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Low-temperature, High-magnetic Field Structural Research a Reality

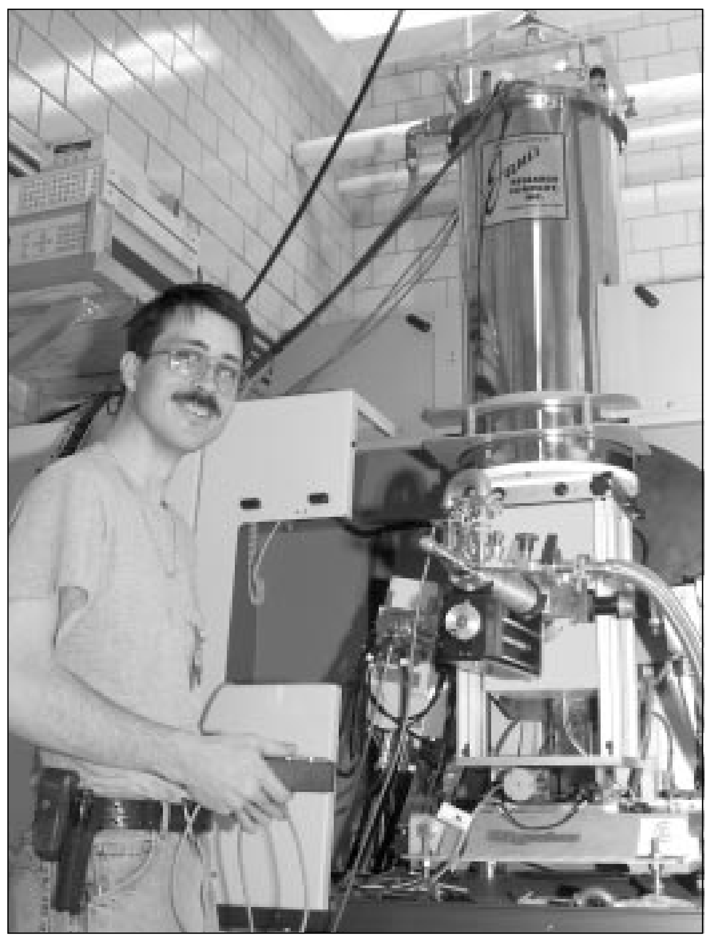
Gschneidner, Pecharsky and co-workers put together new research tool

A new, custom-built piece of equipment in the basement of Spedding Hall is set to give a group of Ames Lab researchers an unprecedented look at the effects of low temperature and high magnetic field on the crystal structure of materials.

Seven years ago, senior scientist Vitalij Pecharsky came up with the idea of integrating a superconducting magnet and a cryostat with a powder X-ray diffractometer. The superconducting magnet creates and maintains a strong and homogeneous magnetic field around the specimen. The cryostat allows material samples to be lowered to temperatures down to 4 degrees Kelvin (the temperature of liquid helium). The combination allows a sample to be subjected to a magnetic field of up to 35 kiloOersteds (kOe) at any temperature from 4 to 300 degrees Kelvin.

The diffractometer probes the sample with a high intensity X-ray beam and records how the crystal planes within the material diffract the beam. The recorded spectrum then can be matched against known patterns — like fingerprints — for specific materials. If the crystal structure was previously unknown, the arrangement of atoms in the crystal lattice can be determined with high precision by analyzing the scattered intensity using standard techniques. In either case, the researchers now have a tool that enables them to determine how the two thermodynamic variables — temperature and magnetic field — are influencing the structure.

"There's nothing individually special about these pieces of research equipment," Pecharsky says, "but they've never been combined in a lab outside of a synchrotron or neutron scattering facility to allow this level of study at low temperature and high magnetic field simultaneously." He adds that researchers at Keio University in Japan have a somewhat similar apparatus, but with much more limited capabilities. *continued on page 2*



Research technician Roger Rink shows off the X-ray diffractometer/cryostat that has been assembled in the basement of Spedding Hall. The diffractometer, which uses an X-ray beam to show the crystal structure of materials, was outfitted with the cryostat — the tall shiny cylinder — to allow study at low temperature.

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"From the beginning, we were aware of severe geometrical constraints imposed by the superconducting coil located inside a cryostat," says Pecharsky, "and we worked closely with our vendors to ensure that an adequate amount of diffraction data can be collected so we'd be able to determine small changes in crystal structure with high precision. This is something that has never been achieved before, and we are proud of this unique capability."

Combining the separate pieces of equipment has been no small feat. A rotating anode Rigaku wide-angle X-ray diffractometer was acquired first so the cryostat (with the integrated superconducting magnet) could be custom-built by Janis Research to meet a set of both physical and scientific specifications. However, the beauty of the system is that the diffractometer can still be used as a stand-alone research tool.

"It's been used every day it's been available," senior metallurgist Karl Gschneidner, Jr. says about the diffractometer, "so we've gotten quite a bit of use and new structural information out of it while we waited for the rest of the pieces to come together."

With the cryostat ordered, the next step was to figure out how to position the 500-pound piece of equipment precisely — within a thousandth of an inch — in relationship to the X-ray beam. Pecharsky, Gschneidner and research technician Roger Rink turned to Terry Herrman, manager of the Lab's R&D Engineering Services group, for help in solving the problem.

"The slides (adjustable metal rails) we've used previously for other equipment could only handle about 40 pounds," Herrman says, "so it took some searching to find a vertical slide system that could handle that kind of load." In fact, the researchers and Herrman joke about the bulky, "blast-proof" control box that came with the unit — evidence of its industrial heritage — that was replaced with more standard lab controls.

"Alignment is very simple," says postdoctoral research fellow Aaron Holm, who is in charge of operating the equipment. "Once we get the horizontal x and y coordinates lined up, the vertical alignment takes no time at all." The unit performs as expected, and initial testing has also proven the unit to be quite temperature stable, Holm adds. Long-term temperature stability is critical for successful experiments because a single data collection run can take anywhere from one to twenty hours.

The setup not only required a slot in each side of the cryostat housing to provide openings through which the X-ray beam can "shine," but there also were necessary precautions to provide shielding for the operators from the X-ray radiation. (Oh, in addition, they had to install a bigger door in the room to get the equipment in.)

The only hitch in the equipment thus far is a problem caused by the strong magnetic field generated by the split-coil superconducting magnet located inside the cryostat. It generates a field so powerful that it pulls the tube-like metal body inside the cryostat out of position. As a result, the slots for the X-ray beam shift by a fraction of an inch and partially obstruct the X-ray beam, causing considerable drop in the measured intensity. Rink is in the process of shimming the innermost part of the cryostat so it can't flex. Once that's complete, the equipment will be calibrated and put into service.

"We have a long list of things to look at," Gschneidner says, starting with the gadolinium-silicon-germanium alloys with giant magnetocaloric, magnetoresistive and magnetostrictive properties that his group discovered a number of years ago. The equipment will allow a close investigation of the materials at various combinations of temperature and magnetic field to see if additional, and as yet unknown, crystalline phases exist.

"We have suspicions of what to expect," Gschneidner says, "but if we knew for sure, there'd be no need for conducting further research. As always, there are a few surprises awaiting us. That's what makes research so much fun."

To test materials, fine metallic powders of the various materials are mixed with a cyrogenic varnish, Pecharsky says. The mixture is then spread over an area roughly one-inch square and about 1/64th of an inch thick. Once the sample dries, it's polished smooth and placed inside the cryostat, and the cryostat is positioned in the diffactometer.

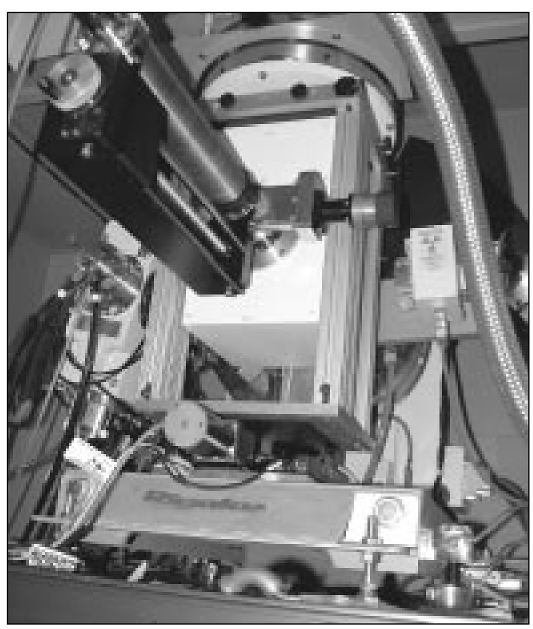
"The varnish keeps the powder in place when it's subjected to the magnetic field," he says. "The varnish also remains noncrystalline, even at low temperature, so it doesn't interfere with the X-ray diffraction pattern and the determination of the crystal structure of the powder."

Though it's taken seven years to get to this point, it's obvious the group feels it has been well worth both the wait and the effort.

"Low-temperature facilities with magnetic-field capabilities exist in many places and so do X-ray diffraction units," Pecharsky says, "but except for a few national user synchrotron and neutron diffraction facilities around the world, none have the ability to study materials at both low temperature and high magnetic field using X-ray diffraction. Our in-house, on-site system is truly unique and should greatly further our understanding of the critical relationships between structure and properties of rare earth and other magnetic materials."

The combined cost of the equipment, and the work to integrate the components is roughly $650,000, including $116,000 for the custom-built cryostat. Funding has been provided through the DOE Office of Science's Materials Science Division. ■

~ Kerry Gibson



The diffractometer's X-ray beam is focused on the sample through the slot in the side of the cryostat. Engineering Services designed and built the carriage — the frame supporting the cryostat — to allow the 500-pound piece of equipment to be positioned with thousandth-of-an-inch precision.