# INquiry

*Science and Technology at the Ames Laboratory*

2001

**New superconductor**
**is on the scene**

**Chemical separation technique**
**wins R&D 100 Award**

**Polymers and quasicrystals —**
**an unlikely mix**

**Novel metal filter**
**cleans up**

AMES LABORATORY

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

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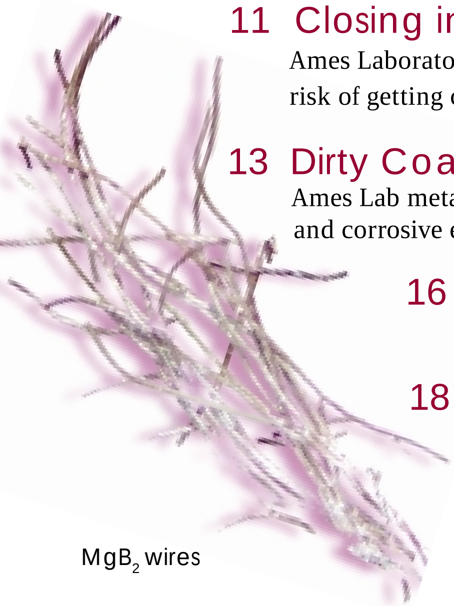

$MgB_2$ wires

polymer solution

(front cover) Magnesium diboride, or $MgB_2$, wire segments, called "angel hair" because the growth looks like angel-hair pasta, are pictured near a U.S. dime for scale. [*Physical Review Letters* 86 (2001) 2423]

(back cover) New high-performance polymers at various stages, including the starting crystalline materials, a liquid polymer reaction mixture and a final, isolated solid polymer.

# from the Director

We begin this year's edition of *Inquiry* magazine with congratulations to Ed Yeung, who has won a 2001 R&D 100 Award. Ed's chemical separation technology potentially could revolutionize the diagnosis of diseases and development of treatments. This is Ed's fourth R&D 100 Award and the 14th for Ames Laboratory.

Ryszard Jankowiak and Gerry Small are also making headway in the war on disease. Their new biosensor chip technology can be used with urine samples to obtain immediate information about DNA damage from carcinogens, providing valuable information for cancer risk-assessment.


On the materials front, our physicists made quite a splash in the scientific and popular press when they became the first to describe the mechanism of superconductivity in magnesium diboride. Then the physicists went a step further by mapping the material's properties and creating the first-ever magnesium diboride wire segments.

Another material has been of special interest to two other Lab scientists. They have developed a process that could finally offer industry a commercially viable way to recover neodymium from the tons of magnetic scrap piled up in warehouses across the country awaiting recycling. The method they've discovered is simpler to use and less expensive than existing technology — two attributes that are music to the ears of industry.

Also on the environmental front, Iver Anderson has developed a new metallic filter for use in power plants. The new filter, which is made out of a nickel-chromium-aluminum-iron alloy, would allow the next generation of coal-fired power plants to cleanly burn high-sulfur, dirty coal. More complete combustion in these plants removes most of the harmful gases, while the filter prevents corrosive and abrasive particles in the exhaust gas from reaching and damaging the power-plant turbines.

These are just a few of the fascinating research efforts underway at Ames Laboratory that you'll read about in this issue of *Inquiry*. I invite you to review the many other ways in which our scientists are working to find solutions to our nation's energy problems.

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Editor
Steve Karsjen

Managing Editor
Saren Johnston

Art Directors
Michael Marti
Janice Weedman

Photographer
Dennis Sailsbury

 For additional information about Ames Laboratory or topics covered in this publication, please contact:

Editor, *Inquiry*
Ames Laboratory
111 TASF
Ames, Iowa 50011-3020
(515) 294-9557

www.external.ameslab.gov

# *Old Material Makes a* New Debut

*by Saren Johnston*

For the most part, it was a wallflower for 50 years. Plain and simple, no one paid it much attention. No one suspected there might be more to the nondescript tan powder than met the eye.

Magnesium diboride is a relatively inexpensive metal compound that can be purchased in powder form from most standard chemical supply companies. Until this year, there was nothing remarkable about it — at least nothing that was known. But the material had never been investigated for superconductivity — whether it had the ability to conduct electricity perfectly, without resistance, when cooled to temperatures near absolute zero (minus 459 degrees Fahrenheit).

That all changed in January when Jun Akimitsu of Aoyama Gakuin University in Tokyo announced he and his research team had discovered that magnesium diboride becomes superconducting at 39 Kelvin (minus 389 F), nearly twice the temperature of current intermetallic, low-temperature superconductors. The news had experimentalists around the world rushing to duplicate and confirm the Japanese findings.

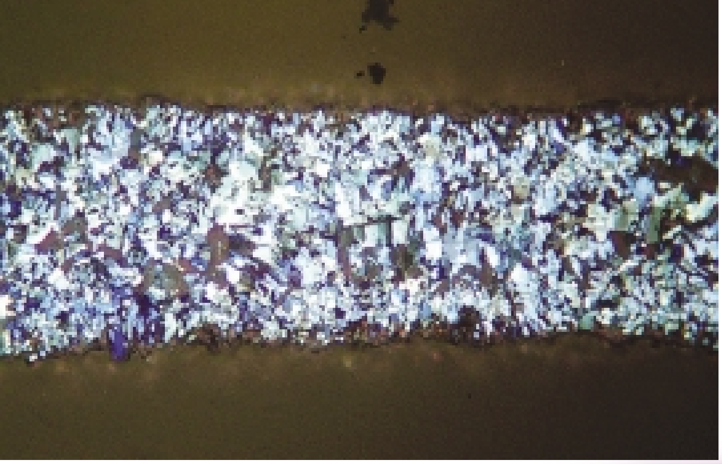
But a team of Ames Laboratory physicists, including Paul Canfield, Doug Finnemore and Sergey Bud'ko, was the first to describe the mechanism of superconductivity in the material. "One of the things that allowed us to make progress quickly was that we figured out how to make high-purity powders of magnesium diboride simply, in a two-hour, turnaround process," says Canfield. "We ran several cycles a day, and then made isotopic substitutions." (Isotopes of an element have the same number of protons, but differ by how many neutrons are within each atomic nucleus.)

## Figuring out the superconductivity

The physicists were very quick in getting the highest purity samples of $MgB_2$ with the highest transition temperature — the temperature at which a material becomes superconducting. Their experiments showed that $MgB_2$ sample pellets containing boron isotopes with an atomic mass of 11 became superconducting at 39.2 K (minus 389 F), while pellets containing boron isotopes with an atomic mass of 10 became superconducting at 40.2 K (minus 387 F). By changing the mass of the boron, the physicists saw a 1.0 K upward shift in transition temperature. "We wanted to understand the mechanism of superconductivity in the material," says Canfield. "And we found that the shift in transition temperature caused by the change in boron mass is consistent with standard models of intermetallic superconductivity."

Conventional intermetallic superconductors conform to the Bardeen, Cooper, Schrieffer theory of superconductivity, commonly called the BCS theory, which explains that the electrons in superconductors are grouped in pairs and that the movements of all the pairs in a single superconductor are interrelated — they make up a system that acts as a single entity.

When an electrical voltage is applied to a superconductor, all the electron pairs move together coherently as a unit. The movement creates a current that will flow indefinitely, even when the voltage is removed, because the electron pairs meet no opposition. This pairing takes place because the electrons are attracted to each other by creating and detecting vibrations in the lattice. These vibrations determine a material's superconducting transition temperature. The smaller the mass of the superconducting material, the higher the frequency of the vibrations and the higher the transition temperature.



Exposing boron fibers to magnesium vapor for approximately 120 minutes results in $MgB_2$ wire segments that are approximately 98 percent dense.

If a superconductor is allowed to warm up past its transition temperature, the electron pairs separate and the superconductor becomes a normal conductor. In all previously studied intermetallic compounds, the electron pairs usually break apart when the temperature climbs above 20 K (minus 424 F). But, amazingly, the electron pairs remain together in $MgB_2$ up to 40 K (minus 387 F).

The 1.0 K shift in superconducting transition temperature produced in the boron isotope experiments of Canfield and his colleagues is strong evidence that the electrons in $MgB_2$ are interacting as described in the BCS theory of superconductivity. This would classify $MgB_2$ as a conventional superconductor, but with an extremely high and unconventional transition temperature. And this is, as Canfield says, "a big, hairy deal."

A cross-section of $MgB_2$. Note: cross section also reveals the central tungsten boride core of the original boron fiber.

## How good is $MgB_2$?

After addressing the mechanism of superconductivity in $MgB_2$, the experimentalists then mapped out the basic properties of the material. "We do this for every material that comes down the pike," says Finnemore. "We wanted to see to what fields $MgB_2$ remains superconducting, what types of currents it can carry in the superconducting state, and how much current it can carry through the grain boundaries in the material. So we did conventional measurements on what we thought was an exotic material, only it turned out to be kind of ordinary — the exotic thing is its superconducting temperature."

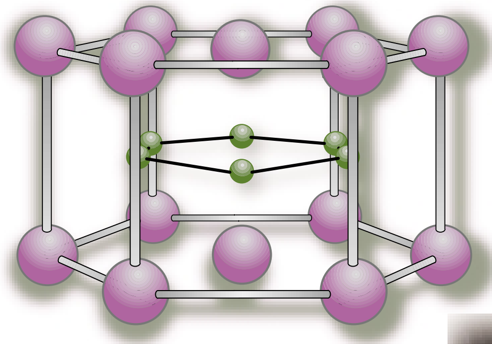
Data the researchers collected on the properties of the $MgB_2$ pellets showed the intermetallic material would carry enormous electrical currents, even though the currents had to jump across a dense array of grain boundaries. This property is far different from that found in ceramic high-temperature superconductors.

Ceramic superconductors operate at higher temperatures than the intermetallics, some over 100 K (minus 280 F), and they require less costly cooling arrangements. But intermetallic compounds are better at carrying current across grain boundaries, making it easier to synthesize superconducting wires that can carry large currents.

## Wonderful wire

Soon after mapping the properties of $MgB_2$, the experimentalists took on and met the challenge of creating wire from the material, starting with boron fibers. In essence, the researchers came up with a way of turning straw into gold. They placed the boron fibers in a tantalum tube with excess magnesium and heated it up. "As the magnesium vapor diffused, the boron sucked it up — what we were able to do was turn boron fibers into magnesium diboride wire," says Canfield. "We've been making lengths of five centimeters (2 inches). We call the growth 'angel hair' because it looks like angel-hair pasta."

The researchers found that $MgB_2$ wire has as sharp and as high a



The crystal structure of $MgB_2$.

(left to right) Ames Laboratory researchers Sergey Bud'ko, Paul Canfield and Doug Finnemore are moving forward with their research on magnesium diboride, making longer wires of the record-setting, low-temperature superconductor.

superconducting transition temperature as the powders they developed and that the wire is at least 80 percent dense. Development of the wire also allowed the researchers to measure the material's resistivity — its ability to carry electricity in the nonsuperconducting state. The measurements revealed that $MgB_2$ has a resistivity approximately 20 times lower (better) than that of the reigning niobium-based superconductor at its transition temperature.

"We made the wire in five-centimeter segments, but boron monofilaments are made in kilometer lengths. This opens the possibility of developing a continuous process in which boron monofilament is made and then transformed into this wire," says Canfield. But he cautions, "Magnesium diboride is brittle."

Finnemore adds, "You can't spool it and give someone a mile of the stuff. It will present challenges to coat and protect the wire, similar to those for previous intermetallic superconductors containing niobium and tin."

The work of the Ames Lab physicists to understand the physics of $MgB_2$ progressed at an amazing pace. Following the January 10 announcement of superconductivity in the material, the Ames team addressed the mechanism of superconductivity in the material, mapped its properties and developed wire segments along with what appears to be a means of making long wires — all in a month's time.

Canfield is quick to point out that there is much more work to be done, but he notes that $MgB_2$ holds promise for being the next low-temperature superconductor of choice. Because of its higher superconducting transition temperature, wire made from the material would not have to be cooled to temperatures as low as for niobium-based superconductors, reducing cooling costs associated with the quantities of liquid helium required to operate such systems. Also, there is a greater abundance of magnesium and boron than niobium.

The bottom line, according to Canfield: "With magnesium diboride, we have something that maps onto the existing technology of intermetallic wires, but with a factor of two higher in transition temperature. However, only time will determine the material's usefulness." ◆

**For more information:**
Paul Canfield, (515) 294-6270
canfield@ameslab.gov

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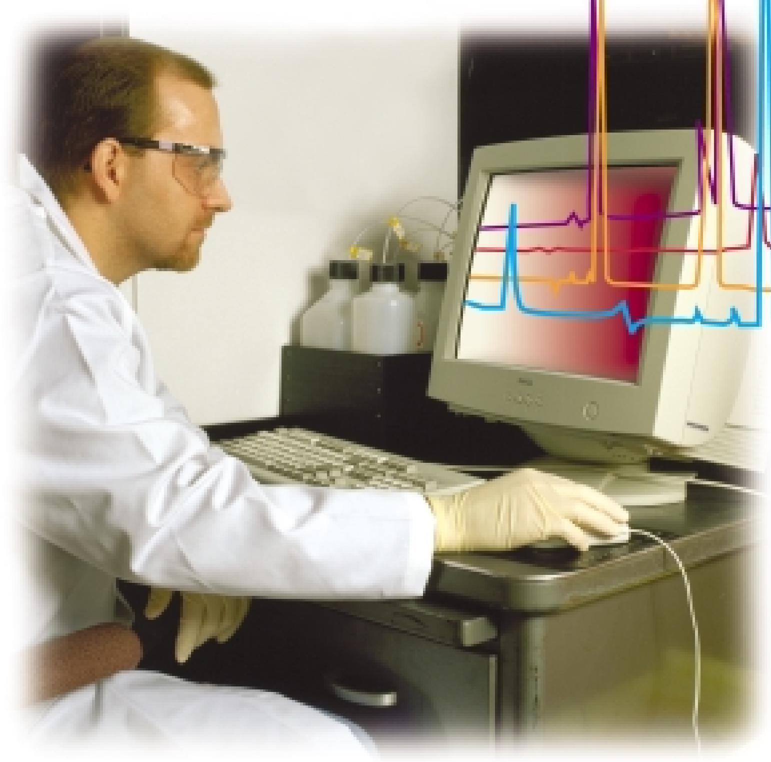
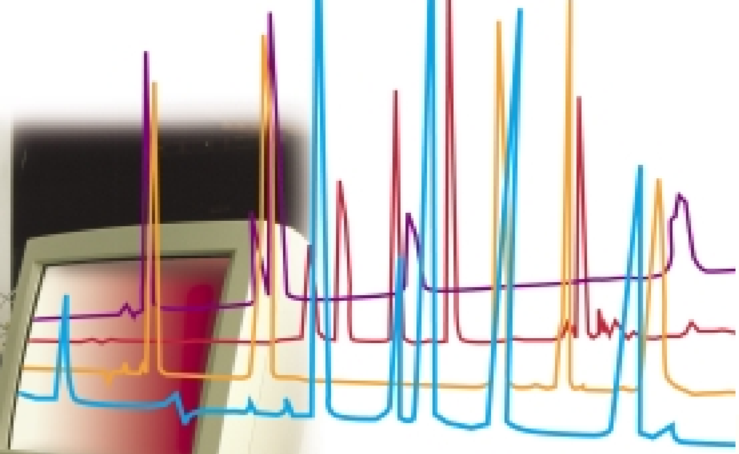




# *Molecular Roller Coaster* Analyzes Compounds

Jeremy Kenseth, a former Ames Laboratory scientist now with CombiSep, Inc., evaluates output from the MCE 2000, a multiplexed capillary electrophoresis machine based on technology developed at Ames Laboratory.

*by Robert Mills*

Visualize a pack of various molecules in a compound, all mingling in a solution. Then imagine molecules being whisked away from the crowd, one at a time, into a tiny tube by an invisible force. About halfway through this molecular roller coaster, the molecules are flashed with ultraviolet light beamed through a small window as they scream by.

Although the molecules may or may not be having fun, the ride is quite revealing from a chemical viewpoint. The type and quantity of molecules in the compound can be determined from the time it takes various molecules to take their ride and from the amount of ultraviolet light they absorb. Imagine, then, the vast quantity of information that can be gathered by running 96 of these roller coasters at the same time.

That, in short, describes the operation of a new instrument created by Ed Yeung and his team of researchers at the Ames Laboratory. Called multiplexed capillary electrophoresis using absorption detection, this method promises to be faster, better and cheaper than alternative technologies.

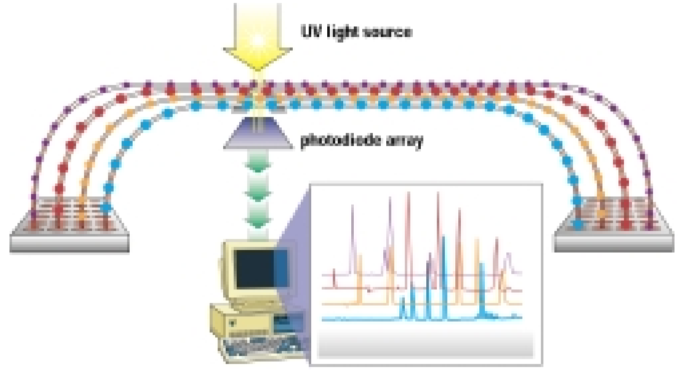
This breakthrough is only the latest from Yeung, director of Ames Lab's Chemical and Biological Sciences Program and a distinguished professor of chemistry at Iowa State University, who has a long history of innovations in analytical chemistry. However, Yeung is particularly excited about this technology, as it has wide-reaching potential.

"Any kind of chemical measurements that involve separation can, in principle, be fitted to use this technology," Yeung says. Indeed, such technology has applications in several burgeoning fields, including combinatorial chemistry, drug discovery, genomics and proteomics (the study of protein expression and function). Such instruments are finding increasing utility in pharmaceutical, genetics, medical and forensics laboratories, and may someday be used in doctors offices.

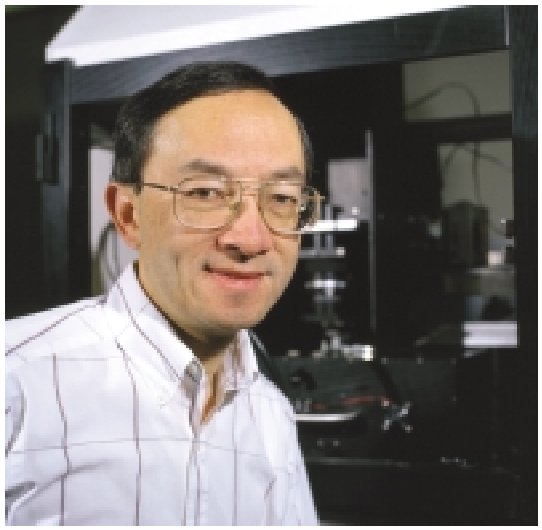
## By size and by charge

Capillary electrophoresis works on the same principles as gel electrophoresis, commonly used in DNA fingerprinting. An electrical current (the invisible force) causes molecules to migrate at different speeds, according to size and charge. The capillaries are silica tubes about 2 feet long, with an inside diameter of 75 microns (about the diameter of a human hair). Because these capillaries can disperse heat so well, an electrical charge of up to 20,000 volts can be used. The high voltage means separations can be done in as little as 15 minutes. "Absorbance detection" describes how the system detects molecules. Ultraviolet light is focused through a tiny window to illuminate the capillaries, and the amount of light absorbed by the migrating molecules is detected by an array of photodiodes. Absorbance detection makes Yeung's approach more widely applicable than other forms of capillary electrophoresis.

The ultraviolet absorption data, along with the time it takes the molecules to migrate, are sent to a standard personal computer. Custom software processes the data to generate charts called electropherograms, revealing the type and quantity of the separated molecules. The data can also be exported in tabular form to standard software packages.




"Multiplexed" means that the machine runs several samples at a time by using multiple capillaries. The machine works with 96-well titer plates to fit standard laboratory equipment. The result is automated, high-throughput analysis, a much-sought-after technology in today's biotech industries.



Ed Yeung's quest to improve chemical separations led to development of multiplexed, absorbance-based capillary electrophoresis.

## Faster, better, cheaper

Alternatives to multiplexed capillary electrophoresis with absorption detection include high-performance liquid chromatography and capillary electrophoresis with fluorescence detection.

"HPLC is a widely accepted practice," says Shelley Coldiron, president and co-founder of CombiSep, Inc., a new company formed to turn Yeung's technology into a commercial instrument. "We have to educate people and let them know we can operate comparable to what they're getting with HPLC."

Another factor working in CombiSep's favor is that its technology uses less solvent than HPLC. "We use 1,000 times less solvent than HPLC," says Coldiron, noting that this results in lower environmental hazards and related costs. Although HPLC systems cost much less than CombiSep's machine, they are limited to one analysis at a time. "We can take 96 of those individual instruments and do the same analyses with one of ours," says Coldiron.

Capillary electrophoresis systems that use laser-excited fluorescence detection are also made in multiplexed versions that can run 96 analyses at a time. These machines play an essential role in DNA sequencing; in fact, Yeung won an R&D 100 Award for developing just such an instrument in the 1990s.

Yeung's latest technology, however, can handle a wider range of compounds, thanks to its ability to detect molecules using ultraviolet light. "Only about 10 percent of all compounds fluoresce naturally," says Coldiron, "so you have to put optical tags on those compounds that don't fluoresce naturally." The "optical tags" are fluorescent dyes that are expensive and may be toxic.

What's more, multiplexed systems can be slow at processing all the data that results from running 96 simultaneous analyses. "It's kind of self-defeating. You're generating all this data but you get bottlenecked in data processing," Coldiron says. To sidestep this hurdle, CombiSep has written software that can process the data in approximately 15-30 minutes, according to Coldiron.

## "Right here, and quickly"

Yeung began his Ames Lab research on the technology in 1998 when he realized there were limitations on existing capillary electrophoresis technology. Things progressed quickly from there. "At the point of publication, we realized it would be commercially viable, so we filed for a patent," says Yeung. Yeung soon helped start what became CombiSep to develop and sell instruments based on the technology. "I realized we needed to move extremely fast," he says. "We had to do something right here, and quickly."

In addition to Yeung and Coldiron, the company's founders include Marc Porter, an ISU chemistry professor and director of ISU's Microanalytical Instrumentation

In capillary electrophoresis, an electric charge causes molecules to migrate down capillary tubes at different speeds according to size and charge. A combination of ultraviolet light and photodiodes are used to detect the molecules. The data is then processed and displayed in graphs called electropherograms.

Center, serving as vice-president; Steve Ringlee, an area entrepreneur; and Roy Strasburg, director of R&D.

The market for high-throughput analysis technologies such as multiplexed capillary electrophoresis is "hot" right now, according to Coldiron. CombiSep is striving to get into this market as quickly as possible, especially as it is competing with several large companies in the fast-growing, multibillion-dollar market for these instruments. "There's always someone out there developing similar ideas," Coldiron says.


The MCE 2000 is a multiplexed, absorbance-based capillary electrophoresis system made by CombiSep, Inc. The system can perform 96 separations simultaneously through its array of 96 capillary tubes (inset).

## Prototype to product, pronto

For such a complex technology, CombiSep has moved extremely fast. Although officially founded in December 1999, the company really did not get rolling until April 2000. Since then, the company has made and tested several prototypes of its MCE 2000 multiplexed capillary electrophoresis instrument. CombiSep shipped its first machine to Procter & Gamble Pharmaceuticals for evaluation in late February 2001. Five instruments are in various states of assembly. Moveover, the company raised $1 million in start-up funds.

CombiSep has been able to move quickly for several reasons, according to Coldiron. First, Yeung had already proved that the technology would work. Second, CombiSep has been able to hire top-notch technologists.

Ed Yeung received a 2001 R&D 100 Award for development of multiplexed capillary electrophoresis technology, making him a four-time R&D 100 winner. The *R&D Magazine* program, now in its 39th year, honors the top 100 products of technological significance marketed or licensed during the previous calendar year. Since 1984, Ames Laboratory has earned 14 of the prestigious awards.

These include the technology's co-inventor, Ho-Ming Pang, as well as founder Strasburg and Jeremy Kenseth, both Ph.D. chemistry graduates from ISU. Third, the company has contracted resources and expertise from Ames Lab and ISU to accelerate its development rather than hiring its own staff. ISU's Center for Advanced Technology Development and Center for Industrial Research and Service have both assisted the company.

CombiSep also contracted with Ames Lab's Engineering Services Group to fabricate various components and to design and build the electronics that go into the machine. Jerry Hand, supervisor of the Lab's mechanical development shop, says the CombiSep project is "pretty much" like the others his group tackles. Much of the effort consists of fabricating components made of aluminum and other materials using the shop's lathes, mills and wire electrical discharge machining system. Hand says his entire group has worked on the CombiSep project. In addition, the electronics tech shop of the Lab helped design the electronics for the prototype. ◆

**For more information:**


Ed Yeung, (515) 294-8062
yeung@ameslab.gov



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# Polymers *Plus* Quasicrystals *Equals* *A Puzzling Interaction*

*by Mary Jo Glanville*

Valerie Sheares examines the color and viscosity of a newly made polymer.

Sometimes trying something that really shouldn't work can lead to an amazing discovery. That's what happened to Valerie Sheares, an Ames Laboratory associate and Iowa State University assistant professor of chemistry. The discovery, a polymer-quasicrystal composite, has the best characteristics of each of the constituent parts. It's opened the door for a variety of innovative uses. Why it works, however, remains a puzzle — one that Sheares is eager to solve.

It was her expertise with polymers combined with an introduction to quasicrystals that kindled Sheares' curiosity. Shortly after arriving at Iowa State in 1996, she heard Pat Thiel, Ames Lab Materials Chemistry Program director, discuss quasicrystals and their unique properties.

Discovered in the 1980s, quasicrystals typically are aluminum-rich alloys of specific compositions. They are extraordinarily hard, have low coefficients of friction under certain test conditions, and don't conduct heat as well as most metals. "All things that in one material are pretty outstanding, especially considering that the material is a combination of common metals," Sheares says. "They exhibit properties not found in the constituent metals, but when you put them together in the right combination, under the right conditions, you get this outstanding set of properties."

## Enticing possibilities

As Thiel described some of the unique properties of quasicrystals, Sheares started thinking about the possible benefits of combining quasicrystals with polymers. Polymers — large molecules that consist of repeating chemical units joined together — typically are filled with all kinds of materials, such as calcium, silica, alumina or carbon black. "Almost no commercial plastic is unfilled," Sheares explains. "There are all kinds of things mixed into a polymer. The polymer really becomes the matrix that is holding the filler together. These other things are dispersed throughout to alter the properties." As a result, polymers are used in a wide array of products, everything from grocery sacks to automobile tires.

In addition, polymer processing is well understood, according to Sheares. They are processed into films, fibers and shapes used in thousands of products. Even in the planning stages, the benefits of a material with the unique quasicrystal properties that could be processed as easily as polymers had enticing possibilities.

## An improbable mixture

The fundamental question of whether polymers and quasicrystals would mix, however, was not guaranteed. "We tried things that shouldn't have worked, but we tried them because we were just thinking from a polymer standpoint," Sheares explains.

In particular, polymers don't like to mix with each other, let alone with other materials. Coupling agents generally are used to increase the adhesion or interaction between the polymer and the filler by attaching to both surfaces, but the nature of the quasicrystal surface prevents the coupling agent from being able to attach.

"Quasicrystals have a low-surface energy — they don't like contact with other things. It's like Teflon®, that's a low-surface energy material, so things do not stick. That gave us the idea that these things aren't going to want to mix with themselves, much less with polymers," she notes.

Considering these barriers, Sheares' experiments showed surprising results. "We dissolved the polymer in a solvent and added it to a quasicrystal powder. We wanted to see it disperse in the liquid," she says. Even though the heavier, metallic powder sank, it was evenly dispersed on the bottom with no clumping together. That was the first indication that the two materials would blend rather than run away from each other.

The next step was to mix a polymer powder with a quasicrystal powder and to process the mixture using compression molding, one of the most common techniques for fabricating materials. The mixture was poured into a mold, and heat and pressure were applied, causing the polymer to become molten or soft. The research team of Sheares, Paul Bloom, an ISU graduate student, and K.G. Baikerikar, an Ames Lab scientist, worked diligently, adjusting the timing and temperature to maximize how evenly the particles dispersed before the material was cooled down. Using electron microscopy, the researchers examined the new composite, slicing into it to confirm that the quasicrystals were evenly dispersed throughout. This characteristic is key because the closer the interaction between the polymer and the filler, the stronger the material. Tests confirmed that the composite's mechanical properties were increased, yet the thermal properties remained unchanged, verifying that in these tests, the quasicrystals behaved just like other hard fillers used in polymers.

In jars, from left: a polymer solution, isolated polymer and pure quasicrystal filler are the ingredients for a new composite that is highly resistant to wear, nonabrasive and easy to process.

## Amazing combination

Next, the researchers examined the wear properties of the composite. Essentially, the tests involve a steel ball held in contact with the composite while the composite rotates on a turntable. Then the composite and steel ball are examined for wear. The results were impressive — quasicrystals, as a filler, significantly improved the wear-resistance of the polymers.

Even more amazing, the composite exhibited a nonabrasive characteristic — neither the composite nor the steel ball showed significant signs of wear. "Almost everything that is hard is also abrasive. If the material itself doesn't wear, it's probably wearing very hard on the other surface. Quasicrystals in polymers are not like that. They are not scratched, and they don't scratch other surfaces," Sheares explains.

While the first polymers tested were high-performance, subsequent tests with commodity polymers also have been positive. "We have gone through every type of polymer you can imagine. It doesn't really matter what the matrix is. It performs similarly," Sheares adds.

With the success in the laboratory, Sheares applied for a composition of matter patent. "We are the first people on record to make a polymer quasicrystal composite, so we want to own that composition," she says. Approval on the patent application is pending.

Thiel, who also is chair of the Iowa State chemistry department, calls Sheares' accomplishments very exciting. "Valerie is very enthusiastic and has clear expectations of what she wants to do. She wasn't afraid to try something that nobody expected to work. As a result, she's opened up a whole new area of study both in terms of the practical ramifications and the fundamental questions related to the nature of the interaction between polymers and quasicrystals," Thiel says.

## New hope for hip replacements

One application Sheares and her Ames Lab colleagues currently are working on grew out of conversations between Bloom and another graduate student, Brian Anderson. Surya Mallapragada, Anderson's major professor, is a member of the Materials Chemistry Program and an ISU associate professor of chemical engineering.

One of Mallapragada's research areas involves the use of ultra-high-molecular-weight polyethylene in replacement hip joints. "The big problem with polyethylene is that there is a lot of wear. The polyethylene is in the form of a cup. A metal ball rotates in the polyethylene socket, and there is constant friction causing wear on both materials," Mallapragada explains. Not only does this impact the mechanical integrity of the replacement joint, but as the ball and socket wear, particles from these materials lead to bone loss, causing the joint to loosen, she says.

With its nonabrasive, wear-resistant characteristics, Sheares' composite appears to offer a solution. Her research group filled the ultra-high-molecular-weight polyethylene with quasicrystals and did the wear test. "They performed just beautifully, just like they had done in every other polymer," Sheares says.

The next hurdle was determining how the composite behaved with living cells. The initial test to establish its biological compatibility was positive, i.e., it's not causing cell death. Sheares emphasizes that this was a quick, short-term test, but it means they can move on to much more extensive biological testing. "It's pretty exciting. It is an application that you can see immediately how it could be utilized," she says.

Sheares removes a sample from a polymerization mixture to isolate it for characterization.

## Searching for clues

The investigation into polymer-quasicrystal composites is far from over. Even as Sheares gets more feedback from industry with ideas for applications, she is searching for clues that will help her answer some very fundamental questions about polymer-quasicrystal interactions at surfaces. What is that interaction? Why do the quasicrystals disperse?

In some respects, the success in the laboratory has hindered finding the answers. "If it only worked with certain types of polymers, then we could look at why one works, but another doesn't. It works with every polymer. It's great, but it doesn't help us explain why," she says.

Unraveling this mystery is a top priority. After all, if you know how and why something works, you can make it work even better. ◆

**For more information:**
Valerie Sheares, (515) 294-2474
vsheares@iastate.edu

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DOE Office of
Basic Energy Sciences

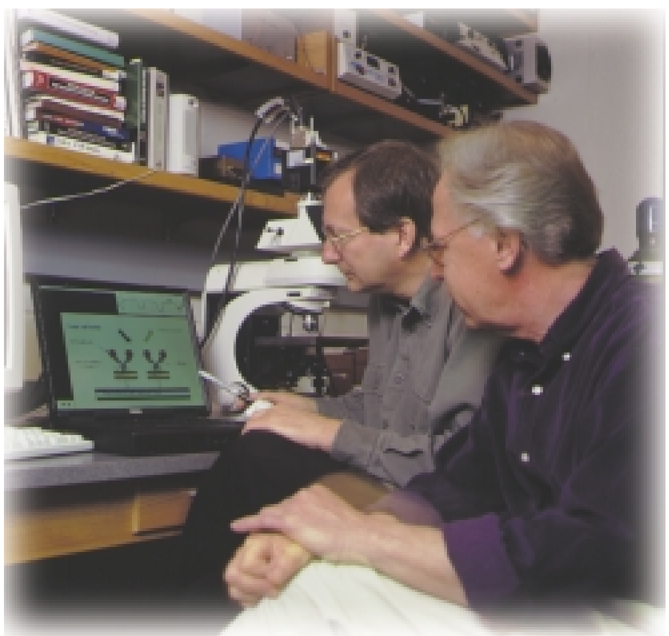
# Closing in on Cancer

## New biosensor technology may ease cancer risk-assessment

*by Saren Johnston*

The sooner you find it, the better. But the indications that an individual may have an increased risk of getting cancer are often difficult to spot. Tucked away, these clues lurk in the twists and turns of the body's DNA.

Uncovering the clues has traditionally been a complex and time-consuming practice, but now Ames Laboratory researchers have come up with a highly sensitive and selective method to speed that process.



Ryszard Jankowiak (left) and Gerald Small study a design for the second-generation biosensor chip.

Gerald Small, an Ames Lab senior chemist and an Iowa State University distinguished professor, and Ryszard Jankowiak, an Ames Lab senior scientist, have developed a unique biosensor technology that provides immediate information about DNA damage from cancer-causing pollutants called carcinogens. Damage to DNA, which carries the genetic code of life, is a critical first step in the development of cancer.

### Tracking adducts

When carcinogens enter the body and are activated, they can react with the DNA to form DNA adducts, chemical compounds in which the carcinogen is attached to the DNA. If the body's natural defense systems do not properly repair the damage caused by these adducts, the result can be the birth of a renegade cell. Uncontrolled proliferation of such a cell results in cancer.

A reliable way scientists can assess cancer risk is to keep track of DNA adducts formed in human cells. Small and Jankowiak are developing a novel means for detection of certain DNA adducts that can be found in urine. The newly developed biosensor chip technique is simpler and potentially more practical than previous methods.

"This scientific advance holds the promise of making it easier and less expensive to identify cancer-causing chemicals in the body, giving physicians a warning sign before the cancer grows and spreads," says Secretary of Energy Spencer Abraham.

Jankowiak leads the biosensor chip research project, which also includes Marc Porter, an ISU chemistry professor and director of ISU's Microanalytical Research Center. Jeremy Kenseth, who recently received his Ph.D. under Porter's supervision, and Scott Duhachek, a former postdoctoral fellow who worked with Small and Jankowiak, also made significant contributions to the project.

The biosensor technology is based on a unique gold chip that was constructed by Kenseth. The chip can be used to detect fluorescent DNA adducts — adducts that emit light when excited by a laser. Bound to the chip's surface are special antibodies, proteins that serve as the body's natural defense system against infectious agents. Scientists can develop antibodies in the laboratory to be so selective that they will preferentially bind a specific DNA adduct.

## The chip in action

The biosensor chip includes a single-layer linker molecule that adsorbs to the bare gold. The chip with the linker molecule can be exposed to a drop of solution containing an antibody specific to the DNA adduct of interest. The linker serves as a coupling agent, binding the antibody to the chip. This process creates an active surface area that can, in turn, bind molecules of those adducts specific to the antibody when the chip is exposed to a liquid sample. With the new biosensor chip technology, scientists could test for the presence of a certain adduct in a sample of urine by simply dipping a chip containing the corresponding antibody into processed urine. The adducts of interest would bind to the antibody and fluoresce when scanned with a laser beam at low temperature — minus 4 Kelvin (minus 452 degrees Fahrenheit). The data gathered from the laser scanning would then be used to produce a detailed fingerprint for adduct identification, providing vital information for cancer risk-assessment.

"The biosensor chip technology has the potential to play a significant role in the advancement of cancer research," says Ames Laboratory Director Tom Barton. "It demonstrates, once again, the diversity of Ames Laboratory's scientific efforts and the commitment of our scientists to perform cutting-edge research that may improve the lives of people throughout the world."

## The glycerol effect

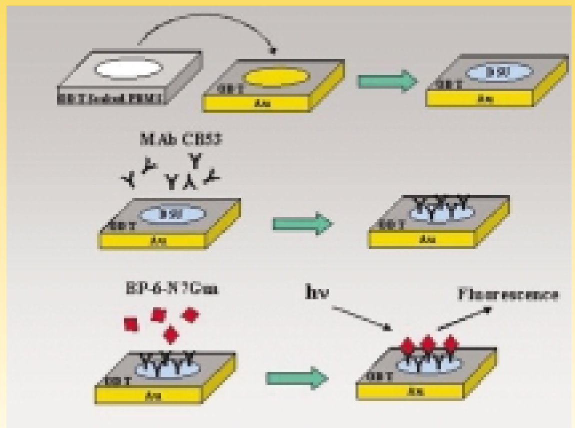
Jankowiak explains that initially they were unable to detect fluorescence at room temperature. However, he notes that the addition of a thin layer of glycerol to the surface of the chip led to a dramatic increase in fluorescence intensity at room temperature. "Cooling the chip further increased the intensity by a factor of 10," he says.

Although Jankowiak is still investigating the glycerol-induced fluorescence enhancement, some insight already has been achieved. For example, he says, "We know that the distance from DNA adduct to the gold surface has increased by a factor of two, since the antibody adopts a more upright orientation on the surface in the presence of glycerol. This accounts for the significant increase in the observed fluorescence intensity due to the decrease in surface quenching by the underlying gold." However, he notes that a great deal more research is needed to determine whether it would be possible to take advantage of the glycerol effect in the manufacture of biosensor chips.

The biosensor research team is investigating other enhancements to the chip technology, including making chips with upright orientation of the antibody and increased distance between the bound DNA adduct and the supporting surface. The team is also looking at developing chips with several addresses for different antibodies that bind different adducts, and using surfaces other than gold.

"The more adducts that can be identified, the more complete the picture of DNA damage resulting from exposure to mixtures of carcinogens," says Small.

Jankowiak adds, "We are currently looking at adducts implicated in breast and prostate cancer. One day this technology could lead to significant advances in precancer diagnosis." ◆




A gold-coated silicon wafer is the starting material for constructing Ames Lab's new biosensor chip that can track DNA adducts. Treating the chip with specific chemicals allows it to bind DNA adducts to its surface. Coating the chip with a thin layer of glycerol greatly enhances the emission spectra produced when the chip is exposed to a laser beam at low temperature.


**For more information:**
Ryszard Jankowiak, (515) 294-4394
jankowiak@ameslab.gov



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# Dirty Coal, Clean Power

## Metallic filter holds key to clean-burning, coal-fired power generation

*by Kerry Gibson*

Iver Anderson thinks the solution to the rolling power blackouts in California and parts of the East Coast may lie under the rolling black soil of Iowa's farm country. "Iowa is sitting on top of huge deposits of coal," says Anderson, an Ames Laboratory senior metallurgist. "The problem is that it's high-sulfur, dirty coal."

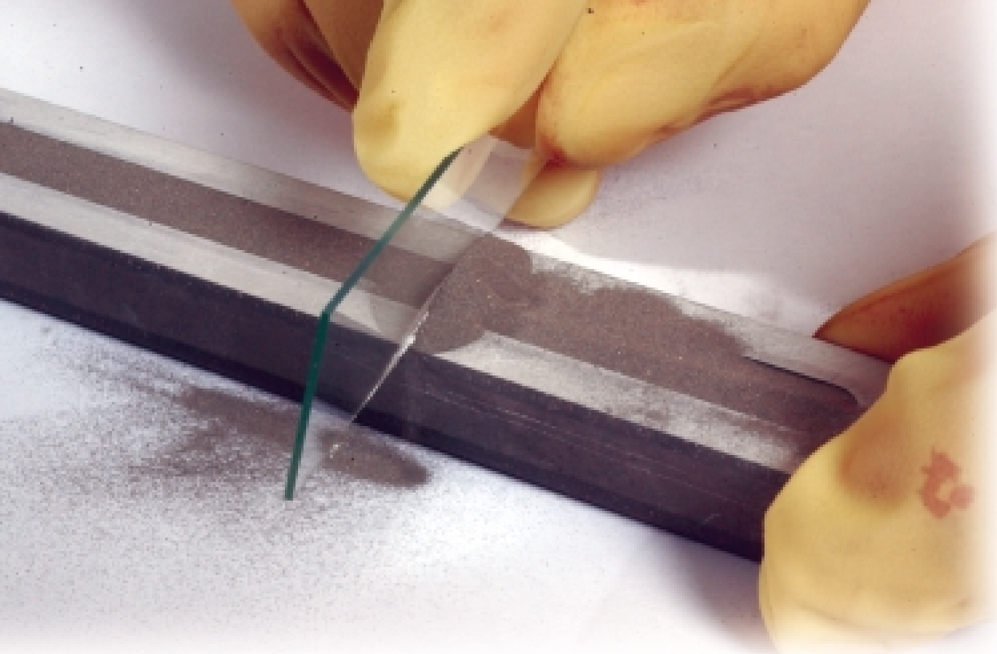
That's why trainloads of cleaner-burning coal from western states pass by every day on the Union Pacific's east-west line, just a couple blocks away from Anderson's lab in the Metals Development building. As those trains rumble past, Anderson and colleagues Bob Terspstra and Brian Gleeson are closing in on a new material to filter the nasty ashes and dust that result from burning "dirty" coal.

"The technology to burn dirty coal cleanly has existed for some time," Anderson says. "Demonstration plants have proven that pressurized-fluidized bed combustion and integrated gasification combined cycles are highly efficient, low-emission power-plant concepts. The high pressure and high temperature volatilize, or burn off, most of the pollutants, even those in the exhaust gases."

But there's a catch, quite literally, with these systems. The flue gases contain fine particles of fly ash. High in sulfides, chlorides and sodium compounds, these particles pose an abrasive and corrosive threat to the turbines that drive a power plant's generators, as well as to air quality. To prevent these particles from reaching the turbine blades (and the atmosphere), the hot gas is passed through clusters, or banks, of cylindrical "candle" filters. Open on the bottom end, these three-inch-diameter filter tubes are about four feet long and currently made out of a ceramic material that can trap particles as small as one micron.

"The ceramic filters do a good job of standing up to the heat and

Senior metallurgist Iver Anderson holds a high-pressure gas atomization nozzle used to produce powdered nickel-chromium-aluminum-iron alloy (contained in the jar). The powder is spread in shallow molds and sintered in a vacuum furnace to form porous metal filter material (foreground) that can be shaped.



Powdered nickel-chromium-aluminum-iron alloy is placed in a test mold and struck off in preparation for sintering, a process that bonds the spherical powder particles together.

the nasty oxidizing-sulfidizing environment created by the gases," Anderson says, "but they're very delicate. Ceramics can crack easily and if even a single candle filter breaks, the filtration ability of the whole bank is lost. So these plants must have several banks of filters. Switching to a fresh filter bank is much better than shutting down the whole power plant to change one filter tube."

As more and more particles collect inside the tube-shaped filters, the amount of air passing through decreases. To keep each tube operating efficiently, the fly ash is periodically knocked off by an internal blast of compressed air, a process called backflushing. Since the flue gases are about 850 degrees Celsius (1,562 degrees Fahrenheit), the abrupt change in temperature caused by the compressed air can also crack the ceramic material. For new combustion technologies to move from demonstration plants to widespread use in the power industry, a new, tougher filter is needed.

"It's the last big hurdle to seeing this technology take off," Anderson explains. "Power companies are in the business of generating power and making money, not constantly changing filters. You want a filter assembly that is rugged enough and has a long enough life that you can essentially forget about it."

To find those properties, Anderson's research team looked at developing rugged metal filters from nickel-, cobalt- and iron-based "superalloys" developed for the aerospace industry. These high-strength metals can withstand high temperatures and aren't affected by thermal shock during backflushing. The researchers selected a nickel-based alloy that maintains its strength at high temperatures, but more importantly, develops a protective scale when it oxidizes.

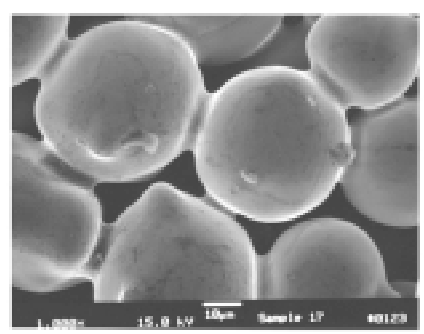
"The nickel-chromium-aluminum-iron alloy we chose contains a sufficient amount of aluminum to form a protective film of aluminum oxide," Anderson says. "Once an aluminum oxide layer forms, it prevents further oxidation. It's why structural aluminum can be left unpainted without rusting away."

While ceramic filters need to be thick for strength, a superalloy metal filter may be quite thin, giving it an airflow efficiency advantage. To create these thin, permeable sheets of metal, Anderson uses a process called tap-densified loose powder sintering.

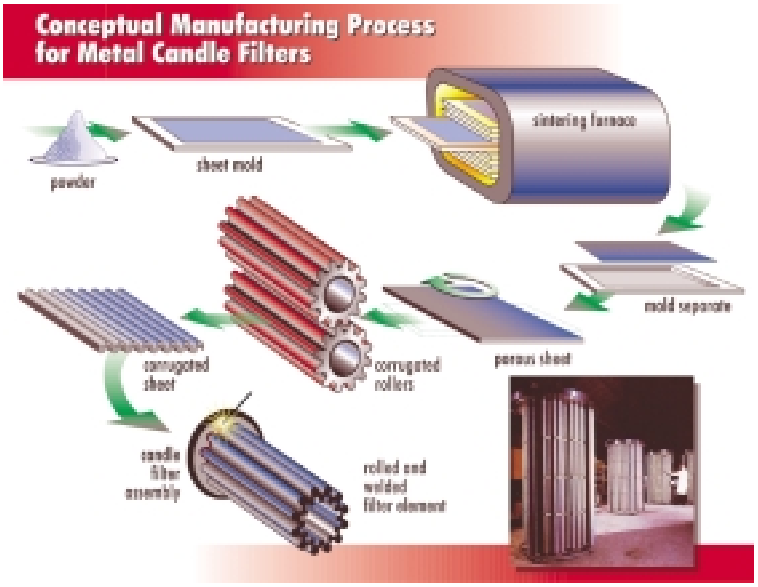
He starts by converting high-purity molten superalloy into a fine powder using a high-pressure gas atomization system. As the hot metal passes through a nozzle, a high-pressure jet of nitrogen gas breaks up the stream of liquid superalloy into millions of tiny metal spheres. The resulting powder is then sorted, by screening, into spheres only 25-45 microns in diameter.

After spreading the sorted metal powder out as a thin layer (0.5 millimeters) in a shallow "cookie sheet," Anderson heats the metal in a vacuum furnace. This sintering process bonds the powder particles together, forming strong, smooth joints between the spheres, but leaving air gaps as well.

"In tests we conducted, our alloy experienced only a moderate drop in yield strength going from room temperature to operating temperature (850 C)," Anderson says. "And



As this micrograph shows, the sintering process fuses the spherical metal powder particles together, forming strong bonds, or necks, while leaving small gaps that allow air to pass through.




The metal filter material would be formed into candle filters to remove corrosive and abrasive fly ash from the exhaust gases of next-generation, coal-fired power plants.

our porous sample turned out to be about six times stronger at operating temperature than a porous sample of an iron-aluminide material being developed by another research laboratory. That strength allows us to go with a very thin filter body."

Given those encouraging results, the researchers tried a series of bend radius tests to see how well the metal could be formed. According to Anderson, the material was ductile enough to enable it to be formed into corrugated tubes, an important feature not only for strength, but for dramatically increasing the amount of filter surface area.

While the group will tinker with the alloy mixture and conduct additional corrosion testing, the next big step will be to perfect a technique for welding or crimping the longitudinal seam to close off the tube and for adding a mounting flange and cap on the open ends.

As that work progresses, Anderson hopes to try out the sintering process on high-capacity commercial equipment with the help of Mott Metallurgical, a Connecticut-based metal filter manufacturer.

"The key to getting the best bonded neck structures — the fused joints between the powder particles that give the material its ductile strength — is keeping the powder material clean," Anderson says. "You have to use pure material and keep it that way during atomizing and sintering to permit proper bonding of the spheres."

He also hopes to test the filters at a DOE demonstration power plant run by the University of North Dakota. The prospects of what could happen if that testing is successful brings an excited grin to Anderson's face.

"I think the filter could have a great impact on the electric power industry worldwide," he says. "I'm a conservationist at heart, but of the resources available, we have a much greater reserve of coal than anything else. It's even right here in Iowa.

"Making it possible to burn dirty coal cleanly would provide us the stopgap that we need until we can develop the ultraclean hydrogen conversion (fuel-cell based) power plants or use completely renewable resources, such as wind or solar."

Such filters could have an impact on diesel combustion engines as well.

"The technology exists to produce highly efficient diesel engines — two to three times more efficient than gasoline engines," Anderson says, "and you'll probably see diesel hybrid cars very soon. The problem is that the emissions are either high in particulates and low in nitrous oxides or low in particulates and high in nitrous oxides — but not low in both.

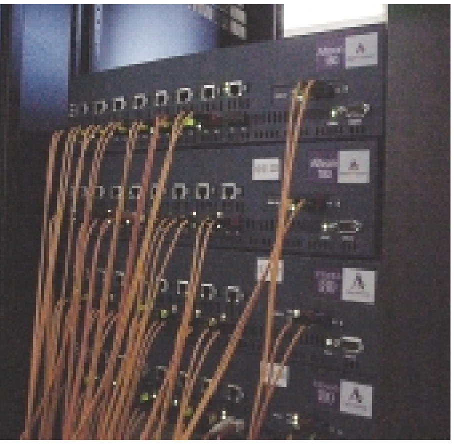
"Ideally, you'd use a particulate filter in combination with the low-nitrous-oxide-producing engine," he adds. "Exhaust emissions from burning common diesel fuels are high in sulfur. We've already addressed that problem, so our filter could possibly work in this application as well. And because diesel fuels can be formulated in so many blends, including soy oil, we'd have all sorts of options." ◆

**For more information:**
Iver Anderson, (515) 294-9791
andersoni@ameslab.gov

**Research funded by:**
DOE Office of
the Deputy Assistant Secretary
for Coal and Power Systems

## "Fast-talking" Clusters

Researchers at Ames Laboratory's Scalable Computing Lab have extended their investigation into communication technology for cluster computers thanks to a National Science Foundation Major Research Instrument grant awarded to Iowa State University's Center for Physical and Computational Mathematics. The $300,000 MRI grant includes $190,000 from NSF and more than $100,000 in matching funds from ISU's Institute for Physical Research and Technology, Iowa State University and the ISU departments of physics and chemistry.



To increase cluster computer efficiency, Ames Laboratory researchers are looking at ways of optimizing communication throughput with the Gigabit Ethernet switch as well as with alternative switch technologies.

Scalable Computing Lab researchers are using the MRI funds to improve interconnect solutions for cluster computers, personal computer or workstation networks that can operate at speeds comparable to today's commercial parallel computers, but for a fraction of the cost. Making message-passing between computers in a cluster faster and more efficient is the primary goal of the MRI-supported research effort.

The SCL scientists believe cluster computers could be very appealing to the larger scientific and academic communities. "A university department or a research group can't afford to buy a supercomputer, but they can afford to put a good cluster together," says Mark Gordon, director of Ames Lab's Applied Mathematics and Computational Sciences Program and head of the SCL.

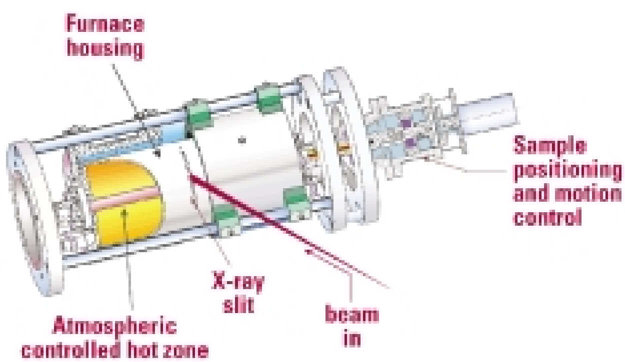
Gordon emphasizes that solutions to grand-challenge problems, such as the design of new materials and catalysts, the development of viable methods for environmental remediation, and the search for the origin of life will depend on state-of-the-art computational hardware and applications software to take advantage of modern computers. "The high-performance computing environment of the future will undoubtedly include scalable cluster computing," he says. (Scalable means the ability to increase, or "scale up," computer processing power to run the same job in less time.)

"In the case of clusters, we want to figure out how to best manage the hardware and get computers talking to each other with a minimum amount of time used in communicating and a maximum amount of time in actually doing the calculations," Gordon adds. "That's what the MRI grant is all about." ◆

## Building on Success

When researchers at Ames Laboratory developed a compact laboratory furnace, it marked a huge leap forward in the ability to understand what happens to a material's crystal structure as the material is heated and cooled. That success has led to the building of two additional furnaces for other DOE laboratories and a refined design intended to make the furnace easier to operate.

Used in conjunction with a powerful X-ray beam, such as that produced by the synchrotron at Argonne National Laboratory's Advanced Photon Source, the furnace allows researchers to capture in a few seconds the patterns created as the crystal structure of the material diffracts the beam. By studying changes in the diffraction patterns as




the material is heated and cooled in the furnace, scientists quickly gain an understanding of what happens when a material transitions from one type of solid to another or from solid to liquid and back again.

The original furnace is being used at the sector of the APS facility operated by the Midwest Universities Collaborative Access Team. According to scientist Matt Kramer, one of the developers of the furnace, two copies of that furnace are being built by Ames Lab's Engineering Services Group.

"One is being built for Systems Research and Instrumentation, which is the developmental CAT for Argonne National Laboratory," says Kramer. "The other one is for the High Temperature Materials Research Laboratory at Oak Ridge National Laboratory."

The furnace's fame isn't limited to this country. Kramer's group designed and built an even smaller unit for the European Synchrotron Radiation Facility, located in Grenoble, France. That collaborative effort was aided by ESRF researcher Larry Margulies, a former Ames Lab graduate student who worked with Kramer, senior materials scientist Bill McCallum, and assistant metallurgist Kevin Dennis on the original furnace design.

Even with the furnace's success, Kramer's group is looking to improve upon it by making it quicker to set up and easier to use.

"With the present furnace, it takes a substantial amount of time, up to 36 hours, to get things properly lined up," he says. "The new design we're working on will be more modular and make it easier to position the sample in relationship to the beam, so we can spend more time doing research instead of setting things up. Also, if we need to swap out components, we don't have to go through the entire alignment process again." ◆

## BAM Continues Amazing Development

A material that rivals industrial diamond in hardness continues to amaze the researchers who developed it and attract interest from a variety of industrial sectors. The material represents a breakthrough technology that could have a substantial impact on the machining industry, which spends $300 billion each year in labor and overhead in the United States alone.

But hardness is only one of several unique properties possessed by the boron-aluminum-magnesium alloy, nicknamed "BAM." Preliminary tests show it stands up well in cutting both concrete and stainless steel, giving it a definite advantage over diamond-coated cutting tools in slicing up steel-reinforced concrete.

"Diamond does a good job of cutting concrete and masonry," says researcher Bruce Cook, "but wears quickly when cutting steel due to the chemical reaction between the carbon in the diamond and the iron in the workpiece. Our alloy seems to cut both concrete and steel equally well without much wear."

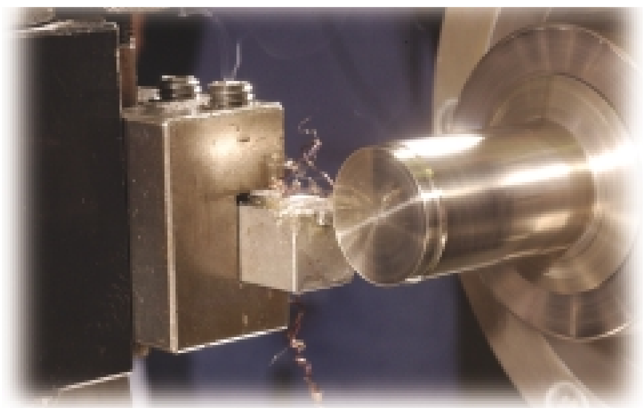
Though Cook and fellow researchers Alan Russell and Joel Harringa expected BAM to perform well as a cutting tool, they were surprised to find that the material cuts without getting hot. Even when the cutting edge is spinning off red-hot ribbons of lathe-turned stainless steel (somewhere around 700 degrees Celsius, or 1,292 degrees Fahrenheit), the tool stays cool enough to touch with a bare fingertip.

"There's very little heat transport, and we think that's due to fine grain size and the complex crystal structure within the material," says Cook. Bruce Harmon, Ames Lab deputy director and director of the Lab's Condensed Matter Physics Program, and graduate student Younben Lee are also conducting further calculations on the fundamental electronic structure. "We're interested to see if there's a relationship between the electrical transport and thermal transport that may help explain how ultrahard materials perform," Cook adds.

Cook's research team has also boosted the scale of production of the material, in part to keep up with requests for samples. Button-sized samples have been replaced by much larger wafers, nearly 2 inches in diameter and about three-quarters of an inch thick. But given BAM's low density (about 2.5 grams/cm$^3$), the wafers lack the expected heft of conventional tungsten carbide used to slice through concrete and steel.

Since discovery of the material was first announced in October 1999, more than 100 companies and research facilities have requested samples.

While in-house testing continues, including recent successful trials in machining titanium, Cook hopes to learn as much or more from the various companies that



A sharpened wafer of BAM, glued to a cutting tool blank with household epoxy, shaves off ribbons of stainless steel. Low heat transport properties keep the cutting tool cool, even though the stainless steel gets red-hot.

have received samples. For example, a manufacturer of tooling for industrial woodworking equipment returned a sample that had been shaped using electric spark erosion. And a producer of industrial gases and surface coatings is investigating the material as an extremely durable coating for various industrial applications.

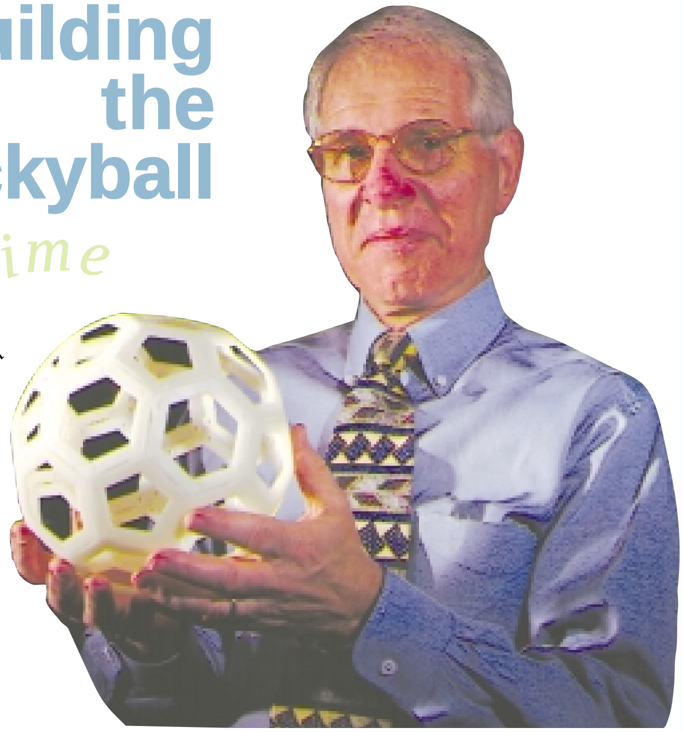
"They may not realize it, but these companies are providing us with valuable information," Cook says. "It gives us a much broader perspective. At the same time, most of the research and development is being conducted by scientific staff so they can supply test data to support the various applications. It really helps our understanding of the material." ◆

(See more INtouch on inside back cover.)

# Building the Buckyball

## A Bowl at a Time

by Kerry Gibson



Ames Lab senior chemist Peter Rabideau holds a buckyball model.

Showing a new, naturally occurring compound to a research chemist is, in a way, like throwing down a gauntlet. The unspoken challenge being issued — create this in the lab.

For Peter Rabideau, that gauntlet has been the buckyball — a curious hollow sphere formed by 60 atoms of carbon. Rabideau, an Ames Laboratory senior chemist, has moved a step closer to meeting that challenge by developing a practical means of producing bowl-shaped segments — buckybowls — that could eventually be pieced together to form the complete ball.

"Chemists are always interested in being able to synthesize things whether you need them or not," Rabideau says, pointing out the synthesis of cholesterol as just one example. "It's a big thing just to show you could do it, so there's an element of that to it.

"But there's also the practical side of being able to put things inside the buckyball, such as precursors for supertough coatings or drug-delivery systems, that may have some real novel properties if one could figure out how to do that," he says.

Buckyballs have intrigued chemists since the uniquely structured molecules were first discovered in 1985. The carbon atoms align to form a hollow structure similar to the pattern of panels found on a soccer ball. They get their name from architect/engineer/philosopher Buckminster "Bucky" Fuller, who pioneered the concept of the geodesic dome.

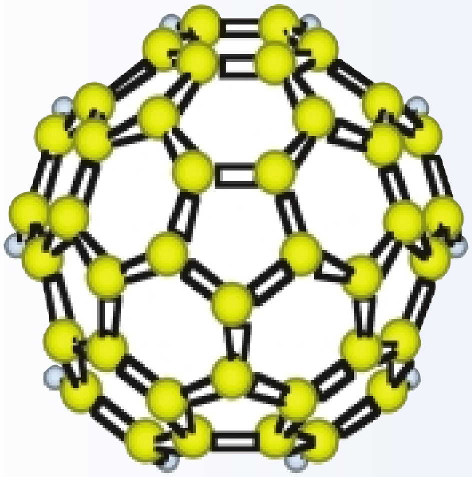
"It turns out that geodesic stability on the molecular level is also rather remarkable," says Rabideau, who also serves as dean of Iowa State University's College of Liberal Arts and Sciences. "Carbon atoms like to be arranged in this particular formation and are extremely stable."

Since the structure is very stable and hollow, chemists have envisioned a whole new array of applications if they could find a way to put other atoms or compounds inside the buckyball.

Unfortunately, the only way to produce $C_{60}$ is to replicate the environment of interstellar space with a process that basically involves arcing carbon rods, but this reaction takes place only at high temperatures. The high temperatures make the reaction hard to control, so it's extremely difficult to try to produce buckyballs with something inside them. So the search turned to finding a way to "build" a buckyball from scratch.

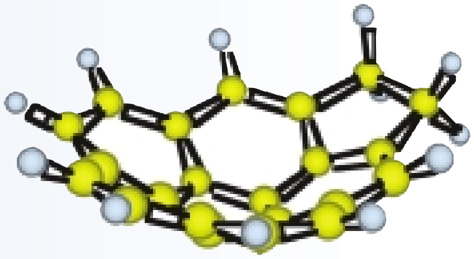
"If you took a buckyball apart, it wouldn't be a stable entity because it would have dangling bonds," Rabideau explains. "But if you took it apart and put hydrogen atoms on the dangling bonds to stabilize the structure, you'd have a chemical compound that we call a buckybowl."

It's the curved shape of these compounds that Rabideau finds intriguing. Most polynuclear aromatic hydrocarbons, such as graphite, are flat. And it was these "flat" compounds that he spent the early part of his career studying at the University of Chicago, under the guidance of the man who literally wrote the book on the subject, Ron Harvey.



As Rabideau's research progressed, so did his advancement through the academic ranks. By 1990, he was a dean at Louisiana State University and had come to an impasse in his research.

"We had answered a lot of the questions that we originally asked, so



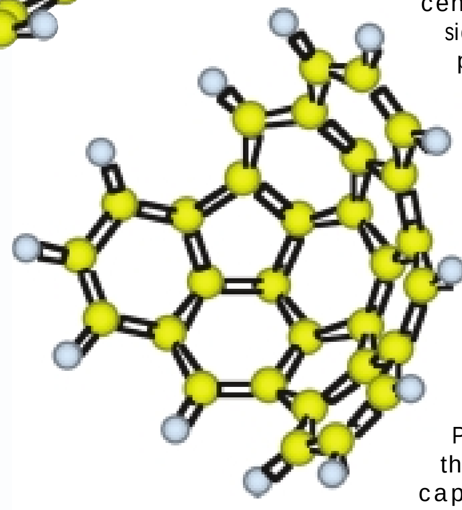
it was a matter of where the research was going," Rabideau says. "I had also become a dean, so I had to do a reality check on whether or not I wanted to continue to do the active research.

"My feeling was that if I was going to remain active in research, we would have to revitalize our program and find some new things to do," he says. "About that time I saw a publication on corannulene ($C_{20}H_{10}$) and some of these curved-surface compounds, and decided that's the direction we would take."

Corannulene was first synthesized in 1966 at the University of Michigan by a long and difficult 17-step process that produced quantities weighing just a few milligrams. Then in the early 1990s, another group of researchers synthesized corannulene using pyrolysis — heating a material until it decomposes. It was this breakthrough that grabbed Rabideau's attention.

"The problem with this (gas-phase) technique is that you must do it in vacuum, so by definition you're still working with very small amounts of material," Rabideau says.

What Rabideau and fellow researcher Andrzej Sygula developed was a solution-phase synthesis using dilute sodium hydroxide in water and acetone that produces tetrabromocorranulene — a molecule of 20 carbon atoms with four bromine atoms attached. This process is detailed in the following article: Sygula, Andrzej and Rabideau, Peter W. 2000. A Practical Large Scale Synthesis of the Corannulene System. *Journal of the American Chemical Society* 122 (26): 6323-6324.



Unlike more common polynuclear aromatic hydrocarbons, such as graphite, which are flat, corannulene molecules and related compounds are curved. This curved, or bowl shape, of 20 carbon atoms is aligned with five atoms in the center. The remaining atoms form six-sided polygons, around this central pentagon, giving the bowls their geodesic pattern. Without hydrogen atoms (shown here in blue), dangling bonds on the carbon molecule would be unstable.

The trick now for Rabideau is to figure out a way to link or combine three bowls together, sans the hydrogens, to form a complete $C_{60}$ buckyball, an extremely stable molecule that has yet to be synthesized.

Piecing the ball together could have the added benefit of being able to capture another atom or metal inside the ball, which would open a whole new array of applications.

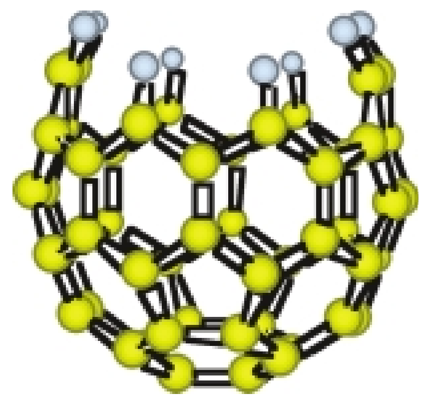
The process allows production of 25-gram samples, a thousandfold increase over the pyrolysis method.

"The process is really pretty simple," Rabideau says. "In fact, the final step could almost be done in your garage using household chemicals." Though it hasn't been a particular goal of his research, Rabideau adds that a feasible commercial synthesis could easily be developed.

The synthesis of tetrabromocorranulene has also had a silver lining. While the bromine atoms can easily be removed to form corannulene, it's advantageous to leave them on and add to them.

"We now have a way of elaborating that particular molecule in a lot of different ways," Radideau says, pointing to diagrams of just eight of the many variations. "It's better than if we'd discovered a way to go directly to corannulene because we can use the bromines to do other things, though our focus is on the fundamental chemistry involved."

Though building a buckyball is still in the distance, researchers now have an unlimited supply of bowl material



to study. By unlocking the properties of these bowl-shaped compounds, Rabideau hopes to eventually discover a way to combine the bowls into a sphere.

"If we could figure out a few critical reactions, we might ultimately be able to synthesize $C_{60}$," Rabideau says. "That would, in principle, allow us to build $C_{60}$ with a hole in it so we could trap something inside, such as an atom or metal, and then close up the ball." ◆

**For more information:**

Peter Rabideau, (515) 294-3220
rabideau@iastate.edu

**Research funded by:**
DOE Office of Basic Energy Sciences

# Random Acts of Brightness

by Saren Johnston

## From disordered systems comes coherent light

Like musicians in a marching band, waves of laser light move "in step" with one another. As a result, the waves spread only slightly, even over great distances.

Laser light differs from the light produced by other sources, such as electric light bulbs or the sun. The light from these sources is a confusion of many wavelengths, traveling in all directions. It is called incoherent light. But a laser produces an intense, narrow beam of light that travels in only one specific direction, much like a marching band moves in unison along a parade route. This is called coherent light.

Producing laser light, light waves of a single frequency or just a few frequencies that "march" together along a straight path, requires a gain medium — a substance that will amplify light. The gain medium must also have a precise and orderly atomic structure that will allow it to store energy and then release the energy at the right time and in the right direction.

Although lasers are technological marvels of exactness and order, physicists have speculated for years about the possibility of achieving laser light with disordered systems.

Costas Soukoulis, an Ames Laboratory senior physicist, has gone beyond speculation. He and former Iowa State University graduate student Xunya Jiang, now working at DiCon Fiberoptics, Inc., near Berkeley, Calif., have developed a theoretical model that simulates the phenomenon of random lasing, in which photons that follow random paths create a multiple-light-scattering laser.

Theoretical physicists Costas Soukoulis (left) and Xunya Jiang look over a transparency displaying their design of the mechanism of a laser system. The transparency is one in a series of visuals the two researchers have developed to accompany their popular, invited talks on the theory of random lasing.

### Regular lasers

In a regular, or periodic, laser the amplifying substance is positioned within the laser's cavity. It might be a solid, such as a crystal or a semiconductor; a gas or a mixture of gases, such as helium-neon; or a liquid dye.

All in all, the production of laser light might be thought of as a continuous round-trip journey. When excited by an outside energy source, perhaps a flashlamp or another laser, the electrons in the amplifying substance get "pumped" to a higher energy state where many will spontaneously emit incoherent light waves. The excited electrons that haven't spontaneously emitted photons of light will begin to fall to a lower energy level, the metastable state, where they "hang out" for awhile, just waiting to release their excess energy as light so they can return home to the ground state. These are the electrons that will eventually emit coherent laser light.

The release of coherent light is called stimulated emission. It takes place when electrons in the metastable state are stimulated to fall to the ground state by photons coming in from other electrons that are falling from their metastable states. When an electron falls from this stimulation, it also produces a photon of light in exactly the same direction and with exactly the same energy and phase as the stimulating photon. Then, the process repeats itself. The laser light produced is reflected back and forth between mirrors positioned in the laser's cavity, a process that amplifies the light many times. Energy gain becomes greater than energy loss, and an intense laser beam is created.

## Random lasers — the BIG SURPRISE

But what happens if instead of possessing an orderly arrangement of atoms that produce synchronized, focused wavelengths, the structure of the light-amplifying substance is one that scatters light quite efficiently in all directions? Can such a substance produce laser light?

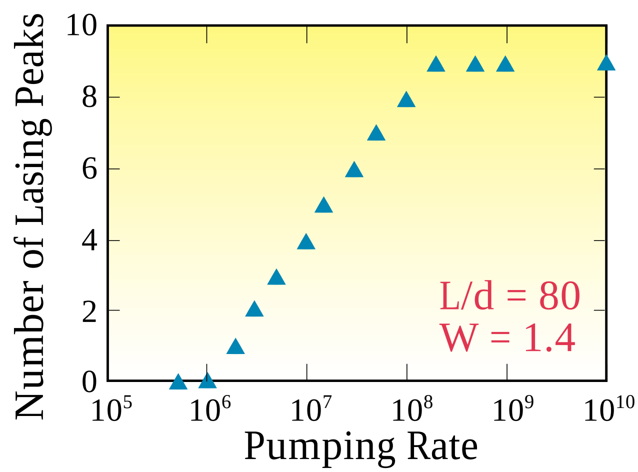
Soukoulis' and Jiang's model says, yes it can. A new kind of laser can be created using photons that follow random paths. The disordered system does not impede lasing — it accounts for it. The process goes something like this: When light is shined into a substance that scatters it well, the photons get bounced in random directions. If this happens often enough, it's likely that the trajectory of the photons inside the gain medium will be extremely long and that the photons will travel many times through the same crystal grains, ricocheting from side to side as they go. Under these conditions, light can be amplified tremendously. The process is similar to the way light from an ordinary laser travels back and forth between the two mirrors in the cavity. If the electrons in the disordered gain medium get pumped to a higher energy level while traveling their long, random paths, the result could be amplification to laser light. The gain medium, whether a crystal powder or a material containing random scatterers, would, in effect, become a laser.

## Explaining the experiments

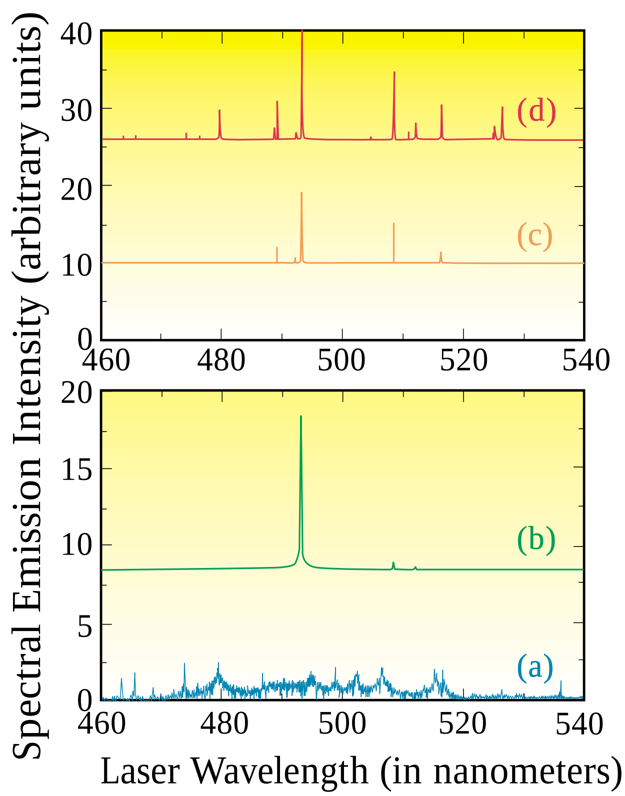
Soukoulis' and Jiang's theoretical work represents some of the very first efforts to understand and describe the mechanics of random lasing. They have interpreted the investigations of experimentalists Hui Cao of Northwestern University, Ad Lagendijk of the University of Amsterdam and Val Vardeny of the University of Utah, breathing logic and reason into the laboratory observations and giving credibility to the random-lasing phenomenon.

"The first thing we wanted to do was to see if we could understand all the narrow, sharp lasing peaks the experimentalists were seeing in the output spectrums of disordered systems, such as the zinc oxide nanocrystals Cao was studying," says Soukoulis, who is also an Iowa State University physics professor. "So we tried to simulate the real experiment in the computer using a complex numerical technique called finite difference time domain. We did a one-dimensional version of a three-dimensional experiment."

Jiang adds, "The advantage of the FDTD method is that from our computer simulation we can see the dynamic process of the random system and how the electric field is building up inside. We are able to follow the evolution of the electric field."




Using their FDTD model, Soukoulis and Jiang discovered that the number of lasing peaks in a random system will increase as the pumping rate increases. However, the model also revealed that once the pumping intensity reaches a maximum value, the lasing peaks no longer increase in number, but, instead, saturate to a constant value.




The above figure shows Soukoulis' and Jiang's findings for random laser systems based on their FDTD computer model. With an increase in the pumping rate — a (no lasing), b, c, d — there is an associated increase in the intensity, or brilliance, of the emission spectra; enhanced definition of the lasing peaks; and an increase in the number of lasing peaks to the point of saturation to a constant value.

The ability to track the population of the electrons as they are pumped to higher energy levels and then fall to metastable and ground energy states has allowed Soukoulis and Jiang to arrive at a number of important results.

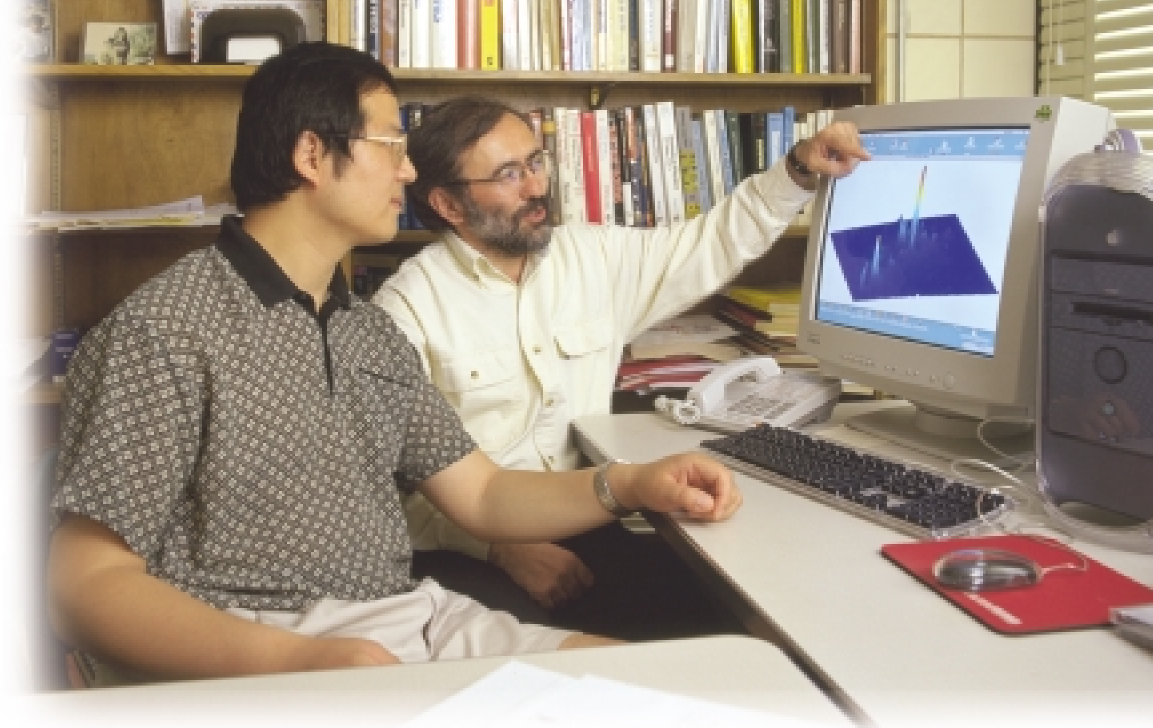
Using their computer simulation of random lasing, the two theorists are able to determine the threshold value of lasing for the amplifying substance. Knowing the threshold value is critical because it is the amount of energy needed to pump the electrons in the amplifying substance to the intensity where lasing takes place. "One very important thing we discovered is that the threshold value of lasing decreases as disorder increases," says Soukoulis. "So, the more the concentration of scatterers, the lower the threshold of lasing."

Through their one-dimensional model, Soukoulis and Jiang also found that the sharp peaks the experimentalists had observed in the output spectrums were coming from specific lasing modes of the disordered systems. "From our theory, we can really solve and understand these beautiful peaks," says Soukoulis. He explains that for disordered systems the lasing has a greater probability of occurring in some particular regions of the system than in others. As the electrons in the amplifying random substance are pumped to a higher energy level, the lightwaves retain their shape, but they become bigger and bigger. Eventually they will start lasing.

In addition, the model revealed that if the pumping intensity is increased above a maximum value, the number of lasing modes remains the same. They do not increase anymore, rather; they saturate to a constant value that is determined by the degree of randomness in the system and its length.

## Predictions and possibilities

"With our one-dimensional model, we can predict exactly where the lasing modes will be in a given random system, which mode will lase best, and the wavelength, which determines the color of the emission light," says Soukoulis.



Teacher and student, Soukoulis (right) and Jiang, study an image of the localized lasing peaks produced by scattered light from zinc oxide nanocrystals. The image was supplied by experimentalist Hui Cao of Northwestern University.

The ability to predict the lasing modes in a given random system is critical when considering some of the potential uses for random lasers. The novel devices hold promising properties for brightening the pixels in flat-panel displays. By reducing the size of the phosphor grains that emit light in these displays, it might be possible for the electron emitter in each pixel to excite the phosphor's electrons and initiate lasing, which would brighten the pixels.

Another possible application for random lasers lies within the medical arena. Used with chemical sensors, these devices could provide a sophisticated, noninvasive tool for diagnosing problems within the human body. And paint-on random lasers may one day light the way to more efficient and economical search-and-rescue missions to identify downed ships and airplanes, and even individual passengers. ◆

**For more information:**
Costas Soukoulis, (515) 294-2816
soukoulis@ameslab.gov

**Research funded by:**
DOE Office of Basic Energy Sciences

# New Life for Old Scrap

## Process recovers valuable neodymium from magnetic waste

*by Kerry Gibson*

Ever own an expensive wool suit that went out of style or got a little too small? Even though you can't wear it, the price tag makes it too hard to part with. So, the suit hangs at the back of the closet, gathering dust, on the chance that you'll drop some pounds or the fashion will come back into vogue.

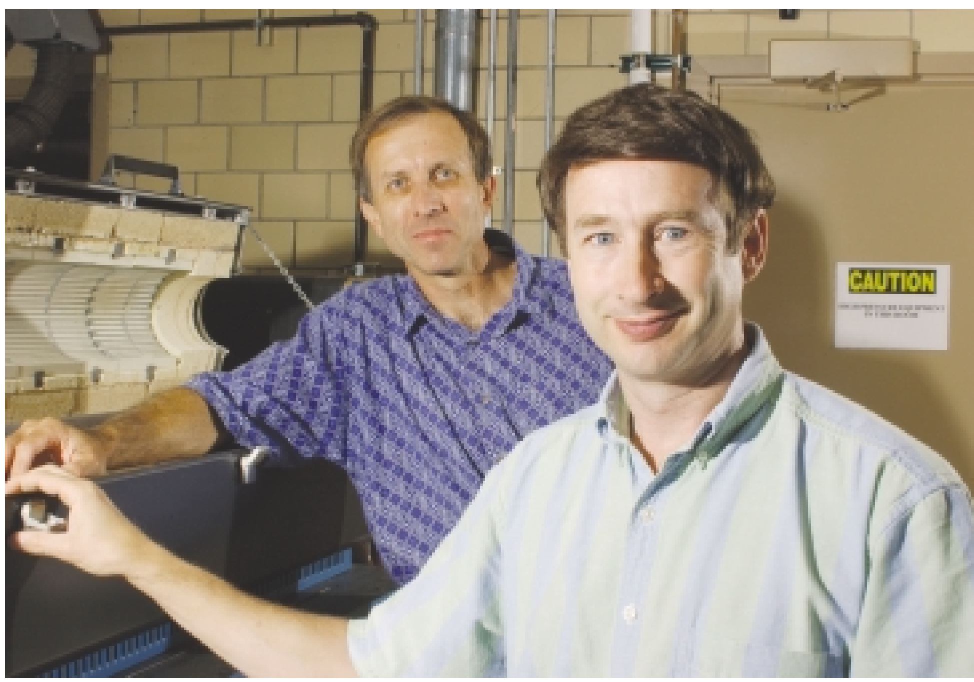
In a way, that's similar to the situation faced by manufacturers of high-energy neodymium-iron-boron magnets. These rare-earth magnets allow the production of smaller, more powerful and more efficient electric motors that are used to make everything from portable CD players to automotive power windows more compact, lighter in weight and easier on batteries.

Despite those good characteristics, the magnet material is brittle and processing it creates a larger than normal amount of machining waste. This leftover scrap can't be reformed into magnets or easily recycled. But because it's roughly 29 percent neodymium (by weight) that's valued at $30 per kilogram — just slightly less per ounce than the price of silver — it's worth too much to dispose of. So it's been stockpiled on the chance that it might someday have a use.

## Molten magnesium solution

That day could be here thanks to Scott Chumbley and Alan Russell. The two Ames Laboratory researchers have refined a process that makes it commercially viable to recover the neodymium from tons of stockpiled magnetic scrap.

"The neodymium-iron-boron material decomposes peritectically — it changes composition — when heated to its melting point," says Chumbley, lead researcher on the project. "So it can't just be melted down and reused. But it's too valuable to throw away, so there are literally warehouses full of 55-gallon drums of the stuff waiting



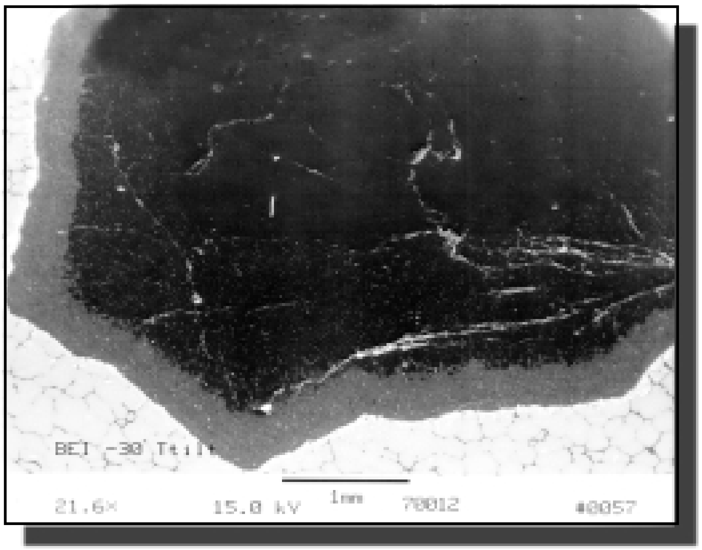

Researchers Scott Chumbley (right) and Alan Russell stand near the vacuum furnace in which test samples of Nd-Fe-B magnet scraps are immersed in molten magnesium. A cutaway view of a stainless steel crucible (title graphic above) shows Nd-enriched magnesium surrounding the darker magnet particles.

to be recycled."

Until now, the best separation method available was to dissolve the neodymium-iron-boron scrap in acid, then perform a series of chemical extraction and reduction steps. However, the complexity and expense of such a method was impractical for large-scale, commercial recycling.

Building on research pioneered and patented by Ames Laboratory researchers Tim Ellis and Rick Schmidt in the mid-1990s, Chumbley focused on using molten magnesium to extract the neodymium from the magnet scrap. Neodymium is soluble in liquid magnesium. In fact, the magnesium-casting industry routinely adds neodymium and other rare-earth elements to make alloys that are corrosion-resistant and offer improved weldability.

The recovery process is relatively simple. After receiving a solvent bath to remove machining residue, crushed pieces of magnet scrap are immersed in liquid magnesium that's been heated to 800 degrees Celsius (1,472 degrees Fahrenheit). The magnesium leaches the neodymium from the scrap particles. The liquid magnesium-neodymium ($Mg_{12}Nd$) solution can then be poured off, leaving the iron-boron particles behind.




A micrograph of a Nd-Fe-B magnet particle (black area) shows the two-phase zone, the lighter area along the edge, in which the Nd is still tied up in an iron compound ($Nd_{12}Fe_{17}$). The Nd then migrates from this region and spreads evenly throughout the liquid magnesium.

## Two-phase zone

What Chumbley and Russell discovered was that there is actually a two-phase zone that forms as the magnet material is submerged in the liquid magnesium. Initially, most of the neodymium is still tied up in an iron compound ($Nd_{12}Fe_{17}$). Then the neodymium gradually moves into the magnesium at the interface between the iron compound and the liquid magnesium. Over time, the composition of neodymium in the magnesium rises at a fairly constant rate and is spread evenly throughout the liquid, while the width of the transitional, two-phase region increases.

The resulting alloy is roughly 30 percent neodymium, making it perfect for use as feed material for the magnesium-casting industry. And it should substantially lower the cost. Currently, a typical magnesium alloy casting contains only two percent neodymium by weight, yet the neodymium accounts for 40 percent of the raw materials cost.

"It would give them a product that is exactly what they're already accustomed to using," Chumbley says. "They wouldn't have to retool or change any of their processes."

## Tweaking the process

The researchers are looking at the effect that variables, such as the size of the magnet pieces, temperature and duration have on the process. In addition to recovering the neodymium, the leftover iron-boron scrap could be recycled as well, particularly for low-grade iron castings where composition isn't critical.

Russell has fielded a number of inquiries about the process from metal producers and recyclers, but so far "no one seems to be on the verge of licensing the technology," he says. One company, a magnesium-casting firm, has requested samples of the magnet material to take a closer look at the process. ◆

**For more information:**

Scott Chumbley, (515) 294-7903
chumbley@iastate.edu

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DOE Office of Basic Energy Sciences

# INtouch

## Coming to the Aid of Midwest Crime Labs

Ames Laboratory and Iowa State University's Institute for Physical Research and Technology hosted the second meeting of the Midwest Forensics Resource Center in May. Just one year earlier representatives from the two organizations had met with Iowa crime lab officials to discuss the types of research, services and training that would be most helpful to their facilities and to suggest funding sources for the proposed center.

This year's meeting reflected how much the effort to establish the MFRC had grown. Attendees represented eight Midwestern state crime labs; three universities; the Federal Bureau of Investigation; the National Institute of Justice; the Department of Energy; and the Bureau of Alcohol, Tobacco and Firearms.

The MFRC will be located at Ames Laboratory and serve as a central point for regional training, education and research in forensic science. It will draw on the expertise of faculty and staff members at Iowa State University, the Institute for Physical Research and Technology, Ames Laboratory and other professionals throughout the region.

To carry out its mission of helping regional crime labs develop more efficient and reliable methods of analyzing crime-scene evidence, the MFRC will focus on four major goals:

- Develop customized training for regional partners.
- Conduct short-term, case-related forensics research projects.
- Develop a forensics curriculum for ISU and associated universities.
- Conduct long-term research into new and improved forensics techniques.

### From Ames Lab to Crime Lab

**Representatives from Ames Laboratory and the Iowa Division of Criminal Investigation load a fingerprint development "glove box" for transportation from Ames Lab to the DCI's crime lab in Des Moines, Iowa. An example of the MFRC's effort to ease problems encountered in the crime laboratory, the fingerprint-development glove box should improve and speed the fingerprinting process.**

**The device was designed and built by Ames Laboratory and IPRT in cooperation with the DCI. It provides a controlled temperature and humidity environment in which to develop latent fingerprints and allows criminalists to view and handle evidence during processing.**

## Science Bowl 2001

The last Saturday in January has become a tradition at Ames Laboratory. It's Science Bowl Saturday. Volunteers know it; students know it; teachers know it. And the event, although held during the coldest month of the year, never fails to produce some hot competition. In 2001, teams from 42 Iowa high schools braved the unpredictable Iowa winter weather to match wits in the annual Ames Laboratory/Iowa State University High School Science Bowl. Ames High School won the regional competition and advanced to the DOE National Science Bowl.


Students collaborate on a bonus question.

# Inquiry

Ames Laboratory
111 Technical and Administrative Services Facility
Ames, Iowa 50011-3020
(515) 294-9557
http://www.external.ameslab.gov

AMES LABORATORY

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

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