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IMR
Institute For Materials Research

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THE OHIO STATE UNIVERSITY

Super Insights into "Superalloys"

By Michael Mills, Professor of Materials Science and Engineering; research funded by the Air Force Office of Scientific Research

Improving the efficiency of turbine engines for both aerospace propulsion and land-based power generation requires materials that can withstand the incredible stresses and temperatures that are generated during combustion of the fuel. "Superalloys" are the only class of materials that presently can provide the required strength and predictable performance at elevated temperature that engine designers require. These remarkable materials, which consist primarily of an FCC solid solution and ordered γ' precipitates, have been developed and improved for decades through a largely empirical process, without a thorough understanding of the basic physics that govern their strength. A research team led by Materials Science and Engineering Professor and IMR Associate Director Michael Mills attempts to address this problem. The researchers, including Dr. Libor Kovarik and Dr. Raymond Unocic, have combined deformation experiments, detailed substructure characterization and state-of-the-art modeling techniques to provide fundamental insights into the deformation physics.

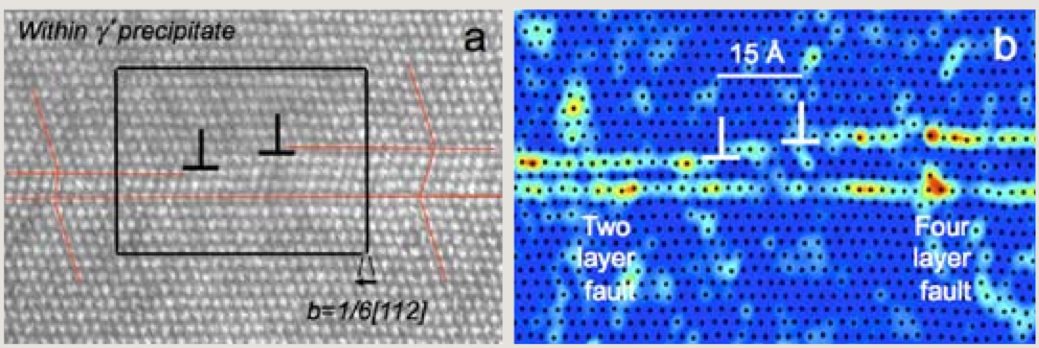


Figure 1.
(a) High resolution scanning transmission electron microscope image of a microtwinning, showing a transition from two to four layers, with the twinning dislocation positions indicated.
(b) Image colorized to show deviation from perfect crystal symmetry.

Continued on page 4

Faculty Spotlight: Jay Gupta

In this issue, we focus on Jay Gupta, Assistant Professor of Physics since 2004. See page 7 for more details about Dr. Gupta's research interests and examples of recent research.

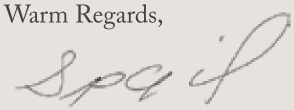
Director's Note

Dear Colleagues,

These are exciting times to be a materials researcher at Ohio State. The Center for Emergent Materials is entering its second year as OSU's first NSF-supported Materials Research Science and Engineering Center. The NSF-supported Nanoscale Science and Engineering Center – Center for Affordable Nanoengineering of Polymeric Biomedical Devices is completing its renewal process. Our state-supported Wright Center for Excellence in Photovoltaics Innovation and Commercialization is maturing into a force in the national photovoltaics scene. The recent award of a statewide Ohio Research Scholars Cluster in Technology-Enabling and Emergent Materials has brought new endowed faculty lines in areas from bio-based materials chemistry to next generation of structural characterization. At the same time, our laboratories continue to expand at an astonishing rate, which has encouraged IMR to begin the development of its Integrated Laboratory Management process in this coming academic year. Examples of new facilities are the first GaN-based MBE system at Ohio State located in our epitaxy hub in Dreese Lab, which is part of the IMR cluster hiring in electronic materials and photovoltaics that brought Professors Roberto Myers and Siddharth Rajan jointly to the Departments of Electrical & Computer Engineering and Materials Science & Engineering, and new core fabrication tools at Nanotech West Laboratory such as an ICP-RIE capability and a sputter deposition system that will soon be integrated within the Nanotech cleanrooms. The culmination of our continued growth and successes is palpable in the excitement that is present at both student and faculty ranks across the colleges and departments who count materials as vital to their strategic research.

In this second IMR Quarterly newsletter, we feature the research of Prof. Dennis Bong of the Department of Chemistry in the area of biomolecular material assembly, new research on "superalloys" by Prof. Michael Mills of the Department of Materials Science and Engineering and IMR Associate Director, and we are thrilled to highlight Prof. Jay Gupta and his work in the Department of Physics through this edition's Faculty Spotlight.

Warm Regards,



Steven A. Ringel, Ph.D.
Neal A. Smith Chair Professor
Director, Institute for Materials Research (IMR)

IMR's New External Advisory Board Created

IMR's newly formed External Advisory Board will hold its first meeting during our 2009 IMR Materials Week event. The Board will conduct a comprehensive review of IMR and its activities to date, and provide valuable feedback and recommendations to ensure IMR's continued future success.

IMR External Advisory Board Members (confirmed as of press time):









Jeff Wadsworth, Chief Executive Office, Batelle and IMR External Advisory Board Chairperson
Timothy Armstrong, Vice President of Research and Development, Carpenter Technology
Robert Chau, Senior Fellow, Intel Corporation
David Eaglesham, Vice President of Technology, First Solar
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Thomas Zacharia, Deputy Director for Science and Technology, Oak Ridge National Laboratory

The IMR External Advisory Board's purpose is to provide IMR leadership with industry and federal lab perspectives and guidance to help ensure the relevance of IMR activities in the OSU and global materials communities. An important goal for the EAB is to assist IMR in maximizing its impact and to enhance its collaborations with partners from the industrial and non-profit sectors, including federal laboratories, by providing advice on both technical directions and mechanisms for interactions with external organizations.

Facilities Updates

Each issue of *IMR Quarterly* includes updates from OSU's materials-allied research facilities. This issue brings reports on four new instruments from Nanotech West Laboratory and the Dreese Laboratories' cleanroom.

Nanotech West Laboratory: 3 New Research Instruments Purchased

OSU's Nanotech West Laboratory recently acquired an Orion-**8 sputtering system** from AJA International, Inc. This UHV instrument features five 3-inch sputtering sources, with two sources optimized for the deposition of magnetic materials. The system is capable of handling samples as large as 6-inch wafers and is outfitted with a load lock for quick turn-around. Other features of the instrument include RF and DC power supplies for sputtering, substrate biasing for sample precleaning and a substrate heater with the achievable temperature as high as 800°C. The system has been tested and accepted by IMR Member of Technical Staff Dr. Denis Pelekhov, and will be available within the next few months to OSU and industry researchers. To schedule training and use of this instrument, please contact ENSL Director Dr. Denis Pelekhov at *dtt@mps.ohio-state.edu.*

Using funds from the Wright Center for Photovoltaics Innovation and Commercialization (PVIC), the Nanotech West Laboratory recently purchased a refurbished **inductively-coupled plasma reactive ion etch (ICP-RIE) tool.** The tool, a load-locked Plasma-Therm SLR770, is now available to users on a limited basis. The tool allows the low-damage, highly-controlled etching of a wide variety of substrates and thin films. CF_4, Ar, and O_2 gases are currently available, and CHF_3 ,Cl_2, and BCl_3 will be available in the near future.


ICP-RIE tool

Nanotech West has also accepted the delivery of a Nanotech Innovations SSP-354 **carbon nanotube deposition system.** The system is a commercialization of technology developed at the NASA Glenn Research Center in Cleveland, and uses a novel organometallic precursor to create high-quality multi-wall carbon nanotubes (MWCNT).

Dreese Laboratories Cleanroom: New Veeco 930 Nitride MBE Installed

A new Veeco 930 **Molecular Beam Epitaxy (MBE) for nitride growth** was installed in June 2009 and now resides in the 095 Dreese Lab cleanroom as part of the Semiconductor Epitaxy and Analsyis Laboratory (SEAL). This new instrument was procured by Professors Siddharth Rajan and Roberto Myers, two new Assistant Professors with joint appointments in the Departments of Electrical and Computer Engineering and Materials Science and Engineering, with resources provided by both departments and the Institute for Materials Research. The addition of the Veeco N_2MBE enhances the growth capabilities of this epitaxy lab, expanding research into a wide array of new technological applications including next-generation solid state lighting, solar power, high data-rate communications, and energy efficient electric transportation systems.

MBE is an advanced epitaxial growth technique, heating ultra pure material to evaporation temperature in an ultra high vacuum (UHV) environment producing a molecular beam of atoms with a mean free path long enough to reach the seed crystal with little or no atomic interruption. An RF nitrogen plasma source is used to create optimal conditions for high quality nitride material and the system is adept to grow materials with group III constituents gallium, indium and aluminum as well as n-type and p-type doping, silicon and magnesium, respectively. In addition, a high temperature effusion cell containing the rare earth dopant gadolinium (Gd) is installed to engineer magnetic properties into GaN for magnetoelectronics and spin electronics. The N_2MBE can produce crystalline material uniform up to 3" diameter and is fully equipped with a high temperature substrate manipulator (1200°C), a standard amenity to properly grow epitaxial nitride based semiconductors. For more information on this tool or the SEAL laboratory, contact Lab Manager Mark Brenner at *brennerm@ece.osu.edu.*

Super Insights into "Superalloys," Continued

One of the team's important findings is that the mechanism of microtwinning is an important deformation mode for Ni-based superalloys deformed at intermediate temperatures (600-700°C). Deformation twinning is classically associated with low temperature and high strain rate conditions, yet this mechanism dominates the deformation of these alloys at higher temperature and very low deformation rates. An example of a microtwin is shown in the "high angle annular dark field" image shown in Figure 1.

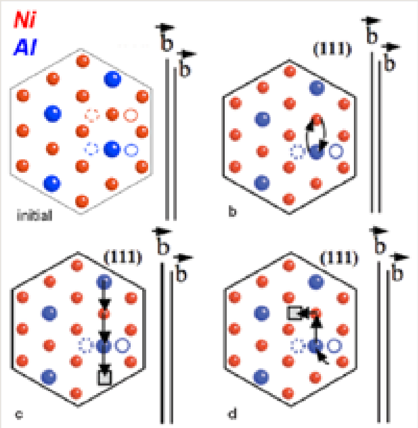

Figure 2. Examples of reordering processes which can convert a high energy, "pseudotwin" (a) into a low energy fault, eliminating high energy Al-Al nearest-neighbors. Process (d) is the lowest energy pathway for reordering as determined using *ab initio* calculations.

This image was obtained using the FEI Titan 3 scanning transmission electron microscope – OSU's most advanced, sub-angstron resolution microscope which is housed in the Campus Electron Optics Facility (CEOF) in the Fontana Laboratories building. The Mills Group has studied microtwinning down to the atomic level using this technique, and it has been unambiguously confirmed that its operation relies on thermally-activated reordering of atoms in the wake of the twinning dislocations (which can be seen directly in Figure 1) within the γ' precipitates. The concept of reordering has also been generalized and is found to be relevant for other planar deformation mechanisms operating at the intermediate temperatures in superalloys. Ab initio calculations have revealed the viable reordering pathways through an evaluation of the energy barriers associated with different reordering scenarios. Several examples of atomic rearrangements explored are shown in Figure 2.

The clear "winner" energetically is the vacancy-mediated process shown in Figure 2d. These results provide a sound basis for understanding the strong temperature and deformation-rate dependence exhibited by these alloys. This knowledge may allow materials scientists and engineers to design superalloys with even better properties, which could translate into enormous cost savings for aerospace and power generation industries and consumers.

For more information about this research, contact Professor Michael Mills *mills.108@osu.edu*

MOCVD Installation at Nanotech West Completed

IMR announces the successful installation and facilitization of **OSU's first Metalorganic Chemical Vapor Deposition (MOCVD) system**. As noted in our last quarterly newsletter, this sophisticated research instrument was acquired by the Wright Center for Photovoltaic Innovation and Commercialization (PVIC) and first delivered to OSU in February 2009. Final process qualification on the new Aixtron 3x2" system was initiated in August 2009. Following certification of thickness uniformity, compositional uniformity and material quality for the range of III-V compounds the system will be available in September 2009 to both OSU and industry researchers. The figure to the left shows the results of a sample completed by Dr. John Carlin, IMR Member of Technical Staff, of a GaAs/AlAs distributed Bragg reflector (DBR) calibration grown on GaAs substrates demonstrating excellent uniformity across the three 2" wafers.

An industry standard technique for the deposition of III-V semiconductors and nanostructures, the new MOCVD capability provides OSU's materials community the flexibility to deposit a full range of device quality arsenide, phosphide, antimonide and dilute nitride III-V compounds. With eight metalorganic sources (including trimethyl aluminum, trimethyl gallium, trimethyl indium, trimethyl antimony, diethyl zinc, diethyl tellurium, carbon tetrachloride, and unsymmetrical dimethyl *hydrazine*), three gaseous sources (including arsine, phosphine and dilute silane) and the ability to reach processing temperatures up to 900°C, the new MOCVD compliments the vast processing and characterization capabilities available at NTW and provides a source of high quality materials for photovoltaics, optoelectronic and high speed electronic devices.

For further inquiries about the new MOCVD capabilities, please contact IMR Member of Technical Staff Dr. John Carlin at *carlin.9@osu.edu.*

IMR–Funded Research: Assembly of Biomolecular Materials

By Dr. Dennis Bong, Assistant Professor of Chemistry

Functional biocompatible and bioderived materials have potential impact in both industrial and biomedical applications, including adhesives and coatings from renewable resources and environmentally sensitive materials for use in tissue culture, targeted delivery and controlled release. Professor Dennis Bong and Ph.D. students Oscar Torres, Yun Gong and Mingming Ma have been exploring new methods to combine the function of biomolecules with the utility of known polymeric scaffolds. This research was funded by IMR over two years through an Interdisciplinary Materials Research Grant. One general aspect of biomaterials is order: function often derives from recognition-controlled molecular organization in aqueous milieu, and thus this research team has focused their attention on mimicking the methods used to assemble Nature's materials, namely peptide and lipid assemblies. The challenge of studying macromolecular assembly not just in synthesis, but also in characterization as these materials occupy a size regime between soluble molecules and solid-state materials: instrumentation and expertise available on campus to IMR researchers were essential in carrying out this research.

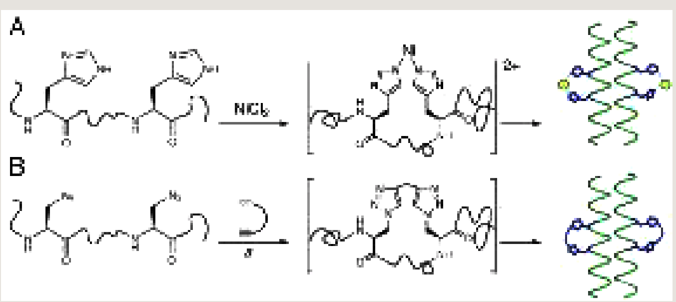

Figure 1. (A) Strategies for helix structure nucleation with i and i+4 crosslinking using metal complexation (top) or double click cycloaddition (bottom).

Peptide Assembly: Metal ion complexation and chemical modification are two new ways of controlling peptide assembly that graduate student Oscar Torres has discovered (1). Peptide helical fold nucleation was triggered by *i* and *i+4* chemical crosslinking of peptide sidechains using: 1) a double "click" cycloaddition with synthetic azido-functionalized amino acids and 2) metal complexation of histidine sidechains (Figure 1). These crosslinking strategies restored peptide dimerization in a folding-incompetent sequence. This method will be expanded to generate novel biomaterials in which assembly and disassembly may be chemically triggered.

Lipid Assembly and Surface Adhesion: Graduate students Mingming Ma and Yun Gong have discovered and analyzed non-native lipid derivatives displaying small molecules cyanuric acid and melamine and found that hydrogen-bonding by these groups in aqueous milieu can modulate lipid membrane interactions, leading to new lipid phases (Figure 2). The aqueous phase recognition of cyanuric acid (CA) and melamine (M) lipid derivatives remains robust and selective in water, suggesting possible applications in new selectively adhesive materials that function in aqueous environment (2). This work is notable and of general interest given the few detailed studies of aqueous phase hydrogen-bonding systems. As such, this work contributes to our understanding of fundamental phenomena such as molecular recognition at the lipid-water interface, membrane chemistry and has possible biomedical and materials applications with regard to selective surface adhesion.

For more information on Dr. Bong's research see his website: http://web.chemistry.ohio-state.edu/~bong/

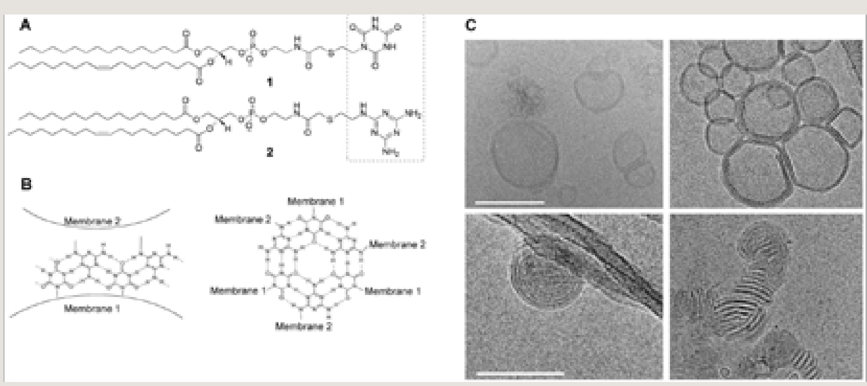

Figure 2. (A) Synthetic lipids 1 and 2, headgroup functionalized with cyanuric acid and melamine (boxed), respectively. (B) Possible hydrogen bonding patterns between lipids in intermembrane contexts. Dotted lines indicate possible hydrogen bonds. (C) (Top Left) Cryo-EM of 1 lamellar phase vesicles formed from lipid 1, (Top Right) lamellar phase lipid 2 vesicles, (Lower Left) hexagonal phase from 1+2 premixed 1:1, (Lower Right) hexagonal phase from lipid 1 vesicle reacted with lipid 2 vesicles. Scale bars (100 nm) in left panels apply to each row.

Reference

1. Torres O, Yuksel D, Bernardina M, Kumar K, & Bong D (2008) Peptide tertiary structure nucleation by side-chain crosslinking with metal complexation and double "click" cycloaddition. *ChemBioChem* 9(11):1701-1705.
2. Ma M, Paredes A, & Bong D (2008) Intra-and intermembrane pairwise molecular recognition between synthetic hydrogen-bonding phospholipids. *J. Am. Chem. Soc.* 130(44):14456-14458.

Faculty Spotlight: Jay Gupta

Jay Gupta joined Ohio State as an Assistant Professor of Physics in 2004. Prior to arriving at OSU, he had been a postdoctoral researcher with Don Eigler at IBM, where he first learned about scanning tunneling microscopy (STM). Jay's research at IBM included studies of carbon monoxide quantum tunneling, single atom spin flip spectroscopy, and hydrogen two-state switching. Jay earned his PhD from the University of California, Santa Barbara working under David Awschalom. Dr. Gupta's research at UCSB involved the measurement and control of electron spins in semiconductor quantum wells and quantum dots using ultrafast laser pulses. He received BS degrees in physics and chemistry at the University of Illinois-Urbana Champaign. The interplay of physics and chemistry has been integral in his research ever since.

Dr. Gupta's current research areas are:

Spin / charge transport through molecules: Spin-based electronics ('spintronics') devices offer inherently new functionalities combined with improved speed and reduced power dissipation. Organic or molecular-based spintronic materials are attractive due to the chemical tunability of strong electronic/magnetic correlations, low-cost fabrication, and intrinsically spin-benign characteristics (e.g. low spin-orbit coupling and small nuclear spin density). However, improved characterization of the interface between active organic layers and electrical contacts is a principal issue in realizing reproducible devices. Using a scanning tunneling microscope, we study charge and spin transport through individual molecules with atomically precise contacts (Figure 1).

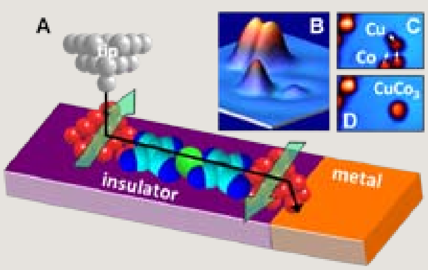

Figure 1. STM studies of single molecule spin transport. (A) Schematic for measuring spin transport through a single molecule with atomically precise contacts. (B) STM image of three TCNE molecules in three distinct charge/configuration states. In the bulk, TCNE is one component of a ferromagnetic organic semiconductor. (C-D) Construction of a ferromagnetic contact from Co atoms on an insulating Cu_2N film.

Spin-spin interactions in semiconductors: Ferromagnetic semiconductors such as Mn-doped GaAs are of interest for spintronic devices that combine both logic and nonvolatile memory. To understand the mechanism for ferromagnetism at an atomic scale, we are studying the magnetic interactions between pairs of Mn using STM. We find that this interaction can be either ferro- or antiferromagnetic, depending on the orientation of the pair with respect to the GaAs crystal lattice. This suggests that growth methods which favor Mn substitution along certain crystallographic directions may improve the ferromagnetism in $Ga_{1-x}Mn_xAs$.

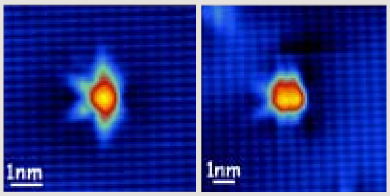

Figure 2. STM images of pairs of Mn atoms, implanted within the surface layer of a GaAs crystal. Rows of Ga atoms in the surface appear as a background grid in the images. (left) a pair of Mn atoms whose spins are ferromagnetically aligned (right) an antiferromagnetic pair.

Evolution of electronic structure in nanoscale clusters: The STM is a versatile tool to monitor the emergence of band structure in nanoscale clusters. For example, we have recently measured the band structure of onemonolayer thick insulating films of Cu_2N, which can be prepared with varying lateral dimensions depending on growth conditions. Our tunneling spectra suggest that the conduction band of the insulator first emerges in islands comprising more than 10 atoms (~ 1 nm^2 in area). Quantum confinement produces a shift toward lower energy as island size increases in the range 1-6 nm^2 , while 'bulk-like' electronic structure is observed for islands > 6 nm^2 in area.

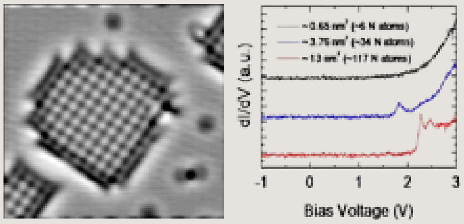

Figure 3. Emergence of the conduction band in onemonolayer thick insulating islands. (left) STM image of a ~5nm^2 Cu_2N island grown on a Cu(100). (right) tunneling spectroscopy showing the evolution of conduction band with island size.

Tip-enhanced studies of surface photochemistry: Optical fields are well known to be locally enhanced near nanostructured metal surfaces. It has been suggested that surface-enhanced optical fields may be used to accelerate photochemical reactions. However it has proven difficult to separate surface-enhanced contributions to the reaction rate from effects due to the wide variety of adsorption sites that are available on nanostructured films. We have built a custom STM with an in situ objective lens to study the influence of tipenhanced optical fields on surface photochemical processes. The advantage of our strategy is that the molecules of interest can be adsorbed on clean, flat single crystal surfaces, so that the adsorption site is well controlled.

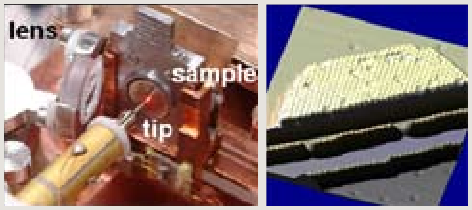

Figure 4. (left) Picture of custom STM stage. (right) STM image of an island of C_{60} on Ag(100). We are studying tip-enhanced photo-polymerization of C_{60} within such islands.

For more information about this research, contact Professor Jay Gupta, *gupta.208@osu.edu*

Request for Proposals:

IMR Facility Grants

For more information
go to our website:

imr.osu.edu
and click on the "Programs" link

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2009 IMR Interdisciplinary Materials Research Grants Awards

IMR's Research Enhancement Program offers three different funding mechanisms to support novel research at The Ohio State University. Our Interdisciplinary Materials Research Grant (IMRG) program provides seed funding to support pioneering interdisciplinary research in materials-allied fields with the goal of generating highly competitive external grant proposals that target large, multi-investigator and center-level opportunities. Grants up to $45,000 are competitively awarded and may be renewable for a second year.

Six new research projects were awarded by the IMR in June 2009, for a total investment of $225,000. Congratulations to the research teams of these 6 new IMRG projects! We hope to see your research results at the 2010 IMR Materials Week!

Metamaterials with Smart Reconfiguration for Broadband RF Antennas
Lead: Marcelo Dapino, Mechanical Engineering;
Co-Applicants: Suresh Babu, Industrial Systems Engineering; John Volakis, Electrical and Computer Engineering.

Economical Platforms for FET-based Protein Detection to Support Sensor Clinical Translation
Lead: Stephen C. Lee, Biomedical Engineering;
Co-Applicant: Paul Berger, Electrical and Computer Engineering.

Use of Electrospun Biomaterials as Carriers of Bone Marrow Derived Stem/Progenitor Cells to Stimulate
Lead: Nicanor I. Moldovan, Internal Medicine;
Co-Applicant: John J. Lannuti, Materials Science and Engineering.

Exploring Electrically Tunable Magnetism in Gd-doped Nitride Quantum Structures
Lead: Roberto C. Myers; Materials Science and Engineering & Electrical Computer Engineering;
Co-Applicants: Ezekiel Johnston-Halperin, Physics; Michael Mills, Materials Science and Engineering.

Synthesis of III-V Semiconductor Nanowire Heterostructures Using Metalorganic Chemical Vapor
Lead: Fengyuan Yang, Materials Science and Engineering;
Co-Applicants: Ezekiel Johnston-Halperin, Physics; Roberto C. Myers, Materials Science and Engineering & Electrical and Computer Engineering.

2009 IMR Materials Week

Join us for this exciting annual event showcasing the most recent discoveries and application in materials research from The Ohio State University and beyond.

This year's meeting will include:

- **Technical workshops and seminars by national experts in a broad range of topics including:**
  - Biosensors
  - Computational materials
  - Magnetoelectronics
  - Photovoltaics
- First year highlights from Ohio State's NSF Materials Research Science and Engineering Center **(NSF MRSEC)**
- **Student poster presentations** featuring work by OSU students in materials-allied fields
- **Evening receptions,** including an exclusive viewing of COSI Columbus' special exhibit "Lost Egypt: Ancient Secrets, Modern Science"

To register for 2009 Materials Week visit our website: **imr.osu.edu**
Blackwell Inn and Conference Center, Columbus, Ohio / August 31– September 3, 2009

2009–2010 IMR Colloquia Series

IMR announces the first colloquium of its 2009–2010 IMR Colloquia Series:
Thursday, October 29, 2009

Kazuhiro Hono
Managing Director of Magnetic Materials Center and Professor of Materials Science, University of Tsukuba
Time and location to be Announced

Institute For Materials Research
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201 West 19th Ave.
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