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Acquisition of a Multi-User Thin Film Synthesis and Processing Facility

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Organization: University of Maine

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Contribution to Project:
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Organizational Partners

Sensor Research and Development Corp
Collaboration on research and development of thin film chemical and biological sensors for warfare agent detection, medical diagnostics, food quality monitoring, and fuel combustion control.

BIODE, Inc.
Collaboration on research and development of thin films for liquid phase sensors and piezoelectric devices.

First Light Technology
Collaborative research on ceramic-based optical coatings for DVD technology.

Fairchild Semiconductor
Collaboration on fabrication and characterization of chemical oxides for bipolar transistor technology.

University of New Hampshire
Collaboration with Prof. James Krzanowski, Mechanical Engineering Department at UNH, on carbide thin films.

Oak Ridge National Laboratory
Collaboration with Dr. Peter Blau, High Temperature Materials Laboratory at ORNL on tribological testing of ceramic thin films.

Sandia National Laboratory
Collaboration with Dr. Charles Barbour on ECR plasma deposition of aluminum oxide films

Other Collaborators or Contacts

Activities and Findings

Project Activities and Findings:
This NSF Instrumentation Grant has been used to establish a state-of-the-art Thin Film Synthesis and Processing Facility to fabricate, characterize, and develop novel ceramic-based thin film materials. The facility consists of instrumentation which allows for precise control of film structures and compositions, including molecular beam epitaxy, ion-assisted deposition, and plasma-based techniques. During the five year period of the award, the research activities focused primarily on three important materials technologies: (1) solid-state chemical / biological sensing films (2) lubricating ceramic films and hard coatings for high temperature applications and (3) epitaxial growth of oxide-based ceramics. The instrumentation was also used to support thin film development projects in optoelectronics and microelectronics in collaboration with Maine industries.

In the Thin Film Synthesis & Processing Facility, four ultra-high vacuum chambers (10-10 Torr) are interconnected by a sample trolley transportation system in order to perform in situ analysis and characterization during film deposition and post-deposition processing. The system was designed in collaboration with DCA Instruments, Inc., Turku, Finland, and assembled in the Laboratory for Surface Science & Technology at the University of Maine using components from several vendors. The heart of the facility is a 24 inch diameter ultra-high vacuum deposition chamber equipped with many well-controlled deposition sources that can be simultaneously focused onto a 2 inch substrate area. These sources include an electron cyclotron resonance (ECR) microwave oxygen / nitrogen plasma source, a 4-pocket electron beam evaporator, a single-pocket electron beam evaporator, a Kaufman ion source, two magnetron sputtering sources with off-axis tilt, and two effusion cells. Film structure and interface characterization is carried out in situ during growth using reflection high energy electron diffraction (RHEED).

Two thin film analysis chambers provide capabilities for x-ray and ultra-violet photoelectron spectroscopy using a hemispherical analyzer (XPS and UPS), Auger spectroscopy using a cylindrical mirror analyzer (AES), azimuthal sample rotation during ion beam AES depth profiling, low energy electron diffraction (LEED), low energy ion scattering (LEIS), and thermal desorption spectroscopy.

A thin film electrical characterization chamber consists of a unique Hall effect probe station that permits in-situ 4-point conductivity and carrier mobility characterization of films under controlled gas exposure from ultra-high vacuum to atmospheric pressure and from room temperature up to 500degC. A mass flow controller / capacitance manometer based gas admission system allows the films to be exposed to a range of pure test gases and gas mixtures at pressures from 10-9 Torr up to 1 atmosphere. This Hall effect system is operated under computer control and is especially geared towards studies of high resistance semiconducting metal oxide films used in chemical sensing applications.

Project Training and Development:
The Thin Film Synthesis & Processing Facility was used to obtain significant accomplishments in the following areas: (1) development of semiconducting metal oxide (SMO) and piezoelectric films for chemical and biological sensor applications (2) studies of oxide-based films exhibiting low friction and wear for applications as hard corrosion-resistant and shock-resistant coatings at extreme high temperatures and (3) characterization of epitaxial oxide thin film growth mechanisms and structural properties.

Thin Films for Solid-State Chemical/Biological Sensors:

The microstructure of semiconducting metal oxide (SMO) films has a major influence on the performance of chemiresistive and surface acoustic wave chemical sensors. Several deposition techniques were used to produce SMO films (WO3, SnO2, RuO2, TiO2, In2O3, ITO, CuO) containing epitaxial, highly textured, random polycrystalline, or amorphous microstructures. These films were also synthesized with various catalytic additives such as Au and Pt to enhance sensor sensitivity and were characterized using electron spectroscopies and diffraction to determine film deposition parameter / microstructure relationships. The capability of performing in situ film conductivity and Hall effect measurements in ultra-high vacuum as well as in controlled gas environments at atmospheric pressure allowed correlations to be made concerning deposition parameter / microstructure / sensor response for several SMO systems. Piezoelectric AlN thin films synthesized using ECR oxygen-plasma-assisted growth have been also developed for use in acoustic-based sensors. Several oxide coatings (SiO2-based and Al2O3-based) have also been developed for liquid phase biosensors. As a result of the unique capabilities of the thin film facility, several sensor related projects were funded in areas such as chemical / biological warfare agent detection (ONR), medical diagnostics from nitric oxide emissions in human breath (DARPA), fossil fuel combustion control (EPA), food quality monitoring (USDA), bacterial detection and speciation (DOE), and sensors for high temperature corrosive environments (NSF-STTR).

Tribological Ceramic Coatings:

In this project, several different ceramic coatings (ZrO2, WO3, AlOx, AlOxNy) were fabricated with the aim of achieving improved friction and wear behavior, particularly in high temperature aggressive environments. Using the thin film facility, film synthesis and processing parameters were directly correlated with microscopic and macroscopic wear measurements. Thin film and interfacial regions were characterized using diffraction (RHEED, LEED, XRD), electron spectroscopies (XPS, UPS, AES), microscopy (AFM) and tribology instruments (pin-on-disk, friction microprobe, and scratch tester). By precise control of thin film microstructure, a wide range of friction coefficients and wear coefficients were produced and evaluated under different environmental conditions. An increased understanding of the structural and chemical stability of interface regions and tribological performance of ultra-thin oxide films was acquired for these important ceramic systems. The thin film facility helped attract DOE-EPSCoR funding to establish a Research Cluster for Micro-Tribology at the University of Maine.

Epitaxial Oxide Films:

Well-defined oxide surfaces have numerous technological applications in catalysis, magnetic media, optoelectronics, etc. and are also needed for more fundamental studies. We used the thin film facility to synthesize extremely high quality oxide films and surfaces via molecular beam epitaxy techniques. Fundamental studies were carried out to investigate oxide film growth mechanisms (NiO, MgO, ZrO2, Al2O3) on oxide substrates (sapphire and silica). In addition to studying interfacial phenomena such as thermal stability, chemical reactivity, interdiffusion and interfacial impurity effects, the synthesis / property relationships of ultra-thin oxide films were investigated through measurements of resulting structural, electronic, chemical, and mechanical properties. We found that ECR oxygen-plasma-assisted growth was extremely effective in producing high quality crystalline material at relatively low growth temperatures (<500degC) and that substrate-mediated lattice matching across the oxide film / oxide substrate interface stabilized certain nonequilibrium bulk phases in thin film form. Thin oxide films grown in this manner have well-defined structures and controlled defect distributions thereby allowing one to study fundamental properties of these complex systems. This work utilized the NSF-supported thin film facility and was also supported by DOE Basic Energy Sciences.

Research Training:
The Thin Film Synthesis & Processing Facility enabled many people to receive training and experience with state-of-the-art instrumentation for fabrication and characterization of ceramic-based thin films. The level of training ranged in scope from first time use to advanced operation and involved faculty, visiting faculty, staff scientists, industrial collaborators, postdocs, graduate students, and undergraduates. The interdisciplinary nature of the thin film activities extended this training to disciplines including physics, chemistry, electrical engineering, chemical engineering, mechanical engineering, microbiology, and food science. Diversity was integrated into the program at all levels including a large number of NSF-REU undergraduate participants.

Outreach Activities:
In addition to research training, the facility was a showcase for exposing and educating members of the public about materials science and thin film science and technology including members of the Maine legislature, business leaders, alumnae, etc. Tours of the facility were also an effective tool for student recruitment and community outreach including students from local middle schools and high schools, Upward Bound program, Boy Scouts, Expanding Your Horizons program for middle school girls, etc.

Journal Publications

Yan Yu and R.J. Lad, "Structure and Morphology of Clean and Magnesium-Dosed Sapphire Surfaces", *Mat. Res. Soc. Symp. Proc.*, p. 317, vol. 317, (1994).) Published

M.D. Antonik, J.E. Schneider, E.L. Wittman, K. Snow, J.F. Vetelino and R.J. Lad, "Microstructural Effects in WO3 Gas Sensing Films", *Thin Solid Films*, p. 247, vol. 256, (1995).) Published
Yan Yu and R.J. Lad, "Chemical Bonding, Structure, and Morphology of Mg / a-Al2O3 and MgO / a-Al2O3 Interfaces", *Mat. Res. Soc. Symp. Proc.*, p. 47, vol. 357, (1995).) Published
D.A. Marshall, J.C. Barbour, D.M. Fallstaedt, A.J. Howard, and R.J. Lad, "Surface Morphology and Microstructure of Al-O Alloys Grown By ECR Plasma Deposition", *Mat. Res. Soc. Symp. Proc.*, p. 497, vol. 396, (1996).) Published
J.C. Barbour, D.M. Follstaedt, J.A. Knapp, D.A. Marshall, S.M. Myers, and R.J. Lad, "Low-Energy Deposition of High-Strength Al(O) Alloys from an ECR Plasma", *Mat. Res. Soc. Symp. Proc.*, p. 235, vol. 403, (1996).) Published
L.J. LeGore, K. Snow, J.D. Galipeau, and J.F. Vetelino, "Optimization of a WO3 Film for Applications in a SAW Gas Sensor", *Sensors and Actuators B*, p. 164, vol. 35, (1996).) Published
J. LeGore, O. Greenwood, J. Paulus, D. Frankel, and R.J. Lad, "Controlled Growth of WO3 Thin Films", *J. Vac. Sci. Technol. A*, p. 532, vol. 15, (1997).) Published
S.C. Moulzolf, Yan Yu, D.J. Frankel, and R.J. Lad, "Properties of ZrO2 Films on Sapphire Prepared by ECR Oxygen Plasma-Assisted Deposition", *J. Vac. Sci. Technol. A*, p. 1211, vol. 15, (1997).) Published
Yan Yu and R.J. Lad, "R-cut Sapphire Surface Spectra by XPS and UPS: Clean Surface and Ultra-thin Mg Films", *Surface Science Spectra*, p. 207, vol. 4, (1998).) Published
S.C. Moulzolf, D.J. Frankel, and R.J. Lad, "A Novel Shutter Design", *J. Vac. Sci. Technol. A*, p. 766, vol. 16, (1998).) Published
S.D. Dvorak, O.D. Greenwood, W.N. Unertl, and R.J. Lad, "Microtribological Properties of Al-O-N Films", *Mater. Res. Soc. Proc.*, p. 222, vol. 522, (1998).) Published
S.C. Moulzolf, R.J. Lad, and P.J. Blau, "Microstructural Effects on the Friction and Wear of Zirconia Films in Unlubricated Sliding Contact", *Thin Solid Films*, p. 220, vol. 347, (1999).) Published
O.D. Greenwood, S.C. Moulzolf, R.J. Lad, and P.J. Blau, "The Influence of Microstructure on Tribological Properties of WO3 Thin Films", *Wear*, p. 84, vol. 232, (1999).) Published
S.C. Moulzolf and R.J. Lad, "Diffraction Studies of Cubic Phase Stability in Undoped Zirconia Thin Films", *Journal of Materials Research*, p. 369, vol. 15, (2000).) Published
R.J. Lad, "Interactions at Metal/Oxide and Oxide/Oxide Interfaces Studied by Ultra-Thin Film Growth on Single Crystal Oxide Substrates", *Surface Review and Letters*, p. 109, vol. 2, (1995).) Published

Books or Other One-time Publications

S.C. Moulzolf and R.J. Lad, "Electron Cyclotron Resonance Oxygen-Plasma-Assisted Deposition of ZrO2", (1998). *Book*, Published
Editor(s): D.A. Glocker and S.I. Shah
Collection: Handbook of Thin Film Process Technology
Bibliography: IOP Publishing, Philadelphia, PA, pp. X3.8.1-4

G. Bernhardt, D. Frankel, R.J. Lad, K. Nason, and T.A. Carbone, "Composition and Thermal Stability of Chemically Formed SiO2 Oxides", (1999). *Proceedings*, Published
Collection: Advanced Semiconductor Manufacturing Conference Proceedings
Bibliography: IEEE/SEMI

R.J. Lad, "Surface Structure of Crystalline Ceramics", (1996). *Book*, Published
Editor(s): W.N. Unertl
Collection: Physical Structure; Volume One of the Handbook of Surface Science
Bibliography: (Elsevier, Amsterdam) Chapter 5

E. Wittman, D.J. Frankel, K. Snow, P. Patterson, and J.F. Vetelino, "Tungsten Trioxide Films for Surface Acoustic Wave Gas Sensor Applications", (1994). *Proceedings*, Published
Bibliography: Proceedings of Fifth Int'l Mtg. on Chemical Sensors, Rome, Italy, pp. 675-678

J.J. Caron J.D. Galipeau, J.C. Andle, C.J. Freeman, and J.F. Vetelino, "SAW Sensors for Chemical Warfare Agent Detection Using Chemiresistive Semiconducting Metal Oxide Films", (1994). *Book*, Published
Collection: Scientific Conference on Chemical and Biological Defense Research
Bibliography: Edgewood Research Development and Engineering Center, Aberdeen, MD, pp. 727-733

J.J. Caron, R.B. Haskell, and J.F. Vetelino, "Surface Acoustic WAve Mercury Vapor Sensor", (1998). *Proceedings*, Published
Bibliography: Proceedings of Seventh Int'l Mtg. on Chemical Sensors, Beijing, China, pp. 124-126

B. Hu, L.J. LeGore, J.J. Caron, D.J. Dwyer, and J.F. Vetelino, "New Mechanism of Spillover Chemistry: Gold Catalyzed Reduction of WO3", (1998). *Proceedings*, Published
Bibliography: Proceedings of Seventh Int'l Mtg. on Chemical Sensors, Beijing, China, pp. 767-769

Scott C. Moulzolf, "Synthesis and Characterization of ZrO2 Thin Films with Controlled Microstructure", (1999). *Thesis*, Published
Bibliography: Ph.D. Thesis, University of Maine

David A. Marshall, "Surface Morphology and Film Microstructure of Al(O) and Al2O3 Thin Films Grown by ECR Plasma Deposition", (1996). *Thesis*, Published
Bibliography: M.S. Thesis, University of Maine

S.David Dvorak, "Microtribological Studies of Aluminum Oxide Based Ceramics Using a New Nanotribometer", (1998). *Thesis*, Published
Bibliography: Ph.D. Thesis, University of Maine

Joseph W. Paulus, "The Morphology of Reactively Sputtered Tungsten Trioxide Thin Films", (1996). *Thesis*, Published
Bibliography: M.S. Thesis, University of Maine

Web/Internet Sites

URL(s):
http://www.ume.maine.edu/LASST/facilities/thin_film_deposition/thinfilm.htm
Description:
Description, photographs, and schematic drawings of the Thin Film Synthesis and Processing Facility within the Laboratory for Surface Science & Technology at the University of Maine constructed under funding from the NSF Academic Infrastructure Award.

Other Specific Products

Product Type: Instruments or equipment developed
Product Description:
As part of the Thin Film Synthesis & Processing Facility, a unique Hall effect probe station was designed, developed and constructed that enables in situ 4-point conductivity and carrier mobility measurements to be performed on thin film samples both in UHV and undercontrolled atmospheric conditions.

Sharing Information:
The instrumentation is available for use by collaborators from academia, government labs, or industry, particularly for semiconducting metal oxide sensor film development.

Contributions

Contributions within Discipline:
The establishment of a Thin Film Synthesis & Processing Facility has enabled advanced materials development projects to be carried out which involve 'in situ' characterization of electronic, chemical, and structural thin film properties and direct correlation with synthesis and processing parameters. The capability of fabricating ceramic-based thin film materials using a broad range of deposition techniques all within the same vacuum chamber has increased the fundamental knowledge base of oxide thin film growth mechanisms and microstructure control as well as provided the necessary fabrication tools to develop new materials for a range of engineering applications.
Contributions to Other Disciplines:
Development of well-defined ceramic thin film materials has a wide reaching impact on several science and engineering disciplines. This is especially apparent in research and development of oxide films for chemical and biological sensing applications. Progress in this area has impacted researchers in areas including food science, microbiology, medical diagnostics, combustion monitoring, and environmental science.
Contributions to Human Resource Development:
In addition to having a large impact on hands-on student laboratory training and outreach activities as discussed above, the project prompted several curriculum based activities. Graduate level special topics courses were developed and taught in thin film technology, vacuum technology, sensor technology and instrumentation, and chemistry & physics of semiconducting metal oxide films. The facility also augmented several undergraduate opportunities including the NSF-REU program, capstone senior projects, and the McNair Scholars program that encourages under-represented groups to pursue graduate education in science and engineering.
Contributions to Science and Technology Infrastructure:

The Thin Film Synthesis and Processing Facility is the only facility of its type within the State of Maine and is available for general purpose use. The facility has been used as a resource by several small spin-off companies as well as larger Maine industries that focus on microelectronics, sensors, paper technology, and composite materials.

Beyond Science and Engineering:

The project has provided infrastructure to help build and establish an Environmental Sensor Research Group at the University of Maine that involves industrial partners in the area of sensor technology. Thin film technology developed in the facility has been used to advance sensors to fulfill a variety of societal needs ranging from air quality and environmental monitoring, hazardous waste detection, industrial process control, food quality monitoring, medical diagnostics, and chemical and biological warfare agent detection.