INquiry 2002

Science and Technology at the Ames Laboratory

New paradigm in sensor technology

Organic chemistry without solvents

Seeking a science trifecta

AMES LABORATORY

A U.S. Department of Energy laboratory operated by Iowa State University

Contents

(front cover) This brilliant blue organic light-emitting device was developed by Joseph Shinar and his Ames Laboratory research group. The versatile, low-cost OLEDs are used as light sources for the fluorescent sensors made by Shinar's group and their collaborators, Raoul Kopelman's group of the University of Michigan, Ann Arbor. Their combined research efforts have resulted in the creation of an integrated OLED/optical chemical sensor in which the detector and the light source that excites the fluorescence are integrated with the sensor films. (see story page 2)

(back cover) Small vials containing fatty acid conversion product are bagged for identification purposes in the reactor lab. The liquid in these vials will be analyzed for dibasic and monobasic acids. (see story page 8)

from the *Director*

I can't imagine that anyone in the U. S. faced with writing an annual summary letter this year would not begin with an observation that this has been an extremely difficult year for our nation. However phrased, it will be an understatement. The events of September 11th and our forthcoming heightened awareness of the international terrorist movement have changed the way we approach many aspects of our daily lives and how we think about life in general.

The plunge of the stock market and the corresponding diminution of our individual retirement holdings have caused us to cease dreaming of a lakeside retirement home, and to view "like a cross to a vampire" the letters arriving with quarterly reports of the status of these funds. Throughout the year, it has been a challenge not to be filled with anger at the corporate executives who, through deceit, filled their pockets at the expense of their own employees and stockholders, and also a challenge not to be filled with concern that Iowa's budget woes were addressed by disappointing reductions in funding for higher education.

Was there anything good about this year? Well yes, as a matter of fact, a lot of good things happened in the Ames Laboratory this year. Particularly timely was the opening of our Midwest Forensics Resource Center that is putting the world-renowned analytical capabilities of the Lab to work on problems in forensic science, especially those that face our rural crime investigation units.

Our scientists continued to receive prestigious awards both for fundamental and applied research. Indeed, watching our basic scientific discoveries be translated into practical uses is one of the true joys of my job. That's why I'm standing in a soybean field on a farm in central Iowa in the above photograph. Ames Laboratory's new Biorenewable Resources Consortium is heavily directed toward Iowa's abundant agricultural resources, such as soybeans. In this issue of *Inquiry*, you can read about how our BRC will turn ordinary crops into high-value products that could enhance economic development in rural states, like Iowa.

Our cover story features a photo of an organic light-emitting device developed by Ames Laboratory physicist Joseph Shinar. This unique light source serves as a compact, versatile and cost-effective alternative to expensive laser systems used to analyze living cells and organisms, and biological compounds.

In the area of "green chemistry," a team of Ames Laboratory scientists led by Vitalij Pecharsky has developed a revolutionary process to combine organic materials in solid state without the use of solvents. The discovery has the potential to dramatically improve our world by eliminating environmentally harmful solvents, such as benzene, used in chemical processes to produce millions of consumer and industrial products.

Physicist Bruce Thompson's expertise in the development of fundamental nondestructive evaluation techniques is being utilized by the Department of Energy in an important project assessing the structural integrity of aging underground storage tanks containing radioactive liquid wastes.

As always, solving our nation's energy problems remains key to Ames Laboratory's mission. The research efforts I've mentioned are just a few examples of how we've supported that mission in 2002. You will read about these and other Ames Laboratory accomplishments in the following pages of this year's *Inquiry*, and I'm confident you will agree that, yes, there were some very good things that happened in this past year. As always, we take pride in our accomplishments and look forward to the challenges ahead.

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Sensible Sensors

The beauty of a new chemical sensor lies in its simplicity

by Saren Johnston

We can envision the whole thing to be about the size of a silver dollar. Ultimately, it should have extremely wide-ranging applications," says Joseph Shinar.

The Ames Laboratory senior physicist is talking about the novel, fluorescence-based chemical sensor he developed in collaboration with chemist Raoul Kopelman from the University of Michigan, Ann Arbor. The new sensor is smaller, less expensive and more versatile than existing technology of its kind, making it a potentially perfect fit for monitoring oxygen, inorganic gases, volatile organic compounds, biochemical compounds and biological organisms. Within the field of molecular diagnostics for biomedical and biochemical research, the sensor could be used for point-of-care medical testing, high-throughput drug discovery, and detection of pathogens and other warfare agents.

Ideally integrated

The new sensor grew out of a basic research effort by Shinar and his Ames Lab research group to study the photophysics of luminescent organic thin films and organic light-emitting devices, or OLEDs, (those that luminesce, or emit light, when a voltage is applied). The University of Michigan researchers, led by Kopelman, were interested in developing fluorescent sensors, so the two groups joined forces to drive the invention of an integrated OLED/optical chemical sensor.

Shinar says the sensor had its beginning with a simple idea put forth by postdoctoral fellow Jon Aylott, who was working with Kopelman. "The invention started with just some scribbled notes about using organic light-emitting devices as light sources for fluorescent sensors," Shinar recalls. "When the idea was first brought up, my own response was, 'Well, there's nothing here. It's so obvious – of course you can excite a fluorescent sensor with an OLED.' You can, but it hadn't been done yet. And the beautiful thing is that you can integrate the two."

Shinar explains that the integration and miniaturization of fluorescence-based sensors is highly desirable because it is the first step toward the development of fluorescence-based sensor arrays that could be used for analysis of living cells and organisms, and biochemical compounds. In general, fluorescence-based chemical sensing devices include three components: a light source that excites the sensing element, the sensing element that produces the fluorescence (usually a fluorescent dye that is used to tag the sample under investigation), and a photodetector that responds to the fluorescence of the sensor. Conventional sensors use lasers or inorganic light-emitting devices as light sources, but they present problems. Not only are they expensive, they are also bulky and cannot be integrated with the other sensor components.

Senior physicist Joseph Shinar and graduate assistant Lijuan Zou watch as graduate assistant Kwang-Ohk Cheon adjusts an organic light-emitting device, or OLED (see cover photo), prior to testing its brilliance.

Stylishly simple

Shinar's and Kopelman's OLED/optical chemical sensor is unique in the simplicity of integration of the detector and the OLED light source that excites the fluorescence. "This is a real advantage," says Shinar. "With this kind of geometry, called 'back detection,' we should be able to use the sensor for in vivo biology applications." (In vivo refers to occurring within the living organism.)

Explaining the back-detection design, Shinar says, "Let's say you have some solution – blood, urine, whatever – on a glass substrate. The solution has lots of compounds in it that you want to detect. Your sensor is in contact with the biological solution on the substrate, and your OLED light source is behind the substrate. It's like a sandwich: sample solution, sensor, substrate, OLED." Powered by a miniature battery, the OLED light source excites the sensor, which fluoresces. When the sensor detects the compound of interest in the sample solution, its fluorescence changes, and the change is picked up by a photodetector positioned behind the OLED.

Shinar says the integration of the OLED light source, fluorescent sensor and detector makes the whole device a lot more compact and should also permit the development of an array of fluorescent sensors to be driven by an array of OLEDs. Describing an envisioned array, he says, "You could have a square with the corners formed from four OLED pixels that would be exciting the sensor in front of them. The light from the fluorescent sensor would come back through the area between the OLED pixels. Each pixel could be 10 microns, so this whole thing could easily be a square of 40 microns by 40 microns – that's your sensor."

Prototype and promising product

Early in 2001, Shinar and his collaborators successfully demonstrated an oxygen sensor prototype in which the OLED was integrated with the oxygen sensor film. "The results were excellent," he says. "The response of the sensor to oxygen was very fast."

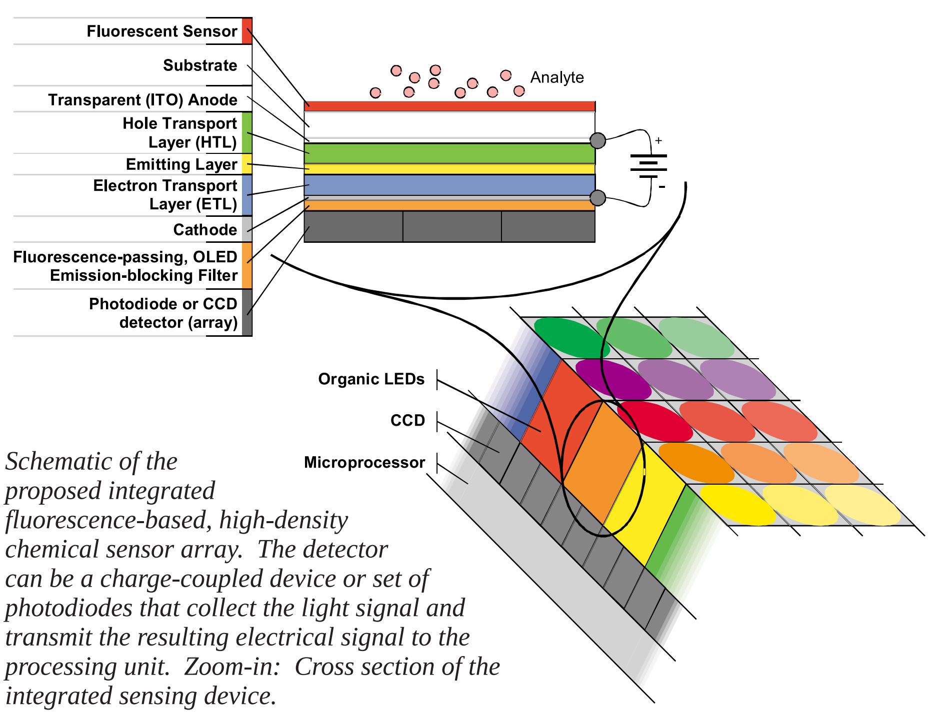
Shinar says they used front detection instead of back detection with the oxygen sensor prototype. Now their goal is to demonstrate the back-detection capability. In addition, he anticipates that the Ames Labratory and University of Michigan research teams will be able to develop a prototype for a glucose sensor in the near future. "The recipe is there, but we're wrestling with the stability of the glucose enzyme, which has a drastic effect on the uptake of oxygen by glucose," he says.

The integrated OLED/optical chemical sensor has tested so well that Shinar is investigating the possibility of starting a business to produce them. "We'd be making the whole sensor device," he says. "It would include packaging the OLED with the sensor, filters, photodetector, power supply and the readout mechanism, which would be digital, either on a liquid crystal display or a regular light-emitting diode – or, it could be an OLED readout."

Because most of the components for the sensor are so cheap, Shinar says almost the whole package could be disposable. The only component that wouldn't be disposable, at least in the foreseeable future, would be the photodetector. "If the sensor is produced in mass volume, I could easily envision its price to be less than $50 for the whole thing," he says. "That's just a guess based on having the photodetector be less expensive, because everything else should be really in the pennies."

The versatility, flexibility and cost-effectiveness of OLEDs offer excellent opportunities for developing OLED/optical chemical sensor arrays and high-density microarrays. Such systems would be able to discriminate between multiple compounds in complex biological samples, such as blood, urine, saliva or airborne particles, creating what Shinar describes as a very economical, compact and practical optoelectronic "nose" or "tongue," suitable for in vivo measurements.

"The big excitement is that a whole new paradigm in sensor technology could emerge from this work – from this basic idea of just integrating the very low-cost OLED light source with the fluorescent sensor," says Shinar. "The integration is really the big step forward." ♦



Schematic of the proposed integrated fluorescence-based, high-density chemical sensor array. The detector can be a charge-coupled device or set of photodiodes that collect the light signal and transmit the resulting electrical signal to the processing unit. Zoom-in: Cross section of the integrated sensing device.

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Shaken, Not Stirred

Creating organic compounds without solvents

by Kerry Gibson

Viktor Balema loads material into a ball-mill vial in a glove box to prevent possible contamination of the sample prior to processing.

As often happens in science, a group of Ames Laboratory researchers didn't set out to discover a revolutionary process to create organic compounds. Study in another area prompted a question and a "let's-give-it-a-try" response.

As a result, their findings that organic compounds can be formed in solid state marks a major advance in both materials science and chemistry where the two fields overlap. The discovery means that environmentally harmful solvents, such as benzene, dichloromethane and others, could be removed from many of the chemical processes used to produce thousands of consumer and industrial products. The initial cost-savings for the solvents alone would be substantial, but when combined with the reduction or elimination of disposal costs for the spent solvents and minimization of chemical waste streams, the overall benefits could be enormous.

Balls replace solvents

The discovery centers on ballmilling, a process routinely used to alloy various metals. Simply stated, the component metals to be alloyed are placed inside a hardened steel vial along with balls of varying sizes. The sealed vial is then shaken vigorously for extended periods of time. Mechanical energy going into the system is transferred to the materials, eventually combining them at the molecular level to form the new alloy.

"We started several years ago trying to find suitable materials for hydrogen storage," says Ames Laboratory senior scientist Vitalij Pecharsky. "As part of that project, we looked into modification of the structure of materials using mechanical alloying. Then we started to look into different classes of materials that were not related to that project, and it took off from there."

Organic materials have very different crystal structures, so they don't readily combine in solid state when mixed together; typically they must be dissolved to break down their crystallinity. Once the materials are in solution, they can be combined and the chemical reaction takes place. The resulting product is then separated out.

Given their expertise in alloying various materials, Pecharsky and a fellow researcher, Ames Lab associate scientist Viktor Balema, decided to explore if ball milling could take the place of solvents in carrying out organic chemical reactions in solid state. Shaking up the materials was easy. The problem was how to tell whether the reaction had indeed taken place during mechanical processing of the solids.

One major tool typically used to study a material's crystal structure is diffraction analysis. X-rays - short wavelength electromagnetic radiation - shone through a material will form a specific diffraction pattern dependent on the crystal structure of the material. This usually allows researchers to identify the solid material based on the spectrum displayed.

"But since our experimental approach is mechanochemical, it reduces the particles to very small size and induces plenty of strain and stress," Pecharsky says. "Essentially, what we end up with is either poorly crystalline materials or materials that aren't crystalline at all, and diffraction techniques are nearly useless."

NMR tracks reactions

For answers, Pecharsky and Balema turned to Ames Lab colleagues, physicist Marek Pruski and chemist Jerzy Wiench, leading experts in solid state nuclear magnetic resonance spectroscopy, known as NMR. In NMR, a strong magnetic

A close-up shows a hardened steel vial and the balls used to process the materials. The vial fits in the cage of the mill, which operates similar to a paint shaker at a paint store.

Vitalij Pecharsky holds a ball-milling vial used to process materials. The materials and steel balls are placed in the mill and vigorously shaken for extended periods of time.

field is used to split the energy levels of the atomic nuclei in the material being studied.

"The transition frequency that corresponds to the splitting identifies the nucleus and the local environment in which the given nucleus finds itself," Pruski says. "In other words, by monitoring the transition frequency, we can fingerprint the specific material."

NMR proved to be perfect for verifying the experimental results. Using materials that produce well-known and well-documented reactions when carried out in solution, Balema and Pecharsky combined these prototype materials in the ball mill and took the resulting products to Pruski and Wiench. With a limited number of possible end products, they were able to quickly match the NMR of the experimental products with commercially prepared samples.

"This was almost born for NMR," Pruski says. "In principle, they have a pretty good idea of what to expect, and we can tell them right away if the expectations are met or not. It doesn't take a lot of handwaving to explain the results. If they had no idea of what the reaction would produce, then we'd have to spend more time pinpointing what it was." (To find out more about solid state NMR spectroscopy, turn to "Atomic Fingerprints," on page 17.)

"The spectra that we collected indicates that the processes do occur in solid state," Wiench says, "ruling out the possibility that heat generated during processing melts the materials, permitting the reaction to take place in a liquid state." Another advantage is that NMR testing is non-destructive and doesn't require any prior or further processing of the material. According to Wiench, this helps eliminate any chance that other factors might influence the reaction.

High yields, one pot

Not everything the team ran through the ball mill was a success, but nearly all of the discovered transformations previously performed exclusively in solution were found to be exceptionally efficient and selective in the solid state. "I would never have believed that solvents could be excluded from all these reactions if I hadn't done it myself," says Balema, adding that milling times varied from one hour to 25 hours, depending upon the reaction being examined.

Another valuable discovery is that the reactions can be carried out consecutively, or as "one-pot" processes, when components required for performing a reaction in stages are ball-milled together in the same vial. "Remarkably, a 'one-pot,' Wittig-type reaction between phosphines, organic halogenides, aldehydes or ketones, and a base is impossible in a solution, but it has been successfully carried out in a mill without a solvent," Pecharsky says.

To date, the group has operated on a shoestring budget, borrowing research time to carrying out the testing. Materials costs have been minimal, according to Pecharsky, and the testing has used existing equipment.

"If we were to go into some more adventuresome experiments, for example to elucidate some of the reaction mechanisms, that takes considerably more effort," Pruski says. "We would need to invest more time in experiments and money in special accessories. We've been able to work around those problems up to now."

Though they only published their findings in the June issue of the *Journal of the American Chemical Society*, Pecharsky indicates that their work has already been cited by another group looking into mechanochemistry.

"It's different work and a different reaction," he says, "but it's all mechanochemistry." They were also successful in carrying out organic reactions in solid state with quite high yields. "Now that we've shown that it can be done without solution, maybe mainstream organic chemists will try it - and most likely succeed, too, which is good. That's how science is done these days. You can't do it alone," says Pecharsky.

Echoing that sentiment, Pruski says the Ames Laboratory effort is truly a multidisciplinary collaboration. "Even though we're in different programs, it's been a good symbiotic collaboration on science at a very basic level," he says. "It's the way a national lab ought to function." ♦

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A 5-millimeter rotor containing the sample of interest is inserted into the magic-angle spinner in one of the solid state NMR spectroscopy probes.

Cleaning Up an Environmental Legacy

by Mary Jo Glanville

Ames Laboratory is playing a key role in a U.S. Department of Energy Office of Science and Technology project to address the environmental legacy left by the production of nuclear weapons during the Cold War.

This legacy includes millions of gallons of radioactive liquid waste stored in 259 huge underground tanks at five DOE facilities. The tank farms are located throughout the United States, with 177 tanks at the Hanford site in Washington, 51 at the Savannah River site in South Carolina, 40 at the Oak Ridge Reservation in Tennessee, 11 at the Idaho National Engineering and Environmental Laboratory in Idaho, and two at the West Valley Demonstration Project in New York.

Many of the tanks, which were built between the 1940s and 1980s, have reached or exceeded their original design life. A number of the tanks are already known to have leaked and as they have aged, concern has increased about additional leakage or collapse. While work is underway to convert the waste into a form better suited for permanent storage, conversion and cleanup is a long-term process.

Assessing Tank Integrity

The five sites, each with unique tank-management issues due to variations in climate, geography and tank construction, are at various stages in the conversion process. As a result, many of these tanks must continue to store hazardous waste. Assessing their structural integrity to determine if corrosion is occurring or cracks are developing is essential to protect people and the environment.

The DOE formed a structural integrity panel in the early 1990s to begin the process of assessing the tanks. The panel gathered extensive information at each of the sites and held 18 workshops over a six-year period to examine the issues and concerns. Bruce Thompson, director of Ames Labratory's Nondestructive Evaluation Program, served as the panel's ultrasonics expert. In 1997, the panel issued its technical report, "Guidelines for Development of Structural Integrity Program for DOE High-Level Waste Storage Tanks."

Ames Lab and the Iowa State University Center for Nondestructive Evaluation have had a continuing role providing assistance to the DOE in developing appropriate NDE for these high-level storage tanks. The linkage between Ames Lab and the CNDE is very beneficial, according to Thompson, who is also CNDE director. "It means DOE has access to the capability and expertise within CNDE and ISU. Because the center actively works with 20 to 25 sponsoring companies, DOE has a window to NDE technology that is being used by a wide range of industries, such as the aerospace and power-plant industries. If you're dealing with some tough problems, it's good to find out how different industries are approaching similar issues," he explains.

The role of Ames Lab moved to a new level when the DOE's Tank Focus Area, or TFA, identified the development and coordination of new and improved technologies for tank integrity assessment as a high priority in September 2000. Ames Lab is coordinating and implementing the project jointly sponsored by the TFA and the Characterization, Monitoring and Sensor Technologies, or CMST, Crosscutting Program of the DOE Office of Environmental Management.

Sharing Information

The project started with visits to each site. Mike Terry, TFA; Glenn Bastiaans, CMST; and Brian Larson and Dave Rehbein, Ames Lab, collected information from technical personnel about tank integrity needs and issues. The first tank integrity workshop was held in fall 2000. The specific goals were to identify significant impediments that might exist to the safe operation and management of the storage tanks, particularly those common to multiple sites; and to establish groundwork for collaborative efforts aimed at eliminating those impediments.

"We talked about what's going right, what's not going well, and what needs improvement. The DOE Order 435.1, "Radioactive Waste Management,"

At the Hanford Site in Washington, 177 radioactive waste storage tanks were built between 1943 and 1985. This is a double-shell tank which contains two carbon-steel liners along the walls and floor and a single steel dome liner. It is enclosed within an outer shell of reinforced concrete. (Photo courtesy of DOE's Pacific Northwest National Laboratory) rose to the top in terms of discussion. It actually mandates the inspection of the tanks, but we discovered there wasn't a perfectly common interpretation of how to accomplish that. So there were benefits of people talking about what this meant," Thompson says.

A second workshop, held fall 2001, took a more technical focus. Participants identified some areas where they needed to compare notes. Thompson explains, "One of these areas was the phenomenon called vapor space corrosion. You might think corrosion would occur below the liquid, but there is some evidence that there has been active corrosion above the liquid and at the liquid/vapor interface."

As a result of this discussion, the Savannah River group organized a workshop focusing specifically on this topic. Kurt Hebert, professor in ISU's chemical engineering department, was one of three expert panelists to hear presentations about experiences at the sites and plans for addressing the concerns. The panel responded to the plans and made recommendations.

In the area of technical assistance, three CNDE projects are progressing well, according to Thompson. One area of need relates to the length of time it takes to ultrasonically inspect the tank walls for corrosion. The size of the tanks, 70 feet in diameter by 70 feet high, translates to a lot of surface area. The current ultrasonic techniques use a tightly focused beam that has to be scanned over the entire surface. This is an accurate but inherently slow process.

New Techniques, Tools

Rehbein, an Ames Lab and CNDE metallurgist, working in collaboration with Sonic Sensors of EMAT Ultrasonics, Inc., has developed a technique to use as a screening tool. The electromagnetic acoustic transducer, or EMAT, inspection system makes use of guided ultrasonic waves propagating along the tank walls to rapidly scan the tank walls and detect thinning or isolated defects that would then be the focus of a detailed inspection. Not only is the EMAT inspection system faster than the conventional system, it doesn't require extensive and expensive surface preparation.

The technique was successfully demonstrated on a mock-up at the Hanford site in September 2001. Now it's a matter of locating the resources for further testing on a full-scale mock-up and on a tank. "We have put a new capability on the table, and now it's ready to evolve to the next level," Thompson says.

Bruce Thompson displays just a few of the many boxes of documentation gathered in the 1990s by a panel reviewing the structural integrities of nuclear waste underground storage tanks located at five DOE facilities.

A second project, being done in collaboration with Pacific Northwest National Laboratory, is designed to obtain data from the tanks' knuckle regions. In double-shell tanks, this is where the sides and bottoms of the steel inner wall are joined. This area is subject to relatively high stresses, but detecting flaws is complicated because it is very difficult to access.

PNNL is developing a system to excite ultrasonic waves in the primary liner using transducers placed in the annular region where there is physical access - between the primary and the secondary liner. The waves propagate around the curvature of the knuckle and find defects near the bottom. The signals are interpreted based on a technique known as Tandem Synthetic Aperture Focusing.

This technique is well established for measurements on a flat plane, so the question is, how does it have to be modified when the waves must go around bends? Ron Roberts, an Ames Lab and CNDE physicist, is developing simulation tools to analyze the wave propagation phenomena as it applies to curved geometry. These tools are designed to provide a means of enhancing the current approach to give detailed information on the size and extent of defects.

The third activity focuses on the concrete walls and domes that cover some of the tanks. These structures have to withstand the load of heavy equipment used to retrieve the wastes. A technique called direct sequence processing allows a signal to be injected from outside the tank, propagate along one of the risers that provide access to the inside of the tank, couple to the concrete and propagate through it to another riser where it can be detected. Measurement of the arrival time of this signal would provide information about the modulus of the concrete, which can be used to determine if there is degradation. A workshop to develop recommendations is being planned.

The challenge of cleaning up the environmental legacy left by the Cold War is extremely complex. "We have been challenged to find the fastest and most cost-effective ways to insure the safety of people and the environment," says Thompson. "To accomplish this we must continue to develop plans and tools to assess the structural integrity of the tanks and thereby reduce the risk of collapse and leakage for as long as the tanks are needed." ♦

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A Sure Bet

Three New Initiatives Move Into the Winner's Circle at Ames Laboratory

by Steve Karsjen

(left to right) Gary Osweiler explains the ISU Veterinary Diagnostic Laboratory's new quick-response bioterrorism database to U.S. Rep. Tom Latham and ISU President Gregory Geoffroy. Congressman Latham had just announced funding for the MFRC and BRC initiatives.

In horse racing, a trifecta is achieved by picking three winners in a row. Ames Laboratory hopes to achieve its own trifecta of sorts in three new initiatives – one in forensics, one in biorenewables and a third in catalysis. And all bets are on a successful outcome.

Midwest Forensics Resource Center

Moving with breakneck speed would be the best way to characterize the pace of activities associated with Ames Laboratory's Midwest Forensics Resource Center, or MFRC. First, there were goals to write and rewrite in order to establish the MFRC at the Lab and Iowa State University. Then there was the rural law enforcement workshop to plan and hold, which was followed by the annual crime laboratory directors' meeting, which included an add-on meeting to discuss preparedness for agro-bio-emergencies. At the same time these projects were being undertaken, David Baldwin, director of the MFRC and Ames Lab program director of Environmental and Protection Sciences, was assembling the draft of the research "call for proposals" for projects to be funded through the MFRC. Successful proposals will receive approximately $55,000 in one-year funding. Final approval of the draft comes next, followed by funding of selected research proposals. All in all, it's been an exhaustive first phase for this Ames Laboratory initiative, but according to Baldwin, the pace was necessary for the MFRC to position itself to meet the growing demand for forensic analyses in areas ranging from counter-terrorism to crime-scene and DNA analysis. "We've tried very hard to be on the edge and ready to go," says Baldwin. "We simply don't want to lose our audience at this point."

This audience of MFRC collaborators includes several high-level government agencies, including the Bureau of Alcohol, Tobacco and Firearms; the FBI; the Department of Justice's National Institute of Justice; the National Terrorism Preparedness Institute; and the Department of Energy. In addition, the MFRC is partnering with crime lab officials from nine Midwestern states, including North Dakota, South Dakota, Nebraska, Kansas, Missouri, Wisconsin, Minnesota, Iowa, and more recently Illinois. Other partners include Iowa State University and ISU's Institute for Physical Research and Technology.

The MFRC got a major boost in January 2002 when U.S. Rep. Tom Latham announced financial support for the center. The $3 million in federal funding was the successful ending to a long story for Ames Lab Director Tom Barton, who years earlier had begun discussing establishing the MFRC with Randy Murch, then deputy director of the FBI Laboratory. Those conversations were held in the cafeteria of the J. Edgar Hoover building in Washington, D.C. "We talked about the problems facing the nation's crime labs – needs and obstacles," says Barton, "and how Ames Lab and Iowa State University could partner to solve them."

Those early discussions eventually led to a variety of research projects being funded by the FBI. Projects ranged from a type of quick-response bioterrorism database (a joint venture between the Ames Lab and ISU's Veterinary Diagnostic Laboratory) that includes information for labs on veterinary response to livestock pathogens to a fingerprint chamber, or glove box, that detects latent fingerprints on things like cans, guns, even plastic bags. The very first glove box is housed in the Iowa Division of Criminal Investigation's Criminalistics Laboratory in Des Moines, Iowa. A second glove box is being used by experts in the crime lab of the Story County Sheriff's Department in Nevada, Iowa. Rural law-enforcement agencies with limited budgets, such as the one in Story County, are considered prime benefactors of the research expertise developed by MFRC scientists says a grateful Story County Sheriff Paul Fitzgerald. "The glove box is certainly something that's going to advance our ability to do research and to train and educate rural law-enforcement officers in forensics," Fitzgerald adds.

Other MFRC research efforts have included projects to restore obliterated serial numbers from steels, aluminum and other metals, and to determine bomb-blast effects upon metals. "As far as new research efforts, we'd expect to fund proposals that support ongoing or existing types of analyses, such as the development of new instrumentation or new methodologies to support crime labs," says Baldwin. He'd also like to see research proposals that support the use of statistical analysis of classes of evidence. Baldwin says investigators can already determine the probability that a human sample matches the DNA of a suspect and the degree of confidence or the significance of that finding. Now he says similar statistical methods need to be developed for analyses to do the same type of matching with bullets, glass and steel. "This is a hot issue right now because of the way DNA has revolutionized the way courts use forensic evidence," says Baldwin.

Research and development assistance to improve forensic capabilities is just one part of the MFRC's four-part mission. Other focuses include casework assistance for crime labs, educating the next generation of forensic scientists and providing forensic training assistance for crime-lab scientists and law-enforcement professionals. In the area of casework assistance, the MFRC is building upon the bioterrorism database idea by developing a database of forensic resources expertise for criminalists. This expertise, for example, would allow a criminalist in the Midwest to find and then consult with technical experts at the Laboratory on a range of topics, such as materials identification.

In addition to casework, the MFRC's training program is also receiving high-level interest. The FBI's Forensic Science Training Unit is considering making the MFRC part of its "virtual" training academy system. In the past, the FBI has provided training solely at its academy in Quantico, Va., but the agency has a limited number of trainers and space so it wants to access the resources of strategic partners nationwide who are qualified to teach training courses in forensics. "This approach fits exactly into the type of training we are equipped to deliver," says Baldwin, who adds the MFRC is hoping to use the capabilities of ISU's Instructional Technology Center to facilitate its training mission.

Ames Laboratory's Todd Zdorkowski (left) and David Baldwin confer with Jane Homeyer, director of the Forensic Science Training Unit at the FBI Academy, at the 2002 MFRC annual meeting. Homeyer urged the MFRC to consider becoming one of the FBI's training partners.

Now that the groundwork has been laid, the next several months will bring a second flurry of activity as efforts are made to enhance all four of the MFRC's mission areas. Those efforts will range from funding more research proposals to fostering more university/crime lab interactions to expanding training programs for forensic scientists to increasing casework assistance. As evidence of this commitment, the MFRC has already begun offering up to 40 hours of casework assistance for criminologists wanting to consult with any of Ames Lab's technical experts. A confident Baldwin believes he'll be able to find the support to help foster all of the MFRC's goals. "Sometimes you work on research in a bureaucracy, and everyone is arguing about what's important," Baldwin says. But that's not the case with the MFRC, he adds. "Everyone is seeing universal importance in everything we're talking about doing."

Biorenewable Resources Consortium

It's no secret that the world, and industry in particular, has a huge appetite for oil and natural gas as energy sources. It's also no secret that at the current rate of consumption these traditional energy supplies will eventually be depleted. The goal of the Biorenewable Resources Consortium, or BRC, is to lessen our nation's dependence on fossil resources and petrochemicals as energy sources by developing agricultural alternatives, such as plant matter, or biomass, to create products and energy. Accomplishing this goal would help improve U.S. competitiveness internationally, provide greater crop diversity for farmers, reduce carbon dioxide emissions from the use of conventional fossil fuels, help form new industries, and enhance economic development in rural states like Iowa.

Ames Laboratory is uniquely positioned to play a key role in the development and use of biorenewable resources as a sustained supply of energy due to its location in the heart of the agricultural belt, which provides access to an unlimited supply of biorenewable resources as well as the ability to access the world's most advanced crop research through ISU. By combining its research strengths with those of its partners, ISU's Plant Sciences Initiative and the USDA's Iowa Agriculture and Home Economics Experiment Station, the Lab hopes to take a leadership role in making biorenewable energy a cost-effective, environmentally safe source for energy and chemical products.

Congressman Tom Latham announced $2 million in federal funding for the BRC in January 2002. Since the announcement, George Kraus, chair of the BRC task force and an ISU chemistry professor, says the BRC has moved quickly to issue a request for proposals for research projects from Ames Laboratory and ISU scientists. Nineteen proposals have been received. Eleven of those were chosen to receive on average $80,000 each. "The proposals are broad-based, ranging from research on issues of fermentation to composites to catalytic reactions," says Kraus. "We believe that a lot of interesting science will emerge."

A key requirement of the BRC research proposals is that they are collaborative in nature, meaning they include interactions between scientists from various disciplines. The goal of this cross-boundary interaction is to develop what Kraus calls "interfacial science."

"We want to bring people together who really haven't interacted with one another much," says Kraus. "Whenever you bring people together from different areas you almost always get different perspectives and, hence, new ideas and strategies for solving problems." Kraus adds the perfect research scenario might include engineers interacting with chemists, who are interacting with food scientists, who are interacting with people from forestry. "There are some pretty ingenious proposals being funded," says Kraus. "I just can't wait to see how some of them actually pan out when we try the science."

Brent Shanks, an ISU associate professor of Chemical Engineering, is one of the scientists whose research proposal has received a grant from the BRC. Shanks proposes taking a biorenewable resource, such as soybean oil, and producing dibasic acids by cleaving, or splitting, the fatty acids contained within. These dibasic acids could be used to replace adipic acid, which is a basic ingredient used in the production of nylon. Nylon is used extensively in carpeting and other products. Industry produces over a billion pounds of adipic acid each year, all of which is produced from fossil fuels. Shanks wants to determine if the adipic acid replacement can be produced from biological sources rather than fossil fuels. "There's a great opportunity before us to get rid of this fossil-fuel-derived material in favor of one that's soybean-oil-derived," says Shanks, who adds that dibasic acids produced from biorenewable materials also have the potential to be cheaper to manufacture than adipic acid made from fossil fuels. It potentially could cost on the order of 40 cents per pound to produce dibasic acids from soybean oil, whereas adipic acid costs around 60 cents per pound to manufacture from fossil fuels. "So not only is there the potential to get rid of a fossil-fuel-derived material, but also an opportunity to provide better economics for industry with soybean-oil-derived material," Shanks says.

Brent Shanks draws a sample of the conversion product from a batch reactor. This sample will be analyzed for yield of the desired dibasic acid product.

An enthusiastic Shanks says he's anxious to get started on his research project and readily admits that without the BRC funding his project might have been a long time in coming. "This BRC seed money will allow us the chance to actually validate that we can do this conversion," says Shanks, who adds that this is very important in a scientific world where funding is often based on results rather than promises of what could occur. At the same time, Shanks is also looking forward to the interactions he'll have with scientists elsewhere at Ames Laboratory and ISU who have backgrounds dissimilar to his. In his research project, Shanks will combine his expertise in chemical engineering with those of scientists whose backgrounds are in catalytic conversion of biological feedstocks, advanced catalytic materials, and oxidation chemistry. "The interplay of this experience will be needed to successfully complete the proposed work," says Shanks.

As for the future of the BRC and of biorenewables research at Ames Lab and ISU, Kraus says it's all in the numbers – numbers of scientists that is. There simply needs to be more researchers working in the biorenewables area, says Kraus. "I think we're primed to do great things, but we don't have all the researchers we need to cover all our bases in biorenewables," he says. "Ames Lab and ISU have a ways to go to catch up to other universities that have a much stronger tradition in biorenewables." But he adds, "We're off to a pretty fast start."

Green Chemistry Catalysis Laboratory

At the same time the MFRC and BRC were receiving funding so was the initiative to establish the Green Chemistry Catalysis Laboratory, or GCCL, at Ames Laboratory and the Center for Catalysis at ISU. Senator Tom Harkin announced $500,000 in funding for catalysis research at ISU.

In order to understand the significance of catalysis research to Iowa, one must first understand catalysis itself and its connection to "green" chemistry. *Webster's New World College Dictionary* defines catalysis as "the speeding up or, sometimes, slowing down of the rate of a chemical reaction caused by the addition of some substance that does not undergo a permanent chemical change." This phenomenon is used widely in the pharmaceutical, chemical and energy industries to manufacture all sorts of products, from pharmaceuticals to cosmetics to cleaner fuels.

Unfortunately, many of the catalytic reactions used in industry to create these products generate large volumes of hazardous pollutants, which is where green chemistry comes into play. Green chemistry refers to chemical processes that do not harm the environment. These processes often occur at room temperatures and ordinary pressures, and use water, preferably, as a solvent. They require the expenditure of less energy, occur much more rapidly and produce significantly fewer unwanted byproducts than competing industrial processes.

According to Kraus, who also directs the GCCL for Ames Lab, Iowa is in a strong position to capitalize on the growing relationship between environmentally friendly catalysts and biorenewable resources. For example, Kraus says there has been a lot of talk about taking ordinary field crops, such as corn and soybeans, and converting them into value-added products. At almost every step of this process, Kraus explains, a catalytic conversion would be required.

A goal of the catalysis laboratory would be to discover new catalysts and chemical reactions that operate in an environmentally friendly manner, which would help the nation replace fossil-fuel-based feedstocks with those based on renewable resources, thus ensuring an adequate energy supply for generations to come. One example of this relationship would be the creation of new environmentally friendly catalysts to convert soybean oil into biodiesel fuel. Another would be biocatalysts to synthesize fine chemicals for pharmaceuticals. Still another would be new catalysts for compounds used in flavorings and fragrances. "We hope some of the catalysts developed in our green chemistry lab and catalysis center will accelerate the process of developing these bio-efforts across Iowa," says Kraus. "Our ultimate goal is to unearth some new science and develop some catalysts that could help create new markets for Iowa products, thereby enhancing Iowa's rural development," he adds.

"Our work could potentially lead to the creation of new industry in Iowa," says Andreja Bakac, Ames Laboratory senior chemist, whose project has received funding through the GCCL. Bakac will use the $50,000 grant to further her work to develop a heterogeneous catalytic system for the oxidation of hydrocarbons. Benzene is an example of a hydrocarbon that she can currently oxidize in a homogeneous solution. The reaction uses visible light as an energy source and oxygen as oxidant, but the homogeneous setup does not allow for easy removal or regeneration of the catalyst after the completion of the reaction. Rather, the catalyst is wasted and must be disposed of, which increases the cost of the process. This problem will be solved if the transfer to a heterogeneous system is successful. "If we can develop this new system and get industry interested in it, perhaps that industry would locate in Iowa," says Bakac, who adds that the timing for the GCCL funding is just perfect. "Without this funding this project would be sitting on a back burner waiting for other money to come in, if and when."

Like the BRC, the GCCL initiative hopes to bring together "teams" of scientists to work on solving mutual problems. David Hoffman, division director of Science and Technology at Ames Laboratory and an ISU professor of chemistry,

Andreja Bakac observes photolysis (chemical decomposition induced by light) in a reaction solution as part of a catalysis experiment.

believes the potential exists for establishing numerous new cutting-edge research teams in catalysis at Ames Lab and ISU. For example, Hoffman envisions research teams consisting of chemists, biochemists, materials scientists and engineers as well as mathematicians. He adds that many of these researchers will have already made individual strides in catalysis research. Creating, fostering and nurturing a synergy between these individuals is a new challenge he expects will quickly take root.

"We have already formed our team and we're working together," says Bakac, whose research project is a perfect example of the synergy of which Hoffman speaks. Bakac is combining her expertise with that of three other Ames Lab and ISU scientists – Marek Pruski, Victor Lin and Brent Shanks – to complete her research project. "We need each other to make this work," says Bakac, who, like Hoffman, believes the team approach is not only good for the research but also for the researchers. Hoffman adds, "We're off to a very smooth start." ♦

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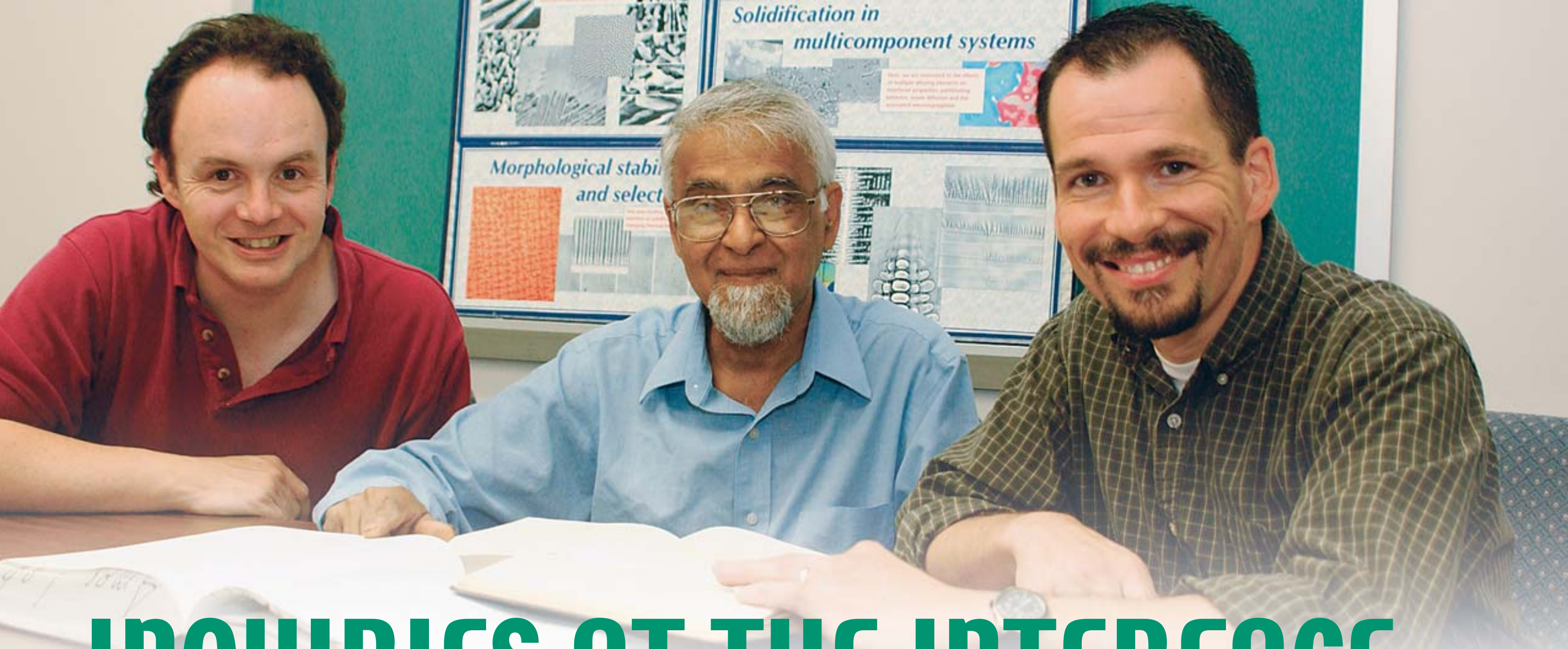


INQUIRIES AT THE INTERFACE

Scientists find small effects play huge role in how materials evolve

by Saren Johnston

You might call them "control freaks" of sorts. Ultimately, they'd like nothing better than the ability to guide what happens every step of the way, and they're making remarkable progress in that direction.

Don't misunderstand – Ames Laboratory researchers Rohit Trivedi, Ralph Napolitano and James Morris are great guys, but relentless in their desire to better understand how microstructures develop in materials. Working toward that objective, they're studying certain properties that exist in metals at the interface between the liquid and solid phases during solidification.

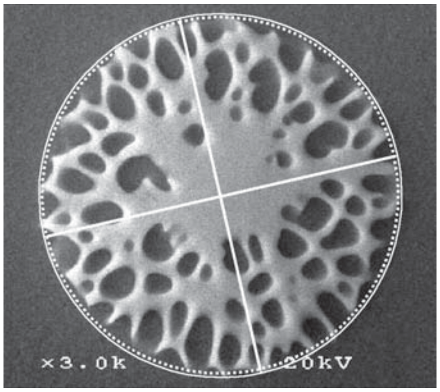
"We want to have control of the microstructure," says Napolitano, physical metallurgist and an Iowa State University assistant professor. To gain that control, he says, "We need to understand the fundamental principles that underlie microstructural evolution, beyond simply tweaking the process knobs." The three scientists are focusing their basic research efforts on that goal – one that may some day allow them and others to tailor microstructural development, providing the basis for new and improved materials.

What lies between

The Ames Laboratory team has shown that there are many subtle variations in microscopic properties at the liquid-solid interface as the solid is "freezing out." The small variations depend upon which crystal face is in contact with the liquid. Different faces, or orientations, give slightly different values for properties such as free energy, mobility, and stiffness (surface tension); and these properties play dramatic roles in how the microstructure of a metal evolves during solidification.

"The structure observed at the interface between a solid and a liquid is strongly dependent upon the crystallographic plane with which the liquid is in contact," says Napolitano. "In metals, this dependence is particularly interesting because the interface is typically 'atomistically rough' and is continuously fluctuating, so that the crystallography can only be represented statistically. The nature of these variations at the atomistic level ultimately governs the overall behavior of the interface," he explains. Accordingly, Napolitano notes that a complementary relationship between his and Trivedi's experimental approach and the theoretical predictions of Morris, a theoretical physicist, has developed quite naturally. "At the end of the day, the fundamental knowledge we're gaining about crystallographic dependence of interfacial properties is critical to the development of various theories concerning solidification, crystal growth, and the natural evolution of microstructure in materials," he says.

Trivedi, physical metallurgist and an ISU distinguished professor, stresses the importance of the interfacial properties he and his co-workers are investigating. "There are some properties that are extremely small, but they have a profound influence on interfacial behavior," he says. "For example, the way a snowflake forms depends on very small factors. It turns out that some of these small factors are really the essential ones in determining shape.



The two-phase structures that form within the droplets during rapid quenching from the liquid state reveal the overall pre-quench equilibrium shapes of the droplets. Rohit Trivedi and Ralph Napolitano use such cross-sections to quantify the deviation from sphericality in the quenched droplets. The deviation is extremely small, but enough to effect big changes in microstructural development.

(left to right) Researchers James Morris, Rohit Trivedi and Ralph Napolitano are studying the fundamental interfacial behavior between liquid and solid phases during the solidification of metals, with the ultimate goal being the ability to control microstructural development.

The same thing is true not only for materials, but for humans, animals, plants – anything that grows."

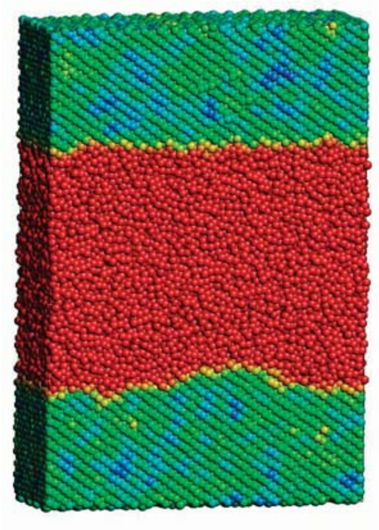
A couple of "firsts"

Trivedi, Napolitano and Morris are paying close attention to the small factors and their effects on microstructural evolution. "We're investigating some very specific quantities, such as the variation of interfacial free energy with crystallographic orientation," says Napolitano. He and Trivedi have developed innovative experimental techniques that provide the first reliable measurements of the minuscule variations in free energy at the liquid-solid interface in metallic systems. Morris' complementary calculations represent the first (and so far, only) effort to theoretically predict the variation in interfacial free energy for aluminum. The combined efforts of the three scientists provide both a direct check between the experiment and the simulation and the opportunity to put forth new solidification theories. Napolitano continues, "By revealing the essential physical behavior of liquid-solid interfaces, these critical experiments, both experimental and theoretical, are facilitating significant advancement in the theoretical prediction of microstructures."

The metallurgists' method

In experiments designed to measure small effects on the property of interfacial free energy, Trivedi and Napolitano have devised a method to selectively melt certain microscopic regions within an aluminum alloy single crystal, forming a dispersion of tiny liquid droplets trapped within the solid. (A single crystal is one in which the atoms are arranged according to a single "plan" or "template.' The orientation is uniform throughout the material, creating a simple, symmetric structure.) The material is then heated to bring the droplet structures to equilibrium (the condition at which no change occurs in the state of a system unless its surroundings are altered). After rapid quenching, the droplet shapes are measured very carefully, and their equilibrium shapes are determined, providing the necessary link to interfacial properties. "Thermodynamics tells us how the equilibrium droplet shape is related to the interfacial free energy," says Napolitano. "These measurements provide a direct means for quantifying the subtle variation of this property with respect to crystallographic orientation. The challenge is to accurately measure the degree to which the droplet shapes deviate from being spherical, and they deviate only by a percent or so."

Trivedi adds, "When you look at the droplets with your eyes, they look like spheres. It's only when you magnify and measure them precisely that you find the spheres are altered in certain directions, so there's a different energy in different directions."



James Morris produces molecular dynamics simulations of coexisting liquid and solid phases, such as the one shown here, to see how the atoms behave at the interface. In this image, the red region in the center represents the disordered atoms of the liquid phase. The blue and green regions on either side represent the ordered atoms of the solid phase. By tracking the fluctuating region between the two phases, Morris can calculate the interfacial free energy.

The physicist's process

Getting a clearer picture of just how little the equilibrium droplet shape deviates from a true sphere is Morris' job. "You're looking at this droplet and saying, 'Well, this droplet isn't perfectly spherical; it has some small asymmetry.' We want to measure that, and we don't want it influenced by dirt in the system or anything else," says Morris. "The deviation is a very small number, but it's very important, and that's where doing the calculations and modeling the atomic fluctuations of the liquid-solid interface have come in."

In parallel with Trivedi's and Napolitano's experiments, Morris selects a model for how the atoms interact to calculate the variations in interfacial properties. To do so, he uses molecular dynamics simulations of the liquid-solid interfaces at equilibrium. His work shows how the atoms behave at the interface and allows him to track their equilibrium fluctuations to calculate interfacial free energy. "You're completely relying on whether the potentials used can provide accurate calculations, and in this case there's been no previous comparison of an experimental measurement with a detailed atomistic calculation," says Morris. "This is a first, and the fact that the comparisons come out nicely suggests that these potentials are suitable."

Small but mighty

Trivedi emphasizes again that the small effects dominate throughout nature. "It's very obvious in biology – think of gene expression in the development of the human embryo. Many things can go wrong, but by and large they don't," he says. "The same thing is true for the development of microstructures in metals. There are very fundamental issues that govern what happens. We would like to know precisely how they influence microstructure formation. To do that, we need to understand the small effects. We study metals, but the principles we hope to generate will have much broader applications." ♦

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BREAK-THROUGH BOOSTER

Process Science Initiative helps materials researchers over the humps

by Kerry Gibson

Marathon runners call it "hitting the wall." It's that point where the legs tighten, momentum drops, the pace slows, and the last ounce of energy has drained away. Without a boost – an energy bar for the body or a word of encouragement for the mind – the finish line seems impossible to reach.

Scientists may not have a term for it, but they too can hit the wall, reaching an impasse where they need help getting a research project back on track. That lift may be collaboration with a colleague, bouncing ideas and theories off a sympathetic ear, or getting access to material or a piece of equipment that's well beyond their institution's research budget.

Like a runner's energy bar, the Ames Laboratory's Process Science Initiative provides materials scientists with the necessary boost to help get them over the hump. Now in its third year, PSI has provided assistance to more than 25 different research projects. The primary assistance comes in the form of access to the Lab's Materials Preparation Center facilities and its personnel providing the materials science expertise.

Access to MPC

"The program is designed to help materials scientists who have reached some type of bottleneck," says Matt Kramer, PSI program manager and an Ames Lab scientist. "It's not a source of summer funding. We really look at how they can benefit from what the MPC has to offer," he adds, noting that it also helps the MPC better fulfill its role as a Department of Energy national user facility.

Proposals are subjected to a review process to make sure they are viable and have a strong materials-processing focus that either looks to improve fundamental understanding of existing processing techniques or explores new techniques for producing novel materials. As part of that review, an Ames Lab researcher with expertise in that particular area is assigned to follow up on a proposal that shows promise, discussing the problems being faced and how PSI may help provide the answers. Ultimately, that Ames Lab researcher is likely to be assigned as the coordinating principal investigator on the project if it is approved.

"That's one of the unique aspects of this program," Kramer says. "We have involvement by our researchers very early in the process, and they work closely with the project throughout."

According to Larry Jones, MPC director, the initiative has helped increase utilization of the center, but also has allowed the center to serve as a "front door" to Ames Laboratory by making researchers and their institutions more aware of the overall expertise and capabilities at the Lab.

"With our ability to fund research projects through PSI, we underwrite some of the risk in preparing new alloys or

Ames Laboratory assistant scientist Amy Ross checks the status of a crystal sample being grown in the High-temperature Bridgman furnace, one of many specialized pieces of equipment found in the Materials Preparation Center.

developing new processes," Jones says, "and bring that good, fundamental science into the public domain. That brings recognition to both the MPC and the Lab. It also provides challenges for us and pushes us to come up with solutions and ultimately expand our capabilities and offer even more to the research community."

Though the first crop of proposals in 2000 was small – four projects from Ames Laboratory and/or Iowa State University that lasted for four months – the program has grown in successive years. Ten projects were funded last year, representing Clemson University, North Carolina State University, the University of Nebraska-Lincoln, Purdue University, and Texas A&M University, along with Iowa State University and the DOE's Brookhaven National Laboratory.

Researchers sing PSI praises

"Fantastic" is just one adjective Richard Figliola, an aerodynamicist studying fluid dynamics at Clemson University, uses to describe the program. Figliola is in the second year of a study with Ames Laboratory senior metallurgist Iver Anderson in modeling high-pressure gas atomizer flows.

Though he has known Anderson and his work with high-pressure gas atomization – a process used to produce high-purity powdered metals – for a number of years, PSI has made it possible for Figliola to work directly with Anderson. Figliola uses experimental data collected by Anderson to model what happens as the stream of molten material is broken into tiny spheres by a high-velocity jet of compressed gas. And the models Figliola has developed, in turn, have assisted Anderson in modifying and improving the atomization equipment. The two hold several patents on the technology.

"We've gone from a very basic approach initially to a pretty sophisticated level both experimentally and theoretically," Figliola explains, adding that the pair has trained a "slew" of graduate students in the technology along the way. "It's been a true partnership. When one of us comes up with an idea, we'll have the other one try it out. The only thing that separates us as a research team is the 800 miles between Clemson and Ames."

In Figliola's case, working with Anderson has allowed him to continue research in an area where he would never be able to afford the state-of-the-art equipment necessary to carry out experimental tests based on his models. Another important aspect of the research is the high-purity of the materials used in the atomization process. Such materials are a hallmark of the MPC.

University of Nebraska researcher Jeff Shield calls the MPC "a one-of-a-kind, world-class facility" that's allowed him to investigate creating nanoscale intermetallic magnetic materials through roll-bonding. The problem with intermetallics, such as neodymium-iron-boron, is they are too brittle to be formed into ultrathin films like metals.

"We spread a tens-of-a-micron-scale Nd-Fe-B powder on copper foil and build up many alternating layers," Shield says. "Then we diffusion-bond the material at 500 degrees Fahrenheit (260 degrees Celsius) to help hold it together, then run it through a rolling mill. When it gets to a minimal thickness, we fold it up, diffusion-bond it again, then repeat the rolling, folding and bonding several times."

Larry Jones (right) discusses a materials request with MPC senior research technician John Wheelock. The Process Science Initiative provides access to the facilities, equipment and staff expertise at the MPC.

As the material is repeatedly rolled, the intermetallic particles fracture, but appear to disperse fairly evenly through the surrounding copper rather than forming in "clumps." Initial findings show the particles are nearing 25 nanomicrons in size, and Shield hopes to publish his results in the future.

"It's been wonderful to work with Scott Chumbley and the rest of the magnetics group on this project," Shield says. "As a university researcher, you might have access to one or two of the pieces of equipment but the MPC has it all – the materials; the bonding, milling and processing equipment; and characterization facilities. It's a total package not found anywhere else."

Shield has more than a passing knowledge of Ames Lab and the MPC. He received his Ph.D. from Iowa State and was a scientist at the Lab during the time he was here. That connection not withstanding, Shield adds that materials scientists everywhere take a keen interest in what's happening at Ames Laboratory.

"Word of mouth is still one of our biggest assets," Kramer says of the PSI program. "As people become aware of the program, they tell their colleagues, and it continues to grow and expand." ♦

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Materials Science and Engineering Division

Ames Laboratory's "Cool Work" Makes a Splash at the G-8 Conference

Ames Laboratory senior metallurgist Karl Gschneidner and his magnetic refrigeration research took center stage for a time at the Conference of G-8 Energy Ministers in Detroit, Michigan, in May 2002. Gschneidner welcomed Secretary of Energy Spencer Abraham to the magnetic refrigeration display, which also featured a working prototype of a room-temperature, permanent-magnet-based magnetic refrigerator built by scientists at Astronautics Corp. of America. Magnetic refrigeration is based on the magnetocaloric effect, or the ability of some materials, such as gadolinium, to heat up when placed in a magnetic field and then cool down when removed from the field. Magnetic refrigeration is more efficient than conventional refrigeration and does not use harmful gases or hazardous chemicals to operate. Following introductions, the Lab's display served as the backdrop for the ribbon cutting ceremony to open the G-8 Conference. The display was one of eight from DOE laboratories and programs chosen by the Office of Science to represent DOE in the exhibit at the conference. Conference participants included energy ministers from the eight most powerful countries – the United States, Canada, France, Germany, Italy, Japan, Russia, the United Kingdom and the European Nation. They work to promote and implement policies geared toward meeting the global energy and environmental challenges of the 21st century.

Karl Gschneidner (left) welcomes Secretary Abraham to the Ames Laboratory display. The Secretary was joined by Argonne National Laboratory Director Hermann Grunder (far right) who, with the Secretary, cut the red ribbon to open the DOE exhibit at the G-8 Conference.

Command Performance

In a command performance, Ames Laboratory presented its magnetic refrigeration display at a special ceremony at DOE Headquarters in Washington, D.C., in May 2002. The ceremony, sponsored by DOE Secretary Spencer Abraham, was held to mark the one-year anniversary of President Bush's National Energy Policy. Secretary Abraham, Secretary of the Interior Gale Norton and EPA Administrator Christine Todd Whitman viewed the magnetic refrigeration display. ♦

DOE Secretary Abraham (right) and Secretary of the Interior Gale Norton listen as Steve Karsjen, Ames Laboratory Public Affairs manager, explains the Lab's magnetic refrigeration display.

ATOMIC FINGERPRINTS

Nuclear magnetic resonance reveals atomic structure of solids

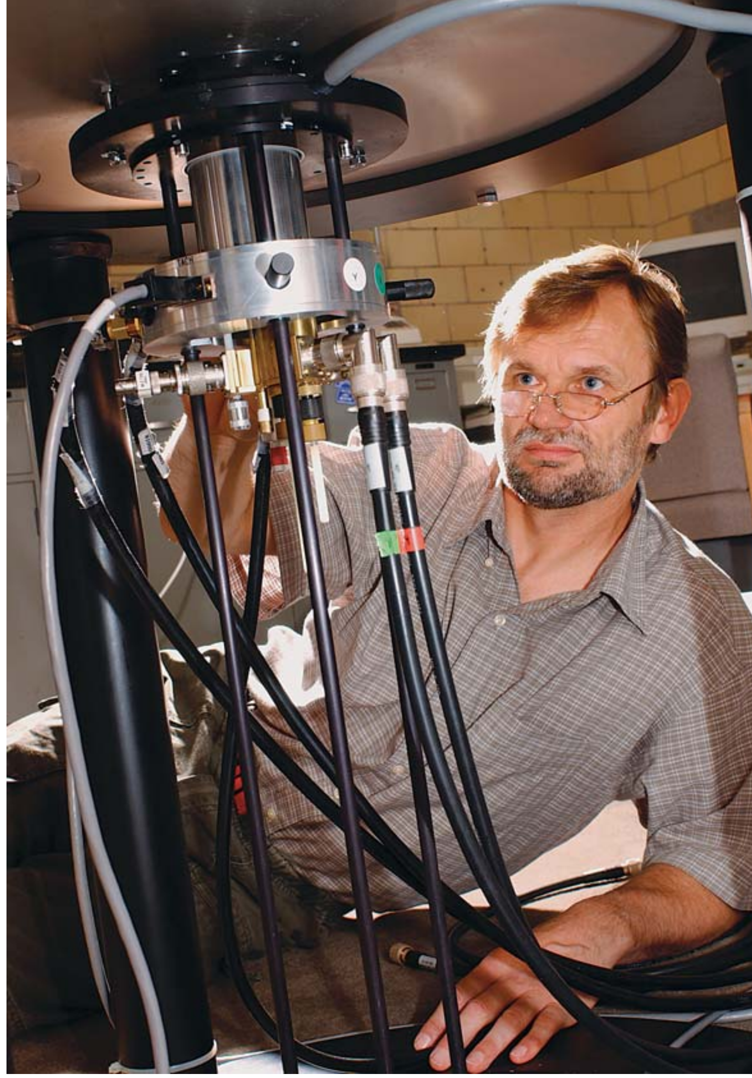
by Saren Johnston

It's a wonderful, investigative tool," says Marek Pruski, talking about solid state nuclear magnetic resonance spectroscopy. "There are many applications of solid state NMR, especially to the study of catalytic materials. The problem is that it is difficult to be really good at this type of spectroscopy and also be an expert in catalysis," adds the Ames Laboratory physicist and solid state NMR guru. Because NMR is a technique that is highly developed and requires fairly extensive knowledge, it usually comes at the expense of catalysis or other applications in chemistry and materials science. "Few people do both really well," says Pruski. "Most of the time, researchers are heavily on one side or the other. That's why it makes perfect sense that this type of expertise exists within the Department of Energy's national labs, where we work as teams."

It's all in the name

Defining NMR, Pruski says, "Nuclear magnetic resonance – the name actually explains what it does. You look at the splitting of the nuclear energy levels in a magnetic field." When most atomic nuclei are put in a magnetic field their energy levels split, and it is possible to probe the transition frequency between those energy levels. (The transition frequency is the characteristic frequency of radiation that the atomic nuclei must absorb or release to undergo transition from one energy state to another.) "Those frequencies are very sensitive to the local molecular environment in which these nuclei find themselves, and provide an excellent fingerprint of that site," Pruski explains. "Monitoring and understanding these frequencies, this is what NMR is all about."

Not all nuclei experience NMR. Only those that possess a nonzero total angular momentum, called spin, can produce an NMR signal, or spectrum. High-resolution spectra, in turn, can provide detailed information about the physical, chemical and biological properties of matter. Solid state NMR spectroscopy, Pruski's specialty, is used to determine the molecular structure and dynamics of solids and, so, can be used to study catalytic materials; glasses; other crystalline and amorphous inorganic and organic compounds; and biological samples.



Marek Pruski adjusts the probe containing the sample of interest that will be examined using NMR spectroscopy. The NMR spectrometer uses a magnetic field of 9.4 tesla – five orders of magnitude beyond that of the earth's magnetic field.

The need for narrow

Securing high-resolution spectra for solids is challenging because Mother Nature does not provide them naturally as she does for liquids. In liquids, the fast, random motion of molecules results in creating narrow spectral lines. But solids are another story. "If you think about a sample that is powdered – a crushed crystal, for example – the spectrum is composed of the contributions from many crystallites, which are randomly oriented with respect to the magnetic field and give resonances at different frequencies," says Pruski. "Furthermore, the contributions from individual crystallites may be homogeneously broadened due to dipolar couplings between nuclear magnetic moments. These conditions cause severe broadening of the spectrum, loss of resolution, and often yield spectra that are very difficult to interpret."

Pruski continues, "In NMR spectroscopy, 'narrow is beautiful,' because we are striving to suppress the broadening interactions to obtain the highest possible resolution." Accomplishing that task in solids requires manipulating the internal interactions in the laboratory space by mechanically spinning the sample; or in the spin space by applying radio frequency, or RF, pulses; or by doing both simultaneously. "In essence, what we're trying to do with these techniques

Marek Pruski displays one of the probes used with the NMR equipment in his laboratory. The turbine in the probe spins the sample at almost two million rpms to overcome the broadening of spectral lines.

is to do what nature does with liquids so we can achieve line-narrowing in the spectrum," says Pruski. "It is usually easier to obtain high resolution for spin 1/2 nuclei, such as ^{13}C or ^{1}H, where the anisotropy of the chemical shift and dipolar couplings dominate the spectra. (Nuclear spin is measured in the units of h/2p , where h is a Planck constant. A total number of protons and neutrons is odd for spin 1/2 nuclei.) However, quadrupolar nuclei, those with spins greater than 1/2, such as ^{17}O, ^{27}Al and ^{23}Na, present a challenge in that quadrupolar interaction brings about broadening of the spectrum. The majority of NMR active nuclei are affected by the quadrupolar interaction. "Of course, how else would it be?" Pruski asks with a laugh.

The quadrupolar quandary

According to Pruski, the quadrupolar interaction involves a coupling between the nonspherical distribution of nuclear electric charge with the local electric field produced by the surrounding atoms. In cases where the quadrupole effects are sufficiently small, the quadrupolar nuclei can be observed by using the same techniques as those used for their spin 1/2 counterparts. More common, however, are the circumstances when these techniques fail. Pruski says this is due to the presence of the so-called second-order effects, which are negligible in cases of chemical shift or dipolar interactions. "As a result, you can't just rotate the sample around a single axis and perform some of the RF tricks you do with first-order effects, and obtain high resolution," he says. "It just won't work. The crystallites in your powder would still have different frequencies of resonance, even though you'd applied those methods. This has been known for quite some time, but people didn't think it was practically possible to provide a sufficiently complex motion to the sample, or do some sort of spin gymnastics that would remove the line-broadening."

However, Pruski notes that two techniques to do just that were developed at A. Pines' lab at the University of California, Berkeley, in 1988, which used mechanically complex devices to achieve the effect of motional narrowing. "One of these methods, called double-rotation NMR, required spinning the sample around two different axes simultaneously at spinning rates on the order of $10^5 – 10^6$ rpms" he says. "You can only imagine the sophistication of a probe needed for those experiments."

In 1995, a mechanically simpler solid-state NMR technique called multiple-quantum magic-angle spinning, or MQMAS, was developed by L. Frydman at the University of Illinois, Chicago. MQMAS only requires spinning the sample around one axis and uses multiple quantum transitions to manipulate the spins and achieve high-resolution spectra.

"This is not a very easy experiment to explain," admits Pruski, "but once you get used to thinking about NMR in terms of coherence pathways and coherence transfers, the idea becomes simple. You basically use a coherence level as another experimental variable that you can control." He explains that two different variables are needed to achieve line-narrowing effect in the quadrupolar case. Rather than using two different axes of rotation to give motion to the sample, as in the earlier methods, MQMAS relies upon using two different coherence states of the spins and changing those states during the experiment by means of appropriate RF irradiation. (Coherence in NMR is a property of the spin density matrix that describes the extent to which interference between the eigenstates of the isolated spin subsystems persist in the entire statistical ensemble.) This provides a two-dimensional correlation spectrum that contains the high-resolution, isotropic information.

Pruski notes that the ideas about manipulating coherences were known earlier from liquid state and solid state NMR spectroscopy of spin 1/2 nuclei. "As it turns out," he adds, "the use of multiple-quantum spectroscopy has also been an incredibly good idea for the quadrupolar nuclei in solid state NMR."

Making the most of MQMAS

Pruski's interests involve the development of new NMR techniques that combine the resolving power of MQMAS with other solid state NMR methods to obtain information about connectivities and distances between atoms in solids. This effort includes the use of double-and triple-resonance experiments in which the correlations between various nuclear spins are investigated using through-space (dipolar coupling) or through-bond (J-coupling) interactions. Several such techniques were developed in his group, including cross-polarization MQMAS; MQMAS with rotational echo double resonance, or MQ-REDOR; and two-dimensional heteronuclear correlation, or 2D MQMAS-HETCOR, NMR. "With proper decoupling schemes, these techniques can provide the information about atomic geometries, including distances, with exceptional resolution," says Pruski.

He and his research associate, Jerzy Wiench, apply their solid state NMR expertise primarily to the field of catalysis. "We are interested in measuring correlations between spins under the high resolution provided by multiple quantum spectroscopy," says

Pruski. "Currently, we are able to measure distances between spins in multispin systems with precision that approaches that of the diffraction methods. We don't need material in a crystalline form to perform such studies, although we do require a large number of spins to generate spectra." He offers an example of how such measurements might be applied in a catalytic system. "For instance, if there is a molecule adsorbed in a zeolite interacting with some catalytically active sites, we can try to measure the distance between certain atoms in that molecule and the active site. This would give us the position of the molecule within the catalyst and a better understanding of the activity of that particular site."

Pruski and Wiench use their solid state NMR capabilities in various collaborative efforts. Among the collaborations in catalysis are ones with Glenn Schrader, an Ames Lab associate and an Iowa State University professor of chemical engineering, and Robert Angelici, an Ames Lab senior chemist and an ISU distinguished professor of chemistry. Projects in these collaborations are funded through Ames Laboratory's Chemical and Biological Sciences Program. The most recent ISU collaborators include Victor Lin, an assistant professor of chemistry, and Brent Shanks, an associate professor of chemical engineering. They have relied on Pruski's and Wiench's expertise in solid state NMR spectroscopy to provide data critical to their work on catalytic systems for nanoscience efforts.

In a Work for Others project, Pruski and graduate assistant Krishnan Damodaran are interacting with a Brazilian company, Petrobras Brasiliero, using solid state NMR spectroscopy to characterize aluminum in zeolites and industrial catalysts.

Pruski emphasizes that his most important external collaborators include professors J.-P. Amoureux and C. Fernandez, from the French universities of Lille and Caen, respectively, with whom he has developed some of the new solid state NMR techniques.

Although Pruski and Wiench remain focused on catalysis, they also seek interactions with others whose research might benefit from the contributions solid state NMR can provide. A current and greatly successful collaboration with Ames Laboratory senior scientist Vitalij Pecharsky and associate scientist Viktor Balema has drawn Pruski and solid state NMR spectroscopy into the world of mechanochemistry. His contributions to their new solvent-free process of creating organic compounds in solid state (see "Shaken, Not Stirred," page 4) has provided the means of verifying Pecharsky's and Balema's experimental results. "It turned out that solid state NMR has been a very efficient tool to analyze not only the products of their mechanochemical transformations, but also to monitor the progress of these reactions," says Pruski. "You can't do that with X-rays. NMR seems to be working very well, and the collaboration has been very interesting and fruitful."

Pecharsky says, "Marek's expertise and willingness to collaborate are first-class. His solid state NMR data were key components in our research on the solvent-free mechanochemical synthesis of molecular compounds because for the first time we were able to obtain decisive proof that certain types of reactions between organic molecules proceed in solid state. This discovery opens up new opportunities and presents new challenges for materials science, synthetic organic chemistry and solid state NMR spectroscopy. We continue to work together to gain better understanding of the mechanisms of these unconventional processes."

Pruski and Wiench will continue to participate in collaborations where their skill in solid state NMR spectroscopy is likely to provide the "atomic fingerprints" needed to resolve questions associated with the structure of solid materials. "We enjoy the collaborations a great deal," says Pruski, "but we are also very interested in improving the efficiency and sensitivity of our experiments and in studying how best to manipulate the spins. The common thread in our basic research has always been the development of NMR techniques – that is a constant that remains." ♦

Jerzy Wiench inserts a 5-millimeter rotor containing the sample of interest into the magic-angle spinner in one of the NMR probes.

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Natural Born Scientist

by Kerry Gibson

Edward S. Yeung, director of Ames Laboratory's Chemical and Biological Sciences Program and an ISU Distinguished Professor, has received the prestigious American Chemical Society Award in Chromatography. Yeung was honored during a special awards ceremony at the 2002 ACS national meeting.

The selection of Yeung to receive the ACS Award in Chromatography speaks to the enormous and impressive volume of research he has produced in the area of chemical separations. To be nominated for this award, the individual must have made an outstanding contribution to the field of chromatography, with particular consideration given to development of new methods.

"It is significant to note that Dr. Yeung's work has contributed to both our fundamental understanding of chromatography and to the way chromatography is being practiced today," says Michael J. Sepaniak, head of the Department of Chemistry at The University of Tennessee, Knoxville. A former student of Yeung's, Sepaniak nominated him for the ACS award.

Among his many research accomplishments leading to the ACS Award in Chromatography, Yeung pioneered the method of multiplexed capillary electrophoresis and in 1995 incorporated the technique into a fluorescence-based DNA sequencer that dramatically reduced the time needed to gather information on an individual's genetic makeup.

Yeung recently combined the technique with absorption detection, creating a new technology that can gather data on a much wider range of compounds. His revamped technology relies on the amount of ultraviolet light molecules in a compound absorb to determine their type and quantity, eliminating the need to attach optical tags to those compounds that don't fluoresce naturally.

In other work, Yeung has developed a method to track the activity of single molecules in solution, a breakthrough that ultimately may have great implications within the fields of medicine, catalysis and biotechnology. With this approach, Yeung was able to elucidate the fundamental mechanisms of chromatography with unprecedented detail.

Yeung has received many awards for his research, including the ACS Fisher Award in Analytical Chemistry in 1994 and the ACS Division of Analytical Chemistry Award in Chemical Instrumentation in 1987. In 2001, he received his fourth R&D 100 Award. He is a Fellow of the American Association for the Advancement of Science.

Chemistry has always interested Yeung, who grew up in Hong Kong. After graduating from high school, he came to the U.S., earning a bachelor's degree in 1968 from Cornell University. Four years later, at the age of 24, he earned his Ph.D. in physical chemistry from the University of California (Berkeley). It was his interest in laser spectroscopy that brought him to Ames as an instructor of analytical chemistry at Iowa State University and an assistant chemist at Ames Laboratory. He's been at Iowa State and the Ames Lab ever since.

"It is easy to do something that is fun," he says of his distinguished career in chemistry, "and it's been something that comes naturally to me … most of the time," adding that it's a bonus to enjoy something "that also pays one's salary."

In what may have been a sign of things to come, he became interested in chromatography through a conversation with Ames Lab researcher Jim Fritz, who won the ACS Chromotography award in 1976. "I found out there are challenging unresolved issues with chromatographic mechanisms and chromatographic instrumentation," Yeung says.

Finding answers to those unresolved issues has been the motivating force behind Yeung's work. As he explains it, the answers have come by breaking a complex problem into a series of questions, then trying to take a novel approach in answering those questions.

"In general, scientific research starts with simple questions on how or why things work in a certain way," Yeung says. "Then, specific studies are designed to answer such questions. Satisfaction comes when one is proven correct in proposing the initial approach or hypothesis, i.e., getting the right answer.

"The biggest challenges are trying to go against conventional theories or standard approaches and trying to develop something different, be it an idea or an instrument," he explains. "Scientists are, in general, conservative, and many of our projects would probably have gone unfunded if the exact plans were laid out in front of grant committee referees."

Not everything comes as easily to Yeung as solutions to complex chemistry problems. For example, he points to two of his favorite diversions.

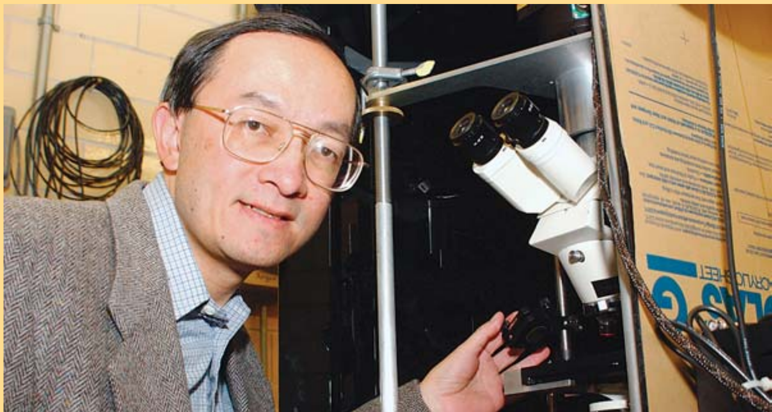
"I play the violin," he says and adds that he enjoys art rather than creating it. "These pursuits are far more difficult to me than chemistry. Perhaps that's why I immerse myself in them whenever I can."

Though natural talent has its place, Yeung appreciates the value of hard work in overcoming obstacles. His music has required that kind of dedication and discipline. "In science, music or art, more advances come from hard work than sheer talent," he says.

He also admits that there's a certain amount of luck involved in making scientific discoveries.

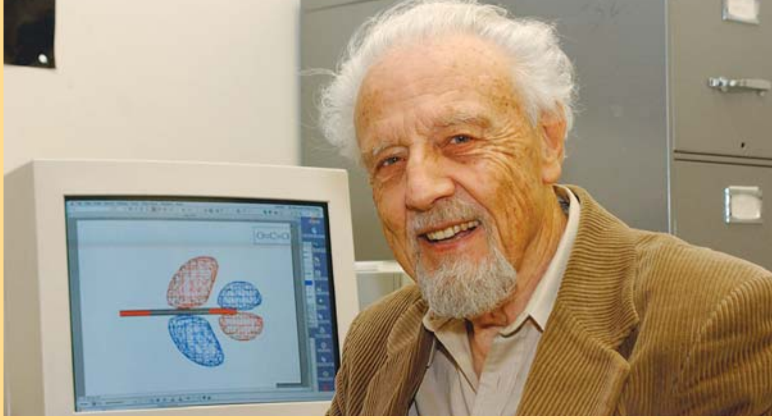
"Science often is opportunistic and it is hard to say what will be discovered next," Yeung says about where his research may take him. "For now, I will settle for learning to play the violin better." ♦

Ed Yeung uses a microscope and a charge-coupled-device camera for single-molecule detection research.



A Giant Among Us

by Saren Johnston



Klaus Ruedenberg with the display of a localized molecular orbital depicting the distribution of electrons in carbon dioxide.

Klaus Ruedenberg, an Ames Laboratory senior associate and an Iowa State University distinguished Professor Emeritus, has received the prestigious American Chemical Society Award in Theoretical Chemistry for 2002. Nominees for this honor must have accomplished "innovative research in the field of theoretical chemistry characterized by depth, originality and scientific significance." His award states that he has seminally advanced many different, important facets of quantum chemistry, encompassing fundamental theory, formal mathematical developments, computational methods and software implementations, as well as conceptual interpretations.

Modest about his accomplishments, Ruedenberg allows that "what we end up doing is somewhat accidental. We choose among the professional options available to us, those that fascinate us and pose problems we feel we will be able to help solve."

He grew up in Bielefeld, Germany, where his father worked for a chemical company. Ruedenberg began his studies by earning an M.S. in chemistry at the University of Fribourg, Switzerland. Finding mathematics and theory more attractive than experiments, he switched to theoretical physics at the University of Zürich where, in 1950, he obtained a Ph.D. in meson field theory with the renowned Gregor Wentzel, whom he followed to the University of Chicago.

There he became a postdoctoral fellow with Roberts S. Mulliken, who two decades later received the Nobel Prize for work in theoretical chemistry. "This was then one of the very few places where one could combine chemistry and theoretical physics," Ruedenberg comments, noting that "theoretical chemistry is one of the invasions of physics into chemistry. Still, the field would not have blossomed," he continues, "if it hadn't been for the subsequent development of electronic computers. I was lucky."

The prominent ACS Award in Theoretical Chemistry honors him as one of the few quantum chemists in the world to be recognized as a leader in establishing the field of theoretical chemistry and ensuring its viability during the last 50 years. Many of the methods and concepts introduced by him have become widely accepted and used. "Professor Ruedenberg is one of the most highly respected and accomplished theoretical chemists in the world," says Mark Gordon, director of Ames Laboratory's Applied Mathematics and Computational Sciences Program and an ISU Distinguished Professor. Gordon, who nominated Ruedenberg for the ACS award, continues, "Klaus' recognition arises not only because of his outstanding research accomplishments, but also as a result of his many contributions to the scientific community as an articulate and inspiring leader."

Among Ruedenberg's most noteworthy contributions is his elucidation of the energetic interactions that cause molecule formation. It has led to profound insights into the basic origin and the physical nature of the chemical bond.

Closely related are his methods for creating a rigorous quantum theoretical foundation for the two-hundred-year-old empirical model of molecules being built from atoms. His approach elucidates how atoms get modified in a molecular environment.

Fundamental theoretical aspects regarding the understanding of chemical reactions have been illuminated by Ruedenberg's studies of molecular potential energy surfaces. His discoveries regarding intersections of energy surfaces are relevant for photochemistry.

In recent work, Ruedenberg has been addressing the problem of electron correlation, a major bottleneck in the quest for accurate predictions of the properties, in particular energies of ground and excited electronic states of large molecules – a fundamental as well as practical goal of theoretical chemistry.

Ruedenberg is a Fellow of the American Physical Society, the American Institute of Chemists and the American Association for the Advancement of Science. His honors include the Midwest Award of the American Chemical Society, membership in the International Academy of Quantum Molecular Sciences, and honorary doctorates from the universities of Basel (Switzerland), Bielefeld (Germany) and Siegen (Germany). For many years, he was editor-in-chief of the scientific journal, *Theoretica Chimica Acta.*

While being a consummate scientist, Ruedenberg has not put aside his early classical humanistic upbringing and remains fascinated by history, literature and, in particular, the human mind and its fundamental motivations. But he believes that in our age basic lessons for these dimensions also flow from the insights of the natural sciences. He observes, for instance, that every stationary state in nature is a dynamic balance between many opposing tendencies – from the motions of electrons to those of the celestial bodies and from the equilibria in chemical test tubes to those in living organisms. He thinks that to be a responsible human means to acknowledge the many mutually opposing forces in oneself and to affirm the internal tensions and efforts that go with working out a harmonious moral balance.

How fortunate for Ames Laboratory that in 1955, when only a very few chemistry departments showed any interest in theoretical chemistry, the young ISU professors and Ames Lab researchers Robert Rundle and Robert Hansen had the foresight to invite Ruedenberg to Ames Laboratory and Iowa State to delve into the harmonies of molecular electrons. ♦

"Lite" Done Right

New parallel library succeeds by going easy on the extras

by Saren Johnston

Better performance for less hassle – that's the advantage of a new message-passing library developed at Ames Laboratory. The library, called MP_Lite, makes it possible to extract optimum performance from both workstation and personal computer clusters, as well as from large massively parallel computers. It supports and enhances the basic capabilities that most software programs require to communicate between computers.

Fewer and faster

MP_Lite might be thought of as a "slimmed-down" version of the message-passing interface, or MPI, standard, a widely used model that standardizes the syntax and functionality for message-passing programs, allowing a uniform interface from the application to the underlying communication network. The MPI standard eases the parallel programming task by providing a common syntax for communicating between computers, making codes portable between widely varying supercomputers.

The full MPI standard contains many options that most people don't use very often. MP_Lite offers only the core functions of MPI, which are enough for most codes. The emphasis is put on implementing these functions in the most efficient manner, providing all the performance without all the extra options. "After all, a Corvette will go fast and look good even without a CD player and electric seats," says Dave Turner, an Ames Laboratory computational scientist and the principal investigater for the MP_Lite project.

Perfecting performance

MP_Lite could be scaled up easily, but its objective is not to provide all the capabilities of the full MPI standard. "Our goal with MP_Lite is to illustrate how to get better performance in a portable and user-friendly manner and to understand exactly where any inefficiencies in the MPI standard may be coming from," says Turner. He explains that the MP_Lite library is smaller and much easier to work with than full MPI libraries. "It's ideal for performing message-passing research that may eventually be used to improve full MPI implementations and possibly influence the MPI standard," he says.

Turner notes that it was "mainly frustration" that led him to develop the MP_Lite library. "Most message-passing packages are large and clunky to work with, and can be difficult to install and optimize. If you run into any errors at all, they give you very cryptic messages that mean nothing unless you actually wrote the library," he says. "So a lot of the reason I got into this project was not just to improve the efficiency, but also to improve performance – make the message-passing more user-friendly."

Offering an example, Turner says, "If two processors are communicating, and one waits a minute for a response from the other one – well, a minute is a very long time in this context – the library should put out a warning into a log file. But that's something that's not done. Most message-passing systems don't tell you what's wrong if a communication buffer overflows or a node is waiting for a message that never gets sent. What if there's a five-

Dave Turner shows how the MP_Lite library is organized in this simplified sketch. Running beneath a full MPI implementation, such as Argonne's MPICH, MP_Lite can pass on its efficiency and performance to the larger library.

minute wait for a message?" he continues. "Something is probably frozen up, so at that point the library should implement an abort and give the user as much information about the current state of the system as possible."

Turner notes that MP_Lite operates with minimal buffering and warns if there are any potential problems. When possible, MP_Lite will dump warnings to a log file and eventually time out when a lockup occurs. "There's a lot of these user-friendly aspects that I'd like to see put into other message-passing systems," he says. "That's one area that doesn't get focused on very often.

"Many of the people developing parallel library codes don't necessarily use them, and it's not in the MPI standard that you have to make them user-friendly," Turner continues. "They're getting paid to add functionality. For them performance and usability have been almost a second consideration, which is unfortunate."

Putting on the "squeeze"

Turner says MP_Lite was "born out of a need to squeeze every bit of performance from the interprocessor communication between nodes in personal computer or workstation clusters, where the Fast Ethernet and even Gigabit Ethernet speeds are much slower than communication rates in traditional massively parallel processing systems." This is due to the fact that although massively parallel systems have processors very similar to those of PCs or workstations, they also have more customized communications systems that allow faster communication rates.

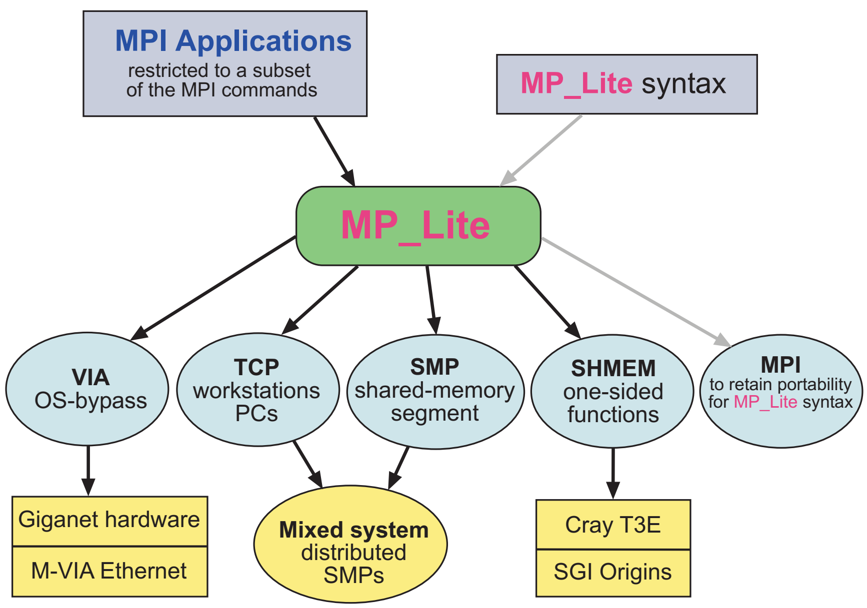
"In PC and workstation clusters, we're catching up to where we're getting at least closer to those rates, but we still need to squeeze everything out that we can," says Turner. "So one of the things that I do is look for where we can squeeze it out. Is it at the message-passing level; in the operating system, itself; or in the driver implementations – there's a lot of evaluation in the whole message-passing, or communication, hierarchy that's involved in the MP_Lite project."

In addition to enhancing performance, another goal Turner has for MP_Lite is to tie it directly to a full MPI library. To do so, he's been working with the Department of Energy's Argonne National Laboratory and running their MPICH library on top of MP_Lite. "By doing this, we can pass the good performance of MP_Lite on to the full MPI implementation," he says. "So we combine the best of both, keeping the efficiency of my library and the greater functionality of Argonne's."

Turner says he named the library MP_Lite for several reasons. The small size of the library's code makes it easy to install anywhere – it compiles in under a minute. There's much less code, so it's more streamlined than MPI. It can also be easily modified to include more of the full MPI functions. And MP_Lite has its own syntax, which is simpler and can be used in place of the MPI syntax. Of course, there's always the fun of the clever response Turner is able to make to people who say to him, "I use this MPI function; why isn't it in your library?" He simply replies, "Well, it's 'lite.' "

MP_Lite is on the World Wide Web at *http://www.scl.ameslab.gov Projects/ MP_Lite/* and can be downloaded free of charge. "I tell people straight out that it's an experimental product under development," says Turner. "It's been a good way of finding out about bugs in the code."

Turner admits that the work on MP_Lite suits him well. "I like the puzzle aspect of it," he says. "I like tuning codes and getting them to run on a scalable computer, and trying to squeeze more performance out of what's there." ♦



MP_Lite delivers nearly the full range of performance as the underlying communication layer, and does so in a portable and user-friendly manner. It can be run on top of transmission control protocol, or TCP, on clusters of workstations; the high-performance, native SHMEM library on Cray T3E and SGI Origin systems; and on any system where MPI is installed to retain complete portability. Performance is achieved by keeping everything simple and clean.

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Thin-film Fabricator

New lab puts researchers on thin-film's cutting edge

by Kerry Gibson

John Snyder adjusts the secondary ion-beam gun that is used in etching the surface of thin films created in the deposition system.

A new state-of-the-art lab is giving Ames Laboratory researchers unprecedented control to create and, perhaps more importantly, duplicate thin films with specific compositions and nanostructures. That ability to control a number of variables allows thin-film "recipes" to be developed and compared, resulting in a better understanding of how thin films work.

Funded by a $530,000 grant from the Roy J. Carver Charitable Trust, via Iowa State University, the centerpiece of the Magnetoelectronics Laboratory is a new, custom-built Indel ion-beam deposition system. It allows researchers David Jiles and John Snyder to easily and quickly create multilayer thin films. As Snyder explains it, the system works like a well-placed break shot in a game of pool.

"To lay down a thin layer of a certain material, you place a piece of it on a target plate," Snyder says. "Then the ion beam blasts that target and a thin layer of the material, maybe only a few atomic layers thick, is deposited on the substrate, usually a silicon wafer. Based on the placement of the beam, we know where the atoms of the target material will wind up."

The material being deposited must be highly pure, and the process must take place under extreme vacuum (10^{-7} Torr or better) and in a dust-free environment, since even microscopic dust could cause variations or breaks in the atoms-thin film. Obtaining high-purity materials is the easy part, thanks to Ames Lab's Materials Preparation Center. Clean-room air handlers control the dust. Achieving such high vacuum, however, is time consuming – it takes several hours, using both mechanical and cryogenic pumps.

To speed up the process and minimize the possible introduction of oxygen, dust or other contaminants, the new system has a turret with six target plates. This allows Snyder and Jiles to load six different materials in advance without having to break and reestablish the vacuum within the deposition chamber. To deposit the different materials, the researchers simply rotate the desired target plate into position.

The chamber also has a secondary ion-beam gun that allows etching of the substrate between layers so the researchers can remove excess material or "clean" the surface before the next layer is deposited. They also designed the chamber so they can heat and cool the deposition substrate, subject it to magnetic fields, and rotate the substrate table.

"By subjecting the substrate to a variety of conditions, we can change how the thin-film material grows," Jiles says. "Particles may orient differently when exposed to a magnetic field or grow differently when heated or cooled during deposition. The beauty of this equipment is that everything is computer-controlled so we can easily recreate a particular 'recipe' for any thin-film wafers we create."

Having that type of control is crucial because minute changes in layer thickness or particle orientation may change the film's properties. For example, magnetic tunnel junctions consist of a thin insulating film sandwiched between two magnetic films. The thinner you make the insulating layer, the less electrical resistance produced. However any gaps in the insulation will cause the structure to short out, just like an electrical cord with frayed insulation.

Magnetic tunnel junctions could replace semiconductor technology now used for a computer's random access memory. When you run a software program, RAM keeps the application accessible and allows users to read data from memory and write new data into the memory. However, most semiconductor-based RAM is volatile, meaning that it requires constant electrical power to operate. If power is lost, so is the data being held in RAM.

"Magnetic tunnel junctions operate magnetically, so they aren't affected by power interruptions," Jiles says. "You wouldn't lose data, and the computer would come on instantly without going through the boot-up process."

Thin films can also possess giant magnetoresistive, or GMR, properties that allow them to undergo dramatic changes in their electrical resistance in response to relatively small changes in the magnetic field surrounding them. Their high sensitivity and small size would make such films desirable for use on the read-heads on next-generation disks, capable of storing 100-500 gigabits of data per square inch.

"The research conducted for the Department of Energy is focusing on the mechanisms of clean, multielement film growth and the structuring of such films at the atomistic to nanolength scale," Jiles says. "It opens whole new areas of research, and we hope to be able to use the facility as a lever to attract research funding and to build other projects around this basic work." ♦

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The Competition Continues

2002 Science Bowl makes it an even dozen

Most of the students who competed in the Ames Laboratory/Iowa State University 2002 Science Bowl were kindergarteners when the popular science competition began 12 years ago. And, unbelieveably, some of them were only preschoolers. Fashions and fortunes, music and markets, ideas and idols – indeed, nearly all things – have changed in those dozen years. But the desire to compete and to win never have and, hopefully, never will because they keep the heart and mind alive. At Ames Laboratory, we have been happy observers of that simple fact each January when we host the annual Science Bowl competition for up to 48 bright and enthusiastic student teams from high schools throughout Iowa.

The 2002 event was no different. The competition was fun, fast and furious throughout the day, ending with a championship match that saw the team from Cedar Falls High School in Cedar Falls, Iowa, winning the regional competition. With that victory, they also won the right to participate in the Department of Energy's National Science Bowl in Washington, D.C., in May. ♦

Students from Cedar Falls High School in Cedar Falls, Iowa, display the $1,000 check they received for their school's science department as a result of finishing in the top 18 out of 64 teams at the DOE's National Science Bowl.

It's a happy moment for Cedar Falls coach Robert Nelson (right) as Ames Laboratory Director Tom Barton congratulates him and members of the Cedar Falls student team on winning the 2002 Ames Lab/ISU Science Bowl

Students from Iowa City West High School in Iowa City, Iowa, say it was "girl power" that led them to a third-place finish in the Ames Lab/ISU 2002 Science Bowl. They were on one of the few competing teams made up of more girls than boys.

Concentration and collaboration are key ingredients in Science Bowl competition, as this team from Ames High School in Ames, Iowa, demonstrates.

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Ames Laboratory is a U.S. Department of Energy laboratory seeking solutions to energy-related problems through the exploration of chemical, engineering, materials and mathematical sciences, and physics. Established in the 1940s with the successful development of the most efficient process to produce high-purity uranium metal for atomic energy, Ames Lab now pursues much broader priorities than the materials research that has given the Lab international credibility. Responding to issues of national concern, Lab scientists are actively involved in innovative research, science education programs, the development of applied technologies and the quick transfer of such technologies to industry. Uniquely integrated within a university environment, the Lab stimulates creative thought and encourages scientific discovery, providing solutions to complex problems and educating tomorrow's scientific talent.

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