Professor Arthur Ruoff retires from MS&E

At the end of the Spring 2006 Semester, Arthur L. Ruoff, the Class of 1912 Professor of Engineering in the Department of Materials Science and Engineering at Cornell University will retire. Professor Ruoff helped form MS&E into a department that produces innovative and cutting-edge research while remaining dedicated to excellence in education. His distinguished career in teaching and research has been an inspiration to faculty and students for fifty years.

Professor Ruoff received his BS, with honors, in chemistry from Purdue University in 1952 and his Ph.D., under the direction of Henry Eyring, in Physical Chemistry and Physics from the University of Utah in 1955. He arrived at Cornell in the fall of 1955 as an Assistant Professor in the Department of Mechanics and Materials. He then moved to the Department of Engineering Physics and Materials Science (EP & MS). In 1965, the Department of Materials Science and Engineering was created and Ruoff, along with thirteen other faculty members, became the first faculty of the newly formed department. This is also the year in which Ruoff became a full Professor with tenure. In 1978 he was named a chaired professor.

Professor Ruoff has dedicated his career to the study of the effect of very high pressure on materials. His research has been marked by many impressive achievements. In 1990 he reached a static pressure of 416 GPa, becoming the first scientist to create a static pressure greater than at the center of the earth, 361 GPa. He has carried out optical studies on diamonds and has obtained x-ray diffraction patterns of tungsten at 560 GPa, the highest static pressure obtained to date. Professor Ruoff has published over 300 scientific publications and has been invited to give talks in 18 countries. He wrote two books on Materials Science published by Prentice Hall in 1972 and 1973, and developed an audio-tutorial course on Introductory Materials Science which has been used in 60 universities.

Throughout his career, Professor Ruoff has won many prestigious awards for his achievements in research. In 1993, Professor Ruoff received the Bridgman Medal for outstanding high pressure research from the Association Internationale Pour L’Avancement De La Recherche Et De La Technologie Aux Hautes Pressions (AIRAPT), the International Association for Research at High Pressure.

Alumni Highlight

Paul Osenar, Ph.D., ’92, a leader in cutting-edge fuel cell technology

The Department of Materials Science and Engineering is proud to highlight graduate Paul Osenar, B.S. ‘92, who exemplifies the department’s commitment to excellence, innovation and leadership in research, product development and technology management.

Paul Osenar’s educational background is in materials science, engineering, polymer science, and organic chemistry, with a B.S. from Cornell University and a Ph.D. from the University of Illinois in Materials Science. Initially, Osenar was drawn to greater Boston to work in contract research at Foster-Miller, Inc. given the wide range of projects and the comfortable transition from the more basic research of graduate school. Projects included hydrophobic coatings for MEMS devices and anti-icing, polymer nanoparticles, high temperature proton exchange membranes, and high speed sausage manufacturing.

Early in 2000, Osenar and several others from Foster-Miller formed Protonex. Leveraging their collective experience in fuel cell materials, Protonex was formed to commercialize fuel cell technology for high performance portable and remote applications. Since 2000, Protonex has...
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FROM THE DIRECTOR

It is with great pleasure that we bring to you the Spring 2006 MS&E News. This year the Department of Materials Science and Engineering celebrated its fortieth anniversary. We are very grateful to our Advisory Board Members, Alumni, Faculty and Students who made this event successful. In the celebration we not only focused on our strong tradition of excellence and innovation in education and research, but on the exciting new directions our department will take to continue its leadership position over the coming decades.

We have many things to look forward to this year.

- We are currently conducting an extensive self-study of our Masters of Engineering (M.Eng.) Program. It is our goal to strengthen and grow this program into an exciting and dynamic educational opportunity for our students.

- As part of our long term strategic plan we are continuing to build an infrastructure that will promote the growth and strengthening of our research programs. This year we are continuing laboratory renovations in the subbasement to give our growing number of undergraduate and graduate students, and new faculty, more of the resources and space required to pursue innovation and excellence in research.

- We are conducting a search for an assistant faculty position in energy and computational materials science. These are areas of strategic growth for our department and will enable us to increase the breadth of research conducted by the department helping us to produce innovative and cutting-edge research in emerging fields.

- We are growing our administrative staff to help support the needs of our growing faculty; we have recently hired a new administrative assistant, Dolores Dewbury, and are pleased to welcome her to our team.

- Our Administrative Manager Julie Delay is now the Director of Human Resources for the College of Engineering. This is an exciting new position for her, and on behalf of MS&E, we extend to her our congratulations and wish her all the best in her new position. We are in the process of filling her position.

- Arthur Ruoff, 1912 Professor of Engineering, will be retiring from MS&E at the end of the Spring 2006 semester. He has been an integral part of this department for many years inspiring countless students and faculty. On behalf of the faculty, students and staff, I wish to extend to him our gratitude for his service and dedication to the Department of Materials Science and Engineering at Cornell University.

- This year our faculty and students have won many prestigious awards and fellowships. I am proud to be Director of a department whose students and faculty continually strive for and succeed in obtaining excellence in research and education.

- This year, for Alumni Weekend, we are hosting a Brunch for our MS&E Alumni, family and friends. Brunch will be on June 10th from 11:00 a.m. - 12:00 p.m., in 260 Bard. Please RSVP for this event to Carol Armstrong at ca20@cornell.edu or (607) 255-9617. I hope that if you are in the Ithaca area, you will stop by and visit the department. We hope to see you for the Alumni Weekend this June.

Emmanuel P. Giannelis, Director, Department of Materials Science and Engineering

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grown to 30 people, securing over $14M in venture capital support. With a number of military and commercial developments underway, Protonex is poised to deliver field trial units in 2006.

As Chief Technology Officer, Osenar is responsible for technical leadership, product development, intellectual property, and corporate strategy. Early on, he had the opportunity to fill a variety of rolls, including HR, finance and business development.

Last September, Osenar took time out of his busy schedule to speak on the Energy and Environmental Technology Panel for MS&E’s Fortieth Anniversary Celebration. He is a devoted husband to his wife Amy and proud father of their 17 month old son Torin. Both the Department of Materials Science Engineering and Cornell University are fortunate to have Osenar as an alumnus and are grateful for the example he sets for our students.
**Graduate Student Poster Presentations**

The Graduate Student Poster Session was part of the Materials Science and Engineering 40th Anniversary Celebration on September 20, 2005. The Session took place in the Duffield Hall Atrium. Below left: Charlie Tracy discusses poster results with students Drew Forman and Anuja DeSilva. Below center George Malliaras, Dean Fuchs and Lara Estroff discuss the Poster Session. Below right: Bill Hudson talks with grad student Craig Weinman.

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**Staff Highlights**

Steve Kriske joined the staff of MS&E in Spring 2003 while finishing the MEng program. After he received his MEng degree, in May 2003, he began working full-time as Undergraduate Laboratory Coordinator for the department. Steve is responsible for assisting faculty in developing, setting up, and running teaching experiments and co-teaches the Junior Lab course. He also coordinates safety training for students and maintains safety standards in lab. In addition to maintaining the undergraduate lab, Steve began working with Professor Michael Thompson on developing an instructional clean room in Duffield Hall in Spring 2005. During the course of this project, he has assisted with the selection and purchasing of equipment on Applied Materials grant, overseen the development of facilities infrastructure and the establishment of access and safety protocol. This project is an important part of the infrastructure development to support the growing teaching needs of faculty and students.

In recognition of Steve’s work on this project, he received the Geyer Award in December 2005. The Geyer Award was established by the department of Materials Science and Engineering in remembrance of Robert Geyer, who was director of administrative operations for over a decade. The award honors contributions to the goals of the department.
Thin films on substrates are the basic building blocks of virtually every micro- and nanofabricated device; from integrated circuits, to microelectromechanical (MEMS) systems, to optics and many others. To improve the performance and reliability of nanofabricated devices, new materials are constantly being sought. In addition, materials in the form of thin films have different properties from those of bulk materials. In particular, thin metal films play important roles as electrical, optical, chemical, and structural elements in most micro- and nanofabricated devices.

The Baker group is working to understand the thermomechanical behavior of thin metal films in a variety of different projects. Some recent results are described below.

**Anomalous Microstructure in Phase Transformed Ta Films**

Tantalum is a refractory metal with a very high melting point. It is of interest because of both its electrical and its chemical properties. Ta thin films can be made in two crystal structures, the common cubic \( \alpha \) phase, and the metastable, tetragonal \( \beta \) phase. The \( \beta \) phase has high electrical resistivity but can be easily patterned using reactive ion etching, a typical nanofabrication method. The \( \beta \) phase is used for thin film resistors and x-ray lithography masks. The \( \alpha \) phase has a much lower resistivity and high chemical resistance and is very difficult to pattern. It is used as a protective diffusion barrier between the copper conductor lines and the silicon substrate in modern integrated circuits.

One can imagine that Ta might be used in a wide range of nanofabricated devices (e.g. microfluidic systems for sensors or biomedical applications) if, for example the \( \beta \) phase can be deposited so that it can be patterned, then converted to \( \alpha \) phase to serve as a layer with electrical or chemical function.

The Baker group has discovered a very interesting and unique new microstructure that arises in Ta thin films that have undergone the \( \beta \) to \( \alpha \) phase transformation. These films are crystalline, but show a very steady rotation of the crystal orientation with position in the plane of the film.

Figure 1 shows an electron beam backscattered diffraction (EBSD) map of the surface of such a film. The color shows the orientation perpendicular to the surface at each point. The color changes smoothly from point to point without crossing any boundaries. To a materials scientist, it seems like this must be impossible. Crystals must always have definite orientations with clear boundaries between them. For example, along the red line, the orientation changes at an almost constant 4°/µm. Baker and his students have found a way to explain this interesting phenomenon in terms of arrays of dislocations (crystal line defects) that are created during the phase transformation.

An analysis of the pixel to pixel rotations in EBSD images reveals that the rotations occur around \( <221> \) crystal axes and lie nearly in the plane of the film. This suggests that an array of edge dislocations lying nearly parallel to the film/substrate interfaces accounts for the rotating crystals. Group members are working to understand how these dislocations are formed and what useful properties this new metal microstructure might have. This work has been done by graduate students Robert Knepper and Ray Fertig, and undergraduates Katherine Jackson, Blake Stevens, and Max Aubain.

**X-Ray Studies of Stress Distributions in Thin Film Metallizations**

Thin metal films on substrates are subjected to very high stresses due to differential thermal expansion between the films and the substrates to which they are attached. This can lead to device failure if the stresses become high enough to crack the film or to peel it off the substrate. Most studies assume that the stress in thin films does not vary with position or direction. In reality, due to the different orientations of the crystallites, or grains, the stresses can vary dramatically from place to place and depending on in what direction one looks. Since failures occur based on the peak stresses rather than the average stresses, it is important to understand how stresses are distributed in such a film.

One way to measure this is to use x-rays to pick out the stress states just from grains having one particular orientation. Since the grains are very small, this requires an intense x-ray beam, such as that provided at the Cornell High Energy Synchrotron Source (CHESS). The Baker group shares the G2 hutch at CHESS with two other research groups, and recently completed construction of a new...
diffractometer system for measurements of the thermomechanical behavior of thin films, as well as many other experiments, in this hutch. The new diffractometer, shown in Figure 2, is based on the “kappa” geometry and has 6 independent rotation axes, which allows virtually any reflection geometry. The system also includes a heating stage that can operate in ultra high vacuum, and line and area detectors that can capture diffraction peaks over a range of angles simultaneously. This new instrumentation will allow us to study a wide range of thin films under controlled, technologically relevant conditions.

In preliminary studies, Baker and his students are looking at the stress distributions in copper metallizations as a function of interfacial chemistry and special aluminum bicrystal films that serve as a model system for studying strain transfer between differently stressed grains. This project has been carried out by graduate students David Nowak and Aaron Vodnick, with assistance from undergraduate student Jeremy Yim.

**Dislocation Interactions and Dislocation Structure in Thin Films**

Thin films are known to be extremely strong compared with the same materials in bulk form. This indicates that it is very difficult to move dislocations—line defects in a crystal that enable plastic deformation—through the film. A question of great recent interest has therefore been, “What stops dislocations in thin films?” To study this problem, the Baker group is conducting dislocation dynamics (DD) simulations at the Cornell Theory Center (CTC) using the computer program “PARANOID” (written by collaborator Klaus Schwarz at the IBM T.J. Watson Research Center). In these simulations, loads are applied to dislocations in a virtual thin film and they move and interact in a way that mimics the behavior of dislocations in a real film.

Figure 3 shows the dislocation structure in one such simulation. The inset illustrates some features of the dislocation structure. The different colors indicate different types of dislocations. After some deformation, dislocations have moved, interacted, joined with each other to form junctions, and annihilated with each other, creating a very complex structure. The portions of the dislocations that run from the top to the bottom of the film are called threading dislocations. As these threading dislocations move through the film, they deposit dislocations at the top and bottom of the film called misfit dislocations.

We have learned several important things from these simulations. For example, while the interactions between threading and misfit dislocations are common, they are weak. They cannot by themselves account for the strength of the film. Instead, Baker and his students found that interactions between threading dislocations are extremely important in determining film strength. This is surprising since threading dislocations are relatively rare in the film. However, the prevalence of these strong interactions is explained by the fact that threading dislocations cluster into small regions of the film. This clustering arises from the inhomogeneous stress state created by the dislocation structure itself. The misfit dislocations relax the film stress unevenly and the threading segments cluster because they are trapped in regions of low stress.

The simulations also revealed that, when the film is unloaded the remaining dislocations move back out of the film generating reverse deformation, another phenomenon that has been observed experimentally but not satisfactorily explained. The DD simulations provide a detailed look into the mechanisms that control stress levels and deformation in thin films and are expected to help people to find ways to improve the reliability of micro-and nanofabricated devices. This work was carried out by former graduate student Prita Pant (now a faculty member at the Indian Institute of Technology–Bombay) and current graduate student Ray Fertig.

Figure 2. The new diffractometer, based on “kappa” geometry, has six independent rotation axes allowing virtually any reflection geometry.

Figure 3. Dislocation structure in a simulation conducted by the Baker group at the Cornell Theory Center. Some features of the dislocation structure are shown in the inset. Different colors indicate different types of dislocations.
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sure and Temperature. In 2004 he was named a Distinguished Alumnus of the University of Utah; in 2005 a Distinguished Alumnus of the Chemistry Department of Purdue University. In addition to awards for excellence in research, Professor Ruoff has received many awards for excellence in education. Even while managing an internationally renown research group he remained dedicated to mentoring and educating his students. In 1956 he won the Westinghouse Award for Outstanding Teaching. He has also won the National Science Foundation Science Teacher Fellowship (1960-1962). During his fifty years of teaching he has mentored thirty-nine students who received their Ph.D.’s and four who received their MS degrees.

In addition, he has had nineteen post-doctoral research associates work with him at Cornell.

Professor Ruoff lead a distinguished career as both a researcher and educator: he has been a leader in his field and a mentor to students for fifty years. His service to Cornell includes being a member of the committees which wrote the research proposals for the Materials Science Center, The National Facility for Submicron Research and the Cornell High Energy Synchrotron Source. He was one of the founding faculty members of MS&E and served the Department as Director from 1978 to 1988. MS&E has been privileged to have had Arthur Ruoff as a faculty member for forty years. His legacy of excellence in research and teaching will continue to be an example to which faculty and students aspire for many years to come.

Professor Ruoff and his wife Enid have five sons:

William: graduated from Cornell in 1978 and has a Ph.D. in toxicology from Illinois. He is the Senior Project Risk Assessor/Toxicologist for URS Greiner-Woodward Clyde in Denver.

Stephen: graduated from the Cornell Materials Science and Engineering Department in 1979. He is CEO of IMR Test Labs in Ithaca, NY, Charleston, SC and Louisville, KY. He is on the Board of the Cornell Center for Materials Research.

Rodney: graduated from the University of Texas in 1981. He has a Ph.D. in chemical physics from Illinois. He was a Fulbright Scholar in Germany. He is the John Evans Professor of Nanoengineering at Northwestern University.

Jeffrey: graduated from Cornell in 1985, and received an M.F.A. at Temple and a Ph.D. at Iowa in film studies. He was a Fulbright Scholar in France and held the Henry Luce Fellowship at Vassar. He is a professor at Dartmouth.

Kenneth: graduated from Harvard in 1989. He has a Ph.D. in Japanese Studies from Columbia. He was a Fulbright Scholar in Japan. He is the Director of the Center for Japanese Studies at Portland State University and the winner of the 2004 Osaragi Prize for Commentary for his book, The People’s Emperor.
**Student Research Highlights**

**Ian D. Hosein**

**Self-assembly of inorganic and polymer base colloids with tailored morphology and composition for the fabrication of novel three-dimensional photonic crystal structures**

My current research focuses on the self-assembly of inorganic and polymer base colloids with tailored morphology and composition for the fabrication of novel three-dimensional photonic crystal structures. A range of new configurations and functionality are being explored for the development of photonic crystals with non-spherical and active colloids as bases. The practical realization of photonic band gap materials (photonic crystals) operating in the near infrared and visible regions relies on ordered lattices of these monodispersed, or uniform sized, nano- and mesoscale particles.

The motivation for this work is to overcome the limitations of traditional colloidal building blocks such as silica (SiO₂) and polystyrene spheres, which have poor optical function (low refractive index) and cannot produce the diverse packing arrangements necessary to fulfill the most promising enhancements in optical properties expected from photonic crystals.

Recently, it was shown that a number of micron scale non-spherical polymer colloids can be crystallized into two dimensional ordered arrays, such as half sphere, pear and asymmetric dimer shape colloids. Three dimensional ordered packing can be further achieved from these ordered monolayers. This is the first demonstration of ordered three dimensional colloidal crystals of non-spherical bases useful for optical applications at visible wavelengths, and current research focuses on understanding the assembly process and gaining further control of particle ordering and single particle orientation.

Additionally, for the first time Zinc Sulfide based colloids of both homogenous and hollow shell morphology have been self-assembled into large scale opaline structures. This exciting advancement makes the advantageous properties of ZnS based photonic crystals, including high refractive index, low absorption, and photoluminescence, available for theoretical study and optical device applications. Current research focuses on gaining tight control of the tunability of these crystals for applications at any desired wavelength in the visible or near infrared regime. The work on ZnS base photonic crystals is being presented at the Spring MRS conference in San Francisco.

**Marleen Kamperman**

**Assembly of block copolymers and high temperature ceramic precursors**

In my research I am developing an easily controlled bottom-up approach towards nanostructured high-temperature ceramics in which block copolymer mesophases are used as templates for inorganic precursors. With this method I am able to make nanostructured ceramic materials that are stable up to 1500 °C. Compared to oxides, they have improved thermal and mechanical properties, as well as enhanced chemical and etch resistivity. These materials have great technological promise in thin film applications of the microelectronics industry as well as in catalysis and separation.

In a “one-pot” approach an amphiphilic block copolymer is blended with a polysilazane, commercially known as Ceraset, which can be regarded as a filler with molecular dimensions. Careful utilization of the block chemistry leads to selective swelling of the hydrophilic block by the polysilazane. This results in a cooperative (co-) assembly of block copolymer and the polysilazane into nanostructured morphologies which can be permanently set by cross-linking the silazane oligomer with a radical initiator. Different mesophases with spheres, cylinders, or lamellae are observed by varying the polysilazane to block copolymer ratio.

The nanostructured hybrid materials can then be converted into high temperature SiCN-type ceramic materials upon calcination. We published this in JACS: Kamperman, Marleen; Garcia, Carlos B. W.; Du, Phong; Ow, Hooiswagen; Wiesner, Ulrich. *Ordered Mesoporous Ceramics Stable up to 1500 °C from Diblock Copolymer Mesophases*. Journal of the American Chemical Society (2004), 126(45), 14708-14709.

I am currently developing hierarchical ordered non-oxide ceramics; I want to show that high temperature silicon-carbon nitride ceramics can be structured on multiple length scales: ~1000 nm, ~100 nm and ~10 nm. Structuring is obtained by combining latex sphere templating, and cooperative assembly of a polysilazane and an amphiphilic block copolymer. Relatively large micron-sized latex spheres self-assemble into an ordered lattice and smaller latex nanospheres are forced to pack closely at the interstices between the micron-spheres.

This template is infiltrated with the ceramic precursor/block copolymer solution, which assembles into nanostructured morphologies. Subsequent removal of the organic material by calcination leaves a three-dimensionally interconnected, hierarchically ordered high temperature ceramic material. A direct application can be found in catalysis, because the catalytic process is thought to occur more efficiently in materials with hierarchical pore size distribution, if diffusion limitations are to exist in a catalyst with unisized nanopores.
MS&E undergraduates receive honors

Katy Bosworth (MSE) is a member of the Ober research group. She received an IBM Fellowship.

David John Herman (MSE) received the Tau Beta Pi Engineering Honor Society Scholarship for 2006-2007.

Kevin Huang (MSE) is an undergraduate who works in the Liddell research group. Kevin is an impressive student and researcher as evidenced by the caliber of the awards he has won this year:
- Nanotechnology Fellowship, Department of Materials Science and Engineering, Cornell University, Spring 2005.
- Barry M. Goldwater Scholarship, Cornell University, Spring 2005.
- A.CerS Hoffman Scholarship Award, Cornell University, Spring 2005.
- Alpha Sigma Mu Honor Society, Department of Materials Science and Engineering, Cornell University, Spring 2005–Present.
- ASM John M. Haniak Scholarship, Cornell University, Summer 2005.

Jonathan Rivnay (MSE) is an undergraduate working in the Malliaras group. His has received the following awards in recognition of his excellence in research:
- Alpha Sigma Mu Scholarship, Fall 2005.
- James L. Gregg Memorial Prize, Dept. of Materials Science and Engineering, Cornell University, Spring 2005.
- Alpha Sigma Mu Materials Science Honor Society, Department of Materials Science and Engineering, Cornell University, Spring 2005-Present.
- 2006 Merrill Presidential Scholar Award.

Jason Slinker (AEP/MSE) is a member of the Malliaras Research Group who received the SPIE (International Society for Optical Engineering) Educational Scholarship. In addition to this award he was a recent recipient of the prestigious Graduate Student Silver Award at the 2005 Fall Materials Research Society Conference in Boston, MA. Jason was chosen as the student finalist representing the meeting session for organic and nanostructured composite photovoltaics and solid-state lighting. This award was based in part on his presentation, “Electroluminescent Devices from Ionic Transition Metal Complexes for Lighting Applications.” Jason was among 22 student awardees representing all fields of materials research. The criteria for selection were:
- Graduate standing in a recognized academic program in materials science, metallurgy, ceramics, or polymers; physics or chemistry; geology or mineral science; electrical, civil, mechanical, mining, or nuclear engineering; or other materials-related field.
- Participation in the 2005 MRS Fall Meeting as an attendee and author or co-author of a symposium paper.
- Outstanding performance in the conduct of this project and promise for future substantial achievement in materials research as judged by the faculty advisor.
- Significant and timely research results.

Scott Warren (MSE/Chemistry) is a graduate student in the Wiesner and DiSalvo research groups, working as part of the Cornell Fuel Cell Institute. Scott has received numerous accolades over the last year for his mentoring work and his research. Scott currently mentors five Cornell undergraduates. Scott’s awards include:
- EPA’s Science to Achieve Results (STAR) Fellowship. Awarded to graduate students who perform environmentally-related research. Only three percent of applicants are awarded this prestigious fellowship.
- Four newspaper and magazine articles profiled Scott’s research and mentoring work (two in the Ithaca Times, one in the Cornell University Graduate News, and one in Great Ideas, a publication of Cornell University).
- Transmission electron microscope award, Cornell Center for Materials Research. This is awarded for producing the most intriguing and scientifically important image on a CCMR transmission electron microscope.