Inquiry

SCIENCE AND
TECHNOLOGY
AT THE
AMES
LABORATORY

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

Letter from the DIRECTOR

It was with great pride that we introduced Secretary of Energy Bill Richardson to the Ames Laboratory since we last published *Inquiry*. Although his visit was short, we were able to provide him an excellent overview of our latest research in the materials, biorenewable resources and high-performance computing areas, and to demonstrate our continued commitment to conducting the highest-quality work for the lowest-possible cost. The comment the Secretary made during his visit that left the most lasting impression with many of us was when he said that not only does he want to see the Ames Laboratory continue, but that it should grow. We are eager to help him achieve that goal.

A second significant event of the year for the Ames Lab came shortly after the Secretary's visit, with the signing of a new five-year contract for the continued operation of the Laboratory. As the signatures were being put on the dotted line, the Lab was once again enjoying an "outstanding" rating from the Department of Energy for our scientific research, a fact which played a key role in the issuing of the new contract.

But we can never be content to rest on our laurels. Science is, to a large degree, about new discoveries – looking outside the box, to use today's catch phrase, to find interesting solutions to old and new challenges. Some of those challenges and our scientists' efforts to solve them can be found in the stories in this issue of *Inquiry*. I invite you to read about our discovery of the properties of what appears to be the second-hardest bulk substance after diamond. The compound could be a lower-priced alternative to the materials now used by industry to rapidly cut and grind the hardest steels.

Also in this issue, learn about the "Octopus," a cluster of 18 computers that can do the work of a much more costly supercomputer. You can also go to the "extreme" and learn about our new low-temperature, high-pressure and high-magnetic-field environment that subjects materials to extreme conditions. This testing helps determine the properties of materials under conditions they may have to experience in actual industrial applications.

Key to research at the Ames Laboratory is our multidisciplinary approach to science. We are constantly linking our scientific expertise, facilities and equipment in new and innovative ways to help find solutions to our nation's energy problems. We take pride in our accomplishments thus far and look forward to the challenges ahead.

Tom Barton

Tom Barton (right) presents Secretary Richardson with a plaque containing ultrapure metals made at Ames Laboratory.

Andreja Bakac injects a sample of saturated hydrocarbons she wants to oxidize into an aqueous solution of uranyl ions. The uranyl absorbs light that provides the energy to drive the oxidation experiment, making the otherwise lifeless hydrocarbons more reactive. (see story page 17)

(back cover) After being placed in the concave top of one of these drop-casting copper split-molds, an alloy button can be heated in an arc-melting system until molten. The molten material then "drops down" into the mold cavity. Once the alloy has cooled, the split-mold is opened to retrieve the desired shape.

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A Gem of a Discovery

Researchers create second-hardest bulk substance after diamond

By Susan Dieterle

Researchers Bruce Cook (left) and Alan Russell

The small piece of material resting in associate scientist Bruce Cook's palm isn't much to look at. If anything, it appears to be a shiny, charcoal-gray shirt button without any holes.

But this rather drab-looking compound has one dazzling characteristic – it is the second-hardest bulk substance after diamond.

And better still, it is expected to be less expensive than the material that now ranks a close third but is nearly as costly as gold.

Cook and two colleagues, associate scientist Alan Russell and assistant scientist Joel Harringa, developed the new material by introducing a small amount of silicon and other additives to an alloy of boron, aluminum and magnesium.

With test results that confirm the hardness of the material, nicknamed BAM, the researchers now hope to expand their research on two fronts. First, they want to further investigate the material's properties and find out whether other combinations of additives might make it even harder. And second, they want to determine how BAM can be produced in large quantities for industries that use ultrahard materials for coating, grinding and machining applications.

"What we're trying to do is combine fundamental science with an opportunity to improve industrial competitiveness in a number of key areas," Cook says. "We're hoping that this material will do that."

If the Ames Laboratory scientists succeed in resolving some of the questions surrounding the compound, the discovery could mean huge savings for manufacturers who use these types of materials in abrasives and cutting tools.

Diamond drawbacks

Diamond, which has a measured hardness of between 70 and 100 gigapascals (the equivalent of 10.2-14.5 million pounds per square inch), costs about $2,000 per pound in powder form. But it can't be used for cutting and grinding steel because it reacts by slowly dissolving into the iron when brought into contact with iron-based materials at high temperatures. In the high-speed grinding that takes place in the auto industry, for example, friction between the steel and the tool produces surface temperatures as high as 1,000 C (1,800 F).

When industries need an abrasive material that works fast and cuts deep into specially hardened martensitic steels, they rely on cubic boron-nitride, which has a hardness of about 45 GPa (6.5 million psi). Cubic boron-nitride doesn't have the iron reactivity problem of diamond, but it costs anywhere from $1,500 to $7,000 per pound because it is produced under conditions of extremely high temperatures and pressures.

"The fact that industries are willing to pay that price for certain uses gives some insight into what a critical industrial process this is," says Russell, a co-investigator on the project. "Cutting iron and steel is an enormous part of the U.S. manufacturing economy."

The BAM compound measures slightly harder than cubic boron-nitride at about 46 GPa (6.7 million psi) and is estimated to cost around $700 per pound. That could substantially reduce the cost of the cutting and grinding tools, enabling manufacturers to trim their production costs.


The substance could be used in cutting and machining applications, such as the surface of this grinding wheel.

"The consumer doesn't see the costs of these materials up front, but it definitely affects the overall price of the product," Russell says.

Hard luck

Cook discovered the hardness of the boron-aluminum-magnesium compound by accident. He was researching its thermoelectric properties in 1992 when he discovered that he couldn't cut the samples he'd made. "We have precision diamond saws in the lab that can cut virtually anything, and we weren't able to cut this material," Cook says. "That caught our attention."

Although the existence of the baseline compound had been known for awhile, the Ames Lab scientists found that the material's mechanical properties hadn't been fully investigated. "When Bruce discovered the hardness, it was unexpected and something that no one had thought to look for previously," Russell says.

It was also an unlikely candidate for a hard material because of the structure of its unit cell, or fundamental building block. "A diamond has eight carbon atoms in a unit cell. It's a very simple, highly symmetric structure," Russell explains. "This material has 64 atoms in the unit cell. If you gave this structure to a panel of experts and asked if it would be hard, they'd say, 'Nah, the crystal structure is all wrong.' But it's extremely hard. And that's the kind of thing that gets scientists salivating."

Cook says the complexity of the chemical structure offers a number of possibilities to vary its physical properties, such as enhancing the compound's hardness by substituting elements such as silicon. "We thought we might be able to change the bonding environment if we added silicon to the structure, and it worked. It made the material harder," he says.

"We would expect that by tweaking the composition, we may be able to push the hardness up a little higher," he adds. "This was the first additive we tried, and it produced a material that's right up there with cubic boron-nitride. But there may be other variations that could further increase the hardness of this material."

To better understand the reasons behind the material's hardness, band-structure calculations are being made by Bruce Harmon, deputy director of the the Ames Laboratory and director of the Lab's Condensed Matter Physics Program, and scientists in his group. With this information, Cook, Russell and Harringa could develop expectations as to which chemical additions would result in the ultimate hardness of the material.

"When the baseline compound was first synthesized in the late 1960s, the computational power to analyze the electronic properties of such a complex structure simply did not exist," Cook explains. "Only recently has the ability to tackle such a complex system existed. Even so, a 64-atom unit cell is an extremely challenging theoretical problem."

Encouraging results

To find out how BAM would fare in a real-world setting, the Ames Lab scientists sent samples to Autodie International, a Michigan company that manufactures tools, dies and molds for the automotive industry. Autodie reported favorable results in its initial tests, Cook says, adding that the company was especially pleased that the material didn't fracture – a common problem for many cutting tools, which are often brittle.

Cook adds that hardness is not the only material property of interest. Wear resistance, toughness, hardness at high temperatures and how well it conducts heat are also important factors affecting the viability of BAM in industrial applications.

During 1998, the scientists used a one-year Department of Commerce grant from Iowa State University's Center for Advanced Technology Development to study the material and possible additives to enhance its hardness. They also received a small grant through the Roy J. Carver Trust.

The researchers are now looking for additional funding for a more extensive study of BAM's preparation and properties. Among their research priorities are a better scientific understanding of the material itself and determining how best to produce large quantities of it.

They also want to investigate the possibility of producing the material as a uniform powder that could be deposited as a wear-resistant coating on surfaces such as bulldozer blades and mining tools. "We know that the two other hardest materials won't tolerate it," Russell says. "This one might."

He says initial tests show that BAM's coefficient of thermal expansion – the change in a material's length in response to temperature changes – is close to that of iron, which indicates that a coating of the material might adhere well to steel.

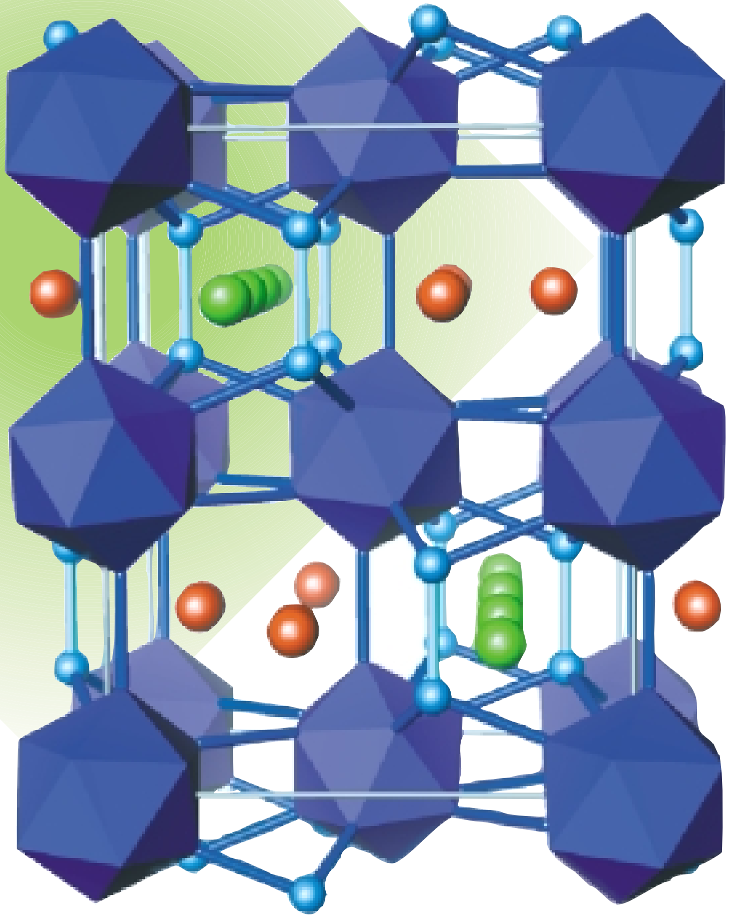

Diagram courtesy of Bruce Harmon

The complex crystal structure of the baseline AlMgB compound without any additives is pictured above. The boron atoms are blue, the aluminum atoms are green and the magnesium atoms are red.

Growing interest

The BAM compound is generating quite a buzz among scientists and manufacturers. Russell notes that researchers from Ames Lab and other facilities are exploring the compound's properties on specialized equipment at Stanford University and DOE's Argonne National Laboratory.

In addition, more than 70 industry representatives have contacted Cook and Russell about a diverse assortment of possible uses for the material, such as testing-probe tips and wire guides for the microprocessor fabrication industry; wear-resistant linings for the petroleum and mining industries; and thin coatings for cutting tools.

"We're excited about the level of interest in this material and the wide range of applications that it could possibly be used for," Cook says. "We want to continue our research so that we can give these companies the information they need in order for them to determine how they could use the material."

Part of that effort involves devising a method for producing larger quantities of the compound instead of the small disks they've been using for research purposes.

"Based on the work we've done to this point, we feel confident that we can resolve the remaining issues and keep moving forward," Cook says. ✳

For more information:
www.external.ameslab.gov/news/boride.html

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(left to right) Karl Gschneidner, Vitalij Pecharsky and Bruce Harmon have giant hopes for the gadolinium-silicon-germanium alloys.

COLOSSAL UNDERTAKING

Ames Lab team launches broad-based effort to better understand promising materials

By Susan Dieterle

The news about materials that scientist Vitalij Pecharsky and senior metallurgist Karl Gschneidner Jr. have studied for the past five years keeps getting bigger and bigger.

Originally noted for their potential in magnetic cooling and freezing applications, the gadolinium-silicon-germanium alloys are now known to possess properties that could be useful in sensors and energy-conversion devices. But first, scientists need a better fundamental understanding of the alloys and why they respond so dramatically to changes in temperature and magnetic field.

To answer those questions about the Gd-Si-Ge alloys and several closely related materials, the Department of Energy has awarded a four-year research project to a team of Ames Lab metallurgists, physicists and chemists led by Gschneidner and Pecharsky. Total funding depends on final DOE budget figures, but could be up to $1 million a year.

"These alloys could be among the most significant materials of the new millennium," Pecharsky says. "I'm sure that we're in for many more surprises and interesting phenomena in the next four years."

In 1995, Pecharsky and Gschneidner began exploring the possibility of using Gd-Si-Ge alloys in magnetic-refrigeration technology and almost immediately discovered the alloys possessed a giant magnetocaloric effect, meaning they heat when magnetized and cool when removed from a magnetic field to a much greater extent than other known alloys. That property made the alloys strong candidates for magnetic cooling and freezing applications. After filing for a patent, the scientists announced their discovery in 1997.

Since then, researchers here and throughout the world have discovered that the materials also possess giant magnetoresistance (a change in the magnetic field triggers a change in the material's electrical resistance) and colossal magnetostriction (the shape or length of the material changes in response to magnetic forces).

Simply put, a relatively small change in the magnetic field surrounding the material produces a tremendous change in its temperature, dimensions and electrical resistance.

"Some materials possess one or two of these properties, but what's unique about this material is that all three changes take place in the same alloy and all three changes are quite large – among the largest ever seen," Gschneidner says.

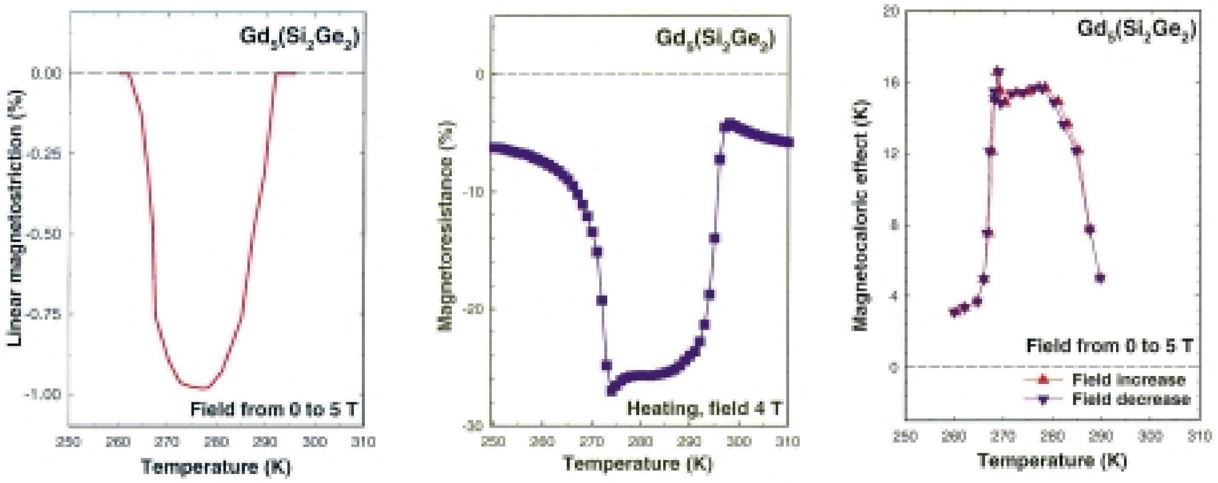
That combination of properties and responsiveness makes the alloys potentially useful in energy-conversion devices, such as systems that transform magnetic energy into mechanical energy and vice versa, and in sensors. Pecharsky says most sensor materials are only effective in certain temperature ranges, but these alloys can be tailored to respond at a variety of temperatures.

In late 1999, the DOE asked its national laboratories to submit competitive research proposals involving complex, advanced materials. The goal of the competition was to select one or two projects for in-depth research that would enable scientists to understand how these types of materials could be used in the future.

Pecharsky, Gschneidner and eight other Ames Lab researchers submitted a proposal to study the Gd-Si-Ge alloys. The other principal investigators are Vladimir Antropov, Scott Chumbley, Bruce Harmon, David Jiles, Tom Lograsso, Gordon Miller, John Snyder and Constantine Stassis. This July, the team was notified that its proposal had been selected for funding.

"We have learned a lot about the properties of this material to date, but we need to understand what causes the extraordinary responsiveness of this and related materials," Pecharsky says. "That's what this project is about."

The team will explore the properties of the Gd-Si-Ge alloys and many closely related materials. The scientists will also develop theories and models that detail the relationship between the composition, structure and properties of the alloys. The models and theories could then be used to engineer the materials for specific applications in the future.



(left to right) The charts depict the colossal magnetostriction, giant magnetoresistance and giant magnetocaloric effect of the Gd-Si-Ge alloys.

Pecharsky will oversee the experimental portion of the research, and Harmon, deputy director of Ames Laboratory and director of the Condensed Matter Physics Program, is in charge of the theoretical work. Gschneidner will serve as project manager.

The three scientists agree that the Gd-Si-Ge alloys are uniquely suited to this type of in-depth materials research. "It has been clear for a very long time that the physical properties are indeed related to both the composition and structure of any material. What has been a mammoth task is to understand how," Gschneidner says.

Pecharsky adds that because clear-cut connections have already been established between the chemistry, crystallography and magnetism of the Gd-Si-Ge alloys, the Ames Lab group is confident that it can succeed in understanding how those properties become so interconnected.

Gschneidner says when scientists understand that process, "we will be ready to make a step having tremendous importance – designing a material with predicted properties and behaviors. That's a dream come true for any scientist involved with materials."

Part of the funding will be spent on a customized X-ray powder diffractometer, which will help show how changes in temperature and magnetic field affect the alloys. The equipment will give Ames Lab the unprecedented ability to study changes in the material's crystal structure at a variety of temperatures as the strength of the magnetic field is altered.

"We're counting on traditional techniques and nontraditional techniques, in the form of the new and quite unique equipment, to help us understand this material," Pecharsky says. "I think we've proposed an extensive and very interesting route to advance the basic studies of these materials."

Harmon notes that the multidisciplinary nature of the Ames Lab team is a definite advantage.

"These are fascinating materials with complex crystal structures and a remarkable combination of magnetic and mechanical properties," Harmon says. "This new funding offers a great opportunity to utilize and extend Ames Laboratory's internationally recognized expertise in the area of rare-earth magnetic materials."

Pecharsky says he and Gschneidner consider themselves fortunate that so many other scientists around the world are interested in the alloys.

"After we first published our findings about the giant magnetocaloric effect in the materials, there were still many unanswered questions about the unusual features that were seen in both the magnetic properties and the crystallography. Other people started looking at the materials with interests far away from magnetic refrigeration," Pecharsky says. "As they started looking at it and we continued our research, the bigger and bigger picture started coming out.

"It's good to know that something you've discovered is considered important by your peers." ✻

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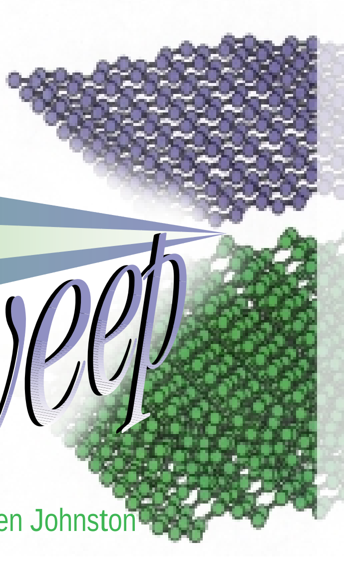
Clean Sweep

Atomic simulations show length of laser pulse is crucial in laser ablation of diamond



By Saren Johnston

Cai-Zhuang Wang and Kai-Ming Ho tend to view materials as marvelous collections of atoms and electrons. As theoretical physicists, they rarely investigate a solid chunk of material sitting on a workbench, preferring instead to explore the material at the atomic level – a world teeming with activity.

The two Ames Laboratory researchers devote their efforts to describing this activity and understanding why atoms interact the way they do in specific situations. The atomic simulations they develop help explain the "why" behind what experimentalists observe in the laboratory.

Over the last 10 years, Wang and Ho have developed a method for doing quantum molecular dynamics simulations of practical materials with far more ease, speed and economy than is possible with conventional first principles methods. The technique, called tight-binding molecular dynamics, is a computationally efficient means of studying the structures, dynamics and electronic properties of complex systems at the atomic level. And TBMD can accommodate changes in bonding within a material due to electronic excitation, a capability that most atomic simulation methods don't possess.

TBMD – a "simple" plan

Wang explains that the interactions between atoms originate from the electrons, so the interactions are best described by quantum mechanics. But full quantum mechanics calculations are too complicated for atomic systems containing large numbers of atoms.

"The idea of TBMD is to keep the spirit of quantum mechanics but simplify the process by developing a model for molecular dynamics simulations that can describe atomic interactions accurately, yet is fast enough to treat systems with large numbers of atoms," says Wang.

Wang's and Ho's research on TBMD won a Department of Energy Materials Sciences Award in 1996. Using TBMD, they have created simulations to investigate such phenomena as carbon clusters and buckyballs, and amorphous forms of carbon and silicon. They have successfully developed other TBMD schemes to simulate how carbon atoms behave in different environments and under different circumstances.

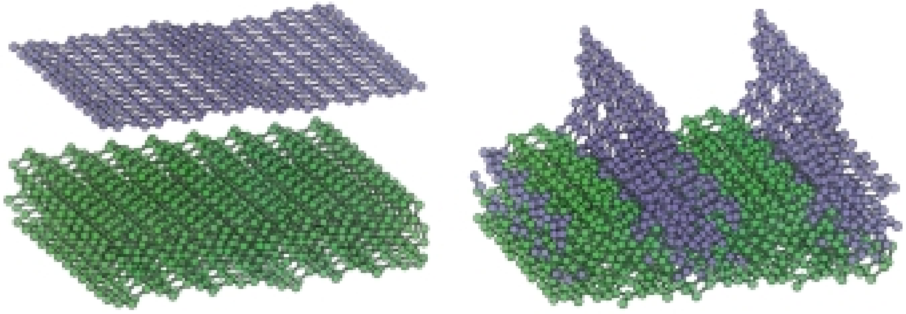
Iowa State University researchers, lead by mechanical engineering professor Pat Molian, wanted to better understand what they were observing in their laboratory experiments on laser ablation of diamond. So Molian decided to contact Wang and Ho. He was hoping to interest the two physicists in doing some unique TBMD simulations that would describe what was going on at the atomic level during diamond ablation with laser pulses of different durations.

The long and short of it

Diamond is popular within the microelectronics and tool-making industries because of its unique properties. It is the hardest material known and has the highest thermal conductivity. It also has a large bandgap that makes it transparent to light in the ultraviolet range. However, the very properties that make diamond such a useful material also make it extremely difficult and expensive to machine – a predicament that Molian thinks could be remedied with pulsed laser technology.

Pulsed lasers provide what scientists call peak power – the amount of power delivered during a single pulse of laser energy. Pulse width, or duration, greatly affects the peak power of a laser because it defines the amount of time the material being studied interacts with the light. Pulse widths of longer durations are generally associated with lower intensity lasers and longer periods

Using an atomic simulation he developed, Cai-Zhuang Wang demonstrates how carbon atoms (purple circles) are peeled from the diamond surface in a layer-by-layer fashion when subjected to femtosecond laser pulses.

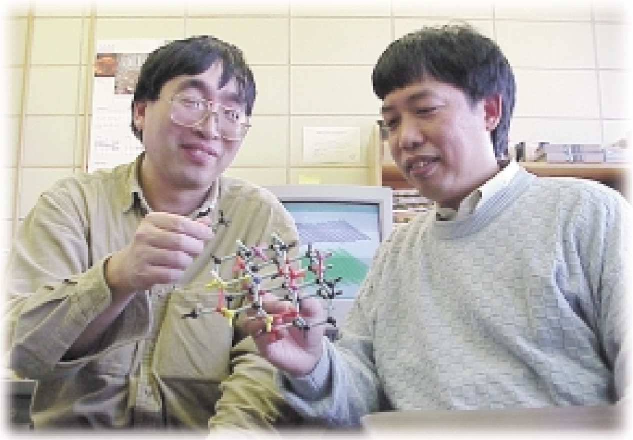


(left) Wang's and Ho's TBMD simulations reveal that femtosecond laser pulses peel carbon atoms (purple circles) layer-by-layer from the diamond surface, leaving a smooth surface after ablation.

(right) TBMD simulations show that nanosecond laser pulses cause thermal melting, resulting in graphite residue (purple circles) that anchors to the diamond surface following ablation.

of light exposure, while pulse widths of shorter durations are associated with higher intensity lasers and shorter light exposure, characteristics essential for high-precision laser machining.

Molian and his colleagues have been investigating the use of ultrashort pulsed lasers for micromachining diamond in their laser ablation experiments. They observed that ablation with nanosecond (one billionth of a second, or 10^{-9} second) or longer laser pulses caused graphite formation and contaminated surfaces. The researchers discovered the graphite, or "diamond dirt," by using Raman spectroscopy, which detects the vibrations in a material. Before nanosecond laser ablation of diamond, the Raman spectra displayed characteristic peaks. But the peaks had disappeared in the spectra produced following nanosecond laser ablation, indicating that the surface was no longer diamond in structure.



Cai-Zhuang Wang (right) and Kai-Ming Ho often construct and study three-dimensional models in the process of creating computer simulations to describe atomic activity.

"Graphite formation is a big issue for many applications, especially electronics, where they don't want any contamination," says Molian. "So we moved on from nanosecond to femtosecond laser technology." (Femtosecond pulses are one quadrillionth of a second, or 10^{-15} second long.)

The switch to the ultrashort femtosecond pulse proved beneficial. The Raman spectra produced both before and after femtosecond laser ablation showed similar peaks, indicating clean surfaces free of graphite contamination. The femtosecond laser pulse had made a clean sweep, removing the top layers of diamond atoms.

Ah – those "whys and wherefores"

"We were very anxious to learn why the femtosecond lasers did so much better than the nanosecond lasers. We could see what was happening, but we didn't know why," says Molian.

Ho says, "They came to us to see if we'd do some simulations that would tell them what was happening atomically – what was taking place on the diamond surface and why the femtosecond and nanosecond laser ablation processes resulted in different structures."

Wang explains, "It usually takes a picosecond for the energy from the laser-excited electrons to reach the atoms. (A picosecond is one trillionth of a second, or 10^{-12} second.) When diamond is irradiated with nanosecond laser pulses, the laser is intense at 10^{-9} second, and the atoms can be thermally excited. This leads to melting and the formation of light-absorbing graphite residues that contaminate the surface layers."

On the other hand, the femtosecond laser pulse is much shorter than the time required for an atom to vibrate, so it is able to eject the electrons into highly excited states while the atoms are still thermally cool. The energy stays in the electron system. The TBMD simulation shows that femtosecond laser ablation peels atoms from the diamond surface in a layer-by-layer fashion through a non-thermal mechanism, leaving a smooth diamond surface.

"The femtosecond laser leaves a very clean cut," says Ho. "With the different amount of energy that is input, there is more surface removed."

The conventional view of material removal by laser is that the material is heated, melts, becomes a liquid or a gas and goes away, Wang explains. "However," he continues, "the physics involved with the femtosecond laser is different than the normal melting picture because the time scale is not long enough to allow the atoms to vibrate. That is some new physics, and the reason why Dr. Molian and his colleagues came to us for the research and TBMD simulations that would help them better understand that process. Our models correlate very well with their experimental findings." ❋

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Research funded by:
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New Life at the Light Source

by Saren Johnston

Ames researchers are tapping into the brilliant X-rays at Argonne's Advanced Photon Source

photo courtesy of Argonne National Laboratory

μCAT
Midwest Universities
Collaborative Access Team

At last, MUCAT has seen the light!

After 10 years of planning and construction, and a whole lot of problem-solving, the undulator beam line developed by the Midwest Universities Collaborative Access Team is up and running at Argonne National Laboratory's Advanced Photon Source near Chicago.

"The beam line is operational. We're actually at the point where we're taking data, getting results and writing papers," says Alan Goldman, an Ames Laboratory senior physicist and chair of the Iowa State University physics and astronomy department. He heads MUCAT, a group of scientists from eight universities and one German institute that was organized in 1990 to develop a sector at the APS.

The APS provides MUCAT scientists and members of other collaborative access teams with a means of tapping into some of the strongest X-ray beams in the world. Goldman and his MUCAT colleagues can use the brilliant, highly focused X-rays produced by the APS third-generation synchrotron radiation source to investigate the molecular make-up of all kinds of materials, ranging from magnets, ceramics and soils to proteins, drugs and asteroid dust.

"The APS is an incredibly versatile tool that allows you to see microscopic details of materials with extremely high resolution," says Goldman. "It can be used by almost any group interested in looking at the structure of materials. Most significantly, because it has wide applications to so many fields, it allows for collaborative research efforts among different disciplines."

Running in circles

The APS synchrotron operates round-the-clock, generating electrons and accelerating them to energies approaching the speed of light. In just a quarter of a second, the electrons can make 200,000 trips around the synchrotron's 368-meter circumference (approximately 405 yards) as they climb to energies approaching 7 billion electron volts.

From the synchrotron, the electrons are injected into a giant storage ring about two-thirds of a mile in circumference. The storage ring is constructed as a set of curves connected by straight sections. Once inside, the electrons travel in a concentrated beam, circling the ring more than 271,000 times per second. The electron beam is guided and focused by hundreds of powerful electromagnets that keep it moving in a circular path.

Researchers have optimum access to the beam through the experiment hall, which surrounds and is separated from the storage ring by a shielding wall. The experiment hall consists of 35 sectors, which serve as laboratory space for the collaborative access teams using the APS.

"Each sector in the APS really consists of two beam lines that tap the synchrotron radiation produced by the storage ring's electron beam," says Goldman. "The insertion device beam line contains powerful permanent magnets that have been placed in the straight sections of the storage ring to either wiggle the beam back and forth or make it undulate – this is how you get synchrotron radiation. But the act of turning the beam around the corner by a bending magnet will also produce synchrotron radiation, so each sector also has a bending magnet beam line," he explains.

The bends, wiggles and undulations produced by the bending magnets and insertion devices alter the electron beam's course and allow it to navigate the straight-aways and curves of the storage ring. As its path changes, the beam sheds synchrotron radiation in the form of X-rays that range in power from 1 kilovolt to hundreds of kilovolts. By way of comparison, Goldman says that standard medical X-rays have an energy of 50-60 kilovolts.

Setting up in sector 6

Although the undulator beam line in MUCAT's sector 6 is now working, Goldman says it's difficult to pinpoint the exact date it became operational.

"There's a constant effort in commissioning that goes on when you're getting beam. And there are lots of problems to solve on these lines because they're extraordinarily complicated," he says.

Fortunately for Goldman and the rest of the MUCAT scientists, they have an outstanding group of trouble-shooters to count on. Doug Robinson, an Ames Lab physicist, is in charge of operations at the beam line. "More than anyone, Doug has really made that thing work," says Goldman. He adds that Ames Lab assistant scientist Eric Zoellner has been at the beam line since the beginning of construction. "Eric has had an awful lot to do with its success, as has Didier Wermeille, a postdoc in my group who's been working on the beam line for about two years."

The building process for sector 6 started from the ground up, or put more accurately, from the wall out. "What the APS gives you in the very beginning is a hole in the wall," says Goldman with a grin. "Basically, what you get to start with is a beam pipe and a flange at the end. You have to build everything else outside to access the storage ring's electron beam."

Everything else includes a lot of expensive equipment, so sector 6 is coming together bit by costly bit. There's an enclosure for the optical systems that condition the X-ray beam so researchers can use it in the experimental station. "All of the instruments and equipment in the optics enclosure condition the beam so we get something clean coming down the pipe and into the experimental enclosure, which is about 50 meters from the storage ring," explains Goldman.

The MUCAT undulator beam line consists of two experimental stations that operate in tandem. And those stations are slowly filling up with the kinds of equipment that will allow Goldman and other MUCAT members to carry on a diverse assortment of research projects. An instrument currently in operation in station 1 is the four-circle diffractometer. Goldman and his Ames Lab colleagues are using the diffractometer to perform magnetic scattering measurements and to study X-ray diffraction from single crystal samples produced at the Lab. The work has brought new information to light about the magnetic structures of the compounds from which the crystals were produced, as well as revealed new magnetic transitions that did not show up under traditional measurements.

Station 1 is also the home of a novel piece of equipment that is one of only a few in the world. The liquid surface reflectometer developed by Ames Lab physicist David Vaknin will be used in conjunction with the powerful APS X-rays to investigate the structure of ultrathin layers of organic materials that may one day be used in novel optical, electronic and biological devices.

APS Collaborative Access Teams

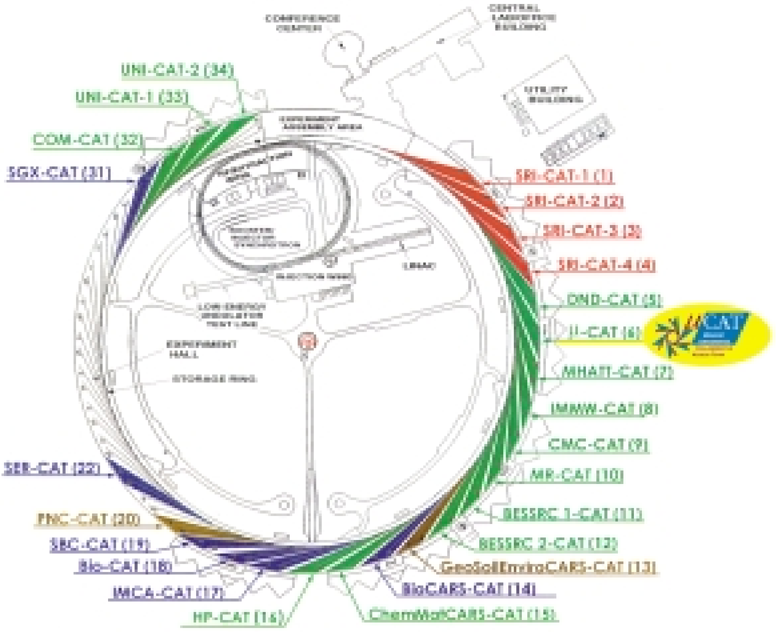

diagram courtesy of Argonne National Laboratory

Small furnace is hot item

A piece of equipment being tested in station 1 on the MUCAT undulator beam line is a new compact furnace designed by Goldman and Ames Lab researchers Bill McCallum, Matt Kramer, Kevin Dennis and Larry Margulies (*see story page 11*). The furnace allows high-temperature powder diffraction studies of complex materials at various temperatures and under the same processing conditions that exist in the laboratory environment. The resulting diffraction patterns reveal what happens to the crystal structure

Alan Goldman (foreground, with white identification badge) heads the group of MUCAT scientists whose collaborative efforts have brought the MUCAT undulator beam line into operation.

as a material heats and cools, information needed to better understand and control the microstructure of new materials.

"I expect the new compact furnace will be one of the most popular instruments on the beam line because it's really rather unique," says Goldman. He explains that the idea was to scale down a basic tube furnace design and make it fit on the four-circle diffractometer to take advantage of the high-energy X-rays produced by synchrotron radiation. "You can control the furnace temperature up to 1,500 C (2,732 F) and also spin the sample in such a way that there are no preferred orientation effects. It's worked quite nicely," he adds.

Always room for more

In addition to the equipment already in place, researchers in sector 6 will soon be able to make use of a new surface science chamber. Funded by the National Science Foundation, the chamber will be constructed in station 2 on the MUCAT undulator beam line. It will be used to study the surface properties of materials and surfaces that are grown in situ using a variety of techniques.

"There's a lot to do, but it's going to be fun," says Goldman. "I like playing with complicated pieces of equipment. And I like seeing things come together and work."

With any luck, Goldman will see that happen with the special high-energy side station, which is being funded and constructed by the German institute, F. Z. Juelich, a MUCAT member organization. As its name implies, the high-energy side station will sit to the side of the main undulator beam line in sector 6. It will operate in parallel with the beam line, supplying energies up to 120 kilovolts compared with the main beam line's lower energies of 3-40 kilovolts.

"The high-energy range is for experiments in which we want to penetrate into the sample more deeply," says Goldman. "The low-energy range will help us with magnetic scattering studies on actinide materials."

Filling up the second hole in the wall

Goldman and his colleagues have a lot on their plates, but not so much that they can't make room for more, especially when it comes in the form of a second beam line for sector 6. "We've been funded to build a bending magnet beam line, which will complement the undulator line and allow a wide variety of standard scattering and spectroscopy techniques to be used," Goldman says. "This is where the high-temperature diffractometer and the compact furnace will live, eventually."

Experiments planned for the bending magnet beam line include studies of phase diagrams, in situ investigations of nucleation and growth, recrystallization of bulk metallic glasses and processing of high-temperature materials.

Getting the new beam line operational will mean a lot of travel between Ames and Argonne for Goldman, who never seems to worry about the time or the miles. "I think one of the things that keeps me going is that there's this instant in time when you're working on a project and you finally understand, or think you have a good way of understanding, what's going on in a material," he says. "And for that instant, you know something that other people don't know. So it's just discovery itself."

And in the end, discovery is the reason Ames Laboratory has strongly supported the MUCAT project, beginning with the 1990 initial planning stages – before the APS was even built. "The Lab's support over the years has been just outstanding," says Goldman. "The APS is a remarkable tool for synchrotron X-ray studies for the general scientific community. And we're going to welcome anyone to come out and do some work." ✻

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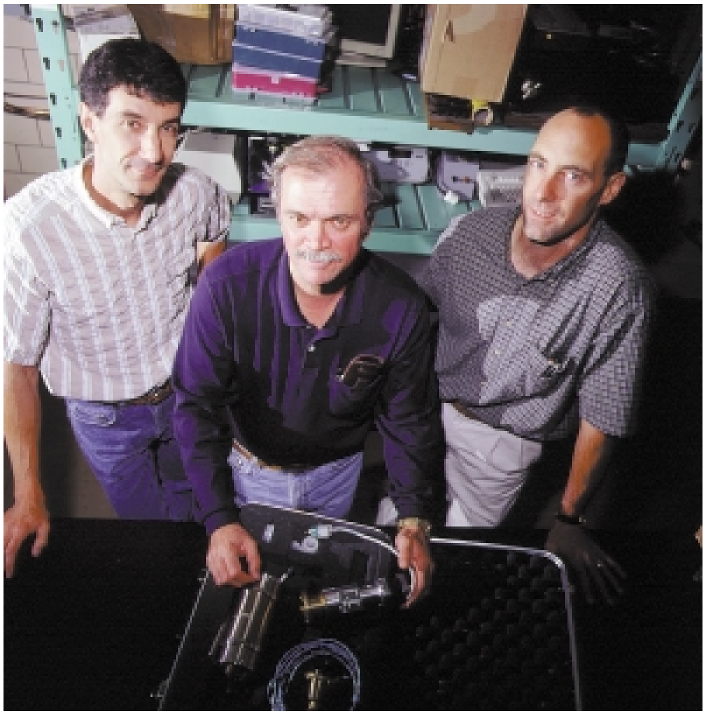
Research funded by:
DOE Office of Basic Energy Sciences

Shedding New LIGHT on Advanced Materials

By Susan Dieterle



(left to right) Researchers Kevin Dennis, Bill McCallum and Matt Kramer display the components of the compact furnace they designed along with Alan Goldman and Larry Margulies.

A unique, compact furnace combined with high-energy X-rays is giving researchers at Ames Laboratory the unprecedented ability to directly record the chemical and structural changes of complex materials at high temperatures under real processing conditions.

This information is crucial to understanding and controlling the composition and microstructure of new materials. It previously took months or years to collect such data through the laborious process of heating, quenching and then analyzing numerous samples. But the Ames Lab researchers can now gather the data in just a few days while getting a more detailed picture of what happens to a material's crystal structure as it heats and cools.

> “The excellent control we have with our furnace means that we can select an exact temperature setting for our measurement and know that the whole sample is that temperature.”
>
> *-Matt Kramer*

The new system is ideal for complex materials such as structural ceramics, superconducting wires and nanostructured materials. The insights gained through the Ames Lab system may speed the development of new materials for use in fields such as aerospace engineering, electrical distribution systems and microelectronics.

“We're seeing details of the phase transitions that I don't think anybody has ever described before,” says scientist Matt Kramer, who helped design the furnace built by Ames Lab's Engineering Services Group.

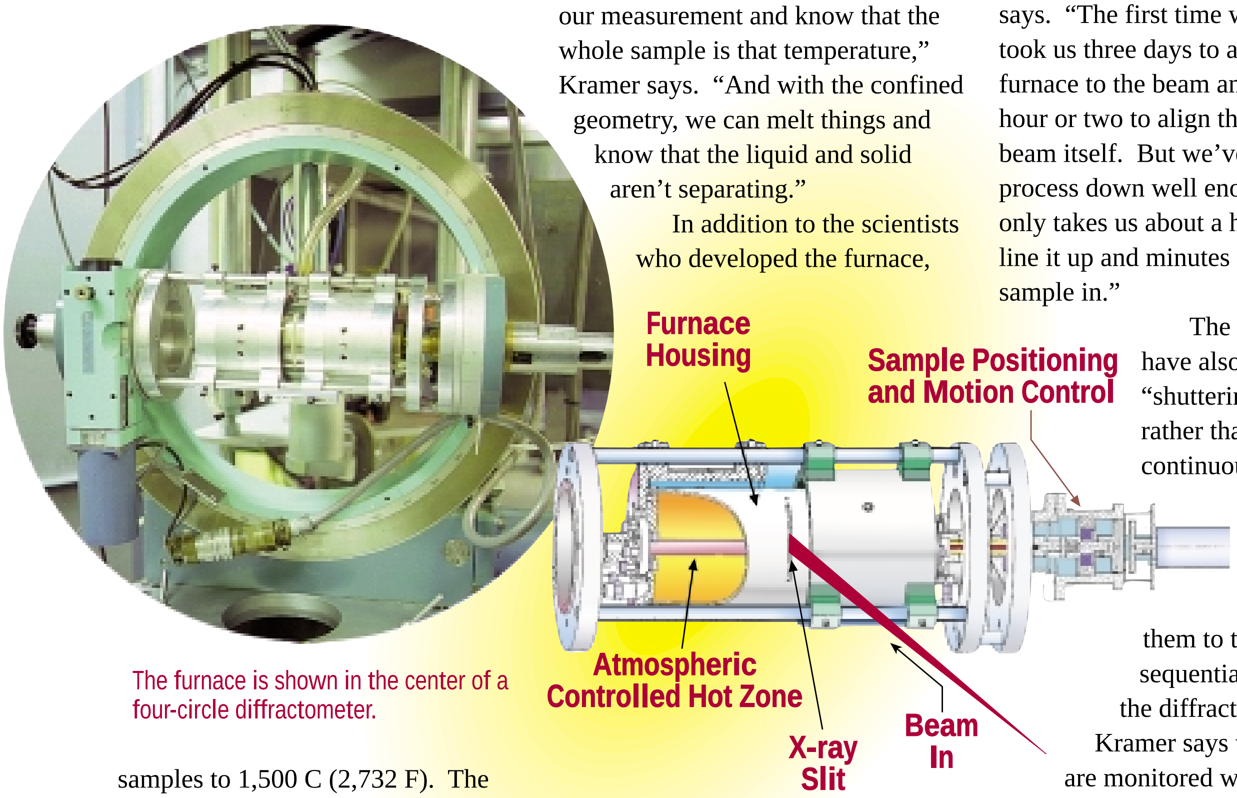
The furnace uses an analytical technique known as X-ray diffraction in which an X-ray beam is focused on a small sample of material. The beam is diffracted by the crystal structure of each material, producing a unique pattern of concentric circles, called “Debye rings.” By capturing images of the changes in the ring pattern as the material is heated and cooled, scientists gain a better fundamental understanding of what happens to the material's crystal structure at various temperatures.

In 1997, Kramer began working with senior materials scientist Bill McCallum, senior physicist Alan Goldman, assistant metallurgist Kevin Dennis and then-Ames Lab graduate student Larry Margulies on a design for a compact, portable furnace that would enable them to rotate samples during an experiment. They also wanted to be able to subject the samples to a variety of environments, such as inert or oxidizing atmospheres, encountered in processing conditions.

What emerged was a scaled-down version of the standard laboratory tube furnace, measuring about 18 inches in length and 6 inches in diameter and capable of heating samples to 1,500 C (2,732 F). The furnace has an indirect, magnetic coupling system that connects to a motor shaft, which rotates the sample.

Samples are held at the end of a long tube and aligned with a 3-millimeter opening in the side of the furnace. The X-ray beam enters through the opening and the diffracted rays emerge through a slot in the furnace.

Kramer says the new system is an improvement over current high-temperature X-ray diffraction systems, in which samples rest on a flat plate. This doesn't allow the sample to be rotated and sometimes causes the liquid and solid phases of the material to draw apart. Also, the flat-plate systems don't always heat the sample uniformly, producing large temperature variations in the material that make it difficult to correlate the temperatures with changes in the crystal structure.



The furnace is shown in the center of a four-circle diffractometer.

"The excellent control we have with our furnace means that we can select an exact temperature setting for our measurement and know that the whole sample is that temperature," Kramer says. "And with the confined geometry, we can melt things and know that the liquid and solid aren't separating."

In addition to the scientists who developed the furnace, the device has been used by Ames Lab scientists Doug Finnemore and Dan Sordelet as well as researchers at DOE's Brookhaven National Laboratory.

The experiments are conducted at off-site facilities where high-energy X-rays of between 35 and 100 kilovolts are available. Most of the experiments take place at the Advanced Photon Source at the Department of Energy's Argonne National Laboratory near Chicago and the Cornell High Energy Synchrotron Source in New York.

At Argonne's APS facility, the furnace has now found a home on a sector reserved for the Midwest Universities Collaborative Access Team, which is operated by Ames Lab and Iowa State University (*see story page 8*).

Before an experiment begins, the furnace must be aligned with the X-ray beam – a painstaking process because the beam itself is about 1 millimeter wide and 0.5 millimeter high. "That's the hard part," Kramer says. "The first time we did this, it took us three days to align the furnace to the beam and then another hour or two to align the sample to the beam itself. But we've gotten the process down well enough now that it only takes us about a half a day to line it up and minutes to put the sample in."

The researchers have also found that "shuttering" the beam, rather than using a continuous ray, enables them to take better sequential images from the diffracted rays.

Kramer says the reactions are monitored with a time resolution of less than two seconds, fast enough to make a virtual movie of images that capture the material's structural transformation during temperature-driven processing.

Kramer adds that colleagues at Argonne and the European Synchrotron Research Facilities have asked about having Ames Lab build similar furnaces for them. He notes that the Ames Lab group is also willing to let other researchers use the furnace. ❋

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Research funded by:
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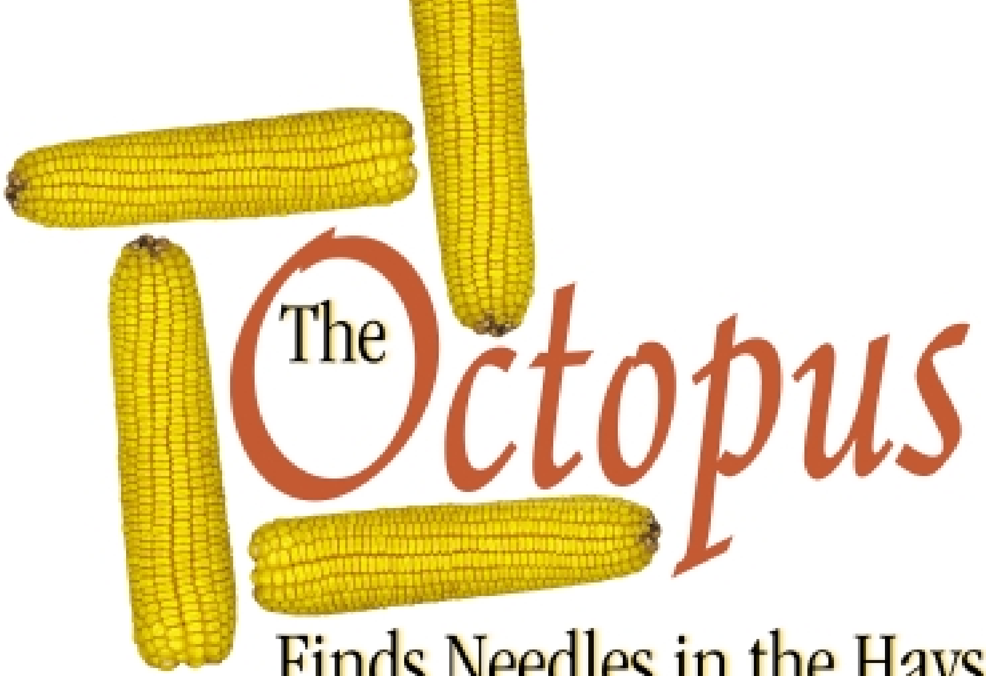


The Octopus Finds Needles in the Haystack

By Robert Mills

It's not impossible to find a needle in a haystack. If you have enough time – or enough arms – it can be done. That's more or less the reasoning behind a "cluster computer" designed by the Ames Laboratory's Scalable Computing Laboratory and built by Iowa State University's zoology and genetics department. Called the Octopus, this machine has 18 computers that function in concert to do the work of a much more costly supercomputer.

In this case, the "haystack" is the huge amount of genetic data being generated by various genome discovery projects. The high-profile Human Genome Project, for instance, will ultimately result in data on the 3 billion chemical base pairs that make up human DNA. Researchers at ISU, working with various colleges around the country, are dealing with similar amounts of data with their maize gene discovery project.

The "needles" in these haystacks are sequences of DNA, or genes, that living cells follow to make proteins. Those proteins, in turn, perform the elemental work of life.

Determining gene sequences and how they express themselves in genetic traits is difficult at best. One challenge is the sheer amount of data involved; just scanning through the human genome data at a rate of 100 bits per second would take close to a year.

Another complicating factor is that only the human genome project can afford the brute force method of direct sequence gathering. Even with the importance of maize – the class of crops responsible for more than 70 percent of the caloric value of the human diet worldwide – researchers must instead find more elegant, less costly ways to crack its genetic code. These methods involve the use of sophisticated computer algorithms acting on huge databases.

Bioinformatics to the rescue

The computational demands of genomics is what led Volker Brendel, an ISU associate professor of zoology and genetics, to instigate the Octopus project. Brendel is a member of the maize gene discovery team and an expert in the nascent field of "bioinformatics." This relatively new discipline taps tools and techniques from biology as well as mathematics and computer science. It relies on sophisticated statistical techniques and efficient software algorithms to interpret genetic data, leading to a better understanding of biological systems.

Like almost anything related to information technology, bioinformatics research can be accelerated by the use of faster computers. To be sure, high-performance supercomputers have the computational horsepower and memory needed to handle gene data with ease. But the cost of these systems starts somewhere north of several hundred thousand dollars. So instead, Brendel considered a cluster computer to be built using run-of-the-mill, and therefore low-cost, personal computer components. Soon, Brendel was in touch with the experts at Ames Lab's Scalable Computing Laboratory.

Cluster computing is one of SCL's specialties. Its scientists have built or designed eight clusters for

Researchers at ISU's zoology and genetics department use a cluster computer built with assistance from the Ames Lab's Scalable Computing Laboratory to help unravel the gene sequence of maize.

Ames Lab and ISU researchers. The SCL also runs two of its own cluster computers, including a $665,000 cluster of dual processor IBM Power3 workstations made possible via a Shared University Research grant from IBM. The SCL team has even written a cluster "cookbook" that provides a how-to on constructing PC clusters (available on the Web at *www.scl.ameslab.gov/Projects/ClusterCookbook/*).

Still, the design of a cluster is a shade more difficult than cooking dinner. Cluster computers achieve high speeds by dividing the work of the software and running various sections simultaneously on individual processors called "nodes." As such, the various components – including processor speed, memory and hard disk space – need to be "balanced" for a particular application to achieve optimal speeds. For the Octopus, SCL researchers ran benchmarks of their own design to determine the most appropriate characteristics for the system.

The SCL also provides ongoing assistance to Brendel's group to help write software that can take advantage of the cluster. "Sticking these machines in a room, turning them on, and so on, is arduous but doable. The true research aspect that we help other groups with is how to get their code to run in parallel," says David Halstead, SCL associate scientist who is sometimes called "Clusterman" around Ames Lab for his expertise in the field.

Plain vanilla, but fast

Each node of the Octopus consists of a 450 megahertz Intel Pentium II microprocessor, 512 megabytes of memory, and a nine gigabyte hard disk. By itself, each would make a fairly powerful desktop computer. Yet, the parts are "plain vanilla," says Halstead. The use of these standard parts translates into low cost, he explains, adding that each node can also have a life after the cluster as a desktop system for word processing and other routine computing tasks.

Brad Powers (left), an ISU senior in biology, and Ames Lab's David Halstead are shown with the Octopus, an 18-node cluster computer they helped build for ISU's zoology and genetics department.

As for the software, "Brendel's algorithms are ideal applications for cluster computing," explains Halstead. "The problem is fairly linear," which means that adding more processors leads directly to faster processing. The $45,000 Octopus has a theoretical capacity of roughly 3.2 billion floating point operations per second, which makes it about half as powerful as the $300,000 ALICE cluster operated by SCL in the Ames Lab. Brendel is also planning to follow SCL's suggestion to add a "terminal room" with desktop systems that can be used either by students as regular computers or as additional nodes for Octopus.

The whole situation "couldn't have worked out better," says Brendel of the design and building of the Octopus, which essentially took about three months. The machine is now up and running in ISU's Molecular Biology Building. "We are still in the infancy of using the machine. Basically, we are using it to speed up sequential code for gene finding and annotation," he says.

Thanks to their low cost, cluster computers are becoming more common in universities and research institutes; in fact, Ames Lab and ISU are home to at least nine such systems. But most clusters are made for physics, engineering, weather forecasting or just to learn more about cluster computing. The Octopus, in contrast, is probably one of the few built specifically for bioinformatics, according to Brendel. What's more, it's located in the same building where computational biologists do their research. "It lets me stay close to my data," he says. ✳

For more information:
www.octopus.iastate.edu

Research funded by:
DOE Office of Basic Energy Sciences
Iowa State University

A material developed by Bill McCallum (foreground) and David Jiles forms the dark ring in the middle of the torque-sensor sample in McCallum's hand. The composite material undergoes slight length changes when magnetized.

On the Road to LIGHTER Vehicles

New sensor material could be part of lightweight power-steering technology

By Susan Dieterle

You might say that David Jiles and Bill McCallum share a mutual attraction to magnetism.

For much of their careers, the Ames Laboratory scientists have developed magnetic materials that can be used in innovative, energy-efficient technologies.

Their latest development is a material that may steer automotive companies toward their goal of lighter, more fuel-efficient vehicles.

The researchers say a 1/4-inch-thick ring of the material could be used in an electronic torque sensor to regulate the steering power provided to a car's wheels by an electric motor. This would enable automakers to eliminate the heavy, energy-draining hydraulic system currently used in power-steering assists.

"Replacing the hydraulic power-steering system with an electrical system that uses this type of sensor should improve the fuel efficiency of a car by about 5 percent," says Jiles, senior physicist. Lighter, more energy-efficient vehicles would use less gasoline, conserving fossil fuels and reducing transportation costs, he adds.

Limited options

Jiles and McCallum, senior materials scientist, looked at a number of possibilities during the past five years as they searched for an inexpensive sensor material that met the specifications of the auto industry. They say only one viable option emerged: a composite consisting of cobalt ferrite (a compound of cobalt oxide and iron oxide) and small amounts of nickel and silver to hold the material together.

"I think we've looked into all of the possibilities, and it's difficult to conceive of a better material at this time," Jiles says. "The fact that it's also a relatively low-priced material makes it very attractive."

He says current power-steering systems use a hydraulic assist that requires continuous pressurization in order to sense and respond to steering changes. This produces a constant drain on the car's power, even when the steering wheel isn't being turned. "Also, the hydraulic system weighs a lot, so there's a significant weight reduction if you can replace it with an electrical system," he adds.

Sensor system

A sensor using a small ring of the cobalt-ferrite composite would be strategically placed on the steering column. As a driver turned the wheel, the magnetization of the cobalt-ferrite ring would change in proportion to the amount of force applied by the driver. The change would be detected by a nearby field sensor that would interpret how much force should be applied to turn the wheels and then relay the information to an electrical power-assist motor. Unlike the hydraulic system, the electrical system would consume minimal energy when the steering wheel was not being turned.

What makes the cobalt-ferrite composite ideal for this application is a property known as magnetostriction, Jiles says. Magnetostrictive materials undergo slight length changes when magnetized. Jiles and McCallum take advantage of that property, but in reverse. In their approach, the turn of the steering wheel would apply stress to the cobalt-ferrite ring, producing a change in the magnetic field it emits.

Cobalt ferrite maintains its magnetostrictive abilities throughout the temperature range specified by the auto industry, from minus 40 C (minus 40 F) to 150 C (302 F). Jiles says that's necessary because automakers don't agree on the best location on the steering column for the torque sensor. Some want it in the passenger compartment while others want it in the engine compartment, where it would be subjected to engine heat as well as winter conditions.

"High-class rust"

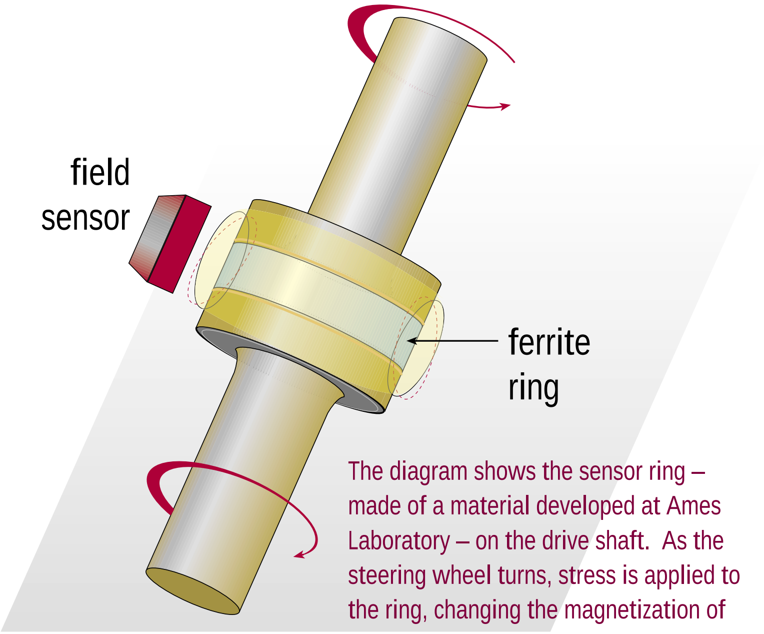
McCallum adds that cobalt ferrite also meets the strength and corrosion-resistance requirements for the sensor material. "This ceramic-metallic composite is similar in concept to materials used in high-strength tool bits where excellent mechanical properties are needed," he says. "And cobalt ferrite is basically high-class rust, so it's hard to corrode any further."

Jiles says the composite is also a cost-effective choice. While other materials may rank higher in terms of magnetostriction, they're too costly to be used in wide-scale production. For example, Terfenol-D is a rare-earth, magnetostrictive compound that Ames Lab helped develop in the 1980s. It possesses a much higher degree of magnetostriction, but can cost up to 100 times more than the cobalt-ferrite composite.

"If you normalize the measurements based on the cost of the different materials, you can see that our cobalt-ferrite material is far and away the best performer," Jiles says.

Dealing with difficulties

The five-year effort to develop an inexpensive sensor material that met the auto industry's needs had its share of frustrations and setbacks. Jiles and McCallum hit several roadblocks that sent them back to the drawing board. As part of the development process, they devised their own software and a unique test bed to measure the performance of the materials they were studying.



The diagram shows the sensor ring – made of a material developed at Ames Laboratory – on the drive shaft. As the steering wheel turns, stress is applied to the ring, changing the magnetization of the material. A field sensor detects the change, interprets how much force should be used to turn the wheels and relays the information to an electrical power-assist motor.

"We'd test some of the materials, and they wouldn't respond in the way that we had anticipated, so we would go back to understand why," Jiles recalls. "But now we have a practical material, and we also have computer simulations that can tell you how it will perform in engineering applications."

Much of the research on the sensor material was funded through a three-year, $820,000 grant they received in 1996 from the Department of Energy's Advanced Energy Projects Division. One of the DOE's primary missions is to engage in research that leads to the development of materials that improve the efficiency, economy, environmental acceptability and safety of energy sources.

Jiles and McCallum have applied for a patent on the cobalt-ferrite composite and plan to continue working with automotive manufacturers interested in using the material in an electronic torque sensor.

"When we began this project, we knew it was possible that there might not be a material that would meet the specifications," Jiles says. "It's satisfying that we've gone through this long search and come up with a material that works." ✻

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Research funded by:
DOE Office of Basic Energy Sciences

ILLUMINATIONS

by Saren Johnston

Excited uranium serves as a photosensitizer, absorbing visible light to drive the partial oxidation of saturated hydrocarbons.

Scientists may one day be using energy from light to activate sluggish hydrocarbons

She studies chemical reactions – that's her job.

The reactions she's investigating may one day lead to new chemicals that can be converted into plastics and other kinds of materials, but Andreja Bakac is focused on the fundamental chemistry that must first take place. Because, as she says, nothing happens without it.

The Ames Lab chemist's special interest is photochemistry – studying the effects of light on chemical reactions. While many photochemical reactions are initiated by ultraviolet light, Bakac is using visible light to do her experiments. Her research centers on the photooxidation of hydrocarbons to industrially important chemicals. But it's not just doing the chemistry that's her concern – it's also doing it in a simple, inexpensive and environmentally safe way.

"Lighting up" the lifeless

Hydrocarbons are chemical compounds that are composed mostly of hydrogen and carbon, and there are lots of them. If you think these compounds may not be relevant to you, think again. Each time you fill your car with gas or adjust the heat in your home, you might remember that petroleum and natural gas are mixtures of hydrocarbons. In fact, petroleum crude oil is the largest source of hydrocarbons.

Bakac's work focuses on saturated hydrocarbons, those that contain only single carbon-carbon bonds, making it impossible to attach additional elements or compounds.

"Were it not for the chemical inertness of saturated hydrocarbons, this class could become an important feedstock for the chemical industry," says Bakac. "They're so plentiful, yet so dead and useless." However, partial oxidation is one way to boot them out of their comfort zone and make them more reactive.

Chemical reactions have to be driven. Any reactions done in the lab must be activated in some way, and the standard method is by heat. This is known as a thermal reaction. But light can also drive chemical reactions and is sometimes a better choice.

Bakac explains that photochemical reactions have some major advantages. "Light is typically a lot cheaper than heat, and it's perfectly clean environmentally. You simply turn on a lamp or move to the window, and there's your energy source."

Of course, oxidation experiments require oxidants, chemicals that give up oxygen easily or remove hydrogen from another compound. Strong oxidants often oxidize things indiscriminately, pollute ground water and are expensive and difficult to handle. "Then you have to consider waste treatment and all the problems associated with it," Bakac reminds us.

Simple air is the oxidant in Bakac's hydrocarbon oxidation experiments. "Air or oxygen – that's as innocuous as oxidants get," she says with a smile. "That's what's so wonderful about these experiments and what makes them so easy."

In addition to light and air, Bakac's experiments need a photosensitizer – something to absorb the light so it can be used in the oxidation reactions. She uses a brilliant yellow aqueous solution of uranyl ions. Its yellow color is what allows the solution to absorb the visible light necessary to drive the reaction. As it absorbs light, the solution turns a neon green. The electronic structure of the uranyl changes, and the uranium becomes more reactive – a condition known as the excited state. The light makes a more energetic uranium that can photocatalyze, or set off, the oxidation of hydrocarbons. Left on their own, Bakac says the hydrocarbons would likely never react, and if they did, the reaction would be so out of control that it would produce many different products.

"Uranyl both increases the reaction rate at which hydrocarbons are oxidized and channels the reaction, leading to improved selectivity in the final product. We've gotten one major product in each of the reactions we've looked at," says a pleased Bakac.

"The way we conduct these experiments is so simple, inexpensive and safe," she continues. "We take a hydrocarbon and uranyl, dissolve them in water, bubble some oxygen or air through the solution, and turn on the lamp. During the reaction, we withdraw samples to monitor product formation."

Bakac notes that the word "uranium" is the only disadvantage of the hydrocarbon photooxidation reactions. "People are afraid of the word because they associate it with radiation pollution," she says.

Andreja Bakac checks to see that the stream of oxygen is sufficient and strong enough to help carry out the hydrocarbon oxidation reaction.

"The fact is, there is no reason to worry because in industrial applications of our experiment, depleted uranium would be used."

In depleted uranium, the amount of the fissile uranium – the isotope that splits apart – has been reduced to well below natural levels. And the United States has a lot of depleted uranium just waiting to be put to use. In Bakac's words, "It's there, it's available and it's safe."

Heterogeneous vs. homogeneous

As simple as the photooxidation experiments are, Bakac says she would still like to make the reactions more efficient. In all of her reactions, the uranium, oxygen and hydrocarbons get mixed together in a homogeneous solution, and nothing is saved when the reaction is over. "It would be ideal if we could take the uranium out, separate it from the final product and then reuse it," she says.

Before her photooxidation experiment can move from the lab to the real world, Bakac says she needs additional funding to develop a heterogeneous system that would make it possible to immobilize the uranium so that when the reaction was over, the catalyst could be removed.

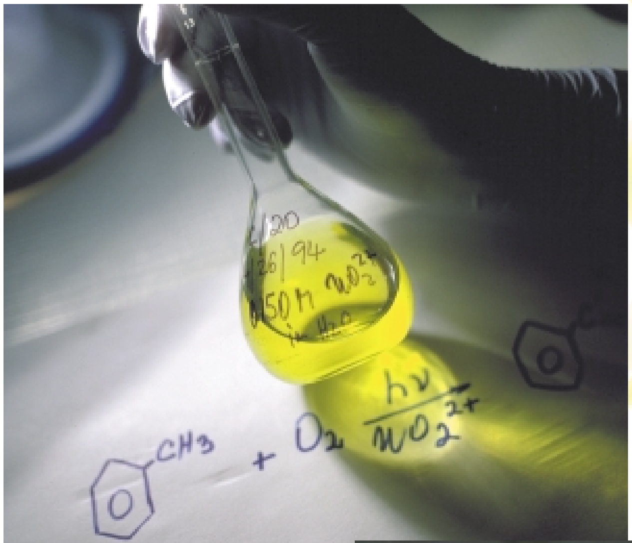
"I want to take a solid support and deposit the uranium onto it so that when it's put into solution the uranium will still stick to that support – it won't wash into the solution," she explains. "There would be a layer of uranium on some inert surface, which would need to have a lot of specific properties, such as certain pore and channel sizes. The molecules would have to be able to go in and come out and react with each other. The support would also have to be thin enough to let the light in."

Bakac says there are some tremendous advantages to the heterogeneous system and the ability to recover the uranium. "You have to be able to give assurances to the community you work in that you are not disposing of uranium in the ground water, soil or anywhere else," she says. "Also, recovering the uranium would make the whole process more cost-efficient. If you can pull the catalyst out and reuse it, that has to be a better way to go."

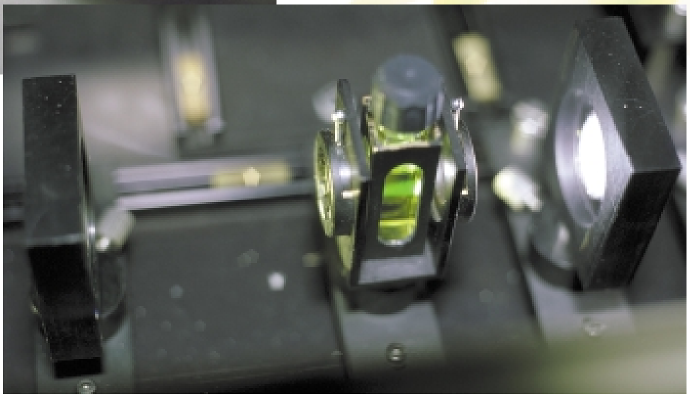
Bakac says that she and her co-investigators, Ames Lab physicist Marek Pruski and Iowa State University chemical engineer Brent Shanks, haven't yet done any experiments on developing a heterogeneous system, but adds that they're looking forward to working on it as funds become available.

Benzene to phenol and toluene to benzaldehyde

In the meantime, Bakac has had some noteworthy success with the photooxidation of a number of different hydrocarbons in homogeneous solutions. She and Yun Mao, a former Ames Lab postdoctoral fellow, were able to oxidize benzene to phenol, a process that is industrially very significant because phenol is an important starting chemical in the manufacture of such products as resins, paints, adhesives, phenolic plastics, synthetic fibers, herbi-



(left) Excited uranium is a catalyst for the photochemical oxidation of toluene to benzaldehyde.



(right) The light from an analyzing beam induces green luminescence of the uranyl ions during a photooxidation experiment.

cides and insecticides.

"Phenol is currently produced in a very cumbersome, multistep reaction," says Bakac. "Yet, we do it very nicely with our uranium and light system, slightly modified for this purpose."

The benzene-to-phenol reaction works great, according to Bakac. "It's a very clean reaction, and we get fantastic yield, which means light is being used very efficiently."

Another notable reaction that Bakac and her colleagues have performed is the photooxidation of the hydrocarbon toluene to benzaldehyde, a very important intermediate in the chemical industry. "The yield is not great," she says, "but this may change in the heterogeneous system.

"Excited uranium has been around for a very long time, yet people haven't tried to use it as a photocatalyst because some fundamental chemistry has not been understood. And you can't develop the application unless you understand the basics," Bakac states.

Methane to methanol

Although she has photooxidized several different kinds of hydrocarbons, Bakac is still hoping to conquer the most difficult reaction. "That would be the oxidation of methane to methanol," she says. "There are huge sources of methane available, but it presents some problems. It's a gas, so the problem of transport is a serious one. Also, it's so unreactive that it really doesn't do anything other than burn. And yet it would be an excellent source of methanol and all of the chemicals derived from it. So this first stage, the oxidation of methane to methanol, is an extremely active area of research right now."

Bakac says the reason they've been unable to oxidize methane to methanol is because the solubility of methane in water is very low, and the fundamental research experiments are done in homogeneous solution. She notes that they might be able to oxidize the methane in a high-pressure reactor equipped with the uranium support they plan to design. "The higher the pressure, the greater the solubility of gas in solvents," she explains. "Eventually we would reach a concentration where the methane would have to react. It's just that the reaction is slow right now, and the high-pressure reactor could speed it up by increasing the concentrations."

Bakac says she's excited about taking on the methane to methanol challenge. She hopes to obtain the necessary funding and get together with Pruski and Shanks to start working on the project.

"The initial effort is just to get the right support for the uranium and see if we can do any of the reactions we've already done in homogeneous solutions," says an enthusiastic Bakac. "It would be a major success if we could get anything to work in the heterogeneous phase at this time. If we could, hey, then anything is possible." ❋

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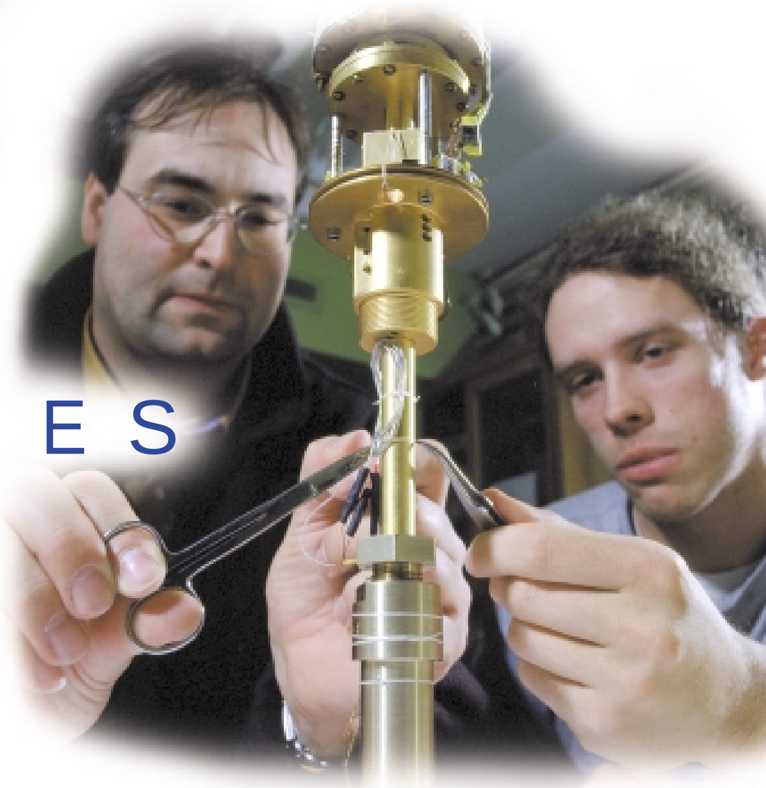
Research funded by:
DOE Office of Basic Energy Sciences

EXTREME MEASURES

By Saren Johnston



Robert Modler (left) and Andrew Thomas prepare an experiment to investigate materials under simultaneous extreme conditions of very low temperature, high pressure and high magnetic field.

Some very complex materials exist on the borderline between different states. Precariously perched, a little nudge is all they need to push them over the edge and drastically change their properties.

Studying the behavior of novel materials, especially those that sit on the brink of becoming something else, is Robert Modler's specialty. What he finds out about how they respond to changes in their environment helps Ames Laboratory researchers better understand these materials and determine their suitability for possible future applications within fields that include computer technology, communications, medicine and the automotive industry.

One of few

Modler, an Ames Laboratory associate scientist, investigates unusual materials under simultaneous environmental extremes of very low temperature, high pressure and high magnetic field. He does his research in a new facility that he designed and set up with Iowa State University graduate student Andrew Thomas. The two researchers spent about a year putting together the equipment for the facility, which houses a helium three-helium four dilution cryostat, a high-pressure cell and a high-field superconducting solenoid to simultaneously create the three extreme environments in one instrument.

"If you want to analyze and understand the behavior of a new material, ideally you would like to know as much as possible about how it reacts in this three-dimensional parameter space," says Modler. "While quite a large number of researchers use one or two of these parameters to investigate materials, only a few really put very low temperature, high magnetic field and high pressure together in one facility. I would guess there are probably only five comparable facilities in the world."

As part of Ames Laboratory's efforts to learn more about complex materials such as exotic magnetic molecules, quasicrystalline materials, semimetals and new magnetic superconductors, Modler subjects them to severe conditions in his lab of extreme environments. Observing how the materials behave in the facility's three-parameter space often reveals unique properties that could prove beneficial to the development of new technologies.

"Chilling out"

To get a good look at the material he's studying, Modler places a sample in the dilution refrigerator, which cools it until the material reaches its ground state – the low-temperature, lowest-energy state at which a material is almost completely free of excitations and vibrations.

The near absence of temperature disturbances in the ground state makes it easier for scientists to learn more about a material's properties. In the ground state, for example, there are fewer atomic vibrations to interfere with the behavior of the conduction electrons – you might even say that the material has "calmed down," which Modler says allows scientists to get a very good picture of how the electrons move about in the material. Alternatively, by increasing the temperature and taking the material out of the ground state, they can see what excitations develop and how they build up.

To achieve the ground state, the cryostat takes the material under investigation through temperatures that range from 300 K (room temperature) to 0.05 K (just above absolute zero). Some intriguing “how cold is cold” facts may provide insight on how chilly 0.05K might be.

“On the Kelvin scale, absolute zero, or 0K, is unreachable,” says Modler. “It would indicate the total absence of heat. As a comparison, our background universe has an average temperature of about 3K, which stems from cosmic microwave background radiation. In our lab, however, we reach 0.05K on a regular basis, which is one-twentieth of a Kelvin above absolute zero. It’s an interesting thought that temperatures this low have never naturally existed in our universe.”

Essential components of Ames Lab’s new combined extremes facility are (left to right): high-vacuum can, heat shield, dilution cryostat with attached high-pressure cell, and the superconducting solenoid.

Adding on

In addition to cooling a material dramatically, Modler can further alter its environment by applying high pressure of up to 20,000 atmospheres. Subjecting a material to high pressure alters the distances between its atoms and can strongly affect or even completely change its properties.

“One atmosphere is equal to the air pressure resting on the surface of the earth,” Modler explains. “Pressure of 20,000 atmospheres is equivalent to about 300,000 pounds per square inch. It can be thought of as approximately the weight of a Toyota Camry on the tip of a medium-sized Phillips-head screwdriver.”

The third parameter of Modler’s lab of extreme conditions is a powerful superconducting magnet for magnetic fields up to 100,000 times that of the earth’s. Just like high pressure, high magnetic field can cause abrupt changes in a material. It can, for example, change how magnetic moments are arranged in a material, causing a different magnetic alignment – a materials property perhaps most notably used in computer hard disks.

Just hanging out and hanging on

“The parameters of temperature, pressure and magnetic field are used for materials research by many scientists throughout the world. But putting all three together in one experiment might ‘scare’ even a tough material,” says Modler. He’s intrigued most by those materials that sit on the borderline between different states. These materials could stay in limbo indefinitely if their environment remained the same, but Modler’s not about to let that happen. His job is to shake things up a bit, and he does it very well.

“By applying just enough pressure, magnetic field or both to a ‘borderline’ material, we can push it over the edge into a new state and bring about drastic changes in its physical properties,” he says. “These materials are unusually complex; you might even call them ‘adaptive’ to their environments. By creating the right conditions, you can get a magnetic material to become superconducting or an insulated material to become metal. To improve our scientific understanding of such materials, we look very closely at how the changing states emerge from each other and how they interact at the borderline. That’s something interesting and not well studied to this point. And through our new facility, we can perform this research very comprehensively.” ✱

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From Guesswork to Formulas

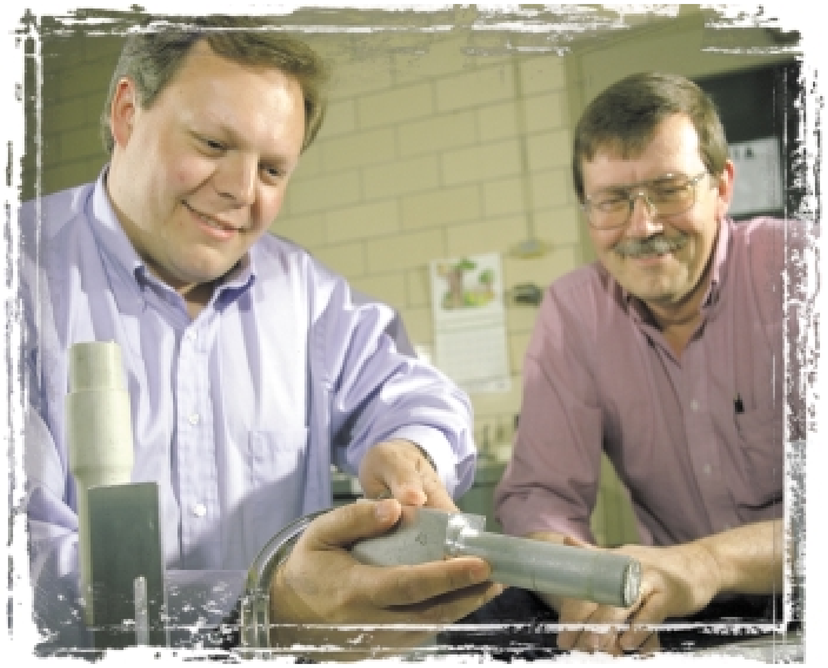
Researchers put a new spin on the study of sintered metals

by Danelle Baker-Miller

Before scientists develop ultrasonic techniques to view fetuses in vivo, old wives' tales were as good a method as any for determining a baby's sex. Did the mother eat peanuts after conception? Does the pillow on her bed face north?

Just as nondestructive evaluation methods like ultrasound have enabled doctors to safely gather more information about a baby before birth, they have made it possible for other experts to eliminate guesswork and understand more about their discipline.

Two Ames Laboratory metallurgists, Jim Foley and Dave Rehbein, have merged the fields of nondestructive evaluation and powder metallurgy, known as P/M, and their expertise. Though not new, the two areas combine to put a new spin on the study of sintered metals. Sintering is a solid-state process, unlike melting, in which compressed metal powder is heated to form a solid mass. During heating, the atoms intermingle across the powder contact surfaces to “heal” gaps, bonding the powders together.



Jim Foley (left) and Dave Rehbein examine an electromagnetic acoustic transducer, an in situ sensor that can measure the sintering process of materials in real time.

Foley and Rehbein explored options for applying ultrasonic techniques to study P/M sintering after Foley became leader of the Lab's Nondestructive Evaluation group a few years ago.

“P/M has needed an in-process sensor for a long time, but nothing works very effectively. When Jim became involved, we met to discuss using the two fields to improve on current techniques,” Rehbein says.

Powdered metals are commonly used today in mass-produced parts made of low-alloy steel, stainless steel, copper, brass and low-strength aluminum. The parts range from gears in automobile steering systems to mountings for rearview mirrors.

But how do engineers know when these bonds are at their optimal strength? How, in the arena of mass-produced parts, can they guarantee that all of the gears produced in a day have a particular strength without breaking them apart to examine their properties?

Enter the electromagnetic acoustic transducer, the tool Foley and Rehbein are exploring for its potential in P/M.

Much like ultrasound on a fetus, EMAT enables them to evaluate bonds in powdered metals in real time, as they sinter in a furnace. The tool's primary benefit is the elimination of wasted time and materials required by the current destructive examination of prototype parts. The data the two researchers can acquire from using EMAT could turn guesswork into formulas engineers could use to predict processing conditions when mass producing parts.

Currently, the work of developing those formulas can be inefficient and time-consuming. Foley says EMAT is a mechanism for smoothing out the rough path of trial and error.

“In an ideal world, you wouldn't need to do any sintering experiments. You'd have an equation and plug in variables to determine the correct processing conditions. But to get to that stage, you need a method for measuring sintering. That's what EMAT is,” he says. “In sintering, the major factors are the properties of the metal, time and temperature. But as with any science, we need to know how these factors interact to create the strongest parts."

EMAT has long been used to detect internal flaws in metals. David Hsu, a scientist at Iowa State University's Center for Nondestructive Evaluation, has used the technique to study airplane wings made of composite materials. To make the paper and resin wings electrically conductive, he covers them with aluminum foil.

"A standard EMAT can be used on anything that conducts electricity, but normally, the technology won't work under high temperatures because heat deteriorates the strength of its permanent magnets," Rehbein explains.

The commercial ultrasonic tranducers Foley and Rehbein use were custom-built by Ron Alers of Sonic Sensors, a company in San Luis Obispo, Calif., to tolerate temperatures up to approximately 600 C (1,112 F). Unlike other ultrasonic techniques that require a couplant of water or jelly, EMAT is noncontact and requires no couplant. Their EMAT unit uses two pairs of permanent magnets and electrical coils, one on either side of a pressed powdered-metal part as it rests in a sintering furnace. The magnetic and electrical fields produce eddy currents that create sound waves inside the metal.

Rehbein likens the ultrasound in EMAT to a microwave oven. "The magnetic and electrical fields come in combination from outside the sample but produce sound pulses inside the sample. Microwaves operate much the same way, by creating heat inside the food rather than by heating the oven itself," he says.

Inside the sample, sound pulses respond to the strength of the metal's bonds by dying out when bonds are weak or bouncing back and forth in pinball fashion when bonds are strong.

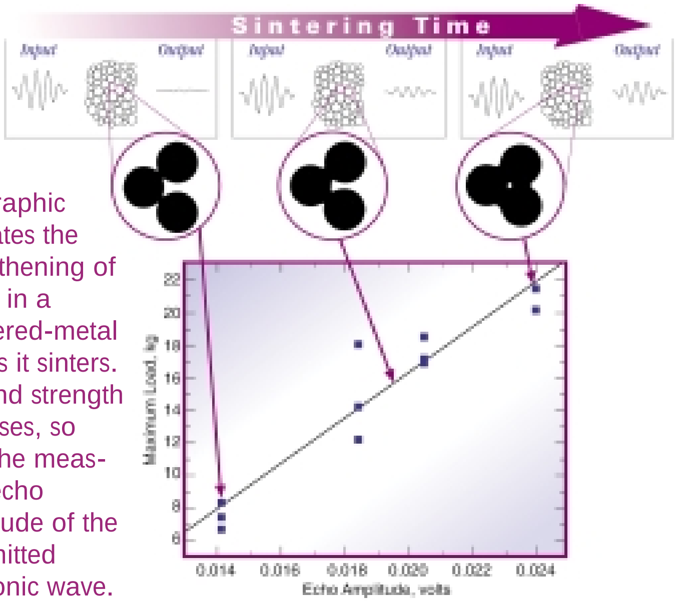
"The weaker the bonds, the more difficult it is for sound waves to propagate. The echo amplitude is low and output in the receiving end of the EMAT probe is weak," says Foley. "But when the bonds become stronger, the wave output more closely resembles the input. We can see much higher peaks on the monitor. In a 100-percent dense piece of sintered metal, the echo amplitude of the input wave would be nearly identical to the output wave," he explains.

On the way out, the sound waves, in combination with the magnetic field, produce electrical voltage that is measured with an oscilloscope and observed on a computer monitor.

Foley and Rehbein are confident they are on the right track – tests show a nearly 100 percent correlation between measured output voltage and the load-bearing capacity of aluminum-copper alloys containing different percentages of silicon carbide after the alloys were cooled and removed from the furnace.

"The data showed an absolutely wonderful correlation," says Rehbein. "I had a hard time believing how well this technique works. Whatever it is in a metal's atomic structure that is controlling strength, it appears to also control echo amplitude. We're going to work with Bruce Thompson, director of ISU's Center for Nondestructive Evaluation, to better understand the connection."

Foley underscores that the EMAT-P/M combination is breaking new ground. "No one else has been able to evaluate P/M parts this way. This is very new science. With EMAT, we can experiment and determine when time and temperature are ideal to create powdered-metal parts strong enough to handle the maximum loads. Eventually, technology like EMAT could be expanded for use in production, even evaluating sintered parts on an assembly line," he says.



The graphic illustrates the strengthening of bonds in a powdered-metal part as it sinters. As bond strength increases, so does the measured echo amplitude of the transmitted ultrasonic wave.

Though EMAT could eventually see the interior of automotive factories, it won't lose its value as an experimental tool. "The technique will still be important as an experimental tool for new alloys. It will always be used as a feedback mechanism to be certain that a metal's properties are as good as they should be," Foley says. The team plans to publish the sintering models they develop. ✻

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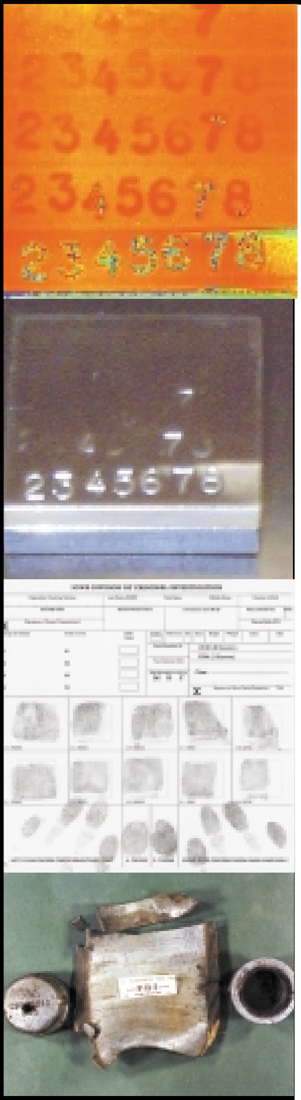
A Helping Hand for Midwest Crime Labs

By Susan Dieterle



Among the current forensic projects at Ames Lab and IPRT are restoring serial numbers (top photo) that have been removed from hard metals (second photo); better fingerprint detection methods (third photo); and analyzing bomb-blast fragments (fourth photo).

In the Midwest, as in most of the United States, crime laboratories struggle with growing caseloads, personnel shortages and a chronic space crunch. Criminalists are challenged with analyzing evidence as quickly as possible, leaving them little time to develop new equipment and techniques that could enhance the speed and accuracy of their investigations.

That's where Ames Laboratory and Iowa State University's Institute for Physical Research and Technology hope to come in. The two organizations are collaborating on a proposal to establish a regional forensics support and research facility at ISU.

The proposed Midwest Forensics Resource Center would draw on the expertise of faculty and staff members at Ames Laboratory and IPRT, a network of multidisciplinary research and technology-development centers on the ISU campus. Ames Lab is part of the IPRT network.

The forensics proposal received $44,000 in seed funding from IPRT to begin developing the partnerships needed to launch the facility, and organizers are working to identify funding sources for the center.

David Baldwin, director of the Lab's Environmental and Protection Sciences Program, says the forensics center would serve three main purposes. First, it would be an arena for the development of new analytical techniques and tools for forensic investigators as well as providing an outlet for technologies developed at Ames Lab and other DOE laboratories.

Second, Iowa State students could work at the facility to gain valuable training prior to graduation, making them more attractive as potential employees. Finally, the center would serve as a regional training facility and resource for local and federal agencies.

The regional aspect of the forensics center will be crucial to its success, Baldwin says. "We don't want to be a burden to any of the states in the Midwest, especially Iowa," he says. "We don't want to be a drain on resources that might otherwise go to the crime laboratories. The center needs to be a regional laboratory that's funded nationally."

Scientists at Ames Lab and IPRT have already begun laying some of the groundwork for the center through forensics research projects for the FBI, DOE and the Iowa Division of Criminal Investigation.

To find out how the proposed center could best serve crime laboratories, Baldwin and others involved in efforts to establish the center met with forensic investigators from eight Midwestern states and four federal agencies in May.

The crime-lab representatives gave input on the types of research, services and training that would be most helpful to their facilities and suggested funding sources for the proposed center.

Carl Bessman, a criminalist with the Iowa Division of Criminal Investigation, says the research assistance his crime lab has already received from Ames Lab and IPRT has proven valuable. "Ultimately, applying good science to the casework is our most important responsibility," he says. ❋

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(left) U.S. Department of Energy Secretary Bill Richardson gets a hands-on demonstration of magnetic refrigeration during his visit to Ames Laboratory.

(below) Ames Laboratory celebrates the 10th anniversary of the DOE National Science Bowl competition. Forty-seven Iowa high schools participated in the annual Ames Laboratory/Iowa State University regional event.

(above) Gen. Eugene Habiger, director of DOE s Office of Security and Emergency Operations (center, with white name tag), tours Ames Laboratory s machine shop.

Inquiry

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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, mathematical and physical sciences. 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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