

# REPORTS



Operated by: Florida State University • University of Florida • Los Alamos National Laboratory • Fall 2001 • Volume 8 • No. 4

## **PROTON NMR STUDIES of SPIN DENSITY WAVE FLUCTUATIONS UP to 1.9 GHz**

**IHRP Awards  
for 2001**

**Schrieffer Symposium**

**PPHMF-IV Conference**

**A New School Year / New  
Challenges for Educators**

**Field Induced  
Superconductivity in a  
Magnetic Organic  
Conductor**

UCLA NMR coil  
and LiVGe<sub>2</sub>O<sub>6</sub>  
sample

44.5 Tesla

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*Published by:*

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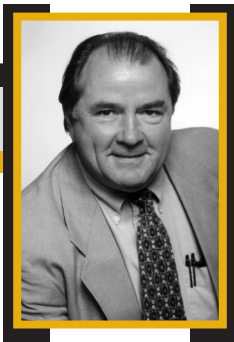
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## From the Director's Desk

Jack Crow

### Honoring NHMFL Chief Scientist, J. Robert Schrieffer

In the last issue of *NHMFL Reports*, I devoted this space to a recap of 2001 NHMFL conference activity, including major meetings on magnet technology, ICR, NMR, EMR, condensed matter and materials physics. I remarked then that the “signature event for the year will surely be a special symposium on October 19 in honor of NHMFL Chief Scientist and Nobel Laureate Bob Schrieffer.”

What I could not imagine then was just how extraordinary the event would be. The program was packed with outstanding talks and discussions and the evening banquet attracted nearly 150 people, including five other Nobelists: Phil Anderson, Alan Heeger, Walter Kohn, Bob Laughlin, and Horst Störmer.

No words of mine could compare with those of others who reflected so eloquently on their relationships with Bob and his life-long commitment to science and the physics community. To give readers a sense for the very special and heartwarming evening, I am pleased to share a few of the accolades.

From **Leon Cooper** (who was unfortunately unable to attend):

*“One of the happiest accidents of my life was to have come to the University of Illinois when Bob Schrieffer was there to work on the same problem that Bob had been assigned for his thesis. It was, as we are all aware, a long time unsolved problem of extraordinary difficulty; we spent*

*many hours together discussing possible approaches and commiserating with one another. (Bob shared an upstairs graduate student office called the Institute for Retarded Studies). ‘I’m never going to finish my thesis’ Bob would say to me. ‘I’m never going to get a job,’ I would respond.*

*Then, suddenly, things came together. We were as runners in a race—handing the baton to one another—each contributing a critical lap while the others rested, exhausted; and together we won. But we were so young, so intense, so immersed in complex calculation, that the months passed as days. We were so involved that we didn’t have the luxury of time to enjoy the experience as it was happening.”*

From **Doug Scalapino**:

*“I remember the first day we met 40 years ago in the ‘cubicle offices’ at the University of Pennsylvania. I was thinking that this arrangement wasn’t suitable even for a postdoc. However, when you arrived and I realized that one of these spaces was to be your office, I was glad that I hadn’t complained! I saw the graceful way you handled it, and mostly I remember how it turned out to be a special place where there was time to talk physics.*

*Most, though, I remember who showed me, by example, how physics could be done. Thanks, Bob.”*

From **Charlie Slichter**:

*“It seems just yesterday that we were together in Urbana, you, Leon, and John cracking the puzzle of superconductivity,*

*helping Chuck Hebel and me explain our experimental results. The excitement of those days and the fun of working with you are still vibrant emotions.*

*I remember clearly the day John stopped me in the hallway of the old Physics Building and said, 'Well, I think we've solved the problem of superconductivity.' What a one-liner! That is how I learned that the three of you had made the big breakthrough. I must have been one of the first to know. This may have been just a day or so after you came back from New York and told John of your idea about how to write the wave function."*

From **Catherine Kallin:**

*"I was postdoctoral fellow at the Institute for Theoretical Physics from 1984 to 1986, and Bob was Director of the Institute. Despite his administrative duties, Bob met regularly with me, Steve Kivelson, and with his graduate student, Daniel Arovas. It was these meetings which resulted in our 'correlated ring exchange' theory of the fractional quantum Hall effect. It was a great environment with great people and I'll always remember Bob for his inspiration and support."*

In addition to words of friendship and praise, Dr. Schrieffer was also presented with other tokens of appreciation, including a unique Steuben crystal sculpture (in recognition of his position as a Corning Glass Fellow at the time of developing the BCS theory), a key to the city of Eustis, Florida, where he attended high school, a letter from President George W. Bush, and a proclamation from the State of Florida, signed by Governor Jeb Bush and all members of the Florida Cabinet.

Special recognition and thanks go to Lev Gor'kov and Nick Bonesteel for reviewing Bob's papers and selecting and editing a significant group of them for a special commemorative edition to be published by World Scientific early next year. This



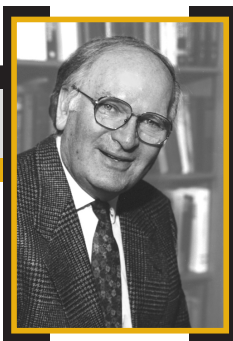
Steuben crystal sculpture representing the BCS theory.

volume will be available to the public, with 10 percent of the proceeds going toward a fellowship at Florida State University. I would also like to acknowledge Janet Patten, NHMFL Director of Government and Public Relations, and Mary Layne, Science Program Coordinator, who were primarily responsible for planning and orchestrating this wonderful event.

Thanks to everyone, in attendance or otherwise, for your support and participation. Finally, I would like to thank Bob Schrieffer for being Bob Schrieffer—a great scientist and a warm and friendly colleague. You can often find greatness in one individual, and warmth and compassion in another, but rarely are they combined in one person. Bob Schrieffer is one of these rare people.

*Jack Crowe*

Director



## From the Chief Scientist's Desk

J. Robert Schrieffer

The NHMFL In-House Research Program has been highly successful as a mechanism to support outstanding projects in the several areas of research pursued in the laboratory. In the past, the in-house component of proposal reviews was carried out by a committee composed of scientists in all of these areas. In order to improve and facilitate the review process, it was decided to form sub-committees whose members would review only proposals in their area of expertise. Other changes, including online submission and review of proposals, also improved the efficiency of the program. We are confident that these new strategies will strengthen what is already an excellent method of initiating novel research that will attract external funding following two years of internal support.

As reported in the Spring 2001 issue of *NHMFL Reports*, the IHRP has undergone changes that are intended to make it more responsive to proposers and improve the expertise in judging the proposals. Along with the new procedures, a new and more efficient way to submit proposals was enacted. For the first time, the 2001 proposals were submitted electronically. All reviewers had access to the IHRP Web site and performed their reviews online. This new submission technique not only saved money, but also time. The IHRP Web site performed quite efficiently, and the future will bring more improvements in submitting not only the proposals, but the semi-annual reports as well.

### IHRP Awards for 2001.

Research Area	# Pre-Proposals Submitted	# Proceeding to Full Proposal Status	# of Projects Funded
Condensed Matter Science	11	4	2
Biological & Chemical Sciences	14	5	2
Magnet & Magnet Materials Technology	8	3	2
<b>TOTAL</b>	<b>33</b>	<b>12</b>	<b>6</b>

### Funded Projects for 2001.

Lead P.I.	NHMFL Institution	Project Title
William Brey	NHMFL	<i>900 MHz NMR Probe Development</i>
Zhehong Gan	NHMFL	<i>High Resolution NMR Above 1 GHz Using Keck Resistive Magnet</i>
Ke Han	NHMFL	<i>Development of High Strength Dispersion Strengthened Conductors for Magnets</i>
William Markiewicz	NHMFL	<i>High Strength Superconductor for High Field NMR Magnets/Enhanced Bronze High Strength Nb<sub>3</sub>Sn Superconductor</i>
Christopher Stanton	UF	<i>Ultrafast Optics of Excitons in High Magnetic Fields</i>
Stanley Tozer	NHMFL	<i>Exploring the Interplay Between Magnetism and Superconductivity in the Heavy Fermions</i>

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**Below are short summaries of each of the new IHRP grants.**

#### ***900 MHz NMR Probe Development***

William Brey, Timothy Cross, and Peter Gor'kov, NHMFL/Tallahassee, CIMAR

A unique opportunity exists at the NHMFL. The development of the 100 mm bore, 900 MHz high resolution NMR (nuclear magnetic resonance) magnet will be a colossal achievement that will demonstrate the preeminence of the NHMFL's superconducting magnet development group. This achievement will be immediately noted by hundreds of NMR spectroscopists worldwide. However, such a magnet is only a component of an effective NMR spectrometer. Even with a state-of-the-art commercial NMR console, the spectrometer is incomplete. NMR probes are needed to expose the sample to spatially uniform radio frequency (RF) magnetic fields at multiple frequencies in complicated pulse sequences involving up to hundreds of pulses timed to generate specific nuclear spin coherence pathways. The probe then couples energy in a single frequency band back to the NMR console for Fourier analysis. Commercial probes that will take advantage of the NHMFL magnet's wide bore will not be developed for many years because there are no commercial widebore 900 MHz magnets available or even planned as commercial products. The NHMFL's core budget will support basic single and double resonance probe development for the 900 MHz magnet. To address the more advanced triple resonance static and magic angle spinning NMR probes, this proposal brings together two of the world's leading probe development groups with the NHMFL's NMR probe development team. The performance level of these probes will, in large part, determine the range of experiments in structural biology of membrane proteins that can be performed with the 900 MHz magnet.

#### ***High Resolution NMR Above 1 GHz Using Keck Resistive Magnet***

Zhehong Gan, Peter Gor'kov, and William Brey, NHMFL/Tallahassee, CIMAR

This proposal explores the feasibilities of using the 25 T Keck resistive magnet at the NHMFL for high-resolution NMR with H resonance frequency above 1 GHz. It proposes the use of small sample magic-angle spinning to reduce the magnetic field homogeneity effect and a newly developed Hetero Nuclear Phase Correction (HENPEC) method to correct the magnetic field fluctuations and drifts of the Keck magnet. The combination of the two methods improves spectral resolution and preliminary data shows that 10 ppb  $^1\text{H}$  line width can be reached for high-resolution NMR. The high-resolution capability would allow exploratory studies of field-dependent NMR phenomena at resonance frequencies above 1 GHz. Two recent developments in protein NMR spectroscopy, the Transverse Relaxation Optimized Spectroscopy (TROSY) experiment and the

field-induced residual dipolar couplings in solution, will be studied at 25 T. The solution NMR experiments using the Keck magnet will demonstrate the high-resolution NMR capability at field strengths not available by superconductive magnets.

#### ***Development of High Strength Composite Conductors for Magnet Applications***

K. Han and R. Walsh, NHMFL/Tallahassee, MS&T

The project suggests three connected approaches to the development and characterization of high strength conductors made of nano-scale  $\text{Al}_2\text{O}_3$  or Ag particle reinforced Cu and nano-scale Cu particles reinforced Ag. The conductors will have the potential to be used for fabrications of high field magnets. The first portion of the project involves both drawing and rolling processes of such materials to a large strain. The second portion of the project is concerned with the deformation at a cryogenic condition, and the third one involves the deformation at a high strain rate (shock loading), where work hardening rate is higher because of the increase of the storage of the defects in the materials. The fabricated conductors will have a range of elastic modulus, strength levels, and conductivities to meet the different requirement of various magnets. Such a conductor has significant impact on the fabrication of the next generation of high field magnets and secures the pre-eminence of the magnet facilities in the NHMFL in the future. The project is also concerned with the rationalization of the deformation mechanism of composites under various conditions. We will compare Cu and Cu+nano-particles in terms of the dislocation structures and other defect formations at room temperature deformation, cryogenic deformation, and shock-loading conditions. This will not only enable us to develop new materials, but also help us to understand the behavior of the Cu+nano-particle materials in pulse magnets, because the conductors in pulse magnets are subject to the cryogenic deformation.

#### ***Enhanced Bronze High Strength $\text{Nb}_3\text{Sn}$ Superconductor***

W.D. Markiewicz, NHMFL/Tallahassee, MST

An improvement in the mechanical strength of composite bronze process  $\text{Nb}_3\text{Sn}$  superconductor would be reflected in improvements in high field magnet designs. It is suggested that significant improvements in the strength of the conductor may be achieved through strengthening of the bronze that is already an integral part of the conductor. Other configurations of high strength  $\text{Nb}_3\text{Sn}$  conductor have been developed, all of which rely on the addition of strengthening material to the base conductor. The stiffness of the wire is increased at the expense of the conductor size and current density. Furthermore, the allowable operating strain may actually be decreased as in the case of tantalum strengthened wires in which the precompression on the  $\text{Nb}_3\text{Sn}$  is reduced. In contrast, increasing the strength of

the bronze already present does not alter the wire size or current density. Because a stronger bronze may increase the precompression, an ideal solution to high strength Nb<sub>3</sub>Sn may be one that balances the changes in precompression with a combination of the high strength bronze and tantalum reinforcement. Strengthening mechanisms for the bronze include solid solution, dispersion, and a novel internal oxidation dispersion. Conductor processing requirements impose severe restrictions on the possibilities for strengthening the bronze. A program is proposed for the development of a high strength bronze that is compatible with use in the bronze process Nb<sub>3</sub>Sn superconductor. A variety of bronze samples would be produced to explore specific strengthening mechanisms. The bronze would be subjected to mechanical tests and used in the fabrication of conductors for electrical tests.

#### ***Ultrafast Optics of Excitons in High Magnetic Fields***

C.J. Stanton and D.H. Reitz, University of Florida, Physics  
J. Kono, Rice University, Electrical and Computer Engineering

This project will significantly advance the optical spectroscopy capabilities of the NHMFL through the development of an ultrafast optics facility and its application to semiconductor quantum structures to investigate the dynamics of excitons in high magnetic fields. Particular emphasis is placed on how we can create and control quantum coherence in excitons that are simultaneously subjected to an intense laser field and an intense magnetic field. Such a system provides an ideal environment in which to address many fundamental and unresolved issues in nonlinear and quantum optics in solids in a well-controlled manner. The laser field manipulates discrete internal states of excitons; whereas, the magnetic field freezes the center-of-mass motion of excitons and tunes the internal energy levels. The large spatial extent of excitons, compared to atoms, leads to strong light-matter coupling, and dramatic non-perturbative phenomena are expected. Such research provides significant insight into the fundamental physics of light-matter interaction and can answer whether light excitation in solids is really quantum, as in quantum optics of atoms.

To achieve this, we have formed a unique interdisciplinary team to conduct this innovative research consisting of three investigators with different backgrounds. Kono is an expert in time-resolved optical studies of semiconductors in high magnetic fields; Reitze's expertise is in ultrafast optics, laser spectroscopy and coherent control; and Stanton is a theoretical condensed matter physicist with a background in electronic, transport, and optical properties of semiconductors and their heterostructures. A key feature of this proposal is our combined expertise in the theory and spectroscopy of semiconductor structures. Each investigator could not separately accomplish all of the objectives of this

program alone. It is essential that a tightly knit, multi-investigator program be established.

#### ***Exploring the Interplay Between Magnetism and Superconductivity in the Heavy Fermions***

S. Tozer, E. Palm, T. Murphy, and D. Hall, NHMFL/  
Tallahassee, Operations

C. Agosta, Clark University, Physics

N. Fortune, Smith College, Physics

115s consist of CeMIn<sub>5</sub> (where M can be Rh, Ir, or Co) and form in the tetragonal HoCoGa<sub>5</sub> crystal structure, which is the same crystal structure as the CuO-based high T<sub>c</sub> materials such as YBa<sub>2</sub>Cu<sub>3</sub>O<sub>7</sub>. The 115s are heavy fermions, have complex magnetic behavior, and often develop a superconducting ground state. The series progresses from the parent compound, CeM<sub>3</sub>, that has an antiferromagnetic (AF) ground state and is a superconductor (SC) at 100 mK under 25 kbar of pressure to CeRhIn<sub>5</sub> with a T<sub>c</sub> of 2.1 K under 16 kbar of pressure to CeIrIn<sub>5</sub> with a T<sub>c</sub> of 0.4 K at ambient pressure and currently terminates with the highest ambient pressure heavy fermion superconductor discovered to date, CeCoIn<sub>5</sub> (T<sub>c</sub> = 2.3 K). The latter also displays an intriguing quasi two-dimensional superconducting state. The 115s provide a wonderful opportunity to observe the subtle interplay between magnetism and superconductivity by combining the extremes of pressure, field, and low temperature.

This team of researchers proposes to continue and extend their current research on these materials using dHvA oscillations coupled with high pressure as a tool to measure the Fermi surface of these materials. Angular dependent pressure studies will be used to observe the evolution of the Fermi surface from AF to SC. In addition, these materials will allow the observation of the Fermi surface as the material changes from a good metal to a heavy fermion as the chemical composition is changed.

They propose the following facilities to perform this work. The first is a pressure cell with a sample space large enough to permit a coil to be placed inside for magnetic measurements. A tunnel diode oscillator system will be constructed to measure quantum oscillations of the surface conductivity at ambient and high pressure. The third development is a compact 2-axis rotator that will allow rotations of the above apparatus *in situ*. All of these will be developed in such a way that they will be user friendly, compatible with both the resistive and superconducting magnets, and available to all of the users of the NHMFL. Future enhancements to these proposed apparatus by other groups might allow for high pressure NMR and heat capacity studies, 2-axis rotator capability at the high B/T and pulsed magnet facilities, and high pressure dHvA studies in the pulsed field environment using available plastic diamond anvils or sapphire.

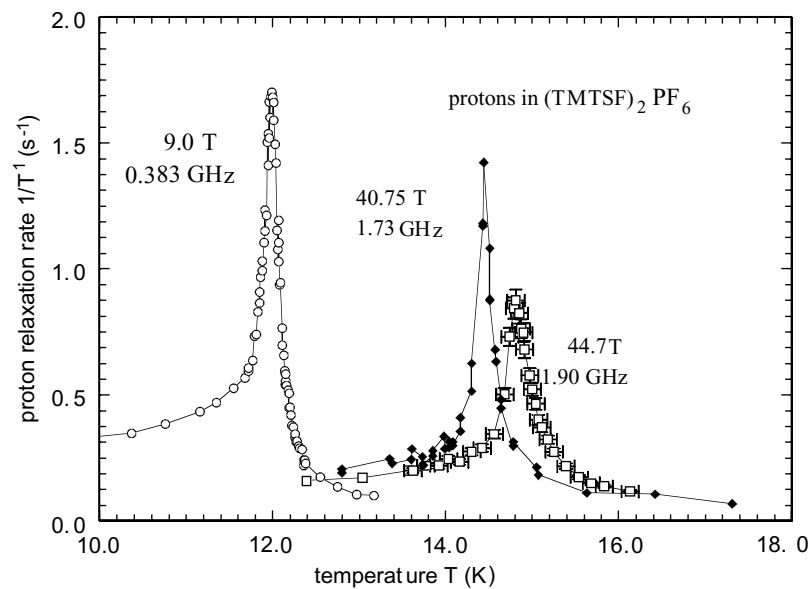
# Proton NMR Studies of Spin Density Wave Fluctuations up to 1.9 GHz

W. Gilbert Clark, UCLA, Physics and Astronomy  
Patrik Vonlanthen, UCLA, Physics and Astronomy  
Philip Kuhns, NHMFL  
Arneil Reyes, NHMFL  
William G. Moulton, NHMFL/FSU

Spin density wave (SDW) order and fluctuations associated with this low temperature phase of quasi one-dimensional conductors, such as  $(\text{TMTSF})_2\text{PF}_6$ , have been widely investigated for a substantial length of time. In part, this is because they reflect the static and dynamic properties of the pinned density wave phase and provide a means to compare these properties with theoretical models for their origin. One NMR measurement of interest is the nuclear spin-lattice relaxation rate  $1/T_1$ .<sup>1-3</sup> For the ordered phase of the SDW not far below the SDW transition, it is generally agreed that thermally excited phase fluctuations of the pinned SDW (phasons) are responsible for  $1/T_1$ . In this regime, the phason fluctuations generate a fluctuating magnetic field whose power spectrum is frequency dependent. Since the fluctuation motion of the SDW also corresponds to polarization charge fluctuations of the condensed electrons that make up the SDW, according to one model,<sup>4</sup>  $1/T_1$  is proportional to the sum of the imaginary part of the SDW dielectric constant over all wave vectors at the NMR frequency. Thus, through measurements of  $1/T_1$  as a function of frequency, one investigates the dynamics of the pinned SDW charge fluctuations on different time scales.

Two other regions of temperature are also of interest in this field. One is the critical fluctuations close to the SDW transition whose temperature ( $T_{\text{SDW}}$ ) is near 12 K at low field. The other is the low temperature region below about 4 K, where there is an unresolved controversy regarding several possibilities, including a simple slowing of the dynamics,<sup>2</sup> additional phase transitions,<sup>1</sup> and opening a gap for a residual conduction electron density.<sup>5</sup>

One of the experimental goals in this field is to extend the frequency of the measurements as high as possible. In a recent set of measurements in the Hybrid magnet at the NHMFL in Tallahassee, we carried out such measurements up to a world record of 1.9 GHz for proton NMR measurements. Here, we give a preliminary, qualitative report of some of this work and describe several special circumstances of the experiments.



**Figure 1.** Proton spin-lattice relaxation rate as a function of temperature at three values of the magnetic field. The peak in  $1/T_1$  occurs at  $T_{\text{SDW}}$  and the rapid variation near  $T_{\text{SDW}}$  is caused by critical fluctuations. At temperatures more than 1 K below  $T_{\text{SDW}}$ , the relaxation is caused by pinned SDW phase fluctuations.



Part of the results are summarized in Fig. 1, where  $1/T_1$  in the critical and SDW regimes is shown for three values of the applied field ( $B$ ) aligned approximately 30 degrees from the  $c^*$ -axis in the  $b^*c^*$  plane. The values of  $B$  and the corresponding NMR frequency are: 44.7 T (1.90 GHz), 40.75 T (1.73 T), and 9.0 T (.383 GHz at UCLA). For all three values of  $B$ , a sharp peak is seen where  $1/T_1$  is dominated by critical fluctuations near  $T_{\text{SDW}}$ . The value of  $T_{\text{SDW}}$  has a weak, quadratic dependence on  $B$ , with values that agree with transport measurements for  $B$  along  $c^*$ .<sup>6</sup> The latter were interpreted using a model of imperfect nesting. Since this temperature dependence disappears for  $B$  along  $b'$ ,<sup>6</sup> there should be an angular dependence to it. Our measurements at 30 degrees from  $c^*$  show a value close to that for  $c^*$ .<sup>6</sup> We plan to extend them to other angles to investigate this aspect of nesting in the material.

Approximately 1 K below the transition, it is seen that  $1/T_1$  decreases monotonically with frequency/field, which indicates that the power spectrum of the density wave fluctuations continues to decrease

with frequency up to 1.9 GHz. Although we had anticipated a change in this behavior that could be attributed to a larger amplitude of phason fluctuations at wave vectors that are too short to be screened and damped by thermally excited quasiparticles, it did not occur.

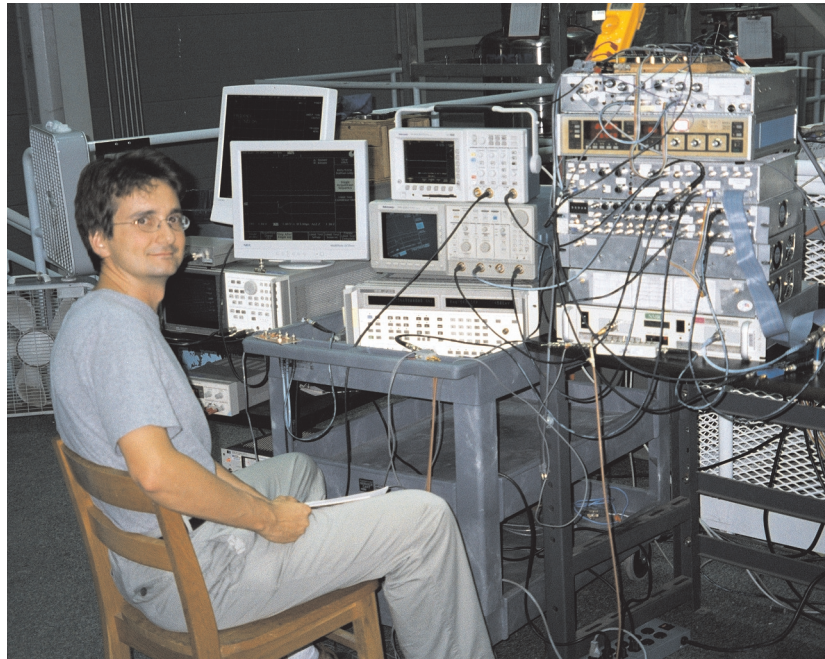
There are two aspects of  $1/T_1$  near the transition that should be noted. The first is that somewhat away from the transition in the high temperature side, its value as a function of the temperature difference from  $T_{\text{SDW}}$  is essentially independent of frequency, including other measurements that go down to 15 MHz.<sup>3</sup> This means that unlike the fluctuations in the ordered phase, those in this part of the critical regime are essentially independent of frequency. Also, they do not display the angular dependence seen in the ordered phase.<sup>2,7</sup> This suggests that they are dominated by amplitude fluctuations rather than phase fluctuations. Another point is that the peak amplitude at the transition appears to decrease as a function of frequency. Unfortunately, on the basis of the present measurements, we cannot tell whether this decrease is a real property of the material or an

artifact of the measurements. The problem is that the peak is very narrow and we had relatively large temperature fluctuations during the run. Subsequently, at UCLA we have identified the origin of the thermal fluctuation problem and decreased it by more than a factor of 20. If future measurements confirm the decrease in  $1/T_1$ , the challenge will then be to determine whether its origin is critical slowing that becomes evident in higher frequency measurements, an effect of the high field on the nesting conditions, or some other mechanism.

A challenging part of these measurements was to complete them in the very short running time available. One of the strategies we used was a continuous saturation method for measuring  $1/T_1$ . It permitted us to carry out individual measurements about 4 times faster than more conventional methods available with our instrumentation and signals. As a consequence, the 68 points shown at high fields in Fig. 1 include three conventional ones that required a total of 20 minutes and 65 that were done in 130 minutes.



**Figure 2.** NMR probe and measurement team. L to R: Phil Kuhns, Arneil Reyes, Patrik Vonlanthen, and Gil Clark



**Figure 3.** Patrik Vonlanthen, the .005-2 GHz NMR spectrometer, and related instruments used for the experiments.

the NMR spectrometer and associated instruments. In the stack of chassis' at the right, the bottom one is the 1 to 2 GHz power amplifier provided by the CMP-NMR group. The next four include the rf power amplifiers and NMR spectrometer brought from UCLA.

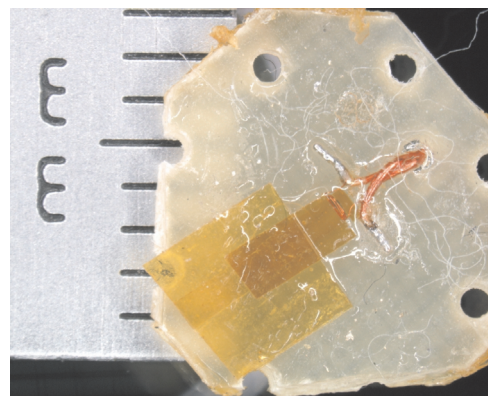
During the recent run in the Hybrid the week of August 20, 2001, we also worked on a project that demonstrated the possibility of doing NMR on very small single crystals. In this case, the goal was to investigate the 1-D antiferromagnetic properties of  $\text{LiVGe}_2\text{O}_6$ .

A key element of the success of our measurements was the level of cooperation and collaboration provided by the staff at the NHMFL. Although the probes and part of the spectrometer were brought from UCLA for the experiments, the rest of the NMR instrumentation and essential collaboration in the measurements were provided by the NHMFL condensed matter physics NMR (CMP-NMR) group. We were also fortunate to have very effective support from the magnet operators, the cryogenics group, the machine shop, the electronics shop, the instrument shop, and those responsible for managing the activities of external users.

Some of the specialized NMR instrumentation used for the experiments along with the persons most closely associated with the project are shown in Figs. 2 and 3. Fig. 2 shows four of us on the platform at the top of the Hybrid magnet. The dewar and NMR probe are at the lower right. Since access close to the magnet is not permitted when the field is ramped up, the long, flexible epoxy glass rod, held by Phil Kuhns and attached to the probe goniometer control shaft, is used to make adjustments during the run. Fig. 2 shows Patrik Vonlanthen with

One of the challenges for this work was that the sample was very small and it had to be rotated about one axis at high field in the NMR probe during the measurements. The probe design steps taken to carry out these measurements are shown in Figs. 4 through 6.

Fig. 4 shows the platform to which the NMR coil and its leads are attached. The platform, which is



**Figure 4.** Epoxy-glass platform with NMR coil, sample, and lead wires attached. The markings on the scale are spaced 1 mm.

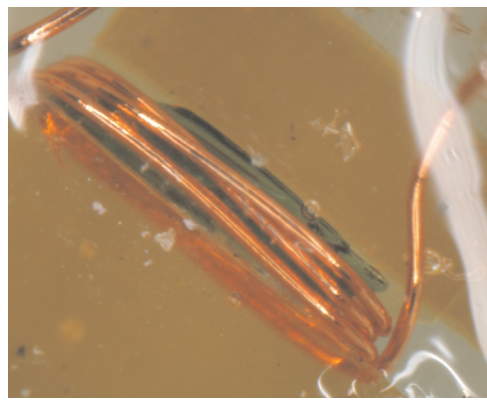


**Figure 5.** Expanded view of Fig. 4.

formed from a thin, epoxy-glass sheet, is attached to a holder that permits rotation of the sample in the magnetic field. A scale with markings one millimeter apart indicates the size of the platform. Fig. 5 is an enlargement of Fig. 4. At the upper right are the larger copper wire leads that connect the NMR coil to the rest of the probe circuit. The structure in the center is the NMR coil wrapped around the dark sample inside of it. The light streaks in the picture are reflections of light from the surface of a thin, clear epoxy layer that encapsulates the sample, the NMR coil, and part of the larger wire leads.

An enlarged view of the coil and the sample is shown in Fig. 6. The coil is three turns of 25 micron diameter wire placed closely on the sample to obtain the best possible signal. This thickness is about 1/2 to 1/3 the diameter of a human hair. The coil is wound around the long dimension of the sample to obtain the desired direction of the 737 MHz radio frequency magnetic field used for the NMR measurements. The sample itself is a single crystal of  $\text{LiVGe}_2\text{O}_6$  grown by Dr. Jean-Yves Henry of the CENG laboratory in Grenoble, France. Its dimensions are approximately 50 microns  $\times$  100 microns  $\times$  0.8 mm. It weighs approximately 15 micrograms and contains about  $4 \times 10^{16}$  of the  $^7\text{Li}$  spins whose NMR signal is measured in our experiments. At the high magnetic fields used, the signal-to-noise ratio of the  $^7\text{Li}$  NMR spin echo signal is approximately 20 for a single transient.

It is of interest to speculate what are the limits on small sample size one could reach by following the approach we have taken and what could be accomplished using nanofabrication technology. With the materials and coil geometry shown in Fig. 6, one could obtain usable results from a sample with  $1 \times 10^{16}$   $^7\text{Li}$  spins and extend that to  $1 \times 10^{15}$   $^7\text{Li}$  spins by averaging 100 transients. By using still smaller commercial copper wire for the NMR coil, we estimate one could obtain useful signals with 1.5 micrograms of sample ( $4 \times 10^{15}$  spins) without averaging and 150 nanograms ( $4 \times 10^{14}$  spins) by averaging 100 transients. Finally, we estimate that with coils produced using advanced nanofabrication technology, one could investigate samples of this material weighing as little as 1.5 nanograms ( $4 \times 10^{12}$  spins) by averaging 1,000 transients.



**Figure 6.** NMR coil (thin wires), sample (dark material inside the coil), and lead wires to the rest of the NMR coil circuit. The NMR coil wire diameter is 25 microns (one thousandth of an inch).

**Acknowledgements.** The UCLA part of this work was supported by NSF Grant DMR-0072524.

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## \$8 Million NIH Grant Awarded for Structural Genomics “Virtual Laboratory”

**T**imothy Cross, NHMFL NMR Program Director and FSU professor of chemistry and biochemistry, along with a large team of researchers from across the country, has been awarded one of the largest grants ever received by FSU from the National Institutes of Health. The award is for \$8.1 million over five years and supports a broad, multi-institutional effort.

The project aims to develop technology for characterizing protein structures that exist in biological membranes. These proteins represent the communication between the outside and inside of all cells—even between the inside and outside of cellular compartments such as the cell nucleus. Because membrane proteins carry out such crucial functions, it is not surprising that drugs, which interfere with the activities of these proteins, can have profound physiological effects. It is anticipated that more than 90 percent of new drugs in the field of neuroscience alone will be targeted toward membrane proteins. One of the primary hurdles in Structural Genomics today is represented by approximately 30 percent of each genome that codes for membrane proteins.

This project is also significant because of the importance of its biological focus: It targets the membrane proteins of *Mycobacterium tuberculosis*, the agent responsible for tuberculosis. These membrane proteins represent many potential drug targets in this bacillus, which is ranked as the #1 cause of infectious disease mortality in the world. In fact, a third of the world’s population is infected with TB. Drug resistance is, today, a paramount concern in the treatment of tuberculosis—new drugs are needed and this project will facilitate the achievement of that goal.

Two core protein production facilities, one at the University of Virginia, led by Prof. Robert Nakamoto and one at Florida State University, plan to attempt the expression of a third of the nearly 1,200 open reading frames thought to code for the membrane proteins of *Mycobacterium tuberculosis*. High yields from overexpression and efficient purification protocols are anticipated from an estimated 30 percent of these ORFs.

Initial protein characterization will include solution NMR and FT-ICR in the National FT-ICR Laboratory headed by Prof. Alan Marshall at the NHMFL. Sample preparation screens will be conducted for more than one type of technology, such as three-dimensional crystallization for

X-ray diffraction in the laboratories of Prof. Michael Wiener (University of Virginia), Prof. Patrick Loll (MCP-Hahnemann University), Prof. Bing Jap (Lawrence Berkeley National Laboratory), and Prof. Cynthia Stauffacher (Purdue University), or two-dimensional crystallization in the laboratories of Prof. Kenneth Taylor (FSU), and Prof. Wah Chiu (Baylor College of Medicine).

Alternatively, newer technologies to the realm of membrane proteins, such as solid state NMR (Prof. Stanley Opella, University of California at San Diego, and Prof. Timothy Cross, FSU), and solution NMR (Profs. Frank Soennichsen and Charles Sanders, Case Western Reserve University) require uniformly aligned lipid bilayer preparations or isotropic solutions, respectively.

In addition to attempting the structural characterization by various technologies, there is an effort led by Michael Chapman (FSU) to combine structural restraints from all of these technologies in a holistic approach for structural characterization. This is based on the premise that for some proteins, no single technology will be able to provide the necessary data to achieve the structure, and consequently a combination of diverse structural restraints will be used.

For the first time, researchers with expertise in a wide range of technologies are collaborating to attack this very difficult scientific challenge. The team of 13 investigators from eight institutions in six states from California to Pennsylvania and Indiana to Texas represent a multidisciplinary group of X-ray and electron crystallographers, solution and solid state NMR spectroscopists, as well as a molecular biologist, and an FT-ICR mass spectroscopist. Internet and communication tools will be used to establish a “Center without Walls,” or Collaboratorium, so that the substantial expertise in each lab associated with membrane protein expression, purification, reconstitution, and sample preparation can be shared in a non-competitive environment. In the post-genomics era, the challenge to solve the membrane protein structures even for a single genome is daunting and consequently the competitive atmosphere between research groups could evaporate if the supply of many proteins was not rate limiting. Membrane proteins exist in a greasy membrane environment that makes it very difficult to prepare the samples for structural characterization. However, the combined expertise of this group provides the best hope to gain this vitally important information for medical science.

# Field Induced Superconductivity in a Magnetic Organic Conductor

L. Balicas, J.S. Brooks, K. Storr, and E. S. Choi, FSU/NHMFL  
S. Uji, National Research Institute for Metals, Tsukuba, Japan  
M. Tokumoto, Nanotechnology Research Institute, National Institute of Advanced Industrial Science and Technology (AIST), Japan  
H. Tanaka, and H. Kobayashi, Institute for Molecular Science, Japan  
A. Kobayashi, The University of Tokyo, Japan  
V. Barzykin, and L. P. Gor'kov, FSU/NHMFL

Physicists concluded long ago, and after several decades accumulating physical evidence, that superconductivity could not coexist with a magnetic ground state. It is thus easy to understand why the recent discovery of a series of ferromagnetic superconductors, like  $UGe_2$ ,<sup>1</sup>  $ZrZn_2$ ,<sup>2</sup> and the borocarbides superconductors,<sup>3</sup> has generated such excitement in the scientific community. Here, according to the conventional understanding of superconductivity, the magnetic moments are expected to play a role quite similar to that of an external magnetic field, which destroys superconductivity either by inducing strong diamagnetic currents or by breaking the spin-singlet state of the Cooper pair (the so-called Pauli pair breaking mechanism).

Given our previous understanding of magnetism and superconductivity, it was remarkable that S. Uji and co-authors<sup>4</sup> recently reported clear physical evidence for a magnetic field-induced superconducting state (FISC) in the  $\lambda$ -(BETS)<sub>2</sub>FeCl<sub>4</sub> layered organic conductor. This observation is particularly unusual since this compound, at zero field and below  $T_N = 8.5$  K, is an *insulating* antiferromagnet. According to Uji *et al.*, at low temperatures the FISC state appears only for fields above 18 T and applied along the conducting planes.<sup>4</sup> Motivated by these preliminary results, we recently mapped out the temperature-magnetic field phase diagram of the FISC state<sup>5</sup> by using AC electrical transport techniques in conjunction with the Hybrid magnet of the NHMFL in Tallahassee.

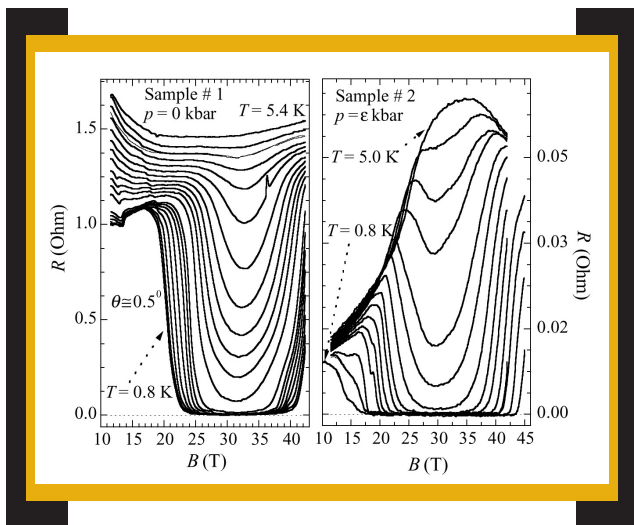
Our magnetic field dependent resistance of a  $\lambda$ -(BETS)<sub>2</sub>FeCl<sub>4</sub> single crystal, under atmospheric pressure, is shown in Fig. 1(a) for different temperatures. Here, the magnetic field  $B$  is applied along the in-plane  $c$ -axis. The main characteristic of the data is that between 18 and 41 T, the resistance of the material drops with decreasing temperature, reaching zero within experimental uncertainties below 2 K in a field range centered near 33 T. In the FISC state and at higher fields, the resistivity drops typically by 2 to 4 orders of magnitude,

putting it at or below the conductivity of copper, and beyond our ability to measure by standard AC lock-in methods. One of the main results of this study is the observation of reentrance toward the metallic state at a temperature-dependent critical field. Since the presence of  $Fe^{3+}$  magnetic moments, coexisting with the FISC state, would suggest a triplet superconducting state that, by definition, is not affected by the Pauli pair breaking mechanism. The fact that the FISC state is re-entrant to a metallic state above 41 T, excludes triplet pairing. Fig. 1(b) shows the resistance of a second single crystal as a function of  $B$  (up to 45 T) and for several temperatures. This sample is immersed 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.

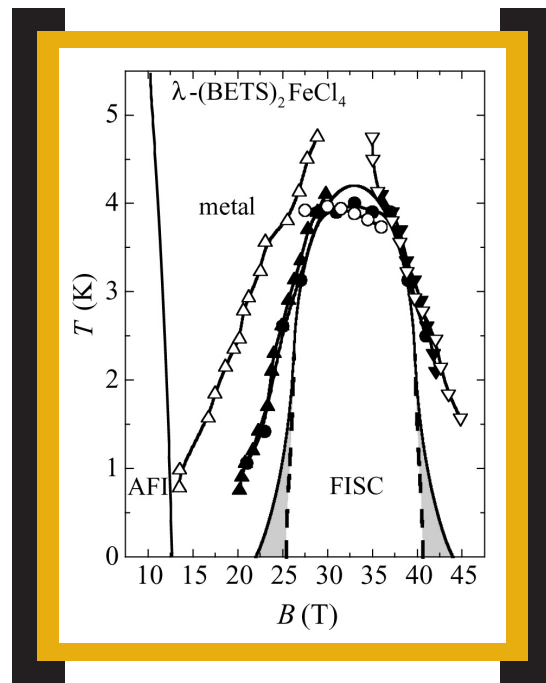
From the isothermal field scans of Fig. 1, we extracted the temperature dependence of the resistance at fixed values of field (not shown). From this set of data and from Fig. 1, we built the phase diagram shown in Fig. 2. Solid symbols describe the diagram of the FISC state at ambient pressure, while open symbols describe its diagram under  $p = \epsilon$  kbar ( $\gg 1$  bar). In both cases, the transition temperature towards the FISC state increases with  $B$  reaching a maximum value  $T_c = 4.2$  K at  $B^* \cong 33$  T for  $p = 1$  bar and  $B^* \cong 31.5$  T for  $p = \epsilon$  kbar.  $T_c$  decreases again for fields above  $B^*$  which makes the diagram symmetric around  $B^*$ . The solid continuous line is a fit of our data to the so-called Jaccarino-Peter effect.<sup>6</sup> The basic idea is that localized magnetic moments of  $Fe^{3+}$   $d$  electrons aligned along the external magnetic interact antiferromagnetically via exchange coupling, with the itinerant  $\pi$  electrons responsible for electrical conduction in this system. The total field  $\mu_B H_{\text{eff}}$  “felt” by the  $\pi$  electrons in an external field  $B_0$  is thus given by:

$$\mu_B H_{\text{eff}} = \mu_B B_0 + J\langle S \rangle \quad (1)$$

We can clearly see from Eq. (1) that, eventually, a strong enough external magnetic field  $B_0$  can *compensate* the internal exchange field  $J\langle S \rangle$  for  $J < 0$ , allowing the condensation of quasiparticles into a superconducting state if they were subjected to attractive interactions. For more details see References 5 through 7.



**Figure 1.** (a) Resistance  $R$  as a function of magnetic field  $B$ , applied along the in-plane  $c$ -axis ( $\pm 0.3$  degrees) of a  $\lambda$ -(BETS) $_2$ FeCl $_4$  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  $p = \epsilon$  kbar ( $\gg 1$  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 where the FISC state is observable.



**Figure 2.** Temperature-magnetic field phase diagram showing the AFI, metallic, and FISC states for a  $\lambda$ -(BETS) $_2$ FeCl $_4$  single crystal vs. in-plane magnetic field at ambient pressure (solid lines and symbols). Solid triangles indicate the middle point of the resistive transition as a function of  $B$  (from Fig. 1(a)), while solid circles indicate the middle point of the resistive transition as a function of  $T$ . Similarly, open triangles and open circles describe the  $T$  -  $B$  diagram of the FISC state under a very small amount of hydrostatic pressure (the AFI is displaced to fields  $B < 8$  T). The solid line is a theoretical fit (see text) to a second order phase transition toward the FISC phase while the dashed line indicates a first order transition from the inhomogeneous so-called LOFF state (after Larkin, Ovchinnikov, Ferrell and Fulde)<sup>7</sup> (shaded area) into the bulk superconducting state.

Consequently, in the present case, and in contrast to what is observed in conventional superconductors, magnetic moments are the essential ingredient for the stabilization of superconductivity at very high magnetic fields.

**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.

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## ATTENTION USERS

**Alex H. Lacerda**  
Director, NHMFL-Los Alamos User Program

As a national user facility, our role is to diversify, educate, and inform the scientific community. With that in mind, I bring to your attention the following article by Prof. G. Barbosa-Canovas, *et al.*, Biological Systems Engineering, Washington State University. Prof. Barbosa-Canovas and collaborators utilized the NHMFL–Los Alamos Facility to explore the effect of high magnetic fields (DC and pulsed) on the growth of microorganisms. The following article illustrates preliminary results. I would also like to take this opportunity to thank the authors for their contribution.

### Exposure of Microorganisms to High Intensity Magnetic Fields

**M. Fernanda San Martín**, Washington State University, Biological Systems Engineering  
**Federico Harte**, WSU, Biological Systems Engineering  
**Gustavo V. Barbosa-Cánovas**, WSU, Biological Systems Engineering  
**Barry G. Swanson**, WSU, Food Science and Human Nutrition

**F**ood processing by nonthermal technologies is currently under extensive research. The use of magnetic fields for microbial inactivation has been suggested as one such technology. Exposure to extremely-low frequency, low intensity magnetic fields has been reported to stimulate proliferation of microorganisms such as *Saccharomyces cerevisiae*<sup>1</sup> and *Pseudomonas stutzeri*.<sup>2</sup> However, microbial inactivation of 1.4, 3.6, and 3.5 logarithmic cycles of microorganisms such as *Streptococcus thermophilus*, *Saccharomyces cerevisiae*, and mold spores inoculated in food samples by exposure to single magnetic field pulses of 12, 40, and 7.5 T were reported by Hofmann.<sup>3</sup> No other results on microbial inactivation due to exposure to high intensity magnetic fields have been reported so far. During the late 1990s, a Washington State University research group showed no inactivation on *Escherichia coli*, *Saccharomyces cerevisiae*, or *Listeria innocua* exposed to 50 to 200 magnetic field pulses neither at 14, 16, nor 18 T.<sup>2,6</sup> The objective of the research at the

NHMFL–Los Alamos Facility was to determine whether exposure to higher intensity fields could achieve inactivation on *Escherichia coli*, *Listeria innocua*, *Bacillus subtilis*, and *Saccharomyces cerevisiae*.

The three bacterial strains and the yeast were incubated and grown as described by Harte, *et al.*<sup>5</sup> until early stationary phase was reached. Cryogenic vials containing the samples were stored in liquid nitrogen until used. After the magnetic field treatment, serial decimal dilutions of the original sample were done in 0.1% peptone solution. Enumeration of surviving organisms as colony forming units per milliliter (CFU/mL) was performed by pour plating method for the three bacteria and spread plating method for *Saccharomyces cerevisiae*.

Cryogenic vials containing the samples were subjected to one of two types of magnetic field treatments at the NHMFL at Los Alamos. Exposure to a static magnetic field of *Escherichia coli* and *Saccharomyces cerevisiae* was done using a 20 T superconducting magnet. The cryogenic vials were attached to a sample positioner (Model No. 1639-8; Cryo Industries of America, Manchester, NH) with a copper mount, a 25  $\Omega$  heater, and a PT-100 temperature sensor. Samples were kept at either 4 or -20°C throughout the treatment. The come-up (0 to 18 T), holding (18 T), and come-down (18 to 0 T) times were 52, 60, and 52 min., respectively.

Exposure of the microorganisms to a pulsed magnetic field was done using a 50 T short pulse magnet. In this case, however, due to the small magnet bore, the sample was attached to a G-10 rod, placed at the center of the magnet, and kept frozen throughout the treatment. Samples were exposed to 1, 2, or 3 magnetic field pulses. Average pulse width was approximately 20 ms and the time elapsed between consecutive pulses was approximately 25 min. In both types of treatment, control samples were obtained by following the same protocol, except that the magnetic field was not turned on.

No significant differences ( $\alpha=0.05$ ) between control and treated samples were observed in log CFU for *Escherichia coli* and *Saccharomyces cerevisiae* exposed to an 18 T static magnetic field neither at 4°C nor at -20°C.

ANOVA analysis showed no effect of the pulsed magnetic field on *Bacillus subtilis* ( $\alpha=0.2291$ ), *Listeria innocua* ( $\alpha=0.4967$ ), or *Saccharomyces cerevisiae* ( $\alpha=0.8195$ ). In the case of *Escherichia coli*, no significant differences were observed either in VRB agar ( $\alpha=0.7869$ ) or nutrient agar ( $\alpha=0.3313$ ).

Our negative results are similar to those reported by Caubet<sup>6</sup> for *Listeria innocua*, *Escherichia coli*, and *Bacillus cereus* exposed to 1 to 6 pulses of a 7 T magnetic field. He observed, however, that spores of the mold *Penicillium cyclopium* exposed to the pulsed magnetic field modified the aspect of the colonies.

Consistent negative results were observed for all the microorganisms studied, regardless of the type of magnetic field used. So far, static or pulsed magnetic fields such as those used in this work, are not a feasible technology for food preservation due to failure to inactivate microorganisms.

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<sup>4</sup> San Martín, M.F., Harte, F., Lelieveld, H., Barbosa-Cánovas, G.V., and Swanson, B.G., Inactivation Effect of an 18 T Pulsed Magnetic Field Combined with Other Technologies on *Escherichia coli*, *Innovative Food Science and Emerging Technologies* (In press).

<sup>5</sup> Harte, F., San Martín, M.F., Lacerda, A.H., Lelieveld, H.L.M., Swanson, B.G., and Barbosa-Cánovas, G.V., Potential Use of 18 T Static and Pulsed Magnetic Fields on *Escherichia coli* and *Saccharomyces cerevisiae*, *Journal of Food Processing and Preservation*, **25**, 223-235 (2001).

<sup>6</sup> Caubet, R., Effets des champs magnétiques pulsés à haute densité de flux sur les micro-organismes, Proceedings of the Technological Training in the Food Industry, Conservation for Tomorrow, 2<sup>nd</sup> Edition, Bordeaux Pessac, France (1999).



## NHMFL Users' Committee November Meeting

One of the most important NHMFL advisory bodies met at the NHMFL in Tallahassee on November 2 and 3. Chaired by Bill Halperin, Northwestern University Department of Physics & Astronomy, the NHMFL Users' Committee provides guidance on the development and use of NHMFL facilities and services in support of the laboratory's multidisciplinary user community.

When NHMFL directors and committee members meet each year, the dialogue inevitably sheds light on important user priorities and issues. These discussions assist management with planning, setting objectives, and responding to user needs. The agenda for the November meeting suggests current topics:

- NHMFL Goals for 2001-2005
- Magnet Discussions, including the availability, repairs and upgrades to various systems, as well as new designs
- High B/T Facility, including plans for increasing the number of users who can be accommodated at temperatures between 1 and 5 mK and fields up to about 10 T
- Instrumentation Discussions, with attention to dilution refrigerators in the DC Field Facility, sources and detectors in the EMR Facility, and other areas
- Facilitating new collaborations between users and NHMFL staff and faculty, with the goal of seeking additional funds for developing new experimental capabilities
- Spring 2002 NSF Performance Review Preparations.

The Users' Committee meets at least annually, with the most recent previous meeting November 17-18, 2000 at Los Alamos. Several key issues discussed at that meeting were the resources devoted to providing dilution refrigerator temperatures in the resistive and Hybrid magnets, committee representation and meetings (including perhaps topical ones), and enhanced integration of CIMAR issues into committee business. After considerable discussion, the committee also made a specific recommendation for a split-coil magnet:

“Based on feedback from a number of users, a split-coil magnet with a field of 20 to 21 T would be acceptable if it possessed what they believed to be the most useful magnet configuration, as described below. The field would be horizontal with a 32-mm vertical access between the coils to allow insertion of the Lab's existing cryostats with that tail diameter, and providing convenient rotation of the cryostat and sample about a vertical axis. The magnet's 32-mm horizontal magnet bore would provide a horizontal access along both 90- and 180-degree paths and could be used for focussed light beams, laser beams, light detection, and telescopic viewing. The *combination* of vertical access and provision for many kinds of magneto-optics experiments (including Raman scattering and where fiber optics are precluded) is significantly more versatile than the choice of a magneto-optics-only vertical-bore magnet with small (say, 10-mm diameter) horizontal ports for optical access.”

Future issues of *NHMFL Reports* will report the ongoing discussions on the split-coil magnet, as well as other topics of interest to users. Users are also encouraged to offer comments and suggestions to Bill Halperin ([w-halperin@nwu.edu](mailto:w-halperin@nwu.edu), 847-491-3686), any Users' Committee member (see next page), or the director of the NHMFL facilities in which they are interested.

## NHMFL Users' Committee

*The laboratory would like to recognize and thank all members of the NHMFL Users' Committee, who are elected by fellow users and serve voluntarily.*

**Ward P. Beyermann**

University of California, Riverside  
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**Stuart Brown**

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Department of Physics

**Michelle Buchanan**

Oak Ridge National Laboratory  
Analytical Chemistry Division

**Nathanael Fortune**

Smith College  
Department of Physics

**Roy Goodrich**

Louisiana State University  
Department of Physics & Astronomy

**Bill Halperin, Committee Chair**

Northwestern University  
Department of Physics & Astronomy

**Steve Hill**

University of Florida  
Department of Physics

**Martin Kushmerick**

University of Washington  
Radiology, Physiology & Biophysics, Bioengineering

**Jim Prestegard**

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**Marion Thurnauer**

Argonne National Laboratory  
Chemistry Division

**Tom Timusk**

MacMaster University  
Department of Physics & Astronomy

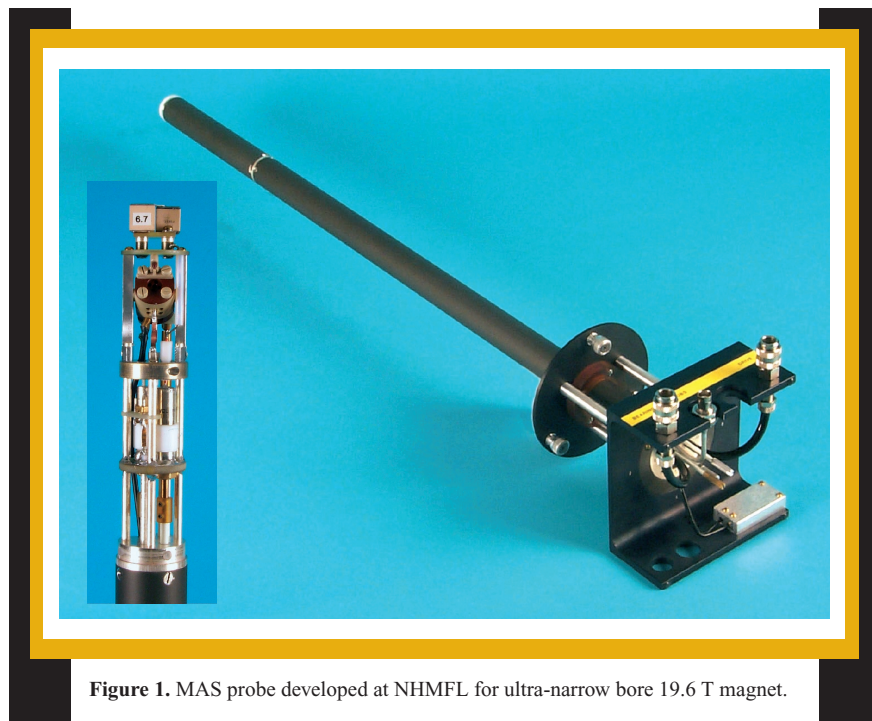
## Ultra-Narrow Bore Magic Angle Spinning Probes Benefit Applications of Solid State NMR at High Field

**Peter Gor'kov, Zhehong Gan, and William W. Brey,**  
NHMFL

**T**wo new solid state NMR probes have been developed at the NHMFL that open up a range of solid state NMR experiments for use at high magnetic fields. The probes are mainly used with the superconductive 19.6 T (corresponding to a  $^1\text{H}$  resonance of 830 MHz) magnet, but can also be used in resistive magnets and the 45 T Hybrid when higher fields are necessary. High resolution solid state NMR requires rapid sample spinning at the so-called magic-angle ( $54.74^\circ$ ) to the

magnetic field to average out the line broadening effect from chemical shift anisotropy, dipolar and quadrupolar couplings. We have obtained a magic-angle spinning (MAS) stator and rotors from our collaborator Dr. Ago Samoson at the National Institute of Chemical and Biophysics of Estonia who holds the world record on sample spinning rate (50 kHz). The bore space for the probes is limited to 28.9 mm inside the thin room temperature shim coils (Doty Scientific, Inc.) in the 31 mm magnet warm bore. The stators were tailored to fit inside the ultra-narrow bore probes and 6 kV variable capacitors with Teflon dielectric (Voltronics) were

used in the rf circuit to deliver a sufficiently high rf field. Both probes can be returned to virtually any nucleus of interest by replacing the high voltage chip capacitors insert attached to the top of the probe head (Fig. 1).



**Figure 1.** MAS probe developed at NHMFL for ultra-narrow bore 19.6 T magnet.

The first probe is designed for low-gamma nuclei ( $^{99}\text{Ru}$ ,  $^{39}\text{K}$ ,  $^{95}\text{Mo}$ ,  $^{109}\text{Ag}$ ,  $^{49}\text{Ti}$ ,  $^{25}\text{Mg}$ ,  $^{67}\text{Zn}$ ,  $^{33}\text{S}$ , and etc.) that can be found in a variety of solid materials such as glasses, zeolites, or semiconductors. The stator accepts standard 4 mm MAS rotors from Bruker Instruments capable of spinning up to 10 kHz. Large sample volume and the high magnetic field provide much needed sensitivity for solid state NMR of low-gamma nuclei. The high magnetic field also reduces the second-order quadrupolar line broadening and rf ring-down time becomes shorter at higher resonance frequencies.

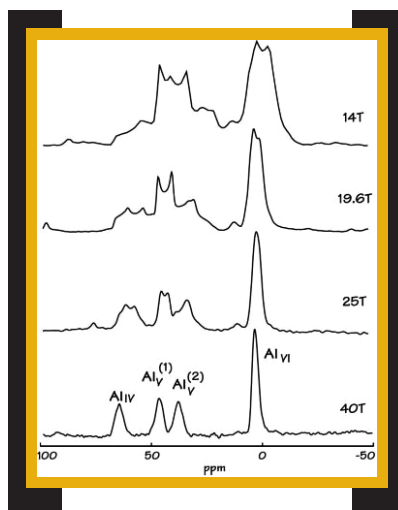
The second probe is designed for its fast spinning speed and is particularly useful for sensitive quadrupolar nuclei such as  $^7\text{Li}$ ,  $^{45}\text{Sc}$ ,  $^{93}\text{Nb}$ ,  $^{27}\text{Al}$ ,  $^{23}\text{Na}$ , and enriched  $^{17}\text{O}$  with large quadrupolar coupling and chemical shift anisotropy. Fast spinning speed avoids the overlap of spinning sidebands. The probe uses a 2 mm rotor capable of spinning up to 50 kHz, although 40 kHz is recommended for

routine experiments with long data acquisition time. Fig. 2 shows the field dependent spectra of an  $^{27}\text{Al}$  crystalline compound illustrating the improvement in spectral resolution by higher magnetic fields and fast MAS. The spectrum acquired at 40 T

from the Hybrid magnet shows the best spectral resolution, as the second-order quadrupolar broadening becomes virtually zero at 40 T. The spectra demonstrate that high resolution NMR is possible with the less homogeneous and stable magnetic field of a resistive magnet.

Both probes are used by external visitors such as L. Butler (LSU), C. Grey (SUNY), G. Hoatson (William & Mary), F. Taulelle (Strasbourg), L. Alemany (Rice), D. Massiot (CNRS, France), S. Martin (ISU), O. Han (Seoul), A. Samosan (Estonia), Ding Ma (DICP, China), A. Stigman (FSU), C.

Lee (Korea), as well as by NHMFL scientists, including Riqiang Fu, Zhehong Gan, Zhiru Ma, and Bretta King. Scientists who might want to use these probes should contact Zhehong Gan at [gan@magnet.fsu.edu](mailto:gan@magnet.fsu.edu) or see the Web site at <http://nmr.magnet.fsu.edu>.



**Figure 2.** The  $^{27}\text{Al}$  spectra of  $^9\text{Al}_2\text{O}_3+^2\text{B}_2\text{O}_3$  obtained at different field strengths, including one from NHMFL's 40 T Hybrid magnet.

# High Capacity Production of > 40% Spin Polarized Xenon-129 for NMR and MRI Applications at the NHMFL

Anthony L. Zook and Clifford R. Bowers, UF, Chemistry

The NHMFL and the University of Florida recently supported the development of a hyperpolarized noble gas generator at UF. We are pleased to report that construction of this facility is now complete, and that its performance for high capacity hyperpolarized  $^{129}\text{Xe}$  production is apparently better than any other laser diode array (LDA) based system reported in the literature. Here, we show the results of the performance tests, and to demonstrate the capability that is now available, several images obtained using the new polarizer will be presented. It is hoped that these promising results will stimulate interest in hyperpolarized noble gas applications that may now be pursued at the NHMFL.

Non-equilibrium methods for enhancing the nuclear spin polarization, and hence the signal-to-noise ratio in NMR experiments, are making a significant impact in certain types of NMR and MRI applications. These enhancement techniques include: dynamic nuclear polarization,<sup>1,2</sup> parahydrogen induced nuclear polarization,<sup>3,4</sup> optical pumping in solids,<sup>5,6,7</sup> and spin-exchange optical pumping of  $^{129}\text{Xe}$  and  $^3\text{He}$ .<sup>8,9</sup> Spin-exchange optical pumping, proven to be one of the more generally applicable methods, involves polarization of the hyperfine sublevels by repeated optical excitation of an alkali metal atom with circularly polarized light. Spin angular momentum exchange collisions between the alkali atom and  $^3\text{He}$  or  $^{129}\text{Xe}$  leads to the accumulation of a large nonequilibrium nuclear polarization, referred to as *hyperpolarization*, of the noble gas. While spin exchange optical pumping at low magnetic field had been known since the 1960's, it was not until the demonstration of Raftery *et al.*<sup>9</sup> that the hyperpolarized noble gas can be separated from the alkali metal and employed in enhanced

sensitivity high field NMR. This led Albert and co-workers<sup>10</sup> to demonstrate that hyperpolarized  $^{129}\text{Xe}$  could be used to obtain *in vivo* magnetic resonance images. Later,  $^3\text{He}$  was applied to the imaging of human lungs,<sup>11</sup> and now the technology of spin exchange optical pumping has even been commercialized<sup>12</sup> due to its excellent potential as a clinical diagnostic. In recent years, the number of spectroscopic applications of hyperpolarized  $^{129}\text{Xe}$  has also grown rapidly.

The key parameters affecting the maximum attainable nuclear polarization of  $^{129}\text{Xe}$  include the Rb vapor density (directly related to the pumping cell temperature), the pumping gas composition and pressure, the laser linewidth, and the laser power.<sup>13</sup> The parameters affecting the maximum attainable nuclear polarization of  $^{129}\text{Xe}$  include the Rb vapor density (directly related to the pumping cell temperature), the pumping gas composition (Xe,  $\text{N}_2$ , and  $^4\text{He}$ ), the pumping time or flow rate, the pumping cell wall coating, the pumping field, and the laser linewidth and power.<sup>13</sup> Two types of near infrared laser systems are currently in common use for Rb-Xe spin exchange optical pumping: the titanium sapphire laser and the LDA. While the power available from the titanium sapphire ring laser is typically only a few watts, the laser line width is much narrower than the Doppler linewidth. The laser power is, therefore, efficiently absorbed by a suitably dense Rb vapor, and for this reason, the titanium sapphire laser is well suited to the production of small quantities (c.a.  $10\text{ cm}^3$ ) of >50% polarized  $^{129}\text{Xe}$  at pressures up to about 10 to 50 mbar.<sup>14</sup> With the advent of the LDA, much higher optical power tunable to the Rb  $^1\text{D}$  absorption line at a wavelength of 794.8 nm is available, but the linewidth of the output is typically on the order of 1000 GHz, a value greatly exceeding the  $\approx 1$  GHz Doppler broadening of atomic Rb (at  $\approx 100^\circ\text{C}$ ). For spin exchange optical pumping of  $^{129}\text{Xe}$ ,

studies have shown that a 98%  $^4\text{He}$ , 1%  $\text{N}_2$ , and 1%  $^{129}\text{Xe}$  gas composition provides the necessary pressure broadening for efficient optical absorption, reduction of spin-rotation relaxation of the  $^{129}\text{Xe}$ , and quenching of the alkali-atom excited state to reduce depolarization due to radiation trapping.<sup>13</sup> With the greater optical power available with LDAs, much higher alkali metal atom densities can be excited, and the rate of nuclear polarization of the  $^{129}\text{Xe}$  gas is proportionally greater due to the increased rate of Xe-Rb spin exchange collisions. The higher rate of polarization enables a flow mode of operation whereby the Xe/ $\text{N}_2$ /He gas mixture passes through the cell continuously. The LDA-based polarized gas generator is, therefore, advantageous in applications requiring large quantities of polarized gas such as magnetic resonance imaging,<sup>15</sup> enhanced NMR spectroscopy using hyperpolarized liquid  $^{129}\text{Xe}$ ,<sup>16,19</sup> and phase-cycled or two-dimensional NMR experiments.

**Table 1.** Survey of  $^{129}\text{Xe}$  polarizer data reported in the literature.

Research Group	Institution	LDA Power (W)	P, %	Xe Flow (sccm)	Production Factor*	Ref.
Appelt	Research Centre Jülich, Germany	50	20	2@26%	40	17
Chupp	U. Michigan	30	3.5	-	-	18
Conradi/Saam	Washington U.	40	5	2.4@26%	12	19
Happer	Princeton U.	50	5	6@71%	30	20
		100	18	3.2@71%	57.2	16
MITI	U. Virginia (15)	120	7	16.6@26%	117	21
	Duke (14)	120	10	12.2 @82%	122	22
	Duram, N.C. (4)	100	21	3	63	12
Pietrass	New Mexico Tech	45	1.5	37@26%	55.5	23
Pines	UC Berkeley	100	10	3@26%	30	24
Rafferty	Purdue	100	4.5	2@26%	9	25
Ripmeester	NRC, Canada	30	6	2@26%	12	26
Segebarth	Université Joseph Fourier, France	150	8	16@26%	128	27
Walsworth	Harvard U.	15	5	-	-	15

Table 1 presents a survey of the  $^{129}\text{Xe}$  optical pumping systems described in the recent literature according to LDA power,  $^{129}\text{Xe}$  nuclear spin polarization produced, and Xe flow rate. Two relevant criteria by which the performance of a polarized gas generator can be evaluated are the maximum polarization achieved and the nuclear magnetization production rate, a quantity proportional to the product of the nuclear polarization and the Xe flow rate. There is a wide variation in the reported values of the nuclear polarization (or signal enhancement factor) and

production factor, but in summary, the polarization achieved in previous LDA systems is substantially lower than the value achieved in titanium sapphire systems.

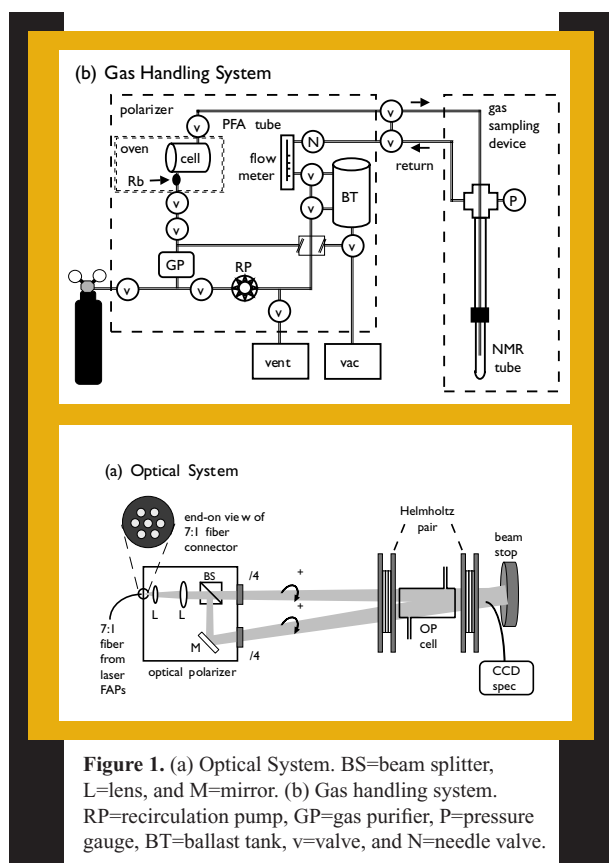
Ideally, a hyperpolarized noble gas generator would simultaneously deliver both high capacity and the highest possible polarization. Previous work suggests that higher polarization and high flow rates may potentially be obtained by increasing the rubidium density and absorbed laser power.<sup>17</sup> However, an experimental investigation to determine if this is so requires a higher power LDA system than has been previously available. The NHFML-UF polarized gas generator employs a 210 W LDA, to our knowledge, the highest optical power yet to be employed in  $^{129}\text{Xe}$  spin exchange optical pumping.

A schematic of the NHFML-UF polarizer is shown in Fig. 1. The laser system consists of seven fiber coupled LDA units, each with an output of 30 W at 795 nm, and with a total combined line width of 1.7 nm (~800 GHz). The individual fiber outputs are combined into a single fiber terminated by a water-cooled output connector. The individual fibers are embedded in a high-thermal conductivity epoxy and are anti-reflection coated to minimize heating at the connector. The unpolarized beam emerging from the optical fiber passes through a collimating lens and then is split into horizontally and linearly polarized beams by a beam-splitting cube. The two beams are circularly

polarized with the same sense of helicity by two separate quarter-wave plates and superimposed at a distance of 80 cm using an adjustable mirror mount. This optical polarizer arrangement permits the full power of the laser system to be circularly polarized even though the output of the LDA system is unpolarized. The 5x12 cm cylindrical optical pumping cell has an inlet and outlet to facilitate continuous flow generation of hyperpolarized  $^{129}\text{Xe}$ . PFA tubing with non-magnetic fittings connects the pumping cell to the NMR probe. After passing through the probe, the gas can either be vented (in applications where it may become contaminated)

or recirculated by a magnetically coupled pump. The flow rate is monitored and adjusted by a needle valve on the return line.

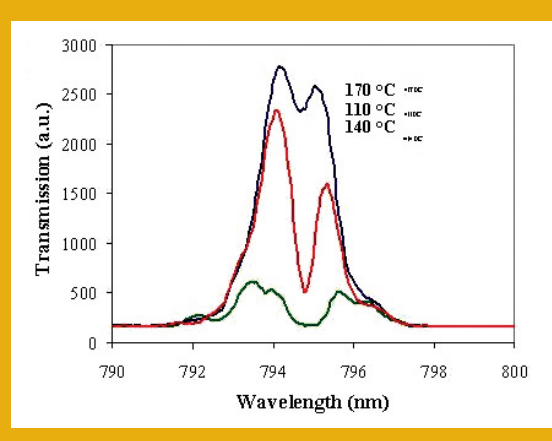
As noted above, the rate of optical pumping of the rubidium vapor depends on the fraction of



the laser power absorbed by the vapor. The main parameters affecting the absorption are (1) the buffer gas pressure and (2) the rubidium density that is determined by the optical pumping cell “conditioning” and the temperature. To obtain optimal absorption a fiber coupled CCD optical spectrometer monitors the transmission of the laser light through the pumping cell. For example, Fig. 2a presents the optical transmission as a function of the pumping cell temperature at a gas pressure of 3400 Torr. At 100 °C, the Rb density is too low and the light is not absorbed, while at 170 °C the density is too high, and in this case the pumping light does not reach the back of the cell. The intermediate temperature of 140 °C represents the optimal situation where the Rb will be maximally polarized over the entire pumping cell volume.

The performance characteristics of the UF/NHMFL hyperpolarized noble gas generator are presented in Table 2. The highest  $^{129}\text{Xe}$  polarization obtained from this system was 46.2% in batch mode and 40.% in continuous flow mode.<sup>28</sup> It appears that these are the highest levels of polarization to be achieved in an LDA-based polarizer, and furthermore, the rate of magnetization production is also higher than any other literature report at any flow rate. These performance characteristics are attributed to the high laser power, narrow laser line width, and efficient polarizer design.

Finally, Fig. 3 demonstrates the capability of performing magnetic resonance imaging experiments on  $^{129}\text{Xe}$  gas and  $^{129}\text{Xe}$  liquid using the UF/NHMFL hyperpolarized gas generator. In each case, a cross section of the cylindrical NMR tube was imaged with a 10 mm high resolution probe using a small pulse angle gradient echo filtered imaging (GEFI) pulse sequence. To obtain the  $^{129}\text{Xe}$  liquid image, the hyperpolarized gas was first condensed as a solid at a temperature several degrees below the melting point. The image was obtained after raising the probe temperature to form liquid Xe in the sample tube. In principle, magnetic resonance imaging with liquid Xe at this level of polarization should yield higher resolution than proton imaging of liquid water.

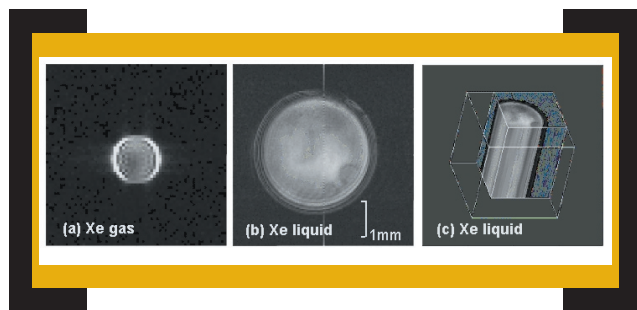


**Figure 2.** Optical transmission of pumping cell as a function of cell temperature.

**Table 2.** UF/NHMFL polarizer characteristics.

Laser Power (W)	P, %	Xe:N <sub>2</sub> :He Composition	Xe Flow Rate (sccm)	Production Factor
210	40	1:1:98	4.8@86%	192
210	23	2:2:96	7.44@26%	171
210	12	5:5:90	18.6@26%	223

In summary, the laser-polarized noble gas generator incorporating a 210 W laser diode array has been constructed and tested, and a nuclear polarization of 46.2% of <sup>129</sup>Xe has been obtained. The <sup>129</sup>Xe nuclear polarization obtained with this new system easily competes with Ti:sapphire based polarizers, but the capacity of the UF/NHMFL LDA system, in terms of the rate of <sup>129</sup>Xe magnetization produced, is far greater. The ability to produce highly polarized <sup>129</sup>Xe at rates in excess of 1 liter-atm/hour will facilitate superior results in 1D and 2D NMR as well as MRI applications. Since the NMR signal obtained from liquid <sup>129</sup>Xe polarized to > 40% exceeds the proton signal of liquid H<sub>2</sub>O, improved resolution in imaging applications should be possible. Potential spectroscopic applications include protein studies, surface NMR, and polarization transfer enhancement on surfaces. In conclusion, we hope that the promising initial results obtained with the NHMFL-UF polarizer will stimulate researchers of the NHMFL in-house and user community to consider experiments using this new instrument. Potential users should contact Russ Bowers, Steve Blackband, Anthony Brennan, or Steve Gibbs.



**Figure 3.** (a) Axial non-slice-selective gradient echo image of polarized xenon gas flow. The increased intensity near the walls of tubing is indicative of wall restricted diffusion of the gas. (b) Single scan gradient echo image of polarized liquid xenon at 186 K. Total experiment time was 20 seconds. Outer ringing of image was caused by clipping of the FID during acquisition. (c) 3D single scan, multiple gradient echo image (5 slices) of polarized liquid xenon in a cylindrical sample cell at 186 K, acquired with a tip angle 4°. The total experiment time was 20 seconds.

**Acknowledgements:** The authors gratefully acknowledge receiving advice and assistance from Stephen Blackband, Sam Grant, and Pete Thelwall. Anthony Zook was supported as a graduate research assistant by NIH resource grant P41 RR16105 (S. Blackband, PI). Funding for this project was provided by the University of Florida and the NHMFL In-House Research Program (C. Bowers, S. Blackband, A. Brennan, S. Gibbs, and S. Smith).

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## A New School Year with New Challenges for CIRL

**A**s the 2001-2002 school year begins, educators at the Center for Integrating Research and Learning focus more attention on outreach to students and teachers. Coming on the heels of a successful and rewarding summer with undergraduates and teachers, we are energized and excited to be offering new and innovative programs to schools statewide. The new school year brings with it exciting challenges as Florida prepares its students for standardized testing in science. CIRL is preparing to take a leadership role in helping students and teachers meet this challenge while offering quality science education.

CIRL kicked off the new academic year with our Fall Ambassador dinner that was co-hosted by RET participants, showcasing educational materials they created during the summer mentorship. One-hundred-and-three teachers, administrators, and community group participants represent nine counties in North Florida and South Georgia, 56 public schools, 10 private schools and 4 charter schools. The ambassadors serve as our conduit through which information about educational outreach and programs reach the wider educational community.

CIRL has been working with local school districts to begin “The NHMFL Classroom” experience for fourth grade students and their teachers. We have targeted 9 underserved local schools and invited their approximately 800 fourth grade students for a day of hands-on science and technology at the NHMFL. Teachers return to their classrooms with materials to continue the experience with support from Center staff when necessary. We are looking forward to our first five classes in November.

In addition to special outreach experiences, CIRL has expanded the outreach that accompanies the tour experience with a new “Observing the Unobservable” outreach that has an ICR/NMR focus. Materials have been re-tooled to address standardized test benchmarks and state standards, and we encourage teachers to

bring students for more than one visit. The ICR/NMR outreach includes a new tour of those areas of the laboratory with demonstrations provided to interpret the equipment and research for students fourth grade and higher. This outreach targets those students who have visited the laboratory before and for whom we can extend the experience to include more complex areas of the NHMFL.

The Center continues to provide statewide training for the *Science, Tobacco & You* curriculum resource. With a new contract from the Florida Department of Health, the Center will conduct 5 regional sessions for 250 teachers primarily trained and 250 teachers secondarily trained by participants in the train-the-trainer workshops. To date, the Center has conducted 61 training sessions, with 2,780 teachers in five states. We continue our trainings in other states throughout the nation.

Recognizing the need to address the underrepresentation of women in the physical sciences, the Center has created a partnership with WIMSE (Women in Math, Science, and Engineering), a newly formed group on the Florida State University campus. The lab will be participating in seminars and lectures focusing on opportunities for women in the sciences.

In addition to ongoing programs, the NHMFL continues to support mentorship programs for middle school and high school students. At the present time, 11 students are serving internships and externships with scientists and researchers at the NHMFL, and we anticipate more than 20 middle school students will work in mentorships during the spring semester.

The Center continues to be optimistic about the state of science education and is working diligently to provide quality professional development workshops for science teachers as well as meaningful experiences for students and the general public. We look forward to our Open House on March 2, 2002, as well as to a full slate of educational activities.



### AMRIS Progress, October 2001.

This report was jointly prepared by Steve Blackband and Art Edison. We are both delighted (Steve) and terrified (Art) by the fact that we just passed the position of AMRIS Director from Steve to Art in July, 2001. Steve was the driving force behind establishing the infrastructure of the AMRIS facility. Now that the installation phase is essentially complete, Art's main job for the next two years will be to help develop AMRIS into a world-class user facility. Please contact Art ([art@ascaris.ufbi.ufl.edu](mailto:art@ascaris.ufbi.ufl.edu)) from now on with respect to AMRIS issues.

**750 MHz Magnet for NMR/MRI:** The stability of the 750 MHz, 89 mm magnet for spectroscopy is still in question. The return of a Bruker engineer in July was partially successful in addressing drift issues; however, for now it appears that good data over long time periods will require the use of periodic gradient shimming. Bruker personnel are returning in October for continuing evaluations. New birdcage geometry rf coils arrived, and thus we now have a complete set of imaging coils (5, 10, 15, 20, 25, and 30 mm diameter) to support the users.

**11.7 T, 40 cm Magnet for MRI:** The ice appears to have been successfully removed from the magnet, leading recently to a re-cooling of the system. Glass ports on the top of the magnet now allow visual access and so far, a few weeks after re-cooling, no ice buildup is evident. Consequently, the magnet is being re-energized and presently stands at 9.4 T temperature stabilizing. In the meantime, another pump burnt out and we are revisiting with Magnex whether in the longer term a larger pump should be incorporated. When the pump issue is resolved, we will complete re-energization to 11.7 T. In the meantime, the magnet filling efficiency is still poor (~40%). At present, Magnex suspects the filling tube equipment. Fingers crossed.

**New Funding and Magnet System:** Dr. Bill Luttge has recently been awarded a U.S. Health and Human Services instrumentation grant (\$960,000) toward the purchase of a human scanner dedicated for research. The grant was entitled "Magnetic Resonance Instrumentation for a National Center for Human Brain Functional Imaging Technology and Image-Guided Surgery (CHBFIT) within the McKnight Brain Institute of the University of Florida (MBI-UF)"—congratulations Bill. This new system, a Siemens Allegra 3 T MR scanner, is a very compact design (only 1.25 m long) designed specifically for human head imaging, though we also intend to use it for animal studies. The new system has been ordered and will be situated on the ground floor of the MBI by converting one of the present ground floor conference rooms. Renovations permitting, we hope to have the system in place by the end of the year. This new magnet will complement the animal instruments in AMRIS, facilitating investigations ranging from molecules to man. The system will be part of AMRIS and run as a national user facility in concert with our other magnet systems. We are presently in the process of interviewing potential faculty hires in support of the system.

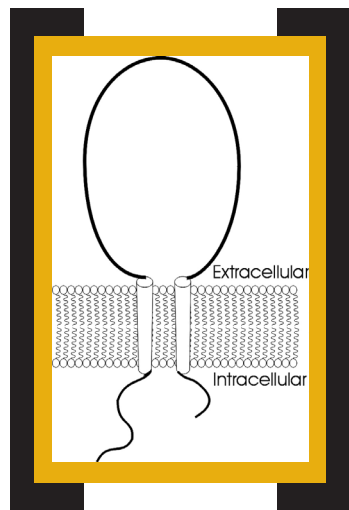
### AMRIS Research Report #8 NMR Structure of a Neuropeptide Bound to Its Receptor

**Arthur S. Edison, Steve Thomas, and Cherian Zachariah**  
NHMFL/UF, Biochemistry & Molecular Biology

One of the greatest challenges in structural biochemistry is to learn how chemical signals are propagated from one cell to another. This necessarily involves learning about receptor proteins associated with membranes that transmit information from the outside to the inside of the

cell. Some receptor proteins, such as G-protein-coupled receptors, are almost entirely buried in the membrane. Researchers such as Dr. Timothy Cross at the NHMFL are developing important new NMR methods to obtain high-resolution structural data from membrane associated proteins. Other receptor proteins have large regions of “extracellular domain” that are not associated with the membrane but are attached to the membrane by one or two transmembrane  $\alpha$ -helices. In many cases, including the one described below, the extracellular domain is the region that interacts with the external chemical ligands, and thus can be studied independently of the membrane-spanning regions.

Our laboratory studies a family of neuropeptides called FMRFamide-like peptides (FLPs). The first identified FLP receptor was the FMRFamide-gated sodium channel (FaNaCh) (REF). No 3D structure for FaNaCh or any of its relatives is known, but amino acid sequence analysis suggests the schematic view shown in Fig. 1. FaNaCh allows sodium to flow into a cell upon activation by the tetrapeptide Phe-Met-Arg-Phe-NH<sub>2</sub> (FMRFamide). The precise function of FaNaCh is not yet known, but several studies have shown that FMRFamide is cardioexcitatory and modulates pain in mammalian brains. The goal of this study is to obtain a structure of FMRFamide bound to FaNaCh, with the long-term hope that this structure will provide a starting point to design molecules to specifically block the peptide-

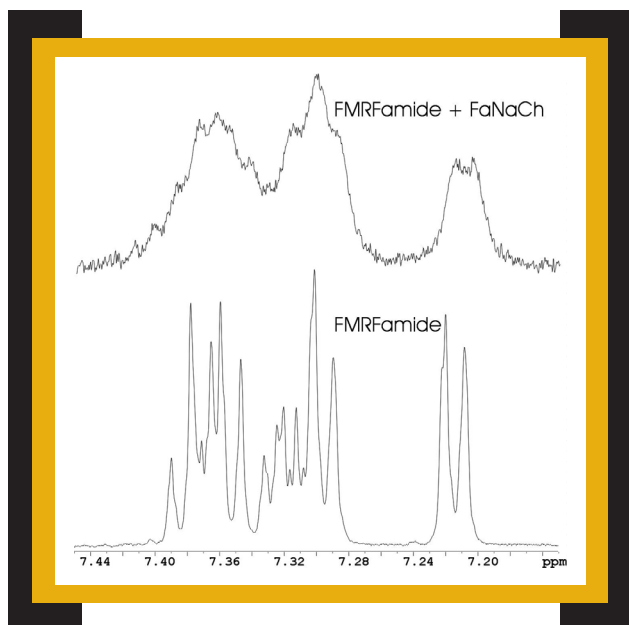


**Figure 1.** Schematic representation of the FMRFamide-gated sodium channel. The N- and C-terminal ends of the protein contain membrane-spanning domains, which are shown as vertical cylinders. The extracellular peptide-binding domain (the large oval) is approximately 450 amino acids and contains 14 highly conserved cysteines. The data in Fig. 2 and 3 were generated with just the extracellular domain expressed in *E. coli*.

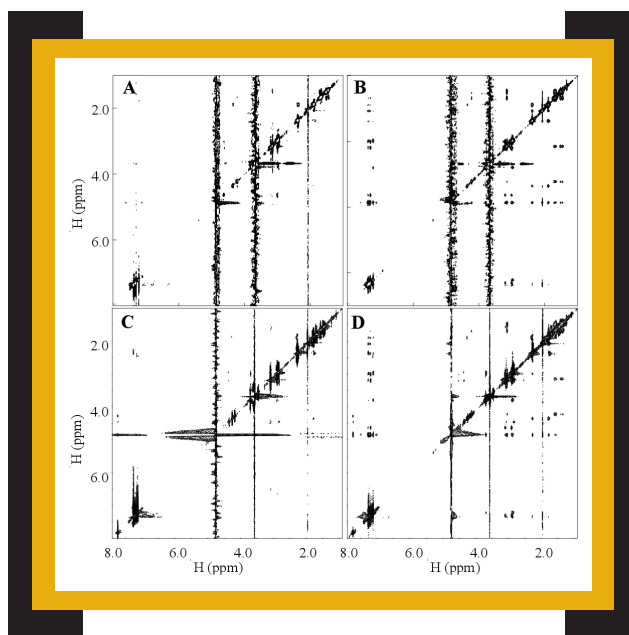
receptor interaction, and help us better learn the function of FaNaCh.

We produced the extracellular portion of FaNaCh (Fig. 1) in bacteria and refolded the protein through dialysis. This was extremely challenging and took well over a year of focused effort, because the extracellular domain contains 14 cysteine residues that need to form correct disulfide linkages in the final structure. The effect of the refolded extracellular domain on the 1D <sup>1</sup>H NMR spectrum of FMRFamide is shown in Fig. 2. The broadening of resonances observed in Fig. 2 shows that the peptide is interacting with the receptor. This broadening is not observed with several similar peptides that do activate the receptor, including Phe-Met-Arg-Phe (no amide). Nuclear Overhauser Effect (NOE) spectroscopy allows distances between protons to be estimated, but the NOE signal goes from positive to negative (and thus disappears) for molecules about the size of a tetrapeptide. Fig. 3 shows NOESY spectra of FMRFamide alone, FMRFamide with FaNaCh, FMRF with FaNaCh, and FMRF with FMRFamide and FaNaCh. The tetrapeptide alone (Fig. 3A) produces no NOESY cross-peaks, as expected. However, with a small amount of the expressed receptor, the same peptide produces many NOESY peaks (Fig. 3B), which arise from the NOEs building up in the receptor-bound state and transferred to the free-state. This is called the transferred NOE (TRNOE), and for moderate to weakly interacting ligand-receptor pairs, it allows the structure of the bound ligand to be determined. We have collected several control experiments to rule out non-specific interactions, the most convincing of which is shown in Fig. 3C and 3D. FMRF (no amide) does not activate the channel, and Fig. 3C shows that it does not interact with FaNaCh. Fig. 3D shows the addition of FMRFamide to the sample from Fig. 3C and shows that the expressed extracellular domain of the FaNaCh receptor is able to bind to FMRFamide but not FMRF. The NMR data in Fig. 3B and others not shown have produced several distance constraints that we have used to obtain a

low-resolution structure of FMRFamide bound to FaNaCh (Fig. 4). We are currently measuring the binding affinity of the peptide for the receptor and are collecting additional NMR data to improve the distance measurements and thus refine the structure.



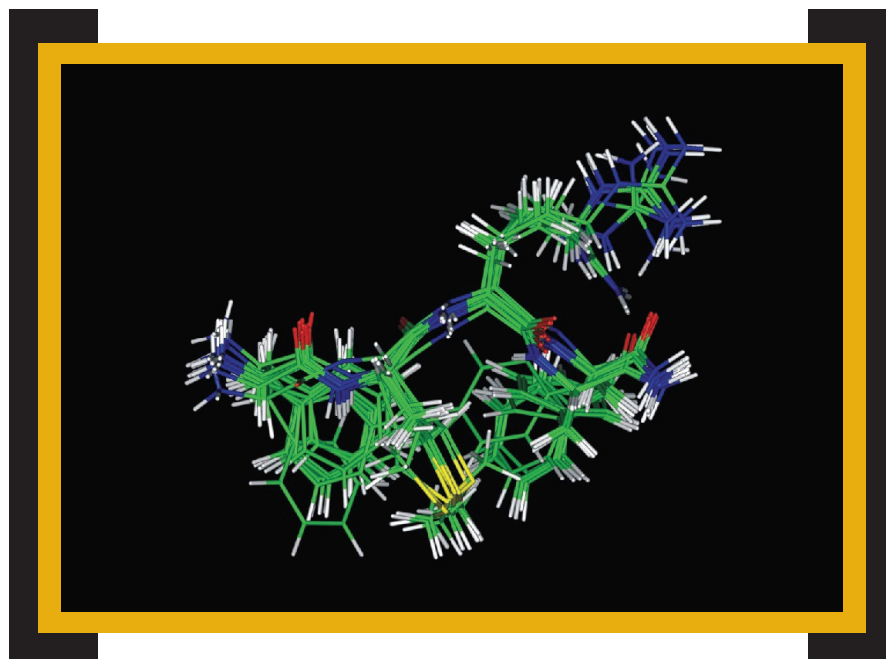
**Figure 2.** 1D  $^1\text{H}$  NMR spectra of the aromatic region of 1 mM FMRFamide alone (bottom) and 1 mM FMRFamide + 20  $\mu\text{M}$  FaNaCh (top). The TSP standard (not shown) is identically narrow in both spectra. The broadening in the top is due to interactions between the peptide and receptor.



**Figure 3.** NOESY spectra for various samples collected on Bruker Avance 750 MHz (A and B) and 600 MHz (C and D) spectrometers at the AMRIS facility in the McKnight Brain Institute of UF. The mixing time for all experiments was 100 ms, but similar results (not shown) were obtained for 50 ms mixing times. Concentrations of peptide in all experiments is 1 mM and receptor is 20  $\mu\text{M}$ . FMRFamide alone (A) shows no significant cross peaks, but FMRFamide + FaNaCh (B) shows many NOE cross peaks. FMRF (no amide) + FaNaCh (C) shows no cross peaks, but addition of FMRFamide to the sample in C produces the same spectrum as B, demonstrating that the receptor binds to FMRFamide but not FMRF.

**Figure 4.** Family of FMRFamide structures bound to FaNaCh obtained from superimposing energy minimized structures from equally spaced points in a restrained molecular dynamics simulation. The restraints were obtained from the NOESY data. The lack of clarity in the family represents our current uncertainty in the structure. The bottom of the structures is completely hydrophobic, and the top has functional and charged groups. Color coding\* is green (carbon), white (hydrogen), red (oxygen), yellow (sulfur), and blue (nitrogen).

\*See <http://magnet.fsu.edu/publications/reports> for color image.



## People in the News



**Timothy Cross**, NHMFL NMR program director and recent recipient of the FSU Distinguished Professor Award has been awarded an \$8.1 million grant from the National Institutes of Health. The project aims to develop technology for characterizing protein structures that exist in biological

membranes. For further information, see page 12 .



**Art Edison**, UF assistant professor of biochemistry and molecular biology recently took over a two-year rotating position as Director of the Advanced Magnetic Resonance Imaging and Spectroscopy (AMRIS) facility in the UF McKnight Brain Institute. Edison obtained his undergraduate degree in

chemistry from the University of Utah and started his NMR career doing undergraduate research in Professor David Grant's laboratory. From Utah, Edison went to the University of Wisconsin-Madison, where he obtained his Ph.D. under the joint supervision of Professors John Markley and Frank Weinholt. After his Ph.D. training in protein NMR and computational chemistry, Edison took a postdoctoral position studying nematode neurobiology with Professor Antony Stretton in the Zoology Department at the University of Wisconsin-Madison. Edison joined the faculty at UF and the NHMFL in 1996; his primary research interest is in structural biology, with a particular emphasis on neuropeptides and other brain proteins and peptides. In addition to several funded projects on structural biology applications, Edison is a core principal investigator on Professor Steve Blackband's recent NIH/NCRR NMR resource grant to develop novel detection methods for NMR and MRI. Edison's major interest outside of the laboratory is music. He plays trumpet, sings, and regularly performs with a "doo-wap, jazz, R&B, gospel, ..." acappella group called Audacity.



**Stephen Hill**, UF assistant professor of physics, has received two grants from the National Science Foundation: "Acquisition of a Micro-Calorimeter Configured with a 10 T Split-Coil Magnet," and "Electron Magnetic Resonance Investigations of Conductors and Superconductors." For more

information on Hill, see *NHMFL Reports*, Summer 2001, page 19.



**Christine Hughey**, FSU Ph.D. candidate and graduate research assistant in the NHMFL ICR Program, won an award for best student poster for her presentation, "Resolution and Chemical Identification of Components in Petrochemicals by Fourier Transform Ion Cyclotron

Resonance Mass Spectrometry," at the annual meeting of the Federation of Analytical and Chemical Spectroscopy Societies in Detroit. That project represents a collaboration between NHMFL and ExxonMobil. In addition to her two trophies, Hughey also received a two-year membership in the Society for Applied Spectroscopy and a subscription to *Applied Spectroscopy*.



**Hiroshi Maeda**, the original discoverer of superconductivity in the Bi-Sr-Ca-Cu-O system and former director of the Tsukuba Magnet Laboratory, is visiting the NHMFL and the FSU Center for Advanced Power Systems for an extended stay of nine months. He will continue to

work on the effects of magnetic fields on the processing of Bi-Sr-Ca-Cu-O superconductors (which are the focus of activities of the laboratory's HTS Magnet and Magnet Materials Group), and the development of a low AC loss Bi-Sr-Ca-Cu-O conductor.

Maeda's research will make significant use of the NHMFL's D.C. magnet facilities; the 33 T resistive magnets and the 45 T Hybrid will be used for short sample  $J_c$  measurements. The large bore resistive magnet will be used in conjunction with the furnace developed by Justin Schwartz's group to facilitate high-field materials processing for magnetic melt processing (MMP) studies.

Maeda, who is affiliated with the Kitami Institute of Technology as a professor of materials science, has collaborations with the National Research Institute for Metals in Tsukuba, Japan and the Institute for Materials Research at the Tohoku University in Sendai, Japan.

**Editor's Note:** We sincerely apologize to Dr. Maeda for identifying him incorrectly in the last issue of *NHMFL Reports* and regret any inconvenience.

## Conference & Workshop Activity

### 42nd Sanibel Symposium

February 23-March 1, 2002

St. Augustine, Florida

Hotel Headquarters: Radisson Ponce de Leon Resort

Abstract Deadline: January 4, 2002

<http://www.qtp.ufl.edu/~sanibel/>

The NHMFL is a partial sponsor of this symposium held by the Quantum Theory Project at the University of Florida. The meeting is dedicated to the memory of Per-Olov Löwdin, who founded the symposia in 1960. Several plenary sessions will be covering subjects that were part of his many and diverse research interests. It will focus on forefront theory and computation in quantum chemistry, condensed matter physics, molecular dynamics, quantum biochemistry, and biophysics. Invited and poster sessions, as well as informal discussions, will include topics on nanostructural materials, quantum control and computing, metals in biology, and protein folding. For more information, e-mail [sanibel@qtp.ufl.edu](mailto:sanibel@qtp.ufl.edu) or visit the conference Web site.

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

May 9-11, 2002

Tallahassee, Florida

Hotel Headquarters: Doubletree

Pre-Conference Registration Deadline: March 8, 2002

<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. Preparation and modification of biodegradable magnetic particles, characterization of magnetic particles, application in cell separation and analysis, applications in molecular biology, and targeted drug delivery will be discussed. Clinical applications of magnetic nano- and microspheres for cancer treatment, hyperthermia, and magnetic resonance contrast enhancement will also be covered. For more information, contact Urs Hafeli at [hafeliu@ccf.org](mailto:hafeliu@ccf.org). Abstract deadline is March 8, 2002.

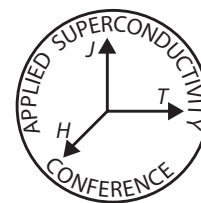
### Applied Superconductivity Conference (ASC04)

October 4-8, 2004

Jacksonville, Florida

Hotel Headquarters: Adam's Mark

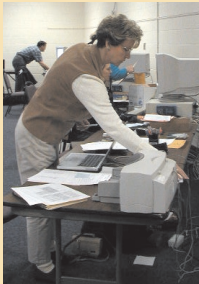
This important international conference is held every two years and typically attracts approximately 1,800 participants. In September 2000, ASC00 was held in Virginia Beach, Virginia; in August 2002, ASC02 will be in Houston, Texas, and in October 2004, ASC04 comes to Jacksonville, Florida. For information,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



# PPHMF-IV Conference

by Rebecca McIntosh

The fourth Physical Phenomena at High Magnetic Fields Conference (PPHMF-IV) was recently held in Santa Fe, New Mexico from October 20-25, 2001. The PPHMF conference is held every three years and represents an opportunity for the global community of researchers to come together to discuss advancements, share results, and plan for future collaborations in the areas of physics at high magnetic fields.



NHMFL Director Jack Crow emphasized that it also represents an opportunity to advertise the NHMFL as a user facility open to the international community of researchers in all disciplines of science.

Unique to this year's event, the conference was kick-started by the J. Robert Schrieffer Symposium on October 19, which honored Dr. Schrieffer's life-long dedication and contribution to the scientific community.

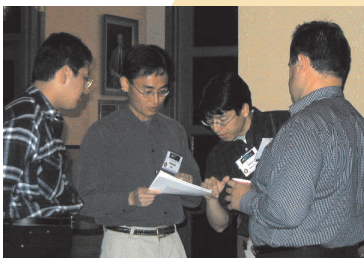
Crow explained that the NHMFL felt combining the celebration with the start of the PPHMF-IV conference was a good match. "Dr. Schrieffer, the NHMFL Chief Scientist, has been a pivotal contributor to the development of the Magnet Lab," said Crow.



Schrieffer, who also stayed to attend the conference, said that this year's event "reflects the real growth and importance of the pulsed field facility [of the NHMFL-Los Alamos]." He continued to say that the NHMFL is proud of what has been achieved there.



The fourth PPHMF conference included four and a half days of invited speakers' presentations, poster sessions, and a tour of Los Alamos, the surrounding area, and the NHMFL Pulsed Magnet Facility



at the Los Alamos National Laboratory. As for the content of the conference, Schrieffer says "the topics haven't changed much over the years, but there are always dramatic new developments." He notes that one such area this year is that of superconductors, with discoveries of new superconductors and the remarkable demonstration of the interface between antiferromagnetism and superconductivity in Tallahassee.

## J. Robert Schrieffer Symposium October 19, 2001

Nearly 150 people honored NHMFL Chief Scientist Bob Schrieffer at a special event in his honor at Los Alamos. Among them were five Nobel laureates and colleagues and students throughout the years. For more information on the symposium, see page 3.



**Nobel Laureates Attending Schrieffer Symposium** (left to right) Walter Kohn, Phil Anderson, Horst Störmer, J. Robert Schrieffer, Bob Laughlin, Alan Heeger



### **Schrieffer's Postdocs and Graduate Students — Past and Present — Attending Symposium**

**1st Row:** left to right: Shoucheng Zhang, Victor Barzykin, Anuvrat Joshi, Dan Arovas, Wu-Pei Su, Daniel Agterberg

**2nd Row:** Bernard Coqblin, Qijin Chen, Robert Paulson, Bob Schrieffer, Catherine Kallin, Piers Coleman, Ted Einstein, Steve Kivelson

**3rd Row:** Nick Bonesteel, James Davenport, Doug Scalapino

## Geochemistry Joins Ocean Research Consortium

In July, FSU, along with Stanford University, joined a select group of American oceanographic institutions that manage the study of ocean sciences globally and plan research. The FSU Department of Geological Sciences' participation in the Ocean Drilling Program (ODP), an international partnership of scientists and research institutions organized to explore the history and structure of the Earth through scientific drilling of the ocean floor, was the impetus for FSU's election to JOI, the 16-member Joint Oceanographic Institution. According to Vincent Salters, a scholar/scientist at the NHMFL, "FSU has had faculty members conduct research on 15 ODP cruises. Over the years, 40 FSU graduate students have sailed on the cruises. For students this sea-going experience is an especially exhilarating one. It gives them the opportunity to conduct research as a team member in an interdisciplinary group of top-rated scientists." Salters concludes that the NHMFL's Geochemistry group is interested in this project because of the close relation of some of its research to ocean drilling and the opportunity to shape future research in an interdisciplinary-multi institutional environment.

# REPORTS

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J. BARDEEN, L. N. COOPER  
Department of Physics

conductivity is presented, based on the interaction between electrons resulting from the fact that the energy difference between the normal and superconducting states involved is less than the phonon energy available to form a superconducting phase. The normal phase is described by the BCS model. The ground state of a superconductor is a combination of normal state configurations. The normal state configurations are virtually excited in pairs of opposite spin and lower in energy than the normal state. The ground state is an average ( $\bar{\psi}$ ), consistent with the mutually orthogonal set of excited states it

### I. INTRODUCTION

facts which a theory of superconductivity explain are (1) a second-order phase transition at the critical temperature  $T_c$  and (2) the heat capacity jump  $\Delta C$  at  $T_c$ .

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SECOND SEMESTER  
GOOD ONLY FOR LAST TERM VALIDATED

SUMMER  
GOOD ONLY FOR LAST TERM VALIDATED



Robert Schrieffer  
M. Bessy Champaign  
DIRECTOR OFFICE OF RECORDS AND RECORDS



Pippard version, and we have the present