

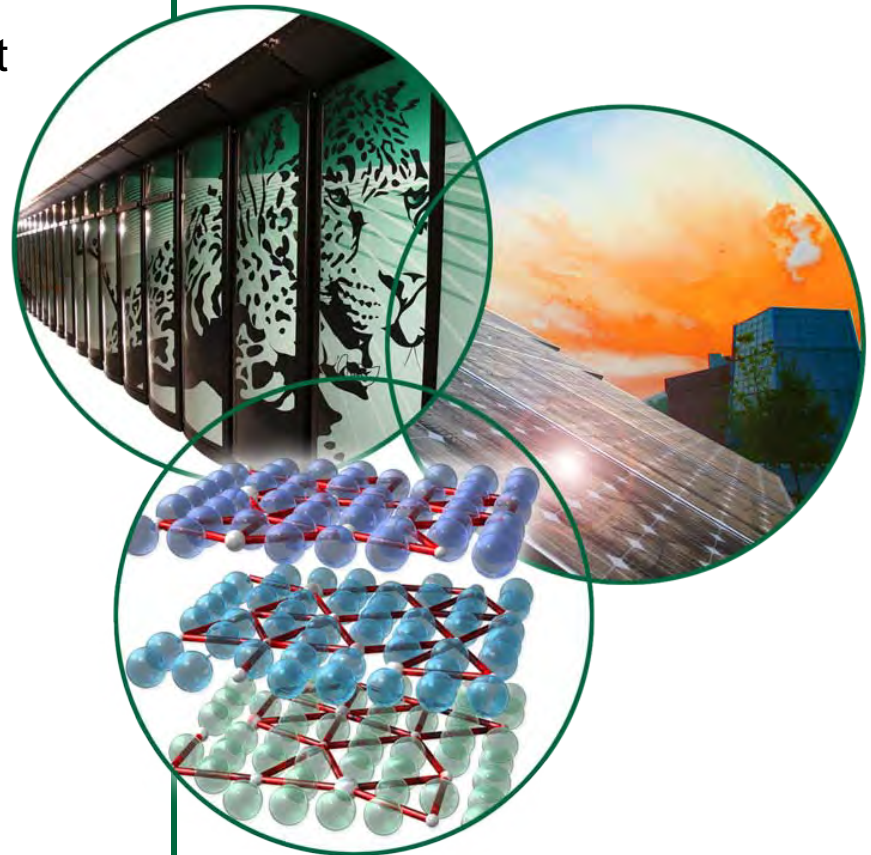
Durability of Diesel Engine Particulate Filters

2011 DOE Vehicle Technologies Annual Merit Review and Peer Evaluation Meeting

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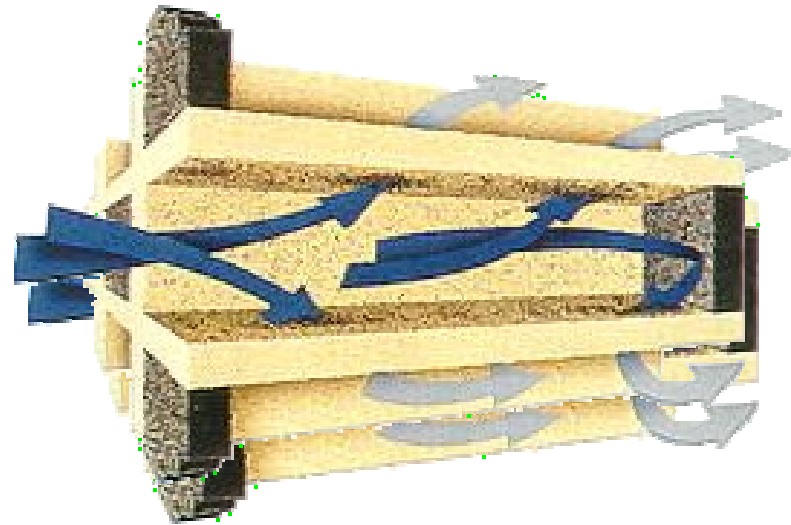
U.S. Department of Energy, Assistant Secretary for Energy Efficiency and Renewable Energy, Office of Vehicle Technologies Program



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Background

- 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



Overview

Timeline

- Start: June 2004
- End: Sept. 2013
- 75% complete

Budget

- Total Project funding
 - DOE-\$2.6M
 - Cummins-\$2.6M
- Funding received:
 - FY10 \$300k
 - FY11 \$300k approved

Barriers* - Propulsion Materials Technology:

- Changing internal combustion engine combustion regimes → Optimize to minimize thermal stresses during regen.
- Cost → Precious metal content

Barriers* - Combustion and Emission Control R&D:

- Cost-eff. emission control → reliable regen.
- Poor durability → Thermal stresses and porosity
- Market perception → Understanding improves public's acceptance

Partners

- Cummins Inc.
- Corning Inc.

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

Objective

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

Relevance to barriers

- **Impact on barriers: Property Data...**

- Input for models to predict behavior accurately. In turn, strategies to mitigate thermal stresses can be formulated for optimized regeneration which *changes engine combustion regimes*
- Predictable behavior allow for better DPFs designed which improves *durability*
- Improved strategies minimize loss, save precious metals improving *cost-effective emission control*
- Net result of above is longer lasting DPFs reducing the amount of precious metals needed or *cost*

Relevance to Vehicle Technologies Goals

- **Advanced Combustion Engine R&D: By 2015, develop materials that reduce the fuel economy penalty of particle filter regeneration by at least 25% relative to the 2008 baseline***
 - Understanding the relationships of the material properties for the filter (and catalyst) substrates enables optimization of porosity, strength, elastic modulus, thermal conductivity, thermal expansion ... leading to thermal management and improved 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
- **Achieve engine system cost, durability and emissions targets***
 - Thrust is to characterize and improve the durability, resulting in the lowest overall cost and preventing emission release in service.

Milestones

- **Milestone10: Continue to determine the change in thermal shock resistance of field tested DPFs and the thermal shock resistance of a second alternate substrate DPF material.**
- **Milestones11:**
- **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.**
- **Initiate characterization of the dynamic and static fatigue response of SiC DPFs (latter part FY11).**

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 regen. This improves durability 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**

Measured DPF properties reflect high porosity

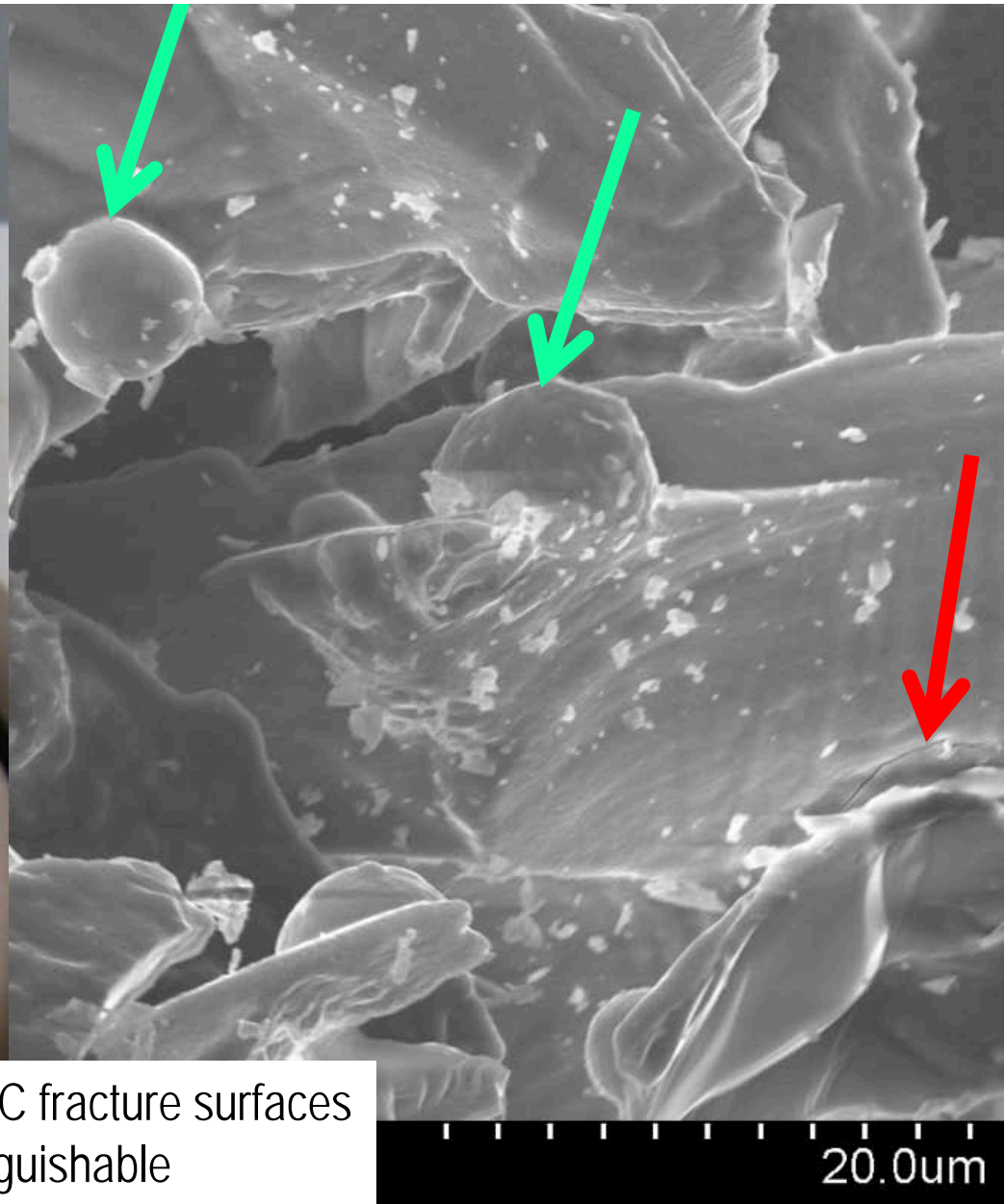
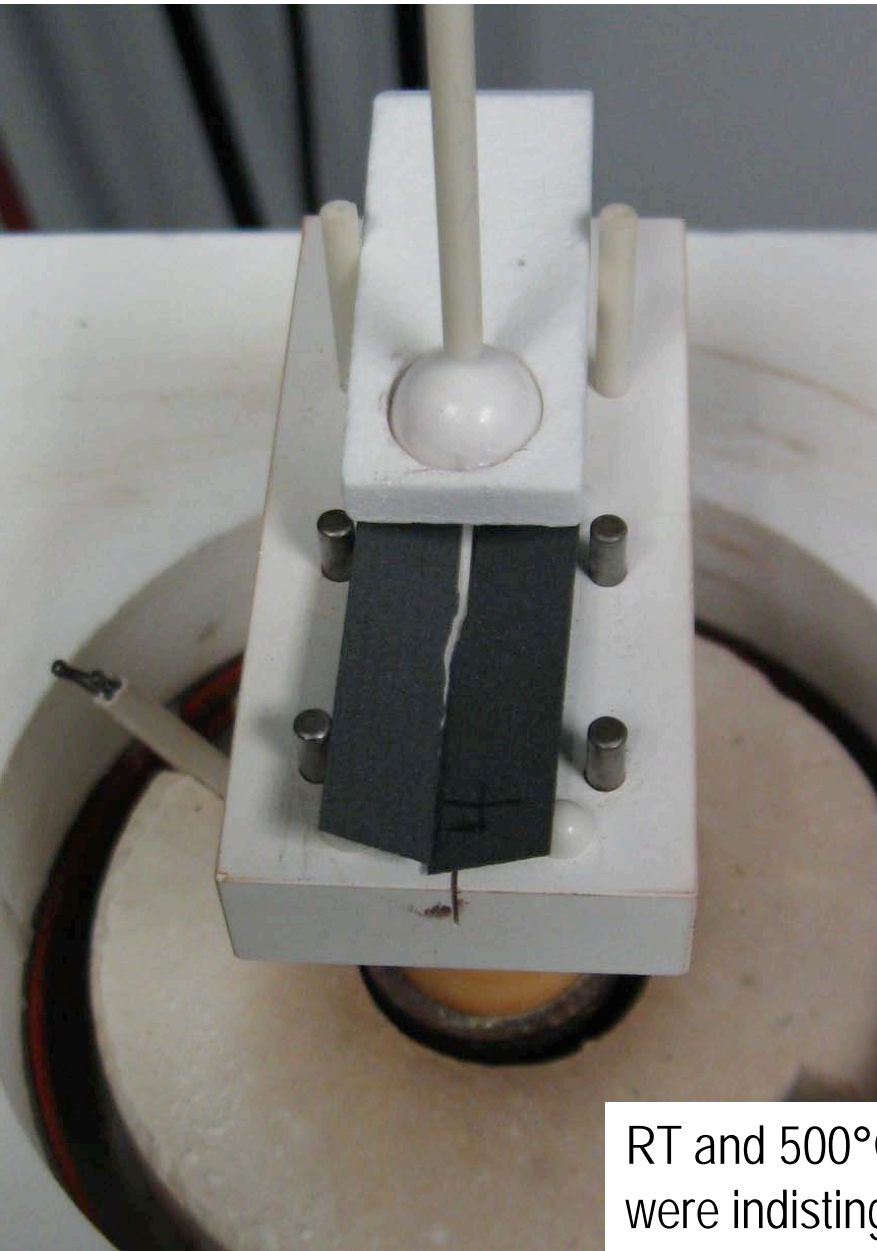
Property/Material	Cordierite [†]	Al ₂ TiO ₅ ^{††}	SiC
Porosity (%)	50.4	51.9	58.3
Young's Modulus (GPa)*	16.2(0.2)	6.19(1.17)	20(2.5)
CTE parallel [¢] (x10 ⁻⁶ 1/°C)	0.48	1.07(0.15)	5.00(0.04)
CTE perpendicular ^{¢¢} (x10 ⁻⁶ 1/°C)	0.99	2.17(0.15)	5.10(0.08)
Strength (MPa) @ RT [¥]	7.33(0.45)	2.98	19.5(0.63)

***DMA (Dynamic Mechanical Analysis); MOI corrected; 2-5 samples, Each remounted ≥ 5 X**

**Linear coefficient of thermal expansion, RT -1000°C
¢parallel and ¢¢perpendicular to the extrusion direction**

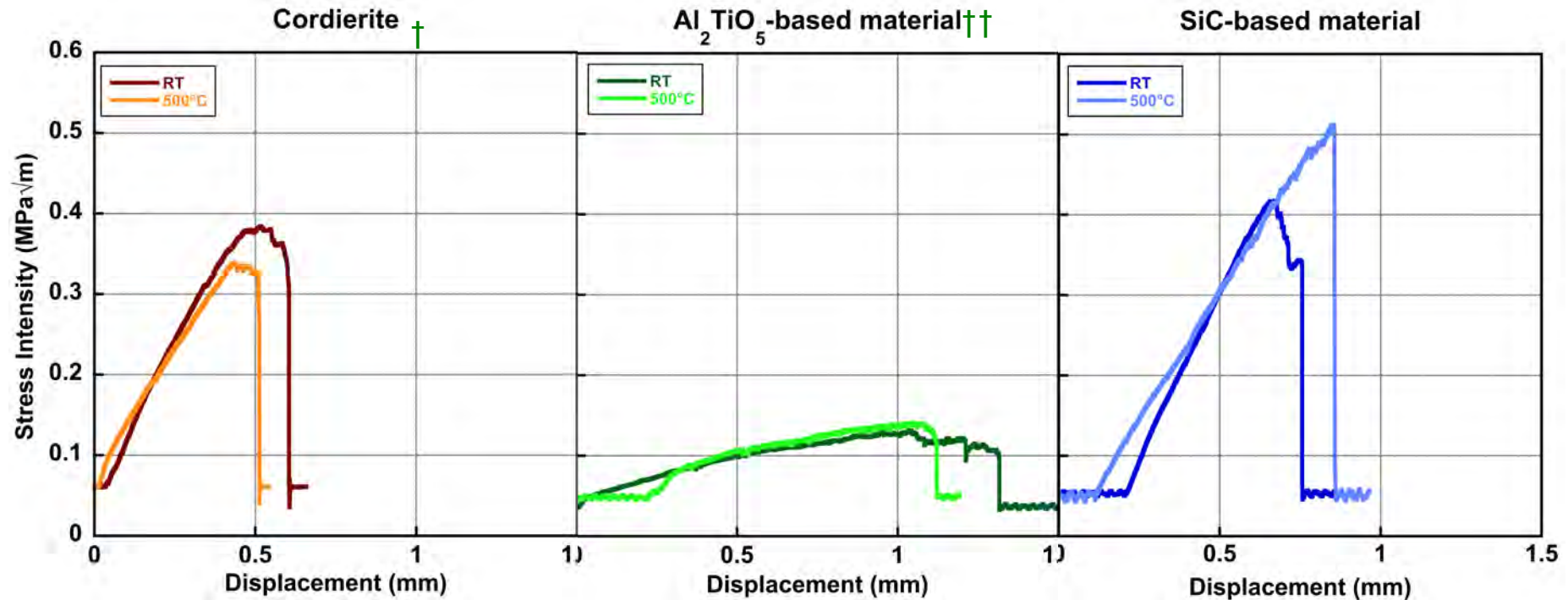
¥ Strength MOI corrected

K_{IC} fracture surface displays nodules in SiC-based material that are silicon rich



RT and 500°C fracture surfaces
were indistinguishable

Double torsion method used to measure apparent fracture toughness as crack length not needed for determination^{¥¥}



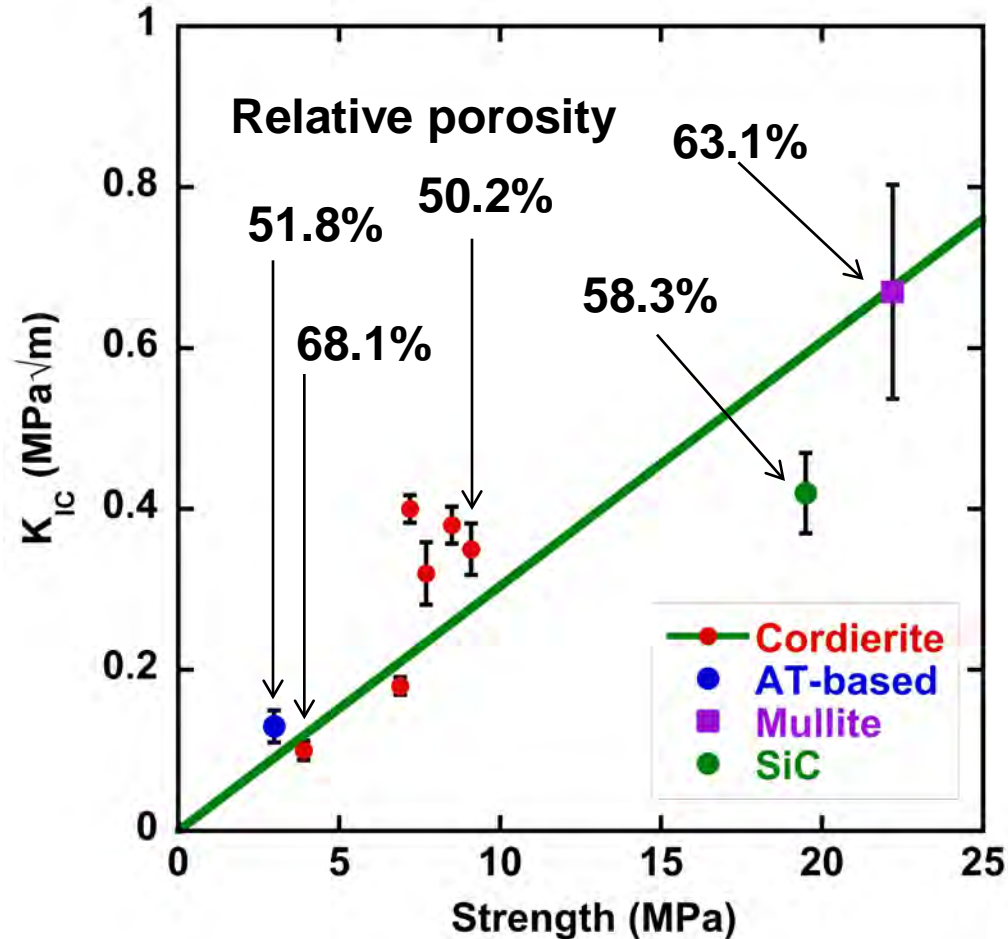
Ave K _{Ic} @RT	0.31(0.02)	0.13(0.02)	0.42(0.05)
Ave K _{Ic} @500°C	0.25(0.04)	0.16(0.02)	0.55(0.03)

- Temperature has negligible effect on toughness of cordierite and AT-based materials; highly microcracked
- Mechanism for increase in toughness for SiC-based material attributed to behavior of free Si (similar to RBSC between RT and 800°C[£])
- Slopes suggest moduli the same or slightly lower at HT

[†] Cordierite data is FY09 work; ^{††} AT data is FY10 work

^{¥¥}Shyam & Lara-Curzio, (2006); [£] Huang & Zhu (2005)

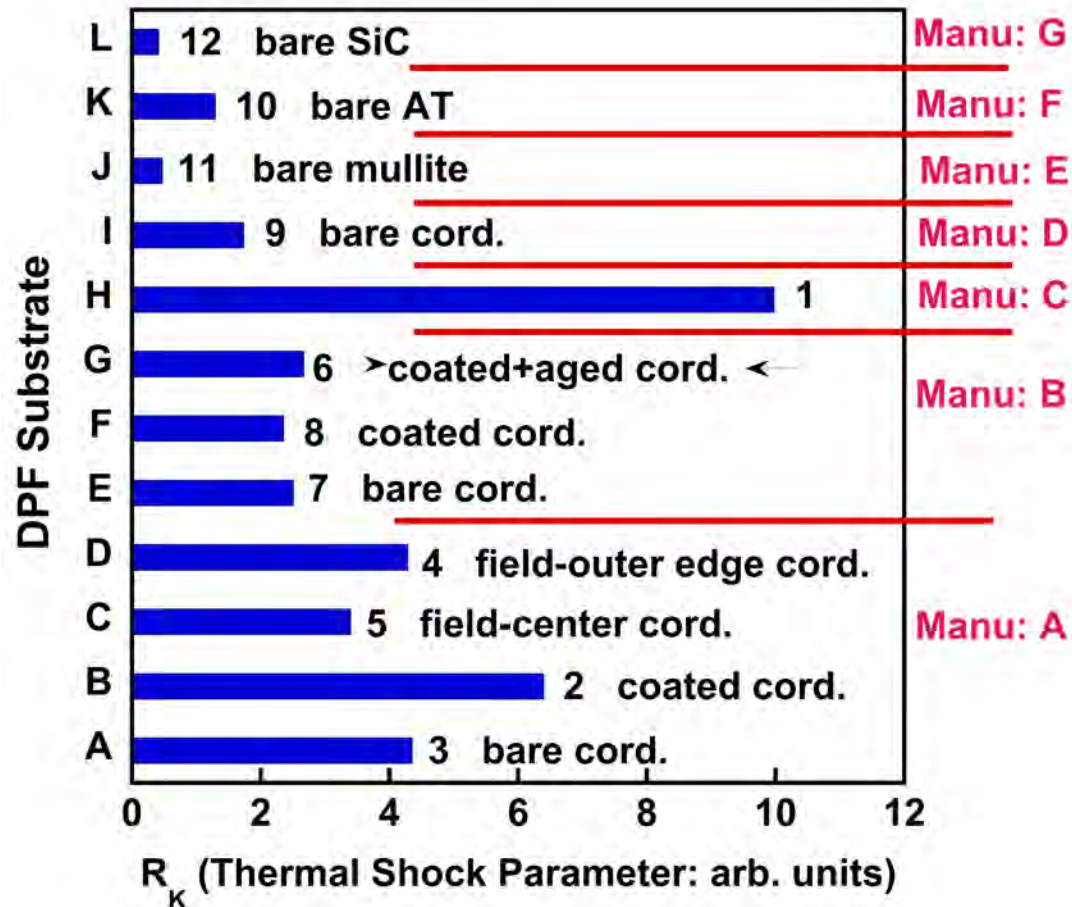
Porosity dominates Properties: e.g. K_{IC} – Strength Correlation



- 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

New thermal shock ranking methodology utilized



$$R_K = K_{IC} / \alpha E$$

- Easier to perform fracture toughness tests than strength (less material and fewer samples)

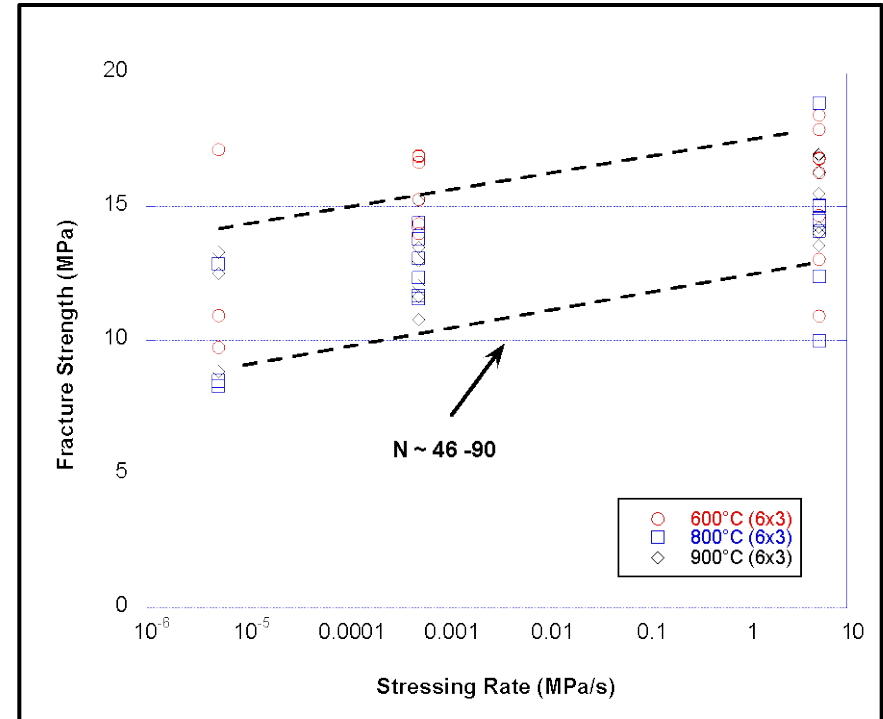
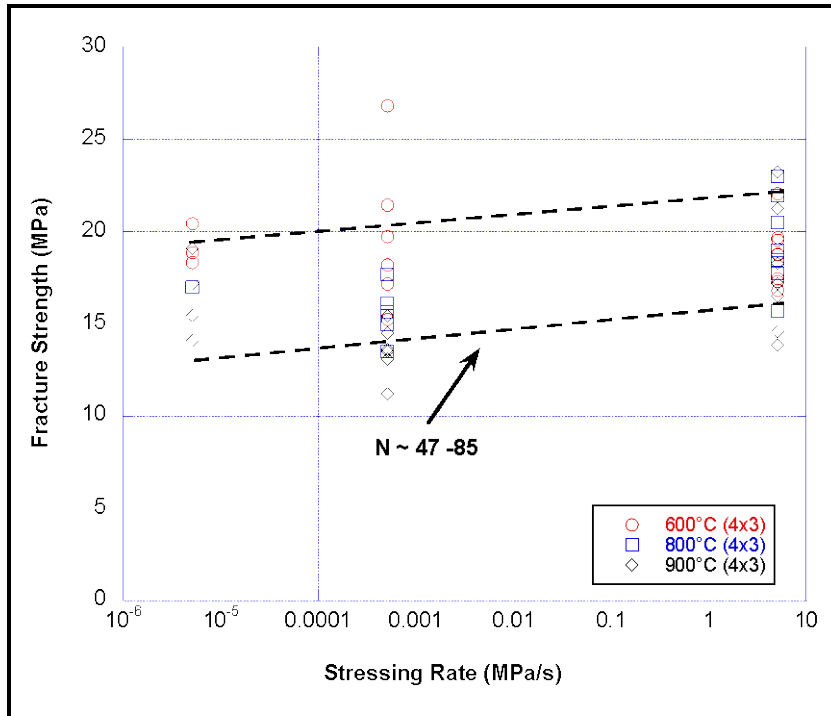
• Properties measured

- K_{IC} , Fracture Toughness (at room temperature) – Double Torsion
- α , Coef. of Thermal Exp. RT to 1000°C – TMA analyzed parallel and perpendicular to extrusion direction
- E , Elastic Modulus – DMA

- Note that the above analysis is highly simplified; assumes:

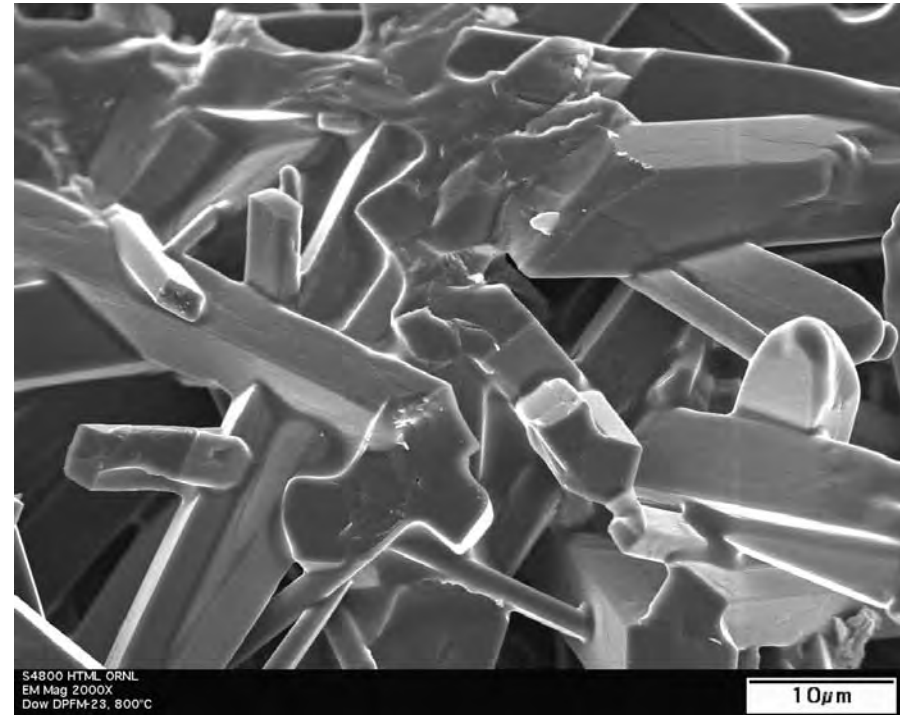
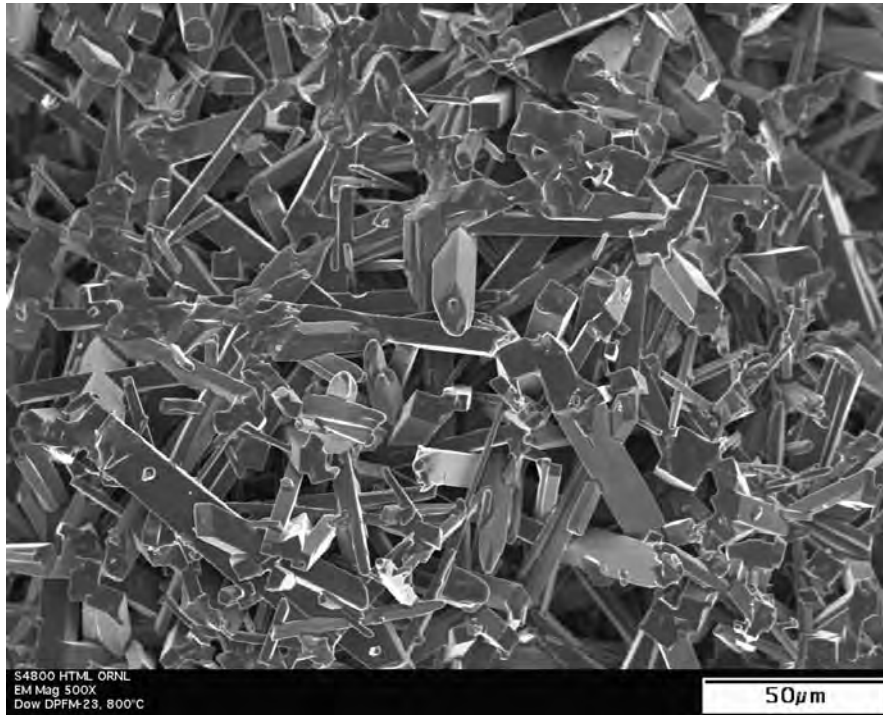
- Homogeneity
- Isotropy

There is a Minor Effect of Specimen Size on the Dynamic Fatigue Response of Mullite ($\text{Al}_2\text{O}_3 \cdot 2\text{SiO}_2$) DPF Specimens




- The dynamic fatigue exponents obtained from the slopes of strength vs. stress rate curves suggest good resistance to slow crack growth processes at elevated temperatures
- The fatigue fracture strength of mullite DPF is 2-3 times higher than cordierite DPF

Fracture Surface of Mullite DPF Specimen Tested at 800°C in air revealed clean and smooth whisker morphology



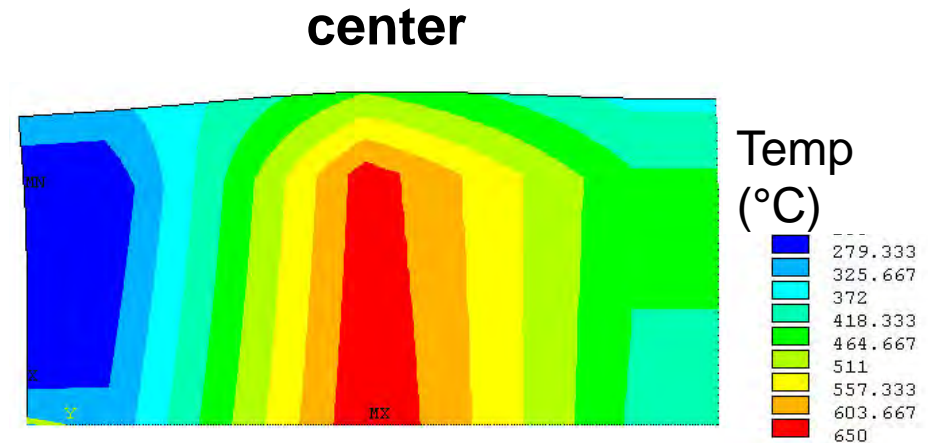
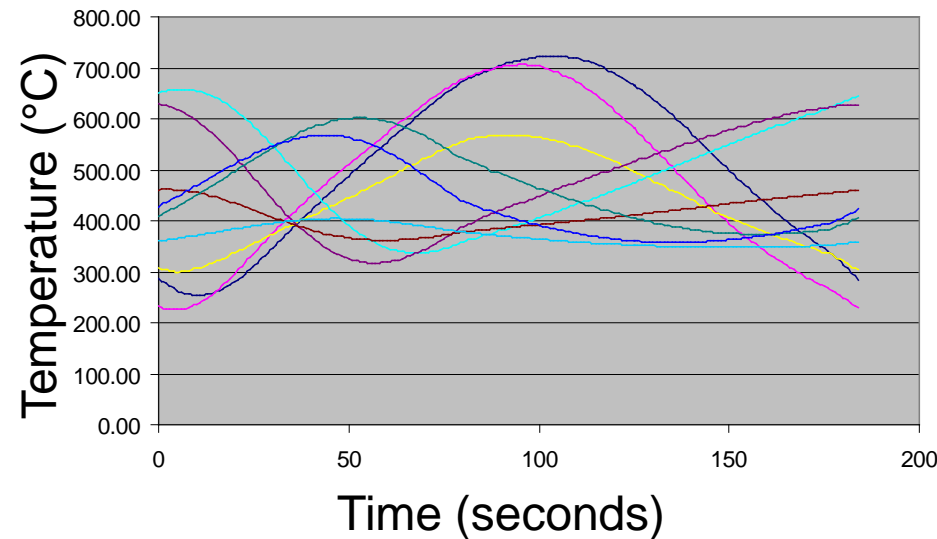
- **No indication of apparent environmental effects on the fracture process, consistent with high fatigue exponent N values**
- **Whisker morphology contributes the highest fracture strength among three DFP materials**

Collaborations and coordinations with other institutions: Partners

-  **(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 (e.g. strength data shown here); exchange of technical information to assist with each others analyses; face to face meetings at least 2X/year
- **CORNING (Industry):**
 - Corning's role is to consult on material application and supply material necessary for testing. Corning has been active for some periods of the CRADA
 - Supplies samples; exchange of technical information

Collaborations and coordinations with other institutions: Tech transfer

- **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)

Future Work

- **Continue to characterize the dynamic and static fatigue response of SiC DPFs (FY11-13).**
- **Continue to study the interaction of washcoat, soot and substrate on properties (FY11).**
- **Characterize the effect of joints between segments on mechanical integrity in multi-element structures (FY11 & 12).**
- **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).**

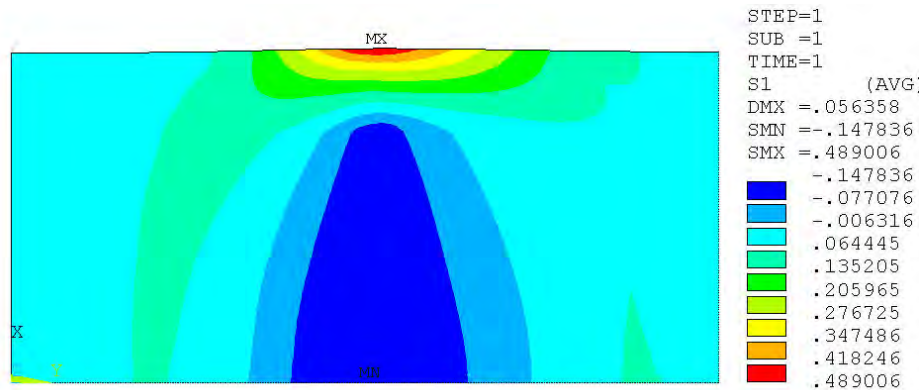
Summary: Property data support modeling which optimize regeneration which changes engine combustion regimes, improves durability, efficiency; reduces cost...VT goals

- Carried out physical and mechanical property measurements on third and fourth alternate substrate DPF materials: SiC and mullite and ranked its relative thermal shock resistance to others (continued from FY2010).
- Refined the relationship between porosity and the elastic-fracture properties for diesel particulate filter substrates.
- Compared properties of cordierite, Al_2TiO_5 -based, SiC-based and $\text{Al}_2\text{O}_3 \cdot 2\text{SiO}_2$ DPF materials

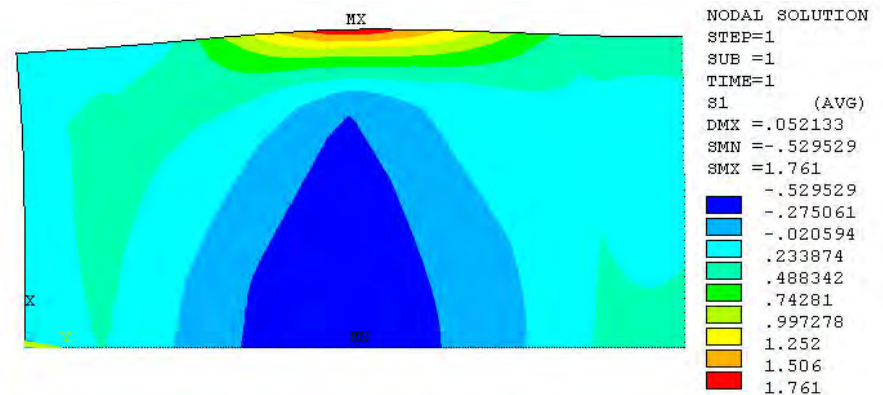
Technical Backup slides

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.



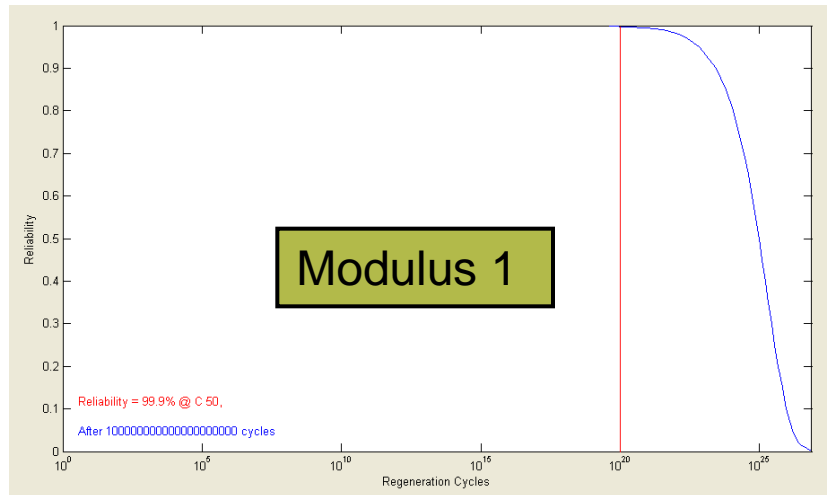
Modulus 1



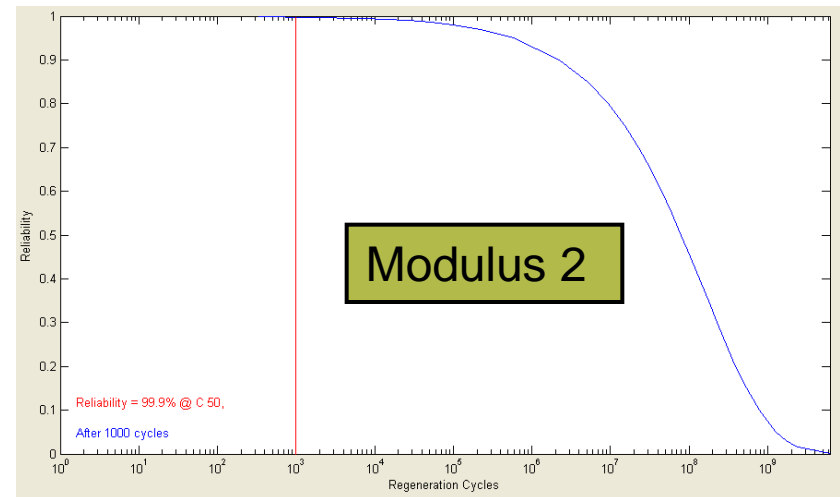
Modulus 2

Collaborations and coordinations with other institutions: Tech transfer3

- **Different stress results (from different modulus inputs) give very different life predictions.**
- **More work to understand effect of modulus measurement is necessary to optimize the stress and life models.**



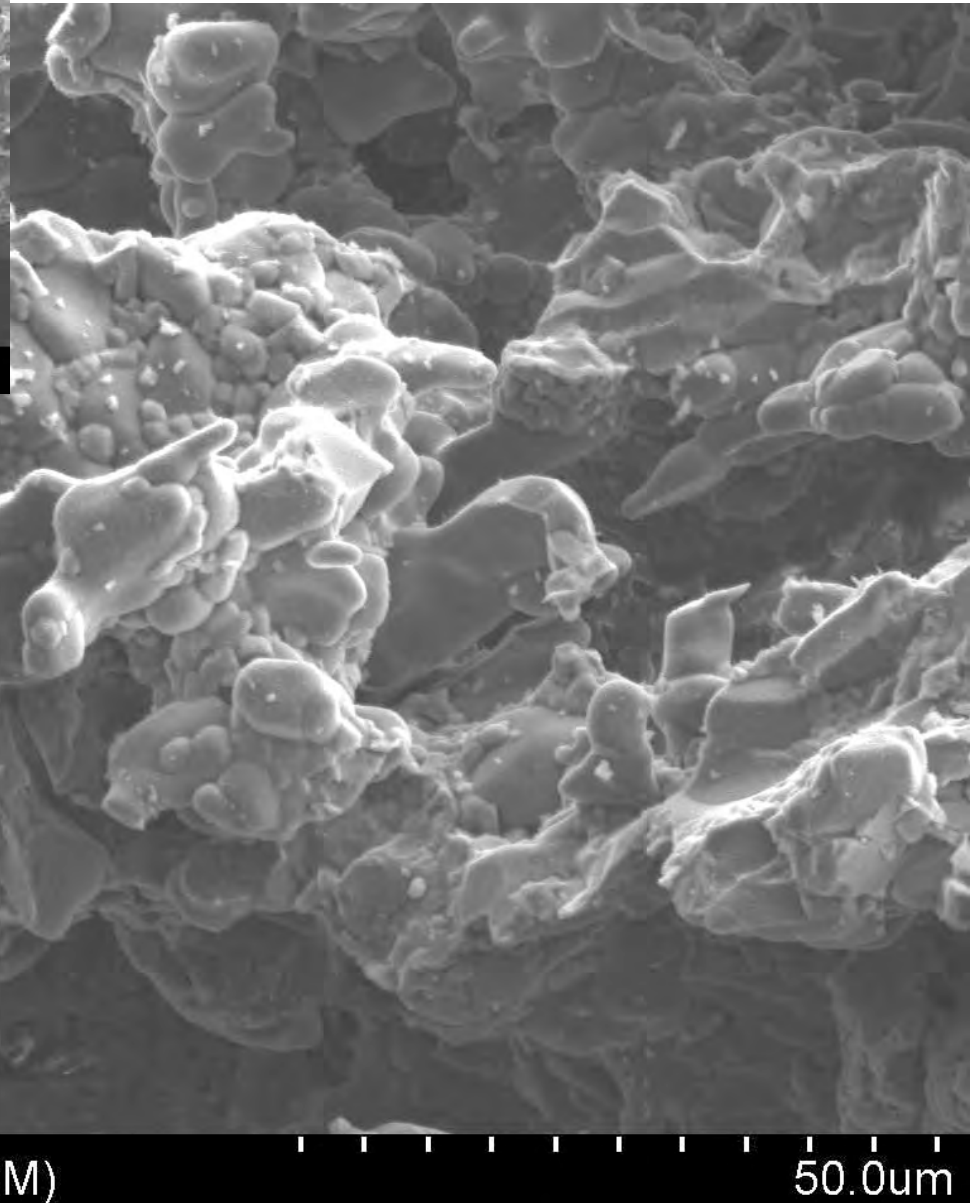
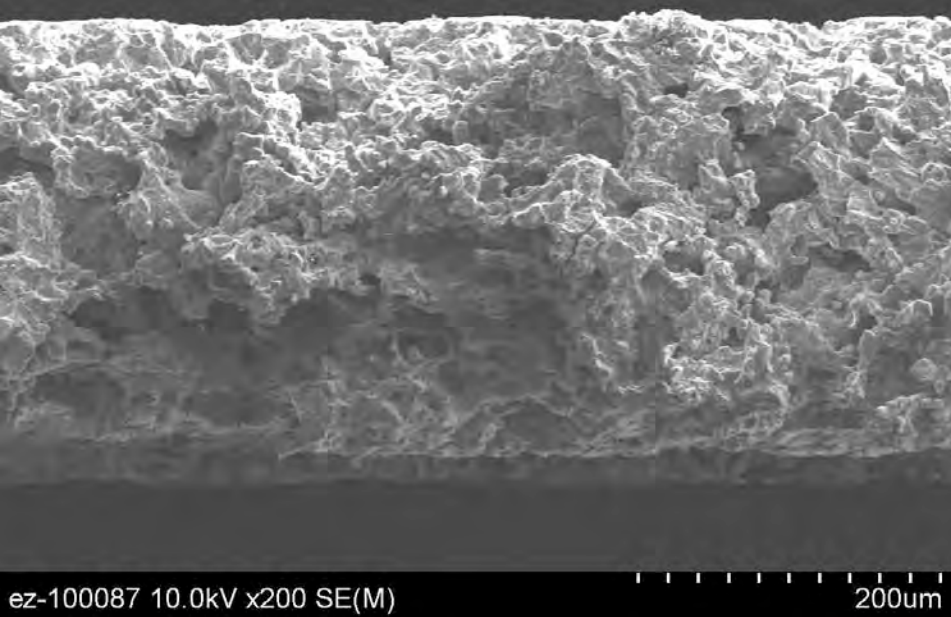
99.9 % component will survive up to **1x10²⁰** cycles.



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

AT DT fracture surface

- microcracked



DPF materials are very porous

- **Density:** Definitions given in ASTM D3766, **ASTM International, West Conshohocken, PA.**

Property/Material	Cordierite	Al ₂ TiO ₅	SiC
Bulk (g/cc)*	1.22	1.65(0.03)**	1.18(0.03)
Skeletal (g/cc)‡	2.46	3.43(0.05)	2.83(0.05)
Relative ρ (%)∅	49.6	48.1	41.7
Porosity (%)	50.4	51.9	58.3
Wall thickness (mm)	~0.38	~0.30	~0.28

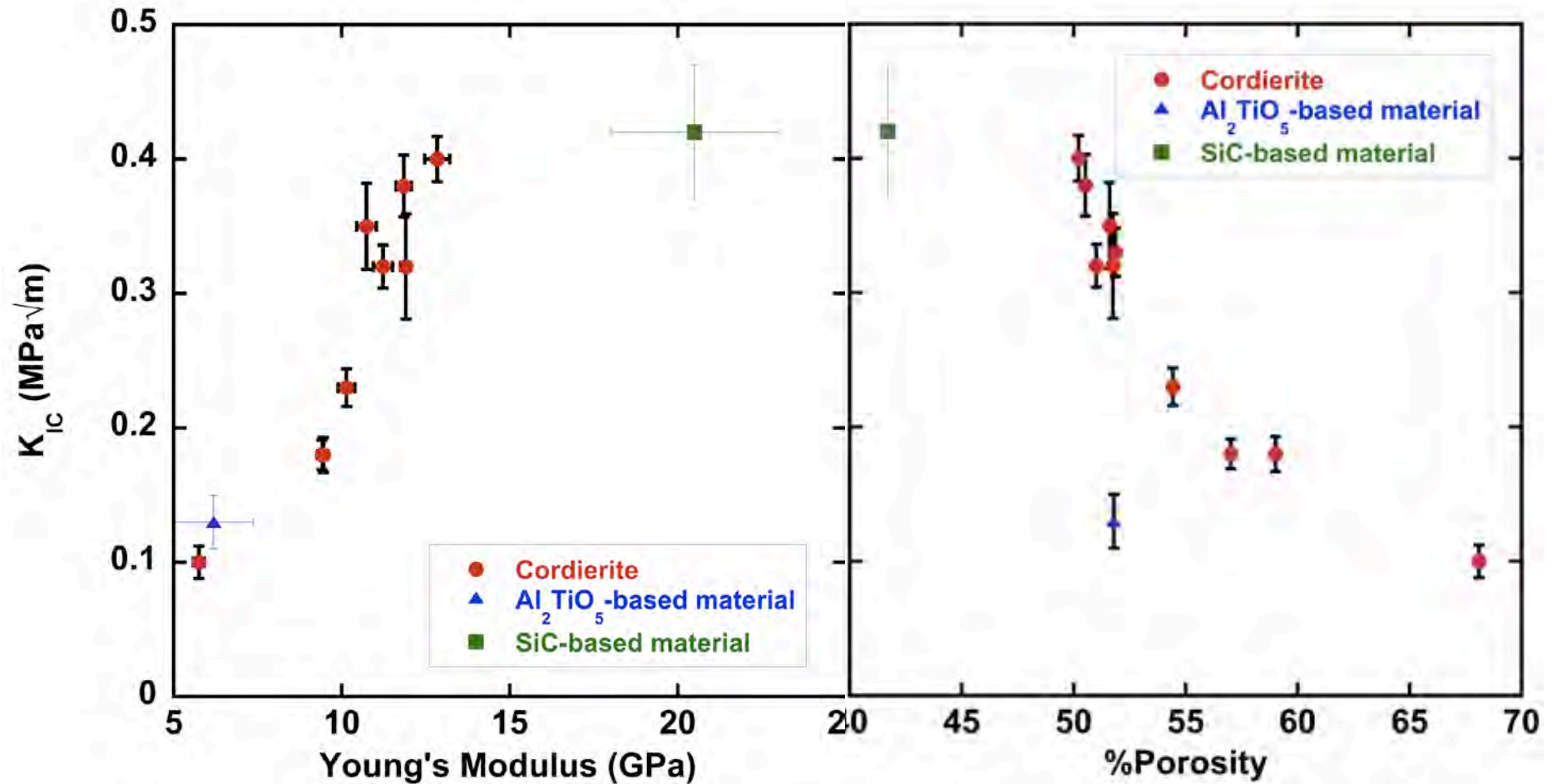
* Geometric volume: includes solids plus all open and closed porosity of plate samples, i.e., no channels

** () values = standard deviation

‡ He pycnometry: volume includes solids plus all closed porosity

∅ Relative density ($\rho_{\text{bulk}} / \rho_{\text{skel}} * 100$)

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