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MAG LAB REPORTS

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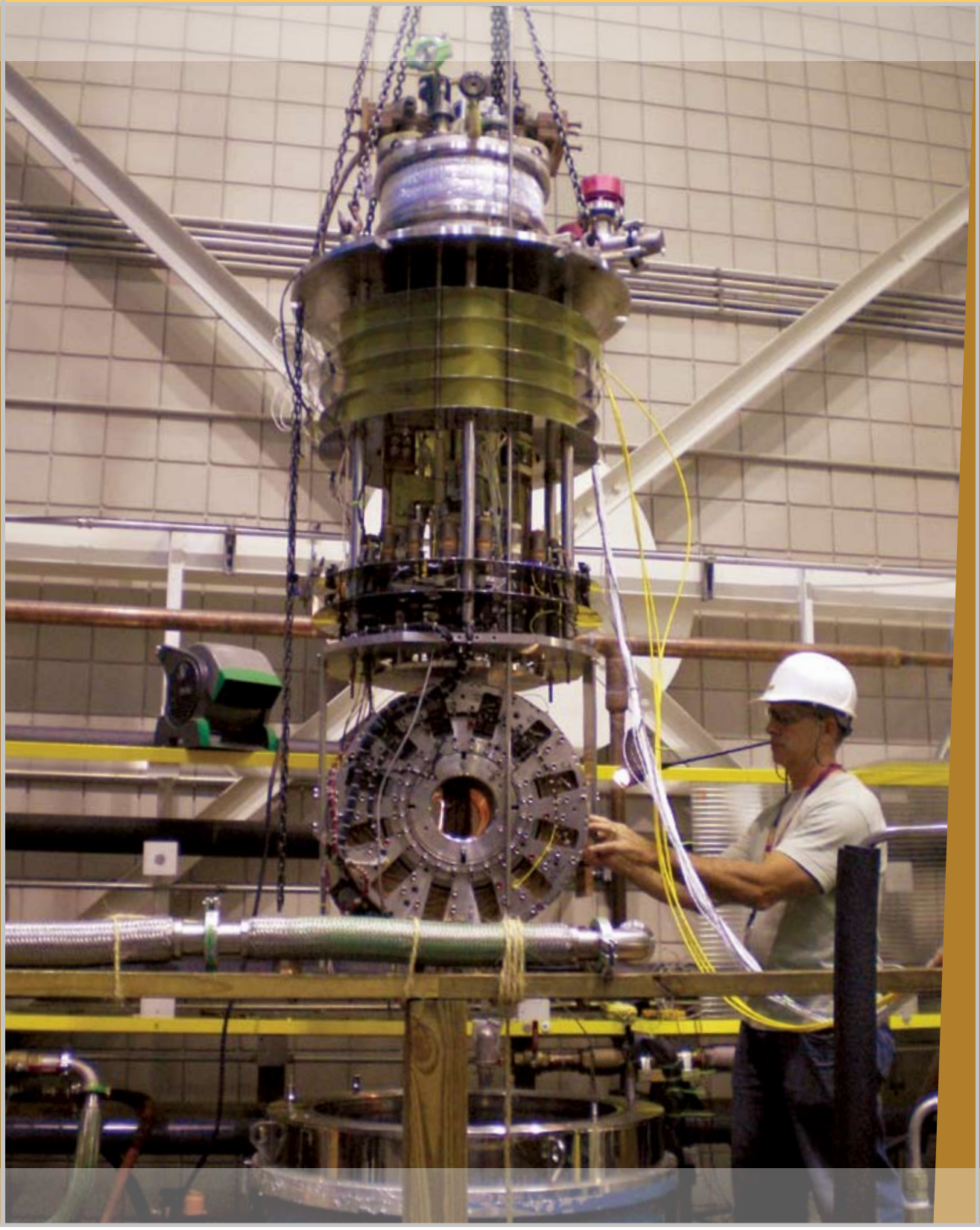

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On the cover:
The superconducting split pair magnet, with a CICC sample discernable protruding vertically through its bore, being lowered into the cryostat in preparation for a test. See page 8 for more.

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Open discussion, excitement at User's Committee Meeting

By Greg Boebinger and Eric Palm,
who provided all of the factual stuff.

Our annual Users Committee Meeting was held in Gainesville from November 6 to 8 and included two day-long seminars preceding the business meetings. One seminar focused on thermometry and thermal measurements in high magnetic fields, the other on new capabilities in magnetic resonance imaging. Both drew scientists from rival laboratories to compare techniques in greater detail than is possible at most conferences.

Greg Boebinger

Foremost on the minds of many users was the budget crunch we've faced during the past year - something that's been discussed in this column before and something that we've weathered so far…having kept the DC magnets up and running without interruption! (something that I feared we would not be able to do when I composed the fall 2008 "Director's Desk" column). I am extremely proud of the MagLab and the user community for their belt tightening and the Florida State University for being such a strong partner as we faced together the additional budget cuts from the state of Florida.

HIGHLIGHTS FROM THE DC, PULSED PROGRAMS:

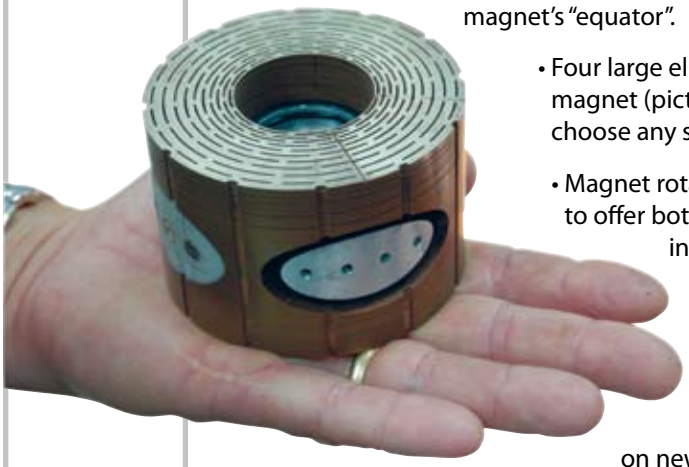
- The User Committee endorsed "flex time," a plan facilitating more efficient use of our DC magnets. Each magnet run will be 35% longer, from 7 to 9.5 hours of magnet time daily. However, by necessity, power consumption will become the limiting factor for each experiment. Each experiment will be assigned a megawatt-hour limit, with consideration of the measurement technique, e.g. "fixed field" measurements versus "swept field" measurements. The magnet control computers will display real-time power usage, as well as the remaining MW-hr budget remaining for the entire run. We hope this will keep the MagLab's power budget under control – or at least "70% contained" (as they say about wildfires - right before they get out of control).

- The users were happy to hear about increased accessibility to high magnetic fields: the imminent completion of the 100 T pulsed magnet rebuild at Los Alamos and the now-expanded High B/T facility at the MagLab / University of Florida. The High B/T user program now offers access to a second microkelvin magnet system and a third dilution refrigerator for "debugging" experiments before loading them into one of the two High B/T systems.

- The users expressed excitement (really!) about the soon-to-be-submitted proposal to construct a terahertz free electron laser at the MagLab, a major initiative to further extend the scientific impact of high magnetic field research (see article on page 5).

- And they were thrilled beyond measure – or at least they seemed pleased – to hear about our new cryogenic systems for the 32 mm bore resistive magnets: a top-loading sorption-pumped He-3 insert with base temperature of 325 mK and a variable temperature insert for ultra-stable temperatures from 1.5 K to room temperature. These systems will increase data throughput by making temperature changes faster and more stable, and will be more reliable than our previous systems (which is manager-speak for "fewer ice plugs and frozen probes").

- Finally, there was healthy skepticism when the MagLab director once again reported that the split magnet was back on track... an annual event held since the late 1950's it seems. The oft-user-requested split magnet project was delayed nine months in 2008 to shift more money to buy DC magnet power. Design work for the split magnet is back on track and we hope to spend roughly $750 k on this magnet in 2009…a big chunk of the total cost for completion by the end of 2011. The split magnet will achieve 25 tesla, with added functionality:



• Optical access not only down the bore of the magnet but also via four ports at the magnet's "equator".

• Four large elliptical ports crossing right through the equatorial plane of the magnet (pictured). This recently-tested MagLab design enables users to choose any scattering angle.

• Magnet rotation to provide either vertical or horizontal magnetic field, to offer both Faraday and Voigt geometries for light scattering, as well as increased flexibility for sample rotation experiments.

Finally, a few key MagLab leadership changes: **Alex Lacerda** has returned to the MagLab as the center leader of the MagLab's Pulsed Field Facility in Los Alamos, after serving almost a year as the interim division leader of the Materials Physics and Applications Division at Los Alamos. **Art Edison** has taken on new responsibilities as director of chemistry and biology at the MagLab, a role that will help focus and direct the MagLab's many initiatives to publicize and grow its research programs in chemistry and biology and related disciplines. MagLab scientists and scientific review committees have proposed many exciting research opportunities and Art will help develop these opportunities and help me to understand, and eventually spell, all those big words that chemists and biologists tend to use. **Steve Hill** is now at the helm of the lab's electron magnetic resonance (EMR) program (more from Steve on page 6). **Nat Fortune** of Smith College has been elected to be chair of the User Committee. We're confident that Nat will bring the same forthrightness and commitment to user issues that made Steve Julian (the outgoing chair) so effective. We thank Steve for his service in the MagLab's user committee and for agreeing to begin serving on our External Advisory Committee as soon as the big ball drops in Times Square.

And on that note, on to the 2009 budget crunch!

Rock and roll,

Gregory S. Boebinger

GREGORY S. BOEBINGER

Let the 'Big Light' shine

By Greg Boebinger

Five workshops, four years, and $2 million later, the design of the *"Big Light"* free electron laser (FEL) is complete and the Magnet Lab is moving forward with a proposal to construct a fourth-generation terahertz (THz) light source alongside its DC magnet facility at Florida State University.

The input of FEL experts, as well as physicists, chemists and biologists from around the U.S. and Europe have defined specifications that offer unprecedented brightness, time-resolution and continuous tunability over the entire terahertz frequency range (roughly 0.3-300 THz).

To minimize costs and time to "first light," the FEL design developed by Jefferson Laboratory assembles components of proven technology – brought together in a novel configuration to provide several unique features:

- Three co-located narrow-band light sources with overlapping frequency ranges
- Co-location of a fourth source, a THz broad-band source
- Ultra-fast THz light pulses (1 psec pulses with a 10 MHz rep rate)
- Ultra-bright THz light pulses (1 million times brighter in the THz regime than third-generation synchrotrons)
- Automatic time-synchronization of near IR, mid-IR and broadband THz sources (< 20 fsec jitter for pump-probe experiments)

The design involves a photo-injected 60 MeV linac for high efficiency, low operating costs and eventual potential enhancement to higher beam-current operation (the initial planned beam current is 3 mA). The FEL is designed in an energy recovery configuration (ERL), the primary benefit for a user facility being the absence of a radioactive beam dump, which in turn means that the accelerator vault can be entered at short notice to perform maintenance and adjustment.

There are three FEL undulators. One, the far-infrared (FIR) FEL, is driven by the injector; the mid-infrared (MIR) and near-infrared (NIR) FELs are in the ERL loop and can be run simultaneously using the same electron bunches. The broadband THz source is also situated within the ERL loop, so that time-correlated pulses from NIR, MIR and THz sources can be used in multi-color experiments.

RESEARCH USING MAGNETIC FIELDS

The *Big Light* source will permit new and enhanced (magneto) optical spectroscopies, pump-probe measurements, pico-second time-resolved experiments, nonlinear absorption and multi-photon techniques across the same frequency range as the orbital, spin and nuclear quantization provided by the intense magnetic fields at the Mag Lab. Other newly enabled experiments will be founded upon the internationally recognized expertise in microscopy, nuclear magnetic resonance (NMR) and Fourier-transform ion cyclotron resonance (FT-ICR) that already exists at the Magnet Lab.

The *Big Light* FEL will be world-unique even without utilizing the Mag Lab's magnetic fields. The Mag Lab's experience running a first-rate user support infrastructure will benefit both high-field and zero-field experiments in *Big Light's* user program.

- **Quantum matter:** resonant spectroscopy of low-energy correlated states, electron spin resonance of d- and f-electron systems, cyclotron resonance of m*~1 systems to 45 tesla, ESR to 1.25 THz with T2 times as short as 1 nanosecond, qubit characterization and manipulation
- **Nanoscience:** Scanned-probe optical nanoscopy for imaging nano-scale phase separation and performance of new superconducting materials
- **Energy:** Sold-state chemistry for catalysis and energy storage, f-electron chemistry for radioactive waste mitigation
- **Complex fluids:** infrared multi-photon dissociation for selective bond breaking, mass spectroscopy for petroleum analysis
- **Macromolecules:** multi-frequency dynamics via spin labelling, vibrational mode coupling via Raman spectroscopy
- **Biomedicine:** nanoscale dynamic imaging of tissue via coherent anti-stokes Raman scattering

As part of its NSF mission, the Magnet Lab strives to enhance existing and create new experimental techniques in response to scientific opportunities. The addition of *Big Light* to the nation's research tools will provide transformational(!) research opportunities in materials, biomolecules and more. Look for an update on *Big Light* in 2009.

The outlook is bright for the Mag Lab Electron Magnetic Resonance user program

By Stephen Hill

Director, EMR User Program

Stephen Hill

Longtime Magnet Lab collaborator Stephen Hill has accepted a position as director of the Electron Magnetic Resonance (EMR) user program and as an FSU physics professor, cementing a relationship that has been building since Hill's postdoctoral work with the lab nearly 14 years ago. Hill comes to the lab from the University of Florida, where he was an associate professor of physics and a Magnet Lab affiliate.

Thanks to outstanding stewardship by Interim Director Peter Fajer, I have the luxury of taking over the reins of an electron magnetic resonance (EMR) program that is in tremendous shape. So far this year, we have hosted or collaborated with 36 different groups around the world; of these, 12 were first time users. The quality of publications in 2008 is extremely high, including articles in *Nature Physics*[1] and *Physical Review Letters*[1-5], with many more in the pipeline (see future editions of these reports).

The EMR group's successes can attributed to significant recent advances in instrumentation, particularly in terms of high-frequency pulsed EPR. Indeed, the Magnet Lab EMR group now boasts the highest-frequency pulsed EPR spectrometer in the world: 336 GHz with 50 ns time resolution[1]. This development can be expected to have a profound impact on studies related to electron-spin-based quantum information processing, thus positioning the Mag Lab EMR group as a world leader in this field. A Zeeman field of 12 tesla corresponds to a thermal energy of 16 K, enabling essentially complete polarization of the electron spin system at pumped liquid helium temperatures. This is extremely important, because fluctuations of the electron spin bath represent the main cause of decoherence in concentrated spin systems, e.g. crystals of molecular nanomagnets[4]. Thus, by polarizing the spin bath, the dominant source of decoherence is completely removed, opening the door for detailed studies of coherent quantum spin dynamics in highly ordered crystalline materials for the very first time[1]. In addition to pulsed EPR capabilities, the group has expanded its high-frequency coverage considerably for high-field measurements (up to 900 GHz).

Next year promises to be even more exciting for EMR at the Magnet Lab. The group is seeking to expand not only its ranks, but also its research portfolio. We are presently searching for a scholar/scientist with expertise in biological EPR. In the next weeks, we will interview several outstanding applicants for this position and our hope is that the successful candidate will start in the first half of 2009. The group also is conducting a broad search for up to three postdoctoral associates in areas of physics, chemistry and biology.

In January of 2009, I will move my own research laboratory (and group) from the University of Florida to the Magnet Lab. This will immediately make available two new multi-high-frequency (8-500 GHz), high-field instruments to the users' program. In addition, several new (funded) capabilities are planned for the next 1-2 years, including: a superheterodyne spectrometer based on solid-state sources providing continuous frequency coverage from X-band to 1 THz; a two-axis superconducting vector magnet system; sub-Kelvin temperatures; and a split-gap magnet with optical access for frequency domain spectroscopy. The broadband capabilities will immediately be compatible with the DC resistive magnets, including the 45 T hybrid; a longer-term goal is to develop a frequency domain spectrometer compatible with the high-field resistive split-gap magnet that is currently under development at the Magnet Lab. We also plan to develop a low-temperature EMR capability for the resistive facility.

An important goal for the coming year will be to engage more students in the activities of the EMR group. Initially, I will bring several graduate students with me to Tallahassee from the University of Florida. I also plan on recruiting additional graduate students from the FSU ranks. My experience has been that well-mentored graduate students can quickly become proficient in the use of highly sophisticated EMR spectrometers, and can also significantly enhance instrumentation and/or computational tools developed in-house. These same students are also able to interact with collaborators/users, collect data and follow the analysis through to publication. Both the productivity and quality of research conducted within the EMR group stands to benefit significantly from the recruitment of more graduate students. The long-term goal will be for the group to support 6 to 8 graduate students (~2 per spectrometer). I also encourage collaborators and users to send their graduate students to the Magnet Lab to participate in experiments. This approach worked extremely well at the University of Florida, and several external graduate student users have already visited the lab this fall.

The integration of research and education will be essential for attracting major new funding to support in-house research. Universities in North and Central Florida (FSU, UF, UCF) currently boast some of the world's leading research groups in the broadly defined area of molecular magnetism, in which EPR has played a critical role. This is an interdisciplinary research field with significant potential for future technologies. Together with faculty at UF and UCF, we plan several activities over the next few years aimed at increasing Florida's visibility in the fields of molecular magnetism and EPR, including the organization of regional, national and international conferences/workshops. The long-term goal will be to establish an International Center for Molecular Magnetism and EPR in North/Central Florida, with joint research and educational programs.

In order to maintain a leadership position in high-field EPR it will be essential to continue development of unique instrumentation. An important first step will be to improve sensitivity and temporal resolution for high-frequency (> 200 GHz) pulsed EPR. This will require a significant investment both in high-power microwave sources/amplifiers and in quasioptical spectrometer components. To this end, we are working with several leading groups in the U.S. and Europe to obtain funding to build the next generation narrow-band pulsed spectrometer for the Magnet Lab. Together with the new scholar/scientist, we also hope to improve existing multi-high-frequency continuous-wave techniques to enable EPR studies of biological (aqueous) samples spanning a very broad frequency range. This will enable studies of complex molecular dynamics to unprecedented time scales.

Finally, in January 2009, the Magnet Lab will submit a major proposal to the NSF to support construction of a THz/infrared accelerator-based light source at the DC Field Facility in Tallahassee, the so-called "Big Light." This proposal submission represents the culmination of a four-year design study involving collaboration between the Magnet Lab EMR group (Hans van Tol), Los Alamos National Lab (John Singleton), and Jefferson Laboratory. The design is a 4th generation light source based on the successful Free Electron Laser (FEL) facility at Jefferson Lab. It uses a photo-injected electron accelerator with a beam energy of 60 MeV, a superconducting RF linac, and undulators within optical cavities to generate the light. It also uses energy recovery to minimize capital and operating costs, as well as minimizing radiation hazards. The light source is tunable, covering the wavelength range from 2.5 – 1100 microns. It has a sub-picosecond pulse structure, with a repetition rate of up to 10.7 MHz, and with 10 μJ per pulse. The defining feature of Big Light's topology is the capability of driving three different FELs. Thus, multiple photon beams will be available for pump-probe studies of materials out of equilibrium. If funded, the Big Light/Magnet Lab combination will be truly unique, having an impact on many areas of research at the lab, as well as opening up new research areas in biology and chemistry. Most importantly, Big Light can be expected to have a major impact on the high-field EMR user program.

For more details concerning the future plans for EMR, please feel free to contact me at shill@magnet.fsu.edu.

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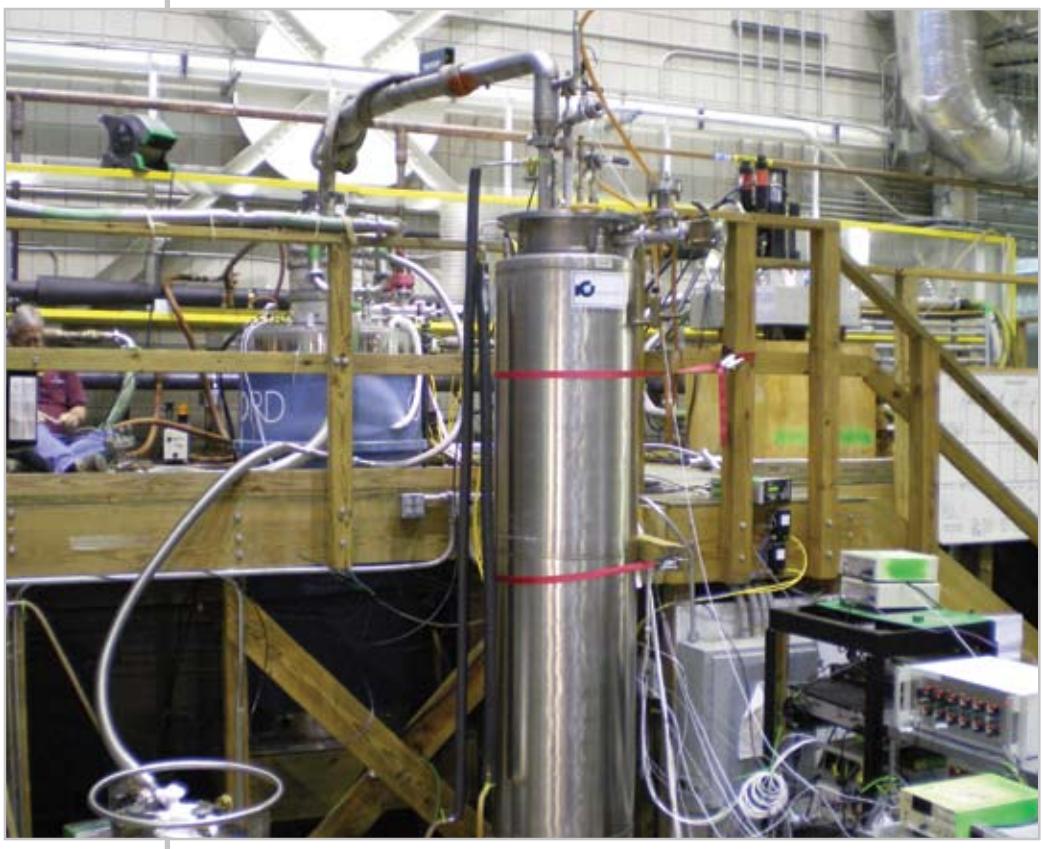
Cable-in-conduit conductor underway for Series Connected Hybrid

By Huub Weijers

Through 2007 and 2008 the Magnet Science and Technology division of the Magnet Lab worked on detailed design and qualification testing of cable-in-conduit conductors (CICCs) for the Series Connected Hybrid (SCH) magnet projects currently underway. One such magnet is funded by the National Science Foundation for installation as a user magnet at the Magnet Lab, and another customized for neutron scattering experiments at the Helmholtz Center Berlin. The qualification testing is now successfully completed, which represents a major landmark not only for the Series Connected Hybrid projects but also for the broader magnet building community that relies on CICCs.



NHMFL employee George Miller on the deck in Cell 16 finalizing the instrumentation. The experiment is being pre-cooled with liquid nitrogen in preparation of a final cool down to 4.2 K (-452 degrees F) in preparation of a test . In the central foreground is the pressurized helium cryostat while the CICC sample and the magnet are in the blue cryostat.

Several years ago problems were discovered with superconducting wire and CICCs within the ITER project, related to strain induced degradation of the superconducting properties that could not be accurately predicted. The problems could possibly affect our Series Connected Hybrid projects as well. A collaborative effort between the Magnet Lab and the ITER community was begun in order to test candidate CICCs. These tests involve currents up to 20 kA, forces up to 25 tons, magnetic field up to 12 tesla in a split pair magnet and a controlled flow of pressurized (up to 5 bar) and slightly heated (5-9 K) supercritical helium through the CICC samples, while measuring voltage signals with sub-microvolt precision. With the development of a new computational model[1] to predict that degradation and a series of 11 full-scale cable tests mostly at the Magnet Lab[2] and available CICC data from other institutions, we have validated the model. Cable tests also confirmed that the selected SCH CICC designs can carry the currents and withstand the mechanical loads as designed.

This is a major landmark in the Series Connected Hybrid project by itself. It's also a huge benefit to the international multi-billion dollar ITER project, a project that former MS&T Director John Miller is now involved with at the Department of Energy's laboratory at Oak Ridge. It is also reassuring to the NSF, as it has been hoping for a timely solution to the previously observed problems with the superconducting wire and CICCs that, through the work at the Magnet Lab, have been solved.

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Fermi surface of superconducting LaFePO determined from quantum oscillations

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R.D. McDonald[3], NHMFL, Los Alamos National Laboratory, USA

LaFePO was the one of the first of the ferro-oxypnictide superconductors to be discovered[1] and has a transition temperature of T_c ~ 6 K. Substituting the pnictogen P for As results in the isostructural compound LaFeAsO, which is non-superconducting and has a spin-density wave ground state but a small amount of electron doping (F replaced for O) that makes this system a superconductor with T_c~26 K[2]. Understanding the details of the Fermi surface of these two isostructural compounds may help to understand the reason for high T_c superconductivity in ferro-oxypnictide. Here we report detailed quantum oscillations measurements, one of the most powerful techniques to determine the details of the Fermi surface topology.

We measure torque magnetometry using piezocantilevers and high quality crystals of LaFePO (size of 200μm x 200μm x 20 μm) in high magnetic fields up to 45 tesla and low temperatures (0.3 K). We find that in the normal state LaFePO has a rich spectra of quantum oscillations (Figures 1a and b) containing at least seven different frequencies associated with the electronic bands centred along the AM direction ($\alpha_{1,2}$, $\beta_{1,2}$ corresponding to minimum and maximum extremal areas of corrugated two-dimensional cylinders) and hole pockets centred along Γ-Z direction (δ, γ, ε) (see Figure 1c). The three-dimensional pocket predicted by band structure calculations is not observed in the current study. The obtained effective mass varies between 1.7-2.1m_e and the mass enhanced for LaFePO is a factor ~2 suggesting a moderate mass effect due to electronic correlations.

The difference in amplitudes of the dHvA signal between the hole and electron pockets is an indication of different scattering rates affecting these orbits. The near-perfect matching between the hole and the electron orbits that we observe suggests that LaFePO (which is non-magnetic) may be close to a spin/charge density wave transition and that magnetic fluctuations are an important ingredient in the physics of the Fe-based superconductors[3].

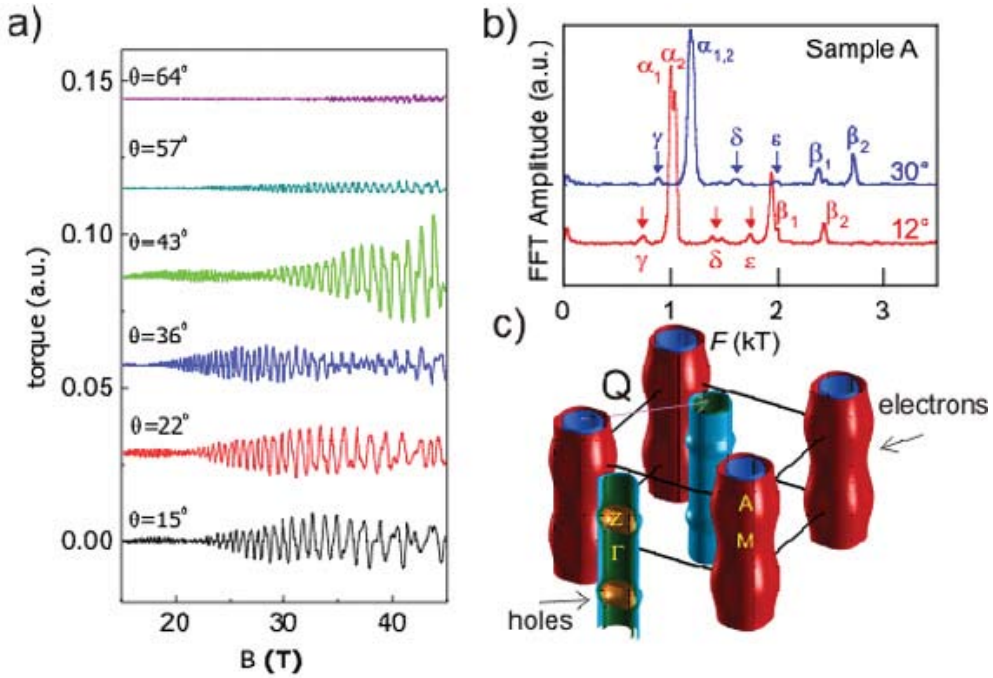

Figure 1a. Torque measurements versus magnetic field in LaFePO obtained in the hybrid magnet at 0.5 K for different orientations θ between the magnetic field and the *c* axis. b) Fourier transform spectra showing the frequencies associated with extremal areas of quasi-two dimensional electron and hole cylinders (see text). c). Fermi surface of LaFePO close to near nesting with the vector Q; the 3D pocket centered at Z is not observed in this study.

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Diffusion MRI at 21 T: Developing New Treatments for Traumatic Brain Injury

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2. College of Human Sciences, Florida State University
3. College of Medicine, Florida State University

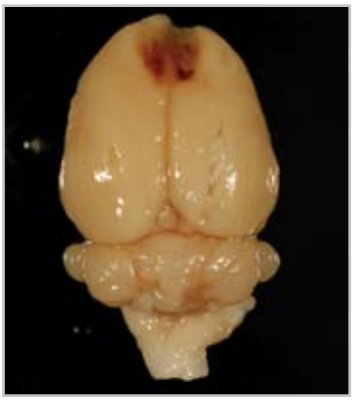

Figure 1.
Medial frontal lobe injury in the adult rat brain. Traumatic brain injury results in a cascade of events starting with edema and inflammation that is leading to a brain cell death and formation of a necrotic cavity. Eventually it is leading to poor behavioral outcomes including impaired learning and memory.

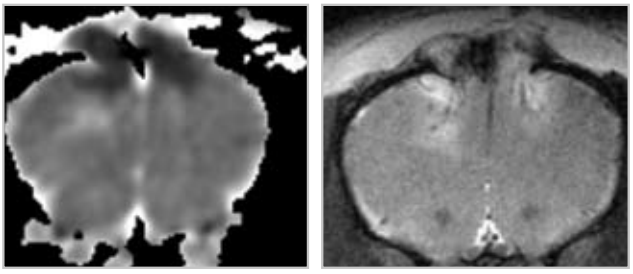

Figure 2 (a, b).
Two MRI images present the same rat and slice position acquired after traumatic brain injury. a - Diffusion map revealing a presence of cytotoxic edema which is noticeable as dark areas. These areas of the brain need a very urgent therapy.
b – RARE MR image showing total edema areas as areas of bright intensity.

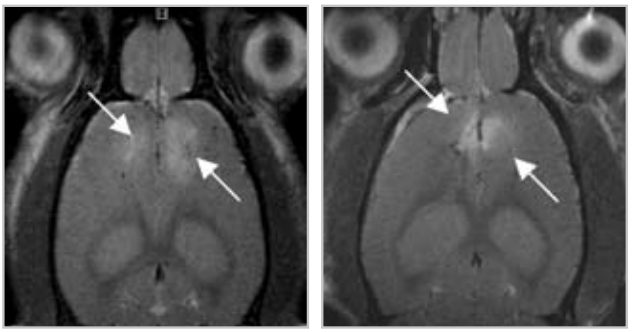

Figure 3 (a, b).
Steroid hormone treatment reduces edema after traumatic brain injury. a - Untreated (vehicle control), traumatic brain injury to the medial frontal cortex induces severe edema 24 hours after injury as indicated by arrows.

b - Treatment with a combination of progesterone and dihydroxyvitamin D (PROG +D) one and six hours after the injury consistently reduced the accumulation of water associated with edema.

TRAUMATIC BRAIN INJURY

Every year 1.5 million Americans sustain a traumatic brain injury (TBI). Of these, about 50,000 die and 235,000 are hospitalized. Currently, as a result of TBI, there are 5.3 million Americans who require long-term or lifelong assistance to perform routine daily activities. The list of devastating impairments that can result from TBI includes loss of both fine and gross motor skills, as well as speech and language abilities. Many patients also experience continuing impairment in learning, memory and cognitive function.

One of the main dangers of TBI is edema, (water accumulation at the site of injury) and swelling. These effects damage brain tissue immediately after injury, and, particularly, the development of edema is associated with poor long-term clinical outcomes. Regrettably, attempts to reduce effects of edema, such as drainage of cerebral spinal fluid, mannitol, and hypertonic saline treatments, have proven to be inadequate in most cases of moderate to severe brain damage.

The development of new treatments to improve outcomes after TBI is limited by our incomplete understanding of the mechanisms responsible for the development of edema after injury. Normally the tight blood brain barrier (BBB) controls the movement of water from the blood vessels into the brain. However, when the BBB is damaged, this allows water to flow into the spaces between the cells of the brain, resulting in swelling known as vasogenic water accumulation. Cytotoxic water accumulation is another source of brain swelling and damage after TBI. Uncontrolled, these and other cellular mechanisms result in neuronal death and a large necrotic cavity (Figure 1).

DIFFUSION MRI AT 21TESLA

This study, for the first time, demonstrates the Magnet Lab's new capability to perform *in vivo* imaging using large rodents. Recently, diffusion MRI for *in vivo* studies was established in our lab to be used with the advanced high magnetic field of 21T. Development of the diffusion pulse sequence included several special measures to prevent natural breathing and other *in vivo* motions from distorting the results of MR imaging. The application of diffusion MRI provided us with an exceptional capability to distinguish two types of edema taking place during TBI. Most valuable is the realization that diffusion MRI represents a unique and very efficient way to detect those parts of the brain, which are subject to a cytotoxic edema (Figure 2). During cytotoxic edema brain cells are incapable of maintaining their low sodium intracellular content and corresponding water is moving into intracellular space, which makes diffusion of that water lower than in normal parts of the brain. This type of brain cell damage is detected in MRI as a dark area on diffusion images of the brain. These brain cells need very urgent help, beginning from the first moments after injury, to avoid future complications.

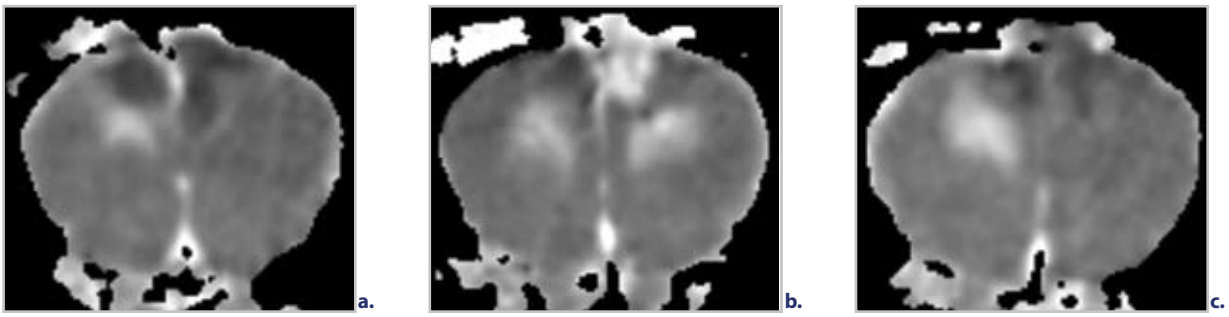

Figure 4 (a, b, c).
Effect of hormone therapy visualized by diffusion MRI: a – untreated animal; (b, c) result of therapy by Progesteron (b) and Thyrotropin Releasing Hormone (c) at one and six hours after injury.

Ultimately, we need new treatments that target both damaged cells, cytotoxic and more time tolerant vasogenic water accumulation. The development of therapies requires a reliable and clear visual separation between the two types of edema and non-invasive detection of changes after treatment to evaluate therapeutic effectiveness. Because diffusion is the most efficient way to visualize regions of the brain that are subject to a cytotoxic edema, we are now using it in conjunction with a number of novel treatments for TBI in a rodent model.

A weight-drop model of TBI in the adult rat was developed that produces an identifiable necrotic cavity and is associated with significant behavioral impairments (Figure 1). This model was used to confirm that administration of hormones at one and six hours post-injury can reduce effects of injury at 24 hours post-TBI exam.

NEW THERAPIES FOR TBI

A joint collaboration between the Magnet Lab, FSU College of Human Sciences, and FSU College of Medicine, as well as graduate students (Silvia Figueiroa, Nicholas Rich) has led to exciting new applications for diffusion MRI to monitor the course of treatment after TBI. In one study, TBI rats were treated with a combination of the hormone progesterone (PROG), which is known to reduce edema, and the active hormone dihydroxy-vitamin D. Both hormones interact with similar nuclear receptors in the brain to regulate neuronal gene expression. Illustration of this therapy can be seen on MR images (Figure 3) performed 24 hours after treatment. The images show that the combination of these two steroid hormones (PROG + D) was more effective than vehicle treatment in reducing edema after injury. We also showed that the combination treatment was better than either treatment alone as indicated by both standard proton imaging and diffusion weighted pulse sequence imaging. These results were confirmed by direct histological measurements of brain edema after injury.

In a second study, TBI animals were treated with progesterone in combination with thyrotropin releasing hormone (TRH). The results of MRI showed the short-term benefits of these treatments against cytotoxic brain damage, which also translated to a significant improvement in learning and memory function of animals in the weeks following TBI. Separate applications of PROG and TRH therapy were both beneficial looking on diffusion MRI (Figure 4) as well as in improving spatial learning and the Morris Water Maze test.

CONCLUSIONS

This work presents the latest achievement of *in vivo* MRI at 21 T. For the first time large rodents have been imaged at this high field MRI scanner. The results demonstrate the power of diffusion weighted imaging at high magnetic fields to detect the short and long-term benefits of brain injury treatments and to predict functional outcomes. It also shows the power of new combinatorial treatments for improving the outcomes after TBI. New tools at the Mag Lab are very valuable for future development of novel therapies for traumatic brain injury that target both vasogenic and cytotoxic edema.

ACKNOWLEDGEMENTS

The *In vivo* MR program at Mag Lab is very grateful to extremely skillful and enthusiastic support from William Brey, Petr Gor'kov, Richard Desilets and Kiran Shetty for their outstanding development of *in vivo* RF probes for MR imaging at 21T. The *in vivo* rat MRI studies were supported by NIH grant R21 CA119177 (PI V. Schepkin) and Hazel K. Stiebeling Fund (PI C. Levenson) and FSU CRC Multidisciplinary Grant Program. The MRI imaging program is supported by Cooperative Agreement (DMR-0084173) and the state of Florida.

Quantum magnetism thaws a spin ice

By Chris Wiebe

The birth of quantum mechanics can be traced back to a number of key experiments that convinced physicists that classical theory cannot be used to describe the atomic and subatomic world. One of these experiments, the low temperature heat capacity of solids, helped to show that (i) energy levels are quantized in matter, and (ii) precise measurements of the entropy, or disorder, within a system could be used to verify models for how atoms move and interact with each other. The natural extension of these experiments was to measure the entropy of magnetic systems at low temperatures to learn about how magnetic spins order. In most systems, the entropy is found to vanish at low temperatures, as predicted by the third law of thermodynamics, but some systems remain disordered or fluctuating down to the lowest temperatures we can measure, usually due to quantum effects. One prominent example of this is liquid helium, which never freezes at ambient pressure at any temperature, but instead forms a superfluid ground state.

Magnetic analogues of fluctuating states ("quantum magnets") such as the ground state of liquid helium are more elusive but the search continues to find model systems to study. The Quantum Materials Group at the Magnet Lab and FSU has recently identified such a system with $Pr_2Sn_2O_7$.[1] The first suspicions of a fluctuating ground state for the magnetic Pr spins began with heat capacity measurements, which are yet again extremely useful for identifying quantum effects in matter. At low temperatures, $Pr_2Sn_2O_7$ was found to have an unexpected amount of "residual spin entropy," which is one of the signatures for unusual magnetism. The physical reason for this spin disorder is rooted in the underlying lattice that the spins reside on – a "frustrated" pyrochlore lattice designed such that the spins cannot order in a conventional fashion. The end result is a fluctuating system of spins that remain dynamic until at least 200 milliKelvin. Recent experiments at the Center for High Resolution Neutron Scattering (CHRNS) at NIST (Maryland) have shown that the spins arrange themselves in local clusters, much like water molecules in ice form in local clusters of chemical and hydrogen bonds.[2] However, unlike water ice, the spins in $Pr_2Sn_2O_7$ are constantly fluctuating, which makes this system a "dynamic spin ice." The study of such states not only provide windows into the quantum world – they are also relevant towards answering questions in high temperature superconductivity, surface physics, and even in protein folding.[3]

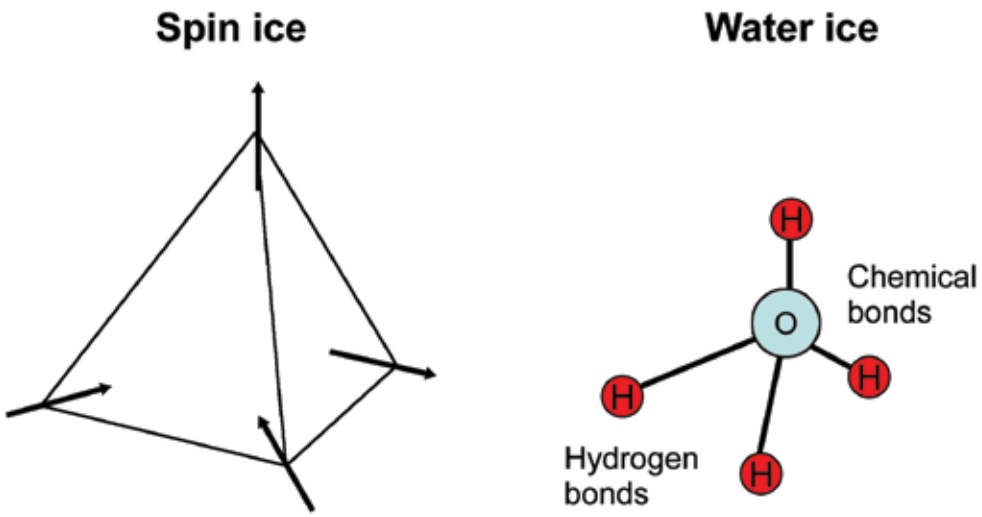

Figure:
The local arrangements of Pr spins in $Pr_2Sn_2O_7$ (two-in, two-out) compared to the bond disorder in water ice (two short chemical bonds, two long hydrogen bonds about each oxygen atom). The difference between the two is the $Pr_2Sn_2O_7$ has spins that are constantly fluctuating, whereas water ice is largely "frozen" into a random orientation of bonds.

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A molecular slinky: How a small peptide can help premature infants take their first breaths

Frank D. Mills[a], Vijay Antharam[a], Seth A. McNeill[a], Suzanne Farver[a], Omjoy K. Ganesh[a], Anil K. Mehta[b], Yilin Meng[a], Adrian E. Roitberg[a], and Joanna R. Long[a,c]

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2. Emory University
3. National High Magnetic Field Laboratory

Inflation of the lungs under ambient conditions requires the formation and upkeep of a stable air-fluid interface in the alveoli. This is accomplished by pulmonary surfactant, a lipid-rich fluid coating the alveoli, reducing the work of breathing and providing a barrier against disease. Inadequate pulmonary surfactant leads to Respiratory Distress Syndrome (RDS), a common condition in premature infants.

Surfactant is 90% lipids and 10% proteins by weight, and its composition varies only slightly among mammalian species. It's primarily made up of phospholipids, including disaturated dipalmitoylphosphati dylcholine (DPPC), monounsaturated phosphatidylcholines (such as POPC), anionic phosphatidylglycerols (such as POPG), palmitic acid, and cholesterol. The two saturated acyl chains in DPPC allow this lipid to pack tightly in a monolayer at an air-water interface. It is thought that the stability of DPPC monolayers is critical to the integrity of the air-fluid interface in the lung. POPC and POPG contain unsaturated fatty acids and increase the fluidity of the DPPC rich lung surfactant, accelerating surface film formation to cover the surface of the alveoli. The resulting monolayer at the interface is hypothesized to contain mostly DPPC while the monounsaturated lipids and surfactant proteins are squeezed out from the monolayer into a surfactant aggregate. These components then form another layer connected to the monolayer.

The lipids in lung surfactant are turned over every five to 10 hours meaning that lipids are constantly being transferred through a network of lipid assemblies to form a stable monolayer (Figure 1). While the lipids in surfactant are important in reducing surface tension and forming a stable monolayer, they cannot act effectively by themselves. Initial clinical studies for potential treatments of RDS using only lipids in surfactant therapies failed, but the use of native lung surfactant isolates met with success. Currently the standard treatment for RDS is administration of lung surfactant isolated from animal sources. Isolated lung surfactant contains small amounts of a key protein, surfactant protein B (SP-B). SP-B facilitates lipid adsorption at the air-fluid interface and is important to intra- and extracellular surfactant trafficking. The hydrophobicity and disulfide bridges in SP-B make purification or heterologous expression of the protein in large quantities impractical.

Lung surfactant replacement therapies containing synthetic peptides are being pursued as an alternative to animal-derived surfactant, as their use would remove the immunologic risks associated with current treatments. They would also allow for greater therapeutic consistency and significantly lower the cost of treatment. One of the most clinically successful synthetic peptides to date is the 21 amino acid peptide KL_4, KLLLLKLLLLKLLLLKLLLLK, which has a periodic hydrophobicity (hydrophilic lysine amino acids separated by four hydrophobic leucine amino acids) similar to the C-terminus of SP-B. The formulation of KL_4

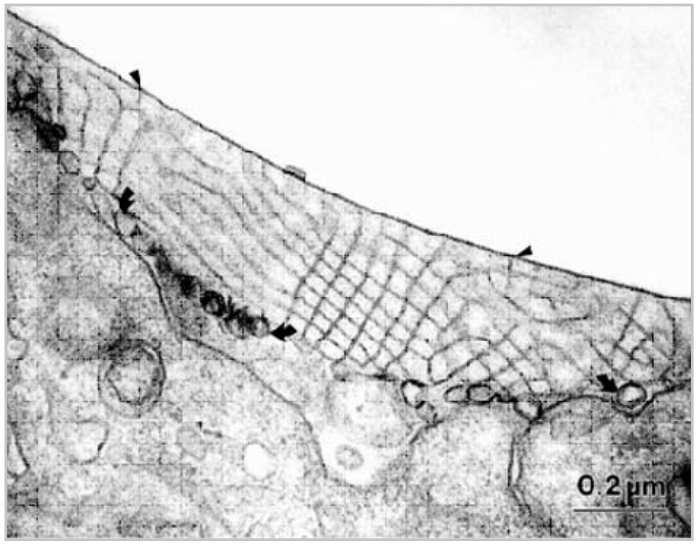

Figure 1.
Transmission electron micrograph of rat intra-alveolar surfactant with the typical lattice-like appearance of lipid assemblies, termed tubular myelin, which is in close contact with the alveolar lining layer (arrowheads). Figure borrowed from Fehrenbach et al., Respiratory Research 2:33 (2001); http://respiratory-research.com/content/2/1/033

with lipids found in native surfactant has shown promise as an alternative to animal-derived surfactant for the treatment of RDS in phase three clinical trials.

A mechanistic understanding of how KL_4 affects lipid properties has proven elusive as the secondary structure of KL_4 in lipid preparations at physiologically relevant concentrations has not been conclusively determined. SP-B has been shown by qualitative spectroscopic measurements to consist of helices lying in the planes of lipid lamellae. Circular dichroism measurements suggest the helical secondary structure of KL_4 is dependent upon the presence of negatively charged lipids (such as POPG) and membrane fluidity, determined by the lipids' degree of saturation. We have also observed changes in the dynamics of the fatty acyl chains in lipid vesicles upon addition of KL_4, consistent with the peptide penetrating deeper into a mixture of disaturated DPPC and monounsaturated POPG lipid bilayers relative to completely monounsaturated POPC and POPG lipid bilayers [1]. Favorable partitioning of a helical peptide or protein into a lipid bilayer results from electrostatic attraction between anionic lipids and basic amino acids as well as a hydrophobic interaction due to amino acids with aliphatic sidechains preferring the hydrocarbon core of the lipid bilayer.

KL_4 represents a unique class of helical, membrane-active peptides, since its primary sequence is intermediate in hydrophobicity relative to amphipathic α-helices, such as antimicrobial peptides, and hydrophobic transmembrane α-helices, which are important in cellular regulation. Its effectiveness in lung surfactant development suggests its unique periodicity may play a role in its ability to mediate lipid trafficking and affect lipid morphology. The spacing of the cationic lysines every 5th residue in KL_4 suggests a structural model in which KL_4 binds to lipid interfaces as an amphipathic π-helix with i→i+5 hydrogen bonding in contrast to a classic, amphipathic α-helix with 3.6 residues per turn and i→i+4 hydrogen bonding. However, the formation of a π-helix by standard peptide sequences is generally energetically unfavorable.

In our structural studies by solid state NMR spectroscopy (ssNMR), we use selective isotopic labeling strategies which allow us to study the conformation of the peptide at physiologically relevant concentrations. In our samples, there are 30-100 lipid molecules for every peptide molecule which leads to significant challenges in terms of sensitivity and making quantitative measurements [2]. Working in collaboration with the NMR probe development group of the Magnet Lab, we have built a magic angle spinning (MAS) probe which allows us to undertake these measurements [3]. This probe makes use of low-E technology [4], giving us larger effective sample volumes and lowering the rf heating during challenging pulse sequences. Using homonuclear dipolar recoupling techniques, we are able to filter out peptide signals from the lipid background (Figure 2) and to probe the backbone secondary structure at specific residues in KL_4, allowing high resolution determination of changes in helicity [5]. In our initial structural studies, we have characterized the structure of KL_4 in a model lipid system containing monounsaturated POPC and POPG lipids by ssNMR and found that it forms neither a classic α– nor a π-helical structure with average (φ, ψ) torsion angles of (-105°, -30°) [6]. More recently we have studied the structure of KL_4 incorporated into 4:1 DPPC:POPG lipid vesicles. This composition mirrors the lipid makeup of the clinical formulation containing KL_4. We have found that KL_4 exhibits two conformations in DPPC:POPG vesicles with the amount of each structure varying between the peptide N-terminus and middle. At the center of the peptide, only a single conformation is

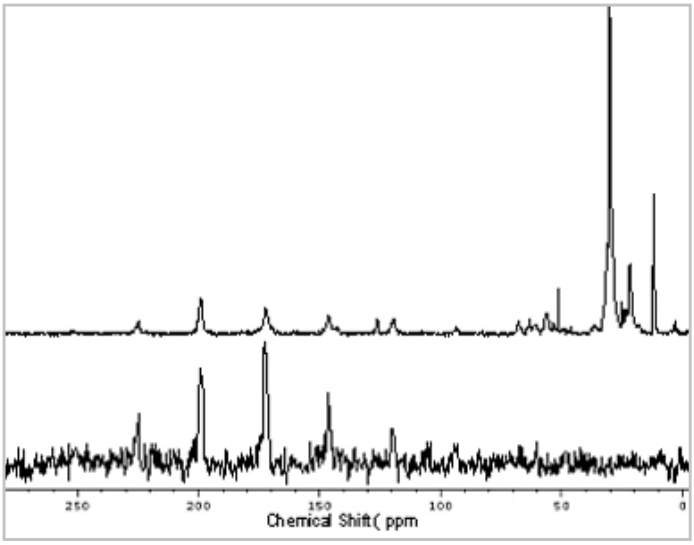

Figure 2.
(Left) CPMAS and DQ-filtered spectra for the 21 amino acid peptide KL4 13C'-enriched at positions L7 and L8 and incorporated into DPPC:POPG lipid vesicles at a peptide:lipid molar ratio >1:50; the signals in the aliphatic region are primarily from the surrounding lipids. (Right) Low-E MAS probe developed at the Magnet Lab.

observed. The first conformation, which is seen at the N-terminus, is similar to the structure found in POPC:POPG vesicles. The second conformation, which is more prevalent, is consistent with average torsion angles of (-63°, -81°) which leads to a lower helix pitch, an increased hydrophobic moment, and is suggestive of i→i+5 hydrogen bonding [7]. If KL_4 partitions deeply into the DPPC:POPG bilayers with the helix axis parallel to the plane of the bilayer, this latter conformation would position the charged lysine sidechains along one edge of the helix where they could preferentially interact with the solvent and polar lipid head groups while allowing the hydrophobic leucine sidechains to be fully partitioned into the lipid interior.

Using molecular dynamics simulations with restraints based on our NMR data, we have been able to replicate the trends we observed in our circular dichroism experiments and examine hydrogen bonding patterns. Simulations for the major conformation in DPPC:POPG vesicles result in two distinct families of structures – the major family is consistent with i→i+4 hydrogen bonding while the minor family is consistent with mostly i→i+5 hydrogen bonding and some i→i+4 hydrogen bonding. The simulation for the conformation in POPC:POPG vesicles shows more fluctuation in the backbone secondary structure. To experimentally verify these hydrogen bonding trends, we measured ^{13}C-^{15}N distances with the REDOR heteronuclear recoupling experiment [8]. The calculated REDOR curves using the MD ensembles of distances for the two conformations agree nicely with experimental data.

The backbone torsion angles for the two helical structures we observe in KL_4, while unusual in classic treatments of secondary structure elements, lie in energetically allowed regions of Ramachandran space. The lipid bilayer environments would favor these conformations as they places all the lysines either on one side of the helix (the POPC:POPG conformation) or along one edge of the helix (the major DPPC:POPG conformation). Although the torsion angles for the second conformation are similar to those for a π-helix, the MD simulations and REDOR data indicate that unlike a π-helix this ensemble has predominantly i→i+4 hydrogen bonding with a minor, yet significant, level of i→i+5 hydrogen bonding.

The structural data for KL_4 in the presence of POPC:POPG and DPPC:POPG vesicles are indicative of two nearly isoenergetic, unique peptide structures. This would allow the KL_4 helix to partition either more deeply or more superficially within lipid bilayers by adjusting helical pitch. The major difference between the POPC and DPPC lipids is a monounsaturated acyl chain in POPC. Our results suggest that altering helical pitch and the saturation of lipid acyl chains play important roles in determining the structure and partitioning of KL_4. A tighter grouping of lysines in the major conformation seen in DPPC:POPG vesicles allows KL_4 to penetrate deeply into the lipid bilayer yet remaining predominantly perpendicular to the bilayer normal (Figure 3). This is consistent with the changes in lipid dynamics we have observed upon addition of KL_4 to DPPC:POPG vesicles.

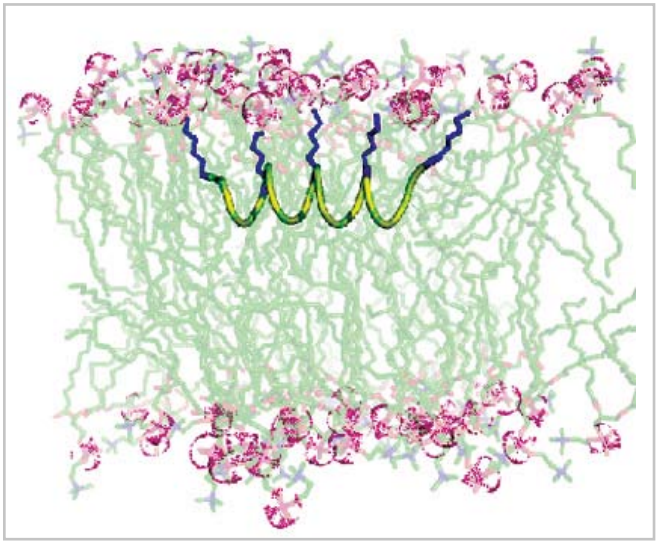

Figure 3.
Model for partitioning of KL_4 into 4:1 DPPC:POPG lipid vesicles. The lysine residues of KL_4 are shown in blue; the length and flexibility of lysine side chains allow them to interact electrostatically with the phosphates in the lipid head groups. However, when all the lipids are monounsaturated the peptide cannot insert as deeply. The properties of KL_4 are dependent on both phospholipid acyl chain and headgroup composition which suggests that KL_4 could play a critical role in lipid partitioning to the lung air-water interface.

The peptide's helical adjustments and variable penetration depth affects the stability and composition of lung surfactant lipid structures by causing positive curvature strain in POPC-enriched domains and negative curvature strain in DPPC-enriched domains. These changes in curvature strain provide a mechanism for lipid trafficking in lung surfactant in a manner that selects for DPPC at the air-fluid interface. Our results illustrate a more thorough molecular model that explains how the relatively simple KL_4 peptide modulates lipid properties; this may enable the development of future SP-B mimetics.

ACKNOWLEDGEMENTS

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Report from the 24th Asilomar Conference on mass spectrometry

New Methods, Instrumentation and Applications of Ion Traps
October 10 -14, 2008
Asilomar Conference Center, Pacific Grove, California

Mass spectrometry conference attendees.

The topic for this mass spectrometry conference varies from year to year. The 2008 Asilomar on Mass Spectrometry was co-organized by Jennifer Brodbelt (U. Texas Austin) and Alan G. Marshall, director of the Magnet Lab Ion Cyclotron Resonance Program. It reprised and updated an Asilomar Conference of similar subject held in 1992 (also co-organized by Marshall). The program was fully subscribed, with 25 talks, 25 posters, and 119 attendees. Sponsored by the American Society for Mass Spectrometry, the conference was also supported by Applied Biosystems/MDS Analytical Technologies, Shimadzu, Waters Corporation, and Thermo Fisher Scientific. Magnet Lab staff (past and present) and users were well represented in the program, as described below.

Marshall opened the conference with an overview, noting that optimal ion trap mass spectrometry performance requires attention to every stage of the experiment: ionization (e.g., use of more basic solvent to extend electrospray negative ionization to non-polar analytes, including polycyclic aromatic hydrocarbons), ion accumulation, mass selection, ion transmission, ion trapping, signal generation and detection, data reduction (e.g., phase correction for up to two-fold increase in mass resolving power), and various ion dissociation techniques. Eugene Nikolaev (Magnet Lab ICR collaborator, The Institute for Energy Problems, Moscow) pointed out that if the number of trapped ions is sufficiently high, then their space charge potential can become comparable to voltages applied to trap electrodes. Single-ion simulation no longer suffices, and it becomes necessary to model the behavior of a million or more ions simultaneously with a supercomputer. He then showed how best to separately calculate the potential contributions from the boundary electrodes, image charge, and ion-ion interactions.

Carol Nilsson (Magnet Lab ICR program staff, now at Pfizer Inc.) discussed how to isolate and identify phosphoproteins and the site(s) of phosphorylation for glioblastoma cells, the most virulent type of brain cancer. Ljiljana Pasa-Tolic (Magnet Lab ICR postdoctoral researcher, now at Pacific Northwest National Laboratory) explained how best to combine high-pressure reversed-phase liquid chromatography and ultrahigh-resolution mass analysis for proteomics, and also introduced a new multi-segmented trapping cell for improved FT-ICR performance.

Christopher Hendrickson (director of instrumentation, Magnet Lab ICR Program) used SIMION analysis to explain and optimize FT-ICR signal detection sensitivity, and reduction of trapping and magnetron sidebands, by appropriate segmentation of the excitation, detection, and trapping electrodes. Hilkka Kenttämaa (Magnet Lab ICR collaborator, Purdue University) described laser-induced acoustic desorption/chemical ionization as a means to achieve uniformly high ionization efficiency for non-polar species such as aliphatic and polycyclic aromatic hydrocarbons, for their successful quantitation in petroleum by mass spectrometry. David Muddiman (Magnet Lab ICR Advisory Panel, North Carolina State University) described atmospheric pressure neutral desorption (with a laser, even without a matrix) and ionization (by electrospray or simply by applying 3 kV to the surface on which the sample is deposited) for application to top-down proteomics, O-linked glycans, and tissue imaging.

Finally, Yury Tsybin (Magnet Lab ICR postdoctoral researcher, now at Ecole Polytechnique Federale de Lausanne) correlated characteristic Electron Capture Dissociation (ECD)/Electron Transfer Dissociation (ETD) fragmentation patterns with peptide structure and hydrophobic/hydrophilic residue distribution, auguring the possibility to infer peptide and protein structure (not just sequence and post-translational modifications) from ECD or ETD MS/MS. Although ECD and ETD share many similarities, ECD detected by FT-ICR and ETD in a multipole ion trap give different product ions due to the effect of ion-neutral collisions in ETD.

Center focuses on teacher support, program evaluation

Fall marked the start of school and a change in focus from summer programming to academic year support for teachers.

This year, the Mag Lab Center for Integrating Research & Learning kicked off a partnership with schools in neighboring Gadsden County, Florida, to provide monthly after-school workshops for K-12 science teachers. The workshops focus on content related to the new Florida Science Standards and on the processes and nature of science. All but one of the public schools in Gadsden County are Title 1 schools. The Center also began its second year of "First Thursdays" after-school workshops for elementary teachers in Leon County.

Science fair season has already begun and requests come in regularly for Magnet Lab scientists to serve as judges at the elementary, middle, and high school levels, as well as for the Capitol Regional Science Fair held each February. We have a core group of intrepid judges who regularly support schools in a three-county area: Eric Hellstrom, Stephen McGill, Maartje van Agthoven, Bill Brey, Todd Adkins, Afi Sachi-Kocher, Mabry Gaboardi, Scott Hannahs, Dragana Popovic, and the entire Center staff.

The Center's research agenda is in full swing with three studies and program evaluation on CIRL programs. The study recently completed by Crissie Grove on teachers who participate in the Magnet Lab's Research Experiences for Teachers program was accepted for publication in the *Journal of In-service Education*. Margareta Pop, who worked with CIRL in 2007-2008 and is now teaching at North Carolina State University, is first author on an article accepted for spring 2009 publication in the *Journal of Elementary Science Education: Research Experiences for Teachers: Motivation, Expectations, and Changes to Teaching Practices due to Professional Development Program Involvement*. Pop will present results of this study at two major national conferences in 2009.

Roxanne Hughes, a Ph.D. student in Educational Leadership and Policy Studies, is conducting ongoing research on the Center's SciGirls summer-camp program for middle-school girls. Her study, *Retention Policies and Practices for Women in Science: A Single Sex Camp and Its Effects on Adolescent Women,* is being presented to the Florida Association for Research in Education and to the Research on Education and Women conference. Brandon Nzekwe, a Ph.D. student in Educational Psychology and Learning Systems, initiated a Research Experiences for Undergraduates tracking study in fall 2008 of the 188 participants in the Magnet Lab's REU program between 1999 and 2008. Using Web-based resources and old-fashioned detective strategies, 60 percent of the participants have been located and some 35 have responded. A Magnet Lab REU facebook page is just one strategy being used to find out where former REUs are, what their terminal degree was or is, if they are in the private sector or academia, and whether they continued on their planned career path. Pat Dixon, director of the Center, is working with former colleague Ryan Wilke, now a measurement and evaluation specialist at the Florida Center for Interactive Media, on a study of science fair participation in the last 20 years. There is no doubt that Center educators and graduate students from Florida State University are making a contribution to the literature of science education and to the informal science education community.

Jose Sanchez, the Center's assistant director and program director for REU and RET, continues to improve the online application system. It's now open for applications earlier than ever before. Summer 2009 programs will accept 12 REU students and 12 RET participants with applications accepted through April 3, 2009 for REU, and March 19, 2009 for RET. REU continues its funding from the core Magnet Lab grant and RET is funded under ESI-0553769.

The Center recently supported an Engineering Research Center proposal submitted by North Carolina State University in partnership with the Center for Advanced Power Systems (CAPS) and FAMU-FSU College of Engineering and Arizona State University to provide educational programming.

The Center will oversee five young scholars from a Title I high school, two RET positions for teachers from that school, and curriculum development to provide graduate students with strategies for classroom visits on renewable energy. As part of its commitment to furthering women and minorities in science, renewable energy activities will be included in the 2009 SciGirls summer camps. Mag Lab Center educators will oversee preliminary data collection to track students' science class choices and college career paths. Still in the planning stages, we expect activities to begin in summer 2009 with the Center for Advanced Power Systems (CAPS) providing mentors and graduate students.

Mag Lab educators continue to find new ways to expand and enhance programming while adhering to the Center's mission to expand scientific literacy and encourage interest in and pursuit of scientific studies among educators and students of all ages.

Science Starts Here

NAME:

Fatma Kaplan

POSITION:

Postdoctoral associate
University of Florida, College of Medicine and Advanced Magnetic Resonance Imaging and Spectroscopy facility
2005-Present

CURRENT WORK:

Fatma works on *C.elegans,* a small soil nematode. She and her colleagues are isolating compounds that *C.elegans* use to communicate with each other, such as finding a mate. They use small molecules to navigate their environment like humans use their five senses.

IN HER OWN WORDS:

"I learned to always be prepared to seize opportunities as they arise.

"I had access to some of the most powerful magnets and most sensitive NMR probes in the world. This allowed me to study natural products, which are found in very low quantities that would have been undetectable with other instruments. I have also had the opportunity to work with a number of the scientists at the Magnet Lab.

"I learned a lot about analytical chemistry and natural products, and I plan to use this knowledge to understand how plants, nonpathogenic nematodes and bacteria interact."

HOW MENTORS MAKE A DIFFERENCE:

"I learned a lot from Dr. Art Edison, whom I consider as a mentor. His open-mindedness to new people and ideas facilitates innovation. Additionally, he uses all of the information available to him to generate ideas and research questions. I think these are important skills for life and research."

"Science Starts Here" showcases young scientists whose career paths have been greatly shaped by their experiences at the Magnet Lab.

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