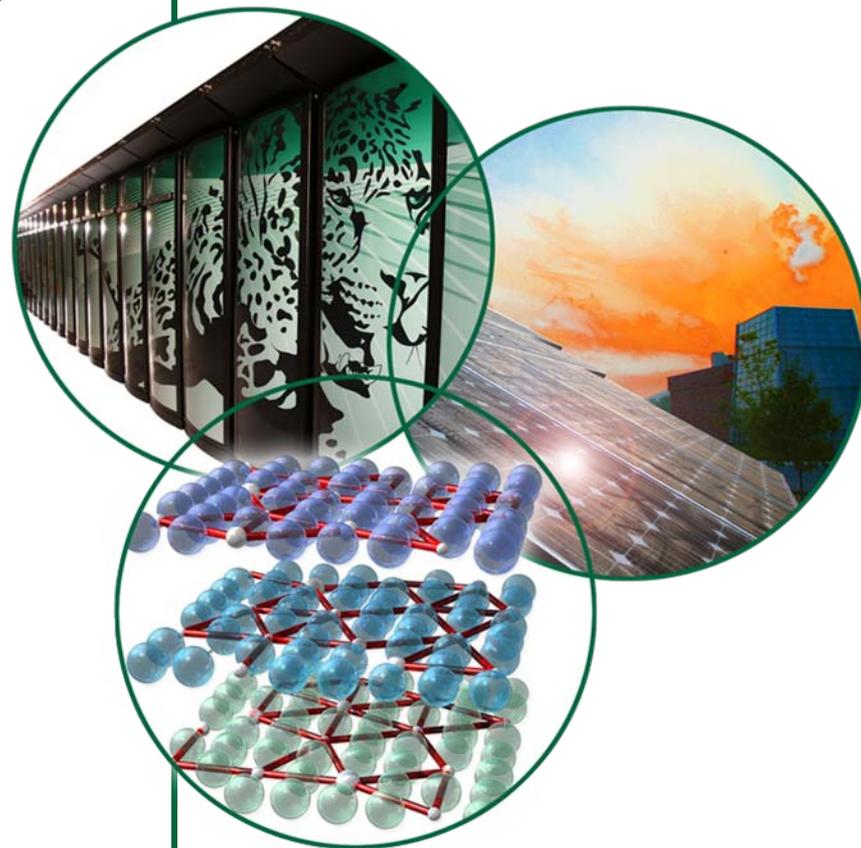


Diesel Particulate Filtration (DPF) Technology: Success stories at the High Temperature Materials Laboratory (HTML) User Program

DOE 2009 Vehicle Technologies Annual Merit Review and Peer Evaluation Meeting

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The HTML User Program - Background

The HTML is a National User Facility that supports the missions of DOE, EERE and the Vehicle Technologies Program in particular, by working with industry, universities and other national laboratories to develop energy efficient technologies that will enable the U.S. to use less petroleum. The HTML is organized into six user centers, which are clusters of highly skilled staff and sophisticated, often one-of-a-kind instruments for materials characterization.

Access to the HTML is provided through the HTML User Program proposal process. Research proposals are reviewed by a committee and approved based on scientific merit, relevance of the proposed research to the mission of DOE's Vehicle Technologies Program, and feasibility. Research is completed within 24 months and normally involves one or more user visits to the HTML.

Both nonproprietary and proprietary research is conducted within the HTML User Program. There are generally no charges for nonproprietary research projects, and users conducting nonproprietary research must agree to submit research results for publication in the open, refereed literature. For proprietary research, the user owns the research data and all costs at the HTML are paid by the user based on DOE guidelines for ORNL costs. A nonproprietary project is complete when the results are published in the open literature and/or presented at a professional conference.

Project ID: IMP04, Shyam

The HTML User Program – FY2008 Activity

During FY2008, the HTML User Program managed 76 user projects from 53 different organizations.

The FY2008 budget for the HTML was \$6,072,283 and was allocated as follows:

- \$1,567,500 for capital equipment purchases**
- \$3,879,483 to support staff participation in user projects**
- \$626,000 for the operation of the user program**

Users cost-share user projects through:

- 1) their direct involvement with HTML staff members during the development of the user project;**
- 2) funding their travel to the HTML to perform research;**
- 3) costs of materials provided by the user or the research performed prior to the user project;**
- 4) their subsequent collaboration with HTML staff members to analyze the data and publish the results.**

The HTML also supports the education and preparation of a new generation of scientists and engineers.

During FY2008, students and professors from 32 universities participated in the HTML User Program. Four of those students received their Ph.D. degree in FY2008 based in part on research performed through the HTML User Program.

Relevance to the VT Program

- The Vehicle Technologies Program supports the HTML User Program and provides an annual budget to address a wide range of materials-related issues in ground transportation systems arising from R&D needs in U.S. industry.
- The user projects highlighted in this presentation address barriers associated with engine efficiency reduction by measures to reduce emissions identified in the Advanced Combustion and Emission Control Technical Roadmap for Light-Duty Powertrains and the Roadmap for the 21st Century Truck Partnership.

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Milestones

- **Research to be planned and performed within two years following approval of HTML User Program proposal.**
- **Research results disseminated by publication or presentation of results so that they are available to the public.**

GE02 Technologies



“Correlation between fracture toughness and microstructure in fibrous and porous monolithic ceramics for use in diesel particulate filters”

Timeline

- Start date: 4/24/08
- End date: 4/23/10
- % complete: 85%

Budget

- Included in the user center allocations from the annual budget of the HTML User Program; users cost-share as previously noted.

Barriers

- Engine efficiency losses due to back-pressure increases and regeneration energy requirements.
- Long-term durability of particulate matter aftertreatment systems
- Cost-effective filter materials

Collaborators

- Users: Timothy Gordon
- HTML Staff: Amit Shyam,
Edgar Lara-Curzio

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User Project with GEO2 Technologies

Research problem: To understand the relationship between the microstructure and mechanical and thermal properties of fibrous diesel particulate filter materials (SiC, mullite).

Approach: Utilized techniques developed at the HTML to prepare test specimens of porous materials and determine their fracture toughness and thermal conductivity. Utilized scanning electron microscopy to characterize the microstructure of these materials.

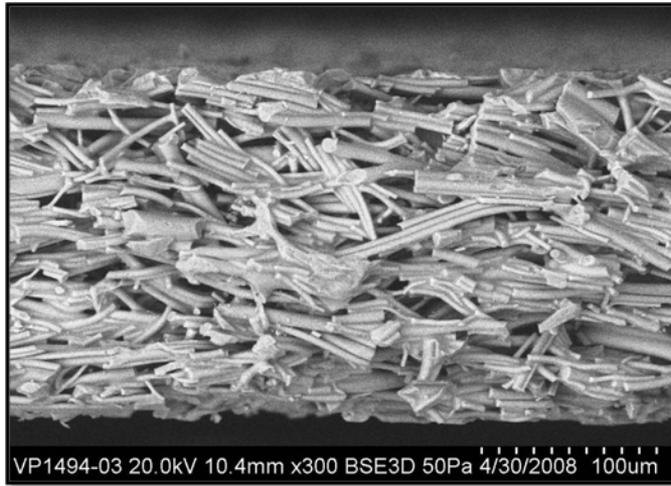
Implications: The development of tough, cost-effective diesel particulate filters with lower pressure drop for higher engine efficiency.



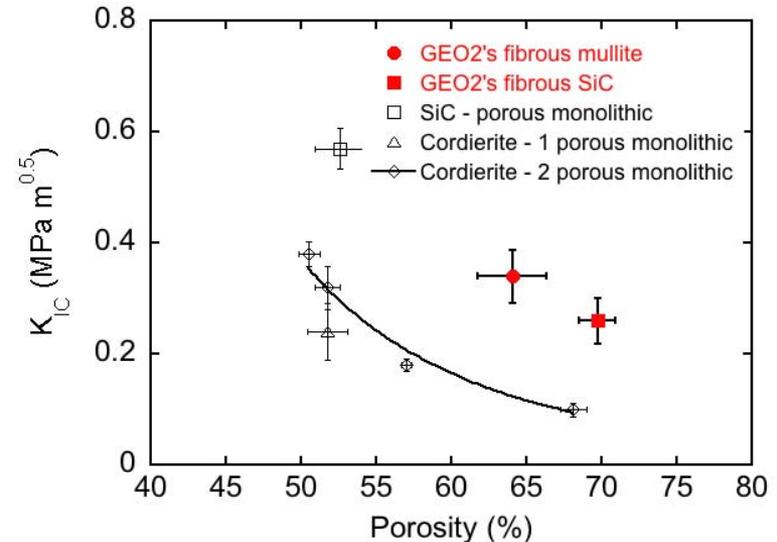
Tim Gordon of GEO2 Technologies sets up a fracture mechanics test at the HTML.

User Project with GEO2 Technologies

Accomplishments



The unique fibrous Cross-Linked Microstructure (CLM™) of the GEO2 substrate is evident on the fracture surface of a mullite test specimen.



A comparison of the fracture toughness of GEO2's fibrous materials and commercially available porous monolithic ceramic DPF substrates.



GEO2 fibrous substrates exhibit high fracture toughness even at high levels of porosity.

Project ID: IMP04, Shyam

University of Wisconsin

“Investigation of soot deposition in the walls of diesel particulate filters”



Timeline

- Start date: 7/29/08
- End date: 7/28/10
- % complete: 90%

Budget

- Included in the user center allocations from the annual budget of the HTML User Program; users cost-share as previously noted.

Collaborators

- Users: Prof. David Foster, Tetsuo Orita, Renato Yapaulo
- HTML Staff: Michael Lance, Larry Walker

Barriers

- Lack of understanding of particulate matter capture and oxidation.
- Engine efficiency losses due to back-pressure increases and regeneration energy requirements.
- Long-term durability of particulate matter aftertreatment systems

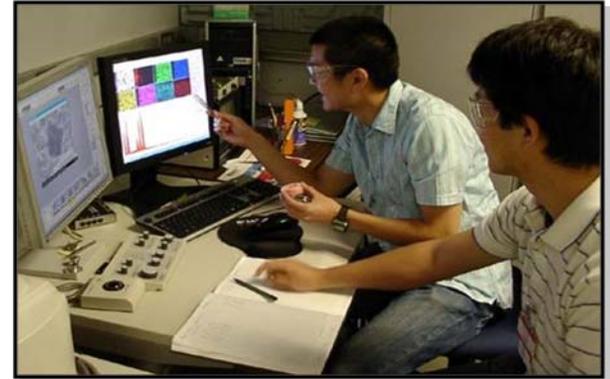
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User Project with the University of Wisconsin

Research problem: To understand the fundamentals of soot deposition in DPFs and to quantify soot penetration depth in DPF walls.

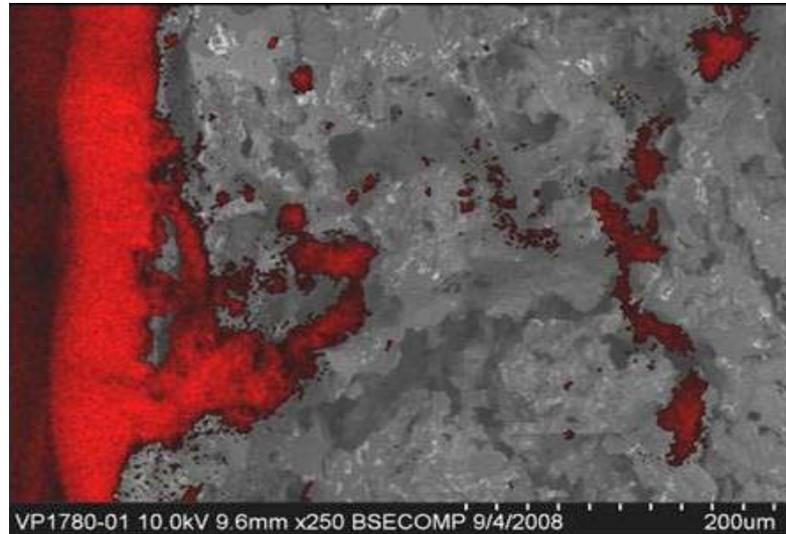
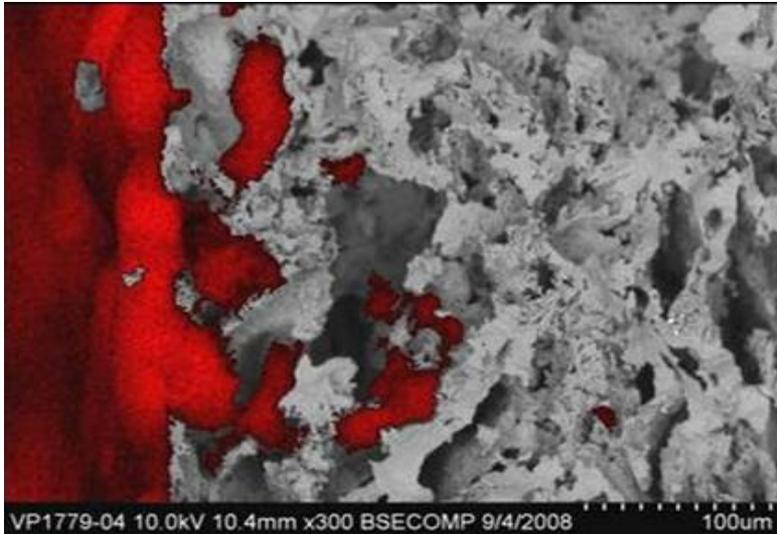
Approach: Utilized UV-illuminated optical microscopy and an environmental scanning electron microscope to determine penetration depth in porous substrates.

Implications: A thorough understanding of the nature of soot deposition in DPFs will enable the development of more efficient filtration systems.



Renato Yapaulo (left) and Tetsuo Orita from University of Wisconsin review results of SEM imaging.

User Project with University of Wisconsin Accomplishments



Images obtained with environmental SEM show soot distribution (in red) comparing Mode 6 and 6x case, both at 8cm/s (these indicate different particulate matter loading schemes).

By understanding the effects of exhaust flow rates and particulate matter characteristics on soot deposition, it will be possible to optimize particulate filter systems.

Project ID: IMP04, Shyam

“Determination of thermal conductivity of soot deposits from transportation fuels”

Timeline

- Start date: 10/8/07
- End date: 10/7/09
- % complete: 80%

Budget

- Included in the user center allocations from the annual budget of the HTML User Program; users cost-share as previously noted.

Barriers

- Lack of understanding of particulate matter capture and oxidation.

Collaborators

- Users: Prof. Eric Eddings, and Ignacio Preciado
- HTML Staff: Ralph Dinwiddie

User Project with the University of Utah

Research problem: To characterize the thermophysical properties of soot deposits from various fuels (e.g., diesel, JP-8).

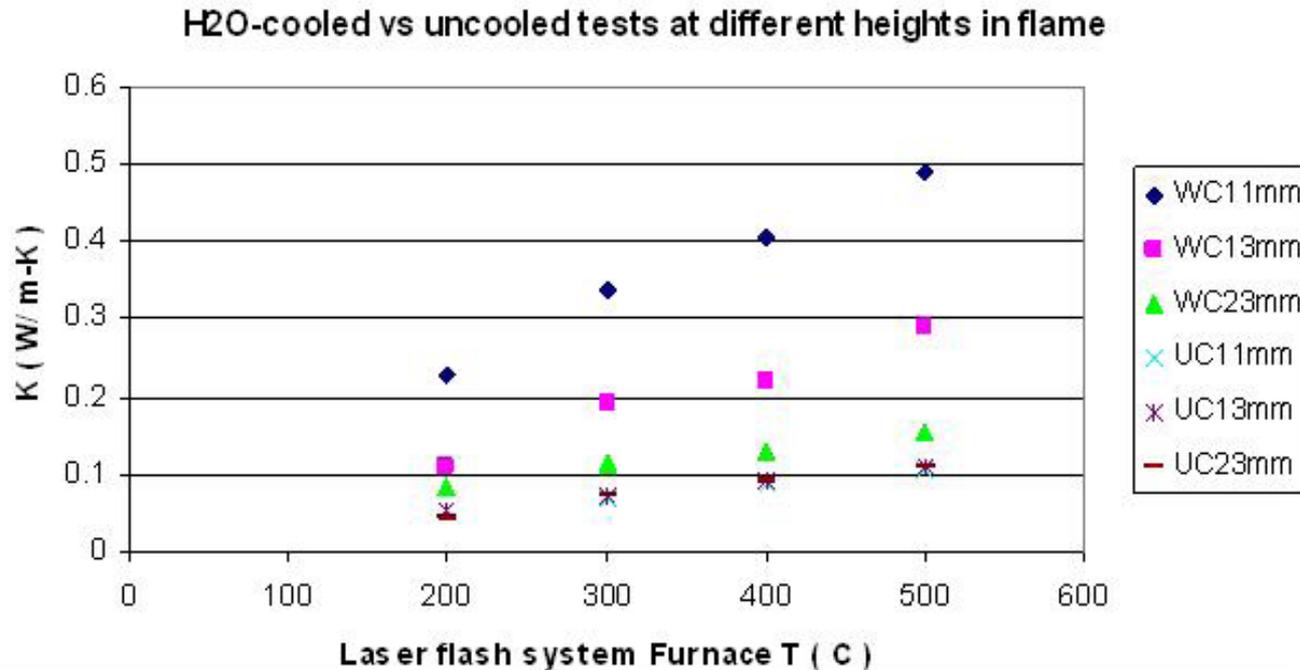
Approach: Used laser flash thermal diffusivity and differential scanning calorimetry to determine the thermal diffusivity and specific heat of soot deposits as a function of deposition temperature.

Implications: The experimental results will support the development of models to understand the effect of deposits in combustion engines.



University of Utah graduate student Ignacio Preciado adjusts settings for a calorimetry test.

User Project with University of Utah Accomplishments



Thermal conductivity for water-cooled (WC) and uncooled (UC) tests at different heights in the flame.



The thermophysical properties of soot deposits were determined as a function of deposition temperature.

Project ID: IMP04, Shyam

Colorado School of Mines

“*In situ* Raman analysis of beta eucryptite pressure-induced transformation”



Timeline

- Start date: 12/6/07
- End date: 12/5/09
- % complete: 100%

Budget

- Included in the user center allocations from the annual budget of the HTML User Program; users cost-share as previously noted.

Barriers

- Long-term durability of particulate matter aftertreatment systems
- Cost-effective filter materials

Collaborators

- Users: Prof. Ivar Reimanis, Timothy Jochum
- HTML Staff: Michael Lance

User Project with the Colorado School of Mines

Research problem: To develop greater understanding of the fundamentals of phase transformations of beta-eucryptite.

Approach: Utilized *in situ* Raman spectroscopy and a diamond indenter to quantify the effect of stress on the beta-eta phase transformation in eucryptite.

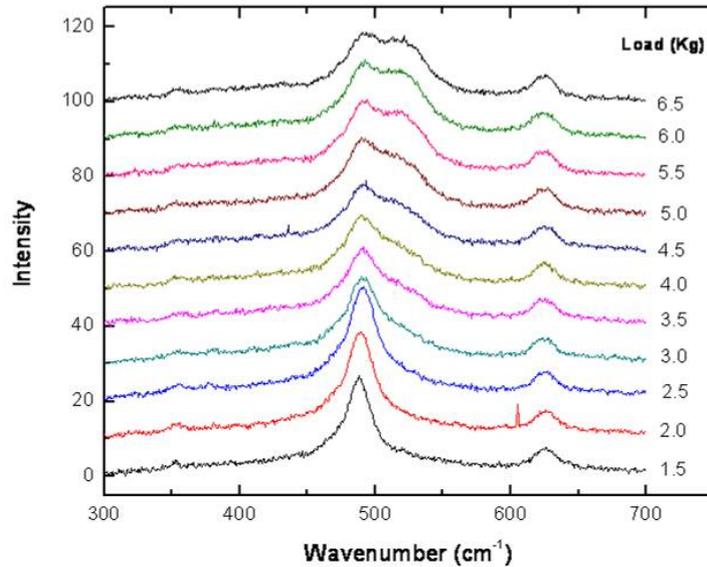
Implications: The development of tough, durable, cost-effective diesel particulate filters based on beta-eucryptite.



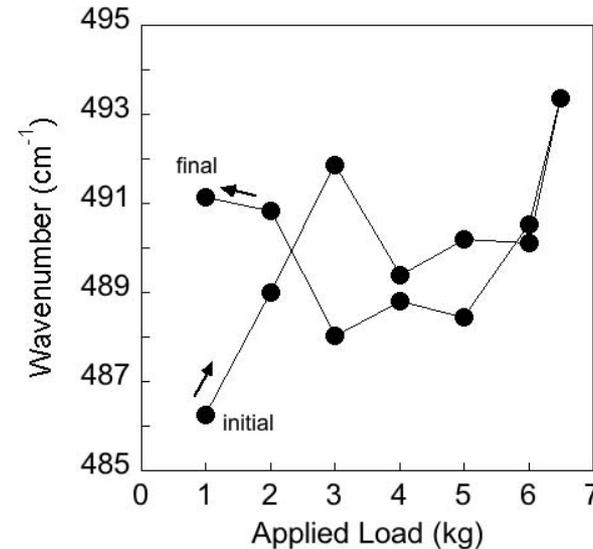
Tim Jochum from Colorado School of Mines collecting Raman spectra on a beta-eucryptite test specimen.

Project ID: IMP04, Shyam

User Project with the Colorado School of Mines Accomplishments



β -eucryptite exhibits a Raman peak near 490 cm^{-1} . The peak at around 625 cm^{-1} is from the diamond punch. At roughly 3 kg of applied load, a shoulder emerges at $\sim 525\text{ cm}^{-1}$ that is associated with ϵ -eucryptite formation.



Frequency shift in β -eucryptite Raman peak position induced by stress.

Phase transformations-induced toughening in β -eucryptite could result in tough, durable diesel particulate filters.

Project ID: IMP04, Shyam

Future Work

- Completion of publications and submission to journal (FY2009).
- Dissemination of results through presentations at technical conferences (FY2009).

User Projects Summary

- During FY08 the HTML user program was involved in user projects with GEO2 Technologies, the Colorado School of Mines, and the Universities of Wisconsin and Utah to advance diesel particulate filtration technologies.
- Test methods developed at the HTML were utilized to determine the mechanical properties of novel fibrous materials developed by a small business (GEO2 Technologies). Using *in situ* Raman spectroscopy, the feasibility of phase transformation toughening in beta-eucryptite was demonstrated, which could lead to the use of this material for tough, durable and cost-effective diesel particulate filters.
- The thermophysical properties of soot deposits were determined, and the effects of engine operating parameters on soot penetration on porous substrates were quantified. Such information will help optimize filtration systems.

Project ID: IMP04, Shyam