Features

Kai-Ming Ho totes this colorful plexiglass model with him when presenting invited talks throughout the world. It provides the perfect example of his original layered lattice design for three-dimensional photonic bandgap crystals. (see story page 8)

(back cover) A single crystal of copper sits in a sample holder while scientists prepare to conduct tests using a technique known as acoustic harmonic generation. The technique may provide the earliest information about cracks and fatigue in materials. (see story page 13)

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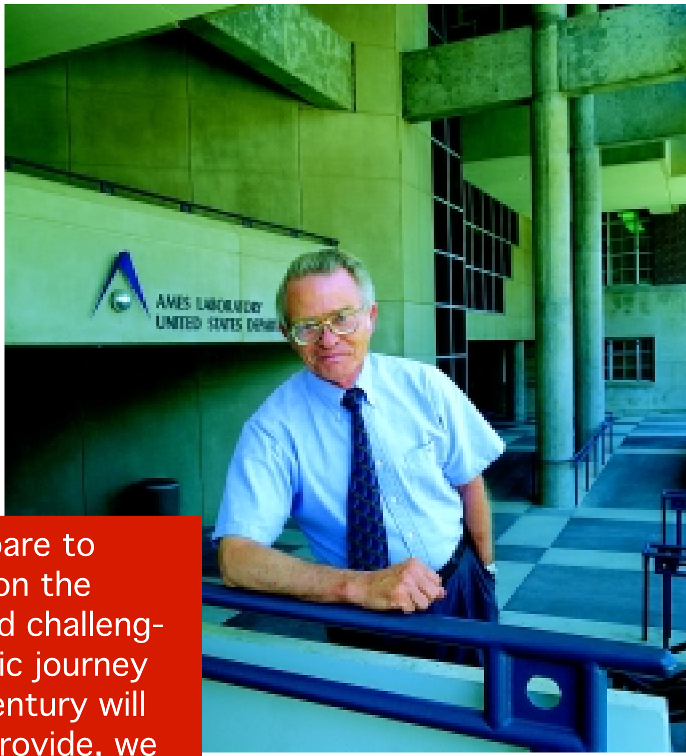
WINNER APEX '99 AWARDS FOR PUBLICATION EXCELLENCE

Greetings from the
Director



Welcome to the 1999 issue of *Inquiry* magazine – our annual mechanism for providing a sampling of the unique and creative research being conducted by scientists at Ames Laboratory.

As this issue is arriving at your office, we are preparing for a significant event in the life of Ames Lab – one that plays a key role in every piece of research featured in *Inquiry* – the signing of our new five-year contract with the Department of Energy. In the strongest way possible, this agreement will provide the foundation from which we will continue to advance the world-class research that has become the hallmark of Ames Laboratory throughout its 52-year existence.

As we prepare to embark upon the exciting and challenging scientific journey the next century will no doubt provide, we look forward to those research opportunities that have yet to present themselves.

As we prepare to embark upon the exciting and challenging scientific journey the next century will no doubt provide, we look forward to those research opportunities that have yet to present themselves. Key to our ability to address those opportunities is the synergistic approach we take in accomplishing our research. Examples of that synergy are prevalent in the stories featured in this issue of *Inquiry*.

AMES LABORATORY

I invite you to turn the page and witness our scientists' substantial efforts to help solve our nation's energy problems. It is an endeavor that is as exciting today as it has been at any time throughout the history of Ames Laboratory.

Tom Barton

(From left) Sina Maghsoodi, Mohammad Nosrati and Iver Anderson stand atop a board supported by small platforms made with the glue they invented.

STICKING *With It*

After several trips back to the drawing board, scientists finally came up with the right recipe for a glue that works on a specialized class of ceramic materials

By Susan Dieterle

The extreme stickiness of a new glue for ceramic composite materials is nothing compared to the stick-to-itiveness of its inventors.

The group of Ames Laboratory researchers went back to the drawing board repeatedly while trying to develop a glue that would hold together parts made of a rugged class of ceramic materials known as silicon carbide composites.

Undeterred by the setbacks, they now have an easy-to-use glue that produces strong joints without the expensive, high-temperature curing process associated with other types of ceramic glues. They hope that this simple, effective joining method will prompt industry to make greater use of ceramic composites in high-temperature settings.

The composites have properties that, in some applications, make them superior to steel and superalloys because they can withstand higher temperatures, do not melt and are less susceptible to corrosion. That makes them ideal candidates for such products as industrial furnace fans and turbines in hot, corrosive environments.

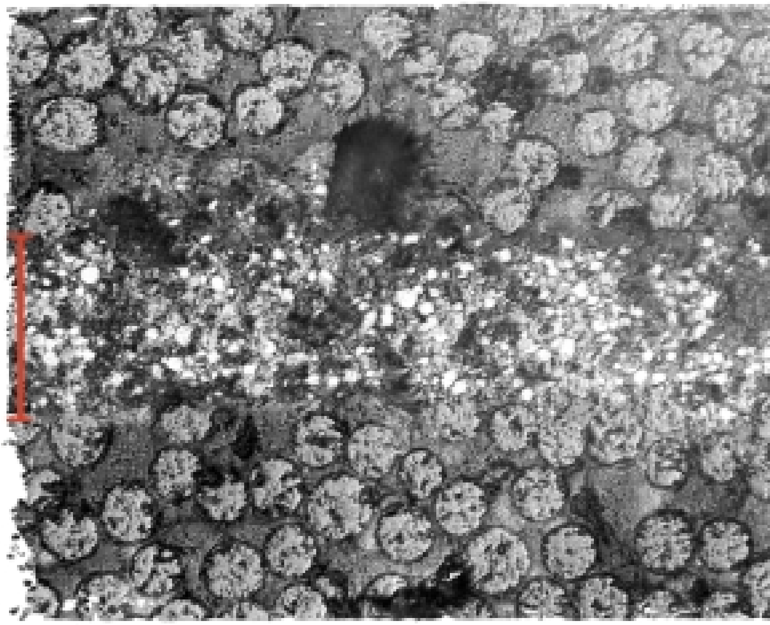
"If we had turbine engines that allowed fuel to burn hotter and industrial furnaces that could operate at uniform, constant temperatures, we would increase their efficiency immensely," says Iver Anderson, director of the Lab's Metallurgy and Ceramics Program. "Ceramic composites could make that possible and would result in a substantial reduction in energy costs for many industries."

Brittle material

Silicon carbide itself is a brittle material that lacks the strength needed for load-bearing applications. So some industries have turned to a composite in which silicon carbide fibers are woven together like a mat and then encased in a silicon carbide matrix. Just as steel rebar strengthens concrete, the fibers strengthen the matrix material. Silicon carbide composites are considered the most developed of the various types of ceramic composite materials and are closest to large-scale commercialization.

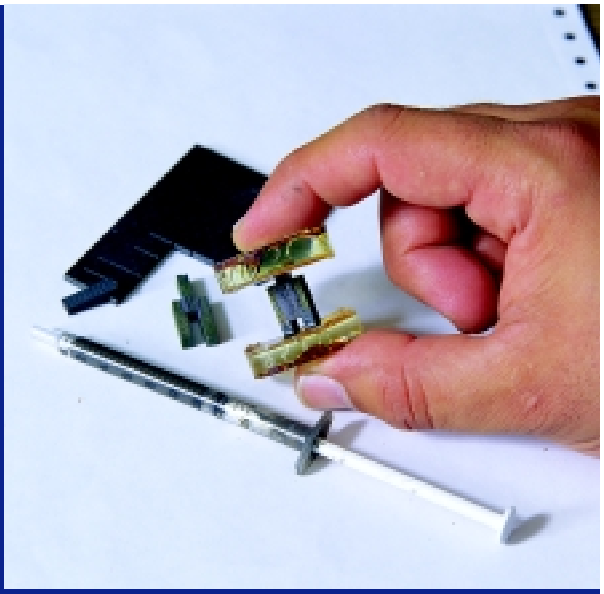
But there has always been a drawback associated with their use – the lack of a satisfactory way to join parts made of the material. "If you want to make a fan, you have to join blades to a hub and a hub to a shaft," Anderson says. "And nobody had a convenient, robust method for doing that."

The problem has been difficult to solve because, although silicon carbide itself can withstand temperatures of up to 2000 C (3600 F), the fibers in the composite material begin to degrade at 1200 C (2200 F). Traditional ceramic-joining methods don't work well because they involve curing the joints at temperatures of at least 1600 C (2900 F), usually in furnaces with inert atmospheres.



A micrograph shows the joint made between two pieces of silicon carbide composite. The joint area runs through the middle of the image, as denoted by the red line to the left.

Three years ago, the Department of Energy asked its scientists to come up with possible solutions to the joining problem. Ames Lab Director Tom Barton, whose background is in materials chemistry, participated in phone conferences regarding the issue and then paired up Anderson and materials chemist Sina Maghsoodi to attend a Salt Lake City workshop where it would be discussed.



The small platforms consist of three pieces of silicon carbide composites that were glued together, with plastic discs at the ends. Seven such platforms supported the weight of the three researchers in the photo at left.

Airport chat

During a long layover in Denver on the way back, the two scientists came up with a promising concept. What if they used Maghsoodi's silicon-bearing preceramic polymers along with Anderson's aluminum-silicon metal powder? The idea intrigued both the chemist and the metallurgist.

Preceramic polymers have several unique properties, Maghsoodi says. They are easily processed into a variety of shapes and forms and bind easily with metal alloy powders. This would give the glue adhesive properties in its "green state" while allowing it to convert to nanocrystalline ceramics at higher temperatures to form a strong joint, he says.

Anderson notes that the aluminum-silicon powder also had several purposes. "Aluminum readily forms a eutectic alloy with silicon; that is, a relatively ductile alloy that has a melting point lower than either aluminum or silicon alone," Anderson says. In addition to lowering the melting point of silicon, the aluminum in the powder would bond with the oxygen that enters the joint when the polymer was heated in air. The result, they believed, would be a joint filled with small islands of aluminum oxide, making it as strong or stronger than the parts themselves.

But getting their concept to work was simpler in theory than in actual practice.

First, a student couldn't make the long-chained polymers Maghsoodi wanted. The scientist ended up using intermediate-length polymers, called oligomers. Fortunately, the honeylike consistency of the oligomers proved preferable to the stiffness of long-chained polymers.

Then, the scientists determined that the alloy powder had too much aluminum and too little silicon, so they had to change the composition of the metal alloy powder.

"It wasn't working very well at

first," Maghsoodi remembers with a rueful laugh.

Getting it right

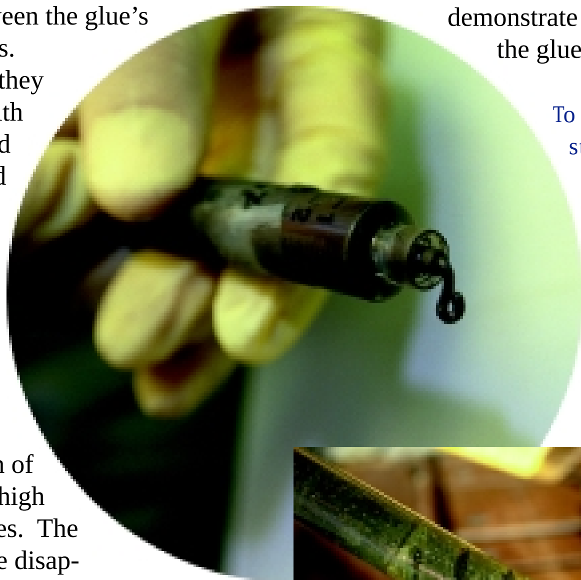
The two scientists, who continued gathering input and direction from Barton, added another member to the team – Mohammad Nosrati, a graduate student in ceramics. Nosrati began experiments to work out the kinks in the glue. He added a pinch of boron to the mixture, strengthening the bonds between the glue's components.

When they came up with a compound that worked well at room temperatures, they turned to ceramist Ozer Unal to measure the strength of the glue at high temperatures. The results were disappointing. "The joints had only 10 percent of their room-temperature strength at 1200 C," Maghsoodi says. "So, we had to go back to the drawing board and find a new formulation."

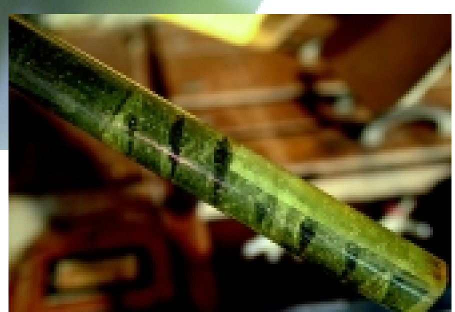
More adjustments followed. In the end, the group had a sticky, grayish paste that could be applied to the joint area with a syringe. From there, the joint can be heated and cured in a regular air atmosphere without clamping pressure. The glue makes a strong joint that can withstand pressures of up to 14,500 pounds per square inch, the equivalent of 100 megapascals.

The source for heating the joints can be something as simple and portable as a propane torch, as Nosrati discovered one evening. "Mohammad was working in a lab on campus late one night and needed to use a furnace that heats to 1500 C, but it wasn't working," Maghsoodi recounts. "He checked it and saw that the heating element was broken."

So, Nosrati smeared some of the glue on the broken ends of the heating element, heated it with a propane torch and then placed it back in the furnace. "It worked," Maghsoodi says, noting that he shows the heating element to interested companies to demonstrate how easily the glue can be used.



To keep the sticky glue from getting on their hands, researchers prefer to apply it with a syringe.



Maghsoodi uses this repaired heating element to demonstrate how easily the glue can be applied in the field.

Growing interest

The glue, which already has two patents with a third in the works, is attracting attention from companies throughout the world. One of those is Amercom, A Synterials Company, a Virginia-based enterprise that conducts research and development work on ceramic-matrix and metal-matrix materials.

Bill Bustamante, Amercom's vice president of business development, is interested in the glue largely because it is air-stable and can be easily used in the field. "That's especially attractive for some of the military applications and for the aerospace industry," he says.

Some of the ceramic composite materials are used in the hot sections of gas turbine engines, Bustamante says. Because the engines are big enough to walk around in, the ceramic parts can be damaged through a worker's carelessness. "The possibility of using this glue to patch or repair a damaged component is very, very attractive," he says.

Maghsoodi has also been approached by companies wanting to know if the glue can be adapted for use with materials other than silicon carbide composites. "We're working with them on small projects to try to help them solve their problems," he says. "So what started as a project for DOE has now increased 100 times through involvement with companies trying to solve their own problems."

The level of interest in the glue makes the scientists glad that, despite the initial setbacks, they stuck with the work. ✤

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The Right Stuff

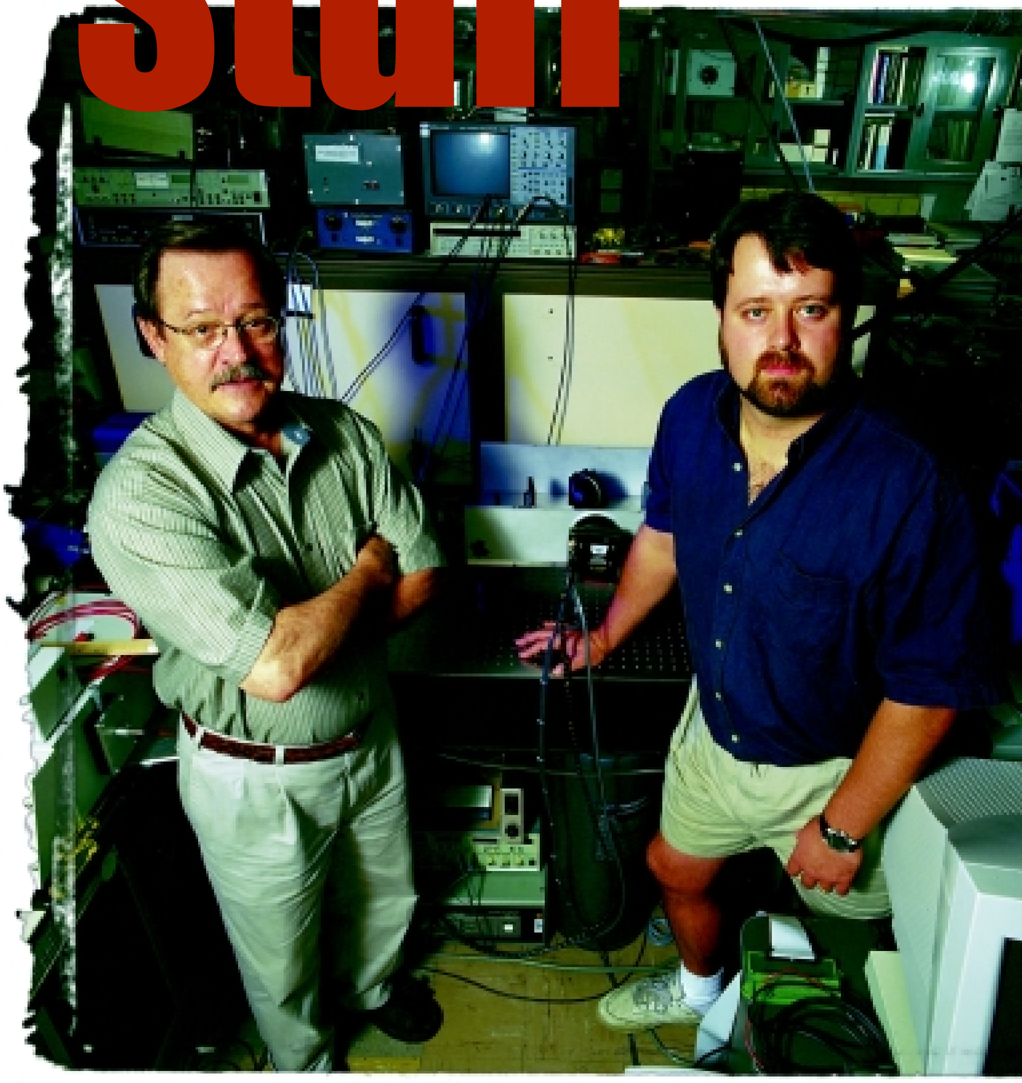
by Steve Karsjen

This was no ordinary bake-off. There were no apron-clad chefs standing around taste testing entries in the chocolate chip cookie contest. In this bake-off, as it was affectionately called by participants, the "recipe" being judged was for the best continuous emission monitor, or CEM, and the prize was the enviable status of being judged the company with the best the CEM industry had to offer.

No small distinction, this honor would not only carry with it bragging rights but an opportunity to impact the way mixed wastes are monitored during the incineration process. Mixed wastes are those containing both hazardous and radioactive constituents. Disposing of mixed waste is an enormous problem for government agencies like the Department of Energy. “It’s estimated the United States has in excess of 165,000 cubic meters of mixed waste,” says Bill Haas, a senior scientist at Ames Laboratory and a technical consultant to DOE’s Characterization, Monitoring, and Sensor Technology Crosscutting Program, the program that along with the Environmental Protection Agency organized the bake-off. “That translates into approximately 800,000 55-gallon barrels of waste needing to be destroyed.”

Mixed waste can be anything from tubing to hoses to paper products. Its common denominator is that the waste is hazardous and radioactive, mostly low-level and mostly the byproduct of government research or weapons production. Currently, the only EPA-approved process for treatment of mixed waste is incineration. As the largest operator of mixed waste incinerators nationwide – with two sites at Idaho National Engineering and Environmental Laboratory, one at the Savannah River Site and another at Oak Ridge National Laboratory – it’s easy to see why the DOE would place importance on finding a safe way to dispose of mixed waste.

Key to operating a successful mixed-waste incinerator is the ability to monitor the materials, or feedstock, that are fed into the incinerator in order to ensure the heavy metal emissions coming out of the incinerator’s smokestack are within



Bill Haas (left) and David Baldwin stand in front of the mercury CEM. Their collaboration helped produce the spark that got the mercury CEM to the development stage.

limits set by the EPA. Incinerator operators can monitor the feedstock two ways: by taking samples and sending them to a laboratory for analysis, which is laborious and time consuming, or by performing periodic trial burns every three to five years to verify that the facility meets EPA emission standards.

During a trial burn, the incinerator is run under a range of conditions, and measurements are made based on EPA standards. “As long as you’re able to operate with emissions that are less than what the regulation says, you’re OK,” says Haas. “However, three to five years is a long time between measurements.” The beauty of CEMs, he says, is their “continuous” operation. “In fact, if everything works out as planned and CEMs are a mandatory piece of hardware on every incinerator, incinerators will not be able to burn waste if the CEM is not working," notes Haas. "What this will provide is a much higher level of protection to the neighbors related to the air they're going to be breathing – air that's not been assaulted, even temporarily, above emission limits."

To determine the state-of-the-art in CEM development, Haas and other partners on the DOE/EPA bake-off team conducted a test in September 1997 at the Rotary Kiln Incinerator Simulator at EPA's National Risk Management Research Laboratory in Research Triangle Park, N.C. Results of the test were expected to help the EPA establish standards for smoke-stack emissions of six toxic metals – arsenic, beryllium, cadmium, chromium, lead and mercury – under the draft EPA Maximum Achievable Control Technology (MACT) rules for hazardous waste combustors.

During the demonstration, seven multimetal CEMs at various stages of development were tested, and results were compared to EPA's reference method for metals emission measurements. "A number of characteristics were taken into consideration in the test, such as sensitivity, speed and data quality, but the most important performance issue was whether the CEMs could quantitatively measure all six metals," says Haas. In the test, the six metals were injected along with fly ash from a coal-fired utility boiler into the afterburner of the EPA's incinerator simulator, and the relative accuracy of each multimetal CEM was calculated. According to Haas, the results of the tests were "disappointing." None of the CEMs tested met performance specifications for sensitivity and data quality in EPA's draft MACT rule for hazardous waste incinerators. Only one of the CEMs tested was able to measure all six metals at the concentrations tested, but the relative accuracy of that CEM varied between 35 and 100 percent, not 20 percent or less as required in the EPA performance specification.

So what did the test accomplish? "Our findings were significant because they helped prevent the EPA from putting in place incinerator emission standards as part of its MACT rules based on the abilities of some monitors that weren't up to standards," says Haas. "Ultimately, we're not there yet in terms of satisfactory CEMs, but through our trials we're moving the process along by providing data from which regulators can make reasonable judgements about standards."

Developing a mercury CEM

The North Carolina test did not go unnoticed by David Baldwin, Ames Lab chemist and program director for the Lab's Environmental and Protection Sciences Program. Baldwin had provided a prototype high-resolution optical spectrometer as part of an inductively coupled plasma-atomic emission system for use by one of the groups involved in the test. Following completion of the tests, Baldwin and Haas began talking about the need to develop an effective CEM specifically for mercury.

Why mercury? "Mercury is near the top of the list of elements that EPA wants much better knowledge and control of," says Haas. "Mercury has a much greater importance than the other five elements simply because it's more common, more easily volatilized and has many pathways to human exposure."

An ordinary pen lamp is a key piece of equipment in the mercury CEM.

Baldwin adds, "Mercury is a huge problem throughout the DOE." Measuring mercury in the waste feed is costly and difficult, especially for heterogeneous waste. Representative sampling is nearly impossible, and mercury is notorious for not being evenly distributed in the waste. Add to that the toxicity of mercury, which increases the risk of personnel being exposed to hazardous chemicals, and you have a major challenge facing the DOE. "DOE is laden with tons of mercury that was used as part of the process to produce uranium at Oak Ridge National Lab," Baldwin says. "In order to decontaminate and decommission places like that, you're going to have to be able to monitor mercury to minimize exposure and danger."

Fortunately, elemental analysis is an area that has been at the forefront of Baldwin's work at Ames Lab, going back several years to when he and his colleagues used an inductively coupled plasma-atomic emission spectrometer to conduct on-site uranium analysis of soil at DOE facilities like the Fernald Plant in Ohio. When that work dried up due to a DOE shift in emphasis from on-site characterization and treatment of contaminated soil to bulk removal and transport to disposal facilities, Baldwin turned his attention to developing systems to support a new DOE thrust, which was analyzing air emissions resulting from incineration of mixed wastes.

"In order to do this, we needed to be able to draw material from air and then be able to analyze it with a smaller ICP than the one-ton spectrometer we had used for soil characterization," says Baldwin, who set about the task of developing a spectrometer system based on acousto-optic tunable filter and echelle grating technologies that would be smaller and provide higher resolution than past spectrometers. At the same time, Baldwin looked at combining this system with a portable air-plasma ICP-AES system, which was being developed at Mississippi State University, to provide the real-time sensitivity and resolution capabilities necessary to perform multi-element analysis in smoke-stacks.

"That's how we got to where we are – from dirt to building a high-resolution spectrometer that can be placed on a smokestack along with an air plasma and can measure emissions from those stacks," says Baldwin. From there it was a natural progression to Baldwin's current effort to develop a mercury monitor. "Since mercury was an area in which we

Ames Lab assistant chemist Dan Zamzow makes adjustments to the echelle spectrometer of the mercury CEM.

were doing exploratory work already, we decided to do experiments to see whether the instrument we built to monitor other metals could also be used as a mercury monitor," he says.

Going beyond conventional CEMs

The mercury CEM begins with an ordinary pen lamp, which shows the many colors that result from excited mercury. The color, or line, of importance to the scientists is the 253 nanometer emission line because it is the one that's absorbed by mercury. "In order to measure the amount of mercury in a cell, you need to be able to measure how much absorption of light is occurring as a result of mercury vapor in the cell absorbing light," says Baldwin. The process sounds simple but it isn't because of two outside influences: interference and scattering.

For the mercury CEM to work effectively, it had to solve the interference and scattering problems. For example, Baldwin says when you burn hazardous materials in an incinerator, you often have molecular gases like sulfur dioxide (SO_2) in the stream. This presents a problem for current CEMs because SO_2 absorbs the same wavelength as mercury, which can throw off the measurement process. "If you're measuring mercury based on absorption of a single wavelength, you may also be seeing a lot of SO_2, which you might think was mercury, but it isn't," says Baldwin.

The mercury CEM also overcomes the second problem, scattering. When chemical or mixed waste is being burned in an incinerator, particulate matter, such as fly ash, is naturally present. "Particles, of course, scatter light which creates another type of interference, this one being scattering rather than absorption as in the case of SO_2," Baldwin says. "Essentially, we do a background correction for molecular interferences, such as SO_2, and for scattering."

Baldwin says a third problem with conventional CEMs is that they're not "hardened" for field use. Typically, the light has to go through the incinerator smokestack and out the other side and into a detector. "It could be 100 degrees with 100 percent humidity in this environment, and the likely result will be that those adverse conditions will cause the monitors to clog or fail to calibrate properly," Baldwin says. "They just don't handle the harsh environment well." Baldwin says what is needed is a high-resolution spectrometer that can measure multiple wavelengths simultaneously and is not prohibitive in size. The mercury CEM addresses both those issues by bringing a high-resolution echelle spectrometer into the equation. "With an echelle spectrometer, you can accomplish both tasks," he says.

The future is bright

Building on the collaborative efforts already existing between Baldwin and Haas, another Ames Lab scientist will soon become involved in helping test the mercury CEM. Glenn Norton, an Ames Lab associate chemist, has funding from DOE to look at commercial mercury analyzers for combustion gases. "He has quite an extensive testing system set up for this type of analysis," says Baldwin, who plans to use Norton's testing facilities and test the mercury CEM being built in the laboratory. "We need to concentrate on making a system that will survive these hostile environments, and then build a fieldable system."

Baldwin's goal is to be ready the next time DOE and EPA conduct another bake-off. "Bill has mentioned there's going to be another mercury test next fiscal year," says Baldwin. "We hope to be there with a system that meets the EPA's performance requirements." ✤

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PHOTONIC FRENZY

The push is on to produce 3-D structures that control light at visible wavelengths

by Saren Johnston

Photonic crystals don't occur naturally. If you want one, you have to make it – not an altogether easy job. But scientists are aggressively pursuing this task, manipulating photons to control electromagnetic radiation for potential applications that include optical switches, microfabricated lasers, waveguides, light-emitting diodes and telecommunications.

Building a photonic crystal requires creating a periodic structure from dielectric material – material that is an electrical insulator or in which an electric field can be perpetuated with a minimum loss in power. Scientists must arrange the photonic material in a lattice structure that repeats itself identically and at regular intervals. If the assemblage is precisely made, the resulting crystal can have a photonic bandgap, a range of forbidden frequencies within which a specific wavelength is blocked, and light is reflected.

With the exception of the bandgap, a properly constructed photonic crystal will transmit wavelengths of light up and down the electromagnetic spectrum. Now scientists can predetermine the bandgap by engineering the lattice spacing of the photonic material to match the wavelengths they wish to block. This fortunate circumstance has given them the ability to control and manipulate light, sending it down assigned routes and around loops and bends.

Proving the gap

A good deal of the credit for light control belongs to an inspired group of Ames Laboratory researchers led by senior physicist Kai-Ming Ho. Ho knows his way around photonic crystals. In 1990, he and Costas Soukoulis, senior physicist, and Che-Ting Chan, a former Ames Lab physicist now at the University of Science and Technology in Hong Kong, theoretically demonstrated the existence of the first photonic bandgap crystal.

"While exploring a number of structures, we discovered that the diamond structure had a bonafide three-dimensional photonic band gap," says Ho. "We were just doing the calculations, so the structure existed only on the computer at that time."

However, it wouldn't be long before the diamond lattice structure that Ho and his fellow theorists developed through computer simulations would become a tangible object. Their design led to the construction of the first successful photonic crystal, which was built later in 1990 by Eli Yablonovitch, a scientist at Bellcore Labs in Red Bank, N.J.

The crystal Yablonovitch fabricated from the Ames Lab design had a bandgap in the microwave region of the electromagnetic spectrum and was produced by drilling a series of carefully positioned holes in a dielectric material. However, drilling holes small enough and precisely enough to position the diamond lattice bandgap in the infrared or visible range of the spectrum where more potential applications exist turned out to be extremely difficult.

After seeing the problem that plagued Yablonovitch, Ho, Soukoulis and Chan started looking for a different way to construct the diamond lattice structure. Ho came up with a variation of the structure that could be built in a layer-by-layer fashion. "The idea was that instead of trying to drill holes, we'd stack alumina rods in alternating layers that ran perpendicular to one another, and we'd position the rods in every layer halfway between the rods two layers away," says Ho. The researchers received a patent for their novel design, which was fabricated in 1992 by former Ames Lab associate physicist Ekmel Ozbay and Gary Tuttle, an Iowa State University associate professor of electrical and computer engineering.

From rods to wafers

Between 1992 and 1994, experimentalists Ozbay and Tuttle built a number of three-dimensional diamond lattice bandgap structures to work in the microwave and millimeter-wave regions.

"Then Ekmel came up with the idea of making the structures smaller," says Ho. "Building the crystal with alumina rods takes quite a bit of time because you have to lay out all the rods and glue them together, stick by stick. So the idea was to make use of wafer technology. Ekmel and Gary would fabricate a whole layer at a time on a wafer and then stack it. That approach was quite successful."

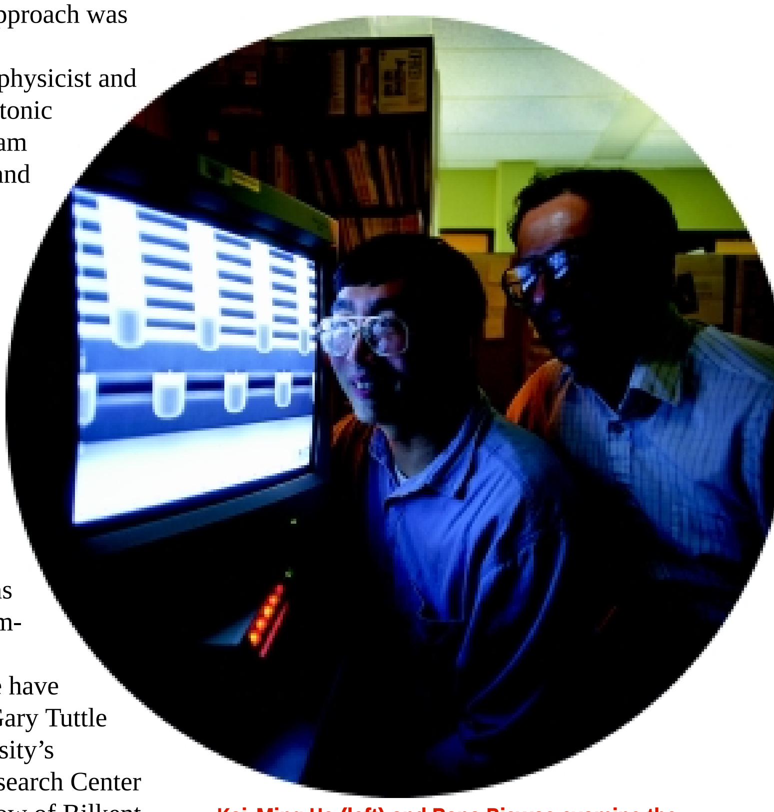
Rana Biswas, a physicist and a member of the photonic crystal theoretical team with Ho, Soukoulis and associate scientist Mihail Sigalas, explains the push to make smaller structures. "We wanted to make these structures work at optical wavelengths, or at least work at infrared wavelengths so there would be applications for lasers and telecommunications.

"Since 1994, we have been working with Gary Tuttle at Iowa State University's Microelectronics Research Center and Ekmel Ozbay, now of Bilkent University in Turkey, to make the structures smaller and smaller," Biswas continues. "We had some success with structures made from metal strips deposited on plastic, but that still didn't solve the problem of getting the bandgap into the optical region."

The collaboration begins

When Biswas was giving a talk at a 1997 American Physical Society meeting, it was not by chance that Shawn Lin from Sandia National Laboratories was in the audience. Lin had been corresponding with Ho and knew that Biswas would be at the APS meeting. "After the session, Shawn came up to me and said he'd like to try to make the Ames structure in his lab at Sandia," says Biswas. "They have a state-of-the-art processing lab at Sandia, probably one of the leading facilities anywhere in the world."

In 1998, Lin's team built the Ames group's structure using bars made of silicon. But instead of leaving an inner air space, they filled the region inside the bars with silicon dioxide to give the structure more strength. Where other groups had



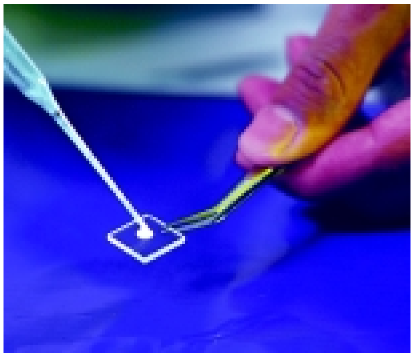
Kai-Ming Ho (left) and Rana Biswas examine the computer image of a photonic crystal built by researchers at Sandia National Laboratories using Ames Lab's original layered lattice design.

failed with this "back-filling" procedure, Sandia succeeded because of their capability to planarize, or flatten, each successive layer of the stacked crystal so well that, as Ho describes the process, "adding another layer was just like starting anew with the initial flat substrate on which the first layer of the structure was readily built." In the final step, the Sandia researchers immersed the whole structure in acid to etch away the silicon dioxide. The resulting three-dimensional diamond lattice structure had a bandgap of about 12 microns. "That's an important region because the carbon dioxide laser operates around 10 microns," Ho explains. "There are lots of interesting uses you can apply these structures to if you can hit the CO_2 laser wavelength."

Later in 1998, Lin's group succeeded in fabricating an even smaller version of Ames Lab's layered lattice design, bringing the bandgap into the region of 1.35-1.95 microns in the near-infrared, a significant achievement because the wavelength used for transmission of telecommunications by optical fibers is 1.5 microns. Photonic crystals with bandgaps at that wavelength could be used to improve the efficiency of optical switches for fiber optic communications.

The combined efforts of Ames Lab and Sandia researchers in designing and fabricating three-dimensional photonic crystals with bandgaps in the near-infrared range of the electromagnetic spectrum was a major step made even more noteworthy because the fabrication method is economical and lends itself to mass production. However, the silicon photonic crystal presents a problem in that it has an indirect bandgap, which lowers spontaneous emission. But Lin thinks he can overcome this problem with certain doping measures.

Further testimony to the success of the Ames structure is the fact that Susumu Noda and Noritsugu Yamamoto, researchers at Kyoto University in Japan, have also used Ho's design to fabricate a layered structure of gallium arsenide rods with a bandgap layer in the infrared. "They use a different technique involving wafer bonding and ion etching, and now they have a sample with eight layers," says Ho.



Colloidal crystals begin "life" on a glass substrate. A few drops of a slurry of titania suspension and polystyrene spheres is all it takes.

Colloidal crystals

While the Sandia researchers investigate ways to improve the emission rate for silicon photonic crystals, researchers on Ho's Ames Lab team are turning their attention from rods to spheres.

Kristen Constant, an Ames Lab associate and an ISU associate professor of materials science and engineering, came up with a novel ceramic technique for making photonic crystals from a nontoxic mixture of polystyrene microspheres and titania. She explains that polystyrene spheres can be purchased in dimensions less than 1 micron in size and can self-assemble into periodic, close-packed structures at optical wavelengths. "This makes them very attractive candidates for fabricating optical photonic crystals," she says.

Graduate student Ganesh Subramania knows just how good a mold the microspheres provide for fabricating photonic crystals. He's the person who makes the crystals, and he does it with surprisingly low-tech and inexpensive equipment.

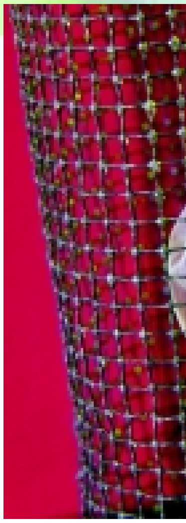
Subramania begins the crystal-construction process by spreading a few drops of a slurry of titania suspension and polystyrene spheres on a glass substrate. The sample spends 24 hours drying in a humidity chamber and is then taken to be "squished," as Constant refers to its fate, in a cold isostatic press. Five minutes of compression helps thin the sample and reduces stress cracks in the heat-treatment process that follows.

Slow baking is the final step. Subramania heats the sample to 520 C (968 F) over five hours, during which time the mold of polystyrene spheres is burned off, leaving behind air spheres in a titania matrix. Shiny regions are visibly apparent in the remaining photonic crystals. These regions have characteristic

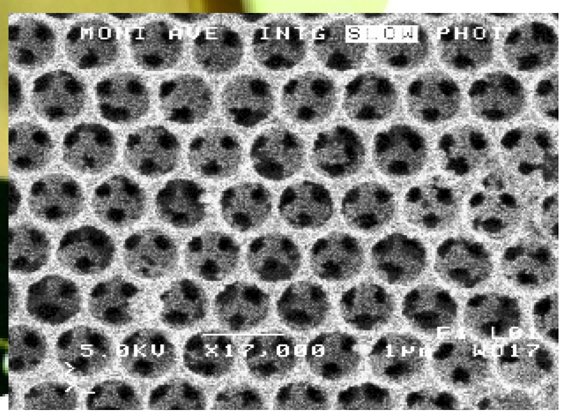
Ganesh Subramania places the colloidal crystal samples in a humidity chamber to dry for 24 hours.

colors that depend on the size of the polystyrene spheres used. The crystals made with smaller spheres (395 nanometers) have bright green regions, and those made with larger spheres (479 nanometers) are salmon-red in color.

"The color indicates that the structure that's present is what we're looking for – basically, a periodic structure at a certain length scale," says Constant. "If we don't see color, either the scale is wrong or the structure is wrong. It's nice because we can visually inspect some of these to see if they have the right structure. Then Ganesh does scanning electron microscopy on the good samples to see the detailed, periodic structure."



Following compression, the sample undergoes a five-hour baking process to burn off the mold of polystyrene spheres, leaving only air spaces in a titania matrix. Kristen Constant visually inspects the resulting colloidal crystal sample for the shiny regions of green or salmon-red that indicate a periodic structure is present.



This scanning electron microscope image of a photonic colloidal crystal shows the close packing of the air spheres.

The crystals are protected from the oily compression of the cold isostatic press by being placed inside a condom in a wire basket. Condoms are used instead of standard laboratory bags because they are cheaper and easier to obtain.

And the beat goes on

Constant hopes they will eventually be able to make large-area optical photonic crystals using the economical and reproducible ceramic technique. Such an achievement would greatly enhance the efforts of scientists to find out more about the control of light emission and propagation in these materials. But there is still work to be done.

Biswas and Sigalas note that although they have now achieved optical photonic crystals with colloidal systems, the structures don't have a fully three-dimensional bandgap like the ones the Sandia team has produced using Ho's original stacked crystal design. On the other hand, the Sandia team is still trying to reach the optical region.

There's plenty of work to be done, but there's also plenty of work that's come out of the photonic crystal research to date. And Biswas says both the theorists and the experimentalists are involved in efforts to expand that work. "One of the next steps we have to focus on is finding new uses for all the research that is coming out," says Biswas. "We also want to partner with industry, and we've had some good feelers." ✤

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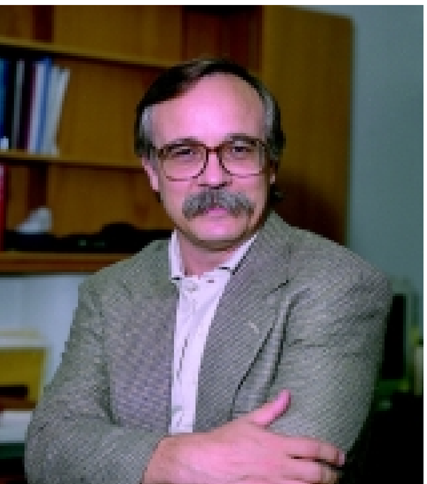
IT TAKES A TEAM

DOE Labs Unite to Advance Computational Materials Science

Ames Laboratory Deputy Director Bruce Harmon recognizes the key role computational modeling has to play in the design, processing and production of better materials with more desirable properties. To advance that effort, Harmon, also the director of the Lab's Condensed Matter Physics Program, helped create and now serves as one of the coordinators for the new Computational Materials Sciences Network.


Bruce Harmon

CMSN is funded through DOE's Basic Energy Sciences Office and came about as a result of interactions between Harmon and Iran Thomas, director of the BES Division of Materials Sciences, as well as the other CMSN coordinators: Chuck Henager of Pacific Northwest Laboratories, Malcolm Stocks of Oak Ridge National Laboratory and Ellen Stechel of Ford Motor Co.

The coordinators planned a series of CMSN workshops that brought together scientists from DOE labs, academia, industry and other government labs to formulate challenging materials science projects that could best be pursued through broad cooperative efforts. Seven broad subject categories were identified:

- Polymers at Interfaces
- Understanding Ductile and Brittle Behavior in Fracture
- Excited-state Electronic Structure and Response Functions
- Microstructural Evolution Based on Fundamental Interfacial Properties
- Bridging Basic and Applied Science in Magnetic Materials
- Oxidation
- Microstructural Effects on the Mechanics of Materials

"CMSN focuses on relevant, interesting and important science," says Harmon. "Larger, more complex problems require interdisciplinary teams, and CMSN provides a means for the DOE materials science community to work together where there's mutual interest in solving significant problems."

Access CMSN at:
http://cmpweb109.ameslab.gov/cmp/ccms.

IBM Grant Gives Huge Boost to Cluster Computing

When IBM officials visited Ames Laboratory and Iowa State University last April, they brought their checkbook with them – figuratively speaking. The visit was to formally announce the company's selection of ISU's chemistry department as the recipient of a Shared University Research grant.

The chemistry department and Ames Lab's Scalable Computing Lab and Condensed Matter Physics Program all benefit from the $665,000 SUR grant that provided 15 dual-processor, top-of-the-line IBM Power3 RS/6000 43P Model 260 workstations. Housed in the SCL where one of the primary objectives is to develop new methods for doing scalable computing, the workstations are helping to advance cluster computing.


IBM workstations are helping advance cluster computing research at Ames Laboratory.

Cluster computing involves networking groups of high-performance workstations to create clusters that operate at supercomputer speed and at a very economical cost.

"The SUR grant is highly competitive," said Mark Gordon, director of Ames Lab's Applied Mathematics and Computational Sciences Program and an ISU distinguished professor of chemistry. "The fact that we received it says that Ames Lab's and ISU's combined expertise is very attractive to IBM, and that they see great promise in our collaborative efforts."

Jeff VerHeul, vice president of Server and Workstation Development, IBM Server Group, said the SUR grant allows IBM, Ames Lab and ISU to work closely to explore the performance of clustered systems. "All three organizations will benefit from the discovery and shared learning currently underway," said VerHeul. "Professor Gordon's team has built a world-class learning laboratory using this new technology to solve real-world scientific problems."

Dan Barnard (foreground) and Jim Foley use acoustic harmonic generation techniques on a single crystal of copper that sits in the middle of the three-tiered apparatus.

Grappling With the Hook

By Susan Dieterle

Two years ago, Ames Lab assistant scientist Dan Barnard uncovered a scientific riddle that has yet to be solved.

Barnard and Otto Buck, former director of the Metallurgy and Ceramics Program, were conducting experiments with acoustic harmonic generation, a technique in which ultrasound frequencies provide information about a material's strength and other characteristics. As Barnard plotted the data from his experiments, he noticed a pronounced downward curve as the amplitude of the frequency decreased. He showed the graph to Buck, who asked in his thick, German accent, "Vat's dat hook?"

Thus was born "the hook" – and the controversy about whether it even exists.

As Barnard and other scientists have grappled with what the hook means, two schools of thought have emerged. One side thinks the sensitive testing equipment is picking up noise from the instrumentation itself, and that the hook doesn't really exist. The other side believes the hook may indicate something new about the microstructure of the materials being tested.

> **"It's always fun to be one of the first to find something new."**
> *— Dan Barnard*

Which camp is Barnard in? "I don't have any preference as far as the real source of the hook," he says. "I'd just like to know what's causing it."

The debate is difficult to resolve because researchers are still trying to understand acoustic harmonic generation. The technique comes from the realm of nondestructive evaluation in which the reliability of materials is studied without the use of invasive procedures. Ultrasonic testing, which involves transmitting high-frequency sound waves into a material to detect imperfections and microstructural changes, is a standard evaluation method. It is often used in a linear fashion, which involves pumping sound energy into one end of a material and then measuring how much energy comes out the other end.

But there is a growing interest in nonlinear acoustic techniques like harmonic generation, Barnard says. While the approach is similar, scientists also measure the faint frequencies emanating from the microstructure itself at double, triple and even quadruple the rate of the original frequency.

"For example, we put in energy at a fundamental frequency of 10 megahertz and then we look at the energy in the harmonics, usually at 20 and 30 megahertz," Barnard says, noting that the harmonics are usually 1/100th or 1/1,000th the strength of the fundamental frequency.

Cracks, dislocations and fatigue in the material produce the second harmonics, but no one fully understands how or why. "We don't have a complete understanding of the sources of harmonic generation and we don't know how they relate to each other," Barnard says. "We want to find out how each source affects harmonic generation, and how they affect it when they're combined."

What they do know is that harmonic generation is more sensitive than linear methods and can detect cracks that aren't visible – even through the most powerful optical microscope. "There are limitations on what kind of information you can get out of the materials with linear acoustic techniques," Barnard says. "Harmonic generation is a lot more sensitive to very fine changes that can occur at the microstructural level and even at the atomic level."

That kind of information could be crucial in settings such as power plants, where high temperatures and radiation can make materials brittle. "You don't ever want a brittle material when you have lots of power or pressure or temperature behind it," Barnard says. "Theoretically, they could use harmonic generation to determine the state of a material and know when to retire it and put in new material."

First, though, scientists need a better understanding of harmonic generation, and they need to adapt the technique for use in the field. Currently, the procedure requires painstaking, time-consuming measurements that wouldn't be possible in industrial settings (*see sidebar next page*).

The experiments that Barnard and Buck began two years ago were part of a collaboration with Oak Ridge National Laboratory to establish a deeper understanding of the phenomenon by measuring samples of copper-aluminum alloys.

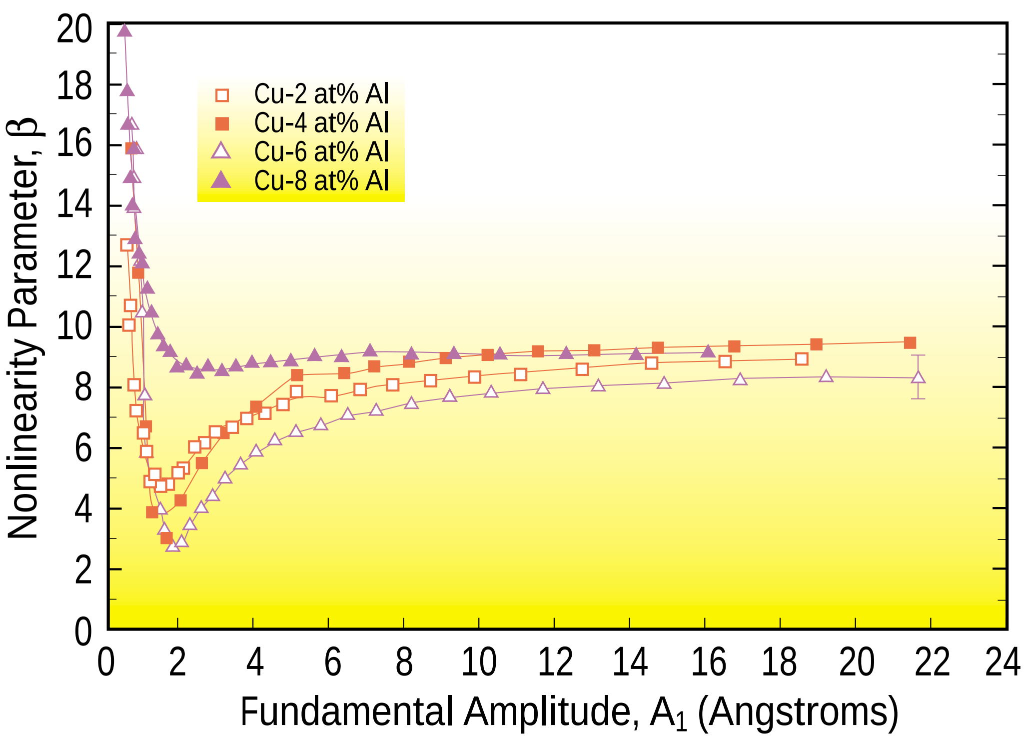
Theory predicted that the data from the experiments would move in a straight line until reaching the "noise floor," or the level of background noise, which would then cause the data to shoot upward. That's why the emergence of the downward hook at low amplitudes was so puzzling.

"We'd never seen it go down and then go back up, and we couldn't think of any experimental reason why it would do that," Barnard says, adding that he has also seen the hook with samples of pure aluminum. In his tests with fused silica, however, there was no hook.

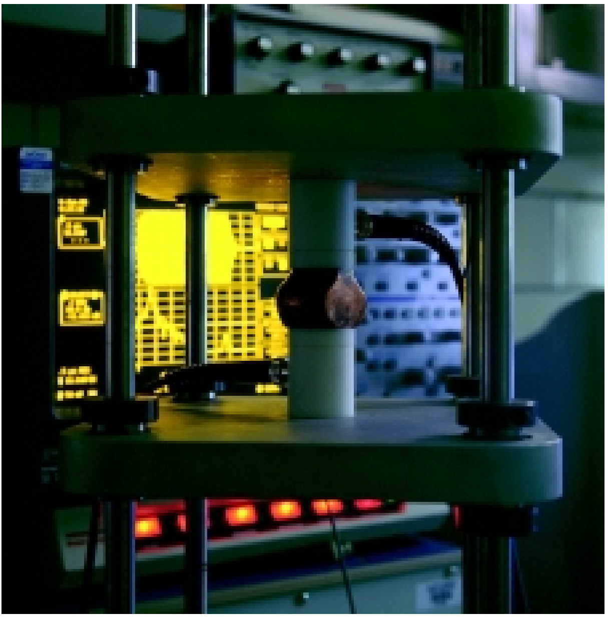
So far, Barnard and others have been unable to provide a definitive answer about the nature of the hook. Scientists at Oak Ridge produced an equation in support of their belief that the hook came from instrument noise, but Barnard says their equation doesn't fit all of the data.

Also, researchers at NASA's Langley Research Center have found curved deviations – some upward, some downward – at low amplitudes in aluminum alloys. John Cantrell, senior materials physicist in the Nondestructive Evaluation Sciences Branch at Langley, says that much of the curvature in his data appears to be linked to instrument noise. When corrected for that factor, the curves disappear – almost.

"If we go down to sufficiently low amplitudes, we still see some variation," Cantrell says. "But we're not sure whether that's due to an instrument offset or whether that's real."



The graphic illustrates the emergence of the downward "hook" at low amplitudes in tests involving four copper-aluminum alloys. Barnard is trying to determine whether the hook is caused by the material itself or by equipment noise.



As short pulses are shot into the copper crystal, the frequencies generated by the microstructure are shown on the oscilloscope behind the sample holder.

Cantrell, who uses different harmonic-generation techniques and equipment, says he plans to test the Ames Lab copper-aluminum samples to corroborate Barnard's findings.

"If it turns out that the hook, or at least part of the hook, is real and not due to instrumentation offset, that would give us some insight into the material physics going on at low acoustic amplitudes," Cantrell says. "But even if it isn't a physical process in the material, the very fact that there's an instrumentation situation has to be recognized. In some cases, you're going to be publishing data from low-amplitude tests, and you have to make sure you don't have instrument offset giving you false positive readings.

"Either way, Dan has made the nondestructive evaluation community aware of a very important situation," he adds.

Notes Jim Foley, who now serves as Barnard's Ames Lab group leader, "The hook is a result – not that we understand that result, but it's a result. Now we have to see what we can do to critically evaluate whether it's a real phenomena or an instrumental phenomena."

Since Buck's death in November 1997, Barnard has continued working with Oak Ridge to better understand the nature of harmonic generation and its contributing sources. In addition, Ames Lab has received funding from the Department of Energy to research ways of adapting the technology for use in the field.

"I think this technique has a definite use in the real world," he says. "And there aren't a whole lot of other people doing this kind of research. It's always fun to be one of the first to find something new."

The Lab recently purchased a more sensitive amplifier system that Barnard hopes will give him new insights into harmonic generation. And it may also help him solve the perplexing riddle of the hook. If it turns out to be a true material phenomenon, Barnard would like to call it the Otto Buck Hook in memory of his mentor.

"If it's an instrumental effect, then I'd like to know what it is and develop a correction for it," Barnard says. "But if it's a material effect, then that's something new, and we want to know what's going on."

And although much of Barnard's time is spent on other research, the hook remains firmly embedded in his thoughts. "When I have time, I go back and do some measurements to see if the hook is still there," he says with a smile. "And it's always there." ✚

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How does it work?

Using acoustic harmonic generation requires patience and precision.

Researchers like Dan Barnard hope to find ways of making the technique easier to use in the field. If they can do that, they'll give industries a powerful new tool that will provide the earliest possible information about cracks and fatigue within a material.

But for now, harmonic generation is used only in research laboratory settings. "Right now, the techniques take so much time that they could never really be applied to industry," Barnard says. "For some tests, we have to polish the surfaces of the material until they're mirror-smooth. On a factory floor, that's just not easy to do."

It usually takes Barnard at least three hours to set up and conduct a single series of tests. After putting the material in the sample holder, he must let it sit for an hour to make sure that it's absolutely still. Then he uses mineral oil to couple the receiving transducer to one side of the sample. Because the transducer is loaded on a spring and rests slightly against the sample to pick up the vibrations during the test, Barnard must let the sample sit for several minutes until the oil "squeezes out" to the thinnest possible layer before taking a calibration measurement. Then he must repeat the process on the other side of the sample with the transmitting transducer.

During the test, ultrasonic pulses are sent into one side of the 1- to 3-inch sample. The pulses must be short enough that they finish going in one side of the sample before they come out of the other. "We're talking about millionths of a second," Barnard says.

A complex network of amplifiers and recorders measures the original signal as it goes into and out of the sample, as well as the "second harmonic" frequency generated by the microstructure at double the original rate.

In addition, Barnard must take a variety of measurements from the equipment itself to ensure the greatest possible accuracy. "If one of those measurements is off, it can skew the results," he notes.

The process isn't easy, but Barnard thinks it's worth it. "Because of the high sensitivity of this technique, I think it has real promise," he says.

Barton Will Help Chart Course for National Laboratory Organization

Ames Lab director Tom Barton has been appointed to the National Advisors Group for the Federal Laboratory Consortium.

The FLC is a quasi-governmental organization involving 711 federal laboratories. Its primary focus is moving laboratory research and technology into the marketplace. The National Advisors Group is a select panel of approximately 15 people who provide guidance to the FLC in pursuing its technology-transfer goals and making the organization more effective. The group includes senior agency officials, laboratory administrators and industry leaders.

"This is a challenging time for federal laboratories, and I look forward to working closely with the FLC as it helps its members strengthen ties with industry," Barton said.

Barton became director of the Ames Laboratory in 1988. Since April 1998, he has also served as director of the Institute for Physical Research and Technology, a consortium of 11 research and technology-transfer centers on the Iowa State University campus. Ames Lab is the largest of the IPRT centers. Barton is also an ISU distinguished professor of chemistry.

Tom Barton

Lab Scientists Apply Analytical Expertise to Forensic Techniques

Ames Laboratory scientists are hoping to convert some of their analytical expertise into techniques that may help local and national forensic investigators.

Representatives from both the FBI Laboratories and the Iowa Criminalistics Laboratory are searching for rapid, more accurate methods of assessing crime-scene evidence. Forensic investigators often use various types of chemical and materials analysis as they search for clues from evidence such as firearm residues, fingerprints and fragments left by explosive devices.

Because Ames Lab scientists use some of the latest analytical techniques available in these fields, law-enforcement agencies are interested in finding ways of adapting these techniques for use in forensic investigations.

Five research projects for the FBI Laboratories – the nation's largest forensic laboratory – are already underway. Along with counterparts from the Iowa State University campus, Ames Lab scientists are investigating techniques that would enable criminal investigators to:

- Restore stamped or engraved serial numbers that had been obliterated from guns and stolen automobiles
- Characterize various types of explosive devices and understand blast effects upon detonation
- Use analytical tools to determine the sources of metal fragments found at crime scenes
- Develop criteria for comparing pieces of trace evidence
- Access an electronic database of facilities and veterinarians throughout the country who could rapidly determine the presence of animal pathogens that could be used as biological warfare agents

Meanwhile, Lab officials are still discussing possible research projects with the Iowa Criminalistics Laboratory, which serves local and state law-enforcement agencies in Iowa.

A New Twist for Tracking Trace Elements

Using his innovative technique that combines ICP-MS with chromatography, Sam Houk can identify ultratrace metals in biological systems at parts-per-trillion levels. As his office decor suggests, the scientist is also a dedicated student of history, and he relishes the opportunity to take an interested "pupil" back in time.

A novel use of an old method is speeding identification of trace metals in biological systems

by Saren Johnston

For good or for bad – the jury is still out – tiny amounts of various metals in our bodies give up their freedom for a chance to cozy up to our DNA. In fact, trace metals can get pretty friendly with all kinds of biological systems, abandoning their freedom in the ecosphere to attach and bind themselves to proteins and other biomolecules. And whether this circumstance is good or bad depends on the element.

"The basic phenomena responsible for the toxicity of trace elements are complex," says Sam Houk, senior chemist. "Some elements like cadmium and lead can replace essential elements like copper and zinc in enzymes, thus interfering with the biological activity of the enzyme. Radioactive elements like uranium, thorium and other actinides are toxic chemically. They can also bind to DNA and cause mutations when they decay. Since the Department of Energy has large amounts of such elements in storage, monitoring their binding to proteins and DNA at very low concentrations is of great environmental importance."

Measuring the metal

Using a novel extension of inductively coupled plasma-mass spectrometry, a well-established analytical technique pioneered at

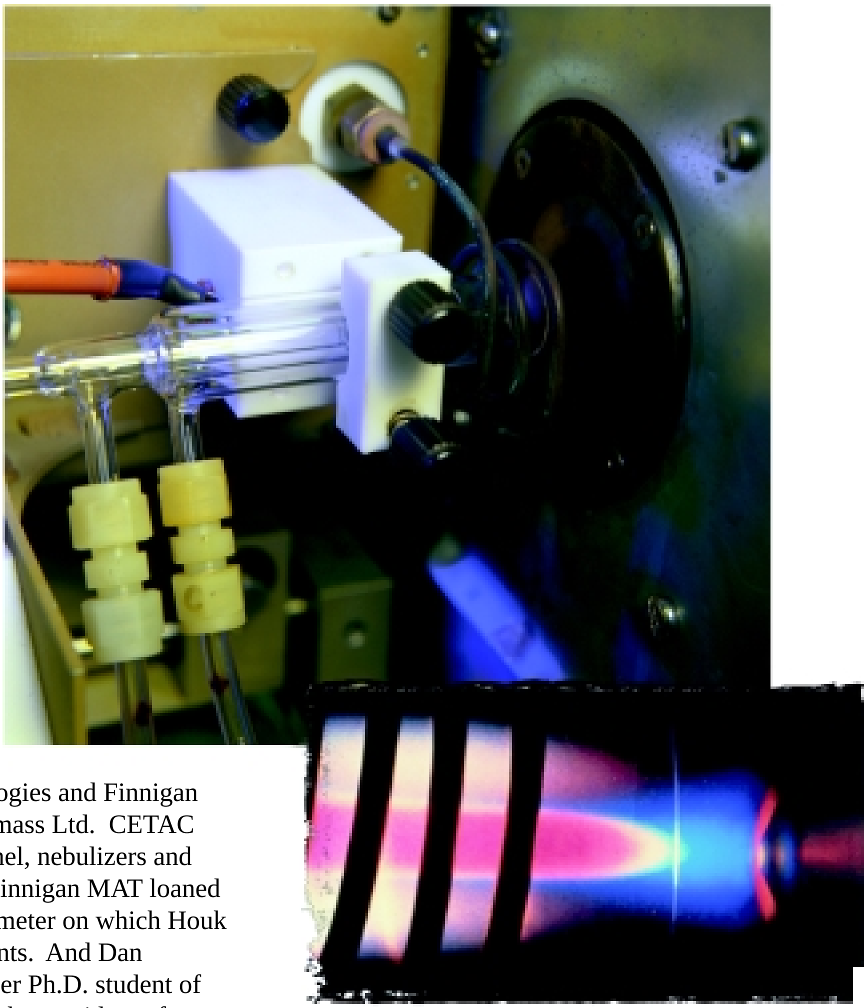
Ames Laboratory, Houk can determine the total amounts of ultratrace metals in biological specimens. His method is faster and more sensitive and selective than conventional schemes. Allowing measurements at parts-per-trillion levels, the ICP-MS technique relies on a special type of chromatographic separation called size exclusion chromatography in connection with a state-of-the-art magnetic sector mass spectrometer and an inductively coupled plasma – a very hot argon gas.

In Houk's new method, chromatographic separation of the compounds of interest provides chemical information, in particular the molecular weights of the proteins. Jin Wang, who recently received his Ph.D. with Houk and is now a postdoctoral fellow at Baylor College of Medicine in Houston, did much of the chromatography work. "It's his specialty," says Houk. "He did almost all of the experiments. He's good at both chromatography and ICP-MS."

The chromatography work is crucial because it reveals the percentage of a trace element in a sample that is bound to particular biomolecules. "If we just put the entire sample in the ICP, it would only measure the total amounts of the individual elements present," says Houk. "It would not tell us that, say, 90 percent of an element is in a particular molecular weight range, and 10 percent is in a different fraction."

Once the chromatography is completed, the sample is automatically injected into a nebulizer that produces a mist of fine droplets. The droplets dry into aerosol particles and pass into a hot argon inductively coupled plasma. In the ICP, they are converted to atomic ions that are measured by the mass spectrometer. The resulting measurements provide data that allow scientists to better comprehend the effects of unusual elements in biology and environmental sciences.

Houk is quick to point out that his development of the new ICP-MS method for trace metal identification was facilitated by interactions with instrument manufacturers Transgenomic CETAC Technologies and Finnigan MAT and Micromass Ltd. CETAC provided personnel, nebulizers and supplies, while Finnigan MAT loaned the mass spectrometer on which Houk ran his experiments. And Dan Wiederin, a former Ph.D. student of Houk's and now the president of Elemental Scientific in Omaha, has served as the primary collaborator on the research.



bottom photo courtesy of Varian Corporation

An open view of the inductively coupled plasma chamber (top photo) looks drab compared to the brilliant emission from the hot argon ICP.

Few limits

Houk's experiment is applicable to almost any element in practically any kind of biological system. "The main exceptions are the common elements that are always present: carbon, nitrogen, oxygen and hydrogen – we don't measure those," he says.

Just as there are few limits regarding the elements to which Houk can apply his methodology, there is also a seemingly endless array of potential applications. Work for the Department of Energy deals with determining the fate of radionuclides in the ecosphere, a major concern in the cleanup of nuclear facilities. The conversion of radioactive elements to atomic ions in the ICP considerably reduces the time required for measuring nuclides with long half-lives from that required by conventional techniques.

"There are also important medical applications," says Houk. "For example, there's a lot of interest in selenium compounds, which are being marketed as health foods and as preventive treatments for cancer and are touted to delay the onset of AIDS symptoms. But there have been few direct measurements of the role of selenium in this regard."

Agricultural applications could involve extracting proteins or other macromolecules from grains and measuring the binding of trace elements. Similarly, researchers could measure the binding of metal ions to environmental compounds, such as humic acids, large polymeric compounds with many carboxylate groups attached.

Houk says his experiment could

also be used to measure trace metals in synthetic polymers if they could be brought into solution. "There is some interest in this since the processing procedures for polymers usually involve exposing the material to metals," he explains. "There's a question about where the metals wind up in the final product."

More selectivity

Houk's experiment can accommodate just about any organic sample as long as it has an inorganic element in it. But now his goal is to improve selectivity in identifying the biological compounds. "Currently, all we get in this particular experiment is a kind of crude separation of compounds based on their molecular weight," he says. "A chromatographic fraction for an element could include many different proteins that contain that element. We want to separate those proteins more effectively and also develop mass spectrometric measurements that will be sensitive to the particular molecule. You see, we get information on the identity of the molecule only from the chromatography, not from the ICP. What the ICP does is give us an extremely sensitive and selective way to find unique inorganic elements."

Houk's experiment is applicable to almost any element in practically any kind of biological system.

To more precisely identify the proteins, Houk says they would probably start with size exclusion chromatography, collect a fraction of the liquid coming out of the chromatographic column and inject it into another kind of chromatographic column that would provide better resolution.

"We might also try capillary electrophoresis, which is an effective way to separate proteins. And we could use different variations of mass spectrometry, such as electrospray MS, which can measure molecular weights and also the sequences of amino acids but is much less sensitive to whether there are small levels of inorganic elements present. These are some of the things we have in mind for the future." ✤

Jin Wang observes the fine droplets of mist created when a sample is injected into the nebulizer following the chromatography process.

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Current research funded by:
DOE Office of Basic Energy Sciences

This ion gauge monitors the quality of the vacuum in the high-resolution vacuum ultraviolet laser system used to measure photoions and photoelectrons.

Basic Needs

"Whether it's basic research or applied research, they are both very useful – it's a matter of time scale, but basic research is always useful." — Cheuk Ng

By Saren Johnston

When chemists try to predict whether the chemicals they want to combine will create a reaction, they must first know if the reaction is energetically possible – if there's enough "oomph" in the atoms to make the reaction go.

Knowing the ionization and bond dissociation energies for the molecules under investigation is critical to accurately predicting reactions, such as electron transfer or oxidation-reduction processes. And Cheuk Ng, an Ames Laboratory senior chemist and an Iowa State University distinguished professor of chemistry, has helped many a chemist make some successful and more accurate predictions. His research in vacuum ultraviolet (VUV) chemistry has provided some of the most accurate ionization and bond dissociation energy data for many molecular species.

Ng considers himself fortunate because he has a dual "playground" in which to investigate molecular dissociation (separation into two or more fragments, creating atoms, ions or radicals) and ionization processes in the VUV range of the spectrum – photon energies in the 6-30 electron-volt region. There, in principle, any molecule can be ionized, a process in which a molecule or atom loses an electron, acquiring a net charge and making the transformation to an ion. To perform his experiments, Ng takes advantage of the third-generation synchrotron radiation source at the Chemical Dynamics Beam Line associated with the Advanced Light Source (ALS) at the Department of Energy's Lawrence Berkeley National Laboratory. Or, he may use the state-of-the-art high-resolution VUV laser apparatus at Ames Laboratory.

To be sure, Ng's research sounds weighty and complicated, but he would argue unequivocally that it is a fundamental thing of beauty. "When I look at the data and understand it, I feel unified with the universe and in harmony with nature," he says. "It's very basic research, but I'm excited about it because every day I learn something new. How much energy does it take to ionize or dissociate a specific molecule? This is a parameter that is fundamental to chemists. Once they know the energetics, they can use them to predict reaction rates. Those rates are needed for modeling problems in atmospheric, plasma and combustion chemistry."

The Berkeley playground

Ng's work at the Advanced Light Source centers around the high-resolution VUV facility he designed for the Chemical Dynamics Beam Line. Specialized instrumentation at the VUV facility allows him to make both photoionization and photoelectron measurements. The spectroscopy he performs provides information on the ionization and dissociation energetics of specific molecules of interest. Photoionization spectroscopy involves detecting the ion(s) produced when one or more electrons are removed from an atom or molecule by absorption of a VUV photon. Photoelectron spectroscopy offers a means of detecting the electron(s) liberated by photon-molecule interaction.

To design the VUV facility, a unique user facility that today serves chemical physics researchers throughout the world, Ng went back in time, in a manner of speaking. “I started to design our experimental station at Berkeley in the summer of 1993,” he says. “I went back to live in a graduate student dorm, and I worked like a graduate student. Everybody asked me which group I was working in at Berkeley, and I told them I had graduated years ago,” he says with a laugh, recalling his brief return to graduate school days.

In 1994, Ng sent graduate students Chia-Wei Hsu and Matt Evans to Berkeley to assemble the photoelectron/photoion apparatus he had designed for the experimental station. “These two students put the whole thing together. I only put together the mechanical design,” says Ng modestly. “In 1995, everything was up and working. Chia-Wei and Matt did all the performance checks for the apparatus, so whenever I traveled from Ames to visit the Berkeley experimental station, I only had to ask, ‘How are things going?’ I feel I’ve become a theoretical experimentalist,” Ng jokes, but with a certain measure of seriousness. “I design a scheme and ask the students to try it and report the results to me.”

"When I look at the data and understand it, I feel unified with the universe and in harmony with nature."

— Cheuk Ng

The VUV facility is considered the best in the world for doing ion chemistry. “The photon energy resolution and energy range we can cover is unprecedented,” says Ng. “Due to the development of several novel experimental schemes, the resolution we can achieve is at least 10 times better than what was available in previous synchrotron-based studies before the VUV facility existed.” Ng notes that they can measure the dissociation threshold

"We scientists live in an obscure corner that may not be related to daily life," says Ng, who often works day and night. "My research inspires me – that's not a feeling you can easily explain. You have to experience it. And as I get older, the teaching becomes more significant. Maybe that's more important than the research," he muses. "My son tells me, 'Daddy, you don't have enough sleep; go back to sleep.' But I cannot explain to him that I can't wait to wake up, see my students and renew."

and thus the bond dissociation energy of a molecular ion to the accuracy of less than 0.001 electron volts. "Many of the things we are doing now can be considered as breakthroughs in the field of VUV chemistry, and the facility coaxes us to do more," he says.

Eyes on the ions and radicals

Some of Ng's work includes looking at radicals, or unstable molecules, to explore the energetics of combustion species for the Department of Energy. "Combustion is almost impossible to understand because it's a radical chain reaction," he says. "When one radical is formed, it initiates another reaction, so there are many, many intermediate species that have not even been detected and for which the energetics and structures are not known."

An aspect of Ng's research important to plasma chemistry involves looking at the reactivities of ions and radicals, which depend on their internal energy states. The state of a molecule is related to how the electrons and nuclei arrange in the molecule. Because of the high resolution capability of the VUV facility, Ng is able to select a given state of a molecular ion and then react it and look at how the internal excitation influences chemical reactivity.

"A molecule can move around – that's what we call translational energy," says Ng. "A molecule can also vibrate – vibrational energy, and rotate – rotational energy. Electrons can also arrange differently with respect to the nuclei," he adds. "This arrangement gives rise to the specific electronic state of the molecule. So all of these things can influence the chemical reactivity of the molecular species. The study and ultimately the control of molecular reactivity are of great interest to chemists. So the VUV facility has opened up opportunities for future investigations in which scientists can really control the internal energy of a molecular ion to a very high degree. For VUV chemistry, I can confidently say that this facility is the best in the world. Many users have come here and told me the same thing. So it's better that they tell me than I say so," Ng notes with a smile.

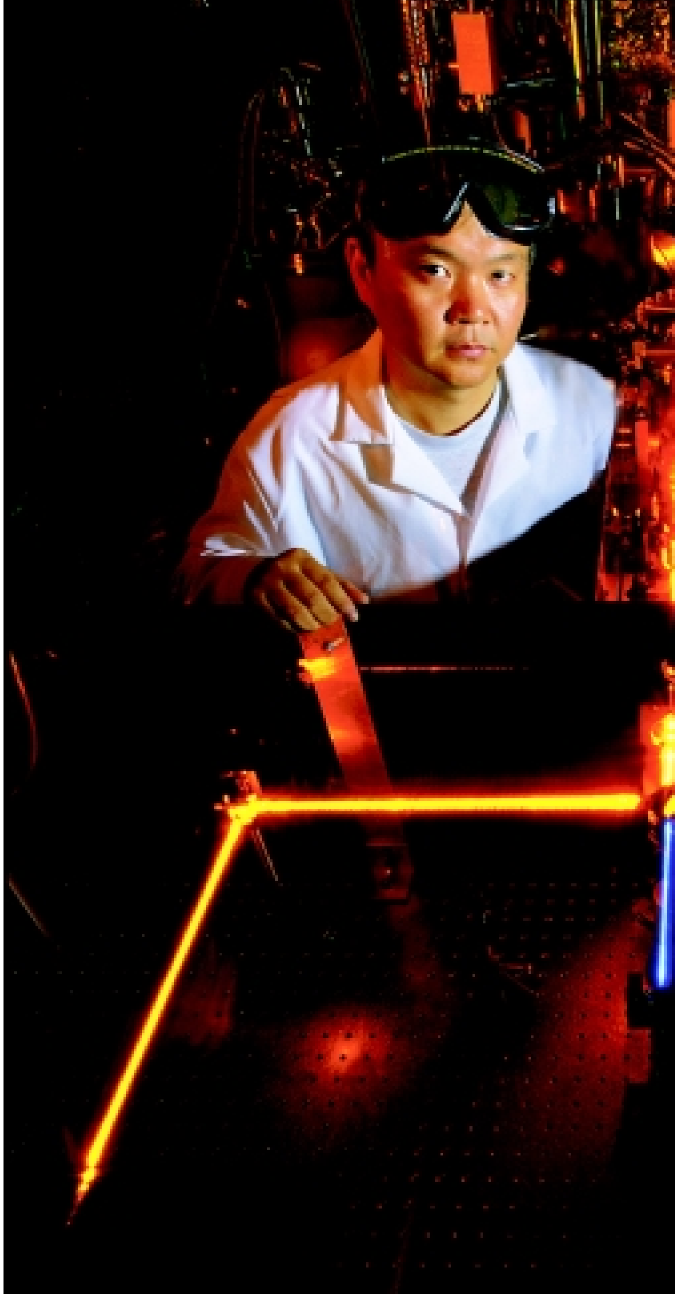
His research is also helping advance knowledge in the field of atmospheric chemistry. The ionosphere, the outermost shield of the earth's atmosphere, is ionized and dissociated by solar ultraviolet and VUV radiation. In order to understand and model the chain reactions that follow the ionization and dissociation processes and cause such phenomena as the aurora borealis, Ng says scientists must first measure the reaction rates and determine how the ions are initially formed. "A lot of research work is needed," he says with conviction.

The home turf

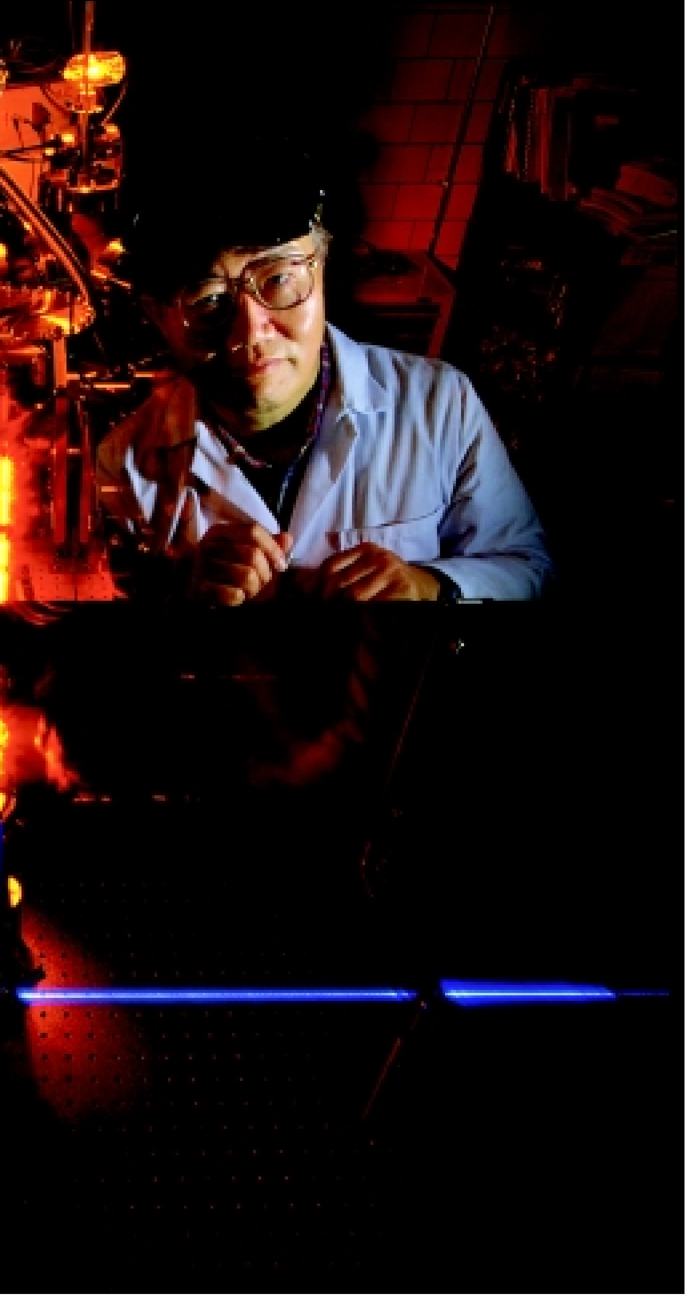
As remarkable as the VUV facility is, Ng can't spend all his time at the ALS. So, with the assistance of fellow chemist Chung-Lin Liao and postdoctoral fellow Yuxiang Mo, he also runs experiments on the high-resolution VUV laser instrumentation at Ames Laboratory.

"The VUV laser has an established resolution better than that we can achieve at the ALS, but it's very difficult to tune," says Ng. "For example, if you want to tune about .2 electron volts, it would take a few weeks for two students to work on it. At the ALS, we can tune through more than 10 times that range in one week, so the throughput or productivity comparison is no match at all. I don't think the laser can win in this case. The resolution is slightly worse at the ALS, but you don't lose that much chemistry and physics. It's really the ease of tunability that makes the synchrotron so attractive."

Ng explains that many of the techniques they have now developed for the ALS facility were previously laser-based. "Now that we have tapped into the synchrotron, I think the laser is gradually losing ground," he says. "But for optical resolution, the laser is still the best. Furthermore, some experiments are easier using VUV lasers."



This time-lapse photo shows Chung-Lin Liao (right) and Yuxing Mo at home in their Ames Lab research space where they use the high-resolution vacuum ultraviolet laser system to perform detailed ionization studies.



Ng and Liao have many ongoing projects in their Ames Lab research quarters. They are focusing on VUV mass spectrometry, VUV and ion-etching reactions, and photoelectron spectroscopy of radicals. "For the studies of radicals, the VUV laser is expected to do a better job than synchrotron radiation because most radicals can be prepared with high specificity by pulsed laser dissocia-tion," explains Ng. "This pulsed radical source matches the pulsed nature of the VUV laser system."

Ng on impress vs. inspire

Two students from Ng's group, Evans and Stephanie Stimson, finished their Ph.D. theses working at the VUV facility at the ALS, and Ng will soon be sending three or four more students to work there. "It's a tremendous educational instrument for our group," he says. "Many people in the world who are known to be experts in VUV chemistry come to the ALS, so these students have the opportunity to interact with them and learn from them, not just from me. Then, after spending four or five years working at the VUV facility, the students will already have connections with well-known scientists."

Ng says there is no better avenue for training students than basic research and that as he gets older the teaching part of his job becomes more significant. "I often have students ask me why they have to learn certain things," he says. "To me, it's very obvious – understanding is really the fulfillment in life. I tell my students, 'There are two ways to make you work hard – one is to impress and one is to inspire. To impress means you apply yourself – if I do this and work hard, I'll get a million bucks in maybe a year or so. Inspire means you don't talk about money – you work night and day. That's a higher level of learning.'

"If I work night and day on a project, it must be fun," says Ng. "Fun is actually the key to being inspired. I would like my students to be inspired. The most important mission to me is not to produce a commercial product but to train students."

No boundaries

As Ng's research matures, the molecules he investigates at the VUV facility are becoming more complex. "We've been working on small molecules because we have to test the techniques we develop," he says. "But we're moving on to more complicated molecules related to combustion.

"The VUV is a user facility, so my job is to convince people that this research is exciting," he adds. "It has been quite a venture in the last year. People come from the United States, Japan, Germany and France because the chemistry is completely different compared to working in a lab. Everybody has his or her own expertise, and I pick up many ideas from users when they come. So to me, the whole world is like a play-ground. I don't feel the boundaries anymore." ✤

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Scientists Nationally Recognized for Quasicrystal Research

A team of Ames Laboratory scientists has earned national recognition for its work with quasicrystals, relatively new materials with potential as highly effective coatings for automotive and mechanical parts and frying pans.

The group received the U.S. Department of Energy's 1998 Materials Sciences Award for "Outstanding Scientific Accomplishment in Materials Chemistry" for its work in understanding the surface properties of quasicrystals.

photo courtesy of P. C. Canfield and I. R. Fisher

This single-grain quasicrystal shows the pentagonal facets that are characteristic of quasicrystal materials.

The annual awards recognize scientists at DOE laboratories for their research accomplishments.

In the late 1980s, scientists discovered that quasicrystal materials were highly resistant to wear and corrosion, and that they had low coefficients of friction – highly desirable properties for coatings on mechanical and automotive parts.

What was lacking, though, was an understanding of why the materials exhibited these surface properties – a shortcoming that Ames Lab researchers wanted to address.

"Five years ago, there were almost no papers in the literature concerning surface properties of quasicrystals," said Pat Thiel, director of the Lab's Materials Chemistry Program. "The Ames group has begun to lay a foundation for understanding this topic and is now internationally recognized for its efforts."

In addition to Thiel, the group includes Dan Sordelet, James Anderegg, Matt Besser, Tamara Bloomer, Alan Goldman, Cynthia Jenks, Matt Kramer and Tom Lograsso. Other collaborators include Martin Gierer from Ludwig Maximilians University in Munich, Germany, and Michel Van Hove of DOE's Lawrence Berkeley National Laboratory.

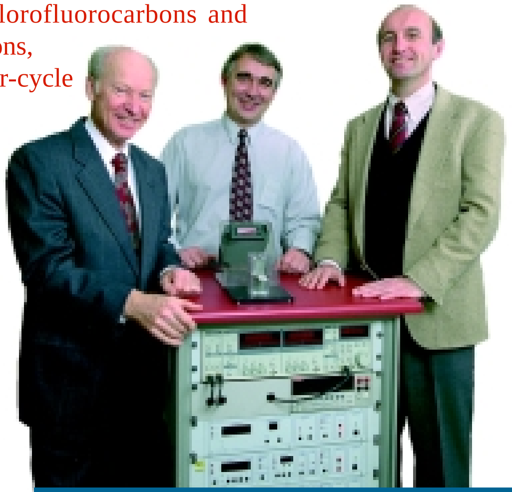
Work Begins on Prototype Magnetic Refrigeration Unit

Scientists at Ames Laboratory have begun working on a prototype cooling unit based on magnetic-refrigeration technology.

Researchers Karl Gschneidner Jr., Vitalij Pecharsky and David Jiles expect the prototype to demonstrate that magnetic refrigeration is a reliable source of cooling power, and is more energy-efficient and environmentally safe than the vapor-cycle systems now used in refrigerators and air conditioners. They will be working with Milwaukee-based Astronautics Corp. of America, the Lab's industrial partner in the project.

If successful, the prototype would be the first magnetic refrigerator capable of sustained operation and generating enough cooling power for commercial applications.

Magnetic refrigeration is based on the magnetocaloric effect – the ability of some materials to heat when magnetized and cool when removed from the magnetic field. Using these materials as refrigerants would provide an environmentally friendly alternative to the volatile liquid chemicals, such as chlorofluorocarbons and hydrochlorofluorocarbons, used in traditional vapor-cycle cooling systems.



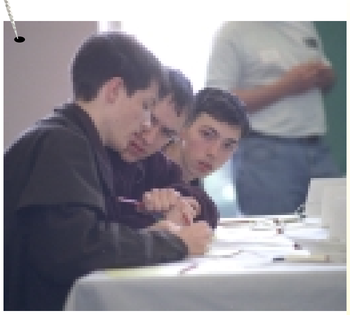
(from left) Ames Lab researchers Karl Gschneidner Jr., Vitalij Pecharsky and David Jiles

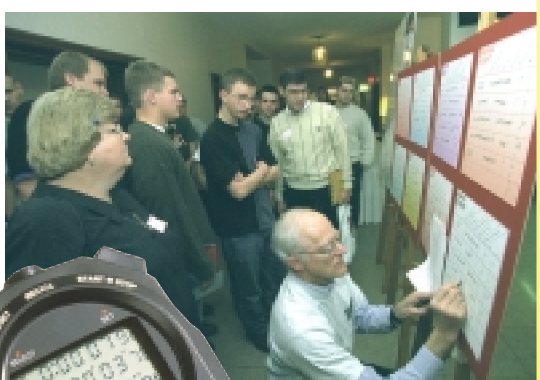
In April, Ames Laboratory and Astronautics signed an agreement to develop a rotary prototype magnetic-refrigeration unit. The U.S. Department of Energy will provide $750,000 in funding over the next three years toward the project, and Astronautics will provide a matching amount through in-kind contributions of personnel, research, services and facilities.

Ames Lab is concentrating on optimizing the refrigerant materials and developing a source for the magnetic field that is more convenient and cost-effective than superconducting magnets. Astronautics will design, build and test the prototype.

"When these pieces of the puzzle are properly put together, it will create a benchmark for all future developments of this new, emerging technology," Pecharsky said.

Ankeny High Trio Triumphs at 1999 Ames Lab/ISU Science Bowl





It was a battle of experience versus quantity in the championship match at this year's Ames Laboratory/ ISU Regional Science Bowl. And experience won out.

Ankeny High School defeated Pleasant Valley High School for the title, becoming the first three-member team to win the annual Ames Lab/ISU event. The competition drew 200 students who demonstrated their academic skills in answering questions from a broad range of science and math topics.

While most of the 40 participating schools had four or five students on their teams, Ankeny decided to field a team of only three players when a fourth student withdrew because of another commitment.

"These three students are seniors who have played together on Science Bowl teams since they were sophomores," said Ankeny coach Mark Maffett. "They decided they knew each other so well that they didn't want to add someone new."

Ankeny went on to participate in DOE's National Science Bowl along with 52 other regional winners. Nationwide, more than 9,000 students from 1,800 schools took part in Science Bowl competitions.

1999 Ames Laboratory
Iowa State University
Science Bowl
U.S. Department of Energy