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### ON THE COVER

*Using his specialized techniques, metallographer Paul Danielson creates a superior sample for the evaluation which, under a microscope, will reveal an in-depth, complete microstructure of the material.*



netlog is a quarterly newsletter, which highlights recent achievements and ongoing research at NETL. Any comments or suggestions, please contact Paula Turner at [paula.turner@netl.doe.gov](mailto:paula.turner@netl.doe.gov) or call 541-967-5966.



## Photomicrographic Image Appears in 2009 Buehler Materials Masterpieces Calendar

Metallographer Paul Danielson produced an optical image of the microstructure of a metallic alloy that appears in the 2009 Buehler Materials Masterpieces Calendar. The image for the month of July shows the alloy viewed under polarized light at 400 times magnification, revealing the as-cast microstructure. The metallographic sample was prepared using an attach-polishing and heat-tinting procedure developed by Danielson to more clearly resolve the details of the microstructure of the alloy via optical microscopy. Twelve images are selected to appear in the calendar each year from submissions by researchers from the international materials science community. Other images selected this year were from organizations including the Naval Surface Warfare Center – Carderock Division, Lehigh University, University of Birmingham (UK), and University of Technology Aachen (Germany). The properties of materials are governed by the underlying microstructure. Thus it is important to understand microstructure in order to improve materials performance.

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*NETL researchers pour molten metal from a bench-scale electric arc furnace during direct smelting tests in cooperative work with Cardero Iron Ore Company, Ltd.*

## NETL Helps Reduce Carbon Footprint of Iron Production

NETL researchers are helping the Cardero Iron Ore Company, Ltd. to reduce the amount of carbon dioxide produced during iron smelting.

NETL is applying its unique capabilities in ore processing to help Cardero process iron ores with reduced carbon dioxide emissions.

Cardero Iron is developing iron-ore sand deposits containing finely divided magnetite ( $\text{Fe}_3\text{O}_4$ ) particles that do not require grinding before further processing. Eliminating the grinding avoids the energy consumed by size reduction – about 10 kilowatt hours per ton of ore.

In an initial series of tests in the cooperative work under a funds-in agreement with Cardero Iron, NETL researchers briquetted 500 pounds of a magnetic

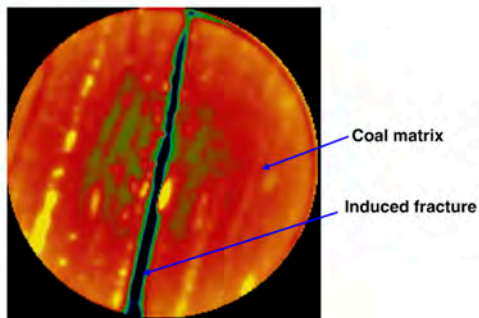
concentrate provided by the company, and then performed direct smelting tests on the unsintered briquettes in an electric arc furnace.

Eliminating sintering, which oxidizes the magnetite to hematite ( $\text{Fe}_2\text{O}_3$ ), reduces the production of  $\text{CO}_2$  by approximately 11 percent.

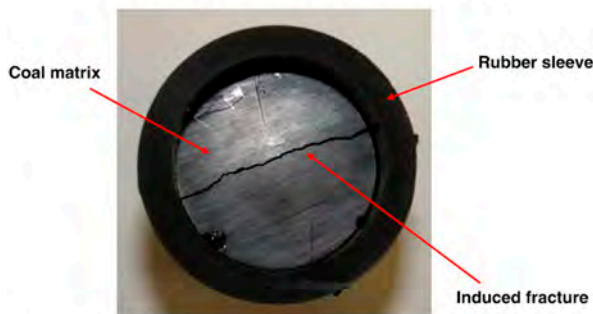
Development of industrial processes that are cleaner and more energy efficient is consistent with NETL goals of reducing  $\text{CO}_2$  emissions.

Cardero Iron is a subsidiary of the Cardero Resources Corporation.

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(a) A typical CT Scan of a Fractured Coal Sample



(b) Visible fracture on the upstream surface

## Researchers Measure Permeability of CO<sub>2</sub> in Coal Under CO<sub>2</sub> Sequestration Conditions

Sorption of gases, particularly carbon dioxide, in coal normally results in swelling of the coal matrix. The coal mechanical behavior can be significantly altered by the plasticizing effect of the carbon dioxide. This usually results in mechanical weakening and loss of permeability to fluid flow. In some cases, permeability loss is so great that traditional flow-through methods of permeability measurement are not possible. NETL uses a specially developed instrument that determines very low permeability by measuring dynamic responses to applied pressure pulses across cores. Quantifying the effects of carbon dioxide sorption is essential for screening coal seams for potential carbon dioxide sequestration. A paper, "Influence of Carbon Dioxide on Coal Permeability Determined by Pressure Transient Methods," submitted by NETL researchers was published in the peer reviewed journal, *International Journal of Coal Geology* 77 (2009) 109-118.

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## Helium Has Major Impact on Characterization of CO<sub>2</sub> Storage Capacity of Coal

Researchers at NETL discovered that exposure to 7 megapascals (1015 pounds per square inch) of helium at room temperature alters the texture of Upper Freeport coal in a lump form. Since helium volume is conventionally used as a measure of "true" volume, the key parameter in interpretation of CO<sub>2</sub> sorption isotherms on coal, this finding has major implications for methods of accurately characterizing CO<sub>2</sub> sequestration in unmineable coal seams and enhanced coalbed methane recovery. The results of the study were reported in an article by Vyacheslav Romanov and Yee Soong, "Helium-Volume Dynamics of Upper Freeport Coal Powder and Lumps," that was included in the basic science section of a special issue, "CO<sub>2</sub> Sequestration in Coals and Enhanced Coalbed Methane Recovery," of the *International Journal of Coal Geology* 2009, 77 (1), 10-15.

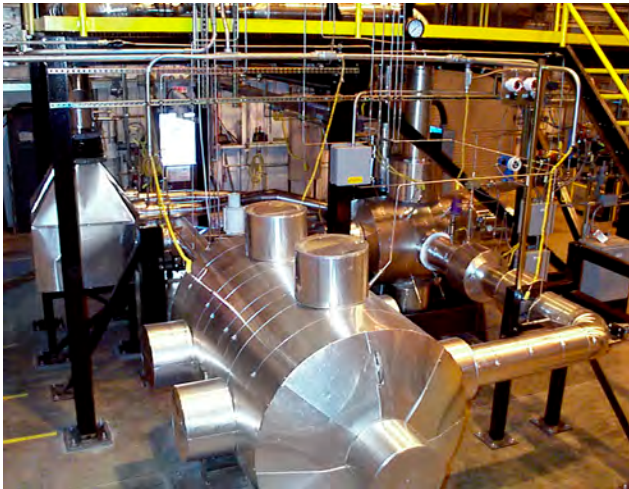
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## NETL Team Writes Book Chapter on New Sensor Technology

A book chapter on sensors for gas detection, written by Michael Buric, Kevin Chen, Joel Falk, and Steven Woodruff, is to be published in *Trends in Photonics* 2008. The chapter, titled "Photonic-Bandgap-Fiber Sensors for Gas Detection," was written at the invitation of the editor, John Canning, who is producing the book for Research Signpost, Kerala, India. The authors are research colleagues in NETL's University Research Initiative project "Raman Spectroscopy for Monitoring of Natural Gas Composition." The book chapter summarizes the results of recent NETL work to develop fast response gas sensors for power generation applications and represents the collaborative efforts of researchers at the University of Pittsburgh and NETL.

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NETL's Hybrid Performance Fuel Cell Test Facility.

## Hybrid Performance Fuel Cell Test Facility Shakedown is Complete

The NETL Hybrid Performance Fuel Cell Test Facility, known as "Hyper," recently completed a full-system safety inspection and shakedown by project personnel and is now back in operation.

The facility is one of only two hybrid hardware simulation facilities in operation worldwide, and it is the only facility that uses hardware to simulate fuel cell dynamics. The advantage of this over a simple numerical simulation is that it can be coupled to a real turbine, heat exchangers, and other balance-of-plant hardware. This allows NETL researchers to explore operational transients that are applicable to large power systems without risking damage to an \$8 million fuel cell.

The complete shakedown of the facility repositions NETL as a world leader in fuel cell turbine hybrid power systems research. Hybrid systems are important because they offer the potential for development of high efficiency fossil-based cycles with carbon capture.

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## Scientists Begin Study of Solid Oxide Fuel Cell with Liquid Tin Anodes

NETL scientists completed the first in a series of fundamental studies of electrode behavior for a solid oxide fuel cell (SOFC) operating with a molten tin anode. Interest in molten metal anodes has risen recently due to their ability to directly consume solid fuel sources such as carbon and coal dust, removing the need for a gasifier. Besides allowing for high fuel utilization, liquid tin anodes also offer a higher degree of tolerance to coal contaminants that poison conventional nickel-based anodes. Standard electrochemical methods (chronoamperometry, impedance spectroscopy) were used to measure oxygen diffusion through the anode and to separate out the multiple interfacial phenomena. Present analysis of the data suggests that more mechanisms are present than just simple oxygen diffusion through the liquid anode, given that characteristic times for transient load equilibration show an order of magnitude faster diffusion times than expected. Follow-on analysis has begun to resolve this identified behavior. NETL's research focuses on isolating and measuring the various kinetic parameters of the anode: tin oxidation at the anode/electrolyte interface, oxygen transport through the liquid tin anode, and fuel oxidation/tin reduction at the fuel/anode interface. Characterizing the rate-limiting step in the anode performance will guide the development of the anode composition and support structure to maximum cell performance.

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*For his research in energy conversion and solid fuels, the American Society of Mechanical Engineers (ASME) has selected NETL employee Dr. George "Geo" Richards to receive ASME's prestigious Percy Nicholls Award.*

## **ASME Selects DOE Employee for Prestigious Percy Nicholls Award**

*Annual Honor Recognizes Achievement in Field of Solid Fuels*

ASME's Fuels and Combustion Technologies Division and the American Institute of Mining, Metallurgical, and Petroleum Engineers (AIME) Coal Division jointly present the annual award for "notable scientific or industrial achievement in the field of solid fuels." Dr. Richards will receive his award during the 34th International Technical Conference on Coal Utilization & Fuel Systems set for May 31 to June 4, 2009, in Clearwater, FL. ASME and AIME created the award in 1942 to honor the scientific contributions of Percy Nicholls in fuels utilization.

"When notified that I would be receiving the Percy Nicholls Award, I was surprised and humbled at the same time," said Dr. Richards. "I thoroughly enjoy energy research, and I didn't expect to be recognized in this way. My colleagues at NETL are a big part of this, and I gratefully acknowledge their many ideas and contributions. We are conducting research ranging from how to capture carbon dioxide from existing coal boilers, all the way to developing new concepts that will increase efficiency, even with carbon dioxide removal. I couldn't do all this without my peers at NETL."

Dr. Richards began working at NETL in 1988 and now serves as the Focus Area Lead for Energy Systems Dynamics in NETL's Office of Research and Development. He earned a Ph.D. in mechanical engineering from Purdue University, while focusing on gas turbine combustion. At NETL, Dr. Richards directs researchers who investigate solid-fuel gasification, carbon dioxide capture, high-temperature fuel cells for coal gases, hydrogen turbines, fuel processing, and fuel flexibility. He also facilitates cooperative research agreements with private industry and universities, as well as concepts related to energy conversion.

In addition to his laboratory responsibilities, Dr. Richards serves as a research advisor for graduate and post-graduate investigators who visit from academic institutions.

## NETL Scientist Receives Professional Achievement Award



Dr. Madhava Syamlal, focus area leader of computational and basic sciences at NETL, is the recipient of one of Illinois Institute of Technology's IIT 2009 Professional Achievement awards. Personal success, outstanding contribution to the field, and recognition by colleagues are all considered for this award, one of the highest distinctions

bestowed upon the university's alumni by previous alumni awards honorees.

A native of Kerala, India, Dr. Syamlal has worked at NETL for over 23 years. He resides in Morgantown, WV. He holds a bachelor of technology degree in chemical engineering from the Institute of Technology at Banaras Hindu University (India) and an M.S. and Ph.D. from the Illinois Institute of Technology (Chicago, IL).

The awards ceremony will take place on April 30 and May 1, 2009, on the campus of the Illinois Institute of Technology.

## NETL Presents Carbon Sequestration 101 to Air and Waste Management Association

At the requests of the Air and Waste Management Association, NETL developed a web-telecast (webinar) to provide an introductory understanding of carbon capture and sequestration. The two-hour session described the DOE Regional Sequestration Partnership and the current technical status of carbon capture and sequestration. The webinar was organized by AWMA as part of its training activities for continuing education credits. Webinar participants were located at 14 broadcast sites across the United States. Recorded versions of the presentation are available from AWMA.

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## Carbon Nanotubes Offer Promise

A paper describing simulations of gases in carbon nanotube (CNT) bundles has been published in *Langmuir* by Matthew LaBrosse, Wei Shi, and J. Karl Johnson. CNTs are promising materials for gas adsorption, separation, and transport with applications to contaminant removal, production of high-purity hydrogen, and so forth. There is a controversy in literature over whether gases can adsorb in interstitial channels (the spaces formed between three or more CNTs). The NETL researchers studied various types of closed, open, and partially opened homogeneous (CNTs having the same diameter) and heterogeneous (CNTs having a distribution of diameters) bundles. Comparing those with experimental results for adsorption capacity, isosteric heat of adsorption, and specific surface area, they found that models including adsorption in defect interstitial channels give the best overall agreement with experiments. The paper is entitled, "Are Defect Interstitial Sites Important?"

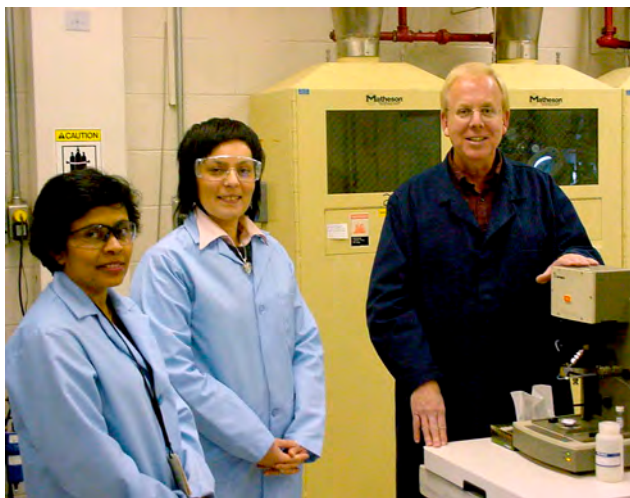
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## NETL Develops Cathode Microcube Materials for Solid Oxide Fuel Cells

Researchers from NETL and West Virginia University have synthesized cathode materials that have unique microstructural features. The goal of this collaborative study, under NETL's University Research Initiative, is to improve fuel cell performance by modifying the cathode architecture. The unique geometry and crystal plane alignment of the microcube material could improve the overall oxidation reaction kinetics and cell performance. The researchers have developed a new synthesis method to scale up manufacture of these single-crystalline LSM microcubes at low cost. The size of microcubes can be tailored from submicron to tens of microns. Tests are being planned to investigate the oxygen reduction reaction rate by using the newly developed LSM solid oxide fuel cell cathode.

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Researcher Ranjani Siriwardane, left; visiting scientist Ewelina Ksepko, center, from the Institute for Chemical Processing of Coal in Poland; and researcher Thomas Simonyi standing in front of the thermogravimetric analyzer, where they test samples for their collaborative chemical looping combustion research at NETL.

## Polish Researcher Collaborates on Chemical Looping Combustion at NETL

Dr. Ewelina Ksepko, a researcher from the Institute for Chemical Processing of Coal in Poland, is conducting collaborative research on chemical looping combustion (CLC) at NETL.

Chemical looping combustion is a novel combustion technique that utilizes oxygen from an oxygen carrier to combust fuels.

Dr. Ksepko's employer, the Institute for Chemical Processing of Coal, signed a Memorandum of Understanding with NETL and the Central Mining Institute of Poland in June 2008.

Three months later, Abbie Layne, director of the Separations and Fuels Processing Division in NETL's Office of Research and Development, and Ranjani Siriwardane, an NETL research scientist, met Dr. Ksepko during the Pittsburgh Coal Conference and discussed the possibility of Dr. Ksepko spending time at NETL as a guest researcher. That led to her coming to NETL to work with Siriwardane.

Dr. Ksepko will be involved in development of novel oxygen carriers for the CLC process. She will be testing the oxygen carriers in the thermogravimetric analyzer and bench-scale flow reactors at NETL. The research will include multicycle tests to understand the stability of the performance and the effect of impurities such as hydrogen sulfide on the performance of the carriers.

A research paper from this collaborative effort will be submitted to a peer-reviewed journal. Dr. Ksepko will be at NETL for about a month but the collaborative research will be continued as a part of the agreement between NETL and the Institute for Chemical Processing of Coal in Poland.

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## Simulations Aid in Designing Co-Feed Gasifier

NETL is applying a Fluent Discrete Phase Model (DPM) to investigate the performance of a proposed laboratory-scale gasifier design under conditions similar to those of existing commercial entrained flow gasifiers. Particle temperatures and conversion histories are being evaluated as functions of reactor length and the fluxes of inlet gas and solids. The solid fuel has been modeled as discrete particles that exchange mass and energy with the continuous gas phase. The mechanisms that cause the exchanges and the resulting source terms are being implemented in the DPM model via custom subroutines developed at NETL.

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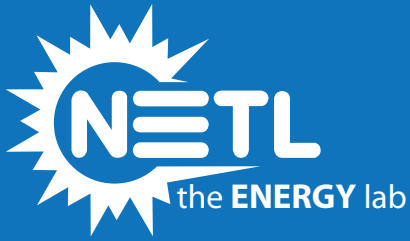
Shown at left is a basalt core within cement at low magnification showing the well cement/rock interface. The photo on the right is a thin section, transmitted light photomicrograph showing the interface between basalt and cement.

## NETL Completes Time-Series Carbon Sequestration Tests

Researchers at NETL have completed a test series to examine the performance of well-bore cement under down-hole conditions that mimic those the cement would encounter when used to seal a CO<sub>2</sub> injection well. Cores from the entire series of tests (28, 56, and 84 days) are being analyzed to determine the effects of the CO<sub>2</sub> environment on the hydration and curing of the cement as well as the interface between the rock and the cement. This helps researchers identify 1) conditions under which CO<sub>2</sub> will leak, and 2) potential methods for sealing the wells. This work supports the goal of the CO<sub>2</sub> Sequestration Program to develop technologies for geological sequestration that ensure greater than 99 percent retention of the CO<sub>2</sub>.

To prepare for the 84-day tests, cement was cast with rock cores from potential CO<sub>2</sub> reservoirs and submersed in fluids designed to simulate the pore waters in those reservoirs. This system was then pressurized with supercritical CO<sub>2</sub>.

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