

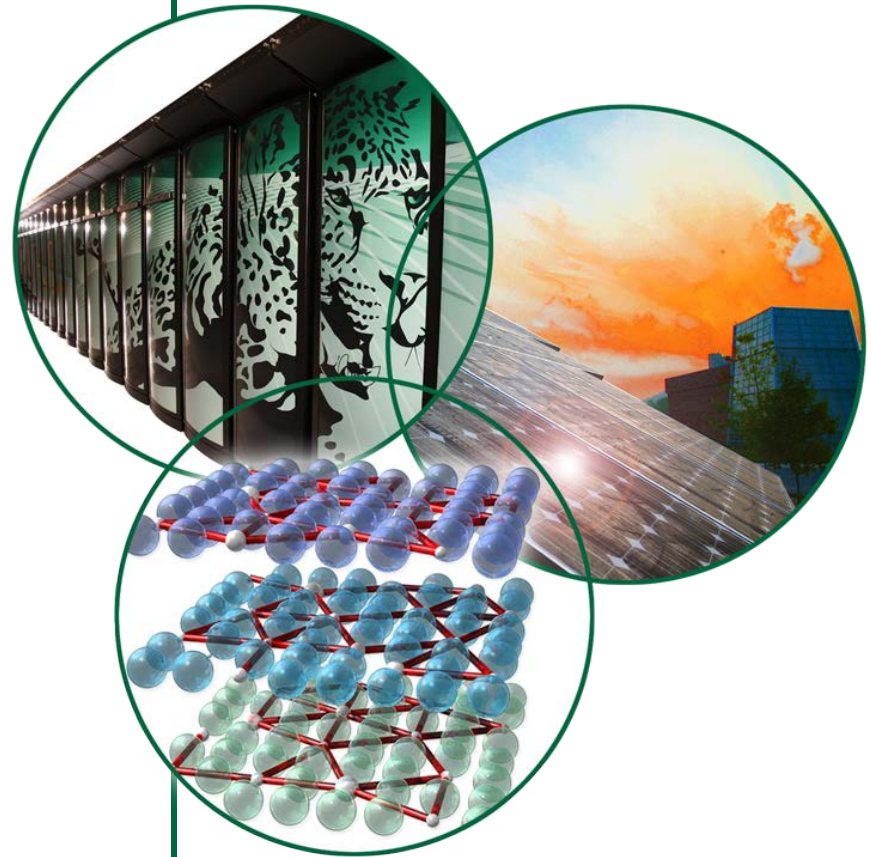
# Durability of Diesel Engine Particulate Filters

2012 DOE Vehicle Technologies Annual  
Merit Review and Peer Evaluation Meeting

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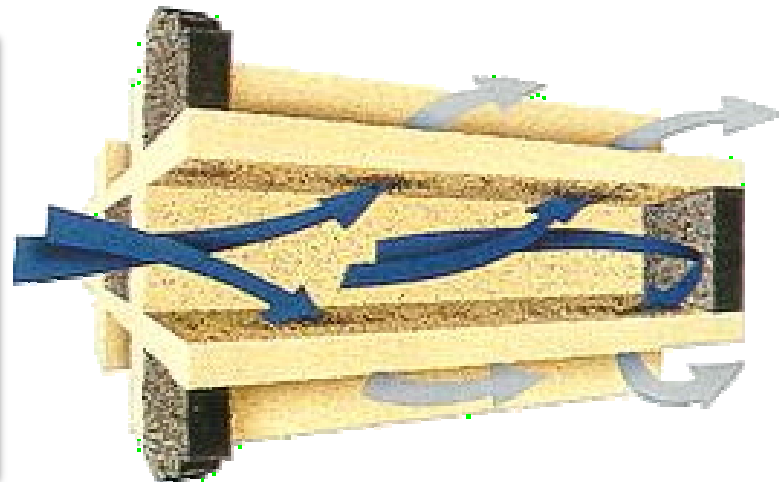
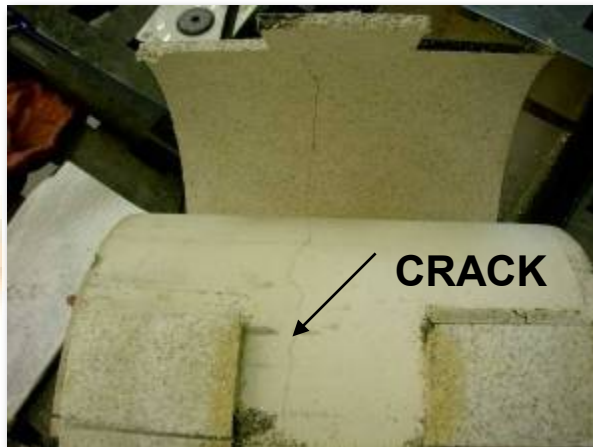
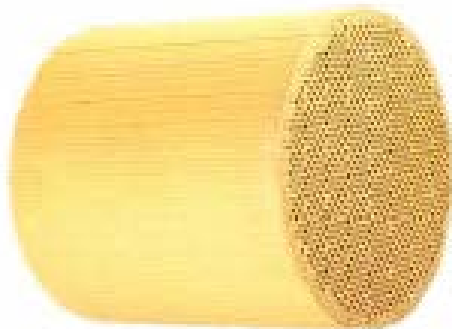
U.S. Department of Energy, Assistant Secretary for Energy Efficiency and  
Renewable Energy, Office of Vehicle Technologies Program



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# Background to CRADA

- Diesel Particulate Filters (DPFs) play a key role and will continue to be a key technology to meet the prevailing stringent regulations.
- Reliable operation for ~425,000 miles required. Reliability could be reduced due to damage induced by thermal stresses.
- Need for improved materials and designs along with life prediction models to optimize reliability and durability, particularly for DPF regeneration.
- Characterization of material properties is needed for model input
- **The CRADA has previously delivered key materials properties information, which enabled application and optimization of the prime path DPF technology, and contributed to down-selecting some alternative materials**
- SiC is considered here



# Overview

## Timeline

- Start: June 2004
- End: Sept. 2013
- 86% complete

## Budget

- Total Project funding
  - DOE-\$2.6M
  - Cummins-\$2.6M
- Funding received:
  - FY11 \$300k
  - FY12 \$300k approved

## Barriers\* - Propulsion Materials Technology:

- Changing internal combustion engine combustion regimes → Optimize to minimize fuel penalty & thermal stresses during regeneration
- Cost → reduce DPF failures & liability

## Barriers\* - Combustion and Emission Control R&D:

- Cost-eff. emission control → reliable regeneration w/ minimized fuel penalty
- Durability → Minimize thermal stresses and porosity, reduce failures & liability
- Market perception → Clean diesel improves public's acceptance

## Partners

- 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...
  - Is input into models to predict behavior accurately. 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
  - Allows for better DPF design which improves durability
  - Results in longer lasting DPFs reducing the liability and failures which reduce cost

# Relevance to Vehicle Technologies Goals

- **Propulsion Materials: 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\***
  - Understanding the relationship of the porosity to the material properties of the filter (and catalyst) substrates enables optimization of porosity, strength, elastic modulus, thermal conductivity, thermal expansion ... leading to improved regeneration strategies and thermal management and filter efficiency.
  - Increases acceptance of clean diesel by the public. Larger acceptance results in larger percentages of conversion to diesel, with the resulting reduction in petroleum consumption
- **Improve the modeling and simulation capabilities for exhaust aftertreatment devices to accelerate the design of the most effective emission control systems\***
  - Characterization of DPF materials provides needed material properties for accurate simulation

# Milestones FY11 & FY12

- **FY11: Continue to investigate the interaction and properties of washcoat, soot and substrate (focus here: SiC substrate properties) on properties of new and field tested DPFs.**
- **FY11: Initiate characterization of the dynamic and static fatigue response of SiC DPFs.**
- **FY12: Complete the determination of strength, fracture toughness, density/porosity/microstructure, and thermal expansion of uncoated DPFs as a function of time at elevated temperatures of 300, 500, 800 and 900°C for a second alternate substrate DPF material.**

# **Technical Approach/strategy:**

- **Application of probabilistic design tools, non-destructive evaluation (NDE) techniques and thermo-mechanical characterization methods to DPF ceramic substrates.**
- **Rank the thermal shock resistance of candidate DPF substrates.**
- **Refinement of DPF service lifetime prediction models based on characterization of field returned filters (Cummins).**

## **...addresses barriers:**

- **The above provides materials behavior and property data to models which optimize regeneration. This improves durability and reduces liability and fuel penalty making emission control more cost effective.**

# **Approach/strategy: Integration within Vehicle Technologies program**

- **Utilizes characterization tools acquired and maintained by the High Temperature Materials Laboratory (HTML) Program**
- **DPF substrate materials used in both DPFs and catalyst systems**

## **Objective**

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



# Background: Why investigate SiC-based DPF

- SiC has not been used in HD industry
- Many differences exist between the LD and HD applications (e.g., much tougher durability requirements)
- Inherent underlying material difference: cordierite and AT are micro-cracked; SiC is not, which was expected to translate into different responses to stress



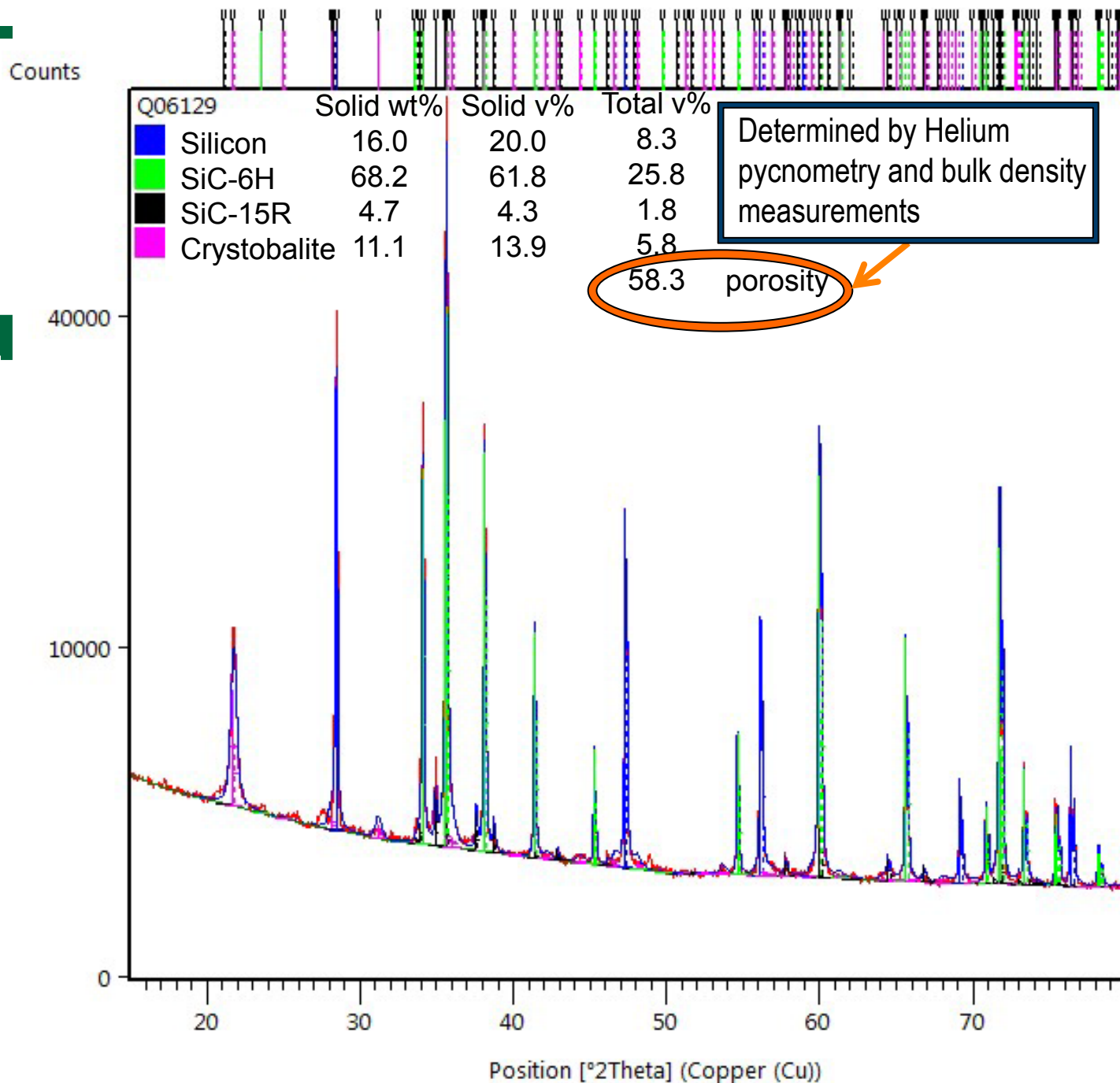
35 mm

Cell size = 1.1x1.1mm

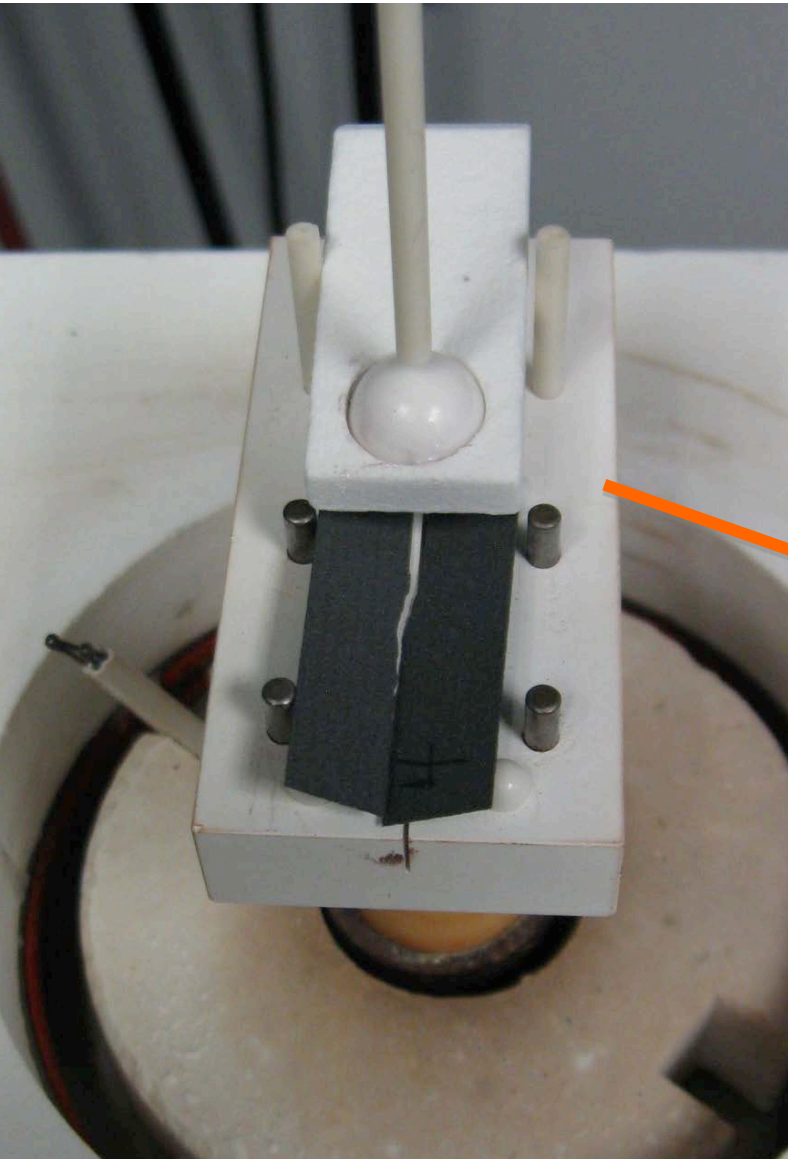
Wall thickness = 0.4 mm

# Tech.Acc. : XRD identified four phases; quantified using Reitveld analysis

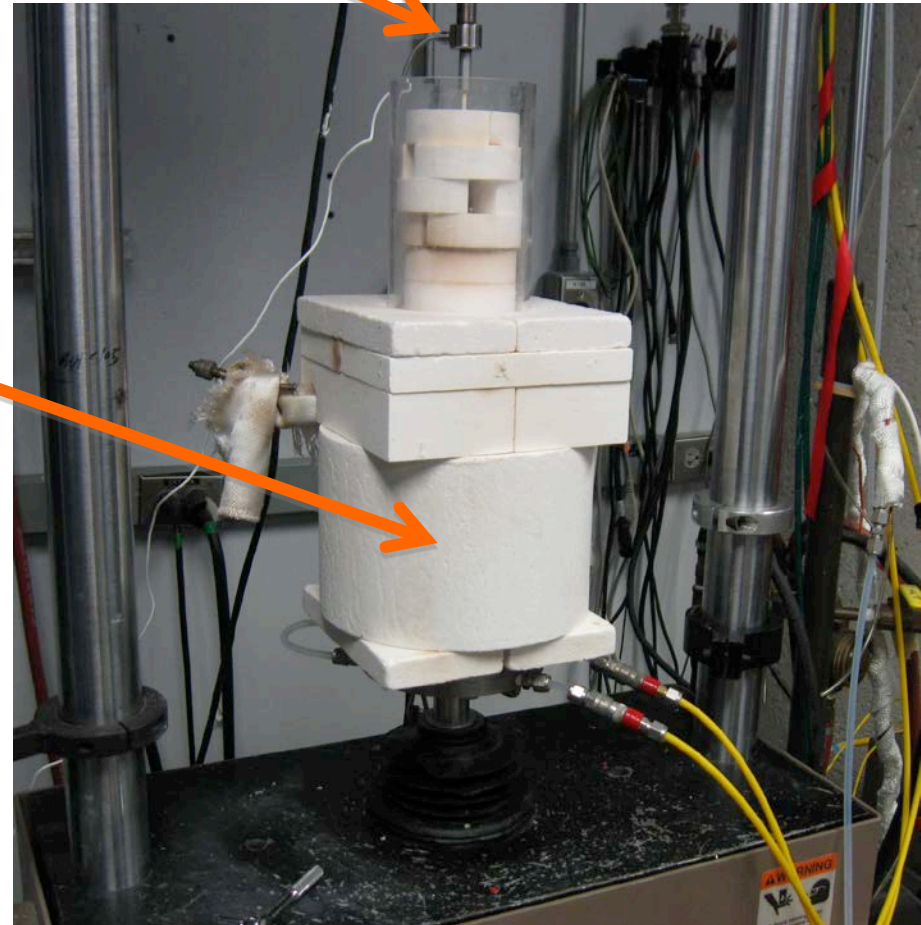
**High Si V% of  
this material  
qualifies it as a  
porous  
siliconized SiC  
(Si/SiC) or  
reaction  
bonded SiC  
(RBSC)**



# Technical accomplishment: High temp fracture toughness experimental Setup

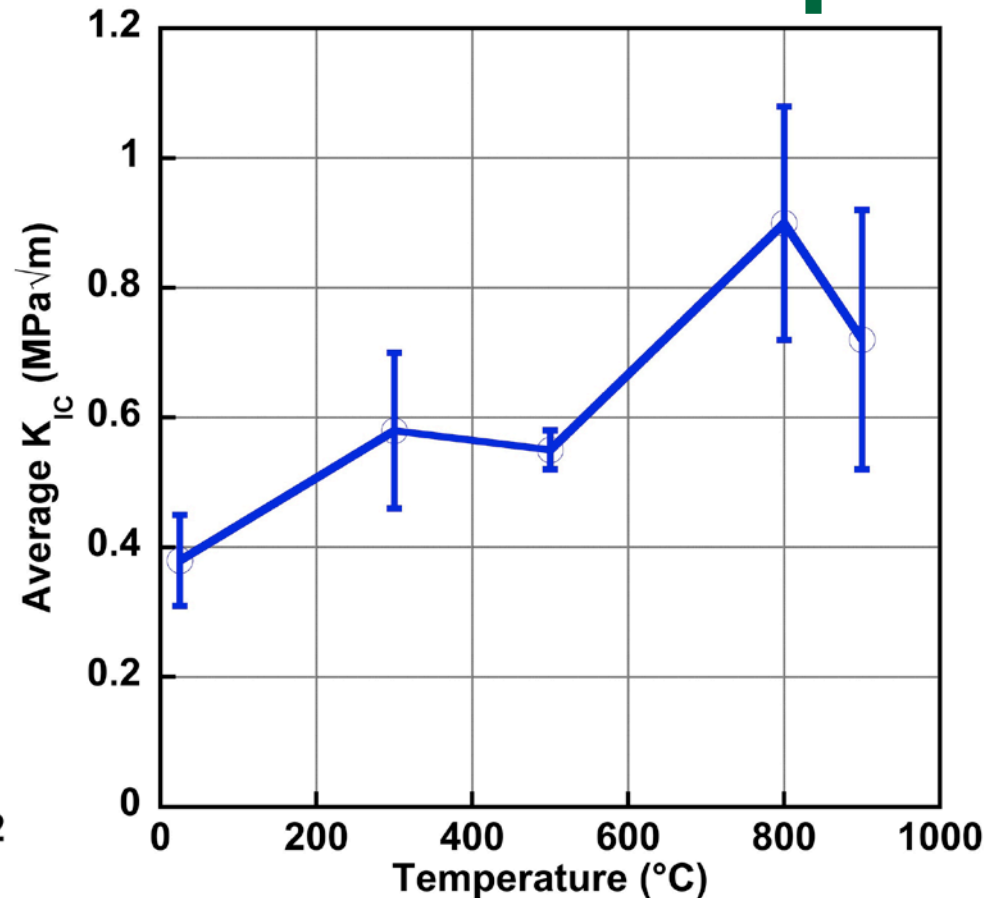
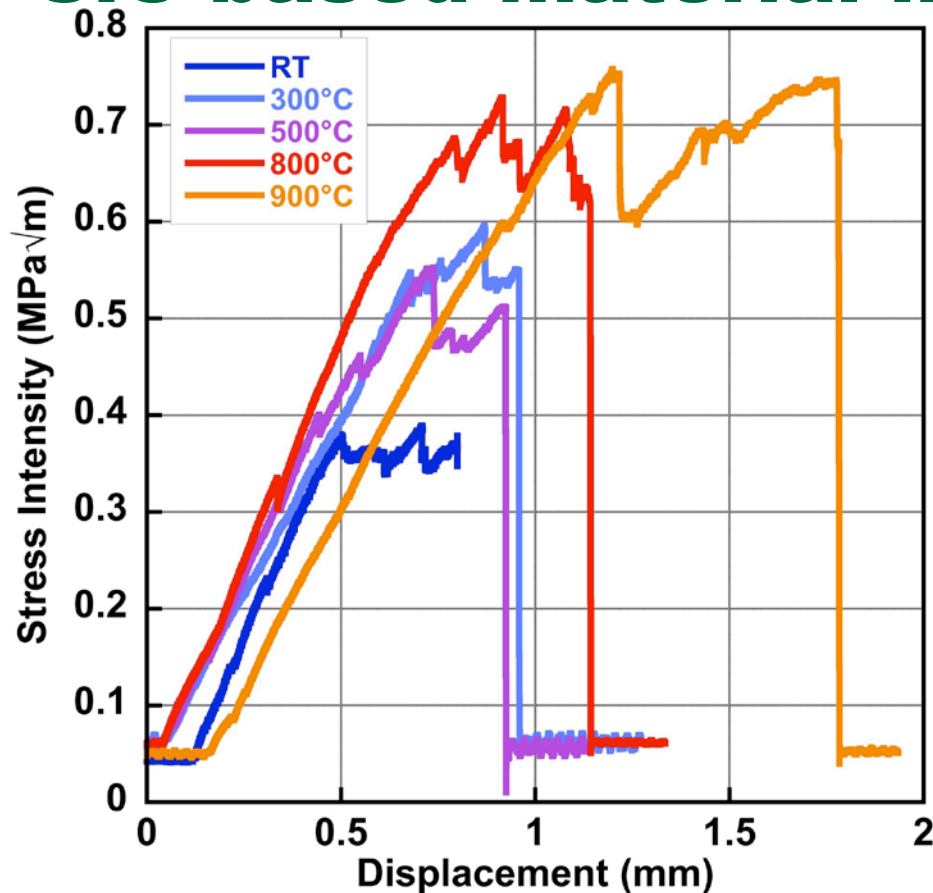


Load cell and  $\text{Al}_2\text{O}_3$  push rod





# Technical accomplishment: Toughness of SiC-based material increases with temp

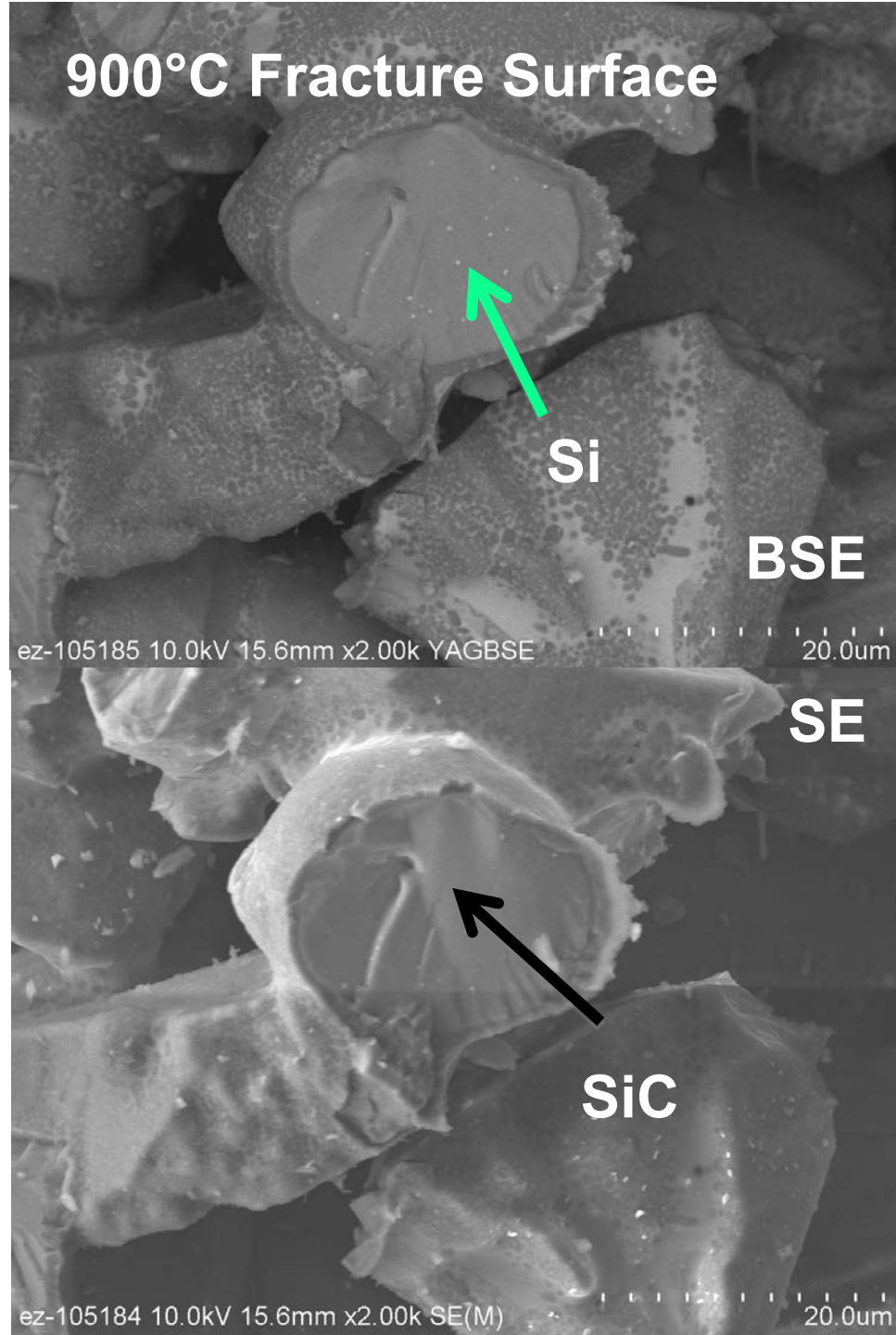


- Maximum load used to calculate  $K_{IC}$
- Non-zero start-finish  $K_I$  due to double torsion jig top load/weight
- Slopes suggest moduli are slightly lower at HT
- Toughening mechanism likely related to free Si.

# **Tech.accomp.: Examined fracture surfaces to look for evidence of toughening mechanisms**

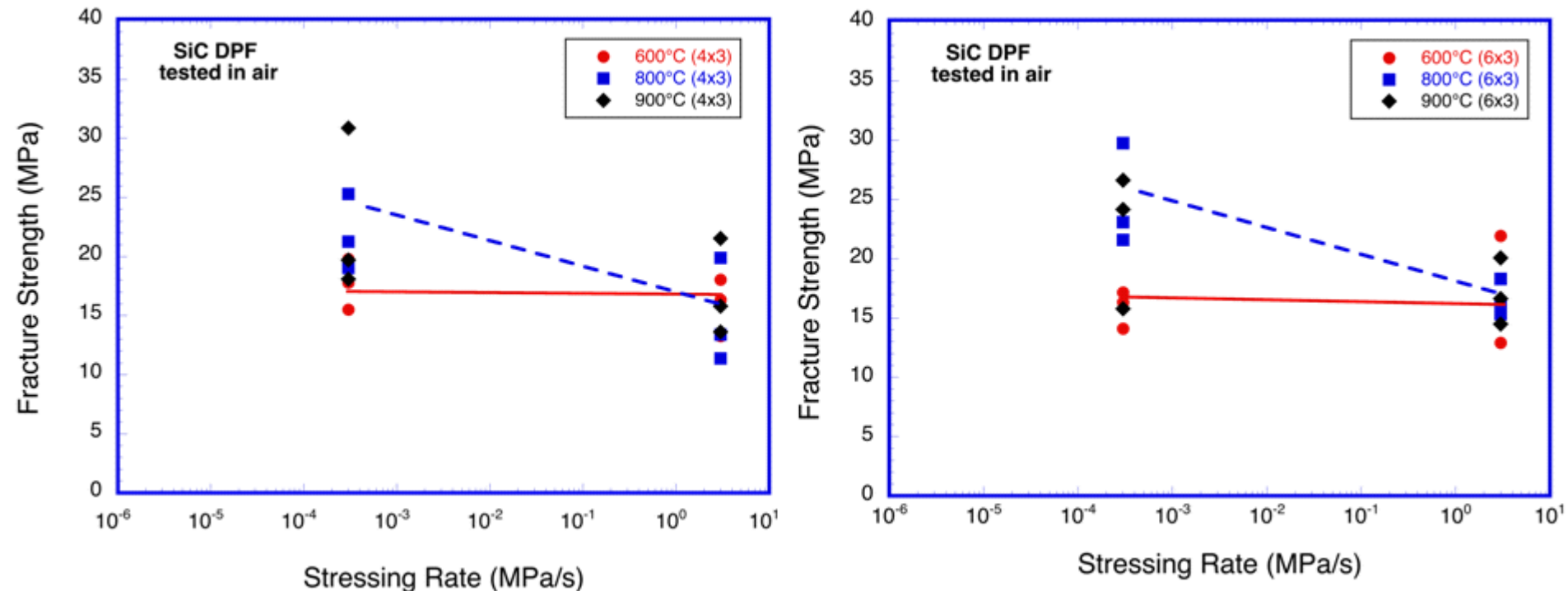
- RT and HT fracture surfaces of double torsion samples were similar, except for distinct oxidation layer in 800 & 900°C samples
- Simple static test showed permanent deformation after 24 hrs at 900°C
- Toughening mechanisms likely include:
  - Crack tip blunting, healing
  - Oxidation layer and/or possibly glass formation to close or fill in cracks/flaws
  - More in Reviewer Only slides

## **900°C Fracture Surface**



# Tech.Acc.: There is an apparent stressing rate effect at temperatures $\geq 800^{\circ}\text{C}$

$\dot{\sigma}$ : 35-45% increase in fracture strength when tested at 0.003 MPa/s (typically this decreases in most materials)



T: 800 & 900 C strengths larger than 600 C at slowest  $\dot{\sigma}$   
Size: No dependence with fracture strength was observed

# Typical Si/SiC strength behavior with temperature\*

\*After:  
Larson and  
Adams,  
AFWAL-TR-  
83-4141,  
1983.

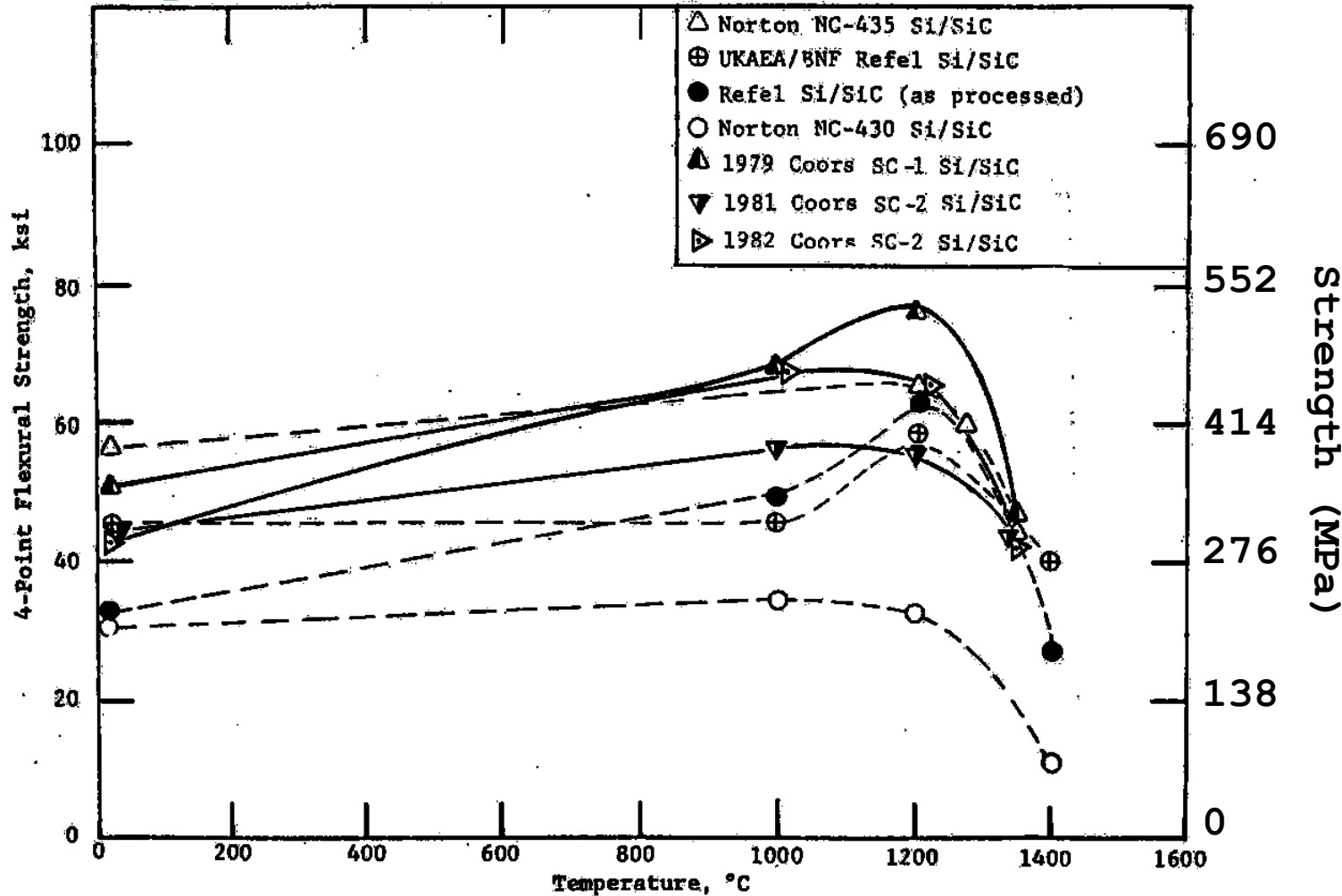
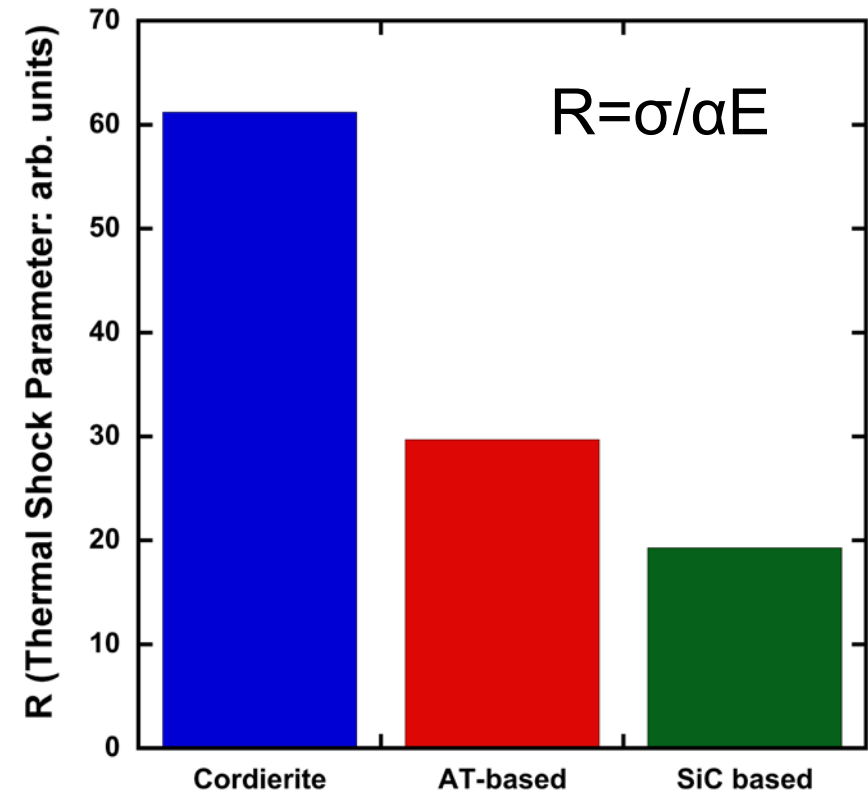
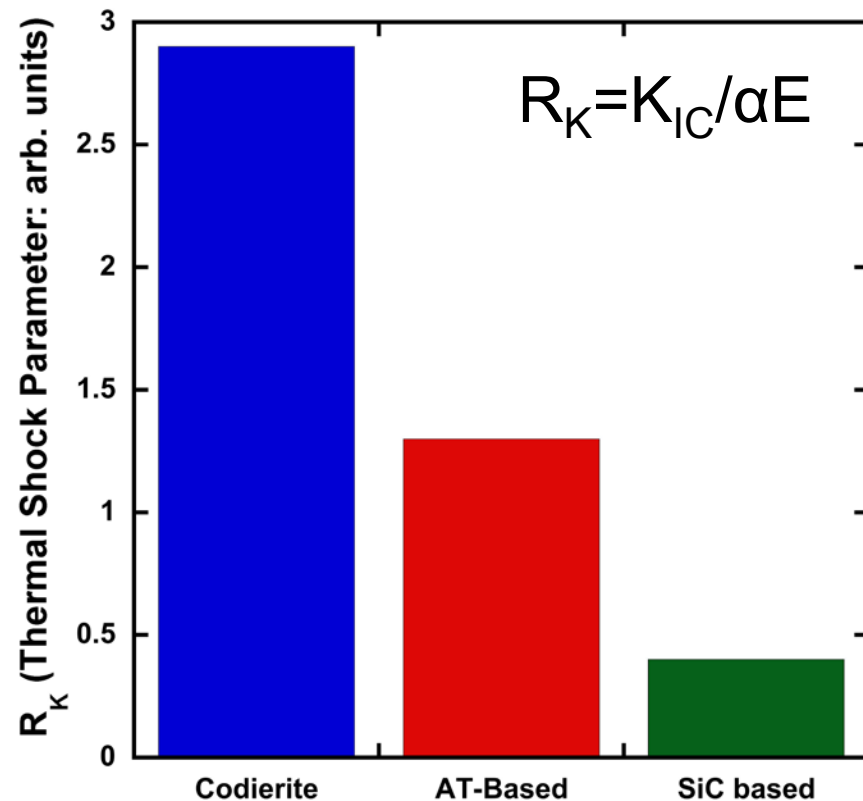


Figure 64. Flexural strength of siliconized silicon carbide materials.

Strength ↑ with T: Tressler et al. (1985,1993); Larsen et al. (1979,1983); Tomlinson et al. (1992); Breder (1995); Huang&Zhu (2005)

# Tech.Acc.: New thermal shock ranking methodology compared to traditional




- Trends are very similar; measured properties used
- $R_K$  - Easier to perform fracture toughness tests than strength (less material and fewer samples)
- Note that the above analysis is highly simplified; assumes:
  - Homogeneity
  - Isotropy

† Data is FY09-FY11 work

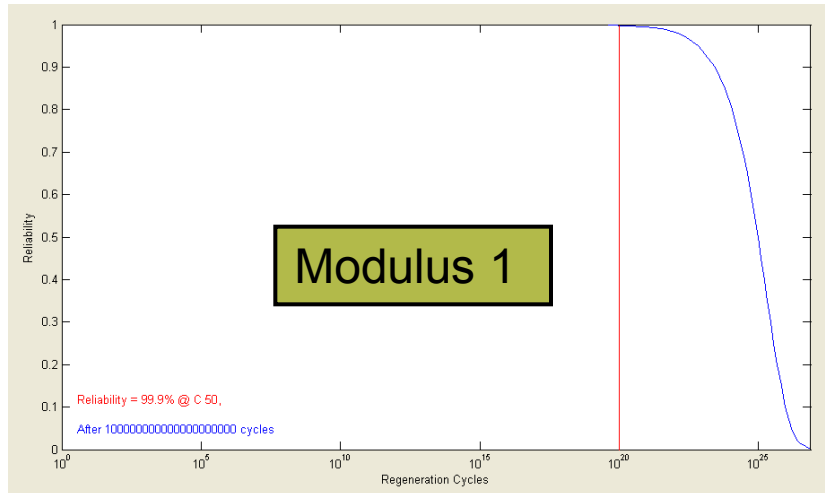


# **Collaborations and coordinations with other institutions: CRADA Partner**

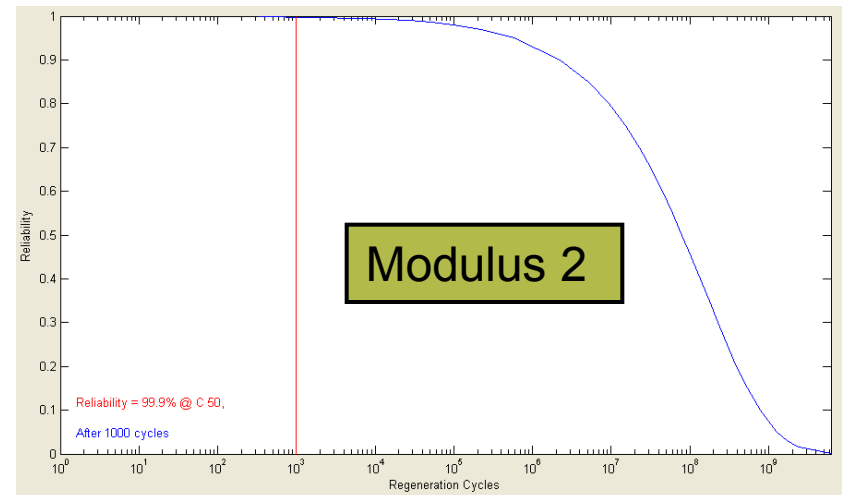
-  (Industry):
  - Cummins' role is to collaborate and guide the work along the most useful path to achieve durability, cost and emissions targets
  - Supplies samples
  - Share experimental results on samples
  - Exchange of technical information to assist with each others analyses
  - Face to face meetings at least 2X/year

# Collaborations and coordinations with other institutions: Tech transfer

- CRADA data used to translate thermal maps into ANSYS stress models and inputs to life prediction code.
- Different stress results (from different Young's modulus inputs) give very different life predictions.
- More work to understand this effect of modulus measurement is necessary to optimize the stress and life models.



99.9 % component will survive up to  **$1 \times 10^{20}$**  cycles.



99.9 % component will survive up to **1000** cycles.

# Future Work

- **Continue to characterize and understand the differences in measurement techniques for Young's moduli.**
- **Continue to characterize the dynamic and static fatigue response of SiC DPFs (FY11-13).**
- **Complete the determination of strength, fracture toughness, density/porosity/microstructure, and thermal expansion of coated DPFs as a function of time at elevated temperatures of a second alternate substrate (FY12 & 13).**

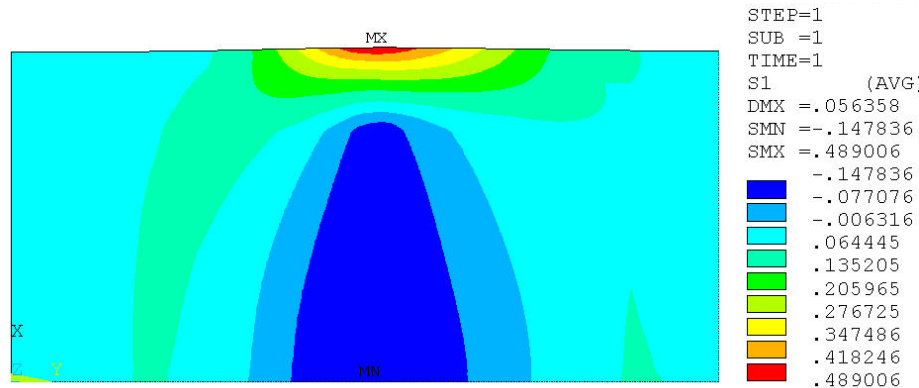
**Summary: Property data support modeling which optimize regeneration which changes engine combustion regimes, improves durability, efficiency; reduces cost...VT goals**

- Data is input into Cummins models and used to modify regeneration regimes to reduce the fuel penalties and improve filter durability (minimize thermal stresses).
- Investigated SiC-based materials as an alternate DPF material to cordierite
- Completed the determination of fracture toughness and microstructure of uncoated DPFs as a function of time at elevated temperatures of 300, 500, 800 and 900°C for SiC-based DPF materials.

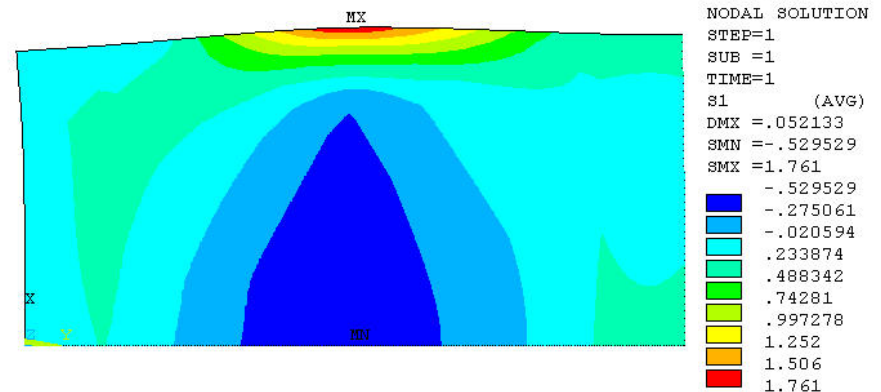
# Technical Backup slides

# Collaborations and coordinations with other institutions: Tech transfer2

- The modulus for stress model was measured by RUS and sonic resonance.
- Values were corrected for porosity and temperature effects.



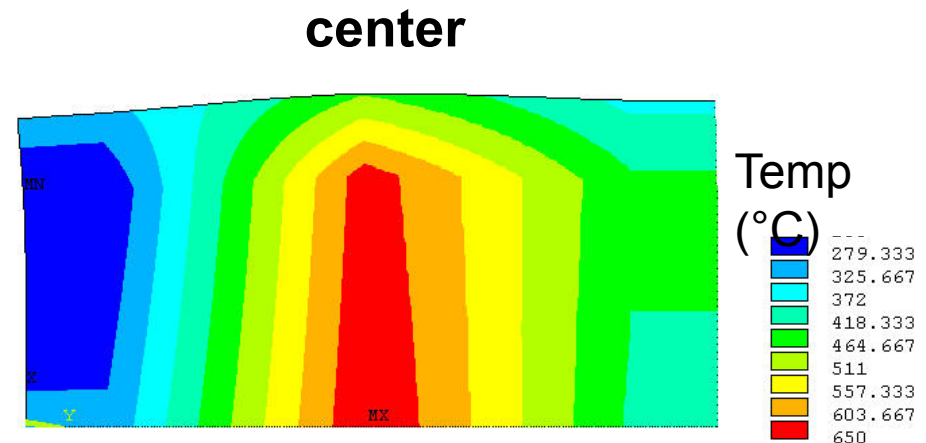
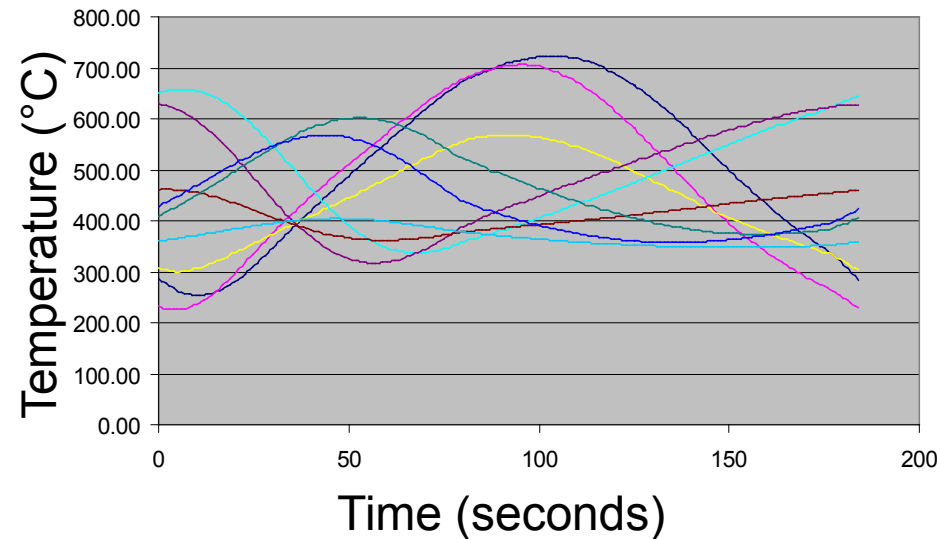
Modulus 1



Modulus 2

# Collaborations and coordinations with other institutions: Tech transfer<sup>3</sup>

- Efforts contributed to refinement of aftertreatment systems Dodge Ram Pickup truck
- CRADA data used to translate thermal maps into ANSYS stress models and inputs to life prediction code.



Thermal cycle temperature distribution (°C)

Temperature distribution at t = 0, (°C)

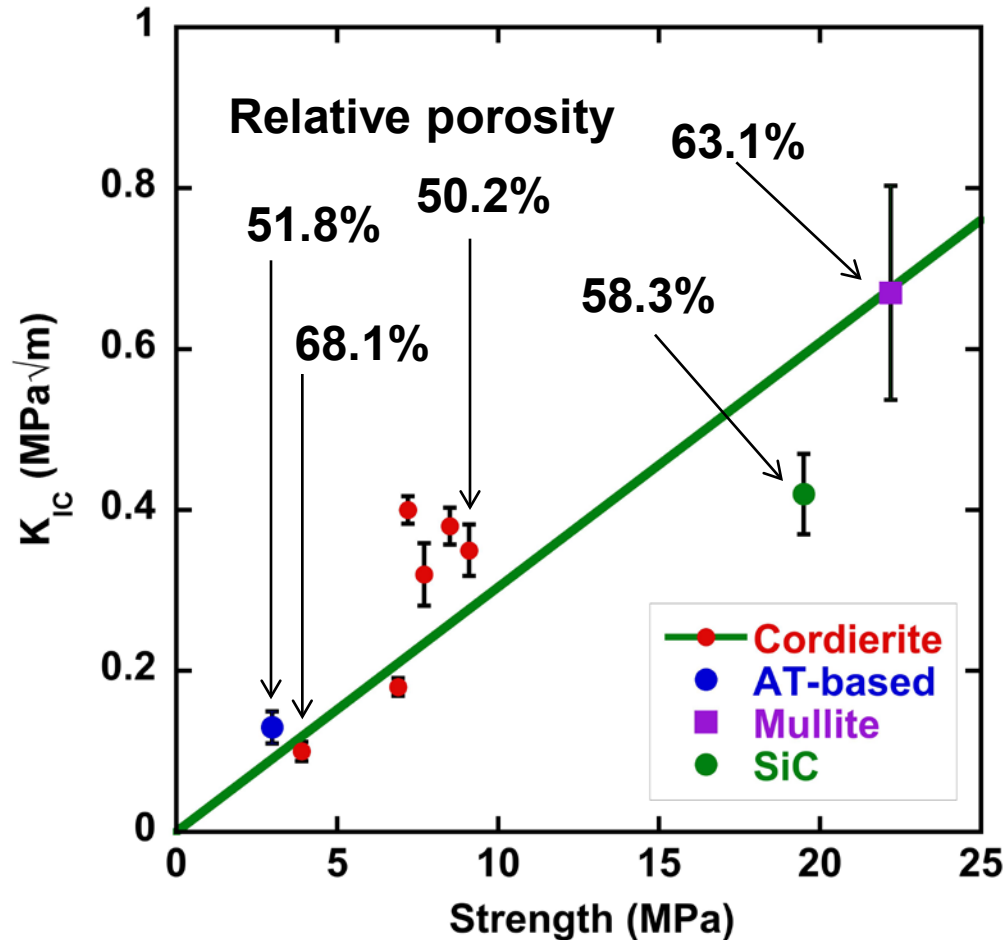
# **Literature for Si/SiC and RBSC heat exchangers suggest mechanisms for $K_{IC}$ increase with temp**

- Toughening mechanism(s) attributed to plastic/softening behavior of free Si at  $T > 1000^{\circ}\text{C}$  in dense materials
  - Crack tip blunting, healing 1
  - Stress redistribution due to grain cluster movement 1,3,7
  - GB softening, sliding 3,6,2
  - Creep cavitation (hard to see here) 1,3
- Behavior is dependent upon volume fraction of Si, temperature, time at temperature and stress level 1,4
- Oxidation and/or possibly glass formation to close or fill in cracks/flaws 5,6

1. Tressler et al. (1985,1993); 2. Larsen et al. (1979,1983); 3. Wiederhorn et al. (1988,1992,1996); 4. Chakrabarti&Das (2001); 5. Tomlinson et al. (1992); 6. Huang&Zhu (2005); 7. Breder (1995).



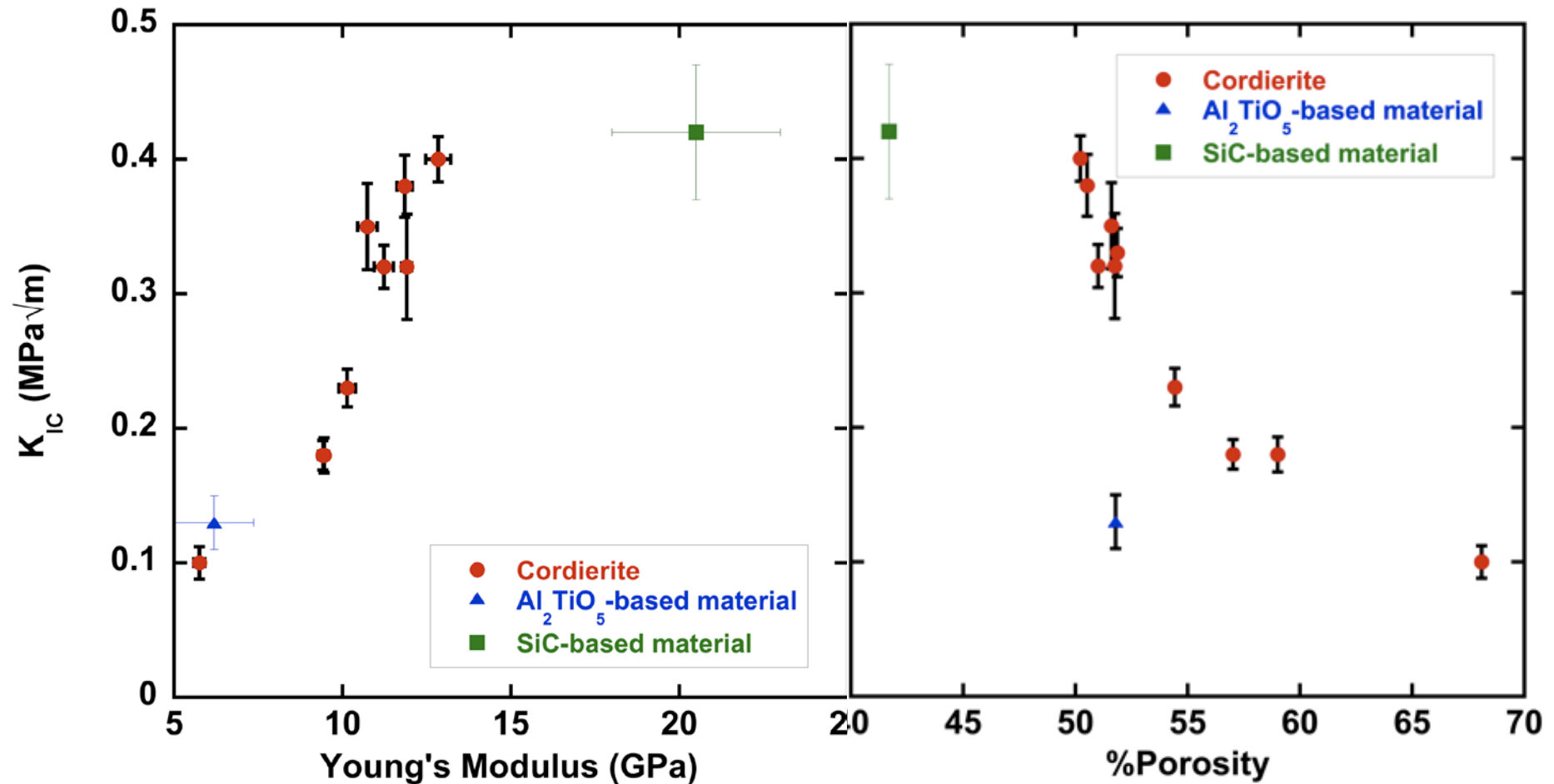
# Porosity dominates Properties: e.g. $K_{IC}$ – Strength Correlation



- Strength values for honeycomb bars corrected for Moment of Inertia (MOI)
- Increasing fracture toughness leads to increasing strength
- It is easier to perform fracture toughness tests compared to strength tests (fewer specimens and less material)

† Cordierite & mullite data is FY09 work; AT-based data is FY10 work

# $K_{IC}$ of porous DPFs appear to have a semi-linear relationship with Young's modulus and porosity



† Cordierite & mullite data is FY09 work; AT-based data is FY10 work