Science & Technology at the Ames Laboratory

# inquiry

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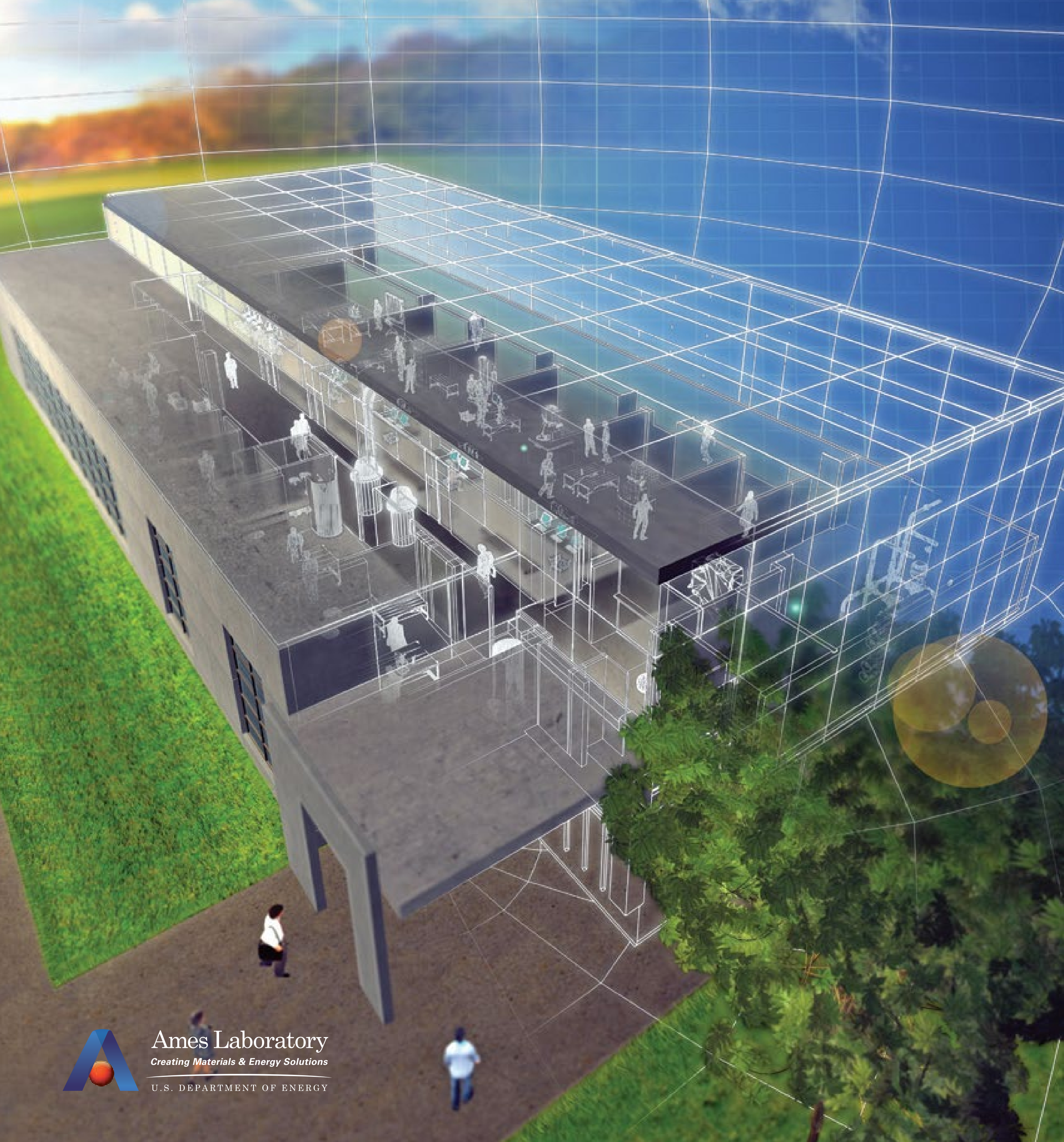

Ames Laboratory
Creating Materials & Energy Solutions
U.S. DEPARTMENT OF ENERGY

# Contents




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Ames Laboratory is a U.S. Department of Energy national 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 Laboratory now pursues much broader priorities than the materials research that has given the Lab international credibility. Responding to issues of national concern, Ames Laboratory 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 and training tomorrow's scientific talent.

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U.S. DEPARTMENT OF ENERGY | Office of Science

# From the Director

## There's a lot going on these days at the Ames Laboratory.

We're in the final stages of construction on our new Sensitive Instrument Facility, a nearly $10 million building that will house an array of state-of-the art electron microscopy equipment. It's Ames Laboratory's first new research facility in more than 50 years. Through a combination of funding sources, including the Department of Energy and our contractor, Iowa State University, the SIF will be outfitted with three new pieces of equipment and an existing transmission electron microscope will be moved and upgraded.

The SIF will significantly enhance our capabilities to characterize materials, particularly at the atomic scale. By better understanding the electronic, atomic and molecular structure of new materials, our researchers can draw correlations between those structures and the materials' properties, such as magnetic, electronic and photonic. You can learn more about the SIF and take a virtual tour of the facility on page six.

And although it's the largest, the SIF isn't the only new thing at the Ames Laboratory. Physicist Ruslan Prozorov's research group has begun collecting data with a new, nitrogen-vacancy magnetoscope that uses defects in diamond's crystal structure—nitrogen-vacancy centers—to visualize the magnetic fields produced by magnetic nanostructures. You can find out more about this on page 10.

When another of our physicists, Adam Kaminski, found that access to synchrotron beam lines was becoming limited, he set out to create an alternative method to conduct angle-resolved photoemission spectroscopy, or ARPES. Using laser light, Kaminski developed a way to study a material's electronic properties right here at the Ames Laboratory, providing both easier access and higher resolution than previously possible. Read about his ingenious discovery on page 12.

> It's Ames Laboratory's first new research facility in more than 50 years.

Ames Laboratory has been a major player in the discovery of giant magnetocaloric materials—compounds that heat up when subjected to a magnetic field, then cool when the field is removed. These materials show promise as a possible replacement for traditional gas-compression technology used in refrigeration and cooling. Our chief research officer, Duane Johnson, and scientist Vitalij Pecharsky recently headed up an international workshop to discuss the current state of these materials and how to advance the technology to make it commercially viable. To learn about the outcome of those workshop discussions, turn to page 14.

Materials discovery, design and synthesis are strengths of the Ames Laboratory and a major reason why our Division of Materials Sciences and Engineering (DMSE) is our largest program. Theoretical models help drive experiments to develop new materials, which in turn—through characterization—help to further refine those theoretical models. Our success in doing this will be evaluated in July as DMSE undergoes its triennial program review.

Given the advances we're making in the areas I've mentioned here, as well as success on a range of individual projects, we feel we're in a strong position to continue to be a world leader in creating materials and energy solutions.

*Adam Schwartz, Director*

# Awards

## Bakac receives Distinguished Service Award

Andreja Bakac

Retired Ames Laboratory scientist Andreja Bakac was recognized for her years of service to the Department of Energy with The Secretary's Distinguished Service Award. Bakac received the award certificate and medallion at an informal presentation at Ames Laboratory on March 6.

The certificate, signed by Secretary of Energy Ernest Moniz, reads: "In recognition of your outstanding research into the mechanistic chemistry of small molecule activation with transition metals and your dedication to the mission of the Department of Energy and the Ames Laboratory. With sincere gratitude for your 38 years of service and stewardship to the Department of Energy, Dr. Andreja Bakac is awarded the Secretary's Distinguished Service Award."

This distinction is given in recognition of continuous and distinctive achievements, within or beyond an individual's area of responsibility, which are of substantial value to the DOE.

Catherine Meis

### Former SULI student named Goldwater Scholar

Iowa State University student Catherine Meis has been named a 2015 Goldwater Scholar, the nation's premier undergraduate scholarship in mathematics, natural sciences and engineering. Meis, a former Ames Laboratory Science Undergraduate Laboratory Internship (SULI) program participant, is a third-year student, majoring in materials engineering with a minor in bioengineering. Meis studied under Reza Montazami and Nastaran Hashemi, ISU assistant professors of mechanical engineering.



### Anderson named 2015 TMS Fellow

Iver Anderson, Ames Laboratory metallurgist, has been named a 2015 Fellow of The Minerals, Metals & Materials Society (TMS). Anderson was one of six Society members who have earned the highest award bestowed by TMS, which recognizes members for their outstanding contributions to the practice of metallurgy, materials science and technology. The 2015 Fellows were recognized at the 144th TMS Annual Meeting & Exhibition held March 15–19, 2015. Anderson was specifically recognized for his inventiveness that led to lead-free solder used in all electronic devices; for seminal contributions to: (1) gas atomization of metallic and polymeric materials, (2) powder metallurgy technology, and (3) rapid solidification processing of a wide variety of materials; for long-time professional leadership as a member of the TMS Board of Directors and chair of numerous TMS technical committees; and for contributions to education, especially for graduate student advising.


*TMS President Hani Heneim, left, presents Iver Anderson with his TMS Fellow certificate during the society's annual meeting.*

### Stephens wins CYtation Award

Ames Laboratory Systems Support Specialist IV Doug Stephens received a 2015 Iowa State University CYtation Award which is presented to ISU Professional and Scientific staff in recognition of individual achievements above and beyond the call of duty, and/or extraordinary performance, or acts in such a way as to make a notable difference in the institution.

Stephens was cited for his leadership in helping upgrade Ames Laboratory's network connections from one gigabit to ten gigabit throughout the four main Ames Laboratory buildings and to the Internet Service Providers (ISU and Energy Sciences Network).

In nominating Stephens for the award, Ames Laboratory Information Systems manager Diane DenAdel said, "Doug's expertise was essential to develop the master plan, implement new switches, and upgrade or modify existing network switches. Other Ames Laboratory Information Technology personnel were involved with various parts of the project, and Doug coordinated the efforts to ensure the success of the project. Doug's efforts made a real difference to the Ames Laboratory and to ISU. It would have been difficult to complete all of this work without Doug's attention to detail and dedication to the overall project."


*ISU Senior Vice President and Provost Jonathan Wickert, left, presents Ames Lab's Doug Stephens with the CYtation Award.*

# SIF — Nearing Operational Status

BY KERRY GIBSON

YEARS OF PLANNING WILL SOON COME TO FRUITION when the Sensitive Instrument Facility (SIF) opens its doors later this year. The $10 million state-of-the-art building, Ames Laboratory's first new research facility in more than 50 years, will house a new array of electron microscopes that will provide researchers an unprecedented close-up look at materials at the atomic level.

"Everything is coming together," said Matt Kramer, Materials Sciences and Engineering Division Director at Ames Laboratory. "We've been able to pull together the necessary funding through partnerships with Iowa State University and the Department of Energy to equip the SIF with what we need to take our research capabilities to the next level."

In addition to an existing transmission electron microscope that will be moved to the new facility and upgraded with newer software and detectors, three additional pieces of equipment will be purchased. Using DMSE equipment funds and an FY14 midyear equipment addition from DOE, Ames Laboratory will purchase a field-emission scanning electron microscope (FE-SEM) for roughly $800,000.

The other two pieces—a focused ion beam microscope, known as a FIB ($1.7 million) and an aberration-corrected scanning transmission electron microscope or STEM ($3.5 million)—are now funded as well. DOE allocated funds to help purchase the FIB. A coalition of Iowa State's Departments of Materials Science and Engineering, Chemical and Biological Engineering, Mechanical Engineering, Chemistry, and Physics, the Deans of Liberal Arts and Sciences and

> This facility will provide a significant boost to the characterization capabilities at both **Ames Laboratory** and **Iowa State University.**

Engineering, the Senior Vice President and Provost, and the Senior Vice President for Business and Finance all contributed to the funding of the FIB and the STEM.

"We are reviewing capabilities of the various manufactures and developing required specifications in preparation for placing an order," Kramer said.

Once the order is placed for the three new pieces of equipment, the work of moving the existing Tecnai TEM and the supporting sample preparation equipment will be scheduled so as to minimize the down time for current microscopy work. Once the new facility is operational, the other two existing scanning electron microscopes and an older TEM, now obsolete, will be removed and the lab space in Wilhelm Hall reclaimed for other purposes.



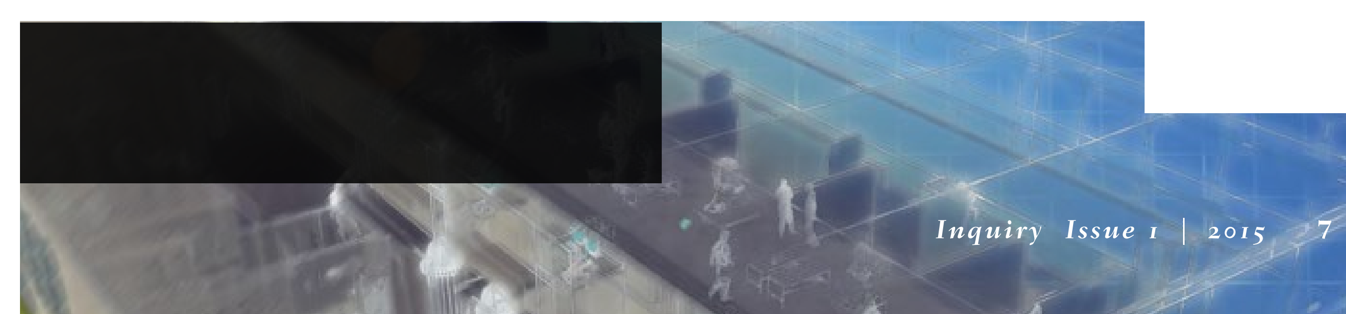
***Top:*** *The new Sensitive Instrument Facility sits near the existing Applied Science II building in the background.*
***Right:*** *Interior view of one of the office spaces.*

Construction of the SIF is wrapping up, however at this point, only four of the six instrument bays are being finished and will house the equipment listed earlier. The remaining space will be allocated based on equipment needs and compatibility that allows the best utilization of that space. A multi-stage commissioning process will take place to insure that the building and its electrical and mechanical systems perform according to specifications before the Lab takes ownership.

"Scheduling could be a bit of a dance to make sure that all of our ISU partners who have funded the equipment get their appropriate access," Kramer said. "I anticipate that it will be in use 24/7. The facility is our space to manage and users will be brought on as Ames Lab associates with all the requisite training in terms of safety, policies and procedures, and how to operate the equipment."

"One benefit of the new equipment is that it allows one to observe the operation of, and even operate, the instruments remotely," he continued. "I'm considering setting up a location on campus where samples could be dropped off. Researchers would book time on the equipment, their sample would be loaded and then control of the equipment would be handed off to them remotely. They could perform their scans without setting foot in the SIF."

"Obviously, we have a lot of details to work out," Kramer said, "but this facility will provide a significant boost to the characterization capabilities at both Ames Laboratory and Iowa State University."




Aberration corrected STEM
0.4nm
Tecnai TEM (existing)
10 µm
Focused Ion Beam (FIB) microscope
1 µm
Field-emission SEM
Conference Room
Office
Office
Repair Shop
Wet Lab
Dry Lab

# Magnetism at Nanoscale

As the demand grows for ever smaller, smarter electronics, so does the demand for understanding materials' behavior at ever-smaller scales. Ames Laboratory physicists are building a unique optical magnetometer to probe magnetism at the nano- and mesoscale.

The device, called a NV-magnetoscope, makes use of the unique quantum mechanical properties of nitrogen-vacancy (NV) centers in diamond. The low-temperature NV-magnetoscope setup incorporates a confocal microscope (CFM) and an atomic-force scanning microscope (AFM).

The NV-magnetoscope will be able to sense the extremely weak magnetic fields of just a handful of electrons with the spatial resolution of about 10 nanometers.

"We want to determine magnetic textures more precisely than ever before, at smaller scales than ever before," said Ames Laboratory physicist Ruslan Prozorov. "Our hope is to understand nano- and mesoscale magnetism, learn how to control it and, eventually, use that to create a new generation of technologies."

*Experimental physicists Ruslan Prozorov (left) and Naufer Nusran (right) and theoretical physicist Viatcheslav Dobrovitski (center) are collaborating to bring the preciseness of quantum mechanics to measuring nanoscale magnetism in Ames Laboratory's NV-magnetoscope. The development of the NV-magnetoscope is a flagship effort of Ames Laboratory's "Magnetic Nanosystems: Making, Measuring, Modeling and Manipulation" research team, led by Prozorov. Theoretical work is done by Dobrovitski and hands-on experimental work is done by a dedicated postdoctoral researcher, Nusran, who has built the experimental setup, the first NV-centers optical magnetometer fitted into the low-temperature AFM/CFM system, first of its kind in the United States acquired from Germany's Attocube.*

## Green Laser Light Excites the NV Center

"Electrons start at low-energy quantum states. And the green laser light 'kicks' them to a high excited state. The rules of quantum mechanics say that those electrons must return back to the lower energy level. If an electron was excited from a non-magnetic level, it always emits red light. However, if it was excited from one of the low-energy magnetic levels, it most likely relaxes back without any emission.

Microwave radiation is used to scramble electrons between low-energy magnetic and non-magnetic states, reaching maximum population of the magnetic states when the interlevel energy difference matches microwave energy. Therefore, by scanning microwave frequency, red fluorescence will cause double-dip spectra, corresponding to two magnetic energy levels, split by the magnetic field (called Zeeman splitting). The distance between the dips is proportional to the magnetic field at the location of an NV center," said Prozorov.

## Detector Counts Red Photons

As excited electrons lose energy and return back to the low-energy state, they emit red light. A detector counts the number of red photons.

## NV Centers "Feel" Sample's Magnetic Fields

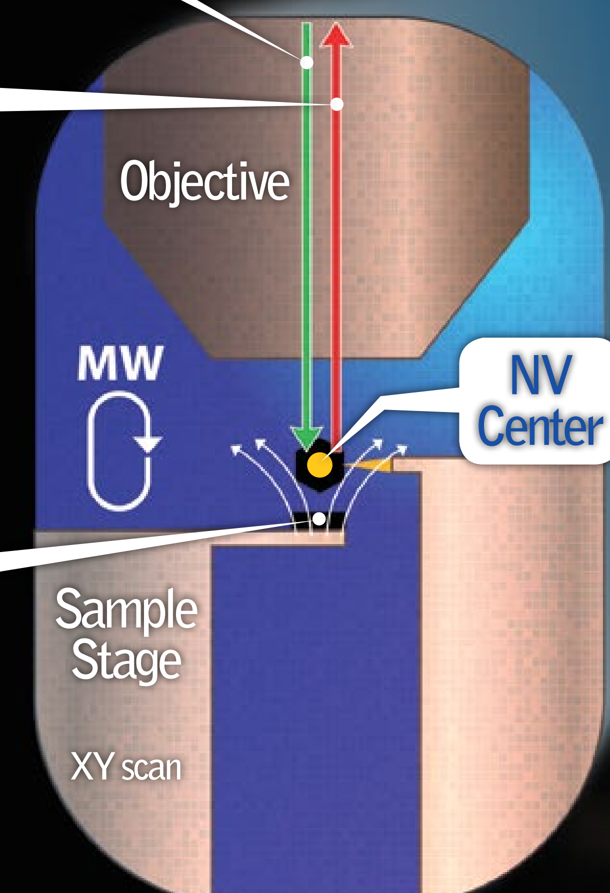
A roughly 100-nanometer-long diamond containing NV centers is attached to the AFM tip. The confocal microscope focuses on a single NV center, collecting red photons only from one tiny area while blocking out outside "noise." The sample of interest is scanned below the NV center. The NV center "feels" the variation of magnetic fields produced by the sample.

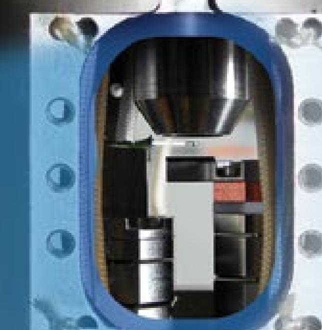
"When the sample of interest is brought close enough to an NV center, the sample's magnetic field is extended to the location of the NV center and affects the center's quantum energy levels. By accurately moving the sample in two dimensions close to the NV center, we can reconstruct the magnetic field intensity map produced by the sample. This, in turn, gives access to the magnetic properties of the sample itself," said Prozorov.

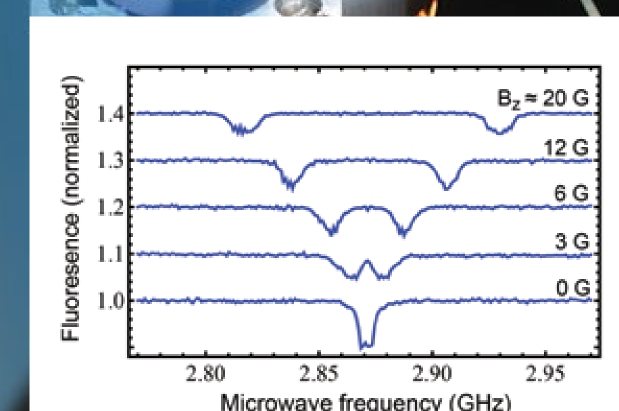
## NV Centers

**Usually, diamonds are most valued when they're perfect and big. But physicists see special value in diamonds' tiny flaws: a certain kind of imperfection, called a nitrogen-vacancy (NV) center, serves as a very sensitive sensor of the magnetic field exactly at the location of the NV center. NV centers are created when a carbon atom is substituted with a nitrogen atom. When there is a missing atom or a "vacancy" nearby the nitrogen atom, this forms the stable pair called the nitrogen-vacancy center.**

**What makes NV centers so useful? Physicists know a lot about how NV centers work. (In fact, Ames Laboratory is home to one of the world's leading experts on NV centers, theoretical physicist Viatcheslav Dobrovitski.) Scientists know how much energy it takes to push electrons from the lowest energy, or ground state, to an excited state and, more importantly, how much energy will be released in form of a red photon when the electron relaxes back to the low-energy level. NV centers' well-defined quantum energy levels are extremely sensitive to a magnetic field. This sensitivity enables the NV-magnetoscope to detect very small magnetic fields – such as that produced by nano- and mesoscale magnetic materials, for example – by reading optical fluorescence emitted by the excited NV centers.**



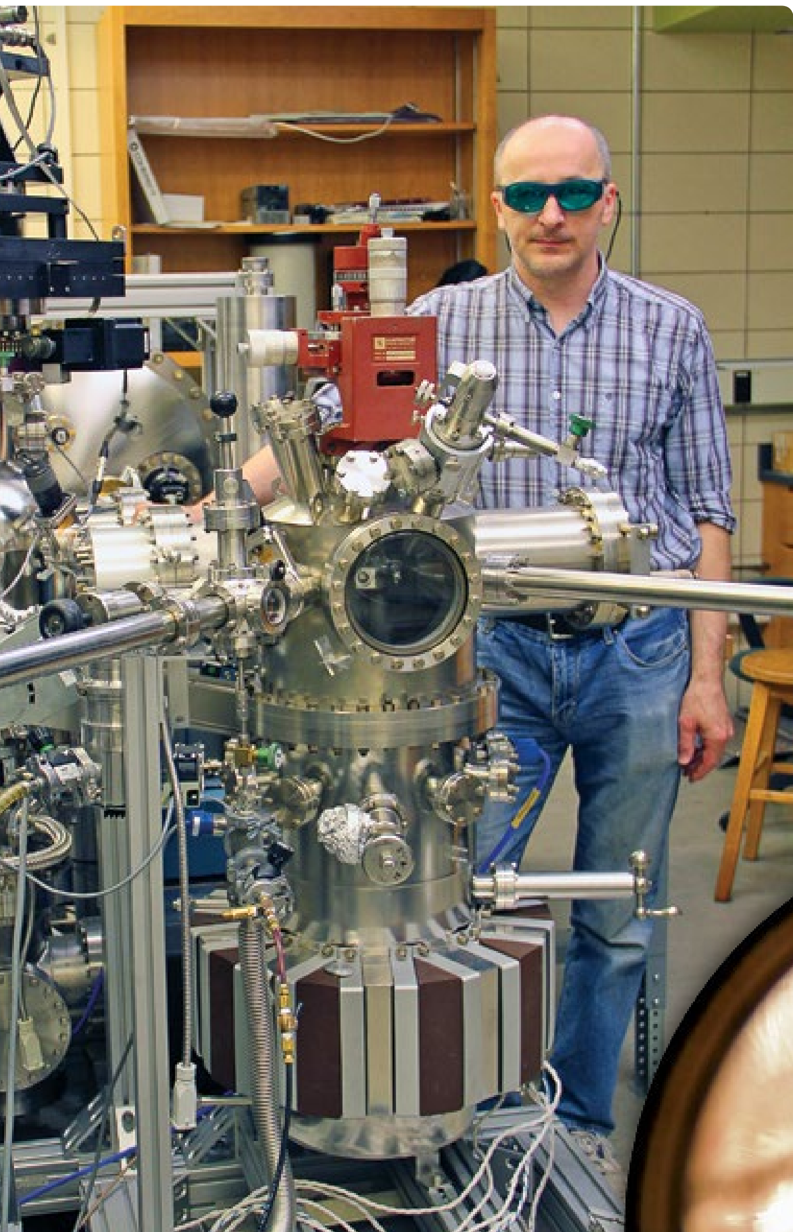



*"NV-magnetoscope measures the intensity of red light fluorescence as a function of microwave radiation frequency. The distance between the two depressions in the plot tells us about the strength of the magnetic field in the location of the NV center," said Prozorov.*

# Homegrown Solution

## for Synchrotron Light Source

BY KERRY GIBSON

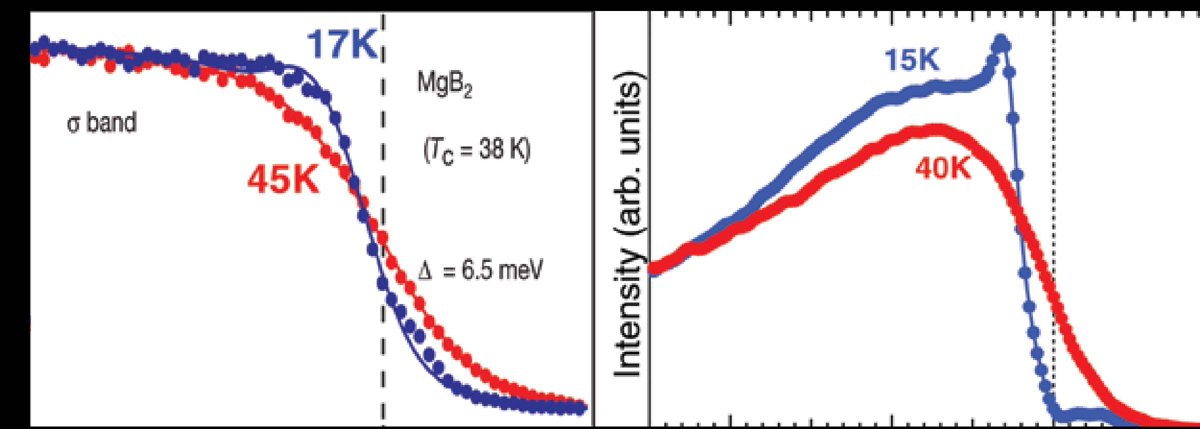


Adam Kaminski stands next to the equipment he assembled to conduct angle-resolved photoemission spectroscopy in his lab using laser light and a potassium beryllium fluoroborate crystal to excite electrons in the material being studied.

It's often said that necessity is the mother of invention. Such was the case for Ames Laboratory physicist Adam Kaminski who took the research challenge he was facing and turned it into a new solution that will help advance his research.

Two years ago the National Science Foundation closed the synchrotron in Stoughton, Wisc. More recently, Brookhaven National Lab closed its synchrotron light source to make way for a more advanced and powerful facility. Concerned that this would leave him without the low-energy light source he needed to study the electronic properties of new materials, he improvised, and the result was the development of a new technique that provides a homegrown, laboratory-based solution.

Kaminski uses a technique called angle-resolved photoemission spectroscopy (ARPES) in which light energy (photons) is directed at a sample being studied. The photons cause electrons in the sample to be emitted into a vacuum. An electron analyzer measures the energy and momentum of these electrons, providing details about the electron properties within the material.




*At left is a graph of synchrotron results of magnesium diboride that show a surface band that curves relatively smoothly. At right are results of the same material from the tunable laser ARPES that shows a dramatically enhanced plot with a sharp peak and a slight dip before leveling off.*

Besides using synchrotron beam lines, lasers could provide the input energy needed, but there were problems with the existing technology. High-energy, tunable lasers offered variable photon energy, but lacked the resolution necessary for good results. Low-energy lasers provided excellent resolution, but the fixed photon energy limited their usefulness.

So Kaminski, who admittedly knew little about lasers, set about finding a way to make a low-energy laser that was tunable. In searching the literature, he found that such a tunable laser had been suggested, but had never been used in ARPES systems. The laser used a potassium beryllium fluoroborate (KBBF) crystal to quadruple the frequency of infrared laser converting photons to the required "vacuum ultra-violet (UV)" range.

Obtaining such a crystal wasn't easy. Kaminski found that the main source for the KBBF crystals, China, had embargoed their export. However, he found a research group at Clemson University that was able to grow him the crystal he needed. He was also able to obtain funding through the DOE Office of Science to build the new system. As an added bonus, the crystal growth and preparation was commercialized by Advanced Photonic Crystals, LLC. This will make them available in U.S. for applications such as UV photo lithography, spectral analysis and defense.

In simple terms, Kaminski's system uses a pair of lasers, with the first acting as a pump for the second one. The resulting beam consists of very short pulses (one quadrillionth of a second) and very high (400 kW) peak power and is directed into a vacuum chamber that contains lenses, mirrors and the above mentioned "magic" crystal. This process quadruples the energy of the photons. By tuning the wavelength of the second laser and rotating the crystal, one can then tune the energy of the produced UV photons. The beam is then focused at the sample in an ultra-high vacuum chamber and a connected electron analyzer measures the electrons emitted from the sample.

"Development of a laboratory-based solution was really important," Kaminski said. "Our beam is smaller, photon flux is higher by one or two orders of magnitude, and energy resolution is better by a factor of five."

For certain experiments, such as Kaminski's, that can translate into significantly better data. As illustrated by the graphs (above), synchrotron results of magnesium diboride show a surface band that curves relatively smoothly. Results from the tunable laser ARPES shows a dramatically enhanced plot with a sharp peak and a slight dip before leveling off.

"Our system has significant advantages," Kaminski said. "It offers much higher resolution. When a researcher has a sample they want tested, we can usually do it the next day."

Kaminski has performed ARPES measurements for a number of research groups at Ames Laboratory as well as researchers at Sandia National Laboratory and Princeton.

"It's great to have the capability to perform measurements right here in the Ames Laboratory," he said, "and it's busy 24/7!"

# What is the future of cool?

## Science community discusses the state of magnetic cooling technology

BY LAURA MILLSAPS

The household refrigerator, sitting in the corner of virtually every U.S. kitchen, has been essentially the same for almost 100 years; air-conditioning, based on the same technology, almost 75 years.

But with the traditional vapor-compression model reaching its limits for advancement in efficiency, cooling still eats up as much as one quarter of U.S. daily energy consumption.

So, what if refrigeration systems could be even better? What is the future of cool?

Ames Laboratory, together with the larger scientific community, believes it could be magnetic cooling, a refrigeration system which exploits the magnetocaloric effect—a temperature change of a material caused by exposing it to a changing magnetic field. Scientists have been attempting for years to push this promising energy-efficient alternative over the gap between fundamental research and applied technology.

This April, Ames Laboratory Chief Research Officer Duane Johnson and scientist Vitalij Pecharsky helped organize and participated in a workshop, Advancing Materials for Efficient Cooling 2015, hosted by the Maryland Nanocenter at the University of Maryland.

There, Pecharsky said, academia, national laboratory scientists, and representatives from industry met to discuss the current limitations of all three types of solid state cooling based on one of three so-called caloric effects—electrocaloric, elastocaloric, and magnetocalorics, Pecharsky's area of research interest.

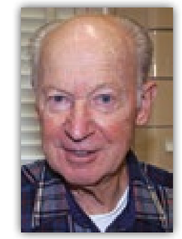
"The first big question is whether this technology is still worth pursuing, and the overwhelming sentiment is 'yes,'" said Pecharsky of the workshop. "The potential gains in energy efficiency are too significant to ignore."

Almost 20 years ago, Ames Laboratory scientists Karl A. Gschneidner, Jr. and Pecharsky announced groundbreaking progress in magnetic refrigeration—they discovered a gadolinium alloy that allowed magnetocaloric cooling to occur at room temperature, and a prototype refrigeration system was built in partnership with Astronautics Corporation of America Ltd.

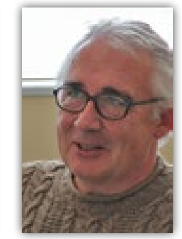
Since then, research at Ames Laboratory and globally has progressed, and there are occasionally demonstration models put forth by industry, but there is still no currently known commercially available product that uses a magnetic cooling system.



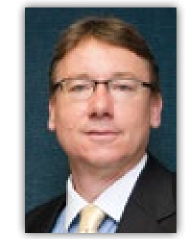
**The potential economic and societal impact is enormous, if you think about all the ways in which we use cooling technology.**



*Karl Gschneidner*



*Vitalij Pecharsky*



*Duane Johnson*

"'What needs to happen next?' is one of the questions the workshop was held to answer," Pecharsky said.

"Everyone agrees we need better materials, it's as simple as that," he said. "We have only a few families of materials that have a reasonable magnetocaloric effect. Almost 20 years ago we called it giant—a giant magnetocaloric effect. Now we know that it was just reasonable, even though at that time it was just about the strongest known. We could make a device with these reasonable materials, but in order for them to become commercially successful, we will need stronger effects that can be triggered by smaller fields."

"We're not quite there on the science side yet," Duane Johnson concurred. "We not only need to have a material with these caloric cooling properties, but ones that are controllable within a range of temperatures for which various forms of cooling systems are used. That's going to be key to getting manufacturers interested."

Even with a superior magnetic material, it's a big leap for manufacturers to make.

"For the consumer, the look and function of a magnetic refrigerator wouldn't change," said Pecharsky. "But for the manufacturer this is a radical departure, replacing a compression unit with an entirely different system. Current refrigeration technology is highly refined, relatively inexpensive to build, and cost-effective to operate. Until science can provide them and their consumers with a big advantage, it won't be financially viable for them to retool production."

That big advantage is a potential 20 to 30 percent reduction in the energy cost of refrigeration.

"That's roughly equivalent to the U.S. import of oil every day, energy-consumption wise," said Johnson. "The potential economic and societal impact is enormous, if you think about all the ways in which we use cooling technology."

Pecharsky said the improvement in materials wouldn't need to be a large one to push the technology over to a successful commercialization.

"If we could incrementally improve the magnetocaloric effect using inexpensive and readily available elements, it wouldn't even begin to push the theoretical performance limits of these materials and would still easily be the more efficient technology," said Pecharsky. "Scientifically speaking, I am confident we can get there."

That is where Ames Laboratory's strength, basic energy sciences, comes in, Johnson said.

"Materials design is a task best served by basic energy research, and we have the history and expertise right here. That's where Ames Laboratory has a great opportunity: for some forward-thinking basic energy sciences research to come together with applied sciences and partner with industry to bring a significant improvement to a technology that nearly everyone uses."

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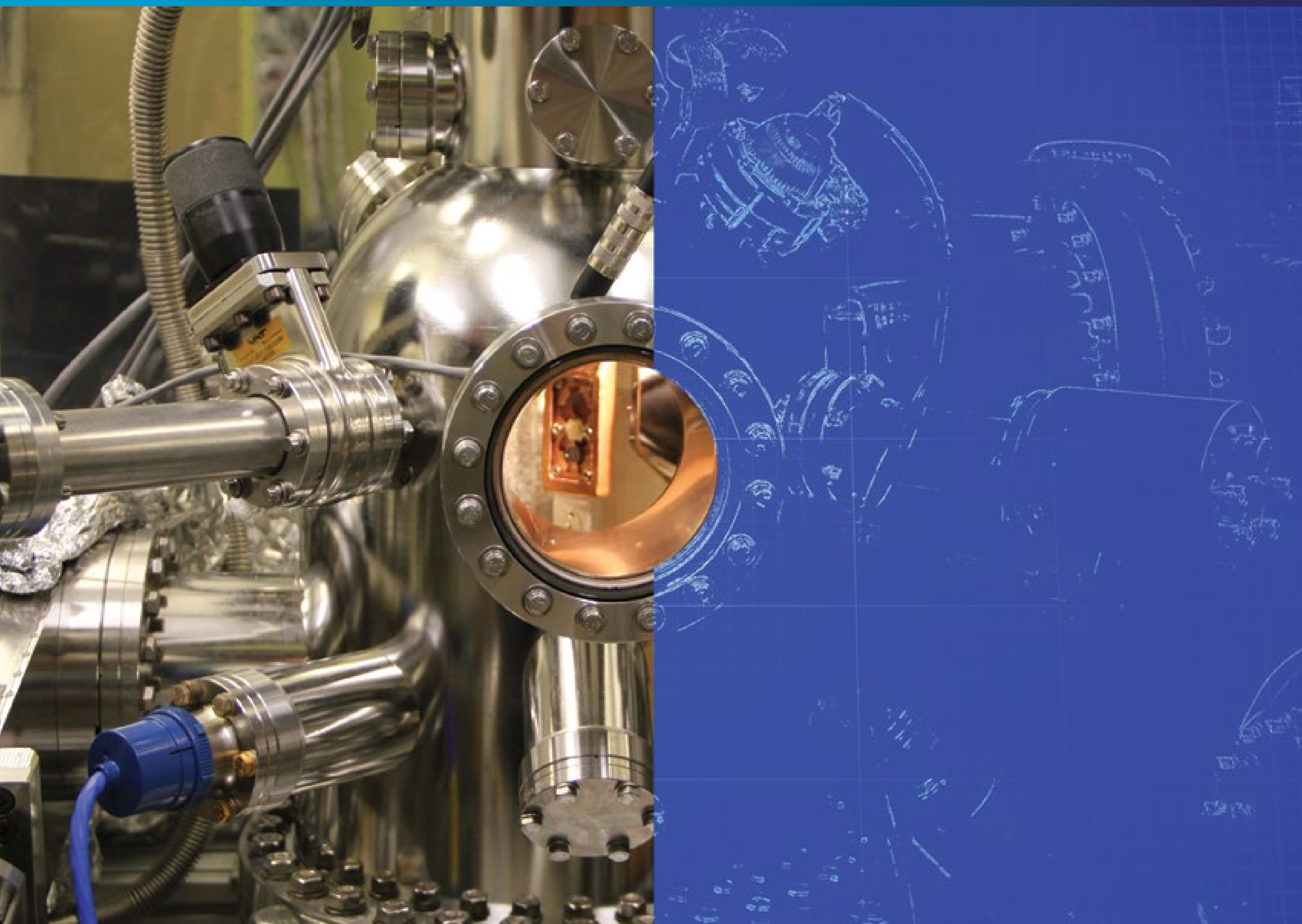

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