R.W. CARPENTER, PROFESSOR

TELEPHONE: 480-965-4549

CENTER FOR SOLID STATE SCIENCE
TEMPE, AZ 85287-1704

FAX: 480-965-9004
INTERNET: CARPENTER@ASU.EDU

May 16, 2000

Final Report

USDOE Grant DE-FG03-95TE00068,
"Energy Resolved High Resolution Dynamic E-Cell Materials Research"

Ray Carpenter, Renu Sharma, and James Mayer
Center for Solid State Science

The purpose of this project was to develop *insitu* materials reaction observation capability in an intermediate voltage high resolution transmission electron microscope. To accomplish this we purchased a GATAN imaging energy filter system, a hot stage, and designed and constructed an environmental cell and real time television image recording system, and installed this equipment on our EM 430 intermediate voltage electron microscope.

The GATAN imaging energy filter enables us to determine composition and information about the electronic structure of small areas within specimens undergoing reactions that change its structure and chemistry in the microscope. The hot stage allows us to control the specimen temperature over a wide range, from room temperature to more than 1000°C, while it is undergoing reaction in the microscope. The environmental cell isolates the specimen environment in a small volume around the specimen itself, for example carbon dioxide gas, from the rest of the microscope. Such gases in the rest of the microscope, the high voltage accelerator for example, would prevent proper operation of the microscope. The real time television recording system enables us to record changes in specimen structure, caused by the reactions we are studying, dynamically as they occur. This dynamic visual record of reaction processes contains much more detailed information of how the reactions of interest occur than can be absorbed by human observers in a single observation of a reaction process. The videotapes recorded during reactions are typically viewed and studied a dozen or more times to extract all the useful information contained on each of them.

The environmental microscope system is now operating successfully on a daily basis. Some publications and conference presentations that describe recent research done using the system are:

Presentations:

Michael J. McKelvy, Hamdallah Bearat, Andrew V.G. Chizmeshya, Renu Sharma, Ray W. Carpenter, and Ken Streib, (invited), "The Role of Dehydroxylation in $\text{Mg}(\text{OH})_2$ Carbonation: Implications for Lamellar Hydroxide Mineral Carbonation," a presentation at the Fourth Carbon Dioxide Mineral Sequestration Forum at Arizona State University, Tempe, Arizona, December 13, 1999.

DISCLAIMER

This report was prepared as an account of work sponsored by an agency of the United States Government. Neither the United States Government nor any agency thereof, nor any of their employees, make any warranty, express or implied, or assumes any legal liability or responsibility for the accuracy, completeness, or usefulness of any information, apparatus, product, or process disclosed, or represents that its use would not infringe privately owned rights. Reference herein to any specific commercial product, process, or service by trade name, trademark, manufacturer, or otherwise does not necessarily constitute or imply its endorsement, recommendation, or favoring by the United States Government or any agency thereof. The views and opinions of authors expressed herein do not necessarily state or reflect those of the United States Government or any agency thereof.

DISCLAIMER

Portions of this document may be illegible in electronic image products. Images are produced from the best available original document.

Michael J. McKelvy, Renu Sharma, Andrew V.G. Chizmeshya, Ray W. Carpenter and Ken Streib, "Mg(OH)₂ Dehydroxylation: A Lamellar Nucleation and Growth Process", presented in Symposium Q at the Fall 1999 Materials, Research Society Meeting in Boston, Massachusetts.

Renu Sharma, Eberhard Schweda, Dirk Naedele, "In Situ Study of Zirconia Stabilization by Anion Exchange (N for O) Using High Temperature, Controlled Atmosphere Electron Diffraction", "Proc. Mat. Res. Society (1999), also to be published in the Proceedings (in press).

R. Sharma and Peter Crozier (invited), "Time and Temperature Resolved Chemical Analysis of Catalysts and Catalyst Supports During Reduction Using E-Cell Combined with GIF", Symposium at Haldor Topsoe, Copenhagen, Denmark, August 1999 (1999).

Michael J. McKelvy, Renu Sharma, Andrew V.G. Chizmeshya, Hamdallah Bearat, Ray W. Carpenter, and Ken Streib, (invited), "Mg(OH)₂ Dehydroxylation: The Path to Brucite CO₂ Mineral Sequestration," an presentation at the Third Carbon Dioxide Mineral Sequestration Forum at the Federal Energy Technology Center, Pittsburgh, Pennsylvania, June 29, 1999.

Michael J. McKelvy, Renu Sharma, Andrew V.G. Chizmeshya, Ray W. Carpenter and Ken Streib, "Mg(OH)₂ Dehydroxylation: A Lamellar Nucleation and Growth Process", presented at the 1999 University Coal Research Contractors Review Conference, Pittsburgh, Pennsylvania, June 1-2, 1999.

Publications

Michael J. McKelvy, Renu Sharma, Andrew, V.G. Chizmeshya, Hamdallah Bearat, and R.W. Carpenter, "Mg(OH)₂ Dehydroxylation: Implications for Enhancing CO₂ Mineral Sequestration Reaction Processes", Proc. 25th International Technical Conference on Coal Utilization & Fuel Systems, (2000) 897-908.

Richard P. Walters, Zhong-Ying Chen, Philip Goldberg, Klaus Lackner, Michael McKelvy, and Hans Ziock, "Mineral Carbonation: A Viable Method for CO₂ Sequestration. Program Plan and Approach," FETC U.S. Department of Energy Global Climate Change Center of Excellence (1999), <http://www.fetc.doe.gov/products/gcc/index.html>.

R. Sharma and Peter Crozier, "In Situ Electron Microscopy of CeO₂ and CeO₂-ZrO₂ Reduction.", Electron Microscopy and Analysis 1999 (1999) 569-572.

L. Diebolt, R. Sharma, M. McKelvy, and W. Glaunsinger, "PreIntercalation Host-Layer "Loosening" and the Role of Elastic Strain: Atomic Level Observation of Early Events in Molecular Intercalation", Molec. Cryst. Liq. Cryst., 311 (1998) 389-96.

Renu Sharma and Karl Weiss (invited), "Development of a TEM to Study In Situ Structural and Chemical Changes at Atomic Level During Gas Solid Interaction at Elevated Temperatures", Microscopy Research and Techniques, 42 (1998) 270-280.

Renu Sharma and Karl Weiss, "An Environmental TEM for High Resolution Imaging, Electron Diffraction, Electron Energy Loss Spectroscopy and Energy Filtered Imaging under Controlled Gaseous Pressure and Temperature". ICEM 14, Vol. III (1998) 551-552.

Peter Crozier and Renu Sharma, "In Situ Oxidation and Reduction of Pd Particles". ICEM 14, Vol. III (1998) 499-500.

Peter Crozier and Renu Sharma, "In Situ Oxidation and Reduction of small Pd Particles on Silica"., Proc. 52nd Ann. Meet. Microscopy Society of America (1998).

END