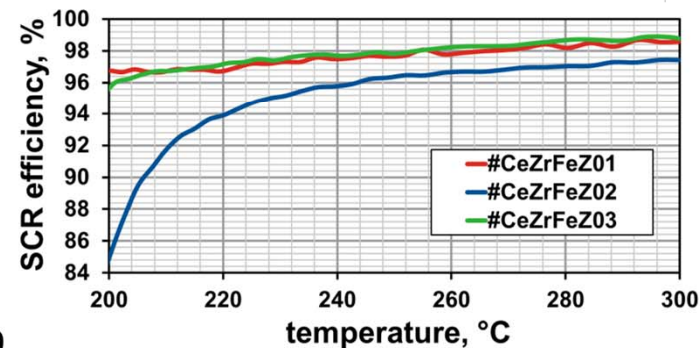


NO₂/NO_x ratio was
adjusted to 50%

The combinations Fe- β -zeolite with CeO₂-ZrO₂ mixed metal oxides

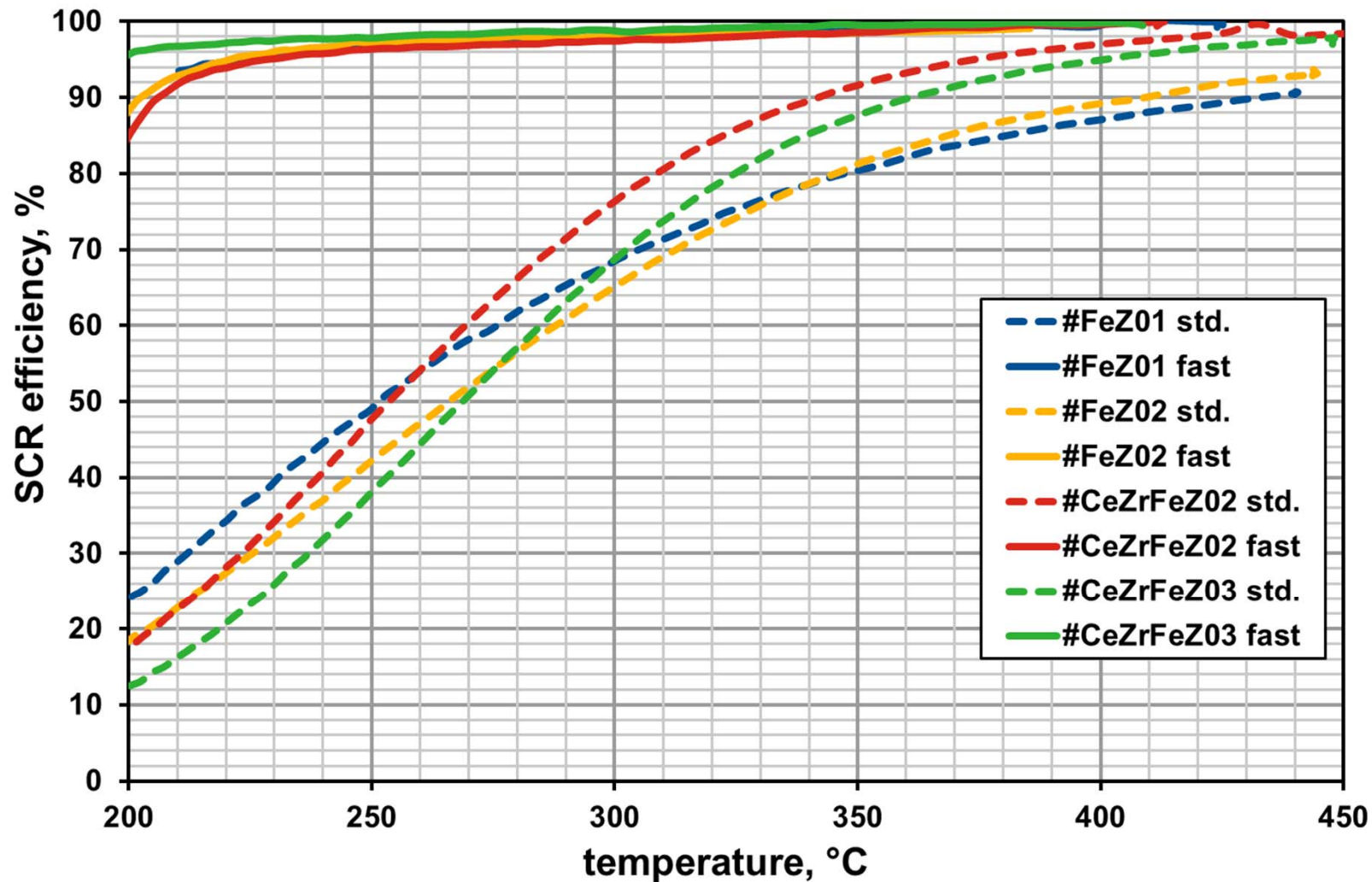


**NO₂/NO_x ratio was
adjusted to 50%**



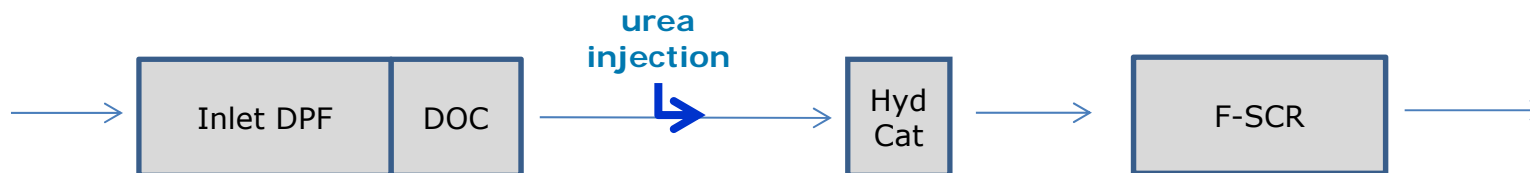
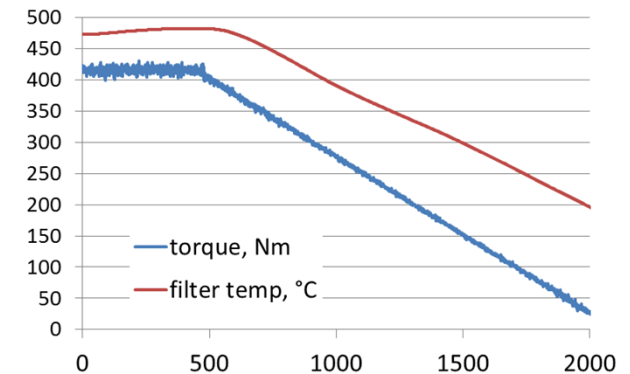
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NO_x conversion under standard SCR conditions compared to fast SCR



Engine bench test setup

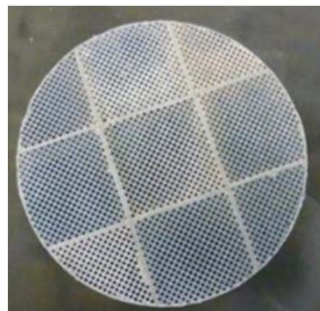
- ▶ OM 904 engine, 4.25L – 4 Cyl. - 129 kW
- ▶ Dynamometer: Horiba T250
- ▶ Gas analyzer: Horiba MEXA6000-FT
- ▶ AdBlue dosing: Emitec Airless urea doser
- ▶ An uncoated DPF was mounted upstream to take the soot out of exhaust mass flow



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Filters with SCR coatings for engine bench tests

	#FeZ01-DPF	#FeZ03-DPF	#CeZrFeZ01-DPF	#CeZrFeZ03-DPF - layered
PN efficiency, fresh	>99.8%	>99.8%	>99.8%	>99.8%
Back pressure cold flow @ 600m ³ /h	6.8kPa	6.9kPa	6.2kPa	6.0kPa
Back pressure @ 140kg/h 480°C	3.4kPa	3.5kPa	3.3kPa	3.1kPa



60g/l SiO₂
60g/l Fe-β-Z

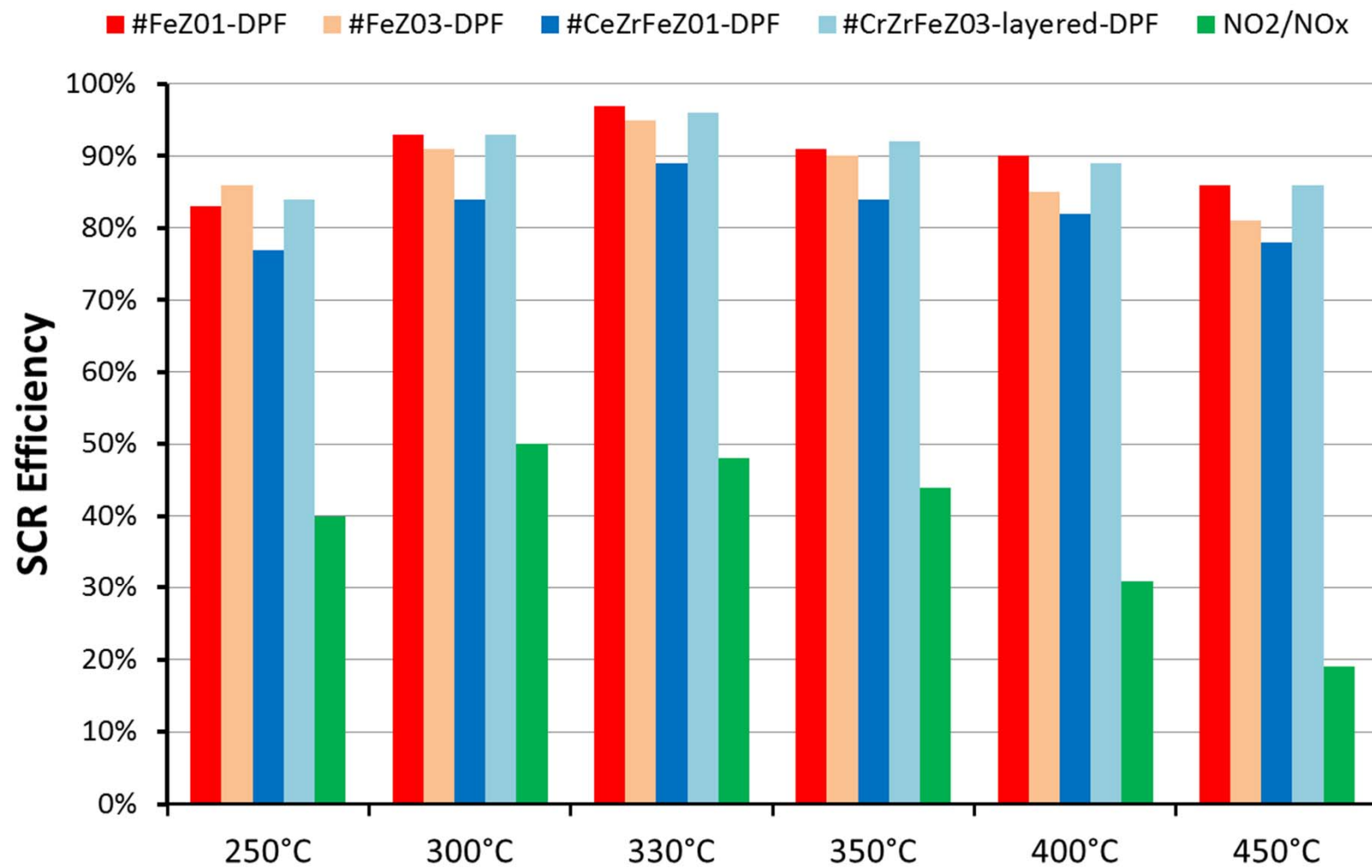


60g/l CeO₂/ZrO₂/Nb₂O₅
60g/l Fe-β-Z



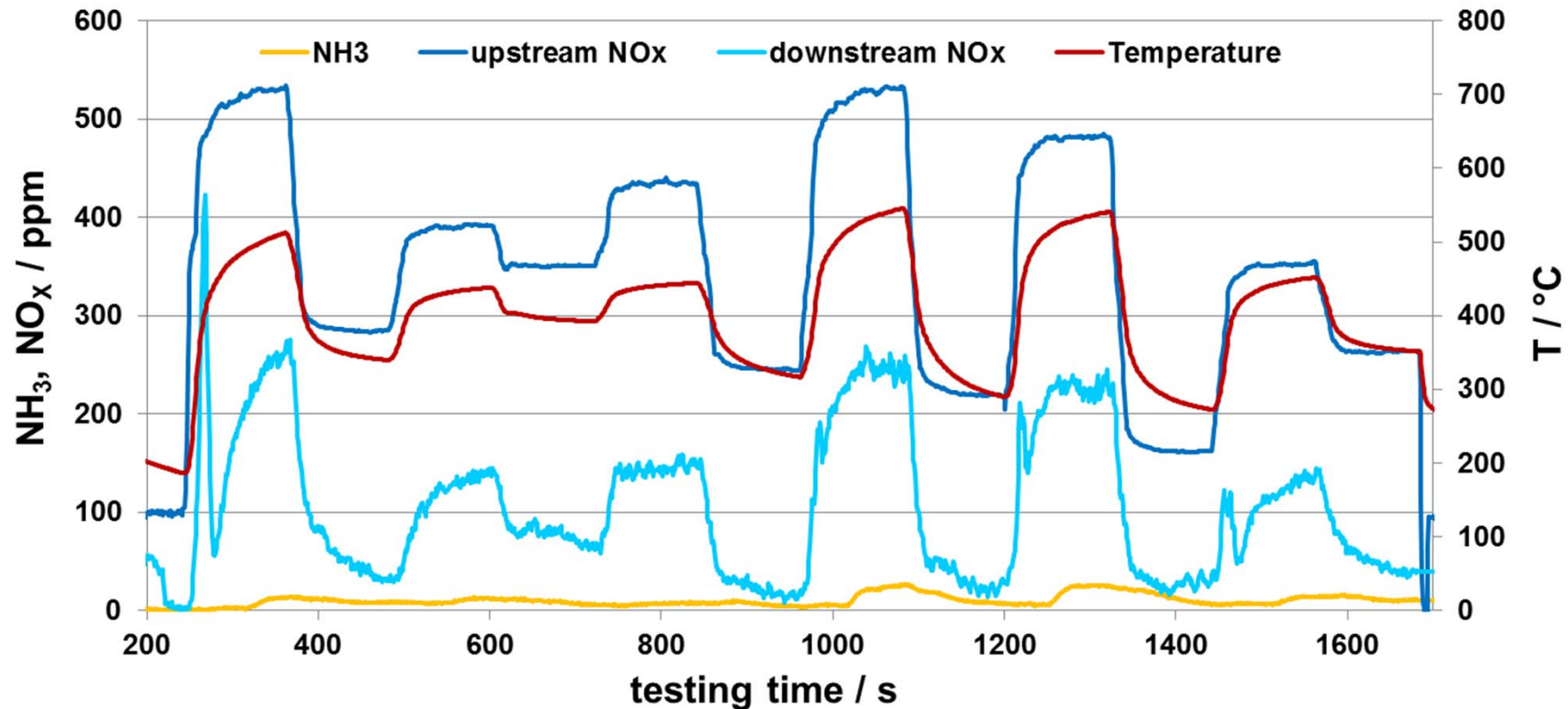
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SCR efficiency for all the test filters



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Performance of #CeZrFeZ03-DPF_layered during ESC



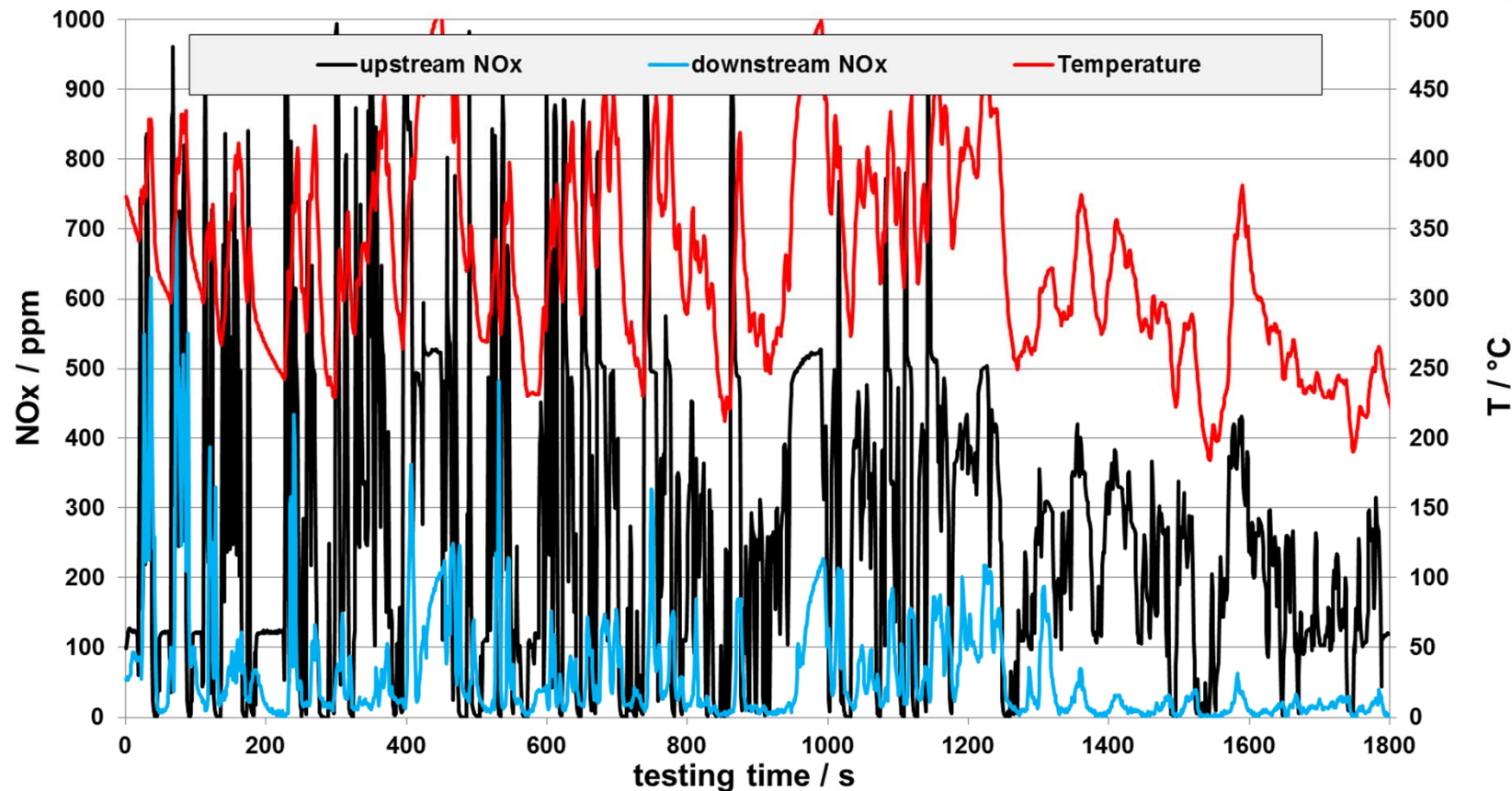
► SCR performance over cycle: **70.0 %**

► Filtration performance over cycle: **99.6 %**



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Performance of #CeZrFeZ03-DPF_layered during ETC



► SCR performance over cycle: **76.3 %**

► Filtration performance over cycle: **99.7 %**



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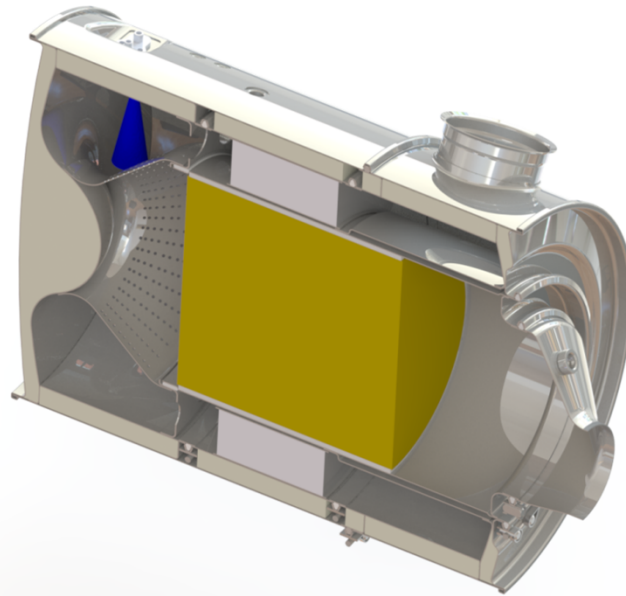
Summary and conclusions

- ▶ 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



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Thank You for your Attention!



For more details see: SAE2014-01-1484



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