NATIONAL HIGH MAGNETIC FIELD LABORATORY

REPORTS

SPECIAL EDITION

VOLUME 13 • NO. 1 • 2006

Reviewing the

2005 NHMFL RESEARCH REPORTS

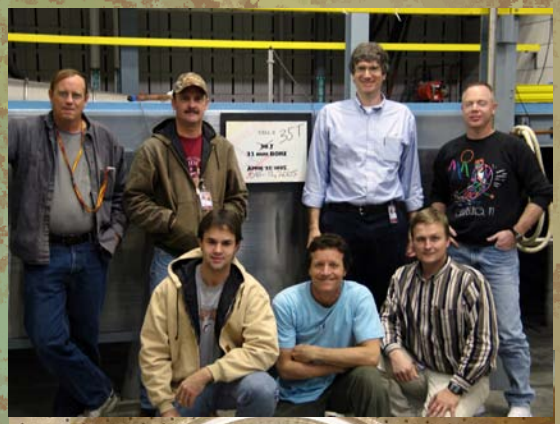
RESEARCH HIGHLIGHTS FROM

Life Sciences

Chemistry

Magnet Science & Technology

Condensed Matter



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NHMFL REPORTS

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PUBLISHED BY:
NATIONAL HIGH MAGNETIC FIELD LABORATORY
1800 EAST PAUL DIRAC DRIVE
TALLAHASSEE, FLORIDA 32310-3706

DIRECTOR:
GREG BOEBINGER

DIRECTOR, PUBLIC AFFAIRS:
SUSAN RAY

EDITING AND WRITING:
KATHY HEDICK & CECI BELL

ART DIRECTION AND PRODUCTION:
WALTER THORNER

www.magnet.fsu.edu

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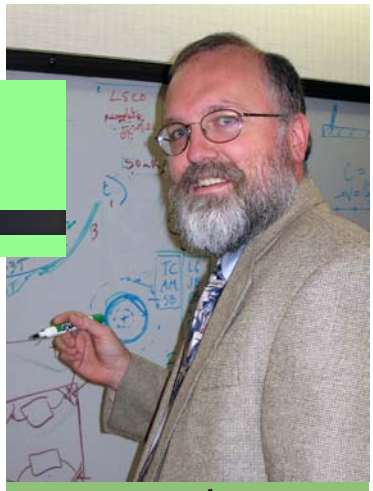
FROM THE DIRECTOR'S DESK



Greg Boebinger

2006 begins with news that **the National Science Board has decided to renew the award for the Magnet Lab** rather than to run a competition for the right to operate the nation's only facility for research in high magnetic fields. This decision comes at the conclusion of a process that began almost three years ago with the National Research Council's commissioning of the Committee on Research Opportunities in High Magnetic Fields. The resulting "COHMAG" report expresses the broad scientific merit and excitement of high magnetic field research. An NSF Blue Ribbon Panel visited the MagLab several times during 2005, which resulted ultimately in the decision to renew. With the NSF decision to renew our award, we intend to compete anyway—compete against our own record of success; compete to support the best science in the world by offering state-of-the-art techniques and instrumentation; and compete for the resources necessary to develop new facilities, magnet systems, and services to increase and expand our multidisciplinary user community.

More welcome news came in the report of the NSF Site Review Committee, which met at the laboratory in December 2005. The Committee noted that "About $7.5 million of facility upgrades, funded by the state of Florida, are scheduled to be completed by June 2006. **These upgrades will provide significant benefits to our DC Magnet User Program** by replacing obsolete and worn equipment with an updated and greater capacity to generate high magnetic fields. Over the last decade, the MagLab has developed and brought into service for users a broad array of magnets. **The [Site Review] Committee commends the MagLab for its ongoing program of continuous maintenance and upgrades, which greatly benefit users.**"

We are grateful for both of the strong endorsements we have received for much of what we do, as well as for the pointed advice for what can be improved.

In this spirit, I am also pleased to announce the membership of the **External Advisory Committee** that will take us through the renewal proposal **(see table, next page).** I am confident that the strength, diversity, and range of expertise represented by the members of this committee will serve the MagLab and its user programs very well.

The laboratory is always seeking to maintain effective communication with our users. The Users Committee http://www.magnet.fsu.edu/users/committees/ maintains our most effective formal dialogue, but equally valuable are the informal communications via our on-site user support, and scientific collaborations among user program and MagLab scientists. As experimental techniques become more sophisticated, we anticipate that the more collaborative mode of user support will become more common. To publicize the expertise of our MagLab scientists, **upcoming issues of**

MagLab External Advisory Committee February 2006	
Magnet & Magnet Materials Technology	
Phil Heitzenroeder	Princeton Plasma Physics Laboratory
Alexis Malozemoff	American Superconductor Corp
Ronald Scanlan	Lawrence Berkeley National Laboratory
Steve Gourlay	Lawrence Berkeley National Laboratory
Science: Biology & Chemistry	
Jean Futrell	Battelle
Robert Griffin	Massachusetts Institute of Technology
David Hendrickson	University of California, San Diego
Stanley Opella	University of California, San Diego
Science: Condensed Matter	
Meigan Aronson	University of Michigan
Dimitri Basov	University of California, San Diego
Paul Chaikin	Princeton University
William Halperin, **Chair**	Northwestern University
Peter Littlewood	Cavendish Laboratory
Cynthia Roberta McIntyre	Renssalaer Polytechnic Institute
Douglas Osheroff	Stanford University
Philip Phillips	University of Illinois-Urbana Champaign
Mansour Shayegan	Princeton University
Frank Steglich	Max-Planck Institute for Chemical Physics of Solids
Horst Störmer	Columbia University
Nai-Chang Yeh	California Institute of Technology
Users Committee Chair – Ex Officio	
James Valles	Brown University

this newsletter will feature these critically important people who, together with our users, account for the diversity and productivity of the MagLab. As the renewal proposal is under development, we hope to communicate to as wide a community as possible the research interests and experimental techniques that the MagLab scientists have developed.

This issue, we are extremely proud to feature the **2005 Research Highlights**. At yearend, we received a record number of 416 research reports from users and Magnet Lab faculty. *All* of the reports were reviewed by MagLab facility or department directors and are available on our Web site http://www.magnet.fsu.edu/. (Click the *2005 Annual Report* image, then use the pull-down menus to view reports by facility, category, PI, or first author.) In January, the **Magnet Lab Science Council, comprising Lev Gor'kov (chair), James Brooks, Rafael Bruschweiler, David Larbalestier, Albert Migliori, Carol Nilsson, and Glenn Walter,** conducted a second review to identify research activities that were particularly noteworthy and representative of the strength, breadth, and interdisciplinary nature of all of the reports. It is these 30 reports that appear in this newsletter as Research Highlights of the outstanding science being conducted by our users and staff across all three campuses of the MagLab.

Let me know what *you* think !

Greg

Greg Boebinger

LIFE SCIENCES

Including: BIOCHEMISTRY & BIOLOGY

Most biological macromolecules function when small molecules known as substrates diffuse into the vicinity of a macromolecule that "recognizes" the substrate. The macromolecule attracts the substrate to an "active site" on its surface where some biological function is carried out and the "substrate" is converted to a "product" before being released. Nuclear magnetic resonance or NMR spectroscopy is a powerful technique for studying molecular structure and molecular motions, but functional processes are not often studied in a direct fashion because we rarely have the opportunity to flow substrates through our small NMR samples. **Prof. Alex Smirnov at North Carolina State University** has worked with the NHMFL (E.Y. Chekmenev *et al*.) to develop a way to solve this challenge. By using thin wafers formed by an array of nanotubes (anodic aluminum oxide) that trap the biological macromolecules, it is possible to change the solvent conditions without reforming the NMR sample. Here ions have been added to the sample, NMR signals are seen to be shifted and when the ions are removed by using a molecular sponge, the signal returns to its original frequency with little loss of intensity. This demonstration will be followed by the straight-forward development of a flow probe such that the sample does not need to taken out of the NMR magnet. It can be expected that the function of a great many biological systems could be studies by this elegant application of a nano-material to facilitate biological studies.

*This research was supported by the **NHMFL Visiting Scientist Program**, which provides financial assistance for new users and early career researchers. VSP pays special attention to increasing the diversity of the MagLab user program.*

FLOW-THROUGH NANOTUBE ARRAYS FOR STRUCTURE-FUNCTION STUDIES OF MEMBRANE PROTEINS BY SOLID-STATE NMR SPECTROSCOPY

E.Y. Chekmenev (NHMFL); A.M. Alaouie (NCSU, Chemistry); P.L. Gor'kov (NHMFL); A.I. Smirnov (NCSU, Chemistry); T.A. Cross (NHMFL/FSU, Chemistry and Biochemistry)

Introduction

Structure determination of membrane proteins remains one of the most difficult problems of structural biology and biochemistry today. One of the principal advantages of NMR spectroscopy over other methods is its ability to study proteins embedded in phospholipid bilayers—a natural environment for membrane proteins. Nanopore-confined phospholipid bilayers introduced recently provide an attractive alternative way for aligning membrane protein samples for solid-state NMR experiments. We studied, for example, the utility of such experiments of an ultra-high magnetic field (19.6 T) NMR ^{17}O anisotropic chemical shift study of ion binding to the gramicidin A (gA) ion channel.

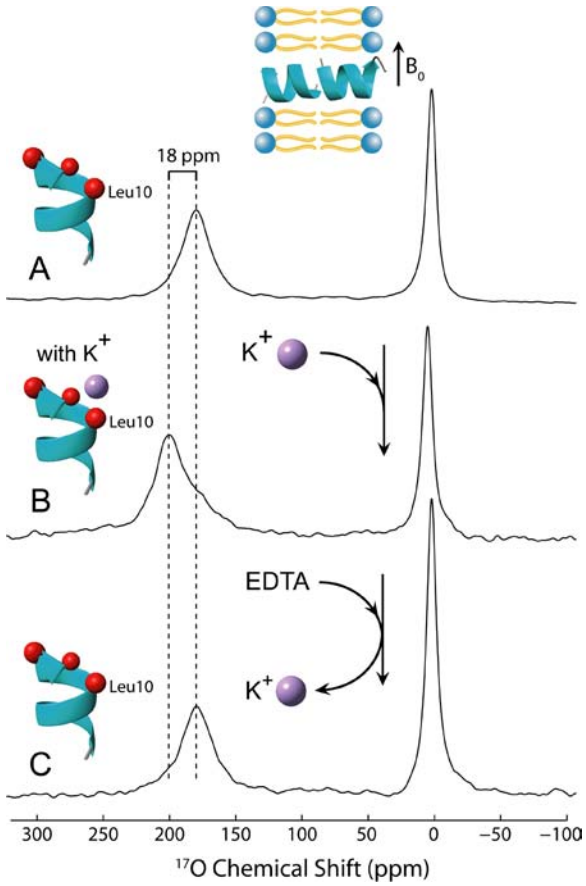


Figure 1. ^{17}O NMR spectra of ^{17}O-[D-Leu10]-gA uniformly aligned in DMPC bilayers in the absence and the presence of KCl (2.4 M) acquired at 40 ºC with at 19.6 T.

Results and Discussion

The Leu10 carbonyl in the gramicidin A pore is one out of three carbonyl sites involved in K^+ binding. We have monitored K^+ binding from the ^{17}O resonance of selectively labeled Leu10 carbonyl when gramicidin A dimers were incorporated into DMPC bilayers and aligned by our Anodic Aluminum Oxide (AAO) method. AAO substrates were positioned with the nanopores aligned along B_0 so the gramicidin A pores would be approximately perpendicular to B_0 (Figure 1, cartoon on top). When DMPC bilayer is in a fluid bilayer phase (i.e., T>24 °C), the tumbling of gA around the pore axis is fast and therefore its NMR spectrum is largely dictated by the average of the two components of the chemical shift tensor, δ_{22} and δ_{33} and the resonance appears at about $\delta=(\delta_{22}+\delta_{33})/2\approx180$ ppm. Quadrupole relaxation largely determines the width of the ^{17}O resonance that is 15-25 ppm at 19.6 T. Since the surfaces of nanotubular bilayers are fully accessible to water soluble molecules, we are able to study reversible effects of K^+ binding to the gramicidin A channel from ^{17}O chemical shift using the same sample without losing its macroscopic alignment. When the sample was exposed to 2.4 M KCl to yield the double occupancy of K^+ in the dimeric channel, or one ion per monomer, a downfield shift of $\Delta\delta\approx18$ ppm was observed (Figure 1B). When K^+ was removed by EDTA wash, the resonance returned to its original position (Figure 1C). Note, that 0 ppm peak is due to natural abundance ^{17}O water signal. We believe that a large number of solid-state NMR-based studies would benefit significantly from the method we described here. Specifically, for the first time solid-state NMR could be used to monitor structural changes in membrane proteins in pH and other titration experiments.

Conclusions

The aligned nanoporous network of AAO substrate provides the advantages of fast accessibility by solute molecules, reversible ion exchange, and titration experiments with physically the same sample of membrane proteins. Thus, one achieves better control of the bilayers by exposing the lipid nanotubes to various solutions and fine-tuning the bilayer properties.

Much of the chemistry conducted by biological macromolecules is initiated at oxygen atoms in these structures and yet one of the most powerful tools available in science has not been able to directly observe these oxygen sites. This tool, known as nuclear magnetic resonance or NMR spectroscopy, is closely related to magnetic resonance imaging (MRI) used commonly in the medical profession. The most common isotope (or variety) of oxygen that is observable by NMR is ^{17}O and spectra of this isotope are very complex. For years NMR spectroscopists have been recording spectra of carbon, hydrogen and nitrogen—the other common atoms in biological systems—in an effort to understand the chemistry occurring at the oxygen sites. Part of this complexity is dependent on the strength of the magnetic field used: the higher the field, the less complex the spectra. The NHMFL's ultrawide bore 21.1 tesla NMR magnet is being used to develop 17O spectroscopy in collaboration with **Prof. Richard Wittebort at the University of Louisville**. Here 17O spectra of small molecules and biological macromolecules are shown. In particular, a molecule that conducts ions across cell membranes (gramicidin A) is being studied to understand how this molecule facilitates this activity. In E.Y. Chekmenev *et al.*, the authors show that the carbonyl oxygens that line the pore of this ion channel interact strongly with ions as they are attracted to the channel and passed from one side of the membrane to the other. The details of *how* the ionic charge is absorbed by the carbonyls are being investigated with this unique tool.

This research was published in J. Am. Chem. Soc., **127**, 11922-11923 (2005).

TOWARDS ^{17}O SOLID-STATE NMR SPECTROSCOPY OF ION-SELECTIVE CHANNELS AT ULTRA-HIGH MAGNETIC FIELDS

E.Y. Chekmenev (NHMFL); L.N. Miller, Y. Mo (FSU, Chemistry and Biochemistry); P.L. Gor'kov (NHMFL); R.J. Wittebort (Univ. of Louisville, Chemistry); K.W. Waddell, (Vanderbilt University, Institute of Imaging Science); T.A. Cross (NHMFL/FSU, Chemistry and Biochemistry)

Introduction

^{17}O, spin 5/2 quadrupole nucleus, has recently been extensively employed to study ion-binding in proteins due to high sensitivity through its quadrupolar coupling (QC) and chemical shift (CS) to the intermolecular interactions. While ^{17}O spectroscopy is considered difficult, advent of high magnetic fields potentially allows functional studies in large ion channels, the Holy Grail for many biochemists today. Here, we provide the progress report on ^{17}O work conducted at the NHMFL when the 21.2 T NMR spectrometer became available.

Results and Discussion

Ultra-high magnetic fields became available recently, and as a result much of the ^{17}O results to date are largely obtained from relatively small molecules such as individual amino acids. Recently we were able to demonstrate that ion binding significantly affects both CS and QC of carbonyl oxygen in polycrystalline peptides (Figure 1-top spectra). Moreover, it was found that ^{17}O is a significantly more sensitive probe for ion binding than the widely used ^{15}N nuclei of the peptide backbone. We also studied ion binding by ^{17}O anisotropic CS in the cation conductive pore of gramicidin A, the binding site

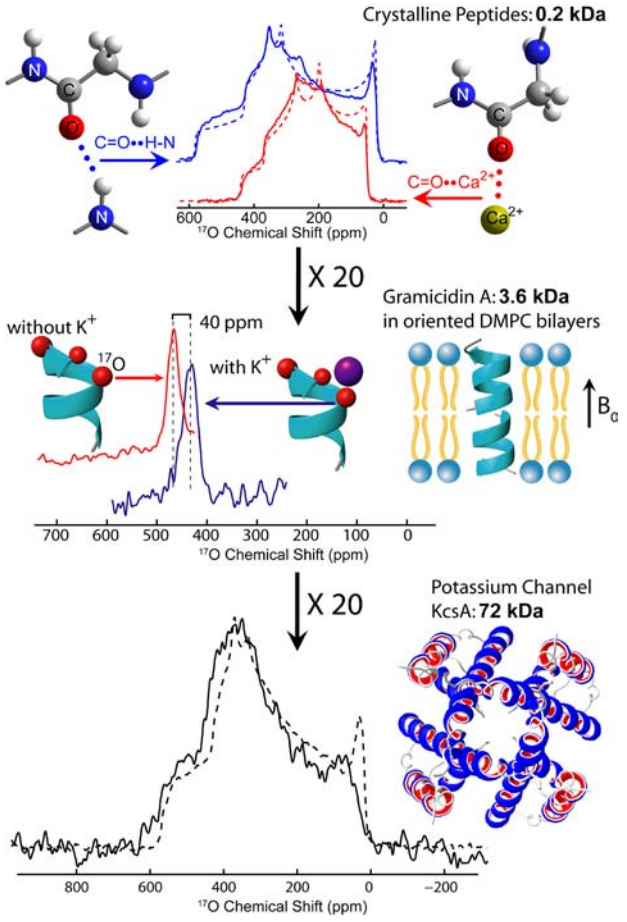


Figure 1. Progress in solid-state ^{17}O NMR spectroscopic studies of ion channels at high field. The top spectrum is acquired at 19.6 T, while the others were obtained from the ultra-wide bore 21.2 T spectrometer.

of which has similar intermolecular interactions that contribute to the biologically important function of highly selectivity and high conductance rates in ion channels. While the sensitivity is always a challenge for NMR spectroscopy, we take advantage of high fields to aid the sensitivity in addition to high ^{17}O isotopic enrichment (~60%), favorable relaxation and orienting the channels, which resulted in reducing the line width from >500 ppm to ~25 ppm, a 20 fold reduction. The insights gained from ion binding effects on CS in the relatively small gramicidin A pore (Figure 1-middle) helps potentially to approach the KcsA potassium channel. The preliminary results (Figure 1-bottom, solid line) suggest librational motions have negligible effects. CS tensor span and CS distribution is similar to those observed in crystalline solids (Figure 1-bottom, dashed line, polycrystalline Gly-Gly-Gly).

Conclusions

Solid-state ^{17}O NMR spectroscopy is a sensitive and accurate probe for ion binding to proteins. Our preliminary results obtained in a full length 72 kDa bundle of KcsA suggest that ^{17}O can be successfully applied to study the mechanism of ion solvation in ion-selective channels, including KcsA, in the native membrane environment.

While biologists have paid a great deal of attention to how biological macromolecules are structured, their attention is now turning to the motions that these molecules undergo in their native environment. Indeed, the motions are critical for achieving the biological function of these molecules. **Professor Dorothee Kern at Brandeis University** has been correlating molecular motions of biological catalysts with their biological function. By studying the motion for the enzyme with and without its substrate bound, the authors (W. Labeikovsky *et al.*) have demonstrated that the molecular motions necessary for the functional activity are present in the catalyst even without the substrate being present. This suggests that the protein is designed not only for its structure, but also for its molecular motions. How proteins are designed for specific motions is not known, but as a result of Dr. Kern's research it is a question that the protein community is beginning to think about.

This research by Dr. Kern continues her investigations reported in last year's annual report and recently published in Nature, **483**, 117-121 (2005).

STRUCTURAL REARRANGEMENTS OF THE PIN1 PROLYL ISOMERASE DURING CATALYSIS

W. Labeikovsky, E.Z. Eisenmesser (Brandeis, Biochemistry); D. Kern (Brandeis/HHMI, Biochemistry)

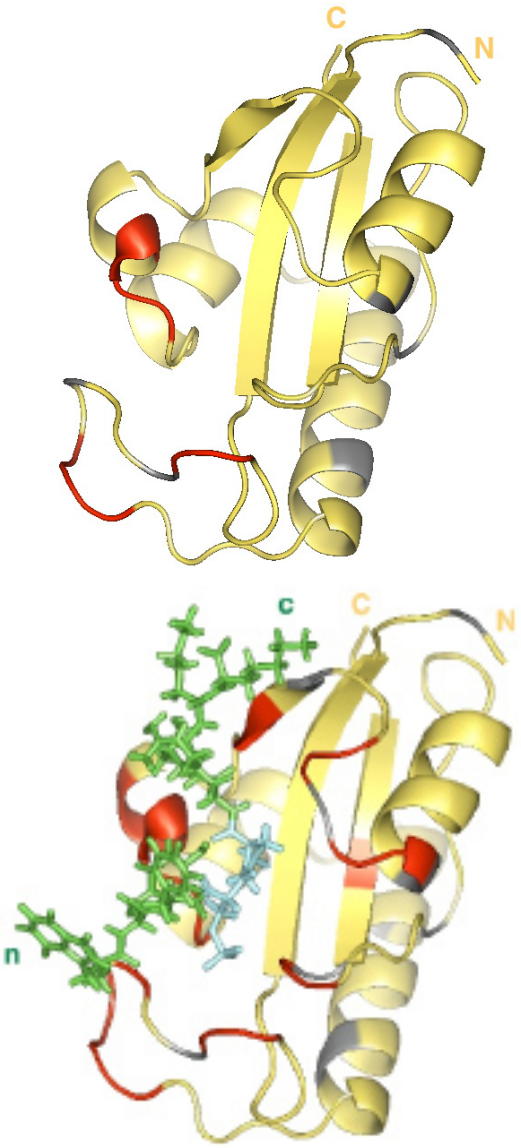
Introduction

Prolyl isomerases (PPIases) catalyze the cis-trans isomerization about the prolyl peptide bonds. Pin1 is unique among prolyl isomerases in that it specifically isomerizes phosphorylated Ser/Thr-Pro (pSer/pThr-Pro) bonds. The acceleration of this isomerization may serve as a second regulatory mechanism in addition to phosphorylation in the form of a kinetic control. Known substrates of Pin1 include a subset of cell cycle proteins (e.g. NIMA, Cdc25, Cyclin D) as well as the Alzheimer protein Tau and the transcriptional activator p53; however, the role of a conformational switch and the mechanism of its regulation by Pin1 is not understood. Here we perform structural and dynamic NMR experiments on Pin1cat during catalysis of a true substrate. Intermolecular NOE data collected during turnover of the peptide substrate together with backbone dynamics measured during catalysis reveals insight into the reaction trajectory. Conformational exchange in the free enzyme similar to the one observed during turnover suggests the pre-existence of dynamics.

Experimental

NHMFL Varian 720 and 600 solution magnets were used in this study.

Results and Discussion



Backbone ^{15}N probes exhibiting conformational exchange in the free enzyme (top) and in the saturated, turning-over complex (bottom) are highlighted in red. Similar conformational exchange suggests preexisting functional protein motions in the free enzyme.

Acknowledgements

We thank Jack Skalicky and Ashley Blue at NHMFL for magnet time. We would also like to thank Lewis Kay, Erik Zuiderweg, and Robert Konrat for pulse sequences and Dmitry Korzhnev for global-fitting code as well as Ming Lei for computational structure modeling tips.

IT The dynamics of molecular motors can be studied by labeling with heavy hydrogen atoms and high resolution mass spectrometry. The technique provides detailed structural snapshots of the protein complex, which complement the static structures obtained by X-ray crystallography. In the work of **Prof. R. Tuma of the University of Helsinki** (J. Lisal *et al.*), the technique was applied to a viral motor that is activated by transport of genetic material through its outer shell. Knowledge of the functional mechanisms of viral proteins can define structural targets for intelligent drug design.

This research was published in Nature Structural & Molecular Biology, **12**, 460-466 (2005).

FUNCTIONAL VISUALIZATION OF A VIRAL MOLECULAR MOTOR BY HYDROGEN-DEUTERIUM EXCHANGE REVEALS TRANSIENT STATES DURING RNA PACKAGING

J. Lisal (U. Helsinki, Biology); T.T. Lam (NHMFL); D.E. Kainov (U. Helsinki, Biology); M.R. Emmett (NHMFL/FSU, Chemistry and Biochemistry); A.G. Marshall (NHMFL/FSU, Chemistry and Biochemistry); R. Tuma (U. Helsinki, Biology)

Results and Discussion

Molecular motors undergo cyclical conformational changes and convert chemical energy into mechanical work. The conformational dynamics of a viral packaging motor, the hexameric helicase P4 of dsRNA bacteriophage ϕ8, may be visualized by hydrogen-deuterium exchange and high-resolution mass spectrometry (with NHMFL's homebuilt 9.4 T electrospray ionization FT-ICR mass spectrometer). Concerted changes of exchange kinetics revealed a cooperative unit that dynamically links ATP binding sites and the central RNA binding channel. The cooperative unit is compatible with structure-based model in which translocation is affected by a swiveling helix. Deuterium labeling also revealed the transition state associated with RNA loading that proceeds via opening of the hexameric ring. The loading mechanism is similar to that of other hexameric helicases. Hydrogen-deuterium exchange provides an important link between time-resolved spectroscopic observations and high resolution structural snapshots of molecular machines.

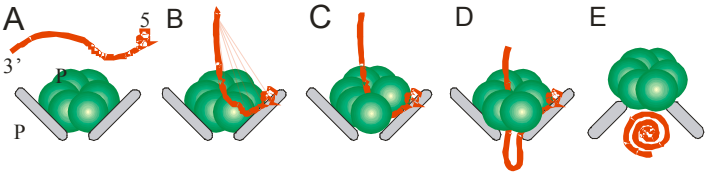


Figure 1. Schematic representation of the progression of single-stranded RNA as it passes through the hexameric pore helicase (204 kDa). H/D exchange reveals that the RNA squeezes through a cleft between adjacent monomers of the hexamer.

Acknowledgements

We thank Ayman Abu Ramadan for the software development. J.L. is supported by Viikki Graduate School in Biosciences, D.E.K is supported by the National Graduate School in Informational and Structural Biology. This work was supported by Academy of Finland grant 206926 (RT), the Finnish Centre of Excellence Program 2000-2005, the NSF National High Field FT-ICR Mass Spectrometry Facility (DMR-00-84173), Florida State University, and the National High Magnetic Field Laboratory in Tallahassee, FL.

References

1 Lisal, J., *et al*., *Nature Struct. Mol. Biol*., **12**, 460-466 (2005).

CHEMISTRY

Including: CHEMISTRY, MAGNETIC RESONANCE TECHNIQUES & GEOCHEMISTRY

Hawaii is the largest volcanic structure on Earth and its height above the seafloor is larger then the height of Mt. Everest. Last year, this group of researchers led by V. Salters of the NHMFL reported on the discovery of depleted material (depleted in elements that prefer a melt) of cumulate rocks that were carried by the basalts to the surface. Their studies this year (M. Bizimis *et al*.) have shown how extensive the occurrence of this component is. The report presented here discusses the identification of long term (in excess of one billion years) depleted mantle material in the Hawaiian peridotites (solid material).

Readers may also be interested in the complimentary research of **J. Blichert-Toft of Ecole Ecole normale superieure de Lyon** and V. Salters, which is available online at *http://www.magnet.fsu.edu/publications/2005annualreport/*.This study shows convincing evidence for the presence of a depleted component in the source of basalts (melts) from the Koolau volcano on Hawaii, although the Koolau basalts are the most enriched on Hawaii. The evidence for this depleted material lies in the radiogenic isotope systems that record the long term history of the magma sources. The identification of the depleted material is difficult, as by its nature the depletion is not easily recognized in melts. Implications of this finding are that the heterogeneities, observed in basalts, represent only a fraction of the heterogeneity present in the source of the basalts. Additionally, these findings show that mantle material can undergo several episodes of melting without refertilization between melting episodes.

THE HAWAIIAN PLUME IS REPLACING THE PACIFIC LITHOSPHERE: EVIDENCE FROM PERIDOTITE XENOLITHS FROM OAHU AND KAUAI

M. Bizimis, V.J.M. Salters (NHMFL, Geochemistry); G. Sen (FIU, Earth Sciences); J.C. Lassiter (UT Austin, Geological Sciences)

Introduction

Subduction of basaltic oceanic crust is a key mechanism for introducing chemical heterogeneities into the Earth's mantle. Together with the crust, however, the associated depleted lithosphere is also subducted. In the mantle, basaltic crust has a lower solidus while the depleted lithosphere has a higher solidus temperature than the ambient mantle. Thus, if a recycled depleted lithosphere is part of an upwelling plume, it could survive melting and be more easily recognized in mantle xenoliths associated with

plume volcanism, than its basaltic counterpart. Here, we report on new isotope data from peridotite xenoliths from Oahu and Kauai, and we show that plume material of recycled origin is recognized in these peridotites.

Results and Discussion

Isotope and trace element analyses were performed at the Geochemistry Division, NHMFL, using a Finnigan MAT-262 TIMS, the ISOLAB (a modified VG 54 MS with Hot-SIMS) and a Finnigan ELEMENT ICP-MS. Two Kauai peridotites overlap the post erosional Kauai lava compositions in Sr-Nd isotope space. A third Kauai peridotite has higher $^{87}Sr/^{86}Sr$ (0.70422) and lower $^{143}Nd/^{144}Nd$ (0.51291) than any published Kauai lava and extends towards more radiogenic Sr (for a given Nd) than the Hawaiian lavas in general (Figure 1). This sample has depleted characteristics with high Mg# (0.925) and Cr# (0.31), and low HREE in cpx. It also has the highest La/Yb ratio and largest Ti depletions yet determined in Hawaiian peridotites. In contrast, three peridotites from SLC have less radiogenic $^{87}Sr/^{86}Sr$ for a given $^{143}Nd/^{144}Nd$ than all other analyzed Hawaiian peridotites and lavas (Figure 1). One peridotite straddles the so-called *LoNd* array, thought to represent mixing between HIMU and EM-I mantle components. In Hf-Nd space, this peridotite falls below the Hawaiian lava array having lower $^{176}Hf/^{177}Hf$ (0.28287) than the most isotopically enriched Koolau lavas suggesting that both depleted and enriched compositions are present in mantle peridotites, perhaps alleviating the need for an enriched pyroxenitic lithology in the Hawaiian plume. The new peridotite data shows greater Sr and Nd isotope variation in the Hawaiian peridotites than previously thought, and supports our conclusions from the Hf-Os systematics for a highly heterogeneous lithosphere. The total Sr-Nd-Hf isotope variability in the Oahu and Kauai peridotites cannot be explained by metasomatism of the Pacific lithosphere with erupted Hawaiian - type melts.

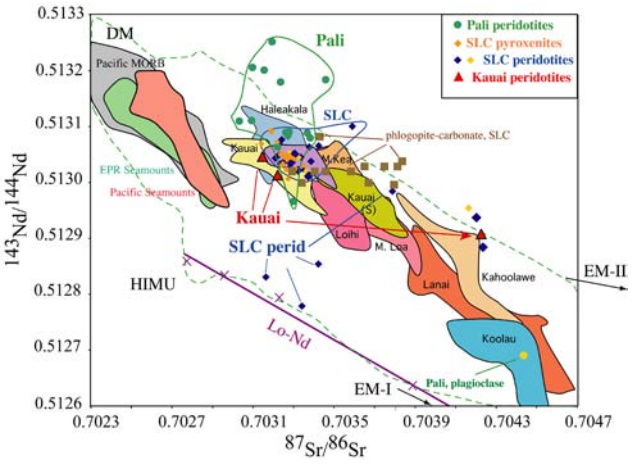


Figure 1. Sr-Nd isotope compositions of Oahu and Kauai peridotites compared with data from the Hawaiian lavas and Pacific MORB and seamounts.

Conclusions

We propose that the observed isotope heterogeneity in the Oahu and Kauai peridotites reflects the presence of plume-derived peridotites within the Pacific lithosphere. The Pali peridotites have compositions compatible with the 90 million year old Pacific lithosphere beneath Oahu. The SLC are best explained as fragments of an ancient (>1 billion year old) recycled mantle lithosphere that is brought to the surface by the Hawaiian plume. The similar temperatures of last equilibration of both the SLC and Pali peridotites (800-1000°C) suggests that plume and Pacific mantle peridotites are juxtaposed beneath Oahu at depths as shallow as 60 km. This provides independent evidence for thinning of the 90-100 km thick Pacific lithosphere by the Hawaiian plume, downstream from the present day plume center under the Big Island. The preliminary Kauai data suggests that such heterogeneity could be observed in the Kauai lithosphere. Additional analyses on Kauai peridotites will test the lateral extent of replacement of the lithosphere by plume-derived material.

Heterogeneous catalysts composed of metal ions supported on an oxide substrate represent an important class of industrial catalysts. Notwithstanding their industrial utility, the active sites that gives rise to the catalytic activity in many of these materials is poorly understood due to their low concentration on the surface. It has long been suggested, however, that they are metal-ligand multiple bonded species such as imido, M=NR, or akylidene, M=CR2, groups that form on the surface. In this study, C. Brown et al. supported vanadium oxide on silica is reacted with aniline to produce a catalytic site. The surface species was unambiguously characterized as a phenylimido species, V=NPh, through the use of $^{51}V/^{15}N$ rotational echo double resonance (REDOR) NMR spectroscopy. This technique allowed the identification of V-N bond formation even at low concentrations. When used in conjunction with proton dephasing studies and vibrational spectroscopy, the formation of a vanadium imido group on the surface could be unambiguously established.

This research was published in the Journal of the American Chemical Society, **127**, (33),11590-11591 (2005).

CHARACTERIZATION OF REACTIVE SITES IN SUPPORTED CATALYSTS BY $^{51}V/^{15}N$ ROTATIONAL ECHO DOUBLE RESONANCE NMR SPECTROSCOPY: FORMATION OF PHENYLIMIDO GROUPS AT SURFACE-BOUND OXOVANADIUM SITES

C. Brown, R. Achey (FSU, Chemistry and Biochemistry); R. Fu (NHMFL); T. Gedris (FSU, Chemistry and Biochemistry); A.E. Stiegman (FSU, Chemistry and Biochemistry)

Introduction

Surface-supported transition metals containing multiply bonded ligand systems such as akylidene and imido groups have long been proposed as key intermediates in heterogeneous catalytic reactions such as olefin metathesis, polymerization, and ammoxidation. In spite of the importance of these proposed species, their positive characterization on catalyst surfaces has been difficult due to their low concentration and transient nature. We report here the generation of metal-imido functional groups at discrete silica-supported vanadium-oxo sites under catalytic conditions. Specifically, vanadium-oxo groups in a silica xerogel matrix react cleanly and quantitatively with aniline in a gas-solid reaction to generate a phenylimido group at the vanadium center with concomitant elimination of water.

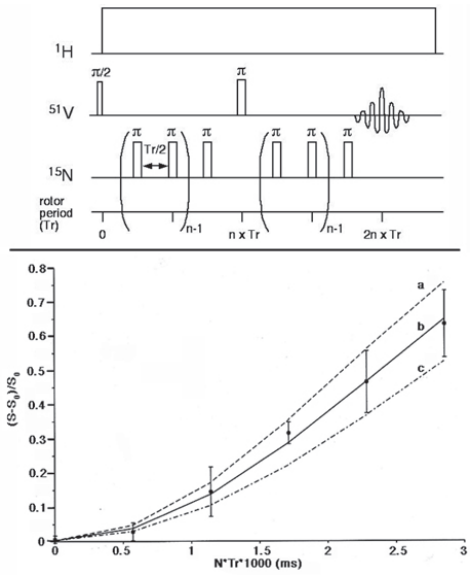
Experimental

Characterization of metal-imido bond formation was carried out using $^{51}V/^{15}N$ rotational echo double resonance (REDOR) NMR spectroscopy where the more sensitive quadrupolar ^{51}V nucleus was monitored while the ^{15}N nucleus was pulsed. While REDOR has been used to characterize a number of dense and porous inorganic solids and both one- and two-dimensional solid-state NMR has been used to characterize surface-grafted organometallic species, we believe this is the first use of REDOR to elucidate such sites.

Results and Discussion

Vanadium oxide supported on silica was fabricated using an established sol-gel derived procedure. Exposure of a monolithic 0.25 mol % V-silica xerogel to vapor phase aniline in a dry nitrogen flow showed systematic changes in the UV-Vis spectrum begin to occur between 120-175 °C with clean conversion to a distinct product observed.

Rotational echo double resonance spectroscopy (REDOR) was used to establish the presence of direct covalent bonding between the V and the N. Because of the low concentration of sites present on the surface, the REDOR fraction was measured by monitoring the more intense ^{51}V NMR signal, while applying two π pulses per rotor period to ^{15}N using the pulse sequence shown in Figure 3a. The ^{51}V echo was measured with and without the ^{15}N pulses and the REDOR fraction, $\Delta S/S_0$, plotted as a function of rotor evolution. Consistent with a short covalent bond length, after 3 msec of dephasing 60 % of the signal is lost. The best fit of the data was with a dipolar coupling constant of 419 Hz. In small molecule analogs, vanadium(V)-nitrogen covalent lengths typically range from around 1.60-1.70 Å for arylimido groups and 1.9-2.0 Å for amido groups. Consistent with these covalent lengths the V-N bond length obtained from the fit was 1.96 (±0.09) Å. While this is longer than expected for an imide, it has been established that bond length estimates determined from fits to the REDOR experiment tend to be longer than the experimental distances found through x-ray crystallography. This has been attributed to molecular motion and thermal dynamics that may partially average the dipolar couplings between two spins.



Conclusions

Discrete catalytic sites containing metal-ligand multiple bonds have been generated on a silica surface. Specifically, vanadium-imido functional groups were generated by a gas-solid reaction.

Acknowledgements

Funding was provided by the Air Force Office of Scientific Research through MURI 1606U8.

Structure, dynamics, diffusion: There is no limit to the questions that you can ask about a sample material with nuclear magnetic resonance spectroscopy (NMR). Unfortunately, NMR is much less sensitive than other spectroscopies, so you won't obtain a good answer unless you have a lot of sample. The sensitive RF sample probe developed by a collaboration of **Bruker Biospin, Inc**., and NHMFL scientists (A.S. Edison *et al*.) is a significant step toward solving the NMR sensitivity problem for biological samples that are available only in small quantities. It combines the sensitivity gains of a small sample coil, the low noise possible at cryogenic temperatures, and the low loss of oxide superconductors to establish a new sensitivity benchmark.

*This research was supported by the **NHMFL In-House Research Program**, which funds approximately six projects per year that meet the criteria of scientific quality on par with other NSF grants and are designed to extend or improve a component of the Magnet Lab user program. This research has been accepted for publication by the* Journal of Magnetic Resonance.

ULTRA HIGH SENSITIVITY NMR: 1-MM HTS TRIPLE RESONANCE PROBE

A.S. Edison, J.R. Rocca (NHMFL/UF); W.W. Brey, S. Saha (NHMFL/FSU); R.E. Nast, R.S. Withers (Bruker Biospin, Fremont, CA)

We report a 600-MHz 1-mm triple-resonance high-temperature-superconducting (HTS) probe for NMR spectroscopy. The probe has a real sample volume of about 7.5 µl, an active volume of 6.3 µl, and appears to have the highest mass sensitivity at any field strength. The probe is constructed with 4 sets of HTS coils that are tuned to ^{1}H, ^{2}H, ^{13}C, and ^{15}N, and there is a z-axis gradient. The ^{1}H coil pair has two features that allow it to be placed very close to the sample: First, the resonators, based on two distributed interdigital capacitors, with a spatial periodicity of only 0.125 mm, place a very low fringing electric field on the sample. Second, the current-carrying fingers are extensively slit to reduce shielding currents that would reduce B_0 homogeneity. The ^{1}H coil pair also has a large height-to-width ratio to produce a homogeneous RF field. The ^{2}H and ^{13}C coils are spiral resonators. Lock sensitivity with such a small sample volume was given higher priority than carbon observe sensitivity, and for this reason the ^{2}H coils are in the second position from the inside, and the ^{13}C coils are in the third position. On the outside are the ^{15}N coils, also spiral resonators to achieve a resonance frequency of 60 MHz. Because the ^{15}N coils are far from the sample, their rather large electric field is acceptable. The simple spiral design, however, supports additional modes at approximate multiples of 60 MHz. Final frequency-trimming relied upon extensive simulations to prevent these modes from interfering with the other coils. The coils are cooled with a commercial Bruker CryoPlatform to about 20 Kelvin, and the sample chamber can be regulated above or below room temperature over a moderate range using a Bruker variable temperature unit. The S/N value of 292±28 for 0.1% ethylbenzene is approximately 3.5-fold less than a standard 600 MHz 5-mm triple-resonance probe (S/N ~1000) with about 70-fold less sample. Thus, the mass sensitivity of the 1-mm HTS probe is about 20 times greater than a conventional 5-mm probe. Commercial 5-mm Bruker 600 and 800 MHz cryoprobes have S/N values approximately 4,000 and 8,000, respectively. The 1-mm HTS probe has a mass sensitivity that is over 4 times that of a 5-mm cryogenic probe at the same field strength and over 2 times that of state-of-the-art 5-mm technology at 800 MHz.

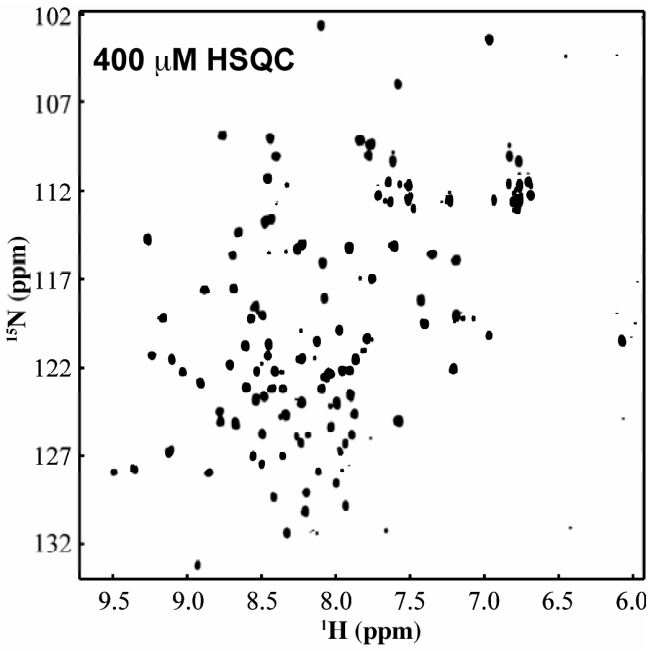


Figure 1. Experimental data from the 1-mm HTS triple-resonance probe. Data were collected using 400 µM ubiquitin using gradient 15N-HSQC and were recorded in 41 m using 16 scans and 128 t_1 increments. High quality data on 8 µL 1 mM samples can be obtained in about 10 min (not shown).

The extremely high mass sensitivity of this probe suggests that it may be useful for metabolomics, natural products, and protein screening applications. Figure 1 demonstrates the utility of this probe for small molecules and proteins with a 2D spectrum of 400 µM ^{15}N-labeled ubiquitin in 8 µl phosphate buffer (pH 5.5) with 10% D_2O for lock.

Acknowledgements

The author would like to acknowledge the support of Dr. Werner Mass and Bruker Biospin, Inc.

References

[1] Brey, *et al.*, *J. of Magn. Reson.*, **179**, (2006), in press.

As the global supply of "light sweet" crude oil is depleted by expanding energy needs, petroleum reserves must evolve toward heavier crude oils that are more difficult to process. Heavy petroleum is comparatively rich in polyaromatic hydrocarbons (PAHs), some of which contain heteroatoms such as nitrogen, sulfur, and oxygen. Variations in petroleum molecular composition directly impact the cost and efficacy of all refinery processes, and efficient removal of heteroatoms is required to minimize environmental impact. The first step toward efficient processing is a fundamental understanding of the types of molecules present in each oil. In this work (S. Kim et al.), NHMFL scientists team with **ExxonMobil** to characterize six similar oils that had undergone varying degrees of microbial degradation due to deposition at different depths below the Earth's surface. Changes in oil composition reveal new chemicals created by biodegradation and the preferred microbial food sources.

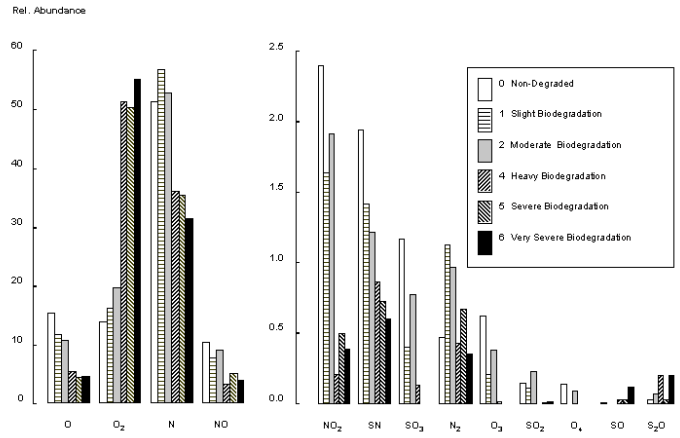
This research was published in Organic Geochemistry, **36**, 1117-1134 (2005).

COMPOSITIONAL CHARACTERIZATION OF THE MICROBIAL DEGRADATION OF PETROLEUM BY ELECTROSPRAY IONIZATION FT-ICR MASS SPECTROMETRY

S. Kim (NHMFL); L. Stanford (NHMFL/FSU, Chemistry and Biochemistry); R.P. Rodgers (NHMFL/FSU, Chemistry and Biochemistry); A.G. Marshall (NHMFL/FSU, Chemistry and Biochemistry); C.C. Walters, K.Qian, L.M. Wenger, P. Mankiewicz (ExxonMobil, Petroleum)

Results and Discussion

A suite of six genetically related oils that had experienced varying degrees of subsurface, anaerobic biodegradation are analyzed by ultrahigh-resolution 9.4 T Fourier transform ion cyclotron resonance mass spectrometry (FT-ICR MS). Electrospray ionization of whole oil samples selectively characterizes all neutral nitrogen compounds and acid NSO compounds, from ~300 to 900 Da, with unambiguous assignment of molecular formulae. Evidence for selective biodegradation is observed in all compound classes. NSO compounds associated with long alkyl side chains are removed, regardless of the NSO core, under conditions associated with moderate (saturated biomarkers unaffected) to severe biodegradation. Changes in aromaticitiy and alkyl distributions of the O_2 species result from simultaneous microbial degradation and generation. The ratio of acyclic to 2-4 ring cyclic O_2-species provides a new parameter to define the degree of biodegradation.



Acknowledgements

We thank D. McIntosh for machining all of the custom parts required for the 9.4 T instrument construction, M.R. Emmett and J.P. Quinn for design and construction of the microelectrospray source, and C.L. Hendrickson for help in optimizing instrument operating parameters. We also thank John Okafor, Phuc Nguyen, and Frank Chen of ExxonMobil Upstream Research Company for conducting the supporting geochemical analysis. Work supported by NSF (DMR-00-84173), Florida State U., and NHMFL. We thank Barry Bennett and a reviewer for their careful proofing and helpful suggestions.

References

[1] Kim, S., *et al.*, *Org. Geochem*, **36**, 1117-1134 (2005).

When exactly does something deviate from the norm? The laboratory mouse is the mammal of choice for exploring genetic variation and for modeling human biology. But to take advantage of the information emerging from mouse genome sequencing efforts, it has become necessary to systematically collect phenotypic information to establish a baseline of normal variability. As part of a worldwide effort, **H. Benveniste of Brookhaven National Laboratory** and colleagues (Y. Ma *et al.*) have constructed a three-dimensional (3D) probabilistic mouse brain atlas database derived directly from Magnetic Resonance Microscopy (MRM) images acquired at the AMRIS facility at the University of Florida. Using 10 excised adult brains of the laboratory mouse strain C57BL/6J, these researchers utilized a 17.6 T widebore magnet and homebuilt MRI coils to generate 3D gradient-echo datasets at an isotropic resolution of 47 μm. These datasets were co-registered, segmented into 20 distinct anatomical structures, and incorporated into three different atlas types: 10 single-specimen brain atlases, a minimum deformation atlas, and a probabilistic atlas. With these resources, genomics researchers can visualize the shapes and volumes of brain structures directly while registering their own data to the 3D space defined by the atlases. Furthermore, this atlas database readily provides quantitative structural information, such as a structure volume, surface area, and local geometric variations, which can be integrated into finer anatomical phenotyping efforts. And because of the inherent digital and 3D nature of the MRM-based atlases, this important resource can be made available on the Internet for widespread use by the scientific community: (*www.bnl.gov/CTN/mouse*).

This research was published in Neuroscience, **135**, 1203-1215 (2005).

A 3D DIGITAL ATLAS DATABASE OF THE C57BL/6J BRAIN BY MR MICROSCOPY

Y. Ma (BNL, Medical); P.R. Hof (Mount Sinai, Neuroscience); S.C. Grant, S. J. Blackband (UF, Neuroscience); R. Bennett, L. Slatest, M.D. McGuigan (BNL, Computer Science); H. Benveniste (BNL, Neuroscience)

Introduction

A comprehensive 3D digital atlas database of the C57BL/6J mouse brain was developed based on MR microscopy images acquired on a 17.6-T superconducting magnet. By using both manual tracing and an atlas-based semi-automatic segmentation approach, T2*-weighted magnetic resonance microscopy images of 10 adult male formalin-fixed, excised C57BL/6J mouse brains were segmented into 20 anatomical structures. The segmentation data were formatted and stored into a database containing three different atlas types: 10 single-specimen brain atlases, an average brain atlas, and a probabilistic atlas. Additionally, quantitative group information, such as variations in structural volume, surface area, MR microscopy image intensity and local geometry, were computed and stored as an integral part of the database.

Experimental

Intact, fixed adult C57BL/6J mouse brains were imaged at 17.6 T with a 3D gradient-echo sequence at an isotropic 47 µm isotropic resolution in 5.5 hrs. One of the brain datasets was chosen as a "representative brain," which was segmented manually into 20 neuroanotmical structures. Subsequent brains were co-registered to this brain and its structures through linear and elastic transformations by means of a guided semi-automatic process (see (1) for complete details). This process generated not only the 10 individual mouse brain atlases but also a minimum deformation atlas based on the minimum spatial transformation required to nonlinearly normalize all brains to the geometric center of the entire population. Further, the 10 brains were used to generate a 3D probabilistic atlas that represents the anatomical variability in the segmented structures.

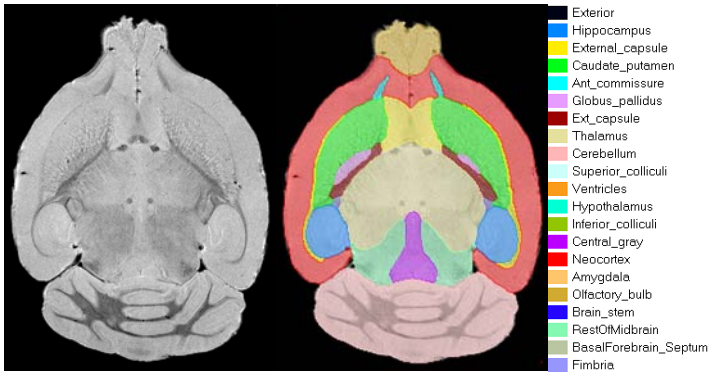


Figure 1. Segmented MRI of mouse brain.

Results and Discussion

Figure 1 presents a single brain MRI segmented into the 20 neuroanatomical structures. Figures 2A and B display the 3D average local deformation map with the intensity of color representing the average distance transformed when the target brains were registered. Figure 2C displays the 3D probabilistic atlas with the color intensity showing the probability of a structure occupying the corresponding location within a segmented region. Areas on the exterior of the brain (cerebellum, stem, olfactory) undergo the greatest deformation and show the biggest statistical variation.

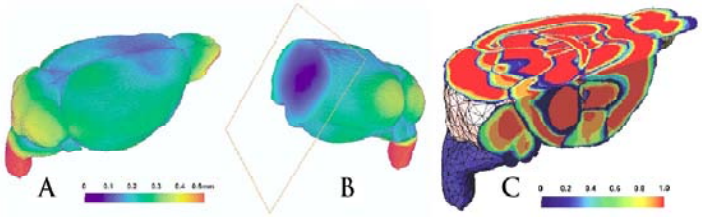


Figure 2. A&B: 3D average local deformation map C: 3D probabilistic atlas

Conclusions

The database augments ongoing efforts with other high priority strains as defined by the Mouse Phenome Database focused on providing a framework for accurate mapping of functional, genetic and protein expression patterns. The database can be browsed and visualized online at site (www.bnl.gov/CTN/mouse).

Acknowledgements

Support provided by the NIH (R01 EB 00233–04; P41 RR16105) and the National High Magnetic Field Laboratory. MRI data were obtained at the AMRIS facility of the McKnight Brain Institute of the University of Florida.

References

[1] Ma, Y. *et al.*, *Neuroscience*, **135**, 1203–15 (2005).

Over the recent past, site-directed spin-labeling EMR has emerged as a powerful and versatile biophysical method to study structure-function relationships in proteins and protein-membrane systems. Advances in high field (HF) EMR methodology extend its applicability even further to address important questions of membrane proteins such as the roles of hydrogen bonding and electrostatics for membrane protein structure and dynamics. Unfortunately, membrane protein samples are quite difficult to handle in HF EMR experiments because of high dielectric losses associated with liquid water.

This report (A.I. Smirnov *et al.*) combines advances in hybrid nanomaterials introduced at **North Carolina State University** with HF EMR instrumentation developed at the NHMFL to construct a nanoporous sample cell to study membrane proteins in the native

lipid bilayer environment. Lipid bilayers are self-assembled into nanotubular macroscopically aligned structures inside nanoporous channels of anodic aluminum oxide that is fabricated as a 60 micron-thick disk. Such lipid structures retain many properties of unsupported bilayers and can accommodate membrane proteins with their native membrane conformations.

*This research was supported by the **NHMFL Visiting Scientist Program**, which provides financial assistance for new users and early career researchers. VSP pays special attention to increasing the diversity of the MagLab user program.*

NANOPOROUS SAMPLE HOLDERS FOR MULTIFREQUENCY/HIGH-FREQUENCY EPR OF FULLY HYDRATED MACROSCOPICALLY ALIGNED SPIN-LABELED MEMBRANE PROTEINS

A.I. Smirnov, A.M. Alaouie, M.A. Voinov (NCSU, Chemistry); J. van Tol, L.-C. Brunel (NHMFL, EMR)

Introduction

Studies of liquid aqueous samples with EPR encounter a problem of high dielectric losses occurring in water at microwave frequencies 2-3 GHz and above. The losses result in non-resonant (magnetic field independent) energy absorption and a rather short penetration depth of electromagnetic field into an aqueous sample. This dictates special arrangements for aqueous samples and specialized sample holders constructed in a way to minimize the Eddy currents through the sample. The latter could be achieved by positioning an aqueous sample within a plane of the magnetic field component of the mm-wave field (*i.e.*, at the electrical field node). In high field/high frequency EPR spectrometers that employ Fabri-Perot resonators this is accomplished by sandwiching an aqueous sample between thin quartz or mylar discs. For example, for water at 20 °C and 250 GHz the sample thickness should be about 18 μm. Such a sample could be configured by putting together two thin quartz discs with one of the disc having a groove etched in. It might, however, be difficult to maintain the 18 μm thickness uniformly throughout the sample. Additional problems arise when macroscopically aligned membrane protein samples are studied with HF EPR. For example, positioning of planar bilayers with director vector perpendicular to magnetic field require a different resonator design. None of the designs described so far allow for convenient examination of physically the same macroscopically aligned membrane protein sample with, for example, at 240 and 9 GHz EPR.

Experimental

Lipid bilayer were prepared from DMPC (1,2-dimyristoyl-*sn*-glycero-3-phosphocholine), doped with 1 mol% of spin-labeled phospholipid 5PC (1-palmitoyl-2-stearoyl-(5-doxyl)-sn-glycero-3 phosphocholine) or spin-labeled ion channel gramicidin A. Hydrated lipid samples were deposited onto 60 μm-thick nanoporous anodic aluminum oxide discs (Whatman, Ltd) to form a macroscopically aligned lipid nanotubular structure. The samples were sealed with a polyvinylidene chloride film with a thickness of 25 μm. 240 GHz EPR spectra were recorded with multi-purpose transient EMR spectrometer equipped with a Fabri-Perot resonatore (NHMFL). Consequently, the same samples were examined with a conventional Varian 9 GHz EPR spectrometer (NCSU).

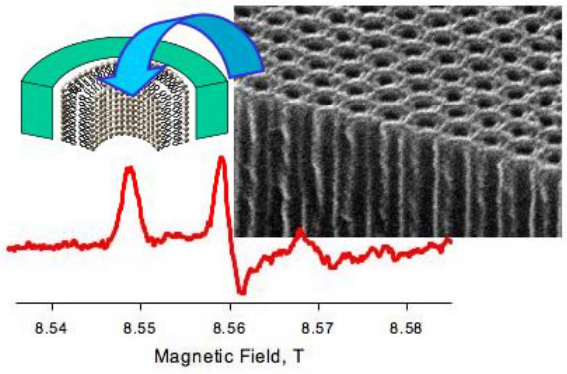


Figure 1. A representative 240 GHz EPR spectrum from DMPC/gramicidin A.

Results and Discussion

Figure 1 shows a representative room temperature 240 GHz EPR spectrum from fully hydrated DMPC/spin-labeled gramicidin A (100:1) aligned by nanoporous AAO. Similar well-aligned spectra were observed from DMPC doped with 1mol% of 5PC. Physically the same AAO discs were used for consequent examination with conventional X-band EPR (not shown). It was determined that polyvinylidene chloride film provides an exceptional moisture seal allowing for several hours of experimentation. We also found that these samples remained to be macroscopically aligned for a period of more than 1 month when stored in a refrigerator at 100 % humidity.

Conclusions

The new technology we describe here allows one to study hydrated and macroscopically aligned membrane proteins with both high frequency/high field and conventional EPR for the first time. Sample handling is also significantly simplified.

Acknowledgements

Support of NHMFL Visiting Scientist Program as well as NIH 1R01GM072897 and DOE contract DE-FG02-02ER15354 (to A.I.S) are gratefully acknowledged.

MAGNET SCIENCE & TECHNOLOGY

Including: CRYOGENICS, ENGINEERING MATERIALS, INSTRUMENTATION & MAGNET TECHNOLOGY

This cryogenics research of S. Fuzier *et al.*, done in collaboration with **GE Global Research Center**—is noteworthy because a better understanding of the properties of liquid helium could lead to development of a medium in which to classify and potentially separate micron and sub-micron particles by size, density and defect. Unlocking the mystery of how to separate very tiny particles has tremendous applications for the pharmaceutical industry and could change how some medications are delivered and how effective they are.

This research was published in Nature Physics, **1**, 36-38 (2005).

PIV MEASUREMENTS OF HE II COUNTERFLOW AROUND A CYLINDER

S. Fuzier, S.W. Van Sciver (FSU, NHMFL & Mechanical Engineering); T. Zhang (GE Global Research Center)

Introduction

The two-fluid model describes He II as if it consists of two interpenetrating fluid components, the viscous normal fluid and the inviscid superfluid. The normal fluid component contains the thermal excitations while the superfluid component carries no entropy. A heat current in He II is carried by the entropy containing normal fluid, which flows at a velocity given by the relation, $v_n = q/\rho s T$, with q being the applied heat flux, ρ being the total fluid density and s the specific entropy. A special case is that of the bulk fluid at rest with the normal fluid flow balanced with flow of the superfluid component such that the superfluid velocity v_s is counterflowing to the normal fluid. Here we report the first Particle Image Velocimetry (PIV) experiments that visualize thermal counterflow around a cylinder immersed in He II.

Experimental

In the PIV technique, one seeds the He II with micron sized particles and tracks their motion to obtain a full field map of the flow. In thermal counterflow, we have previously shown that the particle mainly tracks the normal fluid flow, but may also be affected by the turbulent superfluid component.[1] In our recent study,[2] we placed a 6.35 mm OD transparent circular cylinder in a one dimensional rectangular channel, which is 200 mm long and 38.9×19.5 mm^2 in cross section. The channel is immersed in a constant temperature He II bath with the top end open to the bath while the lower end is closed by a thin film heater. The front wall has a 45×20 mm^2 optical window for imaging the particles with a CCD camera. The cylinder is located inside the channel spanning the full width and orthogonal to the flow.

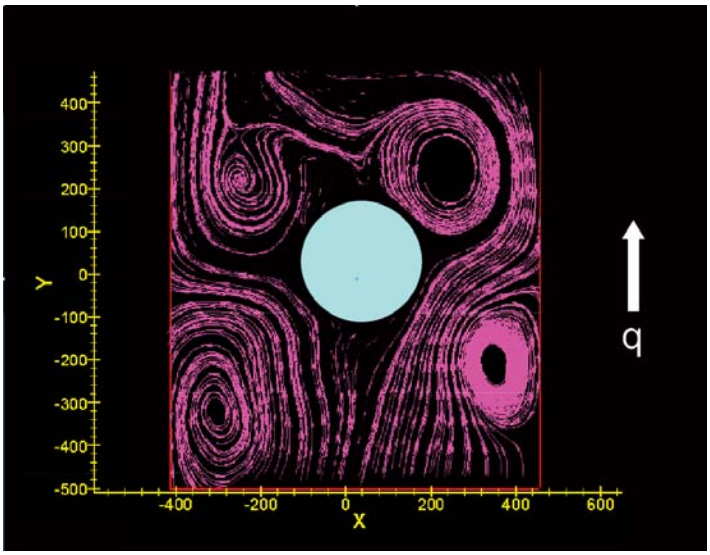


Figure 1. Turbulent structures generated by thermal counterflow around a 6.35 mm OD cylinder. T = 2.03 K and q = 7.2 kW/m^2 of channel cross section, which corresponds to normal fluid velocity of v_n = 23 mm/s.

Results and Discussion

Figure 1 contains an example of the observed counterflow field around the cylinder. These are representative data that actually fluctuate in time and are therefore only typical. They were acquired at 2.03 K and for a heat flux of 7.2 kW/m^2 directed upward, which corresponds to v_n = 23 mm/s. Clearly seen are turbulent structures both below and above the cylinder. However, a more subtle effect is the tendency for the large vortices below the cylinders to cling more closely to the channel walls than do those above the cylinder. Although we have no theoretical explanation for these structures, it is speculated that the large vortices below the cylinder are the result of some form of flow separation occurring in the turbulent superfluid component. Since the normal fluid component interacts more strongly with the suspended particles,[2] however, it is possible that the transverse velocity component of v_n as it flows around the cylinder could push these vortices toward the channel walls. Further work is required to confirm this suggestion.

References

1 Zhang, T. and Van Sciver, S.W., *J. Low Temp. Phys.*, **138**, 865-870 (2005).

2 Zhang, T. and Van Sciver, S.W., *Nature Physics*, **1**, 36-38 (2005).

In the report by G.M. Ludtka et al. high magnetic fields are used to influence the structure and property of steel. In general, the strength of a steel depends both on its chemistry (the combination of atoms included) and the structure of the steel (the way those atoms are grouped together to form "phases"). Thus, two steels of identical chemistry can have very different properties depending on what phases are present. In this **international collaboration of Oak Ridge National Laboratory, Nanyang Technological University, and the University of Cambridge**, it is shown that processing this particular high strength steel can improve the steel's properties by influencing the growth of the desired phase.

This research was published in Scripta Materialia, **52** (6), 461-466 (2005).

EFFECT OF 30 T MAGNETIC FIELD ON PHASE TRANSFORMATIONS IN A BAINITIC HIGH STRENGTH STEEL

G.M. Ludtka, R.A. Jaramillo, S.S. Babu, R.A. Kisner, J.B. Wilgen, G. Mackiewicz-Ludtka, D.M. Nicholson, S.M. Kelly (Oak Ridge National Laboratory); M. Murugananth (Nanyang Technological University); H.K.D.H. Bhadeshia (University of Cambridge)

Introduction

Recently, the effect of a 30 T magnetic field on phase transformations in a high-carbon bainitic alloy was evaluated[1] while continuously cooled at a rate of 1 °C/s. In this work, additional research has been performed to understand the alloying element partitioning characteristics during the pearlite formation. Moreover, the tendency for the pearlite formation under isothermal conditions with and without magnetic field was evaluated.

Experimental

In this study, two steels (see Table 1) were used. The steel identified as "SK" exhibits very sluggish transformation kinetics. In contrast, the steel identified as "FK" is alloyed with Co and Al to enhance the transformation kinetics.

Table 1. Compositions of steel used in the current investigation in at.%

Alloy	C	Si	Mn	Mo	Cr
SK	3.34	3.10	1.89	0.16	1.52
FK	3.43	3.01	1.94	0.13	1.03
Alloy	**Co**	**Al**	**V**	**Fe**	
SK	-	0.02	0.10	Balance	
FK	3.47	2.68	-	Balance	

A 32 mm diameter bore resistive magnet with maximum field strength of 33 teslas at the NHMFL was used.

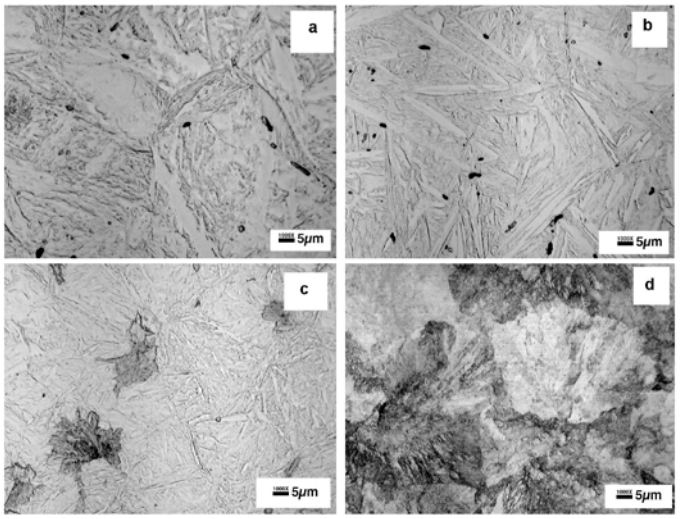


Figure 1. Summary of microstructures attained by continuous cooling from austenitizing temperature with and without magnetic field: (*a*) SK steel – no field; (*b*) FK steel – no field; (*c*) SK steel – 30 T; and (*d*) FK steel – 30 T.

Results and Discussion

The SK and FK steels subjected to continuous cooling from austenitizing temperature without magnetic field did not show any major differences in the microstructure [Figures 1*a* and 1*b*]. On application of a 30-tesla magnetic field, the SK steel showed some localized regions of softer microstructure [Figure 1*c*]. In contrast, the FK steel with an applied magnetic field led to a uniform soft microstructure [see Figure 1*d*].

Conclusions

Although all of the results of this study are not presented here, this research investigated the effects of 30 T magnetic field on the austenite decomposition in two high-carbon steels with different austenite decomposition kinetics. In the steel alloyed with Al and Co, the decomposition of austenite to fine pearlite occurred while continuously cooling from austenite phase field. The pearlite microstructure exhibited a lamellar spacing of less than 50 nm. Atom probe analysis of this pearlite showed enrichment of Cr, Mn, and C and depletion of Si, Al, and Co in the cementite phase. The decomposition of austenite was not extensive during continuous cooling in the alloy without Al and Co due to the sluggish kinetics.

References

[1] Jaramillo, R.A., *et al*., *Scripta Materialia*, **52**, 461-466 (2004).

This work by C. Pantea et al. significantly enhances the state of the art in ultrasound measurements that are used to determine the velocity of sound in a material. These enhancements simultaneously simplify the instrumentation and improve the precision of the measurement.

This research was published in Review of Scientific Instruments, **76**, 114902 (2005).

DIGITAL ULTRASONIC PULSE-ECHO OVERLAP SYSTEM AND ALGORITHM FOR UNAMBIGUOUS DETERMINATION OF PULSE TRANSIT TIME

C. Pantea, D.G. Rickel, A. Migliori (MST-NHMFL, LANL); R.G. Leisure (Colorado State University, Physics); J. Zhang, Y. Zhao (LANSCE-12, LANL); S. El-Khatib (New Mexico State University, Physics); B. Li (State University of New York at Stony Brook, MPI)

The widely applied ultrasonic pulse-echo method uses a short pulse of ultrasound generated by a transducer, often in direct contact with the specimen. The pulse propagates through the material of interest, reflects from the opposite end, and returns to generate a signal in the same transducer: the echo. A measurement of the round-trip travel time and travel distance yields the sound speed from which an elastic constant is determined.

We report an evolution of an all-digital ultrasonic pulse technique for measurements of elastic constants of solids. We use an unambiguous analytical procedure for determining the correct time delay of echoes without any need for actual echo overlap, using a cross-correlation procedure between the buffer rod (BR) and sample (S) signal. We also provide a simple procedure for making corrections for transducer-bond-induced phase shifts.[1] The precision of a measurement made with this system at ambient temperature exceeds one part in 10^7 without the use of mixers, gates, time delays, and other complications normally associated with such measurements.

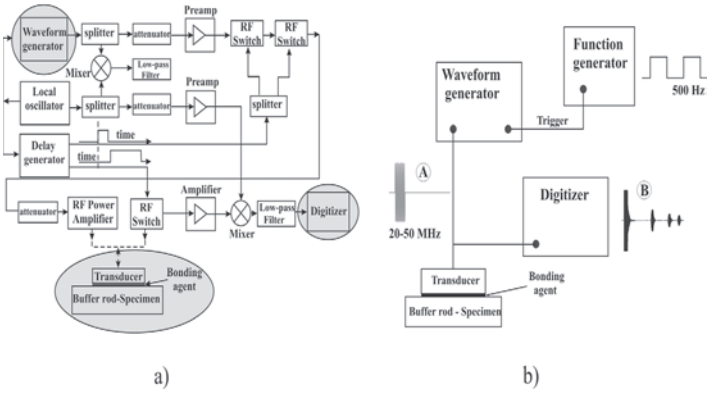


Figure 1. (a) Block diagram of a typical ultrasonic interferometry setup. The blocks marked in gray are the ones we are keeping in the present measurement setup. (b) Block diagram of the present experimental setup.

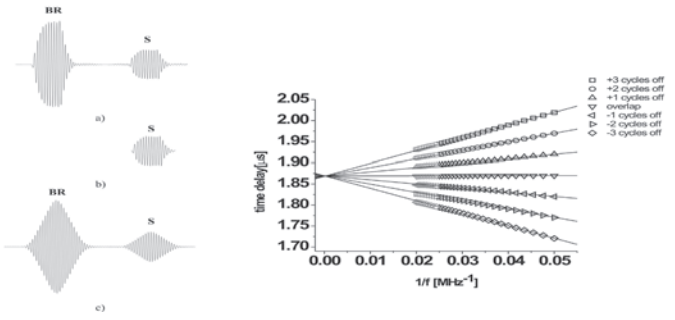


Figure 2. Illustration of the cross-correlation process. (a) BR (buffer rod) and *S* (specimen) signals as collected on the digitizer, (b) the *S* echo signal, and (c) result of cross-correlation procedure. Right side: Measured time delay between the BR and *S* signals vs. the inverse of frequency of the excitation signal.

The present approach has the advantage that it eliminates the need for complicated electronics (see Figure 1) used in ultrasonic interferometry, where two gated sinusoidal pulses need to be generated, spaced in time at different values as a function of the specimen size. The need for only one excitation pulse is a great advantage and can be obtained with a single gating procedure.

Also, we demonstrate that substantial oversampling of the measured signal is not necessary if individual cycles in the BR and *S* signals are fitted to a polynomial function. The use of cross correlation and different overlap peaks determines the correct overlap condition unambiguously. We also show that excitation frequencies higher than the natural transducer resonance should not be used because of additional errors introduced by the presence of an additional frequency component at the natural transducer resonance.

Acknowledgements

This work was performed under the auspices of the National Science Foundation, the State of Florida, and the U.S. Department of Energy.

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T.M. Schaub *et al.* of the NHMFL and Florida State University report an exciting development that opens the powerful technique of Ion Cyclotron Resonance Spectroscopy up to a new class of materials. This technique can accurately determine the chemical basis of a large number of industrially important materials (including but not limited to crude oil). The information derived from these measurements holds the possibility of drastically improving many industrial processes with positive economic—as well as environmental—consequences.

This research was published in Analytical Chemistry, **77**, 1217-1324 (2005).

INSTRUMENTATION AND METHOD FOR ULTRAHIGH RESOLUTION FIELD DESORPTION IONIZATION FOURIER TRANSFORM ION CYCLOTRON RESONANCE MASS SPECTROMETRY OF NON-POLAR SPECIES

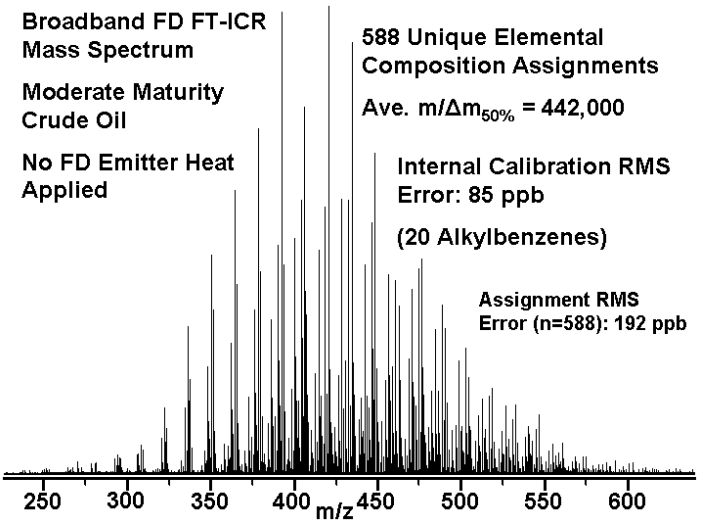
T.M. Schaub (NHMFL Tallahassee); C.L. Hendrickson (NHMFL Tallahassee and FSU, Chemistry & Biochemistry); J.P. Quinn (NHMFL Tallahassee); R.P. Rodgers, A.G. Marshall (NHMFL Tallahassee and FSU, Chemistry & Biochemistry)

Experimental

We have interfaced a commercial field desorption ion source to our 9.4 T FT-ICR mass spectrometer for high resolution, high mass accuracy measurements of non-polar species. The FT-ICR MS instrument includes a liquid injection field desorption ionization source, octopole ion guides, external octopole ion trap capable of an axial potential gradient for ion ejection, capacitively-coupled open cylindrical ion trap, and pulsed gas valve for ion cooling. Model compound responses with regard to various source and instrument conditions provide a basis for interpretation of broadband mass spectra of complex mixtures.

Results and Discussion

As an example, we demonstrate broadband speciation of a Gulf Coast crude oil (see Figure), with respect to numerous heteroatomic classes, compound types (rings plus double bonds), and carbon number distributions.



Acknowledgements

The authors thank Daniel McIntosh for the manufacture of all custom system components, Bernhard Linden for technical assistance with the FD source, Steven Beu for the SIMION modeling, and Kuangnan Qian for helpful discussions regarding petroleum mass spectrometry. This work was supported by ExxonMobil Research and Engineering, NJ, NSF (DMR-00-84173), Florida State University, and the National High Magnetic Field Laboratory in Tallahassee, FL.

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CONDENSED MATTER

Including: KONDO/HEAVY FERMION SYSTEMS, MAGNETISM & MAGNETIC MATERIALS, METAL-INSULATOR TRANSITIONS, MOLECULAR CONDUCTORS, OTHER CONDENSED MATTER, QUANTUM FLUIDS AND SOLIDS, SEMICONDUCTORS & BASIC AND APPLIED SUPERCONDUCTIVITY

KONDO/HEAVY FERMION SYSTEMS

Essential efforts were concentrated on studying magnetic quantum critical points (mQCP) in a few Kondo/Heavy Fermion systems in attempts to establish degree of universality of the phenomenon. Generally, QCP marks a change in the ground state (T=0) driven by an external parameter, such as pressure, alloying, etc. In particular, such change in the ground state at zero temperature is often driven by strong enough magnetic field, e.g., metamagnetic (MM) transitions or suppression the antiferromagnetic (AFM) state by field in the rare- earth (RE) and actinides intermetallics. The basic challenge is whether the 4f- or 5f- electrons on the high field side of a QCP become fully or partially localized.

In the report by C. Capan *et al*., a MM QCP at 28 T in $CeIrIn_5$ shows an upturn in resistivity and non-Fermi liquid behavior; however, the de Haas-van Alphen experiments do not show much changes in the Fermi surfaces' sizes.

For the Kondo material $CeIn_3$, the research of A.V. Silhanek *et al*. indicates against universality of behavior near mQCP.

METAMAGNETISM AND NON-FERMI LIQUID BEHAVIOR IN $CeIrIn_5$

C. Capan, R. Goodrich, J.F. DiTusa (Louisiana State University, Physics); L. Balicas, T. Murphy, E. Palm (NHMFL); R. Movshovich, E.D. Bauer, M.F. Hundley, J.D. Thompson, J.L. Sarrao (LANL)

Introduction

Quantum phase transitions correspond to a continuous ground state transformation at T=0 driven by quantum fluctuations. One of the intensively investigated issues is the possibility of a Fermi Surface volume change at a quantum critical point. In this context, the metamagnetic transition, corresponding to a non-linear increase in magnetization of a paramagnet, have been focus of attention since it might involve such a change, even tough its microscopic mechanism is not fully established. Moreover, strong deviations from Fermi Liquid theory reported in $Sr_3Ru_2O_7$ have raised the possibility of a metamagnetic quantum critical end-point.[1] $CeIrIn_5$, a heavy fermion compound with a recently discovered metamagnetic transition at high fields,[2] offers yet another playground for such investigations. In a preliminary study, we observed a field induced non-Fermi Liquid behavior in both resistivity and specific heat up to 17 T.[3] Here, we report a low temperature study of transverse magnetoresistance and quantum (dHvA) oscillations of cantilever magnetization in a $CeIrIn_5$ single crystal, using the 33 T magnet with the portable dilution refrigerator at NHMFL in Tallahassee.

Results and Discussion

The temperature dependence of the resistivity, shown in Figure 1, is obtained from field sweeps between 20 T-32.75 T. For fields below metamagnetic transition (28 T), resistivity has an upturn for temperatures below 0.3 K. The upturn becomes more pronounced near the transition. The power law fit with an exponent 1.5 (solid

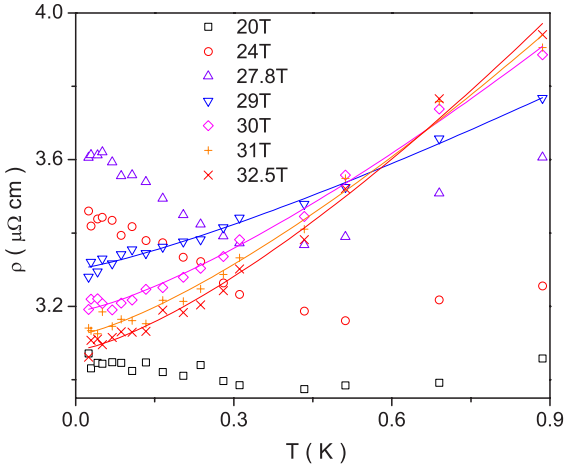

Figure 1. Resistivity vs. temperature in $CeIrIn_5$ for H||[001].

lines in Figure 1) above the transition is indicative of non-Fermi Liquid behavior. Figure 2 shows the FFT spectrum of the dHvA oscillations resolved in cantilever magnetization at 51 mK in the same field range. The frequencies correspond to the quasi-2D "α" and "β" sheets of the Fermi Surface, in good agreement with a previous report.[4] A more detailed analysis revealed that the frequency of the orbits increase slightly across the transition, pointing to a Fermi Surface topology change, not accompanied by an effective mass enhancement.

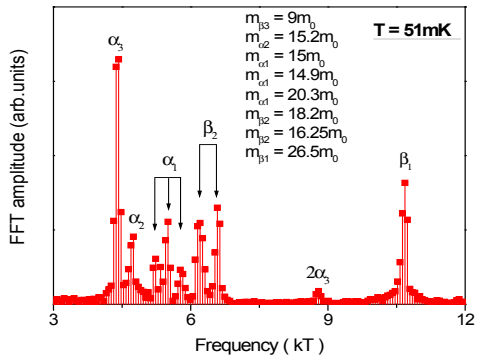

Figure 2. FFT spectrum of the magnetization oscillations.

Taken together with our earlier report of non-Fermi Liquid behavior in the low field phase up to 17 T, this is evidence for a metamagnetic quantum critical point in $CeIrIn_5$. The unusual upturn of resistivity close to the metamagnetic field indicates that disorder effects might be important near this quantum critical point.

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NON-LOCAL QUANTUM CRITICALITY IN HIGH-SYMMETRY SYSTEMS $CeIn_{3-x}Sn_x$

A.V. Silhanek (MST-NHMFL, LANL); T. Ebihara (Shizuoka University, Japan); N. Harrison, M. Jaime (MST-NHMFL, LANL); K. Tezuka (Shizuoka University, Japan); V. Fanelli (MST-NHMFL, LANL); C.D. Batista (T-11, LANL)

When the Neel temperature T_N of an antiferromagnet is tuned to absolute zero at a quantum critical point (QCP), the uncertainty principle leads to a divergence in the characteristic lengthscale of the fluctuations of the staggered-moment order parameter.[1] In itinerant electron antiferromagnets, strong on-site correlations often cause the renormalized Fermi bandwidth T* to become comparable to T_N. A potential locally critical scenario arises in which the extent to which the d- or f-electrons locally contribute charge degrees of freedom to the Fermi liquid becomes subject to fluctuations at the QCP[2,3] and their effective localization is conditional upon the inequality $T_N > T^*$ being satisfied necessitating $T \mapsto 0$ at the QCP as depicted in Figure 1(a). Several f-electron antiferromagnets, including $CeCu_{6-x}Au_x$, $YbRh_2Si_{2-x}Ge_x$, and $CeRhIn_5$, appear to provide examples of such behavior as function of pressure p, magnetic field H, or chemical substitution x. No experiment, however, has yet been able to gauge the extent to which local criticality requires the spin fluctuations to be two-dimensional (2D). Were this an absolute requirement, the unambiguously three-dimensional (3D) spin fluctuation spectrum of cubic $CeIn_3$ should then provide the essential f-electron counterexample to local criticality. In such a case, one might expect a quantum critical spin-density wave (SDW) scenario[4] in which T* remains finite at the QCP (see Figure 1(b)).

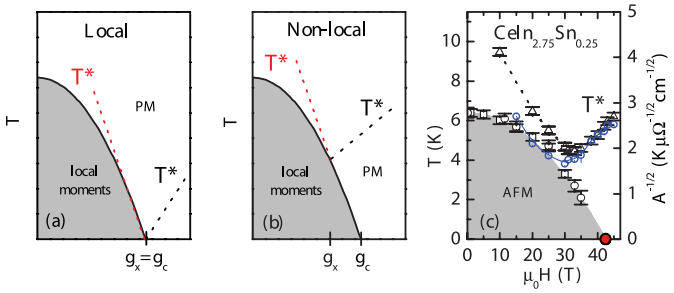

Figure 1. Schematic of antiferromagnetic quantum criticality tuned at $g = g_c$ according to (a) the locally critical scenario and (b) the SDW scenario, where T* represents the Fermi temperature. The parameter g can correspond to p, H or x, depending on the system. (c) T,H phase diagram of $CeIn_{2.75}Sn_{0.25}$ extracted from Cp(T) (○ symbols) and ρ(T) data (□ symbols), with the grey region represents the antiferromagnetic (AFM) phase under the fitted $T_N = T_{N,0}(1 - (H/H_c)^2)$ curve. Δ symbols delineate maxima in $\partial\rho/\partial T$ that are approximately representative of the Fermi bandwidth T* in the paramagnetic (PM) region. Blue circles represent $A^{-1/2}$ in the low temperature limit $T << T^*$.

In the present work we utilize the fact that Sn-substitution of only 8 % of the In sites in $CeIn_3$ (yielding $CeIn_{2.75}Sn_{0.25}$) reduces T_N to ~6.4 K so as to enable quantum criticality of the same type II antiferromagnetic phase as in pure $CeIn_3$ to be tuned by a static magnetic field $\mu_0 H_c = 42 \pm 2$ T (see Figure 1(c)). Electrical transport and thermodynamic measurements reveal that m* does not diverge, suggesting that cubic $CeIn_3$ is representative of a critical spin-density wave (SDW) scenario. The existence of a maximum in m* at a lower field $\mu_0 H_x = 30 \pm 1$ T may be interpreted as a field-induced crossover from local moment to SDW behavior as the magnitude of the antiferromagnetic order parameter falls below the Fermi bandwidth.

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MAGNETISM & MAGNETIC MATERIALS

In the report by L. Balicas *et al*. important information is found concerning the nature of the metamagnetic transition in $Sr_4Ru_3O_{10}$. The latter material is a typical ferromagnet when field is applied along the c-axis. For the in-plane field around 30 T a metamagnetic transition takes place with the strong change in magnetization. Study of the Shubnikov- de Haas oscillations in the in-plane resistivity (at slightly inclined fields) reveals the appearance of the new frequencies above the transition, meaning considerable changes in the structure of the (almost cylindrical) Fermi surfaces spectrum.

*This research was supported by the **NHMFL In-House Research Program**, which funds approximately six projects per year that meet the criteria of scientific quality on par with other NSF grants and are designed to extend or improve a component of the Magnet Lab user program. It was also supported by the **Visiting Scientist Program**, which provides financial assistance for new users and early career researchers. VSP pays special attention to increasing the diversity of the MagLab user program.*

SHUBNIKOV DE HAAS EFFECT IN HIGH QUALITY SINGLE CRYSTALS OF $SR_4RU_3O_{10}$

L. Balicas, Y.J. Jo (NHMFL); N. Kikugawa (U. St. Andrews, Physics); K. Storr (Prairie View A&M U., Physics); Z.Q. Mao (Tulane U., Physics)

Introduction

$Sr_4Ru_3O_{10}$ is the triple-layered member of the Rudlesden-Poper series with $n = 3$. Although this material has not been as widely studied as the other ruthenates in the series, earlier work by Cao *et al.*[1] revealed that its ground state has intriguing characteristics. When the field is applied along the *c*-axis typical ferromagnetic behavior occurs, while for field applied along the in-plane direction a first-order metamagnetic transition accompanied by critical fluctuations is observed. One possible way to understand its metamagnetic behavior within a mean-field theory is by considering the eventual proximity of its Fermi level to a van Hove singularity.

Experimental

We performed a detailed electrical transport study at low temperatures using the hybrid magnet in high quality single crystals of the tri-layered ferromagnetic metal $Sr_4Ru_3O_{10}$. We observed Shubnikov de Haas oscillations for several frequencies.

Results and Discussion

The most prominent orbits have two-dimensional character and display frequencies F_α = 9959 T, F_β = 3949 T, F_γ = 1877 T corresponding respectively to 73.5, 29.2 and 13.9% of the area of the orthorhombic first Brillouin zone (FBZ). Several additional orbits, mostly with frequencies below 2 kT have also been identified, whose spectral weight and frequencies are shifted by a metamagnetic transition induced by an in-plane field component.

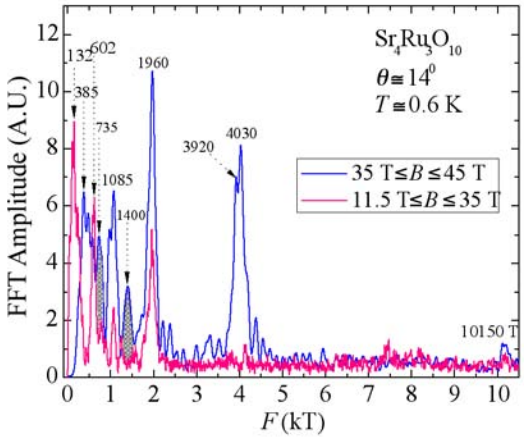

Figure 1. The FFT spectrum of the Shubnikov de Haas signal of a $Sr_4Ru_3O_{10}$ single crystal for two ranges of magnetic field, i.e., above and below a metamagnetic transition. Shaded areas indicate the existence of peaks in the FFT spectrum that are not seen at lower fields.

Conclusions

Our results indicate the reconstruction of the Fermi surface at the metamagnetic transition. The mechanism remains to be determined.

Acknowledgements

LB acknowledges support from the NHMFL In-House Research Program.

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Low temperature ^{133}Cs NMR data are presented by V.F. Mitrovic *et al*. for the Cs_2CuCl_4 single crystal. Cs_2CuCl_4 is a S=1/2 2D frustrated antiferromagnet compound that reveals exotic phases in the presence of magnetic fields. The reported NMR data provide clear evidences for a contra-intuitive behavior with the increase of the applied longitudinal field: namely, it leads to the increased complexity of the local spin textures. That fact demonstrates the important effect of the quantum fluctuations in this spin liquid system.

*This research was supported by the **NHMFL Visiting Scientist Program**, which provides financial assistance for new users and early career researchers. VSP pays special attention to increasing the diversity of the MagLab user program.*

MAGNETIC INDUCED PHASES IN THE FRUSTRATED QUANTUM AF Cs_2CuCl_4

V.F. Mitrovic, M.-A. Vachon (Brown University); R. Coldea (Oxford University); A.P. Reyes, P. Kuhns, T. Murphy, E. Palm (NHMFL)

We report ^{133}Cs NMR data on Cs_2CuCl_4 single crystals. This compound is a 2D S=1/2 frustrated Heisenberg antiferromagnet (AF) on an anisotropic triangular lattice. It is one of only two candidate materials to show strong evidence for 2D spin liquid behavior. Furthermore, a magnetic field stabilizes exotic phases, as shown by neutron measurements.[1] Our NMR measurements at the 60 mK reveal multiple field-induced phase transitions in a magnetic field applied parallel to the Cu chains, *b*-axis. The phases are distinct by their local spin structure. Contrary to common belief, the complexity of the local spin structure increases with applied longitudinal field. Our measurements were performed at the millikelvin facility on the 18 T magnet and with the top-loading ^{3}He/^{4}He dilution refrigerator.

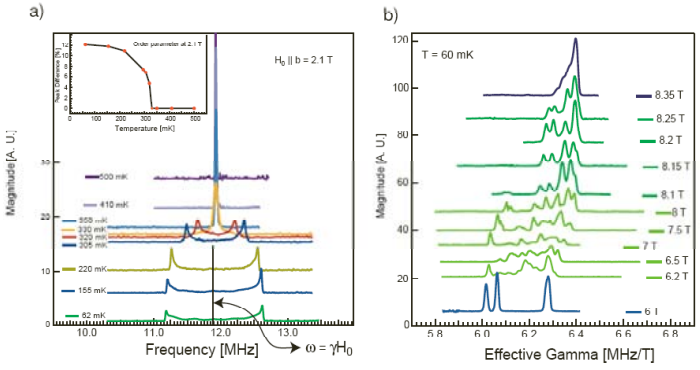

Figure 1. (a) Temperature dependence of ^{133}Cs(B) Spectra at H = 2.1 T. Solid line denotes zero shift position. Inset: Temperature dependence of the order parameter, defined as the difference between extrema peaks. (b) Section of the field dependence of the spectra at T = 60 mK showing a field induced phase transition around H = 6.2 T

Results

In Figure 1(a) we show a temperature dependence of the ^{133}Cs(B) NMR spectra at H = 2.1 T. A double peak structure centered on the Larmor frequency is observed below T = 330 mK. This represents the local spin density probability distribution of an incommensurate phase. The order parameter in this state is defined as the difference between two peaks and its temperature dependence is shown. This difference, related to a sub-lattice magnetization M_s, obeys the power law $[(T_N\text{-}T)/T_N]^\beta$ with $\beta \approx 0.34$. This value is in agreement with the theoretical value for a 3D XY spin model. In Figure 1(b), we present a section of the spectra between 6 T and 8.35 T at T = 60 mK. The abrupt change around 6.2 T is associated with a field induced quantum phase transition. The spectra converge into one single peak (not shown) above the saturation field. We find that the complexity of the local spin texture increases with applied longitudinal fields, signaling that the quantum fluctuations effect are important in the intermediate field regime.

Acknowledgements

Work is supported in part by the Brown University Solomon Research fund. Work at NHMFL is supported by the NHMFL Visiting Scientist Program through the NSF and the state of Florida.

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The idea in the report by S.E. Sebastian *et al*. is that by going down to the lowest temperatures possible in the strong enough magnetic field one can realize in Han Purple pigment, $BaCuSi_2O_6$, the crossover from 3D to 2D regime of the enhanced quantum fluctuation near the BEC quantum critical point (QCP).The experimentally found scaling behavior near the QCP agrees with the theoretical predictions for the 2D criticality.

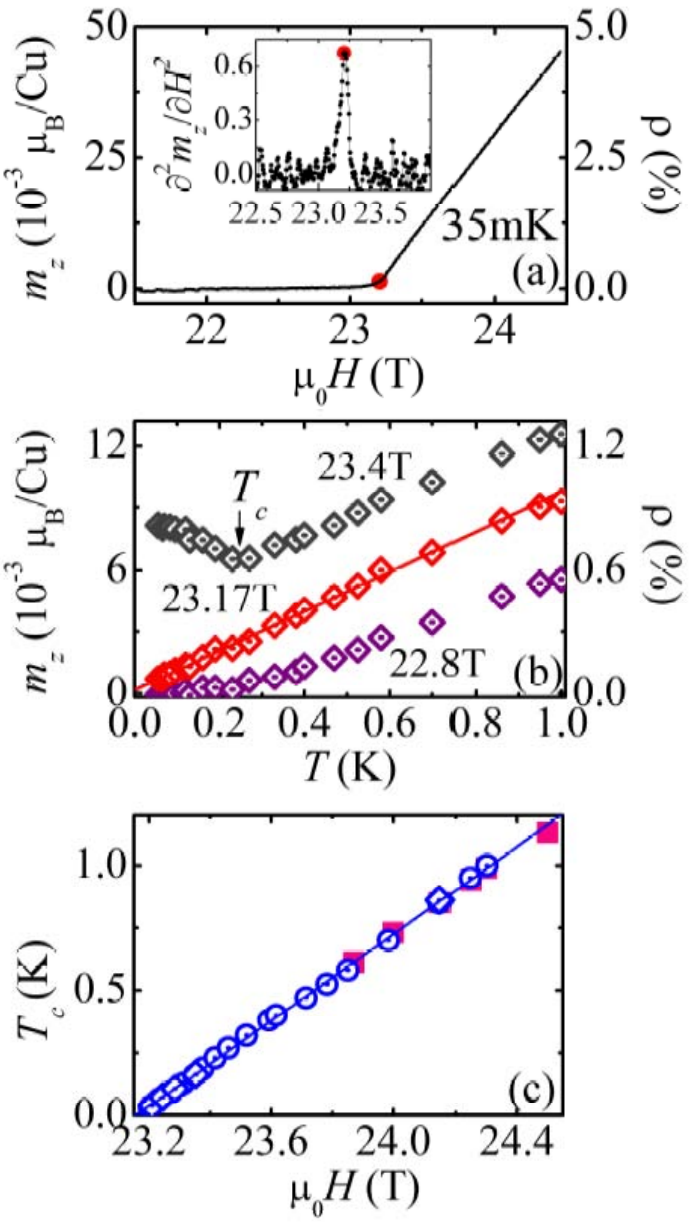
This research has been accepted for publication in Nature.

DIMENSIONAL REDUCTION AT A QUANTUM CRITICAL POINT

S.E. Sebastian (Stanford, Physics); N. Harrison (LANL, NHMFL); C.D. Batista (LANL); L. Balicas (NHMFL, FSU); M. Jaime (LANL, NHMFL); P.A. Sharma (LANL, NHMFL); N. Kawashima (U. Tokyo, Physics), I.R. Fisher (Stanford, Physics)

Low dimensional systems exhibit enhanced quantum fluctuations, and hence offer a rich opportunity to study novel states near a quantum critical point (QCP). The possibility of realizing two-dimensional (2d) quantum criticality embedded within a three-dimensional (3d) manifold has been extensively theoretically

explored in the context of systems such as frustrated magnets, layered superconductors, free-standing liquid crystal films, sliding Luttinger liquids, and lipid bi-layers. However, there has as yet not been an experimental realization of 2d quantum criticality in any bulk 3d system. Measurements in the 33 T magnet with dilution fridge temperatures have enabled for the first time, an experimental realization of dimensional reduction in a 3d lattice of s = ½ spins, in which geometrical frustration reduces system dimensionality, resulting in a 2d-QCP. In this sense, the very notion of dimensionality can be said to acquire an "emergent" nature: although the individual particles move on a 3d-lattice, their collective behavior occurs in lower-dimensional space. The system



The figure shows the magnetization m_z as a function of field H, the magnetization as a function of temperature T and the phase boundary T_c as a function of H, all determined using magnetic torque. The linear behaviors in all these quantities are the direct consequence of a 2d QCP.

we study is the Mott insulator $BaCuSi_2O_6$, which comprises coupled layers of Cu^{2+} s = ½ pairs (spin dimers).[1] As verified by experimental power scaling measurements, the high-field magnetically ordered state can be interpreted as a Bose-Einstein condensate (BEC) of triplets. We report the experimental observation of a continuous crossover in critical scaling behavior near the QCP from 3d to 2d BEC universality. This provides experimental evidence for inter-layer decoupling at the QCP, leading to purely 2d-quantum fluctuations of the BEC order parameter.

Acknowledgements

This work is supported by the NSF and DOE. We would like to thank Eric Palm and Tim Murphy for high quality user support.

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There is a block of interesting reports [V.S. Zapf *et al*., Magnetostriction in the BEC Compound $NiCl_2$-4SC$(NH_2)_2$; D. Zocco *et al*., Thermodynamic Measurements of Ni$(Cl_{1-x}Br_x)_2$-4SC$(NH_2)_2$'; V.S. Zapf *et al*., Angle-dependent Properties of $NiCl_2$-4SC$(NH_2)_2$; and the representative report presented here] ,where, by making use of different experimental tools, it was shown that Ni S=1 spin degrees of freedom in the organic quantum magnet $NiCl_2$-4SC$(NH_2)_2$ may also exhibit BEC in a range of low temperatures and fields. The BEC QCP was assigned at H_{c1} in the (T,H)-plane for the measured XY antiferromagnetic phase diagram.

BOSE-EINSTEIN CONDENSATION OF MAGNONS IN $NiCl_2$-4SC$(NH_2)_2$

V.S. Zapf, D. Zocco (NHMFL, LANL); C.D. Batista (LANL); M. Jaime, N. Harrison, A. Lacerda (NHMFL, LANL); A. Paduan-Filho (Instituto de Física, Universidade de Sao Paulo, Brazil)

Introduction

We have investigated Bose-Einstein Condensation (BEC) of the Ni spin degrees of freedom in the organic quantum magnet $NiCl_2$-4SC$(NH_2)_2$ (DTN) using thermodynamic measurements to dilution refrigerator temperatures at the NHMFL at Los Alamos. This is one of the first quantum magnets to exhibit BEC, and the first one based on Nickel. XY antiferromagnetic (AFM) order occurs for fields between H_{c1} = 2.1 T and H_{c2} = 12.6 T and below T = 1.2 K. We show that the quantum phase transitions (QPT) into the ordered state at H_{c1} can be understood in terms of Bose-Einstein condensation. For a BEC, critical field-temperature phase diagram

is expected to follow a power-law temperature-dependence H_c-$H_{c1} \sim T^{3/2}$.[1] We mapped the phase diagram of this compound and searched for this power-law behavior using specific heat and magnetocaloric effect measurements.

Experimental

The calorimeter consists of a sapphire plate suspected from thin strings, on which the sample, a ruthenium oxide thermometer, and a solid state heater are mounted. The measurements are conducted in a dilution refrigerator in the 20 T magnet at the NHMFL in Los Alamos. Specific heat is measured by a semi-adiabatic heat pulse technique, and the magnetocaloric effect is measured by sweeping the magnetic field up and down while monitoring the temperature. The ruthenium oxide thermometers were calibrated in fields using the compensated region of the 20 T magnet at NHMFL at Los Alamos.

Results and Discussion

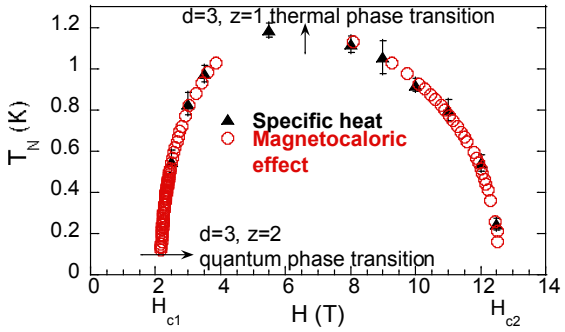

Figure 1. Temperature-field phase diagram of $NiCl_2$-$4SC(NH_2)_2$ for magnetic fields along the c-axis.

The phase diagram determined from specific heat and magneto-caloric effect measurements is shown in Figure 1. More details can be found in Ref. 2. The power-law exponent α where H_c-$H_{c1} \sim T^{\alpha}$ is difficult to extract since it is only valid as $T \mapsto 0$, so any fits over a finite temperature range will distort the behavior. The exponent α was thus determined by fitting the low-temperature data points in Figure 1 between base temperature and various maximum temperatures T_{max}, and extrapolating to $T_{max} = 0$. Our results are consistent with the BEC prediction of $\alpha = 1.5$.

Acknowledgements

This work was supported by the DOE and the NSF through the National High Magnetic Field Laboratory. A.P.F. acknowledges support from CNPq (Conselho Nacional de Desenvolvimento Científico e Tecnológico, Brazil).

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QUANTUM FLUIDS & SOLIDS

A remarkable recent development in the theory of quantum computation has been the realization that exotic states of matter, so-called topologically ordered states, which include certain fractional quantum Hall states, may provide a natural medium for storing and manipulating quantum information. In these states, quantum information is stored globally, throughout the entire system, and so is intrinsically protected against decoherence. Quantum computation is then carried out by moving quasiparticle excitations around one another in 2 dimensions, forming "braids" in 3 (=2+1) dimensional space-time. Because these braids are topologically robust (i.e., they cannot be unbraided without cutting one of the strands), the resulting computation is insensitive to error. In this work by L. Hormozi and others at FSU and **Lucent Technologies**, it was shown, *for the first time*, how to compute braids for carrying out quantum computation using quasiparticles conjectured to exist in the recently observed ν=12/5 fractional quantum Hall state.

This research was published in Physical Review Letters, **93**, 140503-1 – 140503-4 (2005).

QUANTUM COMPUTING WITH NONABELIAN QUANTUM HALL STATES

L. Hormozi, G. Zikos, N.E. Bonesteel (FSU, Physics); S.H. Simon (Lucent Technologies)

Introduction

A quantum computer must be capable of manipulating quantum information while simultaneously protecting it from error and loss of quantum coherence due to coupling to the environment. Topological quantum computation (TQC)[1] offers a particularly elegant way to achieve this using quasiparticles that obey nonabelian statistics. Such quasiparticles, which are expected to arise in a variety of two-dimensional quantum many-body systems, have the property that the usual phase factors of +/-1 associated with the exchange of identical bosons or fermions are replaced by noncommuting (nonabelian) matrices that depend only on the topology of the paths (braids) used to effect the exchange. The matrices act on a degenerate Hilbert space whose dimensionality is exponentially large in the number of quasiparticles and whose states have an intrinsic immunity to decoherence because they cannot be distinguished by local measurements, provided the quasiparticles are kept sufficiently far apart.

In TQC this protected Hilbert space is used to store quantum information, and quantum gates are carried out by adiabatically braiding quasiparticles around each other. Because the resulting

quantum gates depend purely on the topology of the braids, errors only occur when quasiparticles form "unintentional" braids. This built in protection from error and decoherence is an appealing feature of TQC that may compensate for the extreme technical challenges that will have to be overcome to realize it.

Results and Discussion

Previous work on TQC has taken the form of mathematical "existence proofs" that braiding patterns can, in principle, be found. We have shown[2] for the first time how such braiding patterns can be efficiently found for the case of so-called Fibonacci anyons—a particular kind of nonabelian quasiparticle that is thought to exist in the recently observed ν = 12/5 fractional quantum Hall state.[3] Our controlled-NOT gate is shown in Figure 1.

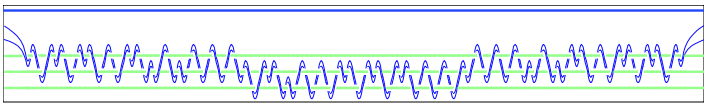

Figure 1. A braid that carries out a controlled-NOT gate between two qubits encoded using triplets of quasiparticles. A NOT operation is performed on the target (green) qubit if, and only if, the control (blue) qubit is in the state |1>.

Conclusions

We have compiled explicit braids for carrying out TQC using a kind of nonabelian quasiparticle that is thought to exist in an experimentally observed fractional quantum Hall state. Our results are the first to give a clear picture of the resources that will be required to carry out this exotic form of quantum computation.

Acknowledgements

This research is supported by U.S. DOE Grant # DE-FG02-97ER45639.

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SEMICONDUCTORS

The three reports published here represent a very substantial group of research activities in the Semiconductor category.

The report by S. Crooker *et al*., shows spectacular imaging of non-equilibrium electron spin polarization and illustrates spin transport, decay, and polarization by reflection in GaAs.

This research was published in Science, **309**, 2191 (2005).

The next report by Y.D. Jho *et al*. is the first observation of superfluorescence from a semiconductor—a spectacular signature of macroscopic coherence that evolves spontaneously in a population of electron hole pairs in a quantum well in high magnetic field.

*This research was supported by the **NHMFL In-House Research Program**, which funds approximately six projects per year that meet the criteria of scientific quality on par with other NSF grants and are designed to extend or improve a component of the Magnet Lab user program.*

The report by Y. Zhang *et al*. describes transport measurements on a new exciting system, showing spin and valley degeneracies breaking in strong magnetic field. *This research has been accepted for publication by* Physical Review of Letters.

IMAGING SPIN TRANSPORT IN LATERAL Fe/GaAs STRUCTURES

S. Crooker, M. Furis (NHMFL-LANL); D. Smith (LANL, Theoretical Division); X. Lou, C. Adelmann, C. Palmstrøm, P. Crowell (University of Minnesota)

Introduction

The ability to control and measure the spin of an electron in semiconductors has recently been proposed as the operating principle for a new generation of spin-electronic, or "spintronic" devices. By taking advantage of the electron's spin degree of freedom, today's charged-based microelectronics may realize significant improvements in operating speed and power consumption. Many designs for functional spintronic devices have been recently proposed; for example, the "spin transistor"—a device whose "on" and "off" states depend on whether the current-carrying electrons are polarized spin-up or spin-down. Proposed schemes for spintronic devices generally require three essential elements: (i) a mechanism for *electrically injecting* spin-polarized electrons into semiconductors, (ii) a practical means for *spin manipulation* and transport, and (iii) an electronic scheme for *detecting* the resulting spin polarization.

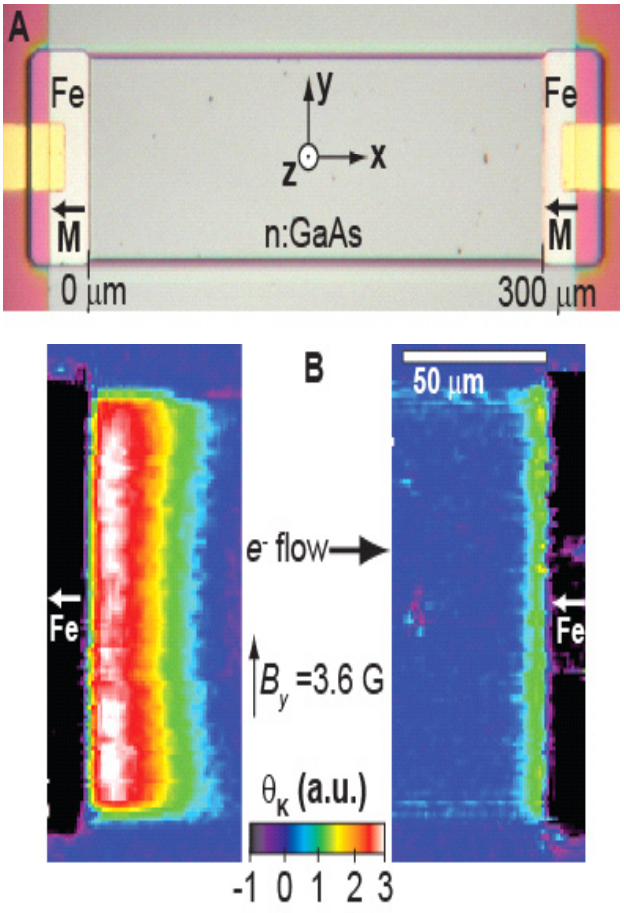

Figure 1. (a) Photograph of a lateral ferromagnet/ semiconductor spin transport device. The Fe/GaAs tunnel-barrier source and drain contacts are magnetized along **–x** as shown. The GaAs channel is 300 μm long. (b) 2-D images of electron spin polarization near the source and drain contacts. Efficient electrical spin injection from the source contact is clearly observed. The spin polarized electrons that accumulate near the drain contact are flowing "upstream" (against the net electron current).

Experimental

We have applied scanning Kerr microscopy to the study of lateral ferromagnet/semiconductor devices that provide an all-electrical means for injection of spin-polarized electrons into semiconductors.[1] These devices, grown and fabricated in the research groups of Profs. Chris Palmstrøm and Paul Crowell at the University of Minnesota, have ferromagnetic iron (Fe) source and drain contacts at opposite ends of a 300 μm long channel of lightly-doped n-type GaAs (see Figure 1a). Each contact is a Schottky tunnel barrier formed by an epitaxial Fe film grown on a thin layer of highly-doped n+:GaAs. This design permits electrons to tunnel directly from the spin-polarized Fermi surface of iron into the n:GaAs channel.

With a small (0.4 V) voltage across the device, Figure 1b shows 2D images of the steady-state electron spin polarization in the GaAs channel. Electrical spin injection from the source (left) contact, and subsequent lateral spin flow in the channel, is clearly observable. The decay length of the injected spin polarization (~50 μm) is much less than the 300 μm channel. Therefore, in this device, injected spins lose their polarization long before they arrive at the drain (right) contact. However, the right-hand side of Figure 2b reveals an appreciable spin polarization in the channel within ~10 μm of the drain. It is possible to demonstrate, through the use of applied stress to the sample substrate, that these "accumulated" electron spins become polarized by *reflection* from the ferromagnetic drain contact, and that they are actually flowing upstream, against the net electron current.[1]

Moreover, these Fe/GaAs tunnel barrier contacts also function as electrical spin detectors (in addition to their role as spin injectors). In this experiment, optically-injected spin polarized electrons are caused to flow through the drain contact, and an in-plane magnetic field (B_y) forces precession of these spins parallel or antiparallel to the magnetization **M** of the drain. The conductance of the device is larger (smaller) when the spin current at the drain is polarized parallel (antiparallel) to **M**, thereby confirming that the electrical conductance is spin-sensitive. All the essential elements of a spin transport device (electrical spin injection, manipulation, and detection) are therefore demonstrated in these structures, an important step towards all-electrical functional spin transport devices.

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ULTRAFAST OPTICS IN HIGH MAGNETIC FIELDS: COOPERATIVE RECOMBINATION OF A QUANTIZED, DENSE ELECTRON-HOLE PLASMA

Y.D. Jho (NHMFL/UF, Physics); X. Wang, D.H. Reitze (UF, Physics); J. Kono (Rice, ECE); C.J. Stanton (UF, Physics); A.A. Belyanin (Texas A&M, Physics); X. Wei (NHMFL)

Introduction

The ultrafast optics facility in Tallahassee uses high magnetic fields and intense ultrashort (<150 fs) laser pulses in combination to investigate electronic excitations in semiconducting quantum-confined systems, with an emphasis on creating and probing quantum coherence in these systems. We have investigated light emission in an $In_{0.2}Ga_{0.8}As$/GaAs quantum well system in a strong perpendicular magnetic field and observed for the first time the emission of superfluorescence (SF) from a semiconductor. The magnetic field fully quantizes the QW system into an atomic-like

system with a series of Landau levels (LLs) and thus strongly increases the density of states (DOS) to accommodate a high-density e-h plasma.[1] The resulting density and energy confinement is sufficient to generate a spontaneous macroscopic polarization that decays through the emission of picosecond superfluorescent pulses.[2]

Experimental

We used a 150 fs, 775 nm, Ti:Sapphire laser system to measure the photoluminescence from the center and edges of the sample as a function of laser fluence F_{laser}, magnetic field B, and excitation spot size and geometry. The 31 T magnet was used in these studies. Emission was collected using optical fibers from the opposite face and cleaved edges of the sample and analyzed with a grating spectrometer equipped with a charge-coupled device detector.

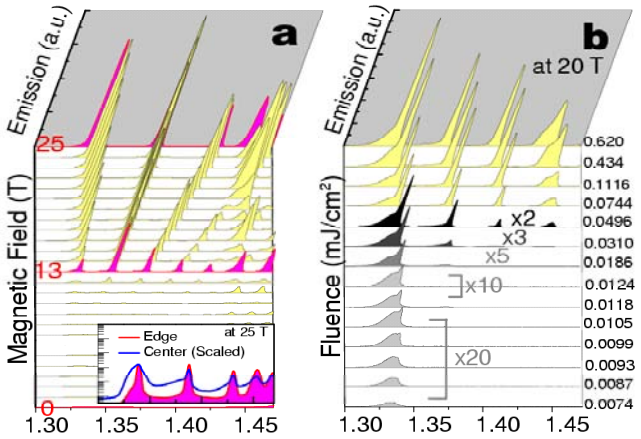

Figure 1. Emission spectra as a function of (a) magnetic field for fixed F_{laser} = 0.62 mJ/cm², and (b) F_{laser} for a fixed 20 T field. To compare the edge (red line) to center (blue line) collection in the inset of Fig. 1(a), the center collection data is multiplied by 5000. The threshold field B_{th} for 0-0 LL in the inset of Fig. 1 (b) increases from 12 to 30 T as the temperature rises from 10 K to 110 K.

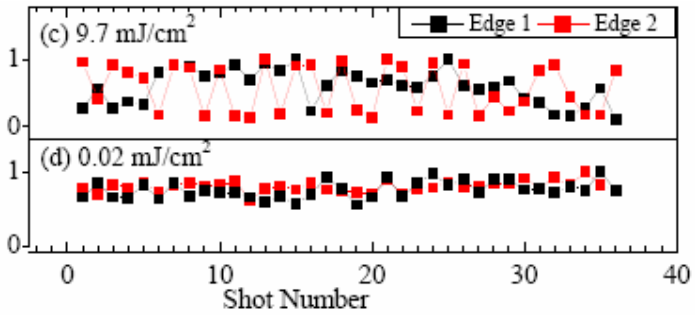

Figure 2. (a) Normalized emission strength for different edges from the 0th LL versus shot number in the SF regime. (b) Normalized emission strength in the ASE regime

Results and Discussion

Figure 1 displays the spectra collected at the sample edge perpendicular to the excitation direction (a) versus B at a fixed F_{laser}, and (b) versus F_{laser}, at a fixed B (20 T) for a temperature of 10 K. A threshold is observed in both cases; inhomogeneously broadened PL peaks (~9 meV) are seen at each interband Landau level transition until F_{laser} and B exceed a threshold value, whereupon a narrow peak (~ 2 meV) emerges from the high-energy side of the broad feature and dominates at high F_{laser}. This transition, along with the associated increase in linewidth at higher B and F_{laser}, is an indicator of SF emission. Figure 2 displays the directionality of the emission for single pulse excitation, obtained by collection from orthogonal sides of the sample. In the SF regime (upper panel), the maximum observed emission strength fluctuates as much as eight times the minimum value, *far greater* than the pump pulse fluctuation. This strong anticorrelation between signals received by different edges indicates randomly changing emission direction on each shot. This is a hallmark of SF: polarization fluctuations grow from initially incoherent quantum noise in the electronic system and reach a macroscopic level, leading to randomly oriented dipole and subsequently random bursts.

Acknowledgements

This research was sponsored by the NHMFL IHRP and NSF (DMR0325499).

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SPIN AND VALLEY SPLITTING OF TWO-DIMENSIONAL ELECTRONS IN GRAPHENE IN THE HIGH MAGNETIC FIELD LIMIT

Y. Zhang, Z. Jiang, J.P. Small, M.S. Purewal, Y.-W. Tan, M. Fazlollahi, J.D. Chudow (Columbia University); J.A. Jaszczak (MTU); H.L. Stormer, P. Kim (Columbia University)

Introduction

Graphene, a single atomic layer of graphite, is a monolayer of carbon atoms arranged in a hexagonal lattice. Such a two-dimensional (2D) system is drastically different from the well-studied conventional 2D systems in that the conduction and the valence band in graphene touch at two inequivalent points (K and K') at the corners of the Brillouin zone, where the Fermi level resides. Around those two points (termed "Dirac points"), the 2D energy dispersion relation is linear and the electron dynamics is thus "relativistic", and described by a 2+1-dimensional quantum electrodynamics.[1,2] Such a unique band structure has profound

implications for the quantum transport in graphene. Indeed, it was recently discovered that graphene exhibits remarkable phenomena, such as an unusual half-integer quantum Hall effect (QHE) as a consequence of the exceptional topology of its band structure.[3,4] In particular, the half-integer QHE observed in high mobility graphene reveals that there is one Landau level situating exactly at the Dirac point, and all the Landau levels have degeneracy of 4, accounting for spin 1/2 degeneracy and the double valley degeneracy. Further investigations of the QHE states near the Dirac point will significantly advance our knowledge of the dynamics of the Dirac Fermions in graphene.

Experimental

We performed magnetotransport measurements in graphene samples in very strong magnetic field, in 33 T and 45 T (hybrid) systems with the ^{3}He cryogenic setup.

Results and Discussion

The splitting of Landau levels n = 0 and ±1, caused by the lifting of spin and valley degeneracies in strong magnetic fields, is observed at T<5 K. In particular, the quantum Hall states ν = ±4 are found to arise from the spin splitting of Landau level n = ±1. The effective Lande g-factor measured at this state is close to 2. The spin origin of ν = ±4 is further confirmed in magnetotransport experiments performed in a tilted magnetic field. The correction to the measured g-factor due to Landau level broadening is also observed.

Conclusions

Both spin and valley degeneracies are all lifted in strong magnetic fields. While the spin lifting is understood, the valley degeneracy lifting poses intriguing theoretical questions.

Acknowledgements

This work is supported primarily by the Nanoscale Science and Engineering Initiative of the National Science Foundation under NSF Grant No. CHE-0117752, and by the New York State Office of Science, Technology, and Academic Research (NYSTAR).

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SUPERCONDUCTIVITY - *Applied*

The ITER fusion reactor is now a formal international project. More than 500 tons of Nb3Sn conductor will be used in the toroidal and central solenoid. Enormous stresses mean that tests of the strain performance of the very high current cables are essential. In the present tests reported by J.V. Minervini *et al*. of MIT, a 36-strand 14,000 amp cable test facility has been built to fit into the 20 T Bitter magnet that will help validate the conductor design for this important magnet system.

CHARACTERIZATION OF Nb_3Sn SUPERCONDUCTING WIRES UNDER STRAIN

J.V. Minervini, M. Takayasu, D. Harris, L. Chiesa (Massachusetts Institute of Technology, Plasma Science and Fusion Center)

A series of tests of the ITER CS model coils confirmed that design and fabrication methods for a large scale niobium-tin cable-in-conduit conductors (CICC) magnet had been well developed for 13 T field operation with a 40 kA pulsed-current of 1.2 T/s. These tests, however, also indicate the importance of further understanding the strain effects of Nb_3Sn conductor cables, including longitudinal load strain and transverse bending effects on individual strands in the large conductor. Degradations due to transverse strain and bending effects on large Nb_3Sn superconducting cables like ITER conductors have been investigated in various ways; bending tests of strands and external transverse load tests of a sub-sized cable using the 20 T, 195 mm bore high-field large-bore DC magnet. This work will contribute to understanding strand and cable degradation effects that need to be included in design criteria and magnet performance analysis.

Fixed Strain Bending Test: Strand bending critical current tests of Nb_3Sn internal tin and bronze processed wires have been continued using two types of Nb_3Sn wire developed by a previous experimental method. The investigation has been expanded over a wide bending range up to 1.4 %. Nb_3Sn wires were heat-treated in straight form, and then mounted in a groove on a Ti-6Al-4V clamping-fixture at room temperature in order to bend the wire to a predetermined radius. The preliminary results of the critical current vs. bending show noticeable difference between the two wires. In the low bending range up to 0.4% the critical currents of IGC internal tin wire decrease, while those of Furukawa bronze wire increase. Microstructure cracking investigation of these bending strand samples have be carried out at the University of Wisconsin, Applied Superconductivity Center after the critical current test, in order to correlate the critical current results with filament breakage.

Single Strand Variable Bending Test: A variable bending device has been designed and constructed for characterizing the critical currents of Nb_3Sn superconducting strand under pure bending. This device allows applying strand bending during a test operation in liquid helium. The strand is placed on a support beam plate that is bent through an evolution of pure bending states. This device is capable to apply a large range of bending strain up to 0.7%. The principal mechanism is similar to the previous work of Goldacker, *et al.* with high temperature superconductor. The challenge to our device development was to establish a given uniform bending to a strand over a large range of applied strain under an electromagnetic force that was generated by the background field and the transport current of the test sample. Verification tests have been performed in the magnetic field of 12 T at 4.2 K. The measured bending distribution along the loaded beam over the sample of about 100 mm length remained within 20% of the intended bending strain. This work will be continued to characterize new ITER wires presently under development.

Effect of transverse stress on the critical current of a 36-strand Nb_3Sn cable: In this experiment, the transverse pressure is applied mechanically. The goal of the experiment is to investigate a critical current degradation of CICC Nb_3Sn cables due to a transverse load. The test cable is composed of 36 (3x3x4) strands resembling the sub-cable pattern 3x3x4x4x6 of an ITER CS cable. To measure the transverse load applied on the cable in the 10-20 MPa range, a strain gauge array is mounted along the outer surface of a sample holder ring while the cable is placed in a groove on the inner surface. The strain gauges measure the surface expansion strains due to the applied transverse load as well as the hoop force due to the electromagnetic Lorentz force of the cable itself. The transverse load is applied to the cable using a conical wedge that transfers a vertical force to a radial, horizontal force. The conical wedge pushes against the sample holder ring while the conical wedge is sliding vertically, so that the cable is squeezed between the ring and the conical wedge. The vertical force is provided with a linear actuator mounted on the top dewar cover flange. The critical current is measured as a function of magnetic fields between 14 T and 10 T at a fixed transverse load. The load is increased in steps. Preliminary tests have been performed and confirmed the probe functionality. A sub-cable of ITER CS model coil wire will be tested early next year.

Acknowledgements

This work was supported by the U.S. Department of Energy, Office of Fusion Energy Science under Grant Number: DE-FC02-93ER54186.

The great hope for cheap conductors of the high temperature superconductors are the coated conductors made from YBCO deposited with excellent biaxial texture on strong Ni alloy substrates. In this work by C.L.H. Thieme *et al.* of **American Superconductor Corp.**, small coils have allowed critical currents of over 300 A in fields up to 19 teslas, validating the capability of this new conductor type to operate in the very high field regime.

SMALL DOUBLE PANCAKE COILS MADE WITH SECOND GENERATION HTS WIRE TESTED IN HIGH MAGNETIC FIELDS

C.L.H. Thieme, D. Aized, D.T. Verebelyi, J. Voccio (American Superconductor Corp.)

Introduction

For the production of Second Generation (2G) HTS, based on a Coated Conductor approach, American Superconductor (AMSC) uses a low cost combination of deformation-textured NiW substrates (RABiTS™) with an epitaxial oxide buffer layer and a solution-based YBCO layer.[1-3] The high field properties at 4.2 K make the 2G conductor interesting for high field NMR, and for this reason small coils are tested on a regular basis at 4.2 K and fields between 10 T and 25 T.

Experimental

In the past year the processing width of the 2G HTS conductor was extended to 40 mm, an important step in the scaling of its manufacture. At the end of all deposition steps the 40 mm wide foil was slit into multiple 4 mm wide insert conductors that were subsequently laminated to copper 155 on both sides for mechanical and electrical stabilization. A second step was the modification of the YBCO chemistry to reduce the difference of the critical current in perpendicular and parallel magnetic fields with regard to conductor plane. This field dependence of the doped YBCO was explored at 20-25 T using a modified ITER barrel that could accommodate tape-like conductors. Second, coil manufacture was developed for the 4.4 mm wide conductor that included improved insulation and epoxy impregnation methods. Several double pancake configurations were tested that used 10 m long conductor pieces per pancake.

Results and Discussion

The doped YBCO conductor was tested in a parallel field orientation. The performance was virtually unchanged compared with the undoped conductor (450 A/cm-width at 25 T). As the parallel field performance can decrease with an increase in the perpendicular field performance, this was an important confirmation of the high field, 4.2 K performance, and of the capability of the slitting and lamination steps at the end of the 2G HTS manufacture. The ITER barrel configuration was very useful even at these high fields, and with a tape-like geometry.

The double pancakes were tested at fields of 0-19 T, the first time this configuration was explored at 4 K and high fields. The assembly stably carried 300 A in fields of up to 19 T. The assembly quenched at 340 A, independent of field, likely caused by the inner joint where current transfers from one coil to the other. Repeated quenching did not damage the coils. Coils were measured again in zero field at 77 K, and the self field performance was exactly the same as before the 4 K measurements.

Conclusions

Second Generation HTS conductors are made using an important scaling step, processing at a 40 mm width and slitting to final conductor with at the very end. The stabilization, applied through a lamination process, has been very effective for 4.2 K, high B performance. The conductor geometry is sturdy enough to allow coil construction suitable for use at fields up to 19 T, without any further reinforcement. The double pancake coils could be safely quenched without sustaining damage. Dopants used to increase the field performance in the c-axis direction could be included without seriously affecting the parallel performance, as was demonstrated at fields between 20 T and 25 T.

Acknowledgements

The authors wish to acknowledge the financial support from NIBIB, NIH, and from the U.S. Department of Energy.

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Measurements by U.P. Trociewitz *et al*. in the 45 T Hybrid magnet show that Bi-2212 conductors made at **Oxford Superconducting Technology Inc**. show a maximum in the pinning force at 4.2 K only at the maximum DC field of about 45 T. An irreversibility field of about 100 T is inferred from this data. The results of this Magnet Lab-corporate collaboration are excellent news for the technology of magnets beyond Nb because these conductors also have critical current density, suggesting a useful superconducting performance well above 30 T—the next big mark for high field superconducting magnets.

Bi2212 SUPERCONDUCTORS IN HIGH FIELD APPLICATIONS

U.P. Trociewitz (NHMFL); J. Schwartz (NHMFL/FAMU-FSU College of Engineering); K. Marken, H. Miao, M. Meinesz, B. Czabaj (Oxford Superconducting Technology Inc., NJ)

Ag-clad Bi2212 superconductor is a potential candidate for the innermost sections of high-field magnets like the proposed 1.3 GHz (30 T) NMR magnet.[1,2] At 30 T, 4.2 K Bi2212 has very high J_c, which makes it not only the conductor of choice but also the only available conductor for this type of application. In a collaborative effort with Oxford Superconducting Technology (OST), the most recent generation of multifilament round wires is currently being studied in a variety of aspects in an effort to creating a data base to allow for reliable parameterization of new magnet designs. Round wire is isotropic and is not sensitive toward a specific field orientation. It can be manufactured in a variety of diameters, it can be aspected if necessary (with anisotropy) and above all has the obvious advantage being more easily implemented in a standard layer-wound approach.

A series of transport characterizations on short round and aspected samples has been completed providing an overview of $I_c(B)$ properties of Bi2212 round wires up to 45 T, as presented in Figure 1. Several aspects become readily clear from the graphs: Bi2212 round wire offers excellent transport properties over a wide range of applied magnetic fields. At an aspect ratio of 1.5:1, Bi2212 wires show only small anisotropy when rotated in field, particularly in the high-field range, offering improved packing density in a winding pack at small trade-off in transport property. Though there is considerable scatter in I_c in the low field range, $I_c(B)$ curves of samples with various geometries and slight variations in composition as well as heat treatment parameters appear to converge at around 10 T where strongly linked current paths take over, which may alleviate some of the restrictions in processing control. At 25 T J_e of Bi2212 wire at 4.2 K is higher than 350 A/mm^2, which is more than double the J_e reported for the best Nb_3Sn wire in the same field range at 1.8 K (150 A/mm^2, 23.5 T).[3] At 31 T the samples retained *n*-values between 14 and 21. Pinning force maximum is just being reached at about 45 T and a fit through the available data applying the Fietz-Webb law,[4] and acknowledging the uncertainty due to extrapolation in the field range above 45 T, the upper critical field is in the range of around 100 T, as shown in Figure 2.

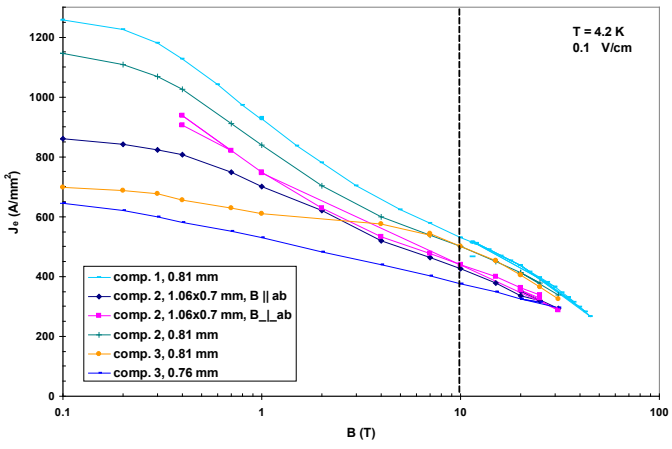

Figure 1. $J_e(B)$ curves for Bi2212 wires.

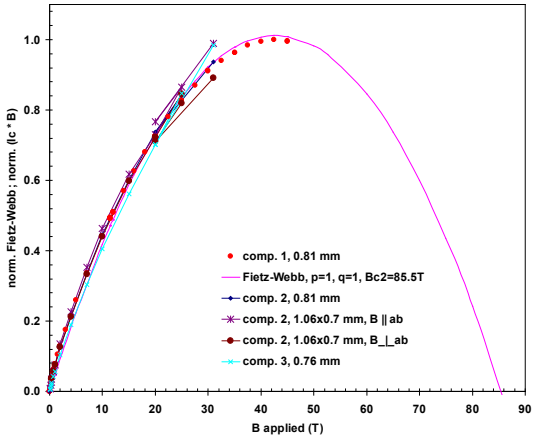

Figure 2. Pinning forces and B_{c2} extrapolation for Bi2212 wires.

A set of test coils with various numbers of turns have been wound to address a variety of processing issues with Bi2212 round wire, like heat-treatment procedures, coil winding approaches, insulation, and conductor homogeneity over longer lengths. Core leakage, which can occur during Bi2212 conductor heat-treatment, is also present in heat-treated Bi2212 wire to some degree. It, however, did appear to neither significantly degrade coil performance nor cause turn-to-turn shorts in coils that were manufactured with glass-fiber sleeve insulated wire. The coils have been successfully tested in a background field of 19 T. To address quench protection requirements of Bi2212 solenoids as part of a high-field magnet system, their quench behavior needs to be studied in detail. Quench experiments on short, 25 cm long, wire samples are currently being carried out in self-field. Quench experiments will be extended on small test-coils soon to study quench behavior of Bi2212 solenoids in a more realistic high field environment.

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SUPERCONDUCTIVITY - *Basic*

In the research of F.F. Balakirev *et al.*, a discontinuity in the doping dependence of the Hall coefficient for $La_{1-x}Sr_xCuO_4$ has been found near the optimal doping (x=.16). Just above approximately the same concentration the temperature dependence of R_H persists down to the lowest temperatures. That contrasts to its saturation for samples with smaller x. The authors consider the two features as a signature of a phase transition near optimal doping.

NORMAL-STATE HALL EFFECT IN HIGH-T_c $La_{2-x}Sr_xCuO_4$ AT LOW TEMPERATURES

F. Balakirev, J.B. Betts, A. Migliori (NHMFL, LANL), G.S. Boebinger (NHMFL/FSU, Physics), I. Tsukada, Y. Ando (CRIEPI, Tokyo, Japan)

We report Hall effect measurements in the normal state of the high-T_c superconductor $La_{2-x}Sr_xCuO_4$ (LSCO). The Hall resistivity was measured by suppressing superconductivity in 65 T magnetic field, thus revealing the normal-state behavior in the low temperature limit down to 0.5 K. The Sr doping x is varied from underdoped (x= 0.08) to overdoped (x=0.22) regimes in a set of 9 thin film samples that were prepared by Pulsed Laser Deposition. The resistivity and Hall voltage were measured simultaneously using digital lockin technique developed at NHMFL.

We find a that R_H (T) saturates in samples with $x \leq 0.16$ in the low temperature limit while in samples with $x \geq 0.17$ the R_H (T) remains temperature dependent down to the lowest temperature measured. Temperature and doping dependence of the Hall number, $1/R_H$, normalized per Cu at high magnetic field in LSCO thin film samples with different levels of Sr doping are plotted on Figure 1a.We find a discontinuity in the doping dependence of the Hall number at around optimal doping level. A similar behavior

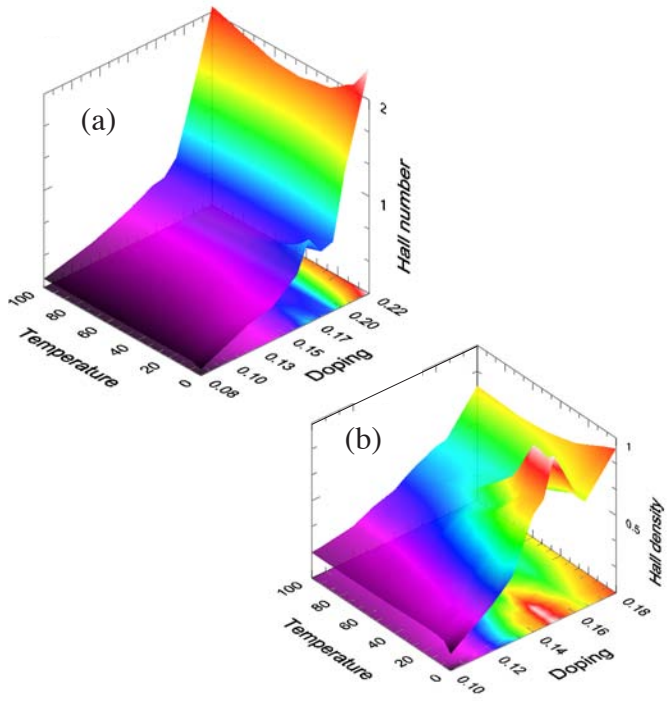

Figure 1. Peaks in the Hall number for two High-Tc compounds **(a)** Temperature and doping dependence of the Hall number at 65 T in $La_{2-x}Sr_xCuO_4$ thin film samples with different levels of Sr doping x. **(b)** Previously published results [1] on the temperature and doping dependence of the Hall number at 65 T in $BiSr_{2-x}La_xCuO_6$ as a function of p, the effective doping.

was observed in another high-T_c compound, $BiSr_{2-x}La_xCuO_6$ [1] (Figure 1b), suggesting a common phenomenon that is generic for high temperature superconductors. This Hall effect anomaly is most commonly associated with a phase transition in the normal state near "optimal doping", the same doping level where superconductivity is most robust.

Acknowledgements

The work at the National High Magnetic Field Laboratory was supported by the National Science Foundation, DOE Office of Science, and the State of Florida.

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1 Balakirev, F., *et al*., Nature, **424**, 912-915 (2003).

In the report by P. Li *et al*., high field measurements of the magnetoresistance, of the critical field, H_{c2}, and of the Hall coefficient in the normal state of the electronically doped $Pr_{2-x}Ce_xCuO_4$ have shown somewhat unusual dependences on the strength of the external field. Although not much information can be extracted directly from the report itself due to its brevity, it is worth emphasizing the importance of obtaining such information for the electronically doped materials to compare with properties of hole-doped cuprates.

RESISTIVITY AND HALL EFFECT MEASUREMENTS IN $Pr_{2-x}Ce_xCuO_{4-y}$ UP TO 60 T

P. Li, R.L. Greene (University of Maryland-College Park, Center for Superconductivity Research and Department of Physics); F. Balakirev (NHMFL-LANL)

We report resistivity and Hall effect measurements in the electron-doped cuprate system $Pr_{2-x}Ce_xCuO_{4-y}$ in pulsed magnetic field up to 60 T. The resistivity and Hall voltage were measured simultaneously using a digital lockin technique developed at the NHMFL. We find negative magnetoresistance (MR) in the underdoped region for all magnetic field values, similar to low field data reported previously.[1] The MR becomes positive at high field in the optimal doped (x=0.15) sample at low temperature. Most surprisingly, we observed a substantial magnetic field dependence of the Hall coefficient at high field (above ~40 T) in optimally doped and overdoped samples (from x=0.15 to 0.19) (Figure 1). A spin density wave induced Fermi surface reconstruction model can be used to explain this phenomenon. We also report for the first time the parallel upper critical field (H//ab plane) for $Pr_{2-x}Ce_xCuO_{4-y}$.

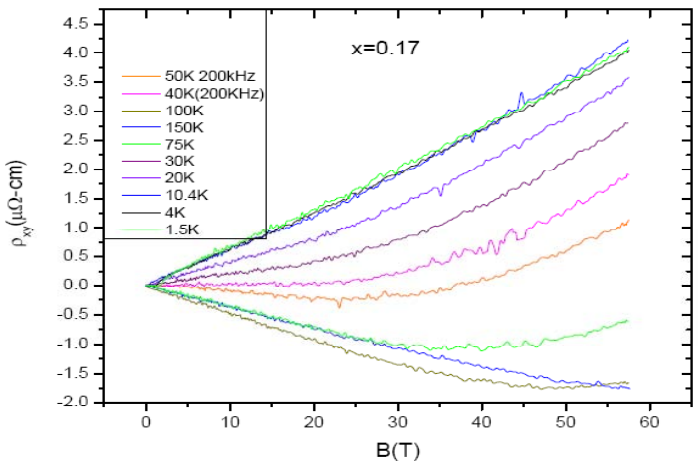


Figure 1. Magnetic field dependence of Hall resistivity in a $Pr_{1.83}Ce_{0.17}CuO_{4-y}$ thin film sample.

Acknowledgements

This work is supported by NSF Grant DMR-0352735. The work at the NHMFL was supported by the National Science Foundation, DOE Office of Science, and the State of Florida.

References

1 Dagan, Y., *et al.*, *Phys. Rev. Lett.*, **94**, 057005 (2005).

In the report by J. Singleton *et al.*, attenuation of the angle-dependent magnetoresistence oscillations (AMRO) with temperature in quasi-2D organic superconductor *k*-$(BEDT\text{-}TTT)_2Cu(NCS)_2$ behaves as T^{-5} (i.e., in a phonon like manner), which is in the remarkable contrast with the T- dependence, T^2, for the in-plane resistivity. This seems to indicate breaking of the transverse inter-plane coherence in the material at finite temperatures.

*This research was supported by the **NHMFL Visiting Scientist Program**, which provides financial assistance for new users and early career researchers. VSP pays special attention to increasing the diversity of the MagLab user program.*

THE COHERENCE CONUNDRUM IN BEDT-TTF SUPERCONDUCTORS; THE PROLONGED DEATH OF INTERLAYER TRANSPORT AS TEMPERATURE RISES

J. Singleton (NHMFL-LANL); P.A. Goddard, A. Ardavan (University of Oxford); R.D. McDonald (NHMFL-LANL); S. Tozer (NHMFL-FSU); J. Schlueter (Argonne National Laboratory)

Introduction

Much recent attention has focused on so-called "bad metals", systems that appear to be Fermi liquids at low temperatures, but whose conductivity falls below the minimum metallic limit as the temperature rises.[1] Examples of this behavior may be found among the cuprates, the ruthenates and organic superconductors.[1,2] A key question in such systems concerns the coherence of the electron orbitals, and whether, as suggested by Anderson and others, it is destroyed by thermal fluctuations as the temperature T rises.[2] Upon such suggestions has a wealth of theoretical supposition been based.

Experimental details and results

To address this question, we have studied magnetic-field-orientation-dependent transport in the crystalline organic superconductor κ-(BEDT-TTF)$_2$Cu(NCS)$_2$ at Ts of up to 45 K.[3] This material was chosen because it possesses a simple low-temperature Fermi surface that has been very well characterized by experiment.[2]

Experiments were carried out using two-axis rotator probes from Oxford and LANL in the 33 T Bitter and 45 T hybrid magnets at Tallahassee. We find that the angle-dependent magnetoresistance oscillations (AMROs) (Figure 1) due to orbits on the quasi-one-dimensional and quasi-two-dimensional Fermi-surface sections are suppressed by rising T with a power-law dependence suggestive of phonon scattering (Figure 1). This result is an interesting contrast with the zero-field resistivity, which follows a T^2 dependence that has been attributed to electron-electron scattering. The coherence peak in the resistivity seen in exactly in-plane fields, and other signatures of a three-dimensional Fermi surface, remain to values of T that exceed the proposed Anderson criterion for incoherent interlayer transport by a factor of order 80. This result suggests that some of the work using the Anderson criterion as an underlying justification may be at best questionable. Theoretical studies of this question are in progress.

References

1 Hussey, N.E., *et al.*, *Phil. Mag.*, **84**, 2847 (2004).
2 Goddard, P.A., *et al., Phys. Rev. B*, **69**, 174509 (2004).
3 Singleton, J., *et al*., in press.

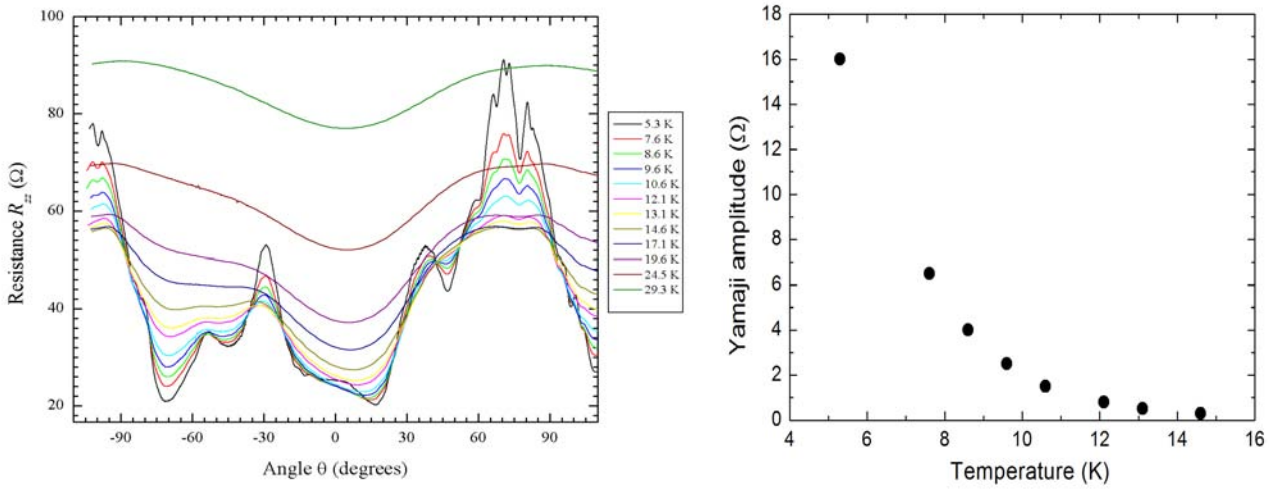
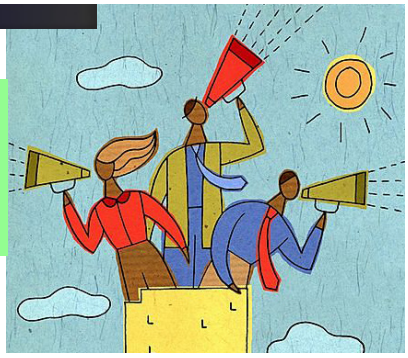


Figure 1. Left; typical AMRO data for a range of temperatures (see inset key); θ is the angle between the field and the normal to the sample quasi-two-dimensional layers. Right: amplitude of a typical AMRO feature (Yamaji oscillation) vs. temperature, showing a T^{-5} dependence suggestive of phonon scattering.

PEOPLE IN THE NEWS



Irinel Chiorescu, an assistant professor of physics at Florida State University and researcher at the Magnet Lab, has been awarded a prestigious Sloan Research Fellowship by the Alfred P. Sloan Foundation. The fellowship is designed to identify and reward early career scientists who show the greatest promise for making outstanding contributions to new knowledge in basic science. As such, it includes a two-year, $45,000 research-support grant. Chiorescu is one of only 116 in North America to receive such an award, and the only faculty member from the state of Florida.

"The Sloan fellowship distinguishes Irinel as one of the very best young faculty members in the country," said Gregory Boebinger, director of the Magnet Lab. "It also shows the level of respect for the faculty and research staff at the FSU physics department and the Magnet Lab." The Sloan Research Fellowships are awarded annually in seven fields: chemistry, computational and evolutionary molecular biology, computer science, economics, mathematics, neuroscience and physics. Since the fellowship program was created 51 years ago, 32 Sloan Fellows have won Nobel Prizes later in their careers, and hundreds have received other honors.

IRINEL CHIORESCU

PETER FAJER

Chiorescu earned his Ph.D. from Joseph Fourier University in Grenoble, France, and is a condensed matter physics experimentalist. His research focuses on quantum spin dynamics and nanotechnologies. "I am truly honored to receive the Sloan award, which strengthens my commitment to succeed in the future," Chiorescu said. "The growing and diverse scientific environment at FSU and the Magnet Lab puts the research goals of my lab within reach."

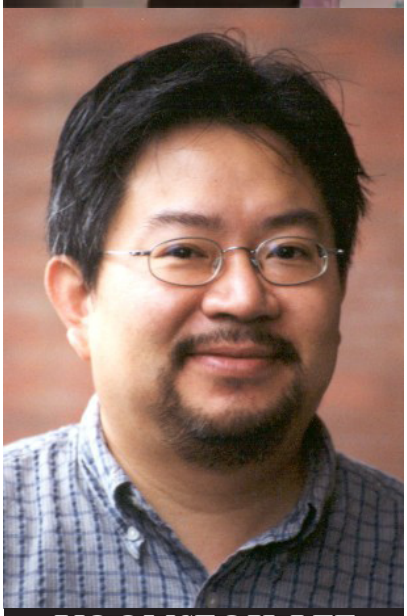
Five physics department faculty members have been named Sloan Fellows; four of them also are affiliated with the Magnet Lab. Besides Chiorescu, the other past winners from FSU are: Pen Xiong (1998), Kun Yang (1999), Vlad Dobrosavljevic (1997), and Nicholas Bonesteel (1996).

Peter Fajer, FSU professor of biology, has accepted the position of **interim director of the Magnet Lab's EMR program**. The Lab's Electron Magnetic Resonance and Free Electron Laser programs will continue to draw on Louis-Claude Brunel's expertise and his leading role in the merging of pulsed EMR techniques with Free Electron Lasers, an important new

Magnet Lab collaboration with the University of California at Santa Barbara. Fajer received his Ph.D. from the University of Leeds, England. He was an FSU Developing Scholar in 1998 and received an FSU Teaching Award in 1998. His general research interest is in molecular structure-function relationships as applied to motility. The organizational level is that of large macromolecular complexes—the interface of single-particle biochemistry/ biophysics and cell biology.

Kristina Håkansson, a Dow Corning Assistant Professor of Chemistry at the University of Michigan and former NHMFL ICR postdoctoral fellow, has won two new honors. She received one of two Analytical Chemistry Grantee Awards from Eli Lilly & Co. (see *Chemical & Engineering News* 2005, **83**, 34, 12 Dec. issue). That award is presented to new professors who have quickly distinguished themselves as leaders in the field of analytical chemistry. It is an unsolicited, unrestricted grant funded at a level of $20,000 per year, renewable for a second year, and will support her research combining ECD (electron-capture dissociation), EDD (electron-detachment dissociation), IR MPD (multiphoton dissociation), H/D exchange, and other structurally informative techniques to determine nucleic acid structure and sequence, characterize protein posttranslational modifications, and map macromolecular contact surfaces. In addition, Dr. Håkansson will receive a five-year $560 K NSF Career Award for related research, in recognition of high performance and potential as an outstanding junior faculty member in chemistry.

KRISTINA HÅKANSSON



YOONSEOK LEE

RICHARD PAGE

Yoonseok Lee, UF assistant professor of physics and director of the Microkelvin Lab, has been selected to receive a 2005/2006 Advisor of the Year Award from the College of Liberal Arts and Sciences. Lee has also been designated as the CLAS candidate for the *University-wide* advising award competition, which will be decided later in the spring. Lee was recognized for all of his efforts as the SPS Faculty Advisor. Lee was named Physics Teacher of the Year in 2004. He began teaching at UF in 2001 and has received several awards and grants, including $450,000 from NSF's CAREER program. He received his B.S. and M.S. in physics from Seoul National University and his Ph.D. in physics from Northwestern University. Lee was a Alfred P. Sloan Research Fellow from 2004-2006 and received an AKPA President's Award for Outstanding Young Researcher in 2002.

Richard Page, a graduate student in the NMR program, received a travel award from ISOTEC to attend the Experimental NMR Conference this April in Asilomar, CA. Only two awards are handed out every year and there are many worthy applicants. Page's poster is entitled "Comprehensive Evaluation of Solution Nuclear Magnetic Resonance Spectroscopy Sample Preparation for Helical Integral Membrane Proteins" and represents a collaborative effort with the research groups of Charles Sanders at Vanderbilt University, Frank Sonnichsen at Case Western Reserve University, Stanley J. Opella at UCSD, and Timothy Cross at the NHMFL. This research represents

SERGEY PANKOV

ANDY POWELL

NEIL HARRISON

a major breakthrough in sample preparation for solution NMR spectroscopy of membrane proteins.

Sergey Pankov is a new postdoc in the Condensed Matter Science group. He received his Ph.D. in physics in 2003 from Rutgers University and his B.S. in math and physics and M.S. in physics from the Moscow Institute of Physics and Technology. From 1997 to 2003 he worked with G. Kotliar, his Ph.D. advisor, on the magnetic energy anisotropy from the spin-orbit interaction in functional formalism. In 2002, he worked at the Kavli Institute for Theoretical Physics in Santa Barbara on the Hertz Millis theory of 2D antiferromagnetic fluctuations in the vicinity of the Quantum Critical Point. From 2003 to 2005 he worked at the Laboratoire de Physique Theorique, Ecole Normale Superieure and collaborated with V. Dobrosavljevic, of the Magnet Lab, on the nonlinear screening theory (extended dynamical mean field theory) of the Coulomb glass.

Andy Powell, of the Instrumentation and Operations group, has been promoted to electronics engineer, replacing Peter Murphy who passed away on June 10, 2005. Powell has a B.S. in electrical engineering from Florida State University. He started working at the Magnet Lab about eleven years ago while attending the FAMU-FSU College of Engineering. Powell has worked in the Electronics Shop his entire Magnet Lab career, where he designs and builds electronic instruments for the researchers. He also designs magnet protection systems, helps maintain the resistive magnet power supplies, and provides technical assistance to in-house researchers and external users.

NEIL HARRISON RECEIVES 2005 LOS ALAMOS FELLOWS' PRIZE FOR OUTSTANDING RESEARCH

Ground-breaking discoveries and outstanding contributions to the field of condensed matter physics have earned **Neil Harrison** a prestigious 2005 Los Alamos Fellows' Prize for Outstanding Research. Harrison, a staff scientist at the Magnet Lab in Los Alamos, New Mexico, was recognized for his work using high magnetic fields to make pioneering discoveries in strongly correlated materials. The Fellows' Prize for Research honors those who make a significant impact in the scientific field.

"I am delighted, certainly grateful, and very much humbled for having been selected for this award at Los Alamos," said Harrison. "I am much indebted to others at Los Alamos, [the Materials Science and Technology] Division and most importantly the National High

NEIL HARRISON

CONTINUED FROM PAGE 38

Magnetic Field Laboratory, who have contributed to the creation of the ideal environment for conducting exciting research in condensed matter physics and material science in high magnetic fields."

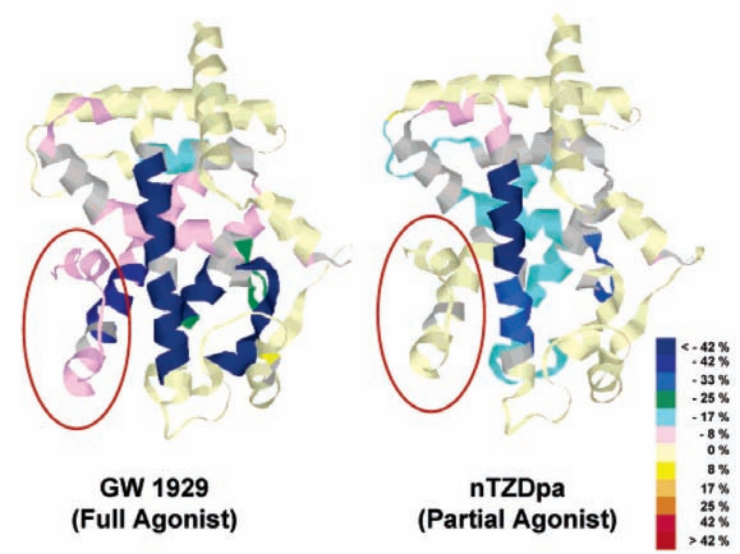
Laboratory employees nominate staff members for the Fellows' Prize. A committee of Laboratory Fellows reviews the nominations and recommends its selection to the director. This year, the committee received nine nominations for the Fellows' Prize for Research. In addition to the honor of being selected, winners receive a plaque and a check for $3,000.

Harrison came to the Pulsed Field Facility as a postdoc in 1996. He earned his bachelor's and doctoral degrees in physics from the University of Bristol in the United Kingdom. Alex Lacerda, associate director for user operations for all three sites and director of the Pulsed Field Facility, had strong praise for Harrison: "He's one of the big reasons why the Magnet Lab is among the most scientifically productive high magnetic field labs in the world."

MAGNET LAB/SCRIPPS FLORIDA COLLABORATION GIVES SCIENTISTS POWERFUL TOOL FOR DRUG DISCOVERY

Researchers at the Magnet Lab and Scripps Florida have developed and evaluated a robust new system for analyzing how drugs bind to proteins. This groundbreaking work could speed the delivery of potential new drugs and improve existing ones. The work, which appeared in *Analytical Chemistry*, is the first published paper to result from a partnership between Scripps and a Florida university [**M. Chalmers**, "Probing Protein Ligand Interactions by Automated Hydrogen/Deuterium Exchange Mass Spectrometry," *Anal. Chem.*, 2006, **78**, 1005-1014 (2006)].

Scripps Florida is a state-of-the-art biomedical research institute currently located in Jupiter, Fla., on the campus of Florida Atlantic University. Scripps announced Florida would be home to its second facility in 2003, with the Florida Legislature agreeing to appropriate $310 million for the organization's start-up costs.



Change in H/D exchange rates for binding of a full agonist (left) and partial agonist (right) to free PPARγ LBD.

The collaborative research is focused on getting a more accurate picture of human proteins, which are the target of most drugs. Understanding the nature of the interaction between a drug and a protein—where the drug attaches and where it doesn't—is one of the keys to drug research, because the exact placement of a drug can determine whether it enhances a natural biological function or counteracts it.

"By pairing the magnet lab's expertise in high-field research with Scripps' expertise in protein dynamics and drug development, we can create a kind of map that shows where drugs bind to the surface of proteins," said **Alan G. Marshall**, director of the lab's Ion Cyclotron Resonance (ICR) program and the Kasha Professor of Chemistry and Biochemistry at FSU. "We can do that because our technology is the best way to generate highly accurate pictures of tiny amounts of protein molecules."

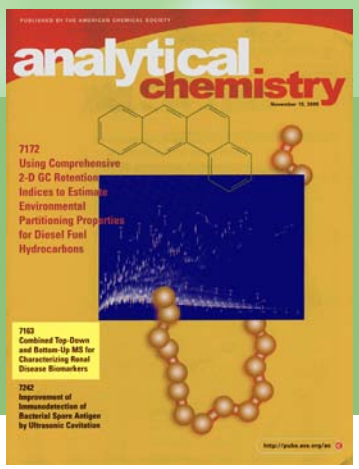
Marshall said the experiment detailed in *Analytical Chemistry* can best be described as "molecular spray painting." Here's how it works: the receptor protein with a drug stuck to it is dipped into a solvent called "heavy water" (deuterium oxide, or D_2O). In the portions of the receptor that can exchange with heavy water (regions not involved in hydrogen bonding), the natural hydrogen atoms are gradually replaced by deuterium atoms, which increase the mass from 1 to 2 mass units. Scientists then dissect the receptor and use the magnet to weigh pieces of it to see which segments of the receptor remain covered up by the drug.

The team saw the potential of probing human protein molecules with this spray-painting technique, but also recognized that the experiment was limited by several factors. Each test that would have to be performed would take anywhere from one minute to several hours, and each measurement would be slow. To ensure the reliability of the experiment, the process would need to be replicated twice more to validate the results, adding additional days to the process.

The paper published in *Analytical Chemistry* lays out the results of research to improve the technical aspects of the experiment. By utilizing the high-field ICR magnet and its powerful spectrometer, coupled with a sample preparation robot, the scientists were able to extract data that show how the drug alters the dynamics of the receptor upon binding. This application of the experiment can measure changes in a fraction of the time—and show those changes over time. And the results are highly reproducible.

"This research is important because it gives us a new and very powerful way to probe the interaction between drugs and proteins," said Patrick Griffin, professor of biochemistry and head of drug discovery at Scripps Florida. "Because we've now solved many of the technical problems, this technique is sure to play an even larger role in understanding the mechanism of action of many classes of drugs."

Now that the data acquisition has been automated, the next step is automating the data analysis. The amount of data generated by the magnet's high-test mass spectrometer is staggering: 1 million data points every second. To analyze the data by hand would take a month. With automated software being developed at the Magnet Lab, the analysis will take just a few minutes.



A recent paper by **Mike Chalmers** (now at Scripps Florida) is featured on the cover of the latest issue of *Analytical Chemistry*, [Chalmers, M.J., *et al*., "Combined Top-Down and Bottom-Up Mass Spectrometric Approach to Characterization of Biomarkers for Renal Disease," **77**, 7163-7171 (2005)]. Chalmers was a postdoc for Alan Marshall in the ICR Program while working at the Magnet Lab.

National High Magnetic Field Laboratory

1800 East Paul Dirac Drive
Tallahassee, FL 32310-3706
Tel: 850 644-0311
Fax: 850 644-8350
www.magnet.fsu.edu

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