NATIONAL HIGH MAGNETIC FIELD LABORATORY

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FROM THE DIRECTOR'S DESK

Our Magnet Lab is in motion. Springtime annually heralds the "Relay for Life," an eighteen-hour charity walk that is near and dear to the MagLab's heart. My thanks to the many folks who contributed, and who walked as part of the MagLab team. I'm sure that several of us lost a few pounds in the process, and can now fit into the suitcoat (see photo) that I hardly ever wear.

Our technology is on the march. This issue highlights recent developments in NMR spectroscopy: using quadrupolar nuclei to probe local electric fields and using high-Tc superconductors to build probes. You also can read about the 35 tesla world record for

our Florida-Bitter DC magnet technology. I highlight here what is perhaps the heaviest magnet technology news of all: the lifting of a nine-ton outsert magnet into its cryostat, an assembly milestone of our 100 tesla multishot magnet. This magnet will provide users later this year their first access to milliseconds above 80 teslas, then 90 teslas, and then higher as we get a "feel" for how the magnet—and its component materials—will behave in this new and extreme regime.

Our managers are flying high. This spring, Guebre X. Tessema, our NSF program manager, will be joining Alex Lacerda, the MagLab's associate laboratory director for User Programs, to tour several of the European magnet labs to see first hand their latest achievements. I continue to enjoy the buzz generated at conferences from research achievements in the MagLab's user programs. Buzz that has me flying around the nation and the world in response to invitations to talk about the MagLab and its research programs…nearly one week every month in the air. In May, Harvard, MIT, and Dartmouth want to hear the latest MagLab news.

Lab Featured in History Channel Show

Magnet Lab Director Greg Boebinger was featured in a History Channel special described as "a cross between a 'NOVA' show and a 'Twilight Zone' episode." The producers of "Alien Engineering" told a group of physicists, astronomers, and engineers to imagine that a flying saucer crashed and was recovered, and then asked them to decode the technology and figure out how the saucer runs. "This project illustrated how scientists approach the unknown: willing to entertain wild speculations, but ultimately requiring that they be replaced by scientific consistency that can withstand the tests of other scientists," said Boebinger. Part 1 of the two-part program included a discussion of antigravity and electromagnetic force fields and a trip to the Magnet Lab. It appeared February 6 and will be rebroadcast in the future.

Chinese Delegation Visits Lab

The Magnet Lab hosted members of the prestigious Chinese Academy of Sciences on February 23. The Chinese officials, who are in the formative stages of creating their own national magnet laboratory, hope to establish mutual understanding, build friendships and explore possible collaborations between the two labs.

Lab Partners in Summer Science Camp for Girls

The Magnet Lab, the Tallahassee Museum, and the Florida State University Office of Science Teaching are partnering with WFSU to produce a free, two-week summer camp for 16 eighth- and ninth-grade girls from the local area. The girls will participate in scientific experiments and go on field

trips. The camp, which will be held July 10 to 17, is funded through a grant with Dragonfly TV, a PBS show that features girls in science. At the lab, the girls will shadow a group of undergraduate female science majors.

ICR Hosts Chemistry Students

The lab's Ion Cyclotron Resonance program in February hosted nine undergraduate chemistry students from Valdosta State University. The visit of Professor Thomas Manning's students was part of an exploratory lab exercise in instrumental analysis in which a valid research project was used to demonstrate the strengths of ICR: high resolution and mass accuracy. After an introduction to the lab's Research Experiences for Undergraduates program (REU), the students learned more about ICR techniques and instrumentation while acquiring data for their research project. Crystal Tabron (sixth from the left), a senior who will graduate from VSU in May, presented the data during an undergraduate poster session at the American Chemical Society Meeting and Exposition held March 26-30 in Atlanta, Ga. For more information, visit http://oasys2.confex. com/acs/231nm/techprogram/P941970.HTM or

http://www.valdosta.edu/~tmanning/.

Crow's Feet Walk Again

More than 70 lab employees walked, sponsored a walker or team, or bought a T-shirt, luminaria or wristband in support of Florida State University's third annual Relay for Life. The three Crow's Feet teams – so named for the lab's patriarch, Jack Crow, who lost his own battle with cancer in 2004 – were chaired by Greg Boebinger, Brian Fairhurst, and Alex Lacerda and raised \$6,659 for the American Cancer Society. This was the second highest

amount raised at FSU.

Relay For Life is an overnight event, held each year at FSU's Mike Long Track, to celebrate cancer survivorship and raise money to help the American Cancer Society save lives. Crow's Feet walkers sporting the lab's trademark colorful (some might say "loud") Tshirts kept the relay going at all times during the 18-hour event, which started at 5 p.m. on March 31 and ended at 11 a.m. April 1.

Metal-Insul

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(at T=0) m Metal-Insulator transitions (MIT) remain poorly understood. The important specifics of the 2D case are that—in the absence of all interactions—the carriers are strictly localized by any disorder. Finite conduction (at T=0) may come about only due to the screened Coulomb interactions between electrons. In addition, the long range Coulomb forces themselves are also known to lead to glassy features for the electronic system by forming deep energy minima in its Free Energy relief.

Experiments by J. Jaroszyński and D. Popović are aimed to shed more light on the MIT issue for the 2D electrons in silicon. To somewhat separate effects of disorder and interactions, the authors studied relaxation processes for conductivity for the extreme regimes of strong deviations of the system away from the equilibrium by a sudden change in the gate voltage and tracing then the temporal conductivity relaxation. Most remarkable is the observed non-monotonic conductivity behavior. Thus, at a sudden decrease of the carriers' concentration, the conductivity first continues to decrease, as if the role of poorly conducting regions would initially prevail, and only after passing through a minimum, it gradually begins to grow up to its new equilibrium value. Details of the experiments are explained below. Qualitatively, the main output from their results is, in my opinion, the clear demonstration of the essential role of dynamical spatial inhomogeneities in the relaxation processes.

Lev Gor'kov, Science Council chair

SCIENCE COUNCIL

Out-of-Equilibrium Dynamics near the Metal-Insulator Transition in a Two-Dimensional Electron System in Silicon

J. Jaroszynski, NHMFL D. Popovic, NHMFL

The interplay of strong electronic correlations and disorder is believed to be responsible for a plethora of new phenomena occurring in many complex materials in the region of the metalinsulator transition (MIT) .¹ For example, there is now evidence of strongly inhomogeneous intermediate electronic phases that emerge between the metal and the insulator in many systems, ranging from manganites, cuprates, and other transition metal oxides, to diluted magnetic semiconductors, and even the Kondo alloys. Even though such inhomogeneous phases are often characterized by glassy fluctuations and response, electron glasses in general remain largely unexplored, in particular in the vicinity of the MIT. In many complex systems, the MIT is accompanied by changes in magnetic or structural symmetry, which complicates the situation even further. On the other hand, low-density twodimensional (2D) electron and hole systems in semiconductor heterostructures, where the MIT has been a subject of great interest and debate,^{2,3} represent particularly appealing model systems for studying the effects of interactions and disorder in a controlled and systematic way.

Previous transport and noise studies⁴⁻⁶ in a 2D electron system (2DES) in Si have shown signatures of glassy behavior at densities n_s lower than some well-defined density n_g , such that $n_g > n_c$ (n_c – the critical density for the MIT) (Fig. 1). The glassy regime was characterized by slow, correlated dynamics, consistent with the hierarchical model of glasses, and was found to persist even when the 2DES is fully spin polarized, indicating that the charge degrees of freedom are responsible for the glassy behavior. Those results also demonstrated the existence of an intermediate $(n_c < n_s < n_g)$ glassy regime, which separates the still poorly understood 2D metal and the glassy insulator. We note that, even though n_g is in the metallic phase (*i.e.* $\sigma(T \rightarrow 0) \neq 0$), the conductivity σ is so small that k_{F} /<1 $(k_F -$ Fermi wave vector, l - mean free path), which violates the Mott limit for the metallic transport in 2D. Such an unconventional conducting regime ("bad metal"), exhibiting a non-Fermi liquid (NFL) $\sigma(T)$,^{4,6} is found in many strongly correlated materials (*e.g.*) cuprates).

In order to elucidate the microscopic origin of the glassy behavior in a 2DES in Si, we have performed a study of the relaxations of σ after excitation far from equilibrium by a rapid change of

Figure 1. The (n_s, T) phase diagram of a 2DES in Si in zero magnetic field. The temperature dependence of conductivity, $d\sigma/dT$, changes sign at an electron density n_s^* , where $k_F l$ -1. The glass transition and the MIT take place at T=0 at densities n_g and n_g , respectively. In high-mobility (low disorder) samples, $n_{\epsilon} \approx n_{\epsilon} \approx n_{\epsilon}$, *i.e.* the onset of glassy dynamics almost coincides with the MIT. In that case, the intermediate, metallic (NFL) glassy phase is absent but it emerges in the presence of a parallel magnetic field.⁶

carrier density n_s , controlled by the gate voltage V_{g} , at low temperatures T. The samples and the measurement technique have been described in detail elsewhere.⁴ Here we show the data for the $2\times50 \text{ }\mu\text{m}^2$ sample, where $n_g(10^{11} \text{cm}^{-2}) \approx 7.5$, and $n_c(10^{11} \text{cm}^{-2}) \approx 4.5.$

The experimental procedure was as follows. The sample was cooled from 10 K to the measurement T with an initial gate voltage V_g^i , corresponding to an initial electron density n_s^i . Then, at time t=0, the gate voltage was switched rapidly (within 1 s) to a final value V_g^f (corresponding to a final electron density n_s^f and $\sigma(t, V_g^f, T)$ was measured. By warming up to 10 K and cooling down again to T with the V_g^f applied, the equilibrium conductivity $\sigma_0(V_g^f,T)$ corresponding to the given V_g^f (or the given final density n_s^f) and T was obtained. At the end of the run, the sample was warmed up to 10 K, the electron density changed back to the same initial value n_s^i , and the experiment was repeated at a different T for the same V_g^f . Finally, the

whole procedure was repeated for different values of final densities n_s^f . Of course, similar measurements were also carried out for various combinations of n_s^i and n_s^f .

Fig. 2 shows a typical experimental run. The relaxations of conductivity, $\sigma(t)$, V_g^f , T), normalized to the corresponding equilibrium conductivity $\sigma_0(V_g^f, T)$, are shown for different T and fixed initial and final densities. In this example, the initial density $n_s^{-1}(10^{11}cm^{-2})=20.26$, corresponding to the regime where $k_F l \sim 1$ (around n_s^* in Fig. 1). The final density $n_s^{f}(10^{11} \text{cm}^{-2})$ = 4.74, just slightly above n_c , the critical density for the MIT. It is striking that σ(t) first overshoots its equilibrium value, goes through a minimum, and only then approaches σ_0 . The minimum in σ shifts to longer times with decreasing T until, at sufficiently low T, it falls out of the time window of the measurements. A detailed and careful analysis of the data reveals⁷ several striking features.

(1) At times above the minimum in $\sigma(t)$, the system equilibrates via a simple exponential process with a characteristic

Figure 2. The relaxations of conductivity $\sigma(t)$, normalized by the corresponding equilibrium value σ_0 , after change of the electron density at several T, as shown. The initial electron density $n_s^{\text{i}}(10^{11} \text{cm}^2) = 20.26$, corresponding to the regime where $k_p l \sim 1$ (around n_s^* in Fig. 1). The final density $n_s^{f}(10^{11}cm^2) = 4.74$, just slightly above n_c , the critical density for the MIT.

time \propto exp(E_A/T), where E_A≈57 K independent of n_s. Therefore, the equilibration time diverges as T→0, signaling a transition at T_{g} =0. The data show that, even though the system is, strictly speaking, glassy only at T=0, at low enough T (*i.e.* \leq 1 K) the dynamics is glassy on all experimentally accessible time scales (even longer than the age of the Universe!).

(2) At times before the minimum in $\sigma(t)$, all the relaxations can be collapsed onto a single, strongly nonexponential curve: $\sigma(t,T)$ σ_0 (T) \propto t^α exp[-(t/τ)^β] with 0<α<0.4 and 0.2<β<0.45, where τ \propto exp($\gamma n_s^{1/2}$)exp(E_a/T), E_a \approx 20 K. Since τ diverges as T \rightarrow 0, the relaxations attain a pure power-law form at T=0. These results are consistent with the continuous phase transition occurring at $T_g=0$. Similar scaling is observed in spin glasses above T_g . Further, the exponent α decreases from 0.4 at the lowest n_s measured, down to zero at n_g , consistent with the glass transition occurring at n_g as a function of n_s at T=0 (Fig. 1). Finally, since the ratio of the Fermi energy to Coulomb energy in 2D systems $1/r_s = E_F/U \approx n_s^{1/2}$, a strong and precise dependence of τ on $n_s^{1/2}$ strongly suggests that Coulomb interactions between 2D electrons play a dominant role in the observed out-of-equilibrium dynamics.

(3) The 2DES equilibrates in a somewhat counterintuitive way: the equilibrium is reached only after the system first goes farther away from equilibrium. An analogous overshooting is known to occur in some other types of glasses but the arguments developed there to explain this behavior are probably not applicable to our system. We speculate that this intriguing overshooting is related to the large size of the applied perturbation in our system, consistent with the results of some recent numerical work, but more experiments are clearly needed to resolve this issue.

In summary, we have observed strongly nonexponential relaxations in a 2DES in Si, which are some of the hallmarks of glassy behavior. Both power-law and stretched exponential relaxations reflect the existence of a broad distribution of relaxation times, which, we believe, is probably due to dynamical heterogeneities that are known to emerge in the out-of-equilibrium behavior of glassy systems. The data strongly suggest that, for $n_s \le n_g$, the 2DES undergoes a transition to a glassy phase as $T\rightarrow 0$, and that the Coulomb interactions between 2D electrons are primarily responsible for the observed out-of-equilibrium dynamics. Further work, however, is needed to clarify the microscopic details of the relaxation phenomena. Measurements in magnetic fields and on 2D systems in other materials are expected to provide valuable information.

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Attention SERS *User Support Scientists: DC Field Facility*

The strength and success of NHMFL users programs and facilities are carefully built around the synergies of the highest field magnets, unique instrumentation, and strong supportive services of faculty and staff. The pages of *NHMFL Reports* are often used to highlight the latest and greatest magnets and to make users aware of new instrumentation. This time, we want to direct your attention to the scientists who work closely with users of the DC Field Facility at Florida State University in Tallahassee. These are the people who express the user's point of view when new magnets are being designed, develop instrumentation for measurements that are possible nowhere else, and help users get and understand the data they need for high quality publications. In addition to all that, we expect them to pursue their own research, both independent of and collaborating with other users.

Each biography is followed by the scientist's email address so that you can easily ask for more information. You can see a list of their publications if you enter the person's name in the search engine at http://www. magnet.fsu.edu/search/personnel/

 Bruce Brandt, director of DC Field Facility *brandt@magnet.fsu.edu*

Luis Balicas earned his Ph.D. in solid state physics at the University of Paris-Sud, Orsay, France, with an emphasis in low-dimensional organic systems that display a variety of phenomena such as unconventional superconductivity, Mott localization, charge/spin density waves, and the quantum Hall effect. His current research interests include quantum spin systems; quantum criticality and heavy fermion physics; unconventional superconductivity in transition

metal oxides and in intermetallic compounds; and fermiology in unconventional metals such as the cuprates, ruthenates, rhodates, and cobaltates. Balicas uses a number of techniques: hydrostatic pressure using piston cylinder cells, linear and non-linear electrical transport (using pulsed techniques), torque magnetometry, and quartz dilatometry. In collaboration with Prof. Ho Bun Chan of the University of Florida and F. Balakirev at NHMFL-LANL, Balicas is currently developing silicon-based micromechanical devices. The plan is to develop the capability to measure magnetic torque with a torsional device(s) or magnetic forces (Faraday magnetometry) with ultra-high sensitivity, and to obtain the absolute value of the moment at high fields. *balicas@magnet.fsu.edu*

Eun Sang Choi graduated from Seoul National University in 1998 with a Ph.D. in physics. His studies focused on transport properties of conducting polymers such as highly doped metallic polyacetylenes. Since graduation, he has been working on highly correlated electron systems, mostly organic charge transfer salts, and his research activities have been related to studying novel ground states of such systems by measuring transport and magnetic properties.

Choi has extensive experience in transport property measurements (electrical, thermal and thermoelectric) under extreme conditions such as high magnetic fields, high pressures, and low temperatures. He has developed techniques for thermoelectric properties (thermoelectric power, Nernst effect) measurements at high field and high pressure, and has also been using and improving the magnetic property measurement techniques, such as magnetic torque, vibrating sample magnetometer (VSM), SQUID magnetometer, and ac susceptibility, that are available for DC Field Facility users.

He is currently developing a piezo actuator driven VSM system. If successful, this will improve the current VSM system of the Magnet Lab in two aspects: higher fields (≤45 T) and better low temperature control. He also plans to develop magnetometers that can measure absolute magnetic moments with better sensitivity than the VSM. According to Choi, "either high field SQUID or magnetic force techniques can be used for this purpose. The absolute magnetization measurements will provide important scientific information by providing quantitative information on the number of spins involved in magnetic transitions, for example." Many users are interested in such measurements. *echoi@magnet.fsu.edu*

Lloyd Engel earned his Ph.D. from Princeton in 1987. His current research program focuses on microwave and rf spectroscopy of twodimensional electron systems that exhibit the quantum Hall effects. This program has produced a number of publications concerning the electron solid and striped phases of such samples. The work is carried out using a broadband technique based on transmission lines capacitively coupled to the two-dimensional electrons. Spectra from

20 MHz to 20 GHz can be measured on a single cooldown of a single sample, typically at dilution refrigerator temperatures. Engel's collaborations with users include transmission line based measurements as well as broadband photoresistivity measurements on a wide variety of systems.

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Scott Hannahs received his degree from University of California at Los Angeles with Gary Williams working with the twodimensional interaction of ion sheets and the 2-D phonons of the helium surface. Since then his interests have revolved around the interplay of dimensionality and other physical parameters such as high fields, angle, and pressure. This work has involved many different physical systems, MgB_2 , HTSC, quantum Hall, organic conductors, and Langmuir-Blodgett films.

Hannahs has developed several techniques for sensitive transport measurements in the DC resistive magnets. Subsequent improvements in the experimental setups at the NHMFL have lead to measurements in the nanovolt range. This is still being developed into a simple facility to measure differential conductance for tunneling measurements. Hannahs has also actively worked on the problem of thermometry at high fields, and especially on the problems at both high and low temperatures. He has automated much of the data acquisition and control systems at the NHMFL.

Most recently, he and his collaborators have developed a technique for angular dependent measurement of heat capacity. This has been used to investigate the properties of such systems as unconventional anisotropic superconductors and Heisenberg frustrated spin insulators. This system will continue to be developed using small custom thermometers and heaters as an easy to use heat capacity system in the range of 20 K to 20 mK. *sth@magnet.fsu.edu*

Jan Jaroszynski is a new member of the DC Field user support group. He earned his master's in physics at Warsaw University. Between 1981 and 2000 he worked at the Institute of Physics, Polish Academy of Sciences, were he completed his Ph.D. and "habilitation." In Warsaw he was involved in experimental studies on diluted magnetic semiconductors as well as in their MBE growth. He was involved in spin dependent transport phenomena well before they were called "spintronics." In 2000 he joined the NHMFL Condensed Matter Physics Group.

His research interests at NHMFL-FSU focus on slow dynamics and conductance noise in a wide spectrum of materials such as silicon transistors and magnetic semiconductors, as well as in manganites, cuprates, and other complex materials. Jaroszyński notes that noise measurement is a very powerful tool for studying materials. For instance, when done in the presence of a high magnetic field, noise measurements can tell whether an observed phenomenon originates from the spin or charge of the particles involved. He is interested in developing hardware and techniques for noise measurements in high fields, in collaboration with others. Jaroszynski also provides to others his expertise on how to reduce unwanted noise coming from the environment and instruments, with a goal to substantially improve the effectiveness and cost of high magnetic field measurements.

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Philip Kuhns received his Ph.D. in Physics from the College of William and Mary, Virginia, in 1984. He completed his thesis work in

PHILIP **KUHNS**

solid state NMR and continued postdoctoral studies at MIT running experiments between 10 mK and 1 K in a top loading dilution refrigerator. He was hired as a staff scientist at MIT and continued running experiments and testing NMR instruments for Japanese Electron Optics, Ltd. He joined the Magnet Lab in its early stages and pioneered NMR in the resistive magnets. His interests are in nuclear magnetic resonance at the extremes, including low temperature NMR and high frequency or high field NMR techniques. Of immediate concern is NMR of microgram samples. His current areas of research interest are exotic ferromagnets, molecular nanomagnets, multiferroics, semiconductors and lowdimensional electron systems. He continues to promote the idea of NMR compatible magnets such as the series connected hybrid and a powered high field superconducting magnet along with acquiring an NMR capable dilution refrigerator for use in the high field magnets. *kuhns@magnet.fsu.edu*

Tim Murphy's education is in low temperature physics, and he supports users at the NHMFL who are doing high field/low temperature research in the Millikelvin Laboratory. His current research interests involve the study of heavy fermion/correlated electron systems at

low temperatures and high magnetic fields in collaboration with Stan Tozer and Eric Palm. They are currently working on the CeMIn_c $(M=Rh, Co, Ir)$ family of materials. CeCoIn₅ is especially interesting due to the emergence of a spatially modulated (FFLO) superconducting state in the high magnetic field-low temperature region of the phase diagram. The experimental techniques utilized for these studies are magnetization/susceptibility, transport, and surface conductivity/penetration depth. Murphy also is currently collaborating with Eric Palm and Chuck Swenson (NHMFL Magnet Science and Technology) on the development of a SQUID magnetometer for use at temperatures down to 20 mK and fields up to 20 T. Funding for this project is being provided through the NHMFL In-House Research Program. Other work includes advancing the current state of the art in low temperature/high magnetic field compatible thermometry, as well as expanding the portfolio of experimental techniques that can be used at the NHMFL. *tmurphy@magnet.fsu.edu*

Eric Palm received his undergraduate degree at Rice University. After teaching high school for two years, Palm went to Texas A&M where he received his Ph.D. studying quantum interference phenomena in HgCdTe field effect transistors. This low temperature physics research continued during a National Research Council postdoctoral fellowship at NIST, where he investigated the integer quantum Hall effect. He joined the NHMFL in the early 1990s, very soon after it opened, to help users do research at low temperatures and the highest possible magnetic fields.

His current research interests include heavy fermion superconductors, reduced dimensional systems, low temperature thermometry (especially in magnetic fields), and novel instrumentation for low temperature research.

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Dragana Popovic earned her Ph.D. from Brown University in 1989. After working as a postdoctoral research associate at the IBM T.J. Watson Research Center, and then as an assistant professor of physics at the City College of the City University of New York, she joined the NHMFL in 1995. She has been leading an independent, externally-funded (sole PI on several NSF grants) research group consisting of graduate students, postdocs, and visiting scientists. Her research program aims to understand the effects of the interplay of disorder and strong electronic correlations, which are relevant to a variety of complex materials. The focus is on the metal-insulator transition in two-dimensional (2D) electron systems, glassy ordering and glassy dynamics both in 2D and in bulk materials, such as cuprates and manganites, and on out-ofequilibrium phenomena.

Therefore, in addition to the more common variables (e.g. carrier density, temperature, magnetic field), *time* appears as a new parameter that presents numerous, unique possibilities even when employing standard electrical transport measurements. Timedependent transport and low-frequency resistance noise spectroscopy represent powerful tools for studying complex, glassy systems in general. They have been used extensively in Popovic's group in recent years, and have resulted in some ground-breaking work in 2D. Popovic's collaborative research includes time-dependent measurements of various systems, where experiments typically need to be carried out continuously over periods of several months. This requires a dedicated cryostat and, thus, it is not suitable for standard one or two-week magnet-time shifts. Popovic's collaborators have included groups not only from within the United States but also from Poland (externally-funded collaboration), Netherlands, United Kingdom, and Japan. *dragana@magnet.fsu.edu*

Arneil Reyes joined the Condensed Matter NMR group in 1997. He obtained his Ph.D. in physics from the University of California-Riverside and did his postdoctoral research with the Condensed Matter and Thermal Physics group at Los Alamos. From there, he joined the Science and Technology Center for Superconductivity at Northwestern University and conducted research on vortex dynamics and on low dimensional organics. He was a regular user before coming to NHMFL.

Reyes is currently in charge of the Condensed Matter Nuclear Magnetic Resonance (CMNMR) User Program at the Magnet Lab with expertise in RF electronics, computer automation, cryogenic and high pressure instrumentation. He co-directs doctoral dissertations of graduate students in the FSU Department of Physics while pursuing a vigorous research program using the high magnetic fields. His research interests include exotic magnetism in strongly correlated electronic systems such as heavy fermion compounds, small-gapped semiconductors, high temperature superconductors, spin glasses, nanomagnets, multiferroics, and lowdimensional electron systems. Over the years, the group has developed NMR instrumentation adapted to the resistive and hybrid magnets. It is unique to the NHMFL because there are no systems for such high fields available from commercial suppliers. These instruments include the first 2 GHz NMR spectrometer suited for the 45 T hybrid magnet. Recent additions to its capabilities include a 340 mK 3 He sorption refrigerator with a 24-hr run time. Plans are in place for the expansion of the program to a full-scale user facility with the addition of a high-homogeneity, fieldsweepable, 17.5 T, superconducting magnet by summer 2006 and a proposed top-loading dilution refrigerator that would allow users

more experimental time than is available in the DC Facility's resistive and superconducting magnets.

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Dmitry Smirnov received his Ph.D. from the A.F. Ioffe Physico-Technical Institute of the Russian Academy of Sciences in 1996. After graduation, he worked at the French National Pulsed Magnetic Field Laboratory as a postdoctoral research associate and as a visiting scientist. He moved to the NHMFL in October, 2003. Smirnov's research interests are in experimental physics of lowdimensional systems with an emphasis on low-temperature magneto-transport and magneto-spectroscopy studies from visible to very far-infrared (far-IR or THz). His current research focuses on intersubband transitions in low-dimensional semiconductor structures, mainly in IR and THz quantum cascade lasers, photoluminescence of 2DEG in the regime of quantum Hall effect, and magneto-transport in carbon nanotubes.

Smirnov is planning to develop a number of experimental techniques including far-IR (THz) magneto-spectroscopy at low temperatures (down to ³ He) based on tunable monochromatic sources (backward-wave oscillators) coupled to an FTIR spectrometer. *smirnov@magnet.fsu.edu*

Alexei Souslov graduated from Leningrad State University (St. Petersburg, Russia) in 1987 and received his Ph.D. at the A.F. Ioffe Physico-Technical Institute of the Russian Academy of Sciences in 1995. He worked at the Delft University of Technology (Delft, The Netherlands); at the Very Low Temperatures Research Centre of the French National Centre for Scientific Research (CRTBT-CNRS, Grenoble, France); and at the University of Wisconsin (Milwaukee, Wisconsin). He moved to the NHMFL in July, 2003.

ALEXEI SOUSLOV

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Stan Tozer studies actinides and related materials using high pressure, low temperature, and both DC and pulsed fields. He has developed many pressure cells to take advantage of the plentiful spectroscopies available at the lab. For example, he designed diamond anvil cells (DACs), rotators, and special cryostat tails, all made of plastic to minimize eddy current heating, for He-3 temperature measurements in pulsed fields. Although he has performed 4-probe electrical transport studies in the past, he has recently combined forces with C. Agosta, incorporating a tuned tank circuit into the DAC, to make contactless conductivity measurements. This technique has permitted them to study the evolution of the Fermi surface as a function of pressure. Even more recently, Tozer has teamed up with Steve Hill's group at UF to use a plastic cell for high pressure EPR studies of nanomagnets.

Tozer's work is not limited to plastic DACs. Metal large volume piston-cylinder cells have also been fabricated for dHvA, NMR (in collaboration with O. Bernal, A. Reyes, and P. Kuhns), and ultrasound studies (with A. Souslov). Metal diamond anvil cells are available in a variety of sizes (the smallest rotates in the 9 mm sample space of a Quantum Design MPMS system). They are used for Hall effect studies, electrical transport, and optical studies. He has also collaborated with G. Schmiedeshoff on a dilatometer for DC field work, and they have reduced the size to make it possible to rotate this cell at millikelvin temperatures in the 45 T hybrid magnet. *tozer@magnet.fsu.edu*

Yong-Jie Wang received his Ph.D. in physics from State University of New York at Buffalo (SUNY at Buffalo). He has been working at the NHMFL since 1993. His main responsibility at the laboratory is to manage the infrared optics program. He and his colleagues have developed an infrared set-up with many different probes that can do both reflectance and transmittance measurements in superconducting and resistive magnets.

Wang's research interests are focused on the magneto-optical study of reduced dimensional semiconductors. His three recent projects are: (1) Systematic optical study of CdMnTe/CdMgTe quantum well structures. The incorporation of Mn into CdTe makes the material magnetic, and many new magnetic infrared optical modes have been observed when the Mn concentration in the well is varied from 0 to 3%. The understanding of these modes is very attractive for the understanding of the fundamental physics as well as the application of this spintronic material. (2) Infrared optical photoconductivity study of graphene. The focus of this project is to measure the effective mass of the graphene at different k values. Graphene is a unique material in which the electron's effective mass changes from zero at k=0 to a finite value at nonzero k. (3) Study of submillimeter wave and infrared radiation-induced resistance oscillations in ultra high mobility 2D electron systems in high magnetic fields. *wang@magnet.fsu.edu*

Xing Wei received his Ph.D. in physics from University of Utah with Prof. Z.V. Vardeny studying photoexcitations in conducting polymers and fullerenes after coming to the United States as a CUSPEA student (CUSPEA refers to the China-U.S. Physics Examination and Application program). He came to the NHMFL in 1997 after completing a Director's Postdoctoral Fellowship at Los Alamos National Laboratory. He is in charge of the visible optics facility and supports users conducting magneto-optical experiments in high magnetic fields, including photoluminescence, transmission, reflectivity, and optical polarization. He has developed many probes and programs to improve automation and facilitate such experiments. In recent years, a major effort has been made to develop an ultrafast pulse optical facility to conduct time-resolved magneto-spectroscopy under high magnetic fields.

Wei's research interests are mainly in the magneto-optical study of low dimensional electronic systems, including carbon nanotubes, III-V quantum structures, and correlated electronic materials such as colossal magnetoresistance manganites, spin-ladders, quasi 1-d or 2-d charge-densitywave materials, Haldane compounds, and molecular antiferromagnets. He is also interested in developing inelastic (Raman) scattering spectroscopy and near-field optical spectroscopy at NHMFL. *xwei@magnet.fsu.edu*

Attention Users **Heat Capacity in the 20 T Dilution Refrigerator** Chuck Mielke, Head of Pulsed Field Laboratory Users Program

The recent addition of a Heat Capacity capability to the 20 T superconducting magnet at the NHMFL/LANL has spawned a remarkable discovery of Bose-Einstein Condensation (BEC) in an organic material. The collaboration between in-house scientists and international users from Switzerland and Brazil is a shining example of how scientists from around the world can come together at the NHMFL and produce exemplary science. The 20 T dilution refrigerator Heat Capacity and Magnetocaloric effect system, developed by Vivien Zapf, is currently on-line and available to users.

Bose-Einstein Condensation Induced by Magnetic Fields in the Quantum Magnet NiCl₂-4SC(NH₂)₂

V.S. Zapf, NHMFL/LANL

D. Zocco, NHMFL/LANL (now at University of California at San Diego, Physics)

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M. Jaime, NHMFL/LANL

- N. Harrison, NHMFL/LANL
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A. Lacerda, NHMFL/LANL

A. Paduan-Filho, Instituto de Fisica, Universidade de São Paulo, São Paulo, Brazil

In traditional Bose-Einstein Condensates, such as dilute gases of cold ⁴⁰K atoms, the bosons form a coherent ground state as the temperature is lowered below the critical temperature T_{BEC} . The Bose-Einstein Condensate (BEC) is formed when the lowest energy state of the system has a finite occupation of bosonic particles. A new set of materials is receiving an increasing amount of attention in which Bose-Einstein condensation can be induced by magnetic fields. Here the magnetic field, not the temperature, tunes the number of bosons from zero to nonzero across a critical field. The quantum magnet $NiCl₂ - 4SC(NH₂)₂$ (see Fig. 1) is one such material, in which the BEC can be induced by magnetic fields, and corresponds to an XY antiferromagnetic state. The $S = 1$ spin triplet of the Ni²⁺ ion is split at zero field by spin-orbit coupling into an $S_z = 0$ ground state and an $S_z = \pm 1$ excited doublet. In applied magnet fields, the $S_z = +1$ level is lowered linearly with field until it crosses the ground state. In the region where the S_z = 0 and S_z = +1 overlap, between H_{c1} = 2.1 T and H_{c2} = 12.6 T,

Figure 2. Phase diagram of NiCl_2 -4SC(NH_2)₂ showing the region of antiferromagnetism/BEC determined from specific heat (black triangles) and magnetocaloric effect (red circles).

antiferromagnetism occurs. The region of overlap is more than 10 T wide due to dispersion of the energy levels by antiferromagnetic interactions among neighboring Ni ions. The antiferromagnetic state can be mapped onto a Bose-Einstein condensate, e.g, by treating the $S_z = 0$ state as an empty state, and the $S_z = 1$ state as an occupied boson. A rigorous theoretical description can be found in Ref. [1]. The relative occupancy of the spin levels depends on the applied magnetic field, which acts as the chemical potential of the system. Due to the tetragonal symmetry of the crystal, the Ni magnetic spins do not have any preferred orientation in the plane perpendicular to the magnetic field and the antiferromagnetic order is XY-like. It is this same symmetry that creates a number conservation among the bosons. Number conservation forces the bosons to remain in the system and macroscopically occupy the ground state at low temperatures, rather than vanishing as the temperature is lowered.

We have investigated single crystals of the organic magnet NiCl_{2} -4SC(NH₂)₂ for the phenomena of BEC using specific heat and magnetocaloric effect measurements taken in a dilution refrigerator and a 20 T magnet at the NHMFL in LANL.² The single crystals were grown by A. Paduan-Filho at the Universidade de São Paulo in Brazil. The phase boundary determined by these methods is shown in Fig. 2, where the antiferromagnetism/BEC occurs in the dome-shaped region under the symbols. The key prediction of Bose-Einstein condensation that we have been able to verify is the power-law dependence of the critical field line H_c - $H_{c1} \sim T^{\alpha}$. The powerlaw exponent α is predicted to be $\alpha = 3/2$ for a 3-D BEC, $\alpha = 1$ for a 2-D BEC and $\alpha = 2$ for an ordinary Ising antiferromagnet. The difficulty in measuring this exponent, which has created problems in many previous investigations of BEC in quantum magnets, is the fact that it only occurs as T approaches absolute zero. We have used dilution refrigerator temperatures and an extrapolation technique to determine α as T approaches 0, and our results from two separate measurements are both consistent with $\alpha = 3/2$, and inconsistent with $\alpha = 1$ or $\alpha = 2$. Thus we have direct experimental verification of Bose-Einstein condensation in this compound. This is the

second-ever compound for which this exponent has been conclusively determined. The other compound, BaCuSi₂O₆,³ found α = 1, consistent with a 2-D BEC.

Our results have also benefited greatly from a collaboration with Michel Kenzelmann and colleagues, who have conducted inelastic neutron diffraction measurements on large deuterated single crystals of NiCl_2 -4SC(NH_2)₂ at the Paul-Scherrer Institute in Switzerland. The neutron scattering measurements probed the antiferromagnetic dispersion relation, and showed that the antiferromagnetic interactions in the plane J_a are ten times weaker than along the tetragonal c-axis J_c . Thus $NiCl_2$ -4SC(NH₂)₂ can be thought of as a weakly coupled 1-D chain system at high temperatures, crossing over to 3-D antiferromagnetic order below ~1 K.

Figure 3. The 20 T superconducting magnet system at the NHMFL-Los Alamos.

- ¹ H.-T. Wang and Y. Wang, *Phys. Rev. B*, **71**, 104429 (2005); and K.-K. Ng and T.-K. Lee, cond-mat/0507663.
- ² V.S. Zapf, *et al*., *Phys. Rev. Lett*., **96**, 077204 (2006).
- 3 S. Sebastian, *et al*., *Nature* (2006), in press.

The Development of High Resolution, High Field 17O Solid State NMR Spectroscopy for Biological and Inorganic Systems

¹⁷O NMR has long been viewed as an important spectroscopy to develop. Its large quadrupole coupling constant (carbonyl oxygens: $C_o \sim 8$ MHz) associated with its spin 5/2 nucleus has generated numerous complications; yet, oxygen atoms are a rich source of electrons and a great deal of important chemistry takes place at these sites in both biological and inorganic catalysts. Biological macromolecular oxygens play key structural roles not only through covalent interactions but, in non-covalent interactions especially hydrogen bonding and electrostatic interactions. Because of this central structural role, the oxygens also play a unique role in numerous functional activities from charge relay mechanisms to electrophilic reactions, from facilitating hydration to forming the key non-covalent interactions associated with ligand binding. In materials, many similar comments can be made about the importance and utility of the oxygen atoms including their role in cooperative phenomena such as phase transitions.

Many researchers have been involved in laying the foundation for present day 17O spectroscopy. The spectroscopy has been particularly challenging because of the second order broadening due to the large quadrupolar interaction and the need for isotopic labeling (natural abundance is 0.037%) to go after complex systems. The sensitivity of the 17O quadrupolar interaction, chemical shift anisotropy and isotropic chemical shift to the electronic environment, however, is spectacular and could potentially be used to mine a wealth of information about the molecular interactions within materials and biological macromolecules. High resolution spectra has been aided by very fast magic angle spinning and very high magnetic fields, both of which are available at the NHMFL for 17O studies. Listed below are a sampling of some of the 17O studies conducted at the NHMFL in recent years capped by the recent efforts at 830 MHz and 900 MHz (external users/collaborators in boldface).

- High resolution 17O NMR spectra of siliceous ferrierite using multiple quantum MAS and Double Rotation (DOR, 18.7 T) spectroscopy. {**Bull, Bussemer, Anupold, Reinhold, Samosan, Sauer, Cheetham and Dupree** (2000) *J. Am. Chem. Soc.* 122: 4948-4958.}
- MAS and Satellite Transition MAS (19.6T) ¹⁷O spectroscopy of three crystalline polymorphs of P_2O_5 combined with *ab initio* calculations of the 17O electric field gradient tensor, quadrupolar

Figure 1.¹⁷O Double Rotation (DOR) and Multiple Quantum Magic-Angle Spinning (MQMAS) spectra of siliceous ferrierite at various magnetic fields (Bull *et al*. (2000) *J. Am. Chem. Soc.* 122: 494- 4958). For quadrupolar nuclei, the peak position is a sum of isotropic chemical and quadrupolar shifts. Spectra at variable fields and variable experiments (DOR and MQMAS) are required to trace the peaks and for the complete determination of chemical and quadrupolar shift contributions. The isotropic chemical shifts can be read from the zero crossing.

University of Melbourne, Z.H. Gan/NHMFL). The 3QMAS experiment at 19.6 T resolves the two peaks indicative of Al-O-Si and Si-O-Si linkages, and the shoulder (lower left) for the Al-O-Si peak from a mass balance perspective. The K-specimen on the right exhibits a greater degree of disorder, with a larger Si-O-Si peak observed. A third peak is clearly seen at approximately 80 ppm in the MAS axis, likely correlating to octahedral Al-O-Al linkages.

Figure 3. ¹⁷O spectra (single pulse with 8 ms recycle delay) of an aligned sample of the ion channel gramicidin A in hydrated lipid bilayers (DMPC) with and without K^+ as a function of temperature. At 40°C the synthetic membrane is above the gel to liquid crystalline phase transition and there is a 40 ppm shift in the anisotropic chemical shift frequency showing exquisite sensitivity to the presence of K^+ in the vicinity of the Leucine-10 carbonyl oxygen. Remarkably, at 9°C (below the phase transition) there is no change in chemical shift upon addition of K^+ to the sample. In the gel phase, the hydrophobic dimension of the bilayer swells potentially ejecting the ion from its customary binding site in the pore of the channel [E. Chekmenev, L. Miller, P. Gor'kov, W.W. Brey & T.A. Cross/FSU & NHMFL].

coupling constant and isotropic chemical shift. {**Cherry, Alam, Click, Brow** and Gan (2003) *J. Phys. Chem. B.* 107: 4894- 4903}

- Single Crystal magic angle spinning ¹⁷O NMR spectroscopy of the Antiferroelectric Phase Transition in Squaric Acid. {Dalal, Pierce, Palomar and Fu (2003) *J. Phys. Chem. A* 107:3471- 3475}
- Determination of 17O tensors using MAS and static spectroscopy up to 19.6 T of potassium hydrogen dibenzoate containing a short hydrogen bond {**Wu and Yamada** (2003) *Solid State Nucl. Magn. Reson.* 24:196-208.
- Single Site 17O labeled gramicidin A observed in aligned samples at 21.1 T with and without K^+ bound. {Hu, Chekmenev, Gan, Gor'kov, Saha, Brey and Cross (2005) *J. Am. Chem. Soc.* 127:11922-11923}
- *Ab initio* calculational and spectroscopic ¹⁷O study of ion binding to four crystalline polymorphs of Gly-Gly-Gly. {Chekmenev, **Waddell,** Hu, Gan, **Wittebort,** and Cross (submitted 3/06)}
- Illustration of flow through Anodic Aluminum Oxide porous substrate trapped lipid nanotubes containing ¹⁷O labeled gramicidin ion channels. {Chekmenev, Gor'kov, Cross, **Aaouie and Smirnov** (submitted 3/06)}
- 17O NMR of carbohydrates illustrates sensitivity enhancement by B_o⁴. {**Sefzik, Houseknecht, Clark, Prasad, Lowary**, Gan and **Grandinetti** (submitted 3/06)}.

The NHMFL offers a wide range of facilities for obtaining 17O spectroscopy. At 16.8 T (¹H frequency of 720 MHz) we offer DOR capability (Fig. 1). At 19.6 T (1 H frequency of 830 MHz) we have a variety of MAS probes with spinning rates from 10 kHz (4 mm) up to 40 kHz (2 mm), but due to the very narrow bore of this instrument there is not variable temperature (Fig. 2). A static probe offering a variety of coils and a temperature range from -80°C to $+120^{\circ}$ C is also available (Fig. 3). At 21.1 T (¹H frequency of 900 MHz) a static HX probe is available with a variety of coils and modest range of temperature from 0°C to 60°C (Several 17O spectra were shown from the 900 in the previous issue of *NHMFL Reports*). Both ¹H decoupled and cross polarized ¹⁷O spectra have been obtained with this probe. A 3.2 mm MAS HX probe with a large VT range is currently under development at the NHMFL.

Jack Toth inspects disks from the latest generation of Florida-Bitter magnets.

Mark Bird, NHMFL

On December 12, 2005, the NHMFL completed testing of a new 32 mm bore Florida-Bitter magnet. The new magnet reached 35.1 T using 19.1 MW of power. The field was measured with a hall probe previously calibrated against NMR up to 30 T. Fig. 1 shows a CAD model of a section of the new magnet and the picture above shows Jack Toth inspecting disks from the latest generation of high-field solenoids.

This magnet constitutes a 2 T upgrade at the NHMFL in this bore and is the highest field resistive magnet available to users worldwide. It surpasses the 33 T, 17.5 MW resistive magnets in cells 9 and 12 at the NHMFL and the copies in Nijmegen, The Netherlands. The magnet is conceptually very similar to the successful and reliable 33 T magnets. Both consist of four nested coils made from the same materials and operating at the same temperature and stress limits.

The primary innovations in this magnet compared with the older 33 T magnet are the introduction of current-density grading to the coils as well as a change in the outer diameter of the inner coils. By using lower current density at the ends of the coils than at the mid-plane, greater efficiency can be attained. The earlier 33 T magnet was based heavily on the preceding 30 T magnet, maintaining maximum interchangeability with it. For the 35 T system, a new global re-optimization was performed resulting in a larger inner set of CuAg coils than the previous version. By dedicating a larger volume of the magnet to higherperformance CuAg material, the overall system "efficiency" was improved despite the fact that the new magnet operates at higher power and field than its predecessor.

DC resistive magnet "efficiency" is defined $E = B(a_1/P)^{1/2}$ where *B* is the flux density, a_1 is the inner radius, and *P* is the power, as shown in Table $I¹$ We see that the new 35 T magnet is more "efficient" than the older 33 T magnet once we adjust for the differences in power consumption.

Figure 1. Section of new 35 T Florida-Bitter magnet at the NHMFL.

Figure 2. Project team including (l to r back row), Scott Bole, Robert Stanton, Mark Bird, Scott Gundlach, (l to r front row) Clint Cates, Jim O'Reilly, Jack Toth pose with new 35 T resistive magnet.

Table I. Parameters of the 35 T and 33 T, 32 mm bore magnets at the NHMFL.

In May 2006, we expect to complete another new magnet: a 32 mm bore, 28 T magnet with field inhomogeneity of ~50 ppm over a 10 mm diameter-spherical volume. This new magnet will use the same Florida-Bitter disk design as the 35 T magnet and will be installed in cell 7 to replace the 23 T magnet that has been operating there since 1995.

¹ J.C. Picoche, *et al*., *J. of Magnetism & Magnetic Materials*, **11**, 308- 316 (1979).

POWERED MAGNET HISTORY @ NHMFL

Pat Dixon, Director of the NHMFL Center for Integrating Research and Learning (CIRL)

The Center for Integrating Research and Learning continues its commitment to enhance classroom science by serving as a resource for teachers who cannot otherwise attend workshops and other professional development opportunities. A plea from teacher Karen Krejci at the Marshall School in Marshall, Alaska, resulted in the Center trying to get materials to her students before the spring thaw! The Magnet Lab provided students with a package of magnets, magnetic field viewers, and a curriculum developed at the Center, *Science, Magnets & You*. In addition, the

high school students at this small bush school requested materials with which to study optics and again the Center was able to help. We are proud to support 121 Marshall School K12 students. One-hundred percent of the students are Native American/Alaskan Natives, and we are looking forward to receiving more photos and hearing about their experiences.

Locally, the Center supports a number of schools with special projects and this semester helped a local fifth grade teacher and former Research Experiences for Teachers participant, Amanda Witters, develop "science totes" for students to take home and share with their parents. Students are competitive about who gets to take them home. Fifth graders share with their parents and siblings science activities and experiments, fiction and non-fiction books about science and scientists, and then record their experiences in a journal that stays with the bag for all to see.

Representing the Center at the RET Networking Session at this spring's National Science Teachers Association national conference is another RET "graduate," Josh Underwood. He will be making a presentation on his experience at the Magnet Lab for teachers from all over the country representing a diverse group of RET programs. We are proud to be able to continue to support RET participants. The fact that Underwood participated in 2004 demonstrates the lasting

relationship that the Center builds with teachers who come to the Lab each summer.

"100 Years Later – Is Einstein Still Relevant?" national conference was hosted by Partners in Science and sponsored by the M.J. Murdock Charitable Trust. Three former NHMFL RET participants were invited to attend: Logan Chalfant, Lincoln High School, Tallahassee, Florida; Richard McHenry, Leon High School, Tallahassee, Florida; and Soon Young Kim, Moanalua High School, Honolulu, Hawaii. Assistant Director of CIRL Jose Sanchez accompanied the educators and spent time comparing RET program features with other program managers. Distinguished speakers Dr. Craig J. Hogan and Dr. Michael G. Raymer gave lectures on Quantum Mechanics and Cosmology. Workshops were provided at General Atomics Lab of San Diego and the University of San Diego on topics ranging from "Using Restriction Enzymes to Identify and Map an Unknown Plasmid" to "Using Neurobiology to Study Genetics & Addiction.

As we prepare for the 2006 Research Experiences for Undergraduates program, it is significant to note that we see quite a few students in the lab who were former REU participants. After the program, they chose to return to Florida State University to pursue graduate degrees in the sciences. At the present time, five former REU participants are at FSU conducting or just finishing graduate studies: Manuel Ramos, Nicole Tibbetts, Rick Clinite, Jason Crowe, and Nathaniel Falconer. Three of their stories follow.

Manuel Ramos, shown below, shares his experience in his own words.

I came to the REU program in summer 2003, which I learned about at my school, the University of Texas at El Paso (UTEP). I worked during that summer with Justin Schwartz, under Sastry Pamidi as a supervisor on "Calorimetric Measurements of AC losses on Bi2223/Ag Tapes." The data that I obtained, as well as the type of research, gave me the opportunity to write a thesis and graduate with honors under the Departmental Honors program in the Physics Department at UTEP. The thesis was published at the school and I also participated and gave an oral presentation at the APS meeting, Texas Section, at Bronxville, Texas in October of 2003.

In May of 2004, I came back to Tallahassee for my graduate studies at the Mechanical Engineering Department of the FAMU-FSU College of Engineering. My graduate advisor is Justin Schwartz. We are working on texture enhancement of Bi-2212/ Ag superconducting wire by high magnetic field background during its partial melt process. This research includes a study of the critical current of the conductor, as well as microanalysis of the structure using SEM and OIM techniques. The thesis results are going to be presented at the Material Science Society Spring meeting at San Francisco in April 2006, and a journal article will be prepared as well. During my two years in the Mechanical Engineering program, I participated in the Applied Superconductivity Conference in October 2004 and presented a poster, "The Angular Dependence of Critical Current of Bi2212/ Ag Superconducting Wires." I also received valuable guidance from Steven VanSciver, Peter Kalu, and Anter El-Azab.

Jason Crowe, currently a third year graduate student in molecular biophysics, works with Teng Ma, chemical/biomedical engineering, and Tim Logan, chemistry. He participated in the 2003 REU program as a student from Clarkson University and then chose to return to FSU for his graduate studies. He describes his research below.

The aim of my research project is to characterize the energy of metabolism in human Mesenchymal Stem Cells (hMSC) grown in a perfusion bioreactor as they differentiate into cardiomyocytes. Metabolism will be explored via NMR spectroscopy and biochemical assays. hMSCs are a pluripotent population of adult stem cells residing in the bone marrow that have the ability to differentiate into cardiomyocytes. hMSCs may have therapeutic potential in combating heart ailments. When delivered to the site of a recent infarct, cardiomyocytes have been shown to integrate and expand within the heart tissue. New cardiomyocyte development within the infarct could strengthen the left ventricle after a heart attack, and could possibly replace scar tissue formation that normally follows heart trauma.

The bioenergetics behind the differentiation of hMSCs into cardiomyocytes is largely unknown. The energy metabolism will be monitored to characterize the metabolic state of the hMSCs, and to characterize how the metabolic state changes as the cells differentiate into cardiomyocytes. 31P NMR spectroscopy allows us to measure the amounts of intracellular ATP, phosphocreatine, and inorganic phosphate, as well as calculate the ADP levels within the cell. This will provide the data necessary to establish a metabolic profile of the creatine kinase pathway. NMR spectroscopy will provide a non-invasive method to determine the metabolic activity of the cells. It will also allow for repeated measurements of metabolite levels throughout the duration of the hMSC growth and differentiation into cardiomyocytes.

The ultimate goal of my research project is to establish a metabolic profile for hMSCs as they differentiate into cardiomyocytes. Once NMR data is collected, the metabolic activity of the creatine kinase reaction can be analyzed. It would then be possible to use the metabolic activity as a marker for where on the differentiation pathway the cells are located.

Nicole Tibbetts, an REU participant in 2000, returned to FSU and the Magnet Lab to pursue her doctoral studies. She not only conducts research and teaches classes, but is an enthusiastic partner with educational programs, working with young people during the summer and assisting teachers during the school year. Here is how she describes her current activities.

I am currently working with Michael Bizimis under the supervision of Leroy Odom, department chair, Geological Sciences, investigating the oxygen fugacity of the ancient earth's mantle through the use of laser ablation inductively coupled plasma mass spectrometry. I also conduct sea turtle research, surveying geomagnetics of deep mineral layers on the shorelines of the coast of Florida and Central America. This work is being conducted with Odom's former Ph.D. student who conducted his research at the Magnet Lab, Jack W. Rink, currently of McMaster University. Rink was recently inducted into the prestigious Explorer's Club in New York City and graced the cover of Nature *in 2005. I am fortunate to work with great people in many disciplines who are now or have been associated with the Magnet Lab.*

Publications include two in review and one published in 2005: "Chemical and Isotopic Analyses of Apollo 16 Glasses: An Integrated Approach," LPSC, 2005, "Chemical Compositions and 40Ar/39Ar Ages of Apollo 16 Impact glasses: Low-Mg High-K Fra Mauro (lmHKFM), submitted to Meteoritics and Planetary Science*, and "Coastal Geomagnetic Anomalies as Navigational Markers for Nesting Loggerhead and Green Sea Turtles," submitted to* Nature*.*

Thousands Drawn to Magnet Lab's Open House

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disguise Talk about pulling them in! More than 3,300 people of all ages spent part of a glorious February day at The National High Magnetic Field Laboratory for a dose of science education disguised as fun.

> It was all a part of the lab's annual Open House, held Feb. 18, 2006, from 10 a.m. to 3 p.m. People arrived before the doors opened and many stayed long after they were closed. The popular community event, in its 12th year, offered activities for all ages, including hands-on science, self-guided tours of the laboratory and interactive demonstrations. The goal? To get the community – and especially children –interested in and excited about science.

And excited they were. Lines formed with children waiting for their chance to shoot a potato out of a cannon, play with a maglev train, build a comet, among many other things. All the while, they learned subtle lessons about biology, chemistry, and physics that often are the spark that ignites a child's interest in science.

Each year at Open House, visitors get a close-up look – and a layperson's understanding – of work underway at the Magnet Lab. Scientists and engineers explain their activities in simple terms or demonstrate a basic science or engineering concept.

The Magnet Lab is a world-class facility serving scientists from across the United States and around the world. While the Magnet Lab boasts a national and international focus, it is very involved with local, regional, and state education efforts, including school group tours, outreach programs, and curriculum products in use throughout the state.

AMRIS Update

Ultra High Sensitivity NMR: 1-mm HTS Triple Resonance Probe

W.W. Brey, NHMFL A.S. Edison, NHMFL/UF, Biochemistry and McKnight Brain Institute R.E. Nast, Varian, Inc. J.R. Rocca, UF, McKnight Brain Institute S. Saha, NHMFL R.S. Withers, Varian, Inc.

It seems that every few months the Magnet Lab sets another world record for laboratory magnets. This is always wonderful news, even for those of us who don't use these magnets. It is exciting to imagine the huge fields and stored energy of these systems. At the other end of the scale, however, a four-year collaborative effort between colleagues in AMRIS, the Magnet Lab, and Bruker has yielded a very small world record: a 600 MHz triple resonance 1-mm high temperature superconducting NMR probe that delivers what appears to be the highest mass sensitivity of any probe at any frequency.

The project was funded by an NIH P41 resource grant to UF, but its origins were established over a decade ago in a company called Conductus. Three of us (Bill Brey, Rob Nast and Rich Withers) and others at Conductus developed the first HTS (high temperature superconducting) NMR probes in the early 1990s. In the mid-1990s, Bruker purchased the HTS NMR probe part of Conductus; Withers and Nast continued at Bruker, and Brey moved to the Magnet Lab. Since this project was completed, Withers and Nast have moved from Bruker to Varian, Inc. The overall goal of the NIH resource grant is to improve the sensitivity of NMR and MRI through novel probes and coils, and we therefore formed a new collaboration to develop the world's most sensitive NMR probe. Much of the design and simulation for the probe was done at the Magnet Lab, and the testing and demonstration of capabilities is being done in AMRIS. The project could not have been completed without an industrial partner because it builds upon the commercial Bruker cryoprobe and cryoplatform, and the final product is in a standard cryoprobe body. That is where the similarities to commercial probes end.

In general, the smaller the NMR sample coil the higher the sensitivity; this is the basis of great success in microsolenoid coils (Olson *et al*., 1995; Li *et al*., 2003). It is also possible to cool NMR coils and preamplifiers to reduce noise in measurements; this is the basis of cryogenically cooled probes, most of which are constructed from standard copper wire (Kovacs *et al.*, 2005). Finally, although they are difficult or impossible to fabricate like standard copper wire, HTS materials can provide extremely high sensitivity for NMR measurements (Brey *et al.*, 1996). We combined small sample volume with cryogenic cooling and hightemperature superconducting (HTS) technology to build a 600 MHz 1-mm triple-resonance probe with a z-axis gradient. The probe has an active volume of 6.3 µL and minimum total sample volume of 7.5 µL. The sample is loaded vertically using small

capillary tubes. Four nested pairs of resonant HTS coils (Fig. 1) produce frequencies for ${}^{1}H$, ${}^{2}H$, ${}^{13}C$, and ${}^{15}N$.

All the planar coils are cooled to about 20 K, and the H , 2H , and 13C preamplifiers are cooled to 77 K using a commercial Bruker Cryoplatform. The 1-mm sample chamber is vacuuminsulated from the HTS coils and is warmed by a stream of air for temperature regulation. The 1 H coil pair (Fig. 2) has a large height-to-width ratio to produce a very homogeneous RF field. To give the best sensitivity, it is placed very close to the sample. Full 3D electromagnetic simulations conducted before the probe was built predicted that the ratio of NMR signal amplitudes following 810° and 90° pulses, a standard measure of B1 homogeneity, would be about 90%. The experimental value is about 87%, in

Figure 1. Design of the 1-mm HTS triple-resonance probe. Coils were constructed by depositing a thin coating of $Y_1Ba_2Cu_3O_{7.8}$ (YBCO) on a sapphire supporting surface.

great agreement with the simulations. The 15N coils (Fig. 3) are constructed as spiral resonators to achieve a resonance frequency of 60 MHz. The simple spiral design supports additional unwanted modes at approximate multiples of 60 MHz. Final frequencytrimming relied upon extensive simulations to prevent these modes from interfering with the other coils. The development of accurate simulations for HTS coils was one of the major accomplishments from this research project and will help with new HTS projects in the future.

Figure 2. Design and simulation of ¹H coils. The ¹H coils have a large length to width ratio to optimize B_1 homogeneity and active sample volume. Electromagnetic simulations were carried out using IE3D (Zeland Software, Fremont, CA) and predicted excellent B_1 homogeneity of 90% for an 810°/90° pulse ratio. This number agrees very well with the experimental value of 86.8%.

The probe was completed the end of summer 2005 and installed in the AMRIS 600 MHz spectrometer in Fall 2005. We have made initial measurements to test the overall performance. One of the most important specifications is the signal-to-noise ratio (S/N). The industry standard for this spec is the height of a certain peak in the spectrum of a solution of 0.1% ethylbenzene in deuterated chloroform following a single 90° pulse. This rather innocentsounding measurement has many complicated nuances that have been optimized to sell spectrometers, and all users should be aware of some of the potential problems: e.g., the definition of the noise varies rather significantly from vendor to vendor; different "standard" tubes with different wall thicknesses will produce different results. When working with very small sample volumes, we have found additional problems: two different "standard" commercially sealed tubes of 0.1% ethylbenzene differed in our measurements by about 20%. Presumably this is due, in part, to the volatility of the chloroform, a problem that would be amplified with very small volumes. Second, in general it is very difficult with small volume samples to keep them dry, especially in Florida! Therefore, we have found that sample preparation is a very important factor that needs to be carefully planned for optimal performance of the 1-mm HTS probe. Given all of these problems and caveats, we obtained S/N values for 0.1% ethylbenzene in a range from 262 to 364 with an average value of 292±28. This is approximately 3.5-fold less than a standard 600 MHz 5-mm triple-resonance probe $(S/N \sim 1000$ with all the caveats) with about 70-fold less sample. Thus, the mass sensitivity of the 1-mm HTS probe is about 20 times greater than a conventional 5-mm probe. Commercial 5-mm Bruker 600 and 800 MHz cryoprobes have S/N

Figure 3. Current distribution in the square spiral ¹⁵N coil. Tuning HTS coils to low frequencies (e.g., 60 MHz for 15N at 14.1 T) is challenging and required a new rectangular coil design. The first resonant ¹⁵N mode is shown, and the red and blue/green colors represent regions with high and low currents, respectively. Extensive computer simulations were required to achieve the correct 60 MHz frequency while simultaneously eliminating parasitic interfering higher order modes, especially 600 MHz.

Figure 4. Experimental data from the 1-mm HTS triple-resonance probe. Data were collected using 22.6 µg ibuprofen dissolved in 10 µL DMSO-D6 for an ibuprofen concentration of 11.1 mM. (a) 1D spectrum collected with 8 scans, (b) 2D COSY recorded in 3 h 49 m using 4 scans and 1024 t_1 increments, (c) ¹³C-HMQC recorded with natural abundance 13C in 16 h using 28 scans and 512 t_1 increments, and (d) ¹³C-HMBC recorded in 13 h 17 m using 32 scans and 464 t_1 increments. We have also collected very good 1D, COSY and HMQC datasets on just 1.7 µg ibuprofen (1.1 mM) (data not shown).

values approximately 4,000 and 8,000, respectively (don't forget the caveats!). The 1-mm HTS probe has a mass sensitivity that is over 4 times greater than a 5-mm cryogenic probe at the same field strength and over 2 times greater than state-of-the-art 5-mm technology at 800 MHz.

How good is the HTS when compared with microsolenoid probes? Unfortunately, these probes are not typically tested with 0.1% ethylbenzene standards, perhaps because the organic solvent could dissolve the seals on the horizontal sample tubes or, for flow systems, damage the flow tubing. The "standard" measurement for these probes tends to be sucrose, but the concentrations used vary quite extensively. We measured the S/N anomeric ¹H from two different 10.0 mM preparations of sucrose in D_2O using 1, 4, and 8 scans in the HTS probe. Each condition was repeated three times for a total of 18 measurements. These yielded a S/N per µmol of 2338 \pm 134 for the active volume (6.3 µl) and 1964 \pm 112 for the total volume of sample used $(7.5 \mu l)$. Comparable measurements in a commercial 1-mm solenoid probe were 2130 for the active volume (1.5 μ l) and 639 for the total volume of sample (5 μ l) (Olson et al., 2004). Thus, the 1-mm HTS probe has better absolute mass sensitivity and much better total volume sensitivity than a microsolenoid probe at the same field strength.

Figure 5. Experimental data from the 1-mm HTS triple-resonance probe. Data were collected using 400 µM ubiquitin using gradient 15N-HSQC and were recorded in 41 m using 16 scans and 128 t_1 increments. High quality data on $8 \mu L$ 1 mM samples can be obtained in about 10 min (not shown).

The 1-mm HTS probe appears ideal to measure very small masses of sample. If there is ample sample, then larger volume probes will provide greater absolute S/N. However, there are important applications where sample is limited or it would be useful to conserve sample: natural products, metabolomics, and protein screening. We have collected initial experimental results for ibuprofen at natural $13C$ isotopic abundance (Fig. 4) and $15N$ -labeled ubiquitin (Fig. 5). The ibuprofen spectra (Fig. 4 a-d) were collected with 22.6 µg in 10 µl DMSO-D6 (11 mM). We also successfully collected data on just 1.7 µg in 7.5 µl DMSO-D6 (1.1 mM) (not shown). We have also acquired protein NMR 15N-HSQC spectra (Fig. 5) in just over 40 min on 400 μ M ¹⁵N-labeled ubiquitin in 8 μ l phosphate buffer (pH 5.5) with $10\% \text{ D}_2\text{O}$ for lock (Fig. 5).

In conclusion, we have designed, built, and tested a novel NMR probe that appears to have the highest mass sensitivity at any field strength. Although we are still learning many things about optimal use of this probe, it is now part of the Magnet Lab user program in the AMRIS facility, and scientists with mass-limited samples are encouraged to contact Art Edison, the AMRIS Director, *(art@mbi. ufl.edu)* to use this exciting probe for their studies.

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- ⁴ D.L. Olson, *et al*., *Science*, **270**, 1967-1970 (1995).
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- * This work was published in the *Journal of Magnetic Resonance*, **179**, 290-293 (2006).

The Stardust spacecraft that left Florida seven years ago recently landed in Utah, bringing with it tiny particles of comet dust that are expected to unlock big secrets about the origin of our solar system. This summer scientists in the Geochemistry Program at the Magnet Lab will study some of those particles as they seek to discover our cosmic ancestry.

With grants from NASA and the National Science Foundation and matching funds from Florida State University, the lab will acquire a mass-spectrometry-based "microanalysis" system for studying this and other extraterrestrial material. The state-of-theart instrument will be housed in the lab's forthcoming Plasma Analytical Facility.

A supplemental grant also will fund educational outreach to local schools and the development of additional science-education materials for teachers through the Research Experiences for Teachers program run by the lab's Center for Integrating Research and Learning (CIRL).

Munir Humayun, an associate professor of geochemistry at FSU and one of the principal investigators on the grant, said the Magnet Lab's new spectrometers will be able to glean 40 times more information from the sample than traditional microanalysis techniques allow.

"The genealogy of the solar system is recorded in comets," said Humayun, a cosmochemistry expert. "These grains of authentic cometary material, together with the new techniques for studying them, will help us develop a deeper understanding of the formation of asteroids and comets. Understanding their origins will help us better understand our own."

Mass spectrometry—a technique for measuring the mass of atoms or molecules—is a key strength of the scientists at the lab. The technique converts molecules to ions that then are separated, using magnetic fields, according to the ratio of their mass to electric charge. Scientists will "shoot" the cosmic dust with a laser,

vaporizing the grains and turning them into an aerosol. That aerosol then will be simultaneously directed to two different mass spectrometers for analyses; one spectrometer will scan the sample for major elements, while the other measures a selection of the trace constituents. This will tell the scientists what elements are in the grains; from that, insights into the processes that formed the comets will be learned.

A covered patio area at the Magnet Lab will be soon converted into the Plasma Analytical Facility.

Educational outreach is a special component of the grant. **Mabry Gaboardi, an FSU graduate student in geochemistry, will work with local teachers** to bring "Comet Tales" —a handson educational program based on the Stardust mission—into the classroom.

"If you want to teach science, you have to capture the imagination of the student," Gaboardi said. "That is why we are so excited to share this NASA mission with local students. The grains Stardust is bringing back will allow us to peer through time into the very birth of the solar system. That should fascinate anyone!"

"Comet Tales" is a two- to three-week science inquiry unit. Fifteen Tallahassee-area classrooms will be selected to participate, based on teacher interest and application. Five each of fifth-, sixthand ninth-grade teachers will receive supplies and assistance to complete the NASA-approved unit "Technology for Studying Comets" with their students.

In this educational unit, students work cooperatively, exploring technology and creating collection tools like the ones used on the Stardust mission. Gaboardi said the focus is on technology, because without recent innovations such as aerogel (see *http:// stardust1.jpl.nasa.gov/tech/aerogel.html)*, scientists would not have the chance to make leaps of learning in space science. During the unit, Gaboardi will visit each classroom to introduce comet properties, answer questions, and assist the students in "cooking up a comet."

After the unit ends, each teacher will choose one student to represent his or her classroom at the Magnet Lab for a day as "Stellar Students." These students will tour the lab, where they will observe research activities in the Cosmochemistry Lab and meet the researchers.

Gaboardi and Humayun also will work with four teachers in the lab's Research Experience for Teachers program this summer, with the teachers ultimately translating their research experience into teaching and learning opportunities for their classrooms.

Munir Humayun explained the science associated with the Stardust Mission at the Magnet Lab's recent Open House (see page 21).

The NHMFL directs a comprehensive set of safety programs directly related to the specific needs and operations of the laboratory. Great care, expertise, and coordination were used in developing these programs in an effort to help ensure the safest possible conditions for NHMFL faculty, staff, users, and visitors.

Research and education in science and engineering may involve a variety of hazards in laboratories and shops. The protection of the health and safety of everyone is a primary concern. At all times, in all laboratories, the use of "Good Laboratory Practices" is required. Individuals are responsible for ensuring that not only themselves, but all others around them, are working in a safe manner.

Listed below are **some** of the "Good Laboratory Practices" that should be followed in all labs at the NHMFL.

AVOIDANCE OF "ROUTINE" EXPOSURE:

Develop and encourage safe work practices. Avoid unnecessary exposure to chemicals by any route; encourage proper personal hygiene (i.e., wash hands prior to leaving laboratory area). Do not smell or taste chemicals. Vent apparatus that may discharge toxic chemicals (vacuum pumps, distillation columns, etc.) into local exhaust devices. Inspect gloves and test glove boxes before use.

CHOICE OF CHEMICALS:

Strive to substitute less hazardous chemicals whenever practical. Limit inventory on hand to chemicals and quantities necessary for laboratory activities. Inspect chemical inventories periodically and dispose of outdated chemicals in accordance with chemical disposal controls.

EATING, SMOKING, ETC:

Do not eat, drink, use tobacco products (smoke, chew, dip), chew gum or apply cosmetics in areas where laboratory chemicals are present; wash hands before conducting these activities.

Do not store food or beverages in refrigerators or glassware that have been used for laboratory operations.

EQUIPMENT AND GLASSWARE:

Handle and store laboratory glassware with care to avoid damage; do not use damaged glassware. Use extra care with Dewar flasks and other evacuated glass apparatus; shield or wrap them to contain chemicals and fragments should implosion occur. Use

equipment only for its designed purpose. Dispose of damaged/unwanted glassware in accordance with associated hazards; sharps and chemical.

EXITING:

Wash areas of exposed skin well before leaving the laboratory.

HORSEPLAY:

Avoid practical jokes or other behavior, which might confuse, startle, or distract another worker.

High Magnetic Fields:

Be aware that strong magnetic fields exist around magnets. Steel, iron or other magnetic objects should be fastened down or kept behind the 100-G line. Be very sure that screwdrivers, wrenches and other hand tools are not left around the magnets.

PERSONAL APPAREL:

Confine long hair and loose clothing. Wear close-toed shoes and long pants at all times in the laboratory. Do not wear sandals, perforated shoes, or cloth sneakers.

PERSONAL HOUSEKEEPING:

Keep the work area clean and uncluttered, with chemicals and equipment being properly labeled and stored; clean up the work area on completion of an operation or at the end of each day.

If it is safe to do so, clean up all small spills immediately.

For large spills and for proper disposal of chemical waste, contact the Safety department at 644-6955 or 644-0233.

PERSONAL PROTECTION:

Assure that appropriate eye protection is worn by all persons, including visitors, where chemicals are stored or handled. Wear appropriate gloves when the potential for contact with toxic materials exists; inspect the gloves before each use, wash them before removal, and replace them periodically.

Use any other protective and emergency apparel and equipment as appropriate. Avoid use of contact lenses in the laboratory unless necessary. If contact lenses are used, inform supervisor so special precautions may be taken. Remove laboratory coats immediately upon significant contamination and decontaminate or dispose of properly.

WORKING ALONE:

Avoid working alone in a building. Prior approval from the principal investigator is required before working alone in a laboratory. Working alone in a laboratory is prohibited when working with a compound of high or unknown toxicity.

This article was contributed by Angela Sutton, NHMFL Environmental Health & Safety manager. She may be contacted at 850-644- 6955 or sutton@magnet.fsu.edu.

PEOPLE IN THE NEWS

Vivien Zapf has recently been converted to a permanent staff member at the NHMFL/LANL. She completed her Ph.D. at the University of California, San Diego with Brian Maple and had postdoctoral fellowships at the California Institute of Technology and LANL. Zapf's expertise lies in low-temperature (dilution refrigerator and up) measurements of thermodynamic properties. She studies the interaction among magnetic behavior, superconductivity, and non-Fermi liquid behavior in felectron compounds. She has also been involved in the study of quantum critical behavior and Bose-Einstein condensation in quantum magnets.

The Office of the Dean of the Faculties at FSU has announced this year's non-ranked faculty promotions and continuing contracts, effective for the fall semester.

For promotion to associate professor and for tenure

Jianming Cao, assistant professor of physics in the Condensed Matter Science group, received his Ph.D. from the University of Rochester. His research interests include ultrafast electronic and structural dynamics in metal nanoparticles and CMR materials. In 2000, he was honored with the Research Corporation Research Innovation Award. His research group has developed a new technique of femtosecond electron diffraction that directly monitors atomic motions on the timescale of a single atomic vibrational period.

For promotion to full professor

Nick Bonesteel, associate professor of physics in the Condensed Matter Science group, received his Ph.D. from Cornell University and his B.S. in mathematics from the Rensselaer Polytechnic Institute. His research interests include condensed matter physics, correlated electrons, quantum Hall effect, and quantum computing. He was honored with an Alfred P. Sloan Foundation Fellowship in 1997. He received the FSU Developing Scholar Award in 2002.

Jianming Cao

Bonesteel

Vladimir Dobrosavljevic

Vladimir Dobrosavljevic,

associate professor of physics in the Condensed Matter Science group, received his M.Sc. and Ph.D. in physics from Brown University and his B.Sc. in physics from the University of Belgrade, Yugoslavia. His research interests include condensed matter theory, metalinsulator transitions, the many-body problem, statistical mechanics and glassy freezing and glassy dynamics. In 1997, he was honored with an Alfred P. Sloan Foundation Fellowship. In 2003, he was honored with the FSU Developing Scholar Award.

For promotion to tenure

Vincent Salters, scholar/scientist and director of the NHMFL Geochemistry group, received his M.Sc. in geochemistry from the State University of Utrecht, The Netherlands and his Ph.D. from the Massachusetts Institute of Technology. His research interests include igneous petrology and trace element and isotope geochemistry in the broadest senses. Most of his research is concentrated on quantifying processes that ultimately bear on the differentiation of the planet. He also researches low temperature processes, especially in the area of speciation of environmentally significant substances and the relationship between speciation and bioavailability.

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