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MRI: Acquisition of a Multi-User X-ray Diffraction System for Advanced Materials Analysis

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Final Report for Period: 09/2007 - 08/2008**Submitted on:** 12/17/2008**Principal Investigator:** Lad, Robert J.**Award ID:** 0521043**Organization:** University of Maine**Submitted By:**

Lad, Robert - Principal Investigator

Title:

MRI: Acquisition of a Multi-User X-ray Diffraction System for Advanced Materials Analysis

Project Participants**Senior Personnel****Name:** Lad, Robert**Worked for more than 160 Hours:** Yes**Contribution to Project:****Name:** Frankel, David**Worked for more than 160 Hours:** Yes**Contribution to Project:****Name:** DeSisto, William**Worked for more than 160 Hours:** Yes**Contribution to Project:****Name:** Vetelino, John**Worked for more than 160 Hours:** Yes**Contribution to Project:****Name:** Tripp, Carl**Worked for more than 160 Hours:** Yes**Contribution to Project:****Post-doc****Name:** Xie, Xinfeng**Worked for more than 160 Hours:** Yes**Contribution to Project:**

studies of wood/ carbon fiber composites

Graduate Student**Name:** Howell, Caitlin**Worked for more than 160 Hours:** Yes**Contribution to Project:**

MS student in biology; studies of wood structure

Name: Zhang, Xuefei**Worked for more than 160 Hours:** Yes**Contribution to Project:**

MS student in Physics; studies of ZrSiN thin films

Name: Sturtevant, Blake**Worked for more than 160 Hours:** Yes

Contribution to Project:

PhD student in Physics; diffraction from langatate single crystals

Name: Stone, Thomas**Worked for more than 160 Hours:** Yes**Contribution to Project:**

PhD student in Physics; studies of AlN/InN multilayer structures

Name: Galimore, Dana**Worked for more than 160 Hours:** Yes**Contribution to Project:**

XRD of photoresist films; IGERT support

Name: McGann, Jason**Worked for more than 160 Hours:** No**Contribution to Project:**XRD of LiTaO₃**Name:** Clark, Aaron**Worked for more than 160 Hours:** No**Contribution to Project:**

XRD of microhotplate structures

Name: Byrne, Mark**Worked for more than 160 Hours:** Yes**Contribution to Project:**

XRD of Au, SiZrON films; supported by IGERT

Name: Li, Lei**Worked for more than 160 Hours:** No**Contribution to Project:**

XRD of cellulose films

Name: Steeves, Michael**Worked for more than 160 Hours:** Yes**Contribution to Project:**XRD of Ru and RuO₂ films**Name:** Kiziltas, Alper**Worked for more than 160 Hours:** No**Contribution to Project:**

XRD of microcellulose

Name: Mba, Joel**Worked for more than 160 Hours:** No**Contribution to Project:**

XRD of chacolgenide materials

Undergraduate Student**Name:** Kirkmann, Tyler**Worked for more than 160 Hours:** Yes**Contribution to Project:**

undergraduate research in chemistry; studies of zeolite powders

Name: Lenferink, Hendrik**Worked for more than 160 Hours:** Yes

Contribution to Project:

senior project in physics and geology; diffraction from carbon thin films

Name: Sylvestre, Jacob

Worked for more than 160 Hours: Yes

Contribution to Project:

XRD of poly-Si films; supported by National Semiconductor

Name: Walsh, Brenna

Worked for more than 160 Hours: Yes

Contribution to Project:

XRD of catalyst powders

Name: Siegfriedt, Abigail

Worked for more than 160 Hours: Yes

Contribution to Project:

XRD of inorganic membranes

Technician, Programmer

Name: Call, Michael

Worked for more than 160 Hours: Yes

Contribution to Project:

diffraction analysis of langatate single crystals

Other Participant**Research Experience for Undergraduates**

Name: Wright, Matthew

Worked for more than 160 Hours: Yes

Contribution to Project:

REU participant and undergraduate senior honors thesis in Physics; diffraction of silver and silver oxide thin films

Years of schooling completed: Junior

Home Institution: Same as Research Site

Home Institution if Other:

Home Institution Highest Degree Granted(in fields supported by NSF): Bachelor's Degree

Fiscal year(s) REU Participant supported: 2006

REU Funding: REU site award

Organizational Partners**National Semiconductor**

studies of poly silicon processing

Zeomatrix, LLC

structure of zeolite materials

Mainely Sensors, LLC

structural studies of piezoelectric materials

Orono Spectral Solutions

structure of porous oxide materials

E-Dots LLC

XRD of Eu nanoparticles

Other Collaborators or Contacts

Oak Ridge National Lab - High Temperature Laboratory - Dr. Andrew Payzant - collaboration on XRD studies of langatite single crystal materials

Activities and Findings**Research and Education Activities:**

A high performance PANalytical X'Pert PRO MRD X-ray Diffraction (XRD) system was acquired and it is providing critical information about the structural properties of materials at the atomic, molecular, and nanometer scale. The instrument consists of an x-ray generator, x-ray mirrors and lenses, a hybrid monochromator, a high resolution goniometer and sample cradle, slits and collimators, a fast multichannel detector and Xe proportional counter, and a high temperature sample stage. Specific measurement capabilities of the XRD system include phase analysis, high resolution reciprocal space mapping, pole figure analysis, rocking curve analysis, reflectometry, stress and texture analysis, and topography.

The XRD system is being used in numerous materials research and education projects at UMaine. Specific materials systems that have been characterized include (i) zeolite powder materials (ii) quartz, LiTaO₃, langasite, and langatite piezoelectric single crystals (iii) Pt-Rh alloy high temperature electrode materials (iv) GaN thin films (v) poly-silicon film structures (vi) silver oxide thin films (vii) As-S-Se gas sensing films (viii) Ni/TiO₂ multilayer structures (ix) ZrSiN and SiAlON films (x) WO₃ gas sensing films (xi) decaying wood samples (xii) carbon nanotubes and thin films (xiii) wood/carbon fiber composites (xiv) AlN/InN multilayer structures (xv) catalyst powders (xvi) cellulose films and fibers (xvii) Ru and RuO₂ films (xviii) polymer photoresists and (xix) inorganic membranes. The XRD system has been used as a training tool for postdocs, graduate students, IGERT students, GK/12 students, REU and RET participants, and undergraduate students. It has also been used for collaborative projects with several industrial partners.

Findings:

The XRD results obtained for the materials systems mentioned above revealed information about crystal structure, crystallographic texture, strain, grain size, roughness, and film thickness. Examples of five specific results are follows.

Example #1: Evaporation of Ag in the presence of an electron cyclotron resonance (ECR) oxygen plasma was used to deposit Ag₂-xO films with a range of stoichiometries onto r-plane sapphire substrates. XRD analysis indicated that the Ag₂-xO films are not single phase but rather contain signatures of coexisting Ag₂O and AgO components. XRD shows that the lattice matching with the r-plane sapphire substrate causes the Ag₂O phase to grow with <002> heteroepitaxial crystallites coexisting with crystallites having <111> normal and random in-plane orientation. The AgO phase also forms with crystallites having <002> heteroepitaxy as well as crystallites with <111> normal and random in-plane orientation.

Example #2: SiAlON and ZrSiN ceramic films were deposited by rf magnetron co-sputtering. XRD indicated that the SiAlON films remain amorphous after processing in high temperature environments. The ZrSiN films were studied as a function of the Si content. At very low Si concentration, the fcc ZrN structure forms with high quality (100) epitaxy on r-plane sapphire as demonstrated by XRD pole figure analysis. Small amounts of Si (up to 6%) added to the ZrN lattice cause the Zr_{1-x}Si_xN films to become polycrystalline, whereas higher amounts of Si (above ~15%) cause the films to become amorphous.

Example #3: XRD was used to analyze biomodification of crystalline cellulose by wood decay fungi. Measured quantities included the average width of the cellulose microcrystals and the percent of crystalline cellulose within the wood as a function of degradation. These data are leading to an understanding of the mechanisms of wood degradation to facilitate the development of more effective decay prevention measures.

Example #4: Small angle (0-2 θ) XRD was measured on ordered, mesoporous silica catalyst support materials to provide information on the periodicity of the 2-D hexagonal array of mesopores which, combined with nitrogen adsorption measurements, allowed the wall thickness to be estimated. The pore periodicity was also measured after supports were impregnated with cobalt or iron catalysts and following reaction measurements. XRD was also used to determine the phase of cobalt and iron oxides in the as-prepared catalyst, after hydrogen reduction, and

post reaction under Fischer Tropsch conditions. Calculation of the activity of the catalyst, expressed in terms of the turn over frequency, requires an estimate of the number of surface cobalt atoms. The linewidths in the X-ray diffraction patterns were analyzed (using the Scherer equation) to estimate the crystallite size, from which the dispersion was calculated.

Example #5: Ordered mesoporous silica films were prepared using surfactants as templates. Typical synthesis conditions include dip-coating of a support with a solution containing the surfactant and a molecular source of silica (tetraethylorthosilicate). Under specific conditions of humidity, temperature, solution aging and solution composition, thin layers of silica containing an ordered mesoporous structure are fabricated. XRD was used to study pore-ordering as a function of temperature and humidity during dip-coating. These films have applications in membrane technology for a wide variety of separations.

Training and Development:

The XRD system has been the centerpiece for training students at all levels, including postdocs, graduate students, and undergrads. Hands-on-experience has demonstrated the nuances of structural analysis using XRD. Several of the users took a newly developed graduate course PHY500-Fundamentals of X-ray Diffraction taught by R.J. Lad and D.J. Frankel, and applied the knowledge to their specific thesis topic. The XRD system has also been highlighted for students and teachers participating in the REU, RET, IGERT, and GK/12 programs at UMaine. Steven Rivers, a visitor on sabbatical from Rhode Island College, participated in the original commissioning of the instrument, and was involved in the characterization of several materials systems.

Outreach Activities:

The XRD system has been featured on several tours of 'nanotechnology research' to different constituents. These outreach groups include the Expanding Your Horizons program for middle school girls; the Maine Principals Association Science Fair; Nanoscience Day offered to students from Colby College; several Maine legislators; middle school students from Brewer, ME and Hampden, ME; high school students from Bangor High School; Girl Scouts from Portland, ME; ROTC students from Lewiston, ME; 4-H groups; and native American students from Indian Island High School, ME.

Journal Publications

Rivers, S.B.; Bernhardt, G.; Wright M.W.; Frankel, D.J.; Steeves, M.M.; Lad, R.J., "Structure, Conductivity, and Optical Absorption of Ag₂-xO Films", *Thin Solid Films*, p. 868, vol. 515, (2007). Published,

D.J. Frankel, G. Bernhardt, T. Moonlight, M. Pereira da Cunha, B. Sturtevant, R.J. Lad, "Stable Electrodes and Ultrathin Passivation Coatings for High Temperature Sensors in Harsh Environments", *The 7th IEEE Conference on Sensors Proceedings, Lecce, Italy*, p. , vol. , (2008). Accepted,

M. Pereira da Cunha, T. Moonlight, R.J. Lad, G.P. Bernhardt, D.J. Frankel, "Enabling Very High Temperature Acoustic Wave Devices for Sensor & Frequency Control Applications", *IEEE 2007 International Ultrasonics Symposium Proceedings, New York, NY*, p. 2107, vol. , (2007). Published,

M. Pereira da Cunha, T. Moonlight, R.J. Lad, D.J. Frankel, G. Bernhardt, "High Temperature Sensing Technology for Applications Up To 1000°C", *7th IEEE Conference on Sensors Proceedings, Lecce, Italy*, p. , vol. , (2008). Accepted,

Xinfeng Xie, Barry Goodell, Yuhui Qian, Michael Peterson, Jody Jellison, "Significance of the heating rate on the physical properties of carbonized maple wood", *Holzforschung*, p. 591, vol. 62, (2008). Published,

B. Sturtevant, M. Pereira da Cunha, R.J. Lad, "Determination of the Absolute Orientation of Langatate Crystals using X-ray Diffraction", *Proc. 2008 IEEE International Ultrasonics Symposium*, p. , vol. , (2008). Accepted,

L.D. Doucette, T.M. Christensen, W.J. DeSisto, R.J. Lad, "CrO₂ (100) and TiO₂ (200) Film Heteroepitaxy on a BaF₂(111) / Si(100) Substrate", *Journal of Crystal Growth*, p. 653, vol. 290, (2006). Published,

Howell, C., Hastrup, A.C., Jellison, J., "The use of X-ray diffraction for analyzing biomodification of crystalline cellulose by wood decay fungi", *International Research Group on Wood Protection*, p. 10622, vol. IRG/WP, (2007). Published,

Howell, C. and Jellison, J., "Biological variability in the oxalate/oxalate decarboxylase system among five isolates of the wood-degrading fungus *Meruliporia incrassata*", International Research Group on Wood Protection, p. 10573, vol. IRG/WP, (2006). Published,

H. P. Pendse, M. C. Wheeler, W. J. DeSisto, B. G. Frederick, A. van Heiningen, "Thermochemical Conversion of Woody Biomass to Fuels and Chemicals", First Annual Conference on Cellulosic Biofuels, p. , vol. , (2008). Accepted,

Books or Other One-time Publications

Xuefei Zhang, "Synthesis and Characterization of Zr_{1-x}Si_xN Thin Film Materials", (2007). Thesis, Published
Bibliography: MS Thesis, University of Maine

J.I. Krassikoff, "Multi-Functional SiAlON Thin Film Coatings for High Temperature Applications", (2007). Thesis, Published
Bibliography: MS Thesis, University of Maine

Caitlin Howell, "Understanding Wood Biodegradation Through the Characterization of Crystalline Cellulose Nanostructures", (2008). Thesis, Published
Bibliography: MS Thesis, University of Maine

Web/Internet Site

Other Specific Products

Contributions

Contributions within Discipline:

The results of high resolution XRD analysis have yielded fundamental information about (i) semiconductor gas sensor films and ceramic coatings, (ii) novel piezoelectric single crystals for acoustic wave devices and sensors, (iii) mesoporous and nanoporous materials for chemical detection, chemical separation, and catalysis (iv) dielectrics for microelectronics, (v) composite materials, (vi) zeolites and inorganic membranes, and (vii) cellulosic and wood structures. In most of these materials systems, the XRD results have been complemented by other analysis techniques.

Contributions to Other Disciplines:

The most significant extension of XRD analysis to other disciplines is the utilization of XRD to probe the structure and degradation of wood by fungi. This project brings together researchers from physics, biology, and wood science disciplines. These results stimulated additional activity in characterizing the structures of cellulose films and nanofibers.

Contributions to Human Resource Development:

The XRD system was used in the lab component of the course 'Foundations of Sensors' developed under NSF IGERT support. GK/12 students also participated in a XRD lab session. Two RET participants had hands-on XRD experience. A new graduate course in Fundamentals of X-ray Diffraction was developed that complements the hands-on training on the XRD instrument.

Contributions to Resources for Research and Education:

The PANalytical X'Pert PRO MRD X-ray Diffraction (XRD) system adds significant capabilities to the overall facilities for nanotechnology research centered within the Laboratory for Surface Science & Technology at UMaine. The XRD system is available for general use by researchers on the UMaine campus and also by collaborators from throughout the State of Maine.

Contributions Beyond Science and Engineering:

The XRD instrument has been utilized in R&D activities carried out by spin-off companies from UMaine technology, including Mainely Sensors, Orono Spectral Solutions, and Zeomatrix. A major project was carried out under funding by National Semiconductor Corporation, located in South Portland, ME, to characterize poly-silicon films, which aided their wafer scale processing technology.

Categories for which nothing is reported:

Any Web/Internet Site

Any Product