NATIONAL HIGH MAGNETIC FIELD LABORATORY

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Collaboration with Grenoble Magnet Laboratory Yields Large Bore Resistive Magnets

The NHMFL large bore resistive magnet—the result of a three-year partnership with the Grenoble High Magnetic Field Laboratory—is nearing completion and should be installed in User Cell 4 in Tallahassee by early spring. A similar magnet was tested at the Grenoble laboratory in France in October, 1997.

The collaboration began in February, 1995, at the International Workshop on Advances in High Magnetic Fields held in Tsukuba, Japan. Representatives attending the workshop from the Tallahassee and Grenoble laboratories agreed to the joint design and construction of magnets to be compatible with both facilities. The outer diameter of the coils would be one meter and the housings at the two facilities would be the same. Two common Bitter coils with inner diameter of 400 mm would be designed to provide as high a field as possible while still being compatible with the power supplies and water pumps of the two different facilities.

All the design work was carried out in Tallahassee and agreement with the Grenoble lab on the common coil design was achieved in July, 1995. The common coils are 575 mm long and are made of high purity copper sheet with axial current grading to maximize the field to power ratio. The Tallahassee configuration provides 11.1 T on axis in a 365 mm bore consuming 13.5 MW of DC power. The Grenoble configuration provides 10.8 T while consuming 13.2 MW.

Both laboratories are installing "insert" coils into these common outer coils to attain higher fields in smaller bores with more power. The Tallahassee insert

 The cooperative agreement encourages leading scientists from the United States and Japan to exchange scientific data and engineering technologies in their respective pursuits of research at the highest magnetic fields available to their scientific communities. In addition, the two international laboratories will share personnel and conduct joint conferences. The implementation of this agreement caps a series of productive exchanges between the two institutions over the last several years. The NRIM has played an important role in materials development, which has allowed the NHMFL to produce the world-record 33 T continuously powered magnet for researchers in Tallahassee. In return,this year the NHMFL built a 30 T magnet system for scientists using the magnet facilities at NRIM.

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NHMFL Director Jack Crow announced the agreement at a press conference on December 8, 1997. The signing ceremony was held in conjunction with the U.S.-Japan Workshop on High Temperature Superconductors, hosted this year by the NHMFL .

Participating in the announcement were (left to right): Hans Schneider-Muntau (NHMFL Deputy Director); Hitoshi Wada (NRIM Director); Jack Crow; and John Graybeal (UF Associate Professor and recent director of NHMFL In-House Research Program). Toshikazu Ishii (Science and Technology Agency of Japan) and Tom Weber (NSF Division of Materials Research) also participated in the press conference, but are obstructed by the podium in this picture.

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COSTALLANDS **U.S. and Japanese Magnet Labs Sign Cooperative Agreement**

REPORTS

Representatives of the National Research Institute for Metals (NRIM) and the NHMFL signed an international collaborative agreement on December 8, 1997, to enter into a cooperative program to facilitate new scientific exchanges, to advance user research facilities, and to drive important new technologies.

GRENOBLE continued on page 4

WINTER 1998

From the Director's Desk

The National High Magnetic Field Laboratory (NHMFL) is almost midway through the second five-year funding cycle. The next two years we will be

devoting considerable time to structuring the vision for the next National Science Foundation renewal. Undoubtedly, a major component of the proposal will be collaborations with the private sector on emerging magnet-related technologies, and I would like to discuss briefly some of the more recent interactions the NHMFL has had with industry.

There is growing interest and opportunities in the area of **power engineering.** This is being driven, in part, by the anticipated deregulation of the utility industry and the need to store larger amounts of power and achieve greater efficiency in transmitting electric energy, especially over longer distances. In addition, the Navy and private shipbuilders are moving to the electrification of ships. The NHMFL has two available research positions targeted for power engineering.

Superconducting magnetic energy storage (SMES)

systems have the unique capability of storing large amounts of energy with a very little loss factor*.* SMES devices could be useful to electric power companies who will need to stabilize the transmission of power over longer distances and possibly across state lines in a "free wheeling" environment. An article on page 7 of this issue of *NHMFL Reports* describes how the NHMFL is assisting BWX Technologies in the conceptual design of a commercial SMES system that utilizes the cable-in-conduit design that the NHMFL incorporated in the 45 T Hybrid magnet system. In the longer term, SMES systems may be designed, developed, and tested for ship utilization. SMES systems will be employed for the propulsion of aircraft launched from ships. The NHMFL is already assisting the Navy in testing a prototype compact SMES magnet designed and built by Westinghouse.

The Navy is interested in the capability to move ships at higher speeds with greater efficiencies. *The electrification of ships' propulsion systems* offers significant potential to decrease the volume and weight of power systems. These factors are essential to the development of high speed ships that would enable the Navy to transport personnel and equipment to troubled spots throughout the world in much less time. This became a problem in the Gulf War when it took the military six months to gear up for the conflict, because 90 percent of personnel and materiel had to be transported by ships. To employ these new electric drive systems successfully, however, new components must be designed, developed, and tested.

NHMFL Interactions with Industry

These systems and components need development testing that can be provided only by the NHMFL. With these R&D goals at the forefront of discussion, the Office of Naval Research and the Society of Research Administrators are hosting a Spring conference in the Gulf Coast region, the home of many shipbuilding enterprises. The NHMFL and the Gulf Coast Alliance for Technology Transfer (GCATT) will be active participants in this conference and dialogue.

The NHMFL is a contributing partner to the *MAGLEV 2000 project,* which is supported by Florida industry, academia, and the state Department of Transportation. The State of Florida has a continuing interest in maglev as a nextgeneration alternative to high-speed rail that will link the metropolitan areas of Miami, Orlando, and Tampa. MAGLEV 2000 recently announced a new simulator designed to demonstrate to the public, in a very visual way, the characteristics of maglev travel at 220 miles per hour over a 20-mile ride through Central Florida. MAGLEV 2000 will begin construction soon of a 1,000-foot segment of a two-mile maglev guideway that will be completed later in 1998. Most thoughts of maglev immediately turn to high speed passenger rail, however, there may be other applications at lower speeds where maglev provides many advantages over conventional systems. For example, the mining industry has expressed strong interest in maglev technology for mining applications to climb steep grades at high speeds, to haul heavy loads, to avoid noxious exhaust pollution, etc. Applications such as these were studied previously by the Bureau of Mines. MAGLEV 2000 will demonstrate at its test site the ability of maglev "hoppers" to move very heavy bulk loads required by the mining industry. With no moving parts or contact with rail surfaces, maglev technology has the potential to be competitive and cost effective compared to traditional inter-modal transportation.

The NHMFL is always seeking input from its external users and the private sector on emerging opportunities and applications of magnet-related technologies. One of the missions of the NHMFL is to encourage and establish public and private sector partnerships that advance magnetic field research and enhance U.S. competitiveness. This mission is taken very seriously by the leadership of the NHMFL, and we look forward to hearing from you and forging new and productive partnerships.

 Jack Crow, Director and Co-Principal Investigator, FSU Don Parkin, Co-Principal Investigator, LANL Neil Sullivan, Co-Principal Investigator, UF

From the

FTICR Offers New Insights and Research Opportunities

John R. Eyler

Fourier transform ion cyclotron resonance (FTICR) mass spectrometry has emerged in recent years as one of the most powerful mass spectrometric analysis methods.¹ lons can be generated from a variety of sources, transferred into a Penning trap, and stored there while being subjected to ion/molecule reactions, laser irradiation, or collisional dissociation. The technique allows ions of extremely high mass to be analyzed, with unsurpassed mass resolving power. The Penning trap requires the presence of a magnetic field, and most desirable figures of merit in FTICR mass spectrometry improve at higher magnetic fields, while many undesirable features of the technique become less important at high fields.²

FTICR mass spectrometry was not included in the original mission of the NHMFL; however, many readers of this column are probably aware that a very active FTICR research program is now in place in Tallahassee under the direction of Alan Marshall. Marshall, as P.I., along with Shenheng Guan and the author as co-P.I.'s, secured funding from the Chemical Instrumentation Program at NSF for a National High Field FTICR Mass Spectrometry Facility. After approximately a year and a half of instrument construction (with continuing projects up to the present time), this facility is now providing state-of-the-art FTICR techniques and instrumentation to a wide range of external users.

Most of the research at the National High Field FTICR Facility has concentrated on large biomolecules and the ions produced from them, since the FTICR approach is almost uniquely suited for studying this important class of ions. Work in our group at the University of Florida (UF) has investigated a range of chemical systems and the ions associated with them, from atomic ions through intermediate size organometallic complex ions to some larger ions of biochemical interest. This report will highlight three projects of current interest: (1) development of inductively-coupled plasma (ICP) FTICR mass spectrometry for high resolution elemental analysis, (2) reactivity and photophysics of partially solvated organometallic complex ions, and (3) high mass resolution analysis of combinatorial libraries. A majority of the studies in each of these areas have been carried out at UF on FTICR mass spectrometers that utilize 3 and 4.7 T superconducting magnets. Each project,

Chief Scientist's Desk *Chief Scientist's* Desk *One of the most remarkable developments over the past decade is the emergence of ion cyclotron resonance (ICR) as a highly accurate technique to study the mass spectrum of a sample. Particularly important is the development of Fourier transform ICR techniques that greatly improve the mass resolving power of the system. Now very large ions can be resolved using the FTICR mass spectrometry facility to better than one part per million. In this issue, Professor John Eyler at the University of Florida reports on three aspects of research using this facility, (1) elemental analysis, (2) partially solvated organometallic ions, and (3) studies of combinatorial libraries. The later promises rapid analysis of thousands or millions of compounds of interest in identifying drugs that bind to a receptor.*

> *FTICR is a prime example of how research can be carried out at two sites (UF and FSU) with strong interactions between these groups leading to an overall program that is considerably stronger than the sum of its parts.*

> > *R. Schrieffer*

however, has benefited in some way from the use of higher field instrumentation in the National High Field FTICR Facility at the NHMFL.

Elemental Analysis Using ICP-FTICR Mass Spectrometry.

Inductively coupled plasma mass spectrometry (ICP- MS ³ is a powerful analytical method for trace elemental analysis. Some of its advantages include low detection limits (sub part-per-trillion), nearly simultaneous multi-element analysis capability, inherent isotopic information, short analysis times, and easy spectral interpretation (compared to optical methods). The mass spectrometers used to date in ICP-MS have been quadrupole and magnetic sector devices, with considerably lower mass resolving power (MRP) than that which is possible with FTICR mass analysis. Higher MRP is frequently needed, however, to separate the peak due to an atomic ion of interest from "interferences." These interferences can be due either to polyatomic ions (hydrides, oxides, argides, cyanides, etc.) that are produced from background species present in the high-energy plasmas that generate the ions, or to the isotopes of other elements that have nominally the same mass as the ion of interest.

FTICR continued on page 5

GRENOBLE continued from page 1

Figure 1. 20 T, 196 mm bore magnet nears completion at NHMFL.

consists of two Florida-Bitter coils electrically in parallel constructed from high purity copper sheet. The insert is electrically in series with the common coils and hydraulically in parallel. The complete assembly should attain 20 T in a 196 mm bore using 20 MW of power. The Grenoble insert consists of 18 polyhelix coils electrically in series constructed from copper-zirconium tubes. Their insert is powered and cooled on separate circuits from the common coils and should attain a field of 36 T in a 34 mm bore using 24 MW of power.

Detailed design of the common coils was completed in Tallahassee in March, 1996, and culminated in a one-week design review with the Grenoble laboratory. Each laboratory has taken full responsibility for design and construction of its own insert coils. Construction of the common coils and housing was a joint effort set up in an attempt to distribute the costs evenly between the two institutions and still maintain tight document control with two laboratories and dozens of machine shops involved. The magnet housings were fabricated by Collet et Amblard of Fontaine, France, and paid for by the Grenoble laboratory. The stamping tooling for the common coils was fabricated by Outillex of Echirolles, France, again paid for by the Grenoble lab. Everything else was paid for by the Tallahassee laboratory including: the copper sheet for the Bitter disks fabricated by Outokumpu of Pori, Finland; stamping the disks by Outillex; endplate fabrication by ECM of Florida; support rings by RV Industries of Honeybrook, PA, etc. By collaborating on the construction in this manner, each of the two laboratories saved approximately \$100,000 in tooling and setup charges.

In October, 1997, the Grenoble laboratory tested their magnet to 10.5 T in a 365 mm bore using 12.2 MW. That magnet was used for a few weeks by the scientific community, and the insert has been installed and should be tested sometime in March, 1998.

All parts were received for the Tallahassee magnet in February, 1997, but assembly was delayed until recently due to other pressing obligations. Stacking of the first coil is about two-thirds complete and the magnet should be operating in User Cell 4 in a few months.

Once the 20 T, 196 mm bore magnet is completed it will be used in that configuration for a variety of large scale experiments including: superconducting insert magnet testing, large scale levitation, and ion cyclotron resonance. One of the first uses of this system will be to test the performance of a high temperature superconductor (HTS) coil under development in collaboration with Intermagnetics General Corporation (IGC) of Latham, NY. This program, which is funded by the National Institutes of Health, has as its main task the design, construction, and testing of a 2.5 T HTS insert coil that will operate in a 20 T background field. This coil will consist of a hybrid configuration made from an outer section of surface coated prereacted Ag/BSCCO-2212 produced by IGC. The outer coil set will consist of five layer wound sections with stainless steel overbanding. The inner coil will be constructed by the Tallahassee HTS Magnet and Materials group using a wind and react process with

sol-gel insulated Ag Alloy/BSCCO 2212 conductor to avoid the bend strain limitations. This coil will be pancake wound of five double pancake sets. The outer coil is designed to have a 160 mm outer diameter and a 52 mm inner diameter (ID). The inner coil will have an ID of 13 mm. The two coils will be operated in parallel to optimize performance.

Figure 2. Drawing of 20 T , 196 mm bore resistive magnet with 2.5 T HTS coil and cryostat installed.

To perform the 2.5 T coil test in the 20 T, 196 mm bore resistive magnet, the system will be equipped with a 165 mm ID liquid helium cryostat for large volume experiments. This cryostat is under construction at Ability Engineering and Technology, Inc., in South Holland, IL, and is due to be delivered to Tallahassee in early April, 1998. The cryostat will be equipped with a high current insert fabricated at the NHMFL for supporting test coils including the 2.5 T HTS coil. A schematic of this facility showing the 2.5 T insert coil installed in the cryostat is shown in Figure 2. The completed nested coil set will be tested in Spring, 1998.

This article was contributed by Mark Bird (850-644-7789, bird@magnet.fsu.edu) and Steven Van Sciver (850-644-0988, vnsciver@magnet.fsu.edu), of the Magnet Science and Technology Group in Tallahassee.

4

FTICR continued from page 3

For the past several years we have been involved in an instrumentation/methodology development program to see if the high MRP of FTICR mass spectrometry might provide benefits when coupled to ICP atomic ion sources. A majority of the funding for this project has been provided by the Director's Fund at the NHMFL, with some instrumentation purchased by the National High Field FTICR Facility. Quite impressive results have been obtained, based on work done both at UF and at the NHMFL. Results of the initial stages of this work have recently been published.4

An example of the very high MRP possible in the analysis of atomic ions is seen in Figure 1, obtained on a relatively low-field (3 T) FTICR system at UF. The atomic ion Ca+ is seen clearly separated from Ar+ (always present in ICP sources since Ar is used as the plasma gas). Detection of trace levels of calcium can now be carried out without "interference" from argon ions, permitting much lower and more reliable detection limits. The results in Figure 1 are the first demonstration ever of the separation of Ca + from Ar + in ICP mass spectrometry. The mass resolving power demonstrated is at least a factor of 5 higher than ever before seen with this technique. Other experiments have shown at least parts per billion sensitivity with this system.

Construction of an ICP-FTICR mass spectrometer system at the National High Field FTICR Facility utilizing a 6 T magnet is nearing completion. This instrument will permit analyses of ions requiring considerably higher MRP to assess the effects of interferences. Other improvements in the NHMFL instrumentation should lead to lowered detection limits (to low or mid- parts per trillion), making this instrument a most powerful addition to ICP-MS elemental analysis.

Partially Solvated Organometallic Ions. One of the major applications of ICR in past years has been the study of (primarily organic) ions in the gas phase, free of solvation, and the comparison of their behavior to that seen in solution. Valuable insights have been gained as to the effects of solvation on inherent ionic properties. In a joint project with Prof. David Richardson at UF, we have been applying a similar approach to study partially and completely ligated organometallic ions of interest to inorganic chemists.5

Recent modifications to our electrospray ionization (ESI) source permit us to generate partially solvated organometallic ions and inject them into the FTICR mass spectrometer for subsequent reactivity and photochemistry studies. Figure 2 gives an example of the solvation of $Ru(NH_3)_4(bipy)^{2+}$ (see figure for structure) with up to 8 acetonitrile ($CH₃CN$) molecules. The characteristic "cluster" of peaks for each degree of solvation is due to the naturally occurring isotopes of Ru. One of the important features of FTICR mass spectrometry is that selected ions can be isolated in the analyzer cell and unwanted ions ejected. Thus, from a collection of ions such as is shown in Figure 2, a complex with a particular degree of solvation (e.g. n=4) can be chosen for further study. In this way chemical and spectroscopic properties of ions with an increasing degree of solvation can be studied.

Figure 1 Figure 2

We have begun investigations into the chemical reactivity of these partially solvated organometallic ions, looking at how charge transfer, ligand substitution, and other bimolecular ion/molecule reactions change as a function of the number of attached solvent molecules. We have also started studying the photodissociation of the ions when they are subjected to tunable radiation from a Nd:YAG-pumped dye laser.⁶ Organometallic ions in general have strong "charge-transfer" bands in the visible

FTICR continued on page 6

FTICR continued from page 5

region of the spectrum and there is considerable interest in how these bands shift as a function of the nature and number of solvent molecules surrounding the ion. We have also noted that the ion-solvent clusters slowly lose solvent molecules as a function of storage time in the FTICR analyzer cell. We believe this is caused by the absorption of blackbody radiation and the subsequent heating it causes in the ion. Other workers have shown that careful analysis of this phenomenon can lead to energetics of dissociation for weakly-bound complexes, so we hope to obtain sequential solvation energies for these organometallic ions by following the rate of solvent loss as a function of cell temperature.

All successful experiments with the partially solvated ions have been carried out on a 4.7 T FTICR system at UF. Recently, however, modifications have been made to ESI sources at the National High Field FTICR Facility at the NHMFL, which will permit additional work to be done in Tallahassee on solvated ions of interest.

Studies of Combinatorial Libraries. The use of combinatorial libraries has revolutionized drug discovery in the 1990's, and further applications are being pursued actively by researchers in both academia and in virtually all pharmaceutical companies. Briefly, instead of synthesizing one compound at a time to test for potential drug activity, a "library" of compounds is synthesized using combinatorial methods, 7 and this entire library is then presented to a receptor (enzyme, antibody, etc.) to see which, if any, of the library components is bound preferentially. The "lead compounds" are usually separated from the receptor, and their structures determined in some manner. Similarities among the lead compounds are used to predict important structural features which should be incorporated into potential drug candidates.

Combinatorial libraries can contain many thousands (or millions) of compounds. This poses severe problems for analyzing the libraries to assess their purity, completeness of synthesis, equal representation of all components, and structure of lead compounds. FTICR mass spectrometry, with its high mass resolving power, exceptional mass accuracy, ms/ms capabilities, and ability to analyze high mass ions, has been successfully applied to several aspects of the synthesis and application of combinatorial libraries.

With as many as $10³$ to $10⁶$ compounds in a combinatorial library, mass spectrometric analysis becomes extremely difficult because there is almost always at least one (and most often several) peak at

each mass in the spectrum. The high mass resolving power of FTICR, however, can be used to separate and identify all ions, even if they have nominally the same mass. Laser and collisional dissociation techniques are being developed to distinguish the structures of compounds of identical mass, as well. Our first work in this area, using FTICR instruments at both UF and the NHMFL, was published about 18 months ago.8

Funded by an NHMFL In-House Research Program grant, we have made considerable progress on other studies related to combinatorial libraries. Maria Wigger, a graduate student in the group of Steven Benner at UF, has demonstrated that FTICR-MS can be used to probe the substrate specificity of enzymes.9 Figure 3 shows at the top a small tripeptide library that mimics possible substrates of the enzyme glutathione-Stransferase. At the bottom is shown the spectrum resulting when the tripeptide library is mixed with glutathione-S-transferase and 1-chloro-2,4 dinitrobenzene (CDNB). Although 16 tripeptides were in the crude library, the enzyme selected only one and catalyzed its reaction with CDNB (the product ion peak is identified on the spectrum and its structure given). The favored tripeptide contained glycine as the amino acid that was varied in library synthesis, and was, in fact, glutathione, the natural substrate of the enzyme. This proof-of-principle experiment suggests combinatorial experiments where two functionalized libraries, each of which targets a different substratebinding pocket, would be presented to the enzyme and the favored substrates detected by observing their unique reaction product. This work was recently described in the News and Features section of *Analytical Chemistry*, which summarizes papers published elsewhere that are deemed to be of great interest to the journal's readers.

Figure 3

FTICR continued on page 7

NHMFL & BWX Technologies Collaborate on SMES

The NHMFL and BWX Technologies (BWXT, formerly Babcock and Wilcox), are joining forces to develop a new Superconducting Magnetic Energy Storage (SMES) system by mid-1999. The novel engineering and design concepts of this system are expected to be of considerable interest to electric utility companies.

SMES systems store large amounts of energy in a DC magnetic field with very little loss for later delivery as AC power. The main components in such systems are superconducting magnets and powerelectronic inverters for the DC to AC conversion. The particular application planned by BWXT is a system for stabilizing power transmission lines, wherein power

would be alternately removed from the line and returned in such a way that fluctuations are damped to an acceptable level.

BWXT was interested in collaborating with the NHMFL on this project primarily because of the laboratory's experience and knowledge gained through the design, development, and manufacture of the 45-T Hybrid system. In particular, they view the Hybrid's cable-in-conduitconductor technology as crucial to their system design. In addition, NHMFL has in place a variety of magnet-component test facilities that are particularly well suited for supporting the development of such a SMES system.

Initially, the NHMFL's role in the project will include assistance with the conceptual design of the SMES magnet. During this phase of the project, which has already begun, key development issues will be identified. The project will then move into the preliminary design and analysis phase and finally to the development and testing phase. The principal technical contacts are Vladimir Karasik of BWXT, and John Miller of the NHMFL. Target date for completion of the preliminary design as well as the development and testing activities this spring. BWXT expects to make delivery of the first completed system in mid-1999.

For further information on the NHMFL-BWXT SMES collaboration, contact John Miller at the NHMFL, 850-644-0929, miller@magnet.fsu.edu.

FTICR continued from page 6

The latest studies at the NHMFL by Ms. Wigger and Weiqun Li, a graduate student of Alan Marshall, have examined noncovalent complexes between domains of the SH2 protein and ligands from peptidic libraries. These protein domains play a major role in the signal transduction processes of the cell and are critically involved in various types of cancer. Preconcentration techniques developed and implemented at the National High Field ICR Facility, in combination with the 9.4 T FTICR instrument there, allow the ionization of noncovalent complexes between the peptides and the SH2 protein in the presence of the vast excess of library compounds. The results of these experiments are providing interesting new insights into the binding characteristics of SH2 domains, and can be applied easily to other proteins.

Acknowledgments. Inductively-coupled plasma-FTICR development has benefited greatly from the work of UF graduate student Eric Milgram, Prof. R. Sam Houk of Iowa State University, FSU graduate student Forest White and Prof. Alan Marshall at the NHMFL, and senior research associates Clifford Watson at UF and Chris Hendrickson at the NHMFL. Experiments on partially solvated organometallic ions at UF have been carried out by Dr. Watson, Prof. David Richardson, and Dr. Kathryn Williams. Combinatorial library studies have involved graduate students Maria Wigger and Joe Nawrocki and Prof. Steve Benner at UF, FSU graduate student Weiqun Li and Scholar-Scientists Chris Hendrickson and Mark Emmett at the NHMFL. Funding from various NHMFL sources has been mentioned in the text of the report and is gratefully acknowledged.

Additional support has been provided by the Office of Naval Research and the Inorganic Chemistry Program at NSF.

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Education Workshops Help Teachers Integrate Science and Math

The NHMFL K-12 Educational Programs group will hold five full-day workshops this winter for Tallahassee area teachers in an important new partnership with the Leon County Schools. NHMFL educators Pat Dixon and Sam Spiegel will be leading "MagLab: Alpha– EdVentures in Science" on February 5 and four sessions of "Integrating Science and Math in the Elementary Classroom" in February and March. The NHMFL was invited by Leon County Schools to offer workshops for their teachers with the schools' Eisenhower funds. Over one hundred teachers signed up and the original three workshops had to be expanded to five to handle the demand.

Ms. Dixon explained the unexpectedly high level of interest this way: "Teachers are always looking for new resources, and there's so little out there on magnetism that links science with kids' everyday lives. In addition, these workshops will help teachers meet the National Science Education Standards in a context that students can understand."

In a very significant way, these workshops will provide new ways for teachers to think of science research institutions. As educators begin to see science facilities as resources for the classroom, the gap between science research and practice and the classroom narrows.

There will be two versions of "Integrating Science and Math in the Elementary Classroom," one for primary grades (K-2) and one for intermediate grades (3-5). Teachers will learn and model strategies and activities for integrating math and science standards in the classroom. The focus of the workshop will be technology-related and hands-on activities.

Dr. Sam Spiegel, director of educational programs at the laboratory, earned his Ph.D. in Science Education from Florida State University in December, 1997. His dissertation was entitled Understanding Teacher Enhancement Programs: Essential Components and a Model.

At "MagLab: Alpha–EdVentures in Science," teachers will receive the MagLab: Alpha curriculum package that was developed at the NHMFL in 1997 by scientists, teachers, and students. It comprises 25 hands-on activities including all the materials—and a CD-ROM that supplements the activities. Teachers will explore ways to implement these materials in their classrooms and integrate the activities with other disciplines.

The MagLab: Alpha workshop is intended to give middle school teachers resources on teaching magnets, magnetism, and related concepts. It encourages middle school teachers to look at science as an interdisciplinary subject. In order to facilitate this concept, teams of teachers—including math, language arts, social studies, and science teachers—will be attending together.

> *For further information about NHMFL's educational activities, contact Director of K-12 Educational Programs Sam Spiegel, 850-644-5818, spiegel@magnet.fsu.edu.*

(TMTSF)2PF6 in the Extreme Quantum Limit

S.W. Tozer, S.T. Hannahs, A. Hopkins, C. Mielke, and Woowan Kang

Because of their highly anisotropic nature, organic conductors of the $(IMISP)_2X$ family (Bechgaard salts) provide experimentalists with a rich variety of properties as a function of temperature and pressure. At the same time, the reduced dimensionality of these systems makes them attractive models for theorists due to their simplified electronic structure. The most conductive axis in these materials is along the staggered columns of aromatic rings that constitute a major portion of the cations. These stack together much like poker chips with the orbital overlaps resulting in a conductivity that is typically 100 to 1000 times greater than that between the chains. One member of this family, $(TMISF)_2PF_6$, is a metal at ambient pressure and room temperature, becomes an insulator at liquid helium temperatures, and is driven into a superconducting state when

subjected to hydrostatic pressures between 0.6 and 1.5 GPa and cooled to temperatures below 2 K. With the application of magnetic fields, this material, which had been superconducting, enters a series of field induced spin density waves [FISDW] that leave the material in an insulating state at the extreme quantum limit (n=0). Burlachkov, Gor'kov, and Lebed',1 however, predicted that these insulating materials may change yet again-back to a metallic or superconducting state at still higher fields. It was not possible to test their prediction until now, because the experiments that required high pressure to put $(IMISP)_2PF_6$ into the correct phase have been limited to the highest DC fields available (33 T) due to problems associated with the effect of pulsed fields on the metal used to make high pressure apparatus.

We have performed the first pulsed magnetic field transport studies of $(TMISF)_2PF_6$ at pressures of 0.8 GPa and

temperatures as low as 350 mK. Our previous measurements, which used a metallic cell weighing only 1 gram, showed clear indications of sample heating arising from the eddy currents induced by the rapidly changing magnetic field. The high thermal conductivity of the diamonds allowed the heat generated in this small metal body to be directed to the sample in much less than a millisecond, dashing any hope of acquiring reliable data. Subsequent measurements to quantify the observed temperature excursion during the pulse monitored the intensity of the photoluminescence from an AlGaAs quantum well, which acted as an optical "thermometer." The results clearly showed that, during a full field 50 T shot, the temperature of the sample was driven from 4 K to approximately 70 K. To eliminate eddy current heating, an electrically non-conductive diamond anvil cell has been developed. The need for a material that could withstand thermal cycling and the small size of the cell (dictated by the 10 mm bore of the experimental pulse magnets) led us to consider commercial ceramics, epoxy composites, and other interesting materials such as warthog tusk. All of these were eliminated as none held the fine threads required to generate the load. We explored a variety

Figure 1. Low field

magnetoresistance clearly shows the quantum Hall plateaus in (TMTSF)2 PF6 at a pressure of 0.8 GPa. Measurements were made at a temperature of 350 mK.

> EXTREME QUANTUM LIMIT continued on page 12

ATTENTION USERS

The NHMFL provides very low temperatures to go with the high magnetic fields. The lowest temperature is found at the Ultra-High B/T Facility in Gainesville (see NHMFL Reports, Summer 1997).

Dilution refrigerators also provide millikelvin temperatures in the Pulsed Field Facility in Los Alamos (one on hand and one ordered for the new 60 T, quasicontinuous magnet). And of course, users of the DC Field Facility in Tallahassee have made good use of the Oxford dilution refrigerator and 18/20 T superconducting magnet system since 1993.

The field range available for low temperature experiments in DC fields has now increased to 33 T with the commissioning of a new Oxford portable, toploading dilution refrigerator for the resistive magnets. (The upper field will increase to 45 T when the hybrid is available.) The technical requirements for this refrigerator are demanding. The long distance from the top of the mixing chamber to the center of the magnetic field (660 mm for the hybrid); small, roomtemperature bore of the magnets (32 mm); sample space sufficient to rotate diamond anvil cells (14 mm); and need to top load the samples all pushed the limits of existing technology. Oxford Instruments' and NHMFL staff have finally achieved the desired base temperature of 25 mK and have found that the refrigerator is unaffected by magnetic fields up to the point where the product of the field and the field gradient is sufficient to trap helium bubbles (see McNiff et al, Rev. Sci. Instr. 59 2474-2476, 1988). We are confident that this problem will also have been solved by the time this article is published, and invite interested researchers to request magnet time using this new refrigerator. Send e-mail to *johnson@magnet.fsu.edu*.

Eric Palm and Tim Murphy also have made several improvements to the existing dilution refrigerator system. Users should know that:

• A micrometer drive was added to the string rotator that was developed for the top loading dilution refrigerator. It provides 0.2 degree resolution and repeatability. The rotator can be moved through 360 degrees if the sample leads allow. On change of direction the backlash is 0.6 degrees. The temperature rise generated for a 90 degree rotation is only 80 mK, so recovery of base temperature requires only a few minutes. Another benefit of the new micrometer drive is that the orientation of the sample does not change as the probe is lifted from the work bench, inserted into the refrigerator, and cooled to base temperature.

Bruce Brandt Director, DC Field User Programs

• A linear motion feedthrough system was developed for the top loading dilution refrigerator. This improvement allows users to move their sample relative to the field center without having to remove the probe from the fridge. This technique is especially useful to experimenters using the cantilever force magnetometer as it allows them to separate force and torque terms without disturbing other experimental conditions.

- Cobalt Nuclear Orientation thermometry has been implemented as a primary low temperature thermometry standard against which to calibrate resistance thermometers for use by experimenters.
- An RF filter was designed and built to reduce noise levels and sample heating. It is installed between the connector on the probe and the breakout box.
- A new computer-controlled gas handling system and a remote operation program called "Timbuktu" enable the staff to perform remote operation/trouble shooting after hours or on weekends without having to drive to the lab. Response time to assist users is shorter and wear and tear on the staff is reduced.
- A new magnet power supply and a new low loss, high capacity dewar were purchased and installed on the 20 T magnet with the dilution refrigerator. The power supply allows uninterrupted sweeps through zero field and improves the resolution of the magnet set point from 0.5 mT to 0.02 mT steps. The dewar allows users to sweep at the maximum sweep rate for greater than 24 hrs. before transferring liquid helium.

NHMFL staff have learned quite a bit about specifying, purchasing, commissioning and using dilution refrigerators in the past few years. We are willing to provide a couple of phone calls of free advice to anyone interested, and welcome inquiries about more extensive consultation. Send e-mail to *palm@magnet.fsu.edu*.

PEOPLE PROFILES

Elliot P. Douglas, Assistant Professor with the UF Department of Materials Science and Engineering, was awarded a Presidential Early Career Award for Scientists and Engineers (PECASE) on November 3, 1997. The honor was presented by Assistant to

Elliot P. Douglas

the President for Science and Technology John Gibbons at a ceremony at the Old Executive Office Building in Washington, D.C.

The PECASE was established by President Clinton in February, 1996, and is the highest honor bestowed by the U.S. Government on outstanding scientists and engineers beginning their careers. Dr. Douglas was nominated by the Department of Defense through the Army Research Office, and the award includes a research grant of \$500,000 over five years.

The goal of Dr. Douglas' project is to understand the structure-property relationships in liquid crystalline thermosets (LCT's), which he explains as follows:

Among the improved properties expected for LCT's compared to conventional thermosets are improved chemical resistance, resistance to environmental changes, and enhanced mechanical properties. These property improvements will lead to fiber-reinforced composites with improved performance, environmental stability, and reliability. Thus, success in this project will lead to an understanding of the structure-property relationships in a new class of materials, and allow them to be used in a wide variety of structural applications.

The main objective for this work is to understand the relationships among molecular structure, phase transitions, curing behavior, and mechanical properties for these unique materials. The morphology of LCT's can be understood as a complicated interaction between the liquid crystalline order and the network structure, which are both in turn affected by molecular architecture and cure behavior. Relating these structural parameters to the resulting properties will provide the basic structure-property relationships. In particular, LCT's will exhibit behavior that is characteristic of both ordered liquid crystals and highly crosslinked thermosets. Thus, the program provides a systematic approach to identifying how these two structural aspects control the properties of the material.

While his work for the U.S. Army doesn't involve magnetic fields, Dr. Douglas is one of the outstanding young NHMFL-related faculty members at UF. In addition, in 1996, the NHMFL awarded him a two-year In-House Research Program grant for work that involves

magnetic fields for processing of polymers—work that is being conducted in collaboration with researchers at Los Alamos National Laboratory, Rensselaer Polytechnic Institute, and The Dow Chemical Company.

More Bright Futures...

Nineteen NHMFL-Affiliated Graduate Students Earned Ph.D.s in 1997

Kathleen Amm, FSU Physics

Synthesis and Properties of Mercury Cuprate Superconductors with Metallic Interfaces

- *Juan A. Caballero*, UF Materials Science and Engineering Growth and Characterization of Thin Films of the Heusler Alloy NiMnSb and its Application to Magnetoresistive Multilayer **Structures**
- *Daniel Q. Duffy*, FSU Physics
- Numerical Studies of Strongly Correlated Electronic Systems *Franz Freibert*, FSU Physics
- Critical Phenomena of Selectively Doped YBa₂Cu₂O₇ *Edgar B. Genio*, UF Physics

Low Temperature Nuclear Quadrupole Resonance Studies of Antimony and Application to Thermometry *Alia Hassan*, FSU Physics

- High-Field ESR Studies of the Doped Spin-Peierls System CuGeO₃ *Patrick Henning*, FSU Physics
	- Thermal Conductivity as a Function of the Critical Temperature in High Temperature Superconductors

Krishna Iyengar, FSU, FAMU-FSU Mechanical Engineering Heat Interception for Construction Structural Support and Current Lead for Cryogenic Equipment

George S. Jackson, FSU Chemistry

Tailoring and Analysis of Excitation and Trapping Potentials in FT-ICR/MS. Applications and Animations of Common FT-ICR/ MS Experiments

Kiho Kim, UF Physics NMR Studies of the Orientational Behavior of Quantum Solid Hydrogen Films Absorbed on Boron Nitride

Jewon Lee, UF Materials Science Engineering Comparison of High Density Electron Cyclotron Resonance and Inductively Coupled Plasma Sources for Etching of Electronic Materials: New Plasma Etch Regimes for Electronic Materials *Hsiang-Lin Liu*, UF Physics

Effects of High Magnetic Field and Substitutional Doping on Optical Properties of Cuprate Superconductors

Thomas Meersmann, Université de Lausanne, Switzerland, Chemistry Relaxation and Coherent Evolution as Competing Mechanisms for Coherence Transfer in Nuclear Magnetic Resonance Spectroscopy

K. Eric Milgram, UF Chemistry

Abatement of Spectral Interferences in Elemental Mass Spectrometry: Design and Construction of Inductively Coupled Plasma Ion Sources for Fourier Transform Ion Cyclotron Resonance Instrumentation

Joseph P. Nawrocki, UF Chemistry

Characterization of Combinatorial Libraries Using Fourier Transform Ion Cyclotron Resonance Mass Spectrometry

Wenhua Xu Ni, UF Physics Designs of Novel RF Coils for Signal-to-Noise Ratio Improvement in NMR

Samuel A. Spiegel, FSU Science Education

Understanding Teacher Enhancement Programs: Essential Components and a Model

- *Forest M. White*, FSU Chemistry
- External Ion Injection Techniques and Biological Applications of Fourier Transform Ion Cyclotron Resonance Mass **Spectrometry** *Feng Xu*, FSU Chemistry

Solvent Roles in Polypeptide Structure and Stability

EXTREME QUANTUM LIMIT continued from page 9

of plastics, too, and settled on a new thermoplastic that is quite hard, has an ultimate tensile strength of 35 ksi, and has good notch characteristics. The plastic cell's small size, 9 mm in diameter and 12 mm long, allows both optical and electrical transport experiments2 to be performed in the pulsed magnets at millikelvin temperatures. This permits investigation of transport properties well into the extreme quantum limit regime of these molecular conductors. Low pressure measurements of (TMTSF)₂PF₆ permitted us to test whether we had successfully eliminated heating of the sample from eddy currents. At pressures of 0.4 GPa, the sample is insulating at low temperatures and any heating due to the pulsing of the magnet would have been clearly observed in the field traces from zero to maximum field and back. No heating was observed.

Transport measurements were made along the c-axis of the

material, the least conductive axis. Compared to the normal 4-probe inline measurement of the a-axis, this provided a stronger signal, eliminated problems with contact resistance, and minimized the open loop induced voltage. Figure 1 shows the low field data and the quantum Hall plateaus that have also been observed previously.3 Data to 50 T (Figure 2) shows the well known rise in resistance at the extreme quantum limit. Above this limit the transport shows a marked frequency and field dependence between 80 kHz and 500 kHz and 25 to 60 T, with the resistance dropping at higher frequencies and fields. We do not yet know whether this drop in resistance is an experimental artifact or a new transition in this fascinating material. Experiments to 80 T using experimental, capacitively driven pulsed magnets are planned for the near future. The arrival of the 100 T pulsed magnet is eagerly awaited so that we can further explore the

phase space in these reduced dimensional systems.

We would like to acknowledge M. Collins, W. Bowman, Y. Eyssa, J. Farrell, A. Harper, T. Keister, B. Lesch, D. McIntosh, M. Pacheco, R. Parmarter, P. Pernambuco-Wise, S. Roybal, C. Stokley, D. Walsh, and V. Williams without whom this study would not have been possible.

References:

- ¹ Lebed', A.G., JETP Lett., *44*, 114 (1986); Burlachkov, L.I., Gor'kov, L.P., Lebed', A.G., Europhys. Lett., *4*, 941 (1987).
- ² The pressures that can be attained in this plastic cell are currently limited by the gasket configuration required for a given experiment. Optical measurements have been performed to 8.5 GPa, but the insulating layer necessary for electrical transport measurements presently limits these measurements to 1.5 GPa at millikelvin temperatures.
- ³ Hannahs, S.T., *et al*., Phys. Rev. Lett., *63*, 1988 (1989); Cooper, J.R., *et al*., *ibid*., *63*, 1984 (1989).

Figure 2. Magnetoresistance of (TMTSF)2PF6 showing the rise in resistance at n=0. Measurements were made at 0.8 GPa and T= 350 mK. The origin of the frequency and field dependence at higher fields has not been ascertained, but may be intrinsic or an experimental artifact. Shots to 60 T closely track this data and display no additional anomalies.

Conference and Workshop Activity

Strongly Correlated Electrons Workshop

March 12-14, 1998 NHMFL and DoubleTree Hotel Tallahassee, Florida

This workshop is the continuation of a series of meetings that have taken place in recent years in Switzerland and Japan, and now will be held in the United States for the first time. The goal of these workshops has been to bring several leaders of the field of strongly correlated electrons together in a pleasant atmosphere for discussions. It is by invitation only, with a core list of participants that has remained constant over time, plus a group of new speakers every year.

VIIIth International Conference on Megagauss Magnetic Field Generation and Related Topics (Megagauss VIII)

October 18-23, 1998 **NHMFL**

Tallahassee, Florida

Complete Registration Information: *http://www.magnet.fsu.edu/ whatsnew*

Plans are progressing for the VIIIth International Conference on Megagauss Magnetic Field Generation and Related Topics slated for mid-October 1998 and hosted by the NHMFL. Over 200 scientists from around the world—including approximately 60 Russians—are expected to gather in Tallahassee to discuss the latest results on the generation of very high magnetic fields at the megagauss level and higher.

WORKSHOPS continued on page 14

Confirmed plenary speakers include:

- C.M. Fowler, LANL, on "Explosive Flux Compression: A Review of 50 Years of Los Alamos Activities,"
- Victor D. Selemir, VNIIEF, Russia, on "Investigation of Magnetocumulative Power Sources and Ultra High Magnetic Fields in VNIIEF," and
- Nobel Laureate J. Robert Schrieffer, NHMFL Chief Scientist, on "Science in Ultra High Fields."

Registration materials will be mailed and available via the NHMFL website by March 1, 1998. The preregistration fee is \$450 by August 1; late registration will be accepted at a higher rate. For further information, check out the website or contact the conference coordinator: Ysonde Jensen, 850-644-0807; *Megagauss@magnet.fsu.edu*; (fax) 850-644-0867. Interested participants also should note related information regarding the PPHMF-III conference.

Physical Phenomena at High Magnetic Fields (PPHMF-III)

October 24-27, 1998 NHMFL Tallahassee, Florida

14

Complete Information & Registration: *http://www.magnet.fsu.edu/whatsnew*

Megagauss VIII and PPHMF-III are planned back-to-back intentionally, because many of the interesting science and research developments in these areas overlap. Consequently, the agendas for the two conferences (specifically, the last day of

Megagauss VIII and the first day of PPHMF-III) are being developed to offer attendees new opportunities for exploration and discussion.

PPHMF-III, which is held just once every three years, is anticipating over 175 participants and forty speakers. The program will include invited and contributed papers as well as posters, covering a broad range of materials and phenomena, including semiconductors, magnetic materials, superconductivity, organic solids, quantum Hall effect, chemical and biological systems, and the technological use of high magnetic fields.

The deadline for abstracts and pre-registration is March 31, 1998. For further information, check out the website or contact the conference coordinator: Mary Layne, 850-644- 3203; *layne@magnet.fsu.edu*; (fax) 850-644-5038.

Yesterday's Technology Meets Tomorrow's

On October 29-31, 1997, the NHMFL hosted the 52nd revival of the Glidden Antique Car Tour. Over 350 remarkably maintained classic vehicles built before 1935 made an official stop at the NHMFL in Innovation Park. The list of entrants—from 33 states—included a 1914 Hudson; 1913 Fiat; 1910 Oakland; 1913 Rambler; 1911 and 1912 Steven-Duryeas; four Pierce Arrows, including a 1917 model; a 1929 Duesenberg; many early Fords; and several Marmons, Packards, Rolls Royces, and Auburns. The vintage vehicles were on display in the laboratory parking lot, as the estimated 800 drivers and guests visited the laboratory.

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INSIDE THIS ISSUE

WINTER 1998 **Volume 5 • No. 1**

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