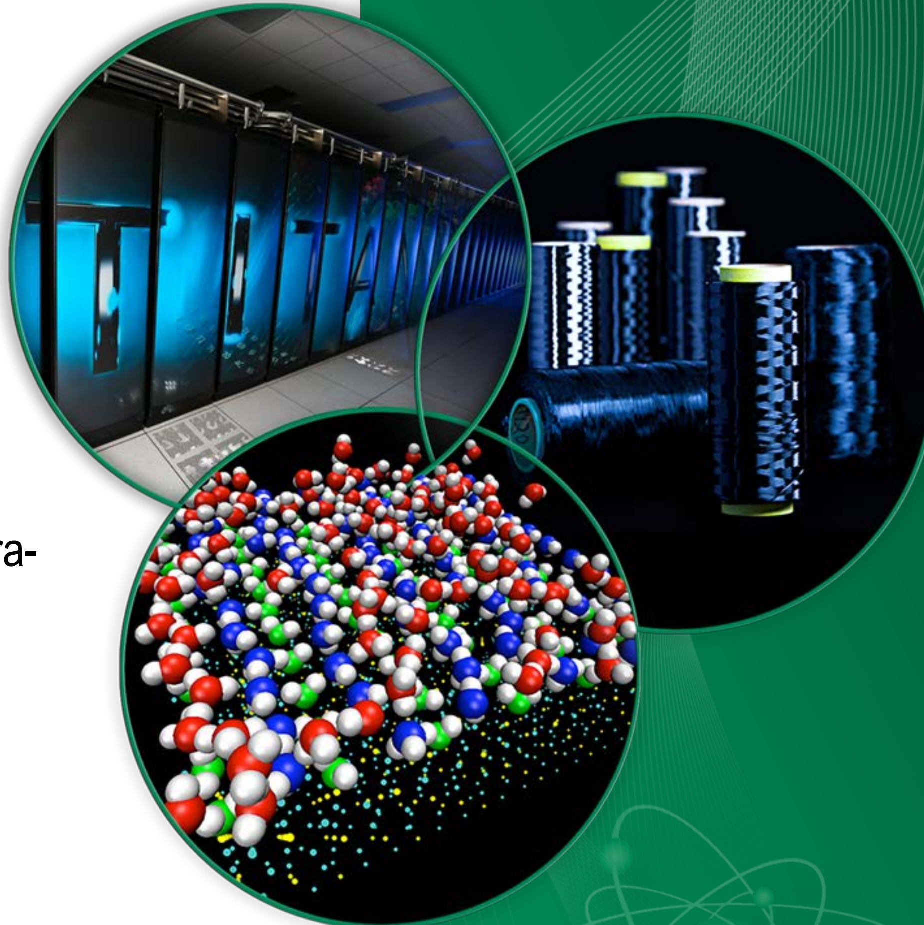
Durability of Diesel Engine Particulate Filters Agreement 10461

Project ID: PM010



2014 DOE Vehicle Technologies Annual Merit Review and Peer Evaluation Meeting

June 20, 2014

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Randall Stafford; *Cummins Inc.*

Sponsored by
U.S. Department of Energy, Energy Efficiency and Renewable Energy, Vehicle Technologies Office

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Objective

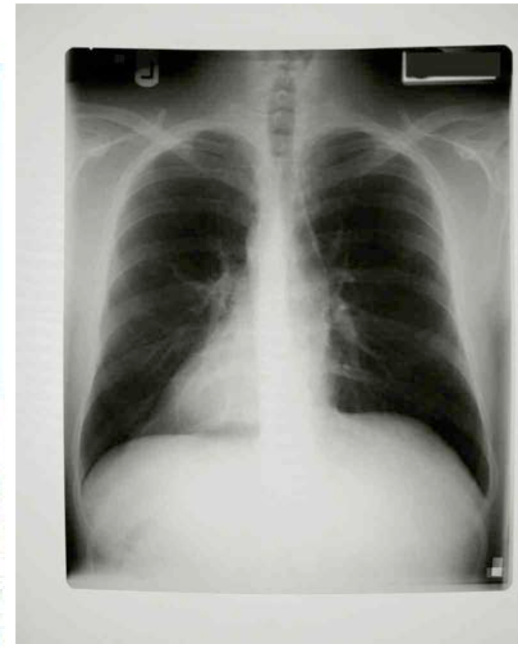
- Implement test techniques to characterize the physical and mechanical properties of ceramic diesel particulate filters (DPFs) and develop analyses and tools for assessing their reliability and durability.

Relevance to Vehicle Technologies' Goals

- "By 2015, develop materials, materials processing and filter regeneration techniques that reduce the fuel economy penalty of particle filter regeneration by at least 25% relative to the 2008 baseline* "
- "By 2015, improve the fuel economy of light-duty diesel vehicles by 40%, compared to the baseline 2009 vehicle* "
  - Understanding the relationship of the porosity to the material properties of the filter (and catalyst) substrates leads to optimized regeneration strategies, thermal management and filter efficiency.
  - Increases acceptance of clean diesel resulting reduction in petroleum consumption

* Vehicle Technologies Program, Multi-Year Program Plan 2011-2015, Dec 2010, pp. 2.5-7, 2.3-2.

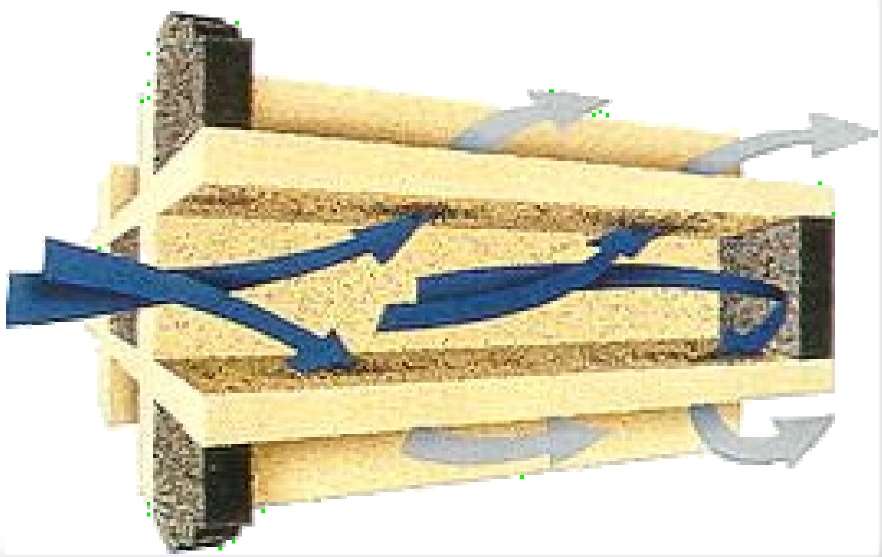
Opportunity/Problem Description:
(Why are we investing in this project?)



- *Reduced emissions AND increased fuel efficiency*

Opportunity/Problem Description:
(Why are we investing in this project?)

- Diesel Particulate Filters (DPFs) play a key role and will continue to be a key technology to meet the prevailing stringent regulations.
- Improving regen strategies: materials properties <u>needed</u> for regen models



Overview

Timeline

- Start: June 2004
- End: Sept. 2015
- 89% complete

Budget

- Total Project funding
  - DOE-$2.8M
  - Cummins-$2.8M
- Funding received:
  - FY13 $170k
  - FY14 $150k (approved)

Barriers*

- Changing internal combustion engine regimes
- Cost-eff. emission control
- Durability
- Market perception

Addressing Barriers by Providing

- Materials information allows reliable and durable regeneration w/ minimized fuel penalty releasing clean exhaust

Partner

- Cummins Inc.

* Vehicle Technologies Program, Multi-Year Program Plan 2011-2015, Dec 2010, pp. 2.3-4, 5, 8; 2.5-7, 8, 9, 10.

Relevance to barriers

- Impact on barriers: Property data generated by this CRADA…
  - Provides necessary input for models to predict DPF regen behavior accurately. In turn, strategies to mitigate thermal stresses can be formulated for optimized regeneration which → *changes engine combustion regimes* and which → improves *durability*/minimizes failures, → improves *cost-effective emission control*
  - Net result of above is cleaner diesel which improves *market perception and reduces emissions*

Approach

Materials identified & provided by Cummins

<u>Cummins</u>
Strength testing, Burner Rig testing

<u>ORNL</u>
Mechanical testing, Microstructural analysis, Thermal expansion; ***Leading to materials properties and selection.***

<u>Diesel Community</u>
Publically available material properties for predictive model development and validation for robust regeneration duty cycle strategies

Utilizes characterization tools acquired and formerly maintained by the High Temperature Materials Laboratory (HTML) Program.

Approach/Milestones FY13 & FY14

- FY13: (1) Determine the origins of the load dependent Young's moduli and quantify the impact of the microstructure on same. (2) Complete characterization of the dynamic and static fatigue response of SiC DPFs. [Complete]

- FY14: (1) Initiate characterization of the dynamic and static fatigue response under the temperature ranges relevant to the engine operating conditions of a fourth alternate substrate material (a zeolite based material). [On-track] (2) Initiate the determination of strength, fracture toughness, density/porosity/microstructure, and thermal expansion as a function of time at elevated temperatures of a fourth alternate substrate material. [On-track]

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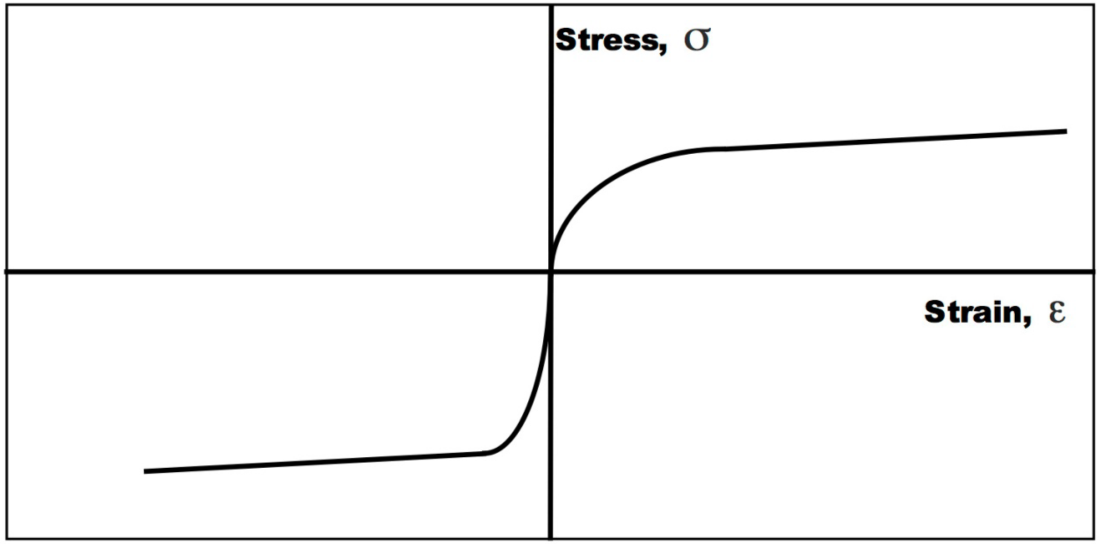
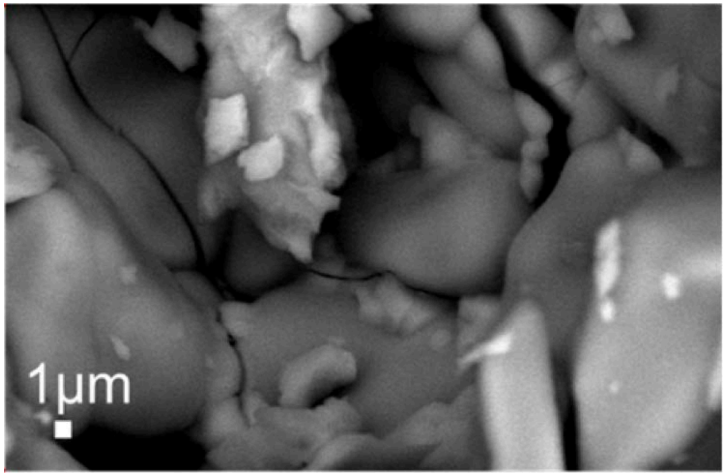
Background: ORNL teams researched and solved apparent discrepancies in Elastic modulus, a *critical* property for regen models. Elastic modulus is difficult to quantify because it is non-linear in DPF materials✪

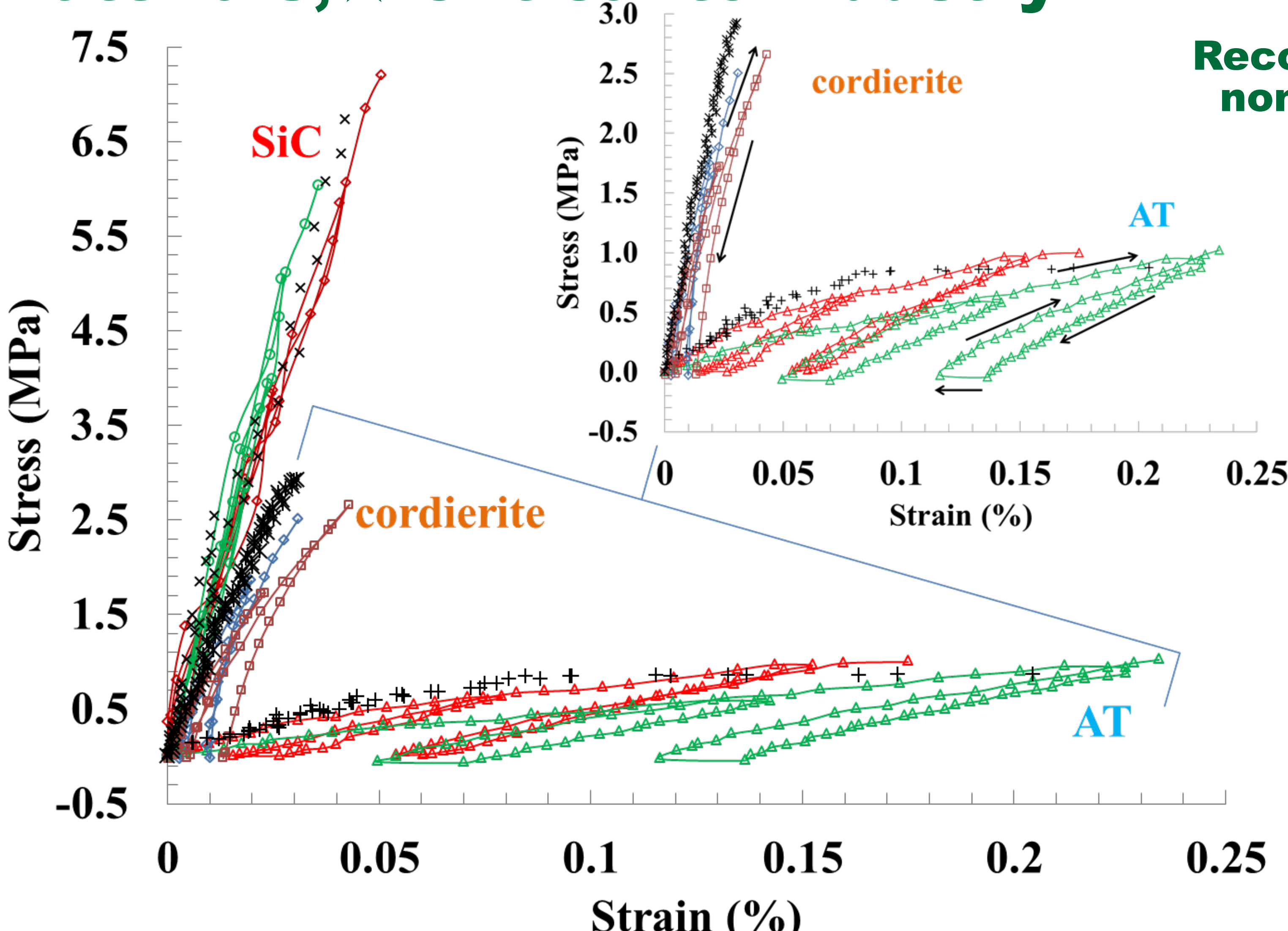




- Dynamic techniques: low loads, high moduli: 10-14 GPa cordierite**
- Static techniques: higher loads, lower moduli: 1-3 GPa cordierite**
- Microstructure: porosity and microcracks and "Honeycomb" structure play a role

✪Wereszczak et al., "Failure Stress and Apparent Elastic Modulus of Diesel Particulate Filter Ceramics," SAE Int. J. Mater. Manuf., **5**(2) 517- 27 (2012).
**Stafford et al., 2012, 37th International Conference and Exposition on Advanced Ceramics and Composites (ICACC 2013).

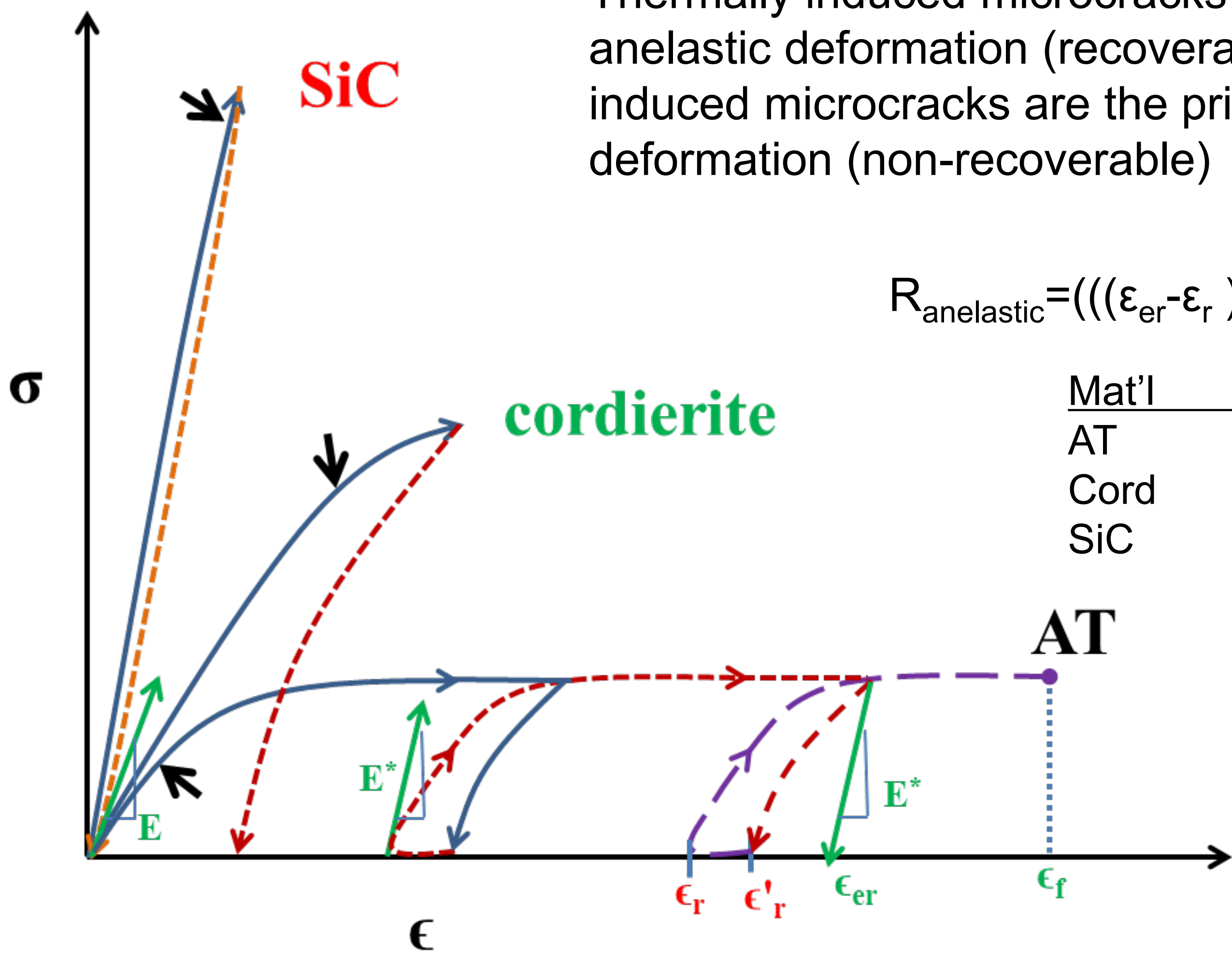
Tech.Acc: Loading-unloading-reloading response of AT, cordierite and SiC based materials;★ critical to industry



Recoverable AND non-recoverable deformation observed

★ A. Pandey, A. Shyam, T. R. Watkins, E. Lara-Curzio, R. J. Stafford and K. J Hemker, "The Uniaxial Tensile Response of Porous and Microcracked Ceramic Materials," J. Am. Ceram. Soc. 97 [3] 899–906 (2014). One of three featured articles for March.

Tech.Acc: Stress-strain response of these materials was non-linear; degree of non-linearity is related to the initial microcrack density and evolution of damage in the material.



Thermally induced microcracks are the primary cause of anelastic deformation (recoverable) and the mechanically induced microcracks are the primary cause of inelastic deformation (non-recoverable) during unloading

$$R_{inelastic}=100^*(\varepsilon_r / \varepsilon_{er})$$

$$R_{anelastic}=(((\varepsilon_{er}-\varepsilon_r)^*100) / \varepsilon_{er}) = (100-R_{inelastic})$$

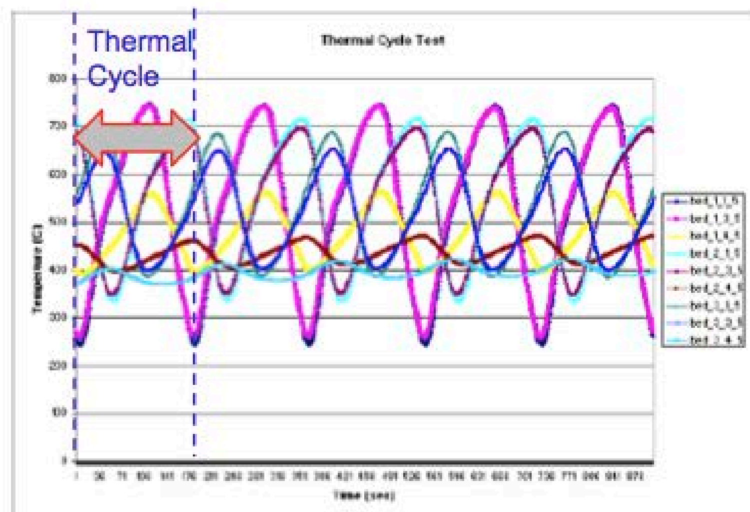
Mat'l	E(GPa)	R_{ane}(%)	R_{ine}(%)
AT	0.9	50	50
Cord	10	67	33
SiC	15	98	2

★ A. Pandey, A. Shyam, T. R. Watkins, E. Lara-Curzio, R. J. Stafford and K. J Hemker, "The Uniaxial Tensile Response of Porous and Microcracked Ceramic Materials," J. Am. Ceram. Soc. 97 [3] 899–906 (2014). One of three featured articles for March.

Tech.Acc: Cummins' burner rig testing of DPF provides simulated measures of lifetime

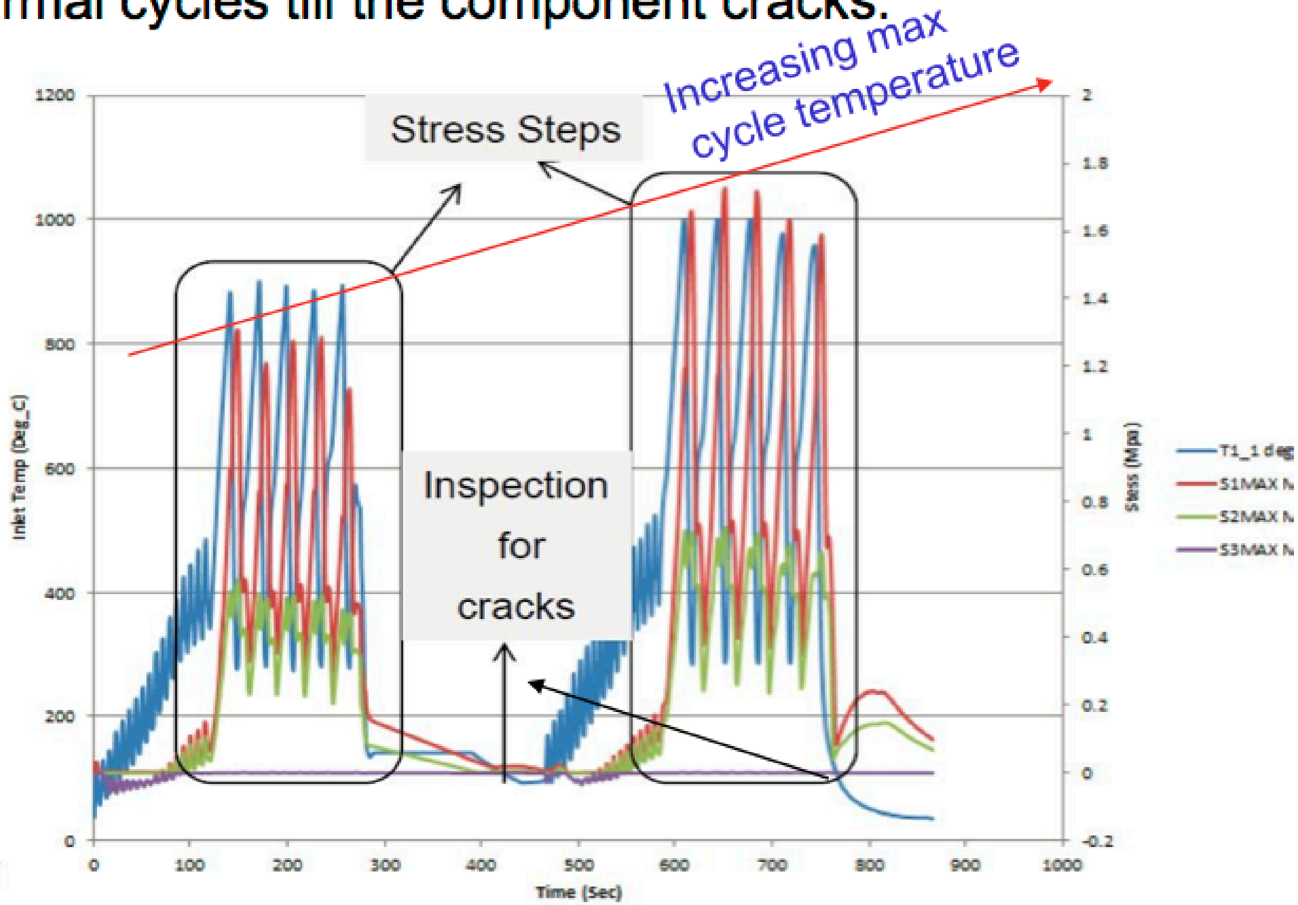
Burner Rig for Thermal Cycle Testing

- A thermal cycle is complete when the target thermocouple completes a 250 – 600/700/xxx – 250 C . The time for one cycle ranges from 3-5 mins.
- Typical temperature profile



Tech.Acc: Cummins' burner rig testing of DPF provides simulated measures of lifetime (cont'd)

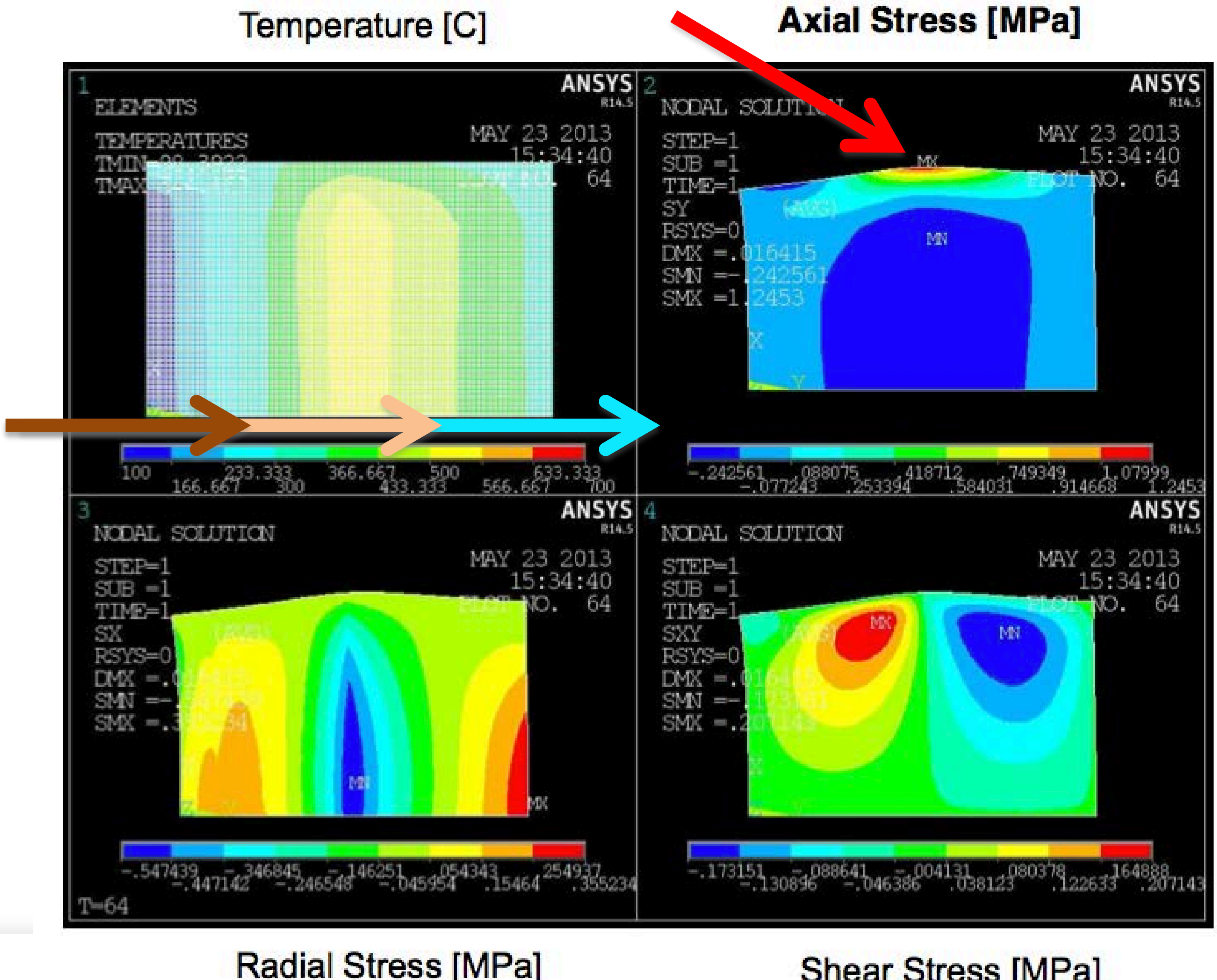
- Burner rig is used to subject the DPF to thermal load by exposing it to high temperature exhaust gases
- Step stress procedure involves increasing max temperature every 5 thermal cycles till the component cracks.



Tech.Acc.: Cummins puts materials data to work in new regen models✣

Maximum Axial Stress can cause cracking

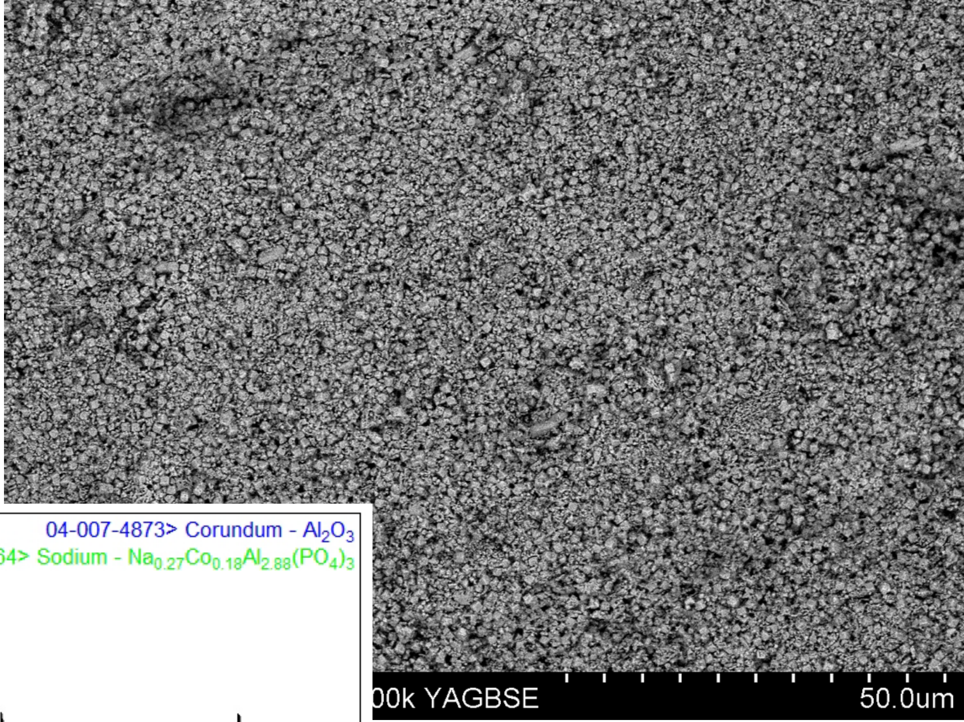
Exhaust flow left to right

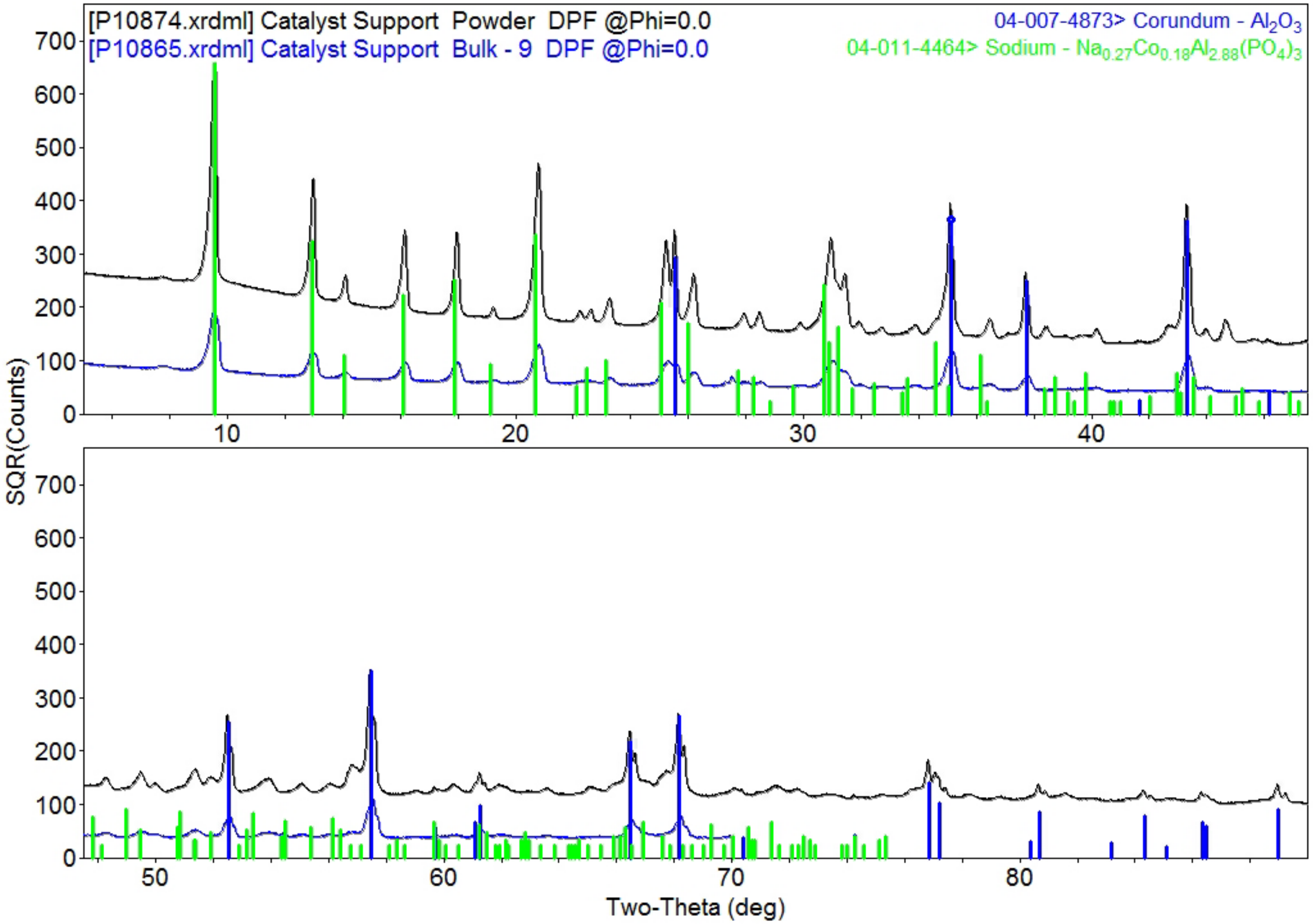


✣ C. C. Su, D. M. Aguilar, R. J. Stafford, "Thermal Fatigue Analysis of Diesel Particulate Filters presented by R. J. Stafford at the 38th International Conference on Advanced Ceramics and Composites Meeting held in Daytona Beach, FL, January 31, 2014

Tech.Acc.: New Material: A zeolite based support, prospective DPF

- 58% porous but finer pore structure observed





- XRD & SEM w/EDAX identify Chabazite Zeolite
- Zeolite GS = 2μm

Response to Reviewer's comments

There were several complementary comments. Thanks.

The repeated criticisms trended into 2 areas:

Project longevity: 10+ years. Its focus has been fairly consistent to experimentally determine the properties of DPF materials in various states: new and used. The properties provide necessary input for models to predict DPF regen behavior accurately. Each year both material characterizations and system studies were performed.

FY2004-8: Cordierite DPF materials.

FY2009-11: Aluminum Titanate (Al_2TiO_5)-based DPF materials.

FY2012-13: Si-SiC DPF materials.

FY2014-15: Zeolite catalyst support that could become a DPF material.

The funding level of this project over 10+ years is consistent with what is currently possible in area of interest 8 within the recent Funding Opportunity Announcement (FOA) Number: DE-FOA-0000991 over a shorter term.

2. Routine work: These samples are >50% porous and are a challenge/not routine to work with. Specimen preparation in non-trivial requiring a meticulous machinist who can innovate (we published a paper, in part, on sample preparation). Over the years we have developed new techniques to measure properties in these highly porous materials (e.g., 2013 our new microtesting rig with DIC to measure Young's modulus artifact free, the release of ASTM standard C1674 in 2008). Fracture toughness testing has been done with a novel double torsion method. We will be exploring other methods with our new microtesting rig.

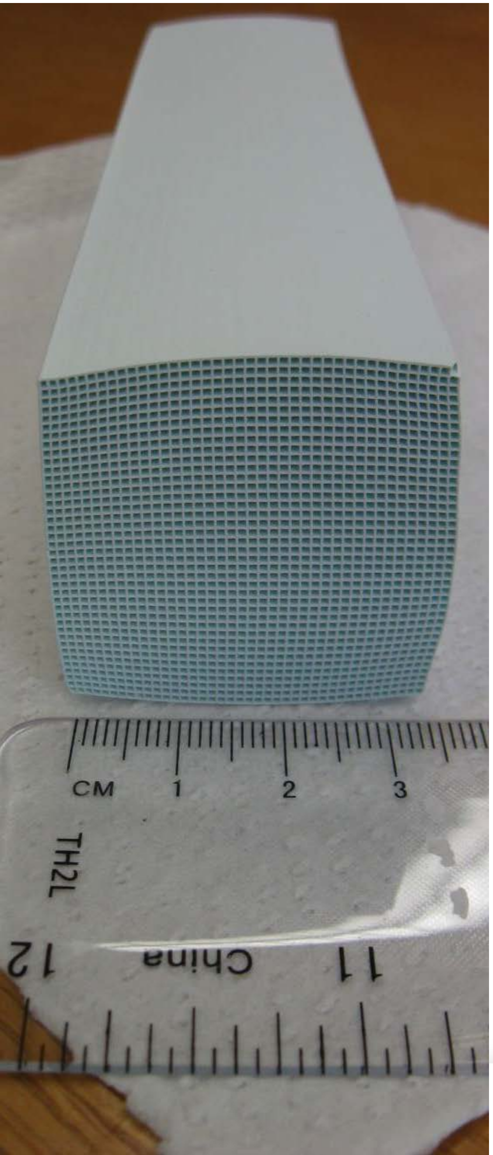
Benefits only one company: These DPF products are commercially available and therefore diesel engine manufacturers should be interested. Our results are made available to the community through the AMR, DOE annual reports and other venues (also see presentations and publications). Cummins' advanced models are proprietary.

"…did not see a relation to petroleum displacement.": A sanitized example is provided in the Technical Backup Slides Section.

Collaborations and coordinations with other institutions: CRADA Partner

- Cummins (Industry):
  - Cummins' role is to collaborate and guide the work along the most useful path to achieve durability, cost and emissions targets
  - Telecons and meetings: Exchange of technical information to assist with each others analyses; Share experimental results
  - Supplies samples
  - Using the materials property information from this CRADA, diesel community can optimize diesel engine operation to minimize the fuel penalty associated with regen and eliminate or reduce DPF failures in-service.

What are the challenges?



- *New zeolite material has very thin walls 0.2 mm*
  - *Machining and testing are difficult and delicate, respectively*
  - *Seeking new tests to resolve as we did in the past to understand the non-linear Young's modulus*
- *How to measure elastic modulus in a regime that corresponds to the actual stress-strain field in application.*
- *Validation of predicted cracking with an actual component*
- *Creation and validation of 3D structure models using the micro level data being developed.*

Proposed Future Work

- Continue to investigate the mechanical properties of the new Zeolite-based substrate.
  - Characterization of the dynamic and static fatigue response under the temperature ranges relevant to the engine operating conditions
  - Determination of strength, fracture toughness, density/porosity/microstructure, and thermal expansion as a function of time at elevated temperatures
- Continue with Young's modulus studies transverse to the extrusion direction as this is needed for models
- Initiate new testing methodologies for measuring fracture toughness/fracture energy of these highly porous materials
- Model development to complement the new testing methodologies for fracture toughness/fracture energy of the highly porous materials:simple 1D to 2D homogenous to 2D cellular to 3D homogenous.

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Summary: CRADA has industry wide impact affecting DPF design and implementation regen cycle strategies

- **Relevance:** Property data generated by this CRADA is input into models accurately to predict DPF behavior during regenereation. In turn, strategies to mitigate thermal stresses can be formulated for optimized regeneration which *changes engine combustion regimes* and which minimizes failures, *improving cost-effective emission control*.

- **Approach/Strategy:**
  - Characterize the materials properties and microstructure of the ceramic DPF substrates supplied by Cummins.
  - Refinement of DPF service lifetime prediction models (Cummins).

- **Technical Accomplishments:**
  - Precise measurement of elastic modulus is important as it affects the lifetime prediction of structures such as DPFs.
  - Non-linear tensile behavior of porous microcracked ceramics that will lead to revised constitutive relations.
  - If failure in this class of materials is controlled by stress or strain. If failure is controlled by strain, AT may be considered an attractive material for higher performance DPF substrates.
  - 2D models developed; 3D models developing (presentation)

- **Collaborations and Coordination with Other Institutions:** Cummins and the Diesel community can input these materials properties into their in-house models

- **Proposed Future Work:** Investigate fracture toughness testing methods; measure the transverse moduli; examine a 4th alternate DPF material (Zeolite)

Technical Backup slides

Return on Investment: An example

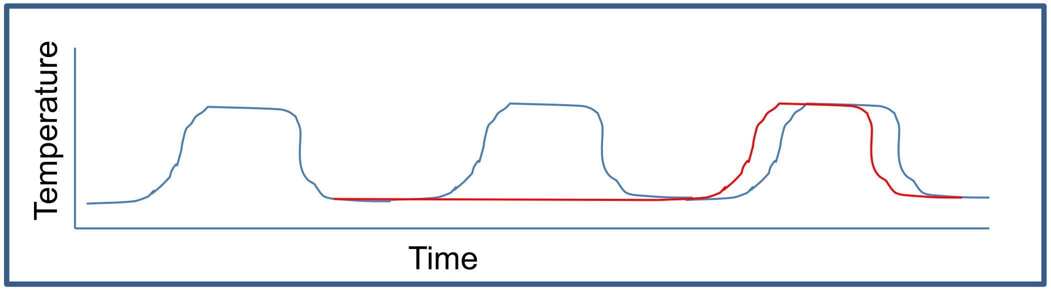
(Is there an ultimate payoff in energy demand reduction, and if so, can it be quantified?)

- Optimized regeneration cycle

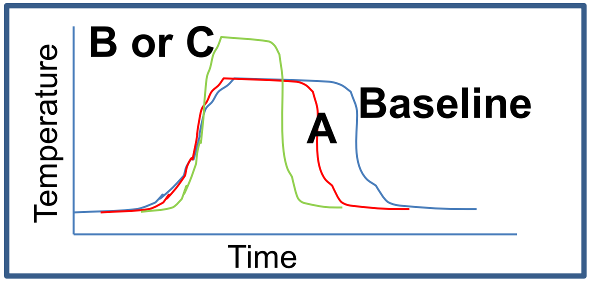
- Fuel economy impact of a particulate filter is driven by:
  - Frequency of regeneration cycle
  - Length of regeneration cycle
  - Temperature of regeneration cycle
  - Total backpressure on engine
  - Backpressure rise with soot loading during operation

Fleet fuel economy affected by material choice

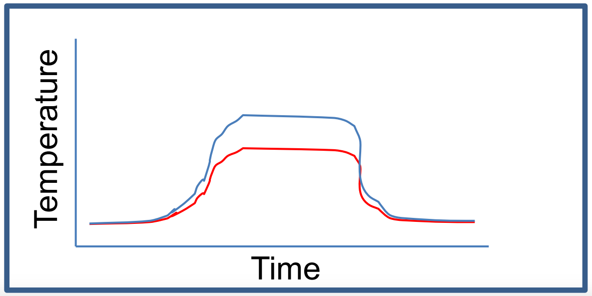
- Levers for specific fuel savings for heavy duty vehicle
  - Model calculations based on steady state rated conditions with 575°C regen and assumption of 1 gallon fuel consumed /regeneration and MPG of truck is 8.
  - Case 1 - Improved soot capacity (longer interval between regen's): Regen frequency
    - 1 regen instead of 2 per time interval
    - Fuel saving ~ 600 gal over rated life (1M miles)
    - 8MPG➔125k gal➔0.48% saved/truck



  - Case 2 - Improved robustness (higher temperature): Reduced duration
    - Baseline
    - A – baseline T, 16% shorter regen
    - B – 20% higher T, 33% shorter regen
    - C – 20% higher T, 50% shorter regen
    - Fuel savings: A 200 gal, B -100 gal, C 200 gal



  - Case 3 - Improved catalyst (lower temperature, burn less fuel): Reduced temperature
    - 20 % lower T • Fuel saving ~ 750 gal



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Expected Impact: (With a successful conclusion, what will the impact be? How will these results get used in industry?)

Public

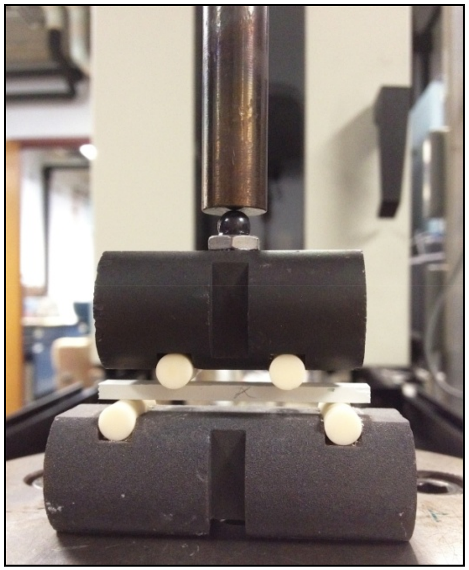
- Materials properties have been shared publicly at conference presentations and publications. Both Cummins and Corning have used these results to improve the durability of the aftertreatment structures.
- Release of ASTM standard C1674 (developed as part of this CRADA) which is being used by manufacturers and end users to share and compare strength data for the aftertreatment materials.
- Development of fracture toughness test to characterize cracking progression and fracture energy which is used in models.

Proprietary

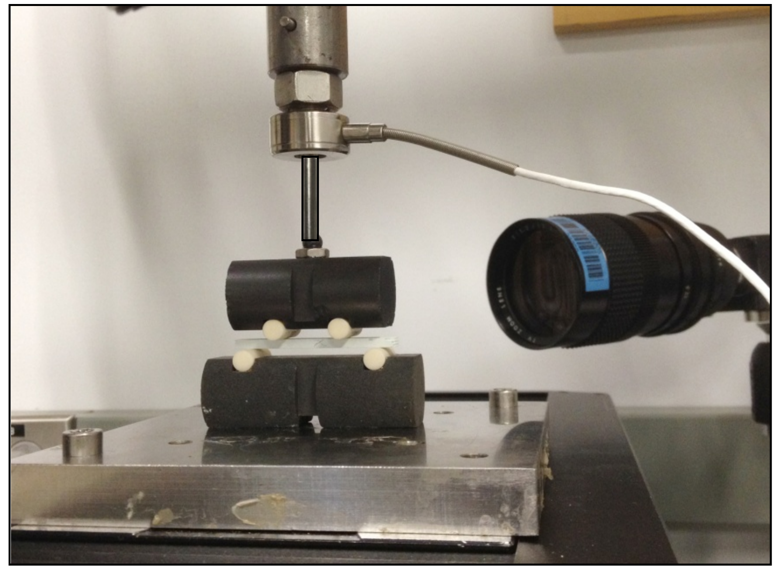
- Third result is progression of models from simple 1D to 2D homogenous to 2D cellular to 3D homogenous.

Remaining Challenge: Low modulus and very thin wall (<200μm) impose a great challenge to mechanically test the zeolite DPF specimens

Zeolite material under the loading pins tends to deform/crush first prior to crack initiation in the tensile stress region



Instron Bench Top Test Machine



Custom Made Micro Indentation with in situ monitoring optical microscope

Tests will be conducted with precision motion control plus in-situ monitoring system to reliably quantify the mechanical properties of Zeolite DPF

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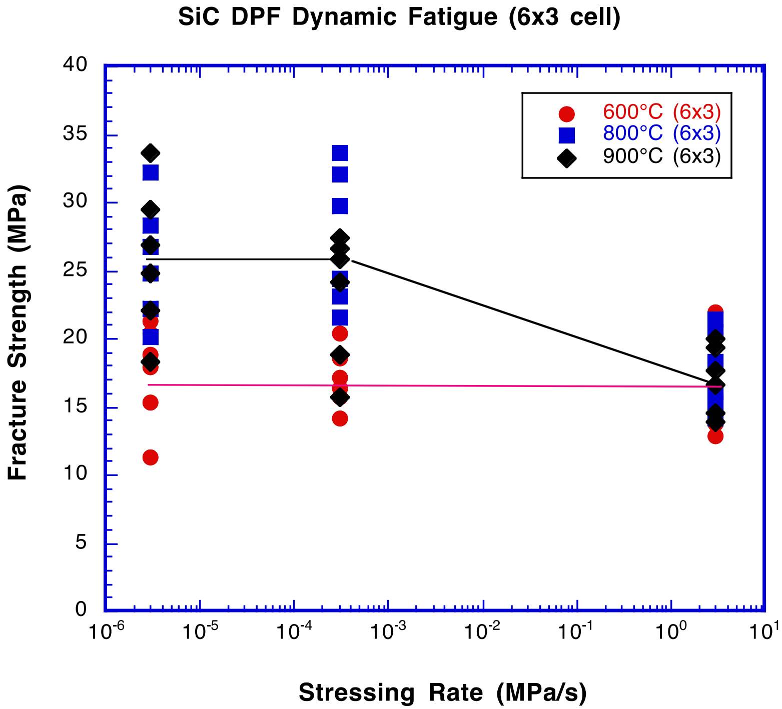
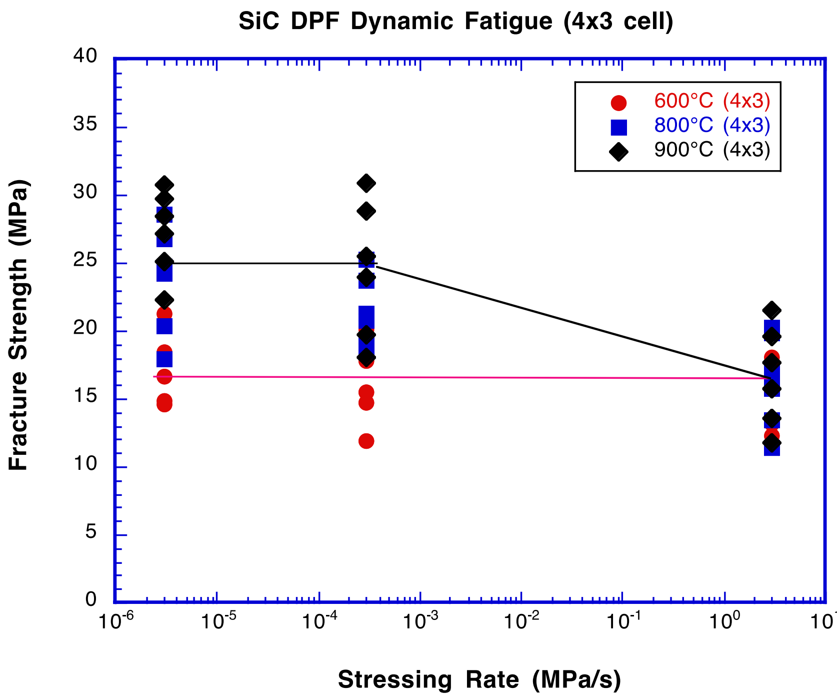
There is an Apparent Stressing Rate Effect at Temperatures ≥ 800°C

35-45% increase in fracture strength when tested at ≲ 0.003 MPa/s





No specimen (cell) size dependence on dynamic fatigue response

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