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Instrumentation

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Cryogenics

Previewing the 2001 NHMFL Annual Research Review



#### **REPORTS • WINTER 2002**

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## **From the Chief Scientist's Desk: Previewing the 2001 NHMFL Annual Research Review 3**





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*The laboratory's major annual report of research has recently been completed and is available on the Internet (http://www.magnet.fsu.edu) in several formats, including CD-ROM. We are proud to devote this issue of our newsletter to highlighting some of the laboratory's outstanding research and engineering activities that are included in the* 2001 Annual Research Review*.*

*As in the past years, the science program of the NHMFL continued to grow in strength and size in 2001. There was strong collaborative research by users and in-house scientists ensuring broad benefits to the scientific community of the unique facilities of the laboratory. This year 322 research reports were submitted in 16 areas of research as compared to 295 reports in 2000.* 



*The number of reports in each of the categories is as follows: biology (49), chemistry (31), geochemistry (10), superconductivity basic (29), superconductivity applied (11), quantum solids (6), kondo/heavy fermions (21), molecular conductors (24), semiconductors (32), magnetism and magnetic materials (45), other condensed matter (13), magnetic resonance techniques (24), instrumentation (10), engineering materials (5), magnet technology (7), and cryogenics (5).*

In order to give a flavor of the overall research *program, we are presenting here a brief overview of selected research programs from each area of research.*







 $\sqrt{\frac{1}{\pi}}$  are investigating the molecular mechanisms of activation in the dinhtherial toxin server. in the diphtheria toxin repressor, DtxR, as a model system for understanding the complex interplay of multiple equilibria involved in regulating protein activity. DtxR is a good model system for these studies because of its small size (226 amino acid residues), its importance in biomedical research, and its rich regulation. The long-term goal of this research is to identify non-metal ion activators of repressor activity to shut down toxin synthesis in people suffering from diphtheria.

In the inactive form, DtxR is incapable of sequence-specific DNA binding, whereas, upon activation by metal ion binding, DtxR binds specifically to the *tox* operator to inhibit transcription. X-ray crystallography structural studies of the apo- and holo-forms of the repressor show that the inactive and active repressor are very similar in structure, differing only by a small rotation of the DNAbinding helix-turn-helix motifs. This small structural difference conflicted with the large differences in biological activity of the two states. We used NMR spectroscopy to investigate possible structural differences between dimeric forms of the apo- and holo-repressors in solution and were immediately faced with a paradox: the 2D  $\,$ <sup>1</sup>H, <sup>15</sup>N correlation spectrum of this 226 amino acid protein, contained only 85 resonances, while "missing" about 140 resonances. After a series of controls, we identified that these 140 resonances, corresponding to the entire N-terminal domain, were experiencing exchange broadening on the intermediate time scale. Spectra of apoDtxR at 4 °C exhibited numerous resonances, consistent with some ordered and some disordered portions of this domain. In contrast, spectra of holo-DtxR were consistent with a higher degree of order in this domain, indicating that metal binding induced a folding transition.<sup>1</sup> This apparent folding transition provides a more plausible explanation for the differential DNA binding of the apo- and holo-repressors than earlier data.



Figure 1. 2D<sup>1</sup>H, <sup>15</sup>N correlation spectrum indicating the presence of ordered and more flexible portions of apo-DtxR at 4 °C.

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MCM-22 is microporous aluminum-silicate that is widely used in the catalytic hydrocarbon conversion reactions.<sup>1</sup> Little attention was paid, however, to the crystallization process of zeolite MCM-22, but it is one of the key factors to understand the chemistry of MCM-22. From its  $^{27}$ Al MAS NMR or  $^{27}$ Al DOR NMR spectra under various fields  $(Fig. 1)<sup>2</sup>$  it is clear that a high resolution was achieved for spectra scanned on a 19.6 T instrument: three distinct resonances attributed aluminum at different T sites were observed. Upon increasing the spinning speed to 35 kHz, additional band at 53.7 ppm is resolved, indicating that the efficiency of high-speed rotor. From the  $27$ Al MAS NMR

<sup>1</sup> Twigg, P.D., *et al.*, *Proc. Natl. Acad. Sci. U.S.A*, **98**, 11259-64 (2001).

(19.6 T, 35 kHz) of MCM-22 in different cystallization stages, it is interesting to note that aluminum at different T sites enter the framework of zeolite consequently and not simultaneously. It is different from the cystallization mechanism of other zeolite like SAPO-34 or HZSM-5 that have been extensively investigated. After calcinations, these four bands become better resolved, suggesting that organic template has a strong interaction with these aluminum species.



**Figure 1.** 27Al MAS or DOR NMR spectra of MCM-22 after 96 h crystallization, A) 9.4 T (8 kHz); B) 16.9 T (DOR); C) 19.6 T (19.9 kHz); D) 19.6 T (35.5 kHz).

- 1 Ma, D., *et al.*, *J. Catal*., **189**, 314 (2000).
- 2 Ma, D., *et al., J. Phys. Chem. B*, **105**, 1770-1779 (2001).

**GEOCHEMISTRY**

 $\overline{\phantom{a}}$  , where  $\overline{\phantom{a}}$ 

Determining the Mineralogy of the MORB Source through Nd-Isotope Analyses of Abyssal Peridotites

Salters, V.J.M., NHMFL Dick, H.J.B., Woods Hole Oceanographic Institution

Interpreting the chemical and isotopic systematics of MORBs<br>in relation to details of the melting process and the physical conditions under which melting takes place hinges on the assumption of compositional similarity or regularity between all MORB sources. For example, the interpretations of regular variations in  $Na<sub>8</sub>$  and  $Fe<sub>8</sub>$  vs. axial ridge depth, thought to reflect a deeper initiation of melting and higher extents of melting beneath shallow ridges, are only valid in the context of chemically and mineralogically homogeneous source mantle.

The presence of garnet-bearing mafic compositions with enriched signatures in the mantle has long been discussed. Garnet-bearing eclogite or pyroxenite blobs or veins in the MORB mantle would likely be associated with plume material and thus, would tend to correlate with the degree of "enrichment" in the MORB source. If residual during melting, garnet pyroxenite can further contribute to the chemically-enriched character of derivative basalts by trace element fractionations that are characteristic of residual garnet.

It has been shown that, in general, the mineralogical, major element and trace element variations of abyssal peridotites are consistent with the major and trace element variations observed in MORB. We have examined the variations in the Sr and Nd-isotopic composition of abyssal peridotites from two ridge segments, the segment near the Atlantis II Fracture Zone (AII FZ) and the 10° to 16°E section of the south east Indian ridge (10° to 16°E) and compared these variations with the Nd-isotope variations in the basalts and. Fig. 1 is a comparison of the Nd-isotopic compositions of the basalts with the data obtained on the peridotites. Basalts from the AII FZ show a normal MORB chemistry and have  $143\text{Nd}/144\text{Nd}$  ratios as low as 0.5130, while basalts from the 10 $^{\circ}$ to 16°E section show an anomalous enriched character and have  $^{143}Nd/^{144}Nd$  ratios ranging from 0.51284 to 0.51302. The REE pattern of diopsides in the AII Fracture zone peridotite show a strongly light REE depleted pattern indicating they are residues of MORB genesis. In comparison, the REE patterns of the diopsides of the  $10^{\circ}$  to  $16^{\circ}E$  area are flat to only slightly light REE depleted and the most enriched REE patterns can be in equilibrium with MORB. The Nd-isotopic composition of the diopsides in the abyssal peridotites confirm the trace element chemistry. Peridotites from the AII FZ have Nd-isotopic composition ranging from 0.513077 to 0.513409, while 143Nd/144Nd ratios for the 10° to 16°E section varies from 0.513031 to 0.513194. Lack of correlations of the isotopes with major or trace element indicators of melt infiltration (Ti-content, Cr/Cr+Al, Sm/Nd, Na-content) indicate that the isotopic variations indeed display the pristine character of the periodtite.

These data further indicate that the Nd-isotopic composition of abyssal peridotites from different ridge segments is more depleted than MORB. The lack of overlap between basalts and peridotites in both segments indicate that the basalts have a contribution of a component different than the peridotite sampled. Two explanations are possible: there are temporal variations in the isotopic compositions of the basalts and isotopically more depleted basalts can be found further off-axis. Although this possibility cannot be ruled out, the lack of depleted basalts along the entire ridge argues against this. The second and more likely possibility is that the MORB source contains an enriched component such as pyroxenite that melts out and contributes to the ridge basalts.



**Figure 1.** Nd-isotopic variations in basalts and peridotites along the South West Indian Ridge. Circles are basalts and squares are peridotites.



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ne of the puzzling aspects of high temperature superconductors is the prevalence of magnetism in the normal state and the persistence of superconductivity in very high magnetic fields. Generally, superconductivity and magnetism are not compatible; but recent neutron scattering results<sup>1</sup> indicate that antiferromagnetism can appear deep in the superconducting state in an applied magnetic field. Superconductivity is suppressed in the vortex core and it has been suggested that antiferromagnetism might develop there.<sup>2</sup> To address this question, it is important to perform electronic structural studies with spatial resolution. We have implemented a high field NMR imaging experiment that allows spatial resolution of the electronic behavior both inside and outside the vortex cores.<sup>3</sup> Outside we find strong antiferromagnetic fluctuations, and localized inside there are electronic states rather different from those found in conventional superconductors.

In Fig. 1, we show the <sup>17</sup>O NMR spectrum and spin-lattice relaxation in the high temperature superconductor  $YBa<sub>2</sub>Cu<sub>3</sub>O<sub>7-x</sub>$ in magnetic fields of 13, 23, and 37 T, the latter obtained in the Hybrid magnet of the NHMFL. The region of the spectrum that is shaded in this figure identifies oxygen nuclei in resonance that

are inside the vortex cores for the applied field of 37 T. This region is defined to be a circle centered on the vortex with a radius of one coherence length,  $\xi$ =16 Å. The fraction of the spectrum associated with the vortex cores is proportional to the applied field and is largest at 37 T. The nuclear spin-lattice relaxation rate is proportional to the joint product of density-of-states from initial and final electronic quasiparticles that leads to a nuclear spin flip. In the vortex core region, we find enhanced relaxation which, from its temperature dependence shown at 37 T in Fig. 2, we associate with antiferromagnetic fluctuations.<sup>4</sup>



Figure 1. The NMR spectrum of <sup>17</sup>O NMR and spin-lattice relaxation in the high temperature superconductor YBa<sub>2</sub>Cu<sub>3</sub>O<sub>7-x</sub> in magnetic fields of 13, 23, and 37 T at a temperature of 11 K. The shaded region of the spectrum corresponds to the vortex core at 37 T.



**Figure 2.** The temperature dependence of the spin-lattice relaxation rate in the high temperature superconductor YBa<sub>2</sub>Cu<sub>3</sub>O<sub>7-x</sub> in a magnetic field of 37 T. The shaded region of the spectrum corresponds to the vortex core at 37 T.

**Acknowledgements:** This work was supported in part by the NHMFL In-House Research program and the Materials Research Center at Northwestern Univ., grant DMR-0076097.

- <sup>1</sup> Lake, B., *et al., Science*, **291**, 1759 (2001).
- <sup>2</sup> Arovas, D.P., *et al.*, *Phys. Rev. Lett.,* **79**, 2871 (1997).
- <sup>3</sup> Mitrovic, V.F., *et al.*, *Nature*, **413**, 501 (2001).
- Mitrovic, V.F., *et al.*, to be published.

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van der Laan, D.C., NHMFL/Univ. of Twente, The Netherlands, Applied Physics van Eck, H.J.N., Univ. of Twente, Applied Physics ten Haken, B., Univ. of Twente, Applied Physics ten Kate, H.H.J., Univ. of Twente, Applied Physics Schwartz, J., NHMFL/FAMU-FSU College of Engineering

To better understand the dominant mechanisms that limit the critical current density in high temperature superconductors, the dependence of the critical current density on magnetic field and temperature of  $Bi_2Sr_2Ca_2Cu_3O_x$  tapes is measured in magnetic fields up to 8 T and temperatures between 4.2 K and 100 K. The results were modeled by describing the current in the tape as two parallel current paths. The critical current density of one path is limited by weak links and that of the other path by intragranular flux pinning. The total critical current density is given by: $<sup>1</sup>$ </sup>

$$
J_{c}(B,T)=J_{cs}(B,T)+J_{cw}(B,T), \qquad (1)
$$

where  $J_{cS}(B,T)$  and  $J_{cw}(B,T)$  are the strong-links and weak-links critical current densities of the tape. The strongly linked current path is described according to the classical flux creep theory. We show that by using a logarithmic dependence of the activation energy, the following expression for the strong-links critical current density is obtained: *n*

$$
J_{cs}(B,T) = J_{cs}(0,T) \left[ \frac{E_0(B,T)}{E_c} \right]^{-} \left[ \frac{B}{B_{irr}(T)} \right]
$$
 (2)

where  $E_c$  is the voltage criterion, and *n* a constant. The magnetic field and temperature dependence of the weak-links critical current density is given as:

$$
J_{cw}(B,T) = \frac{J_{cw}(0,T)}{I + \left[\frac{|B|}{B_0(T)}\right]^{\beta}}
$$
(3)

The critical current density as function of magnetic field for temperatures between 4.2 K and 50 K is shown in a semilogarithmic plot in Fig. 1, while the critical current density as function of magnetic field for the temperature range of  $77$  K to  $100$ K is shown in Fig. 2.

Table 1 lists the parameters used in expression (1) to describe the measurement. The result is that critical current of the weaklinks current path vanishes at a temperature below the critical temperature of the BSCCO 2223 phase. The weakly linked current path has a critical temperature of 86.7 K, while the strongly linked current path has a critical temperature of 106.4 K.



**Figure 1.** The critical current density as function of magnetic field for low temperatures. The points represent the measurement, while the solid lines represent expression.<sup>1</sup>



Figure 2. The critical current density as function of magnetic field for high temperatures.

**Table 1**. Parameter values in the model.

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Value	Unit
106.4	к
319	A/mm <sup>2</sup>
86.7	ĸ
1368	A/mm <sup>2</sup>
0.060	
2.1	т
	V/m
0.8	
	$10^{-4}$

 van der Laan, D.C., *et al*., *IEEE Trans. on Appl. Supercond*., **11**, 3345 (2001).



**V**iscosity measurements on very dilute <sup>3</sup>He-<sup>4</sup>He mixtures  $(x_3=150 \text{ ppm})$  at fields up to 14.8 T and temperatures  $(x_3=150 \text{ ppm})$  at fields up to 14.8 T and temperatures down to 3 mK were performed. The measurements employed a novel vibrating-wire viscometer that incorporates a large-diameter section to limit slip effects. The measurements employed a novel vibrating-wire viscometer that incorporates a large-diameter section to limit slip effects. The <sup>3</sup>He spin polarization achieved was greater than 99% for the highest field and lowest temperature used. We observed a drastic increase of the viscosity due to the polarization.

At low temperatures, the properties of dilute <sup>3</sup>He-<sup>4</sup>He mixtures are characterized by behavior of the 3 He quasi-particles, which behave as a weakly interacting Fermi gas. The transport properties are determined by the *s*-wave scattering between quasi-particles. With spin polarization and vanishing of the minority spin state, there is a decrease of *s*-wave scattering. Thus all transport coefficients are expected to get larger upon polarization.

Fig. 1 shows the line width of the viscometer at two fields, 1.0 and 14.8 T. At the higher temperature the line width is proportional to *T*1/2, which agrees with the expected temperature dependence for the viscosity of a dilute gas. The  $B_0 = 1.0$  T line width has a shallow minimum at about 6 mK. Our base temperature is not sufficiently low to see the *T*-1 behavior expected at temperatures far below the Fermi temperature. At  $B_0 = 14.8$  T a large increase of the line width is clearly seen at lower temperatures. At 3 mK, the line width is nearly ten times larger than that at low field. Although final reduction of our data to field-dependent viscosity values requires a detailed consideration of the hydrodynamics for our viscometer design, the viscometer measurement has shown a huge increase, by a factor of about one hundred, in the viscosity due to the spin polarization.



Temperature ( mK )

**Figure 1.** Line width measured for the viscometer in a  $x_3 = 150$  ppm mixture at 1.0 and 14.8 T.

**Acknowledgements:** This work was supported by the science research program of the NHMFL funded by NSF-DMR-9016241 and the State of Florida.



The dimensionality of the 115 materials appears to be correlated with their superconducting transition temperature. The material with the highest  $T_c$ , CeCoIn<sub>5</sub>, has the most 2D-like Fermi surface (FS) of the three. CeRhIn<sub>5</sub> has a high  $T_c$  (~2.1 K), but only under a pressure of  $\sim$ 16 kbar. At ambient pressures, CeRhIn<sub>s</sub> is an antiferromagnet. The FS of CeRhIn<sub>5</sub> was the subject of one of our recent publications.<sup>1</sup> In order to confirm the link between the superconducting state and FS dimensionality, we would like to observe the FS as a function of pressure in  $CeRhIn<sub>5</sub>$ . If the FS becomes more 2D-like as the critical pressure is approached, then there will be good evidence for making such a correlation.

In order to perform this experiment, we have designed and built small pressure cells, capable of running in a dilution refrigerator and in a rotator. Measuring torque inside a pressure cell is impossible, so we have developed small compensated pickup coils which fit into the cell. Each coil has four to five thousand turns. The filling factor approaches unity because we are able to situate the coil along with the sample in the cell. A small modulation coil

is wound on the exterior of the cell to provide an AC reference signal for the measurement.

We have already measured the FS of  $CeRhIn<sub>5</sub>$  under several pressures. At each pressure, we measure FS frequencies and their amplitude dependence as a function of temperature. From this, we can extract information about how the effective mass of the quasiparticles is changing as the pressure is increased. The figure shows a Fourier spectrum of CeRhIn<sub>5</sub> under  $\sim$ 7.8 kbar. The crystal was oriented so that the a-b axis plane is perpendicular to the applied field.

In these materials, it seems that superconductivity does not appear until the overlap between the *f* electron wave functions is sufficient to allow band-like behavior. There is no reason to believe why the FS should change as a linear function of pressure. Possibly, at some pressure close to the critical pressure, the transition to itinerate behavior will take place. Therefore, further measurements are needed to carefully follow any FS evolution that may occur near the critical pressure.



**Figure 1.** The Fourier spectrum of CeRhIn<sub>s</sub> at ~7.8 kbar is essentially the same as at ambient pressures.

**Acknowledgements:** This work was supported in part by NSF under Grant No. DMR-9971348 (Z.F.). A portion of this work was performed at the NHFML, which is supported by NSF Cooperative Agreement No. DMR-9527035, and by the State of Florida. Work at LANL was performed under the auspices of the U. S. DOE.

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#### **MOLECULAR CONDUCTORS** Magnetic Field Induced Superconductivity in a Magnetic Organic Insulator

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Motivated by preliminary results reported by S. Uji and co-authors,<sup>1</sup> we have recently mapped out the temperaturemagnetic field phase diagram of the field-induced superconducting phase (FISC)<sup>2</sup> in  $λ$ -(BETS)<sub>2</sub>FeCl<sub>4</sub> using AC electrical transport techniques in conjunction with the hybrid and resistive magnets at the NHMFL-Tallahassee. Our magnetic field dependent resistance of a  $\lambda$ -(BETS)<sub>2</sub>FeCl<sub>4</sub> single crystal, under atmospheric pressure, is shown in Fig. 1(a) for different temperatures. Here the magnetic field  $B$  is applied along the in-plane c-axis. Similarly, in Fig.  $1(b)$ we show the resistance of a second single crystal as a function of *B*



**Figure 1.** (a) Resistance *R* as a function of magnetic field *B*, applied along the in-plane c-axis (± 0.3 degrees) of a λ-(BETS)<sub>2</sub>FeCl<sub>4</sub> single crystal (sample #1), at ambient pressure and for temperature intervals of approximately 0.25 K, between 5.4 and 0.8 K. The superconducting state develops progressively with decreasing temperature, but is suppressed for fields sufficiently away from (above or below) 33 T. (We note that since the Hybrid magnet is composed of a superconducting outsert coil in combination with a Bitter type resistive insert coil, the field generated by the outsert is kept constant at approximately 11.5 T, while the field of the insert coil was ramped between 0 and 31.5 T). The FISC transition has a maximum transition temperature  $T_c \approx 4.2$  K near 33 T. (b) As in (a) *R* as a function of *B*, applied along the in-plane c-axis for sample #2. In the present case, the sample is immersed in a fluid medium that induces a very small amount of hydrostatic pressure  $p = ε$  kbar (>> 1 bar) upon cooling. The effect of  $p$ is, on one hand, to considerably decrease the resistivity of this compound and, on the other, to widen the range in magnetic fields at which the FISC state is observable.

<sup>1</sup> Hall, D., *et al.*, *Phys. Rev. B*, **64**, 064506, cond-mat/0011395 (2001).

(up to 45 T) in a liquid medium that induces a small amount of hydrostatic pressure when it solidifies upon cooling. No pressure was applied at room temperature. Notice that the FISC state is now observable in a much broader field range, indicating that this compound, and hence the FISC state, is remarkably sensitive to pressure. The obtained phase diagram is explained in terms of the so-called Jaccarino-Peter effect, see Ref. 2.

**Acknowledgements:** We would like to thank the Hybrid Magnet Group at the NHMFL for their invaluable assistance during these measurements. One of us (JSB) acknowledges support from NSF-DMR-99-71474 for this work.

1 Uji, S., *et al.*, *Nature*, **410**, 908 (2001).

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2 Balicas, L., *et al*., *Phys. Rev. Lett*., **87**, 067002 (2001).



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So far, strong electrical anisotropy in the two-dimensional electron system (2DES) has only been observed at halffilled Landau levels, i.e.,  $v = 9/2$ , 11/2, 13/2, etc. Although the nature of this anisotropy remains uncertain, experimental data and theoretical models point to the formation of a charge "stripe"

In our experiments on samples with two electrical subbands occupied in a symmetric quantum well, however, we find strong electronic transport anisotropies at *fully filled* Landau levels. They are created by very strong in-plane magnetic fields  $(B<sub>in</sub>)$  at very large tilt in the regime of the integral quantum Hall effect (IQHE) at  $v = 4$ , 6, and 8. At these filling factors, the usual *deep minima* in the magnetoresistance occur for the current flowing *perpendicular* to the in-plane magnetic field direction, but develop into *strong maxima* for the current flowing *parallel* to the in-plane field.

The origin of the anisotropy in the IQHE regime is unknown but resembles the observed anisotropy at half-filled Landau levels. Since it only occurs when the two Landau levels cross each other, as evidenced by the disappearance of the Hall plateau, we speculate that in the vicinity of the level crossing a phase separation of the electronic system into spin-unpolarized and partially spinpolarized domains, or "spin stripes," may occur. Unlike the *charge* stripe phase at  $v = 9/2$ , 11/2, etc., here, the charge is uniformly distributed and it is the *spin polarization* that forms the alternating pattern. The resulting stripes of alternating configurations have one-dimensional edge-states and should carry the electric current in a highly anisotropic fashion. In a recent preprint (condmat/0110126), Demler *et al.* suggested an even more intriguing possibility, namely that the observed anisotropy in the IQHE regime might be due to a *skyrmion* stripe phase.



**Figure 1.** R<sub>xx</sub> (solid lines) and R<sub>yy</sub> (dashed lines) around  $v = 6$  for five tilt angles. For  $\mathsf{R}_{\mathsf{xx}}$ , the current runs along  $\mathsf{B}_{\mathsf{ip}}$ , while for  $\mathsf{R}_{\mathsf{yy}}$  the current is perpendicular to B<sub>in</sub>. 2DES density n = 4.2 x 10<sup>11</sup> cm<sup>-2</sup>.



Mahendiran, R., Pennsylvania State Univ., Physics Samarth, N., Pennsylvania State Univ., Physics Jaime, M., NHMFL/LANL

The ferromagnetic semiconductor  $Ga_{1-x}Mn_xAs$  material has been the subject of much recent interest since it can be he ferromagnetic semiconductor  $Ga_{1x}Mn_xAs$  material has incorporated in III-V semiconductor systems with the goal of making "spintronic" devices—utilizing the spin degree of freedom to allow new functionality. We are undertaking a study of a series of  $Ga_{1-x}Mn_xAs$  with various values of x. For this purpose, a series of 24 samples has been recently grown by MBE with Mn compositions spanning the accessible range, and they have been annealed to optimize the ferromagnetic  $T_c$  (such annealing has recently been shown to also result in smooth mean-field-like magnetic behavior).<sup>1</sup>

We are studying how the magnetotransport behavior (including magnetoresistance and Hall effect) and magnetization develop as functions of Mn content and carrier concentration (the two are not equivalent since there can be compensation by defects such as As antisites). Of particular interest will be the behavior near the transitions to insulating behavior at  $x \approx 0.03$  and  $x \approx 0.07$ , which will help us understand how a ferromagnetic ground state occurs without itinerant carriers both at lower and higher Mn doping. We determine the Mn concentration directly by electron microprobe analysis, magnetization by SQUID magnetometry, and magnetotransport at low fields in our 14 T system at Penn State. These transport measurements are not, however, sufficient to determine the carrier concentration of the sample since the negative magnetoresistance is not negligible at these low fields and the anomalous Hall effect is difficult to clearly separate.

At the NHMFL, we have measured magnetoresistance and Hall effect at fields up to 50 T from near room temperature down to well below T<sub>c</sub> on five representative samples of our series of  $Ga_{1-x}Mn_xAs$ . The data have been analyzed to obtain the carrier concentration, and they are being included in analysis of our combined data on the sample series. These systematic measurements should give new insight into the mechanism responsible for the observed ferromagnetism in this material, and provide a basis for evaluating the competing theoretical models of ferromagnetism in the III-V compounds. We are currently finishing analysis of the data and preparing a manuscript for submission to *Physical Review Letters*.

**Acknowledgements:** This research program is supported by DARPA, ONR, and NSF.

1 Potashnik, S.J., *et al*., *Applied Physics Letters*, **79**, 1495-7 (2001).

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Rosenbaum, R.L., Tel Aviv Univ., Physics and Astronomy Lin, S.T., National Cheng Kung Univ., Physics Su, T.-I, National Cheng Kung Univ., Physics

There is little convincing experimental evidence that *bulk* quasicrystals (QCs) of AlPdRe are truly insulating and display activated resistivity behaviors. Using special preparation techniques, bulk QC samples can be prepared to have very large resistance ratios  $R(4.2 \text{ K})/R(300 \text{ K})$  ranging from 30 to 120.<sup>1</sup> These bulk QC samples display *saturation* of their resistance at temperatures below  $4.2 \text{ K}$ .<sup>1</sup> It has been suggested that structural imperfections in the icosahedral structure provide a second conduction path.<sup>1</sup> There has been one published paper reporting Mott variable-range hopping (VRH); but the very small Mott temperature is inconsistent with the Mott formulation.2 S.T. Lin's group has reported a Mott law in the resistivity over a rather limited temperature interval.<sup>3</sup> In contrast, a *QC thin film* of AlPdRe exhibited a Mott VRH law in its resistivity;<sup>4</sup> this film was weakly insulating, having a Mott temperature  $T_{Mott}$  of 3.42 K and a resistance ratio  $R(4.2 \text{ K})/R(300 \text{ K})$  of only 8.7.

We have measured a bulk bar icosahedral AlPdRe sample prepared by arc melting and annealing.<sup>5</sup> This bar sample had a small R(4.2 K)*/*R(300 K) ratio of 13.2. Between 50 mK to 1.6 K, the resistivity followed a Mott VRH law using the expression  $\rho(T)=0.0268 \exp(3.50/T)^{0.227}$  in  $\Omega$ cm. The fit is extremely good, as shown in Fig. 1. The hopping exponent of 0.227 is close to the Mott value of 1/4; and the Mott temperature,  $T_{Mott}$ =3.50 K, is small making this QC bar sample weakly insulating. Note that the exponential fit expands over more than one decade in temperature and gives convincing evidence that this sample is insulating.



Figure 1. Zero field resistance data for a quasicrystal AlPdRe bar sample. The solid line is a VRH activated law. The ratio *R*(4.2 K)/*R*(300 K) is 13.2.

The magnetoresistance (MR) data are extremely similar to those data of the insulating AlPdRe  $\text{film}$ <sup>6,4</sup> namely that the values are positive and *large*. These MR data can be described using the wave function shrinkage model with partial success.<sup>6</sup> This model is suitable for MR data of *strongly insulating* samples. A value for the localization length of 400  $\AA$  was extracted from the fits.

**Acknowledgements:** A portion of this work was performed at the NHMFL, which is supported by NSF Cooperative Agreement No. DMR 9527035 and by the State of Florida and in Tainan, Taiwan, by the National Science Council of the Republic of China (Taiwan) under grant NSC 89-2112-M006-041.

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Ultrasonic Spectrometer for Pulsed **INSTRUMENTATION INSTRUMENTATION High Magnetic Fields** 

Suslov, A., Univ. of Wisconsin-Milwaukee, Physics Sarma, B.K., Univ. of Wisconsin-Milwaukee, Physics Ketterson, J.B., Northwestern Univ., Physics and Astronomy Jaime, M., NHMFL/LANL Balakirev, F., NHMFL/LANL Migliori, A, NHMFL/LANL Lacerda, A., NHMFL/LANL

Ultrasonic velocity and attenuation measurements are powerful tools to study condensed matter systems,<sup>1</sup> especially various phase transitions and collective phenomena. We have constructed a unique ultrasonic spectrometer specifically for the study of condensed matter systems at the intense pulsed magnetic fields at NHMFL, Los Alamos and have used it to study the metamagnetism in  $UPt_3$  and  $URu_2Si_2$ .

The spectrometer is a computer-controlled, phase-sensitive ultrasonic interferometer. A rf signal is split into two channels by a power splitter. One of these signals is gated and sent through the sample: piezoelectric transducers convert the electric signal to a mechanical (sound wave) signal that propagates through the sample with the speed of sound. This attenuated and delayed signal is then mixed with the signal in the second (reference) channel. Phase sensitive detection is done at an intermediate frequency of 60 MHz, and with signal averaging a very high sensitivity in the velocity measurement  $(1:10<sup>7</sup>)$  is achieved.

The short pulse magnets (20 ms, 50 T or 60 T) require approximately 30 to 40 minutes to recover between pulses. In order to collect the maximum amount of data per pulse it is desirable to: (1) digitize and store all echoes in a given pulse train and (2) launch as many pulse trains as possible (need a power amplifier). Typically the ultrasonic pulses are sent at a frequency of 25 kHz or 40 µsec between pulses. During this time the magnetic field changes by 0.2 T.

We use the LeCroy 6810/6310 digitizing systems at the Los Alamos site. During the magnet pulse, a rapid succession of ultrasonic pulses is sent into the sample, and all the information stored. The data is analyzed to obtain the velocity and attenuation information that is then transferred to the computer and stored, freeing up the LeCroy system for the next set of pulses.

For measurements in pulsed magnetic fields the spectrometer has been used in a second configuration, which we call a digital spectrometer. The IF frequency of 12.5 MHz, is extracted by a low pass filter and is digitized by a GaGe-CS 1250 oscilloscope card. The sampling rate is 50 MHz, which allows us to measure four points per IF period. The signal is stored in the memory of the oscilloscope card and later transferred to the computer. The mixing and detecting functions are now performed digitally.

Typically the high accuracy and sensitivity in the ultrasonic measurements is achieved by signal averaging over many pulses; thus requiring a slow sweep rate of the magnetic field. Typical sweep times in the DC fields at Tallahassee were 60 minutes to full field. In the short pulse magnets no signal averaging is possible, since the magnetic field changes by as much as  $0.2$  T between the rf pulses. Also the echo train should be short, so that there is no overlap between echoes of neighboring pulses.

**Acknowledgements:** This research was supported by NSF through grants DMR-9971123 and DMR-9704020, and the NHMFL, Tallahassee and Los Alamos.

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Innovative Bitter Magnet for Uniform Transverse Field over 20 T **MAGNET TECHNOLOGY**

Gavrilin, A.V., NHMFL Bird, M.D., NHMFL

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The NHMFL is designing a high field magnet for condensed matter physics with the field perpendicular to the access tube ("transverse" field). The most attractive innovative option consists of a new type of Bitter magnet in which the plane of the conducting disks is rotated 45 degrees with respect to the central  $axis<sup>1</sup>$  (see Fig. 1). This results in the induced field being oriented 45 degrees off the central axis. A set of two or more such Bitter coils are nested (see figure), with the current flowing in opposite directions within each two adjacent coils. By adjusting the current densities properly, the axial components of the various coils will cancel each other out, leaving only a purely transverse magnetic field. Of course, by tilting the disks at other angles and adjusting the current densities differently, field directions anywhere from purely axial to purely transverse are attainable.<sup>1</sup>

A preliminary design of a three-coil magnet is underway as shown in the Table. This concept<sup>2</sup> is very promising as it suggests a transverse field of 21 T with only 13 MW of power may be attainable, in so doing the maximum stress in the coils does not exceed 400 MPa. The field generated by this magnet is essentially transverse and very uniform over the whole bore along the length of at least 200 mm, as opposed to a Bitter split magnet where an area with high uniformity is very small compared to the bore volume.

**Table 1.** Characteristics of the three tilted coils Bitter magnet (calculated).

Total number of coils	3
Material	Copper
Inner radius (mm)	19
Outer radius (mm)	305
Over-all length (mm)	1195
Rated current (kA)	40
Maximum field @40 kA	20.8
Total power in the	12.62
magnet (MW)	
Total number of turns	



Figure 1. Sketch of the simplest configuration with two nested tilted Bitter coils for transverse field generation.  $B_1$  is the field from the inner coil,  $B_2$  is the field from the outer coil, B is the net resultant transverse field.

1 Gavrilin, A.V., *et al*., "Transverse Field Bitter-type Magnet," USA patent pending.

 $\overline{\phantom{a}}$  , where  $\overline{\phantom{a}}$ 

2 Gavrilin, A.V., *et al*., "Conceptual Design of High Transverse Field Magnets at the NHMFL," presented at the 17<sup>th</sup> Int. Conf. on Magnet Technology, Geneva, Switzerland, Sept., 2001, accepted for publication in *IEEE Appl. Supercond. of 2002*.



Zhang, T., NHMFL/ FAMU-FSU College of Engineering Celik, D., NHMFL Van Sciver, S.W., NHMFL/FAMU-FSU CoE

his study reports the application of Particle Image Velocimetry (PIV) technique to the thermal counterflow heat transport in He II. The PIV measurement of He II thermal counterflow was carried out with a specially designed optical cryostat containing three fused silica windows, two of which are for the laser sheet in and out, and one is for the CCD camera to capture the images. The PIV measurement system consists of a dual head Nd:YAG pulse laser, beam expanding optics, a CCD camera, and a synchronizer to trigger the camera and laser with correct sequence and timing. The thermal counterflow channel with a length of 220 mm and cross section of 20 x 30 mm2 was placed inside the cryostat. At the bottom of the channel is a  $20 \times 20$  mm<sup>2</sup> thin film heater to generate the counterflow. In this experiment, the hollow glass spheres are employed as the seeding particles, which have a true mean density of 165 kg/m<sup>3</sup> and size range from 20 to 50  $\mu$ m.

For each applied heat flux and helium bath temperature, a series of PIV measurements were performed in time sequence just after the heater is turned on and with a time separation of 66.7 ms. Fig. 1 shows a typical measurement result of the thermal counterflow with 1.8 K bath temperature and  $560 \text{ mW/cm}^2$  heat flux. On the left of the figure is the captured image and the right is the PIV analysis result of the velocity distribution. From the experiment results, a parabolic-like velocity profile is observed at the earlier stages, then it becomes flattened when time increases. One reason for this phenomenon is caused by the heater diffusion process, which makes the heat flux homogeneous and the velocity profile flatten. Another reason is due to the development of counterflow from laminar to turbulent. This is indicated by the fact that the development time for turbulent flow as observed in this experiment lies in the time range predicted by Vinen and Chase. The PIV velocity measurements also indicate that most of the region of the flow field has a velocity in the range of  $17$  to  $28$  mm/s. Thermal counterflow heat equation  $q = \rho \cdot s \cdot \nabla v_n$ , however, gives a mean velocity of 40 mm/s. The difference between these two values is mainly attributed to the slip velocity between the seeding particles and the liquid helium. The slip velocity of the particles used in this experiment could be from 2.9 mm/s to 18.13 mm/s, that corresponds to the particle size from 20  $\mu$ m to 50  $\mu$ m.



Figure 1. A typical measurements result of thermal counterflow in He II.

**Acknowledgements:** This work is supported by NSF Chemical Transport Systems Division under grant number: CTS-0001411.

- 1 Chopra, K.L., *et al., Physical Review*, 108, 157 (1957).
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<sup>2</sup> Nakano, A., *et al., Cryogenics*, **34**, 179-185 (1994). 3 Van Sciver, S.W., *Helium Cryogenics*, Plenum, New York, 116 (1986).

## EOPLE IN THE NEWS



**Kee Hoon Kim**, formerly a LANL Director's funded postdoc at the NHMFL, has been awarded an Oppenheimer Fellowship by LANL Director John Browne. The Oppenheimer is the most prestigious postdoctoral appointment at LANL. A maximum of two fellowships are granted each year, and the appointment runs for three years.



**Alan G. Marshall**, FSU Kasha Professor of Chemistry and Biochemistry and director of the ICR Program at the NHMFL, has been chosen to receive the Pittsburgh Spectroscopy Award from the Spectroscopy Society of Pittsburgh. This award is presented to "an individual who has established a career of accomplishmentst toward the advancement

and understanding of spectroscopy." He also received the American Chemical Society Award in Analytical Chemistry, sponsored by Fisher Scientific Company. The award recognizes Marshall "for his pioneering comprehensive development of theory, instrumentation, and analytical applications of FT-ICR ultrahigh-resolution mass spectrometry." Joining 13 other Florida Award winners from the FSU Department of Chemistry, Marshall was awarded the 2002 Florida Award from the Florida Section of the American Chemical Society. Marshall has received eight national or international awards in the past eight years.



**Willy Nerdal**, Associate Professor at Kjemisk Institutt, Universitetet I Bergen in Norway has been working in the NMR Spectroscopy and Imaging Program for the past several months. Nerdal's work extends his membrane efforts into the structure of membrane proteins through solid state NMR of the potassium channel from *Streptomyces lividans* (KcsA). While a

modest resolution structure of this channel has been obtained from X-ray diffraction of crystalline protein in a detergent environment, the solid state NMR structural information will be obtained from the protein in a more native lipid membrane environment. This work should confirm or refute the assumption that detergents represent an adequate environment. If adequate, the solid state NMR data will provide considerable high resolution detail that can refine the X-ray structure leading to a much greater understanding of the channel specificity and conductance efficiency than can be deduced from the X-ray structure alone.



**John Singleton**, a Reader in Physics at Oxford University and member of the Correlated Electron Systems Research Group, has recently accepted an offer to join LANL as a new member of the NHMFL. Singleton is well known to the NHMFL community, having performed numerous experiments in the Pulsed Field and DC Field Facilities. He also recently completed a year-long sabbatical at NHMFL-LANL where he developed techniques for high frequency measurements of metals in pulsed magnetic fields. His research interests include crystalline organic molecular metals and magnets, magnetic oxides, oxide superconductors, superluminal light sources, semiconductors, and biophysics. Singleton will begin working at the NHMFL in mid-summer 2002.



**Jack J. Skalicky**, a former research associate in Chemistry at the State University of New York, Buffalo, has joined the staff of the NMR Spectroscopy and Imaging Program as an Assistant Scholar/Scientist. Skalicky will perform Structural Biology research, with a primary interest in the development and applications of high resolution NMR

of biomolecules in supercooled water solutions. Supercooling provides enhanced sensitivity and often alters the lineshape of resonances due to changes in the equilibrium of various conformational exchange processes. The combination of supercooling and high magnetic fields is especially exciting and will contribute to improved three dimensional structures of proteins and nucleic acids and will provide unique insights into the coupling between dynamic processes and biological function in these molecules.



**Danko van der Laan**, Ph.D. candidate at the University of Twente, the Netherlands, and graduate research assistant in the HTS Magnets and Materials Group at the NHMFL, received the award for "Best Poster in Large Scale Applications of Superconductivity," for his presentation "Magneto-Optical Imaging Study of the Crack Formation in

Superconducting Tapes Caused by Applied Strain" (co-authors: M.W. Davidson, NHMFL; B. ten Haken, Univ. of Twente; H.H.J. ten Kate, Univ. of Twente; and J. Schwartz, NHMFL). The presentation was held at the European Conference on Applied Superconductivity 2001 in Copenhagen, Denmark, where Danko was also invited to give a talk on "Interpretation of the Critical Current in  $Bi_2Sr_2Ca_2Cu_3O_x$  Tape Conductors as Parallel Weak-link and Strong-link Paths." The prize was awarded by the Board of Directors of the European Society on Applied Superconductivity.



**Zoran Zujovic**, Research Associate with the Institute of General and Physical Chemistry in Belgrade, Yugoslavia, is working in the NMR Spectroscopy and Imaging Program with Zhehong Gan. Zujovic is currently conducting research on NMR pulse sequence development and improving the performance of NMR in studies of biological systems in solids.

# CONFERENCES & WORKSHOP A

## 4<sup>th</sup> International Conference on the Scientific and Clinical Applications of Magnetic Carriers

**May 9-11, 2002 Tallahassee, Florida**

Hotel Headquarters: Doubletree *http://www.magneticmicrosphere.com/*

The laboratory is pleased to support this biennial conference being organized by the Cleveland Clinic Foundation in collaboration with IFAB, Rostock, Germany, and FSU. The meeting will be devoted to the development and application of magnetic microspheres in the basic and clinical sciences and will include a visit to the NHMFL. Invited speakers include: Jack Crow, NHMFL Director, "The National High Magnetic Field Laboratory: A National Resource in Support of Bio-Medical Science and Technology" and Kurt Hofer, Professor of Biological Sciences at FSU, "Hypothermia in Cancer Therapy." Professor Tim St. Pierre, a Senior Lecturer in the Biophysics Programme of the Department of Physics at the University of Australia, will give a featured lecture series of 3 half-hours (one per meeting day) about the basic physics knowledge everybody in the field of magnetic targeting should know. For more information, contact Urs Hafeli at hafeliu@ccf.org.

## Applied Superconductivity Conference (ASC04)

**October 4-8, 2004 Jacksonville, Florida**

Hotel Headquarters: Adam's Mark

This important international conference is held every two years and typically attracts approximately 1,800 participants. In September 2000, ASC00 was held in Virginia Beach, Virginia; in August 2002, ASC02 will be in Houston, Texas, and in October 2004, ASC04 comes to Jacksonville, Florida. For information, contact ASC04 Conference Chair Justin Schwartz in NHMFL's Magnet Science & Technology program: 850-644-0874, fax 850-644-0867, *schwartz@magnet.fsu.edu*.

### 15th Conference of the International Society of Magnetic Resonance (ISMAR 2004)

#### **October 24-29, 2004 Jacksonville, Florida**

Hotel Headquarters: Sawgrass Resort

The scientific program of this major international meeting encompasses lectures and posters representing the various fields of magnetic resonance, showing its breadth and interdisciplinary nature. The program reflects the progress and exciting developments of new techniques, theory, and applications in chemistry, physics, biology, medicine, and materials science.

The conference Chairman is Timothy A. Cross, NHMFL/FSU NMR Spectroscopy and Imaging Program Director/Professor. Complete conference information will be available in the near future.





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