NHMFL REPORTS
NATIONAL HIGH MAGNETIC FIELD LABORATORY
WINTER 1997
Florida State University • University of Florida • Los Alamos National Laboratory

Power Supplies Installed for Quasi-Continuous Magnet at LANL

Power converters and their associated controllers were recently installed at NHMFL Pulsed Field Facility at Los Alamos National Laboratory to energize the quasi-continuous magnet, which will produce a 60 T flat-top pulse for 100 ms from five 64 MW converters. The liquid nitrogen cooled magnet, which will be the most powerful of its class in the world and the first of its kind in the United States, consists of nine concentric coils that are grouped electrically into three independent sections, as shown in Figure 1. The converters receive power from the 1430 MVA / 600 MJ inertial energy storage generator.[1] Three of the five converters have been installed and individually tested at rated power and energy with an ohmic-inductive load. The remaining two will be ready by May, along with two "extra" converters for the world's first non-destructive 100 T magnet, to be completed later.

Power Supply Design

Each power converter is supplied with 21 kV from the 1430 MVA / 600 MJ energy storage generator.[2] At the synchronous speed (1800 rpm), 1260 MJ are stored in the generator rotor and shaft train. The generator can release up to half of this energy during

LANL *cont. on page 15*

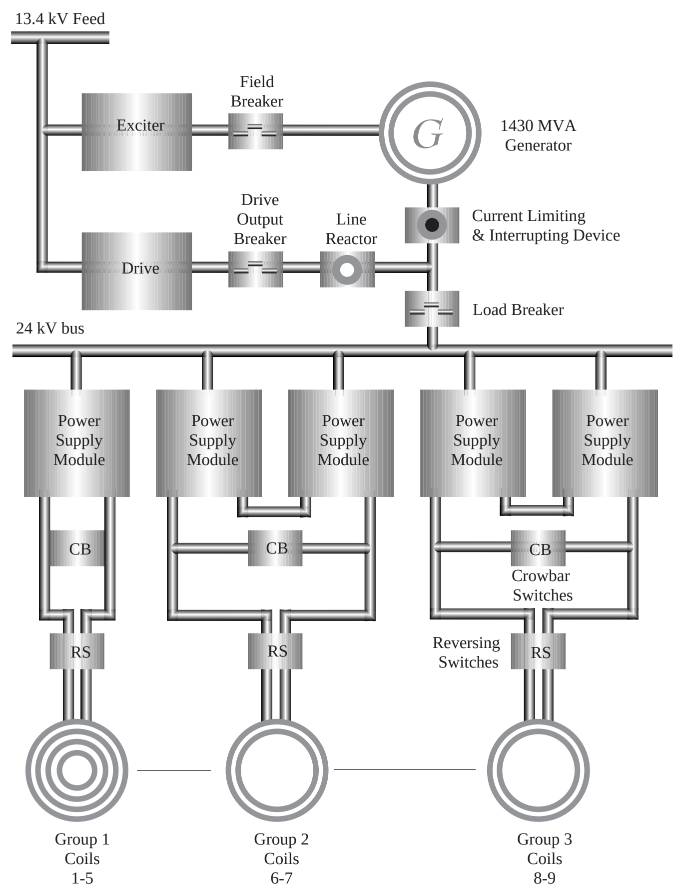


Figure 1. Overall 60 T/100 ms magnet system layout.

Interdisciplinary NHMFL Team Extends Upper Limit for Protein Mass Spectrometry by an Order of Magnitude

Accurate determination of protein molecular weight to within 1 Dalton would be a boon to protein characterization. It would then become possible to: (a) count the number of disulfide bridges (–S–S– is 2 Dalton lighter than 2 –SH); (b) identify deamidation (–NH_2 is 1 Da lighter than –OH); (c) identify such post-translational modifications as phosphorylation and glycosylation; (d) resolve and identify adducts; (e) identify variant amino acid sequences; etc. At first sight, determination of the molecular weight of a neutral protein to within 1 Dalton from measurement of the mass of its gas-phase ion might appear easy. After all, electrospray ionization can now routinely generate abundant multiply-charged gas-phase unhydrated quasimolecular ions, $(M+nH)^{n+}$, for most proteins, and Fourier transform ion cyclotron

INTERDISCIPLINARY *cont. on page 6*

From the Director's Desk

Expanding Research & Technology Opportunities Through Collaborations & Partnerships

The onset of each new year prompts the leadership of the NHMFL to set goals and priorities for the new year. Underpinning every discussion is—first and foremost—fulfilling the laboratory's mission, as articulated by the National Science Foundation. This includes topics often discussed in this space: enhancing user operations; completing all magnet development projects tasked by NSF; developing, implementing, and assessing the impact of an in-house research program; and expanding educational and public outreach in science awareness. With regard to the NHMFL In-House Research Program, I am pleased to direct the reader's attention to the column starting on page 4, *From the Chief Scientist's Desk*, which announces the results of this program's very successful initial solicitation.

This year, I expect that we will be focusing even greater emphasis on two programs established in the early years of the laboratory, but which now offer significant opportunities for expansion of the core facility and enhancement of research programs. Specifically, I am speaking about Interagency and Inter-Institutional Collaborations, and the Industrial Affiliates Program.

Collaborations with the private sector and other agencies offer the laboratory critical avenues through which we may broaden the NHMFL funding base. Important conversations are underway with the National Institutes of Health, NSF, the Department of Energy, NASA, the U.S. Air Force, and other institutions. New collaborations with these organizations and others will be crucial in building the magnetic resonance users programs of the laboratory, for further enhancing the facilities at LANL, for driving new high field magnet research and development activities, and for solidifying our already successful educational outreach efforts.

Partnerships with the private sector—through the NHMFL Industrial Affiliates Program and through SBIR/STTRs, CRADAs, and GOALI—are critical to moving cutting-edge science from the laboratory to the marketplace and to advancing the state of magnet research and technology. They are also essential to regional and state economic development.

This newsletter reports two interesting new alliances. One is an innovative private-state collaboration that will drive the development of superconducting magnets for magnetic levitation trains. The other is a collaboration with the Institute for Plasma Research in India to assist with the design and development of superconducting magnets for a new Steady-state Superconducting Tokamak called SST-1. The tokamak concept may provide the first demonstration of generating useful energy by controlled thermonuclear fusion.

These two examples, and the many others described in the *1996 NHMFL Annual Report* currently in production, foreshadow the opportunities at hand. The NHMFL offers users from a broad range of disciplines—including physics, biology, geology, engineering, materials science, chemistry, geochemistry, and structural biology—world-class facilities at the extremes of high magnetic fields and temperature. It is well positioned to drive the technological advances needed for the 21st century.

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

NHMFL/IPR-India Collaboration on Superconducting Magnet Development

Engineers and scientists of the NHMFL Magnet Science and Technology (MS&T) Group have just begun a collaboration with the Institute for Plasma Research in India (IPR-India) to assist with the design and development of superconducting magnets for a new Steady-state Superconducting Tokamak called SST-1. The tokamak concept—originating in the former Soviet Union about forty years ago as a device for creating, containing, and heating a plasma of hydrogen isotopes—remains the leading candidate to provide the first demonstration of generating useful energy by controlled thermonuclear fusion. It is the basis for ITER (the International Thermonuclear Experimental Reactor), a collaborative undertaking of Japan, the European Community, Russia, and the United States, presently nearing the end of its engineering design phase.

Like the NHMFL, IPR-India is a relatively new institute. The SST-1 (illustrated by the conceptual design drawing of Figure 1) represents its second major step pursuant to a broad mandate from the Indian government to stimulate plasma research and development activities within Indian universities and industries. The first step was the design and construction of its presently operating Aditya Tokamak, which is similar in physical size to SST-1 but lacking in steady-state or long-pulse capability because it uses resistive magnets.

In the initial phase of the collaboration with IPR-India, MS&T will provide only consultation on the design of cable-in-conduit superconductors for SST-1 magnets. Negotiations are underway, however, to integrate a broader program of design, development, and testing into ongoing MS&T programs in such a way that the unique NHMFL resources (facilities and staff) are used to the maximum mutual benefit. IPR-India's interest in NHMFL was initially sparked by knowledge of our development of cable-in-conduit conductor technology for the 45 T Hybrid (see *NHMFL Reports*, Fall 1996 issue).

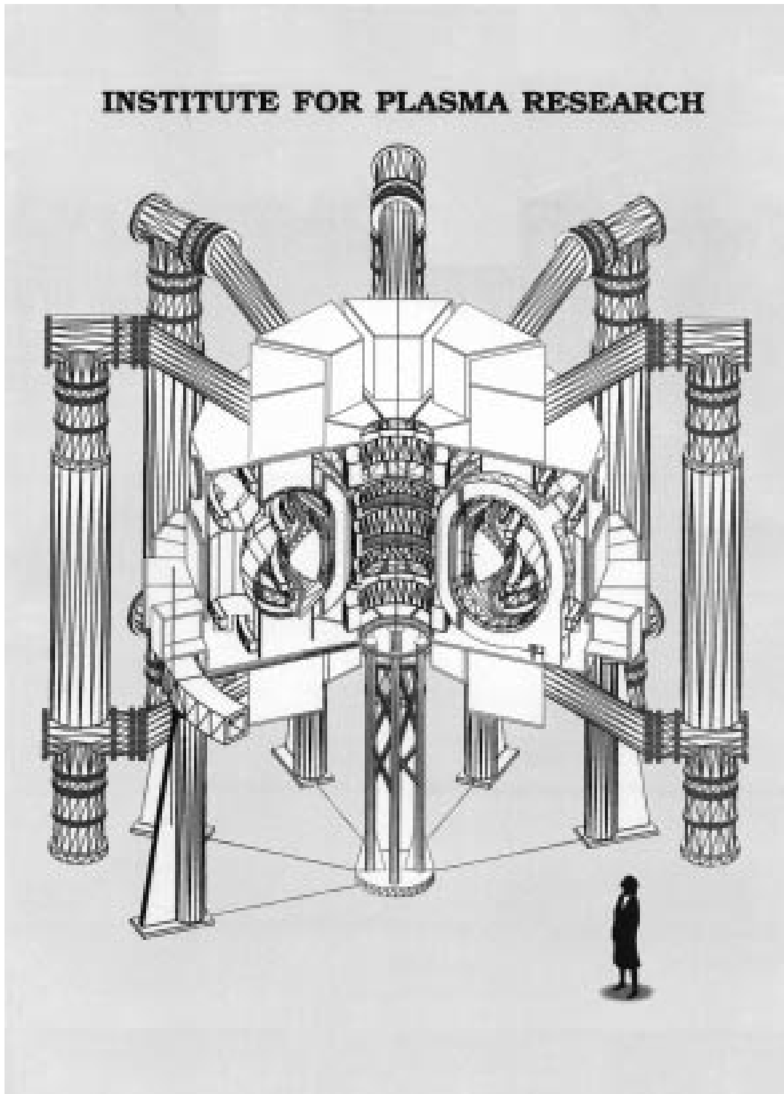


Figure 1. Conceptual drawing of Steady-state Superconducting Tokamak—SST-1.

From the Chief Scientist's Desk

The NHMFL In-House Research Program

The National Science Foundation charged the National High Magnetic Field Laboratory with developing an in-house research program that:

- Utilizes the NHMFL facilities to carry out high quality research at the forefront of science and engineering, and
- Advances the NHMFL facilities and their scientific and technical capabilities.

To this end, the NHMFL developed an in-house research program that not only guides and stimulates magnet and facility development, but additionally provides intellectual leadership for experimental and theoretical research in magnetic materials and phenomena. The NHMFL In-House Research Program (IHRP) seeks to achieve these objectives by funding research projects of normally one to two years in duration in the following categories.

- Small, seeded collaborations between internal and/or external investigators that utilize their complementary expertise.
- Bold but risky efforts that hold significant potential to extend the range and type of experiments.
- Initial seed support for new faculty and research staff, targeted to magnet laboratory enhancements.

The IHRP strongly encourages collaboration across host-institutional boundaries and between internal and external investigators in academia, national laboratories, and industry, as well as interaction between theory and experiment. Projects are also encouraged to drive new or unique research, for example, to be used as seed money for development of initial data that may lead to external funding of a larger program. In accord with NSF policies, the NHMFL cannot fund clinical studies.

Results of the program's first year. A total of 67 proposals were received by the proposal submission deadline of August 16, 1996. The proposals spanned a very significant intellectual breadth, consistent with NHMFL intentions. Their approximate breakdown by research discipline and/or technique is shown in Table 1. Note that several proposals spanned the boundaries between these designations, and thus could be legitimately placed in more than one category. For the magnetic resonance (non-physics) area, the 30 proposals received could be approximately subdivided by technique as: NMR (11), EMR/ENDOR (7), MRI (6), ICR (6).

In order to ensure that funded projects are of the highest quality, each proposal was evaluated against four primary review criteria: research performance competence; intrinsic merit of the research; utility or relevance of the research to the NHMFL mission; and effect on the infrastructure of high magnetic field science and engineering. The review process entailed three steps as follows:

Table 1. Proposals received for the NHMFL In-House Research Program for 1996.

Research Area or Technique (Number of Proposals)	Research Subarea	# of Proposals (per subarea)
Magnetic resonance (30) (non-physics)		
	System & hardware development	11
	Biochemistry	4
	Solid-state chemistry	4
	Biology	4
	Chemistry	4
	Geochemistry	1
	Biomedicine	1
	Hydrodynamics	1
Condensed matter physics (27)		
	Heterostructures /Quantum Hall effect	8
	Oxides	7
	Theory	4
	Heavy Fermions	3
	Quasi-1D systems	2
	General magnetism	2
	Quantum fluids	1
Materials science and engineering (10)		
	Materials science & processing	5
	Applied superconductivity	4
	Biology	1

1. *Initial review by the NHMFL Research Program Committee (RPC).* This committee evaluates research opportunities available to the NHMFL and recommends programs for the use of facilities and resources of the NHMFL. It oversees the IHRP, encourages the formation of collaborative research efforts, establishes worldwide channels for communication, and identifies cutting-edge high magnetic field research programs. The RPC is chaired

Table 2. Approved NHMFL In-House Research Program projects.

Lead PI	NHMFL Institution	Project Title
Brooks, James	FSU (Physics)	*Experimental & Theoretical Aspects of Quasi-3D Quantum Hall Systems*
Brunel, Louis-Claude	FSU (CIMAR)	*Development of a High Frequency Short-Pulse Gyrotron for Pulsed EPR*
Dagotto, Elbio	FSU (Physics)	*Study of Spin Gapped Quasi-1D Compounds Using ESR Techniques & Numerical Simulations*
Dalal, Naresh	FSU (Chemistry)	*High-Resolution Solid-State NMR Techniques & Applications to Materials Science*
Fisk, Zachary	FSU (Physics)	*Doped Hole Physics In Single-Layer Perovskites*
Gibbs, Stephen	FSU (Physics)	*Applications of MR Velocimetry to Flow in Media & Fiber/Composite Manufacturing*
Moulton, William	FSU (Physics)	*NMR Studies Of Superconducting and Magnetic Cuprates & Low Dimensional Electron Systems at High Magnetic Fields*
Douglas, Elliot	UF (Matls Sci.)	*Materials Processing in Magnetic Fields: High-Strength Polymers*
Eyler, John	UF (Chemistry)	*Novel Syntheses & Fourier Transform Mass Spectrometric Analyses of Combinatorial Libraries*
Mareci, Thomas	UF (Biochem)	*High Field, High Frequency RF Coils for NMR Spectroscopy and Microscopy of Small Samples*
Talham, Daniel	UF (Chemistry)	*Comparing Magnetic Langmuir-Blodgett Films to Their Isostructural Solid-State Analogs Using Antiferromagnetic Resonance*
Tanner, David	UF (Physics)	*High-Field Optical Studies of Highly Correlated Metals*
Hammel, P. Chris	LANL (MST-10)	*Very High Magnetic Field NMR Studies of the Cuprate Spin Gap*
Movshovich, Roman	LANL (MST-10)	*Heat Capacity Measurement in the NHMFL 60 Tesla Quasi-Continuous Magnet*
Rickel, Dwight	LANL (DX-6)	*Time-Resolved Photoluminescence Studies of Semiconductor Heterostructures in Ultra-High Magnetic Fields*

by the Director of the IHRP, John Graybeal, and the present membership is: James Brooks, Zachary Fisk, Lev Gor'kov, Alan Marshall, Stephan von Molnár, and Stan Tozer (FSU); John Graybeal, Kevin Ingersent, and Thomas Mareci (UF); Alan Bishop, Chris Hammel, and Joe Thompson (LANL). Proposals deemed to hold the highest merit passed on to the second review step.

2. *Subsequent review by an external peer review panel.* Members of this panel were chosen by the IHRP Director in consultation with the NHMFL Chief Scientist and members of the RPC. When a conflict of interest arose involving the IHRP Director, reviewer selection was performed by the NHMFL Chief Scientist. Reviewer selection was based on the following criteria:

   - Highly distinguished reviewers, leaders in their respective fields, and
   - Absence of conflict of interest or close association with proposal PI or collaborators.

   The identities of the external reviewers were held strictly confidential. The external reviewers evaluated the proposals based on the goals and criteria stated in the solicitation.

3. *Final determination of project funding by the NHMFL Chief Scientist.* The NHMFL Chief Scientist, J. Robert Schrieffer, made the final funding decisions based upon the internal and external review reports.

Of the 67 proposals submitted, the committee recommended that 38 proposals be sent out for external review. As with the internal review, two external reviews were sought for each proposal. In all, 64 reviews were received from 27 external reviewers, an average of 1.7 reviews per proposal and 2.4 reports per reviewer. On the basis of this external review, 15 projects were ultimately approved for funding.

We learned a great deal from the first round and are improving the process: increased number of external reviewers, better balance across the disciplines, more involvement with external collaborators, etc. The second IHRP proposal solicitation was released in late January 1997, with a proposal deadline of April 7, 1997.

John Graybeal, Director of the NHMFL In-House Research Program

J. Robert Schrieffer, NHMFL Chief Scientist

INTERDISCIPLINARY *cont. from page 1*

resonance (FT-ICR) mass spectrometry can determine the ion mass to ppm accuracy at typical electrosprayed protein multiply-charged ion mass-to-charge ratios, $500 \leq m/z \leq 2{,}000$. Surprisingly, however, the monoisotopic mass (see below) of a protein inferred from the mass(es) of its corresponding ions may still be wrong by up to several Da!

For organic molecules of less than ~1,000 Da, determination of molecular weight from the singly-charged molecular (M^+) or quasi-molecular (e.g., $(M+H)^+$) ion is relatively simple. Why then is it so much more difficult to determine the mass of a biological macromolecule? The problem is apparent from Figure 1 (top half). The natural abundance of ^{13}C is 1.066-1.106% relative to ^{12}C as 100%. For a molecule containing *n* carbons, however, the isotopic distribution is a binomial expansion $(0.9889 + 0.0111)^n$, and it is ~*n*% as likely that a given molecule will contain one ^{13}C as that all of the carbons will be ^{12}C. For a protein with hundreds of carbons, hydrogens, nitrogens, and oxygens, the combined binomial distributions for its constituent elements produce a wide spread in natural-abundance isotopic relative abundances (Figure 1, top). Only one resolved peak has a unique isotopic composition: namely, the "monoisotopic" species, $^{12}C_{527}{}^{1}H_{830}{}^{14}N_{146}{}^{16}O_{155}{}^{32}S_3{}^+$ in this case. Every other peak represents an unresolved superposition of several isotopic variants: e.g., the next highest nominal mass peak includes $^{13}C^{12}C_{526}{}^{1}H_{830}{}^{14}N_{146}{}^{16}O_{155}{}^{32}S_3$ and $^{12}C_{527}{}^{1}H_{830}{}^{15}N^{14}N_{145}{}^{16}O_{155}{}^{32}S_3$ (as well as other combinations). Thus, accurate unambiguous determination of protein molecular weight to within 1 Da reduces to correct identification of the monoisotopic mass. For very small proteins (≤5-10 kDa), the "monoisotopic" species, $^{12}C_v{}^{1}H_w{}^{14}N_x{}^{16}O_y{}^{32}S_z$ may be detected directly. For proteins larger than ~ 2 kDa, however, the most abundant peak is shifted upward in mass from the monoisotopic peak by ~1 Da for every 1.5 kDa of molecular weight; worse, at ≥15 kDa, the "monoisotopic" ion abundance is below detectability (<< 1%).

Fortunately, because of the widespread production of proteins (in milligram quantity) isotopically *enriched* in ^{13}C and ^{15}N for various multidimensional heteronuclear FT-NMR experiments, it is equally easy to produce any of the same proteins isotopically *depleted* in ^{13}C and ^{15}N by substitution of appropriate ^{13}C– and ^{15}N–depleted nutrients. In work just published,[1] CIMAR research groups directed by Alan G. Marshall (FT-ICR MS) and Timothy M. Logan (FT-NMR) combined forces to simplify the mass spectrum of FK506-binding protein (FKBP, 107 amino acids, ~11,800 Da molecular weight) very substantially by isolating the protein from *E. coli* grown on 99.95% glucose-$^{12}C_6$ and 99.99% ammonium sulfate-$^{14}N_2$.

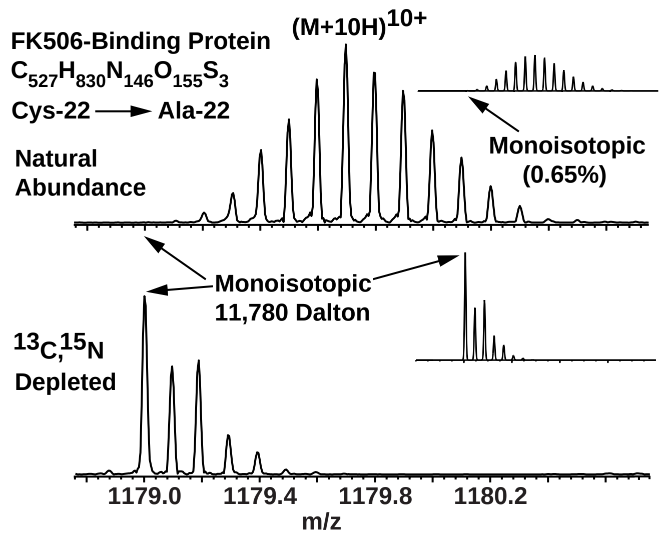


Figure 1.

Figure 1 (bottom) shows the dramatic improvement in mass spectral quality afforded by double isotopic-depletion of FKBP. (Experimental mass spectra are at left, and simulated isotopic distributions at right in the Figure.) First, the monoisotopic species, present at only 0.65% at natural abundance, becomes the largest peak in the mass spectrum of the $^{13}C,^{15}N$ doubly-depleted protein! The molecular weight of the neutral protein is thus determined immediately and unambiguously as 11,780.01 (compared to 11,780.07 computed from the amino acid sequence). Moreover, for a similarly doubly-depleted protein of 80 kDa molecular weight, the monoisotopic peak would still be 1% abundant, and thus easily identifiable. Thus, double-depletion extends the upper molecular weight limit for proteins by about an order of magnitude!

This result offers an excellent example of the synergism at play between different CIMAR research groups. Dr. Logan's group was already making ^{13}C, ^{15}N-*enriched* protein for NMR work, and was therefore perfectly poised to produce the ^{13}C, ^{15}N-*depleted* material for mass spectrometry. Dr. Marshall's group was ready with the world's highest-performance electrospray FT-ICR mass spectrometer to take maximal advantage of the new protein. Many more future collaborations between NMR and ICR groups are anticipated: in fact, Dr. Ming-Daw Tsai's group at Ohio State University has already prepared ^{13}C,^{15}N doubly-depleted p16 tumor suppressor protein for mass analysis.

[1] Marshall, A.G.; Senko, M.W.; Li, W.; Li, M.; Dillon, S.; Guan, S.; Logan, T.M., "Protein Molecular Mass to 1 Da by ^{13}C, ^{15}N Double-Depletion and FT-ICR Mass Spectrometry," *J. Am. Chem. Soc.* **1997**, *119*, 433-434.

NHMFL Joins Maglev Project

In January, 1997, the NHMFL officially entered into a collaboration with the Maglev 2000 of Florida Corporation of Stuart, Florida, to push forward the development of superconducting magnets for magnetic levitation trains. Maglev 2000 was issued a Joint Project Agreement from the Florida Department of Transportation to research, develop, and demonstrate a high technology magnetic levitation transportation system in Brevard County, on the east coast of Florida.

The NHMFL brings to the partnership expertise in high temperature superconducting materials and magnets. These materials are particularly promising for maglev applications due to the flexibility of their operating temperature and thus their ability to operate without liquid cryogens. High temperature superconducting materials are also important because of their ability to operate in the presence of a substantial heat load, which is intrinsic to some maglev designs.

Education at the NHMFL: A Year-Round, Every-Year Commitment

The NHMFL promotes public awareness and interest in science and mathematics by using its unique strengths—world-class research facilities and human resources (i.e. leading scientists and engineers)—to develop educational programs for students at all academic levels. Some of these initiatives have been featured before in *NHMFL Reports*, most recently in the Fall 1996 issue with the article entitled, "Recruitment Begins for 1997 Minority/Women Research Internship Program.

As the laboratory matures (recalling that the facility was only dedicated in October, 1994), educational programs started in the "early years" just seem to be getting better and better. For example:

- The *5th Annual Minority/Women Research Internship Program* mentioned above is experiencing an *outstanding* recruitment season. Over sixty applicants have applied for the summer program; we expect to place twenty-one students at the three NHMFL sites. Other interesting statistics:
    - Twenty-one states and the Virgin Islands are represented in the applicant pool. Understandably, Florida leads the list, but California is following closely.
    - Women applicants are outnumbering male applicants two to one.
    - Forty-six undergraduate institutions are represented.

- For the third consecutive year, gifted middle school students presented the results of their semester-long research to a packed audience that included their fourteen NHMFL mentors, the laboratory's Chief Scientist, J. Robert Schieffer, parents, and local media. The students were part of the NHMFL's education partnership with the Leon County (Florida) schools that offers students opportunities to explore the real world of science, mathematics, and technology under the

***Education** cont. on page 8*

Education *cont. from page 7*

Facilities such as the NHMFL are national treasures because of their world-class instrumentation and leading scientists and engineers. It is critically important to expose and challenge young minds to the research opportunities that exist in their backyards. We hope that this experience at the laboratory will further their interests in science and technology so that someday they will return to conduct their own research.

— Jack Crow, NHMFL Director

mentorship of in-house scientists and engineers. Twenty-four students participated in this year's *Research Mentorship Program*, which concluded with the poster presentation session on January 14, 1997.

The students' research encompassed a wide range of activities including architecturally-designed houses using computer-aided design (CAD) programs; a project to determine the lowest temperature limits of a standard helium-3 cryostat; and the preparation, fabrication, and testing of superconducting tape that may be the basis for major breakthrough opportunities for winding high temperature superconductors in the future.

- For the second consecutive year, the NHMFL will participate in NSF's National Chautauqua Short Course Program by hosting a course for undergraduate college science teachers entitled *Magnetic Fields in Science and Technology*. The course will be held at the NHMFL in Tallahassee from June 12 through June 14. Participants will have a choice between three subtopics: Physics & Materials Science, Environmental Science & Geochemistry, and Biology & Chemistry.

The NHMFL Chautauqua course will provide an opportunity for college teachers to learn about the advances in science and technology promoted through the laboratory; to interact and discuss research and teaching with top scientists and scholars; and to examine and develop strategies that incorporate these new understandings and advances into their curriculum.

For more information, contact Sam Spiegel at (904) 644-5818 or *spiegel@magnet.fsu.edu*.

75th Anniversary Symposium for E. Raymond Andrew

The University of Florida and the NHMFL honored E. Raymond Andrew on the occasion of his 75th anniversary in recognition of his many far-reaching contributions to magnetic resonance, solid state physics, and magnetic resonance imaging. The international celebration was held in Gainesville, Florida, on January 5, 1997.

Dr. Andrew is a graduate of the Cavendish Laboratories, Cambridge, B.A. (with a First in Physics) 1942; M.A. 1946; Ph.D. 1948; D.Sc. 1964. His first work on NMR came shortly after the discovery of NMR at Harvard where he was a Commonwealth Fellow from 1948-49, at the same time Charles Slichter was carrying out his graduate studies. He returned to Scotland as lecturer at St. Andrews, where he carried out seminal NMR studies of solids, of which many experiments are described in texts as classics today: Critical field measurements in superconducting foils, molecular motion in solid hydrocarbons, and self-diffusion in molecular solids.

He moved to University of Wales (Bangor) in 1954 where he became Professor and Head of the Department of Physics until 1964. It was in this period that he made one of the most significant discoveries: the narrowing of NMR lines by magic angle spinning, which has been the foundation of modern high resolution NMR studies in the solid state. In 1964 he was appointed Lancashire Professor and Head of Physics at Nottingham where he became Dean in 1975. At Nottingham, Dr. Andrew continued his work on using rapid rotation of samples for high resolution studies, and went on to make another major contribution to the field of magnetic resonance with his pioneering studies on magnetic resonance imaging. It is thanks to these efforts with other colleagues that the tremendous benefit to mankind of the use of MRI in medicine has been possible. He was elected Fellow of the Royal Society in 1984.

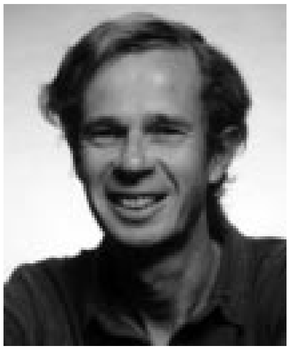
Dr. Andrew left the United Kingdom for the United States in 1983 to join the NMR physics group in Florida where he had enjoyed long friendships with Tom Scott and Jim Brookeman. It seems that every time Dr. Andrew makes a move, he adds another quantum to the advances in NMR: Indeed, he was a major player in the proposal to establish the National High Magnetic Field Laboratory in Florida in 1990. Dr. Andrew continues a very active program at UF with the recent commissioning of the 3 T whole body imaging capability at the Veteran's Hospital Medical Center in Gainesville.

The memorable celebration was sponsored by UF, the NHMFL, Elsevier Science B.V; Nolarac, Corp.; Magnex Scientific; Oxford Instruments; Varian Associates; and John Wiley & Sons, Ltd. Invited speakers included Nicolaas Bloembergen, Paul Bottomley, James Carolan, Stan Clough, Erwin Hahn, Laurie Hall, Jacek Hannel, Sir Peter Mansfield, Vincent McBrierty, Alex Pines, Robert Pound, Hans Schneider-Muntau, Charles Slichter, and Sir Martin Wood.

Florida has enjoyed the benefits of Raymond's work since 1983 when he joined an active group in magnetic resonance at the University of Florida where he was instrumental in establishing the National High Magnetic Field Laboratory in Florida in 1990—the world's leading facility of its kind today.

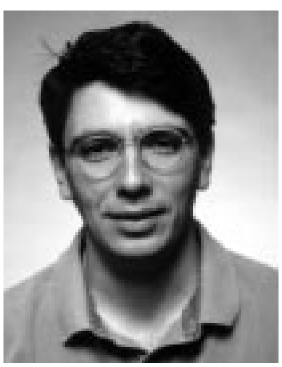
From a Resolution approved by the Florida Cabinet and signed by Governor Lawton Chiles in appreciation for Dr. Andrew's exceptional contributions to science, medicine, and the State of Florida.

People Profiles



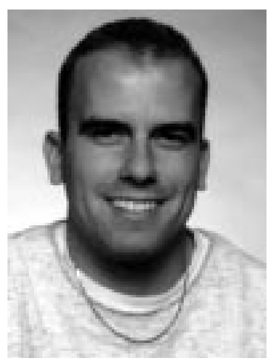
Geoffrey Bodenhausen, a full professor in the FSU Chemistry Department and director of the NMR program at NHMFL from 1994-1996 until his recent move to Ecole Normale Superieure, was elected to the rank of Fellow of the American Physical Society, in the Division of Chemical Physics. This designation is limited to no more than one half of one per cent of the membership each year. His fellowship citation reads, "For his numerous contributions toward making magnetic resonance one of the most sophisticated and versatile methods available for gaining insight into structure and dynamics of molecules in condensed and gas phase."

Dr. Bodenhausen, along with co-inventor ***Riqiang Fu*** were issued a U.S. Patent for a "Method and Apparatus for Broadband Decoupling in NMR with Chirp Pulses" in December 1996.



Vladimir Dobrosavljevic, an FSU Assistant Professor of Physics and a theorist in the Condensed Matter/Theory Group of the NHMFL, has been selected as an Alfred P. Sloan Research Fellow for 1997. This award carries a $35,000 fellowship over two years. Dr. Dobrosavljevic joined the faculty at FSU in August, 1995, prior to which he carried out research at Rutgers University, the University of Maryland, and Brown University. His principal research interests include theoretical studies of disordered electronic systems, the physics of strong correlations, non-Fermi liquid behavior in metals, and the metal-insulator transition.

Alan Dorsey joins the NHMFL faculty at UF as an Associate Professor in theoretical condensed matter physics. He was attracted to the University of Florida because "there were a large number of vigorous research efforts in the physics department and the University; and in my own specialty, this included the NHMFL." His research, which has been funded by the NSF since 1989, focuses on theoretical aspects of magnetic flux dynamics in superconductors. He is particularly interested in the dynamics of quantized flux vortices in superconductors; in his words: "How do vortex pinning and thermal fluctuations conspire to produce some of the unusual transport properties of the high temperature superconductors?" He is also investigating the nonequilibrium aspects of superconductivity. For example, he developed theoretical models that indicate that when a magnetic field is expelled from a superconductor it should form complicated, fingered patterns, which may explain the propensity of some superconductors to trap magnetic flux. He joins UF and the NHMFL from the University of Virginia, where he received the Department of Physics' Outstanding Teaching Award in 1996. For his research accomplishments, he was awarded an Alfred P. Sloan Foundation Research Fellowship from 1991 to 1994.



Forest M. White, a Ph.D. candidate in his final year of research in the ICR group headed by Alan Marshall, has received the FSU Chemistry Department 1997 Graduate Student Award, which is based on research achievement. Forest describes his research as "focusing on the analysis of growth factors from a glioma cell line. Growth factors are small proteins that play a critical role in the growth and proliferation of cancerous tumors, especially gliomas (brain tumors). We are trying to develop an assay for these growth factors that will have comparable limits of detection to current techniques, but will yield more structural information." Following graduation, he plans take a postdoctoral position and continue in academia.

Workshop & Conference Activity

First North American FT-ICR MS Conference

March 13-15, 1997
NHMFL-Center for Interdisciplinary Magnetic Resonance
Tallahassee, Florida

In response to the continued rapid growth and interest in mass spectrometry and Fourier Transform–Ion Cyclotron Resonance mass spectrometry, the first North American FT-ICR Conference will be held at the NHMFL in Tallahassee. Dr. Alan Marshall, the director of the Ion Cyclotron Resonance Program at the laboratory is chairing the conference. Over 100 attendees, representing industry, academia, and national laboratories, are expected for two days of invited oral and poster presentations, as well as contributed posters.

The topic areas will include a broad range of FT-ICR MS topics, including: instrumentation/technique development, biological applications, polymers, mixture analysis, ionization methods, ion-molecule chemistry, ion spectroscopy, and others. A tour of the NHMFL and the local FT-ICR facilities will be provided.

29th Annual Southeastern Magnetic Resonance Conference

October 30 - November 1, 1997
NHMFL and University of Florida
Gainesville, Florida

The 29th Annual Southeastern Magnetic Resonance Conference (SEMRC) will bring together research groups predominantly from the southeastern states of the United States for exchange and discussion of recent advances in techniques and applications of magnetic resonance. The meeting will be well balanced between EMR and NMR and should contain a good contribution from the ICR community, as well. Several internationally renowned scientists are being invited for keynote lectures.

Final conference arrangements are being made. The tentative deadline for registration and submissions is September 12. For more information, contact Dr. Alex Angerhofer (phone, 352-392-9489 or *alex@chem.ufl.edu*) or Dr. Russ Bowers (phone, 352-846-0839 or *russ@physical27.chem.ufl.edu*).

8th U.S.-Japan Workshop on High-T_c Superconductors

December 8-10, 1997
NHMFL
Tallahassee, Florida

Plans are being finalized for this conference, which will be held at the NHMFL in Tallahassee. For updated information, contact conference co-chair, Dr. Justin Schwartz at *schwartz@magnet.fsu.edu*.

Pulse Magnet Development Activities Continue at Fast Pace

The NHMFL's Pulse Magnet Group has just finished successfully testing a series of magnets at the laboratory's Pulsed Field Facility at Los Alamos. The primary aim was to commission a series of four coils to be used as focusing magnets for a $20 million high intensity X-ray radiography experiment at Sandia National Laboratory.

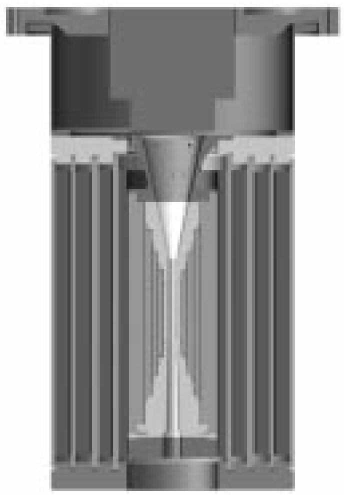


Figure 1. Schematic of 60 T quasi-continuous magnet at LANL.

The on-time delivery to Sandia was the latest project in a series of pulse coils commissioned by external organizations, including the Australian National Pulse Magnet Laboratory, University of Wisconsin, and Harvard University.

A test to failure was performed on one of the two designs of the 60 T, 15 mm bore magnets in use at the pulse facility. The test coil was first tested to 60 T and then further to 65 T. No ill effects were observed with numerous pulses at the higher field. With these findings it seems likely that the user field for this magnet will be extended to at least 63 T, with the possibility of 65 T in some circumstances. Within the next few months, the group plans to test an alternative design for this coil with the aim of extending the user field even further. In addition, they will be testing, in a similar manner, the current 50 T, 24 mm bore magnet to 55 T.

For more information on the pulse magnet development activities of the laboratory, see *NHMFL Reports*, Fall 1996, or contact Paul Pernambuco-Wise, (904) 644-0854, *wise@magnet.fsu.edu*.

High Sensitivity NMR and MRI Without High B/T

Part One: Applications of Optical Pumping in NMR Spectroscopy

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The second part of this two-part article, *Applications of Optical Pumping to Magnetic Resonance Imaging*, will appear in the Spring Issue of *NHMFL Reports*, to be published in May 1997.

Improved sensitivity in NMR and MRI experiments has long been a primary driver for the development of magnets capable of generating the highest possible fields. At the forefront of the current technology are NHMFL's superconducting and resistive magnet development programs. Higher magnetic fields can increase the sensitivity in at least two ways; firstly, the induced EMF in the receiver coil is increased in proportion to the Larmor frequency according to Faraday's law of induction, and secondly, the magnitude of the nuclear magnetism to be detected is increased as a result of Boltzmann's law. The NMR signal is directly proportional to the polarization P of the nuclear spin states:

$$P = \tanh\frac{-\gamma\hbar B}{2kT} \approx \frac{-\gamma\hbar B}{2kT} \quad (1)$$

where γ is the nuclear gyromagnetic ratio, B is applied to field, T is temperature.

The impetus for constructing high field magnets is clearly evident from Eq. 1. By increasing B/T, higher sensitivity should result. In the high temperature regime, the signal is approximately proportional to B/T. Unfortunately, other factors can either reduce or negate the theoretical sensitivity improvement that would otherwise result from higher polarization and better RF reception. Relaxation times can become undesirably long at high field and low temperatures, reducing the ability to signal average. Furthermore, if liquid state samples or living specimens are to be studied, then the freezing point or biological factors will set a lower limit on temperature. Therefore, it is imperative that new techniques be developed for enhancing the initial nuclear magnetization or for increasing the efficiency of the detection mechanism under a variety of conditions. Polarization enhancement schemes that do not depend on high B/T should be pursued in parallel with the superconducting and resistive magnet development programs.

The primary objective of the Bowers' research program at UF is to develop enhanced sensitivity NMR methodologies and to demonstrate their utility with specific applications in chemistry, materials science, and condensed matter physics. The enhancement schemes are based on either the signal gain obtained through increasing the nuclear spin polarization, or via new detection mechanisms that provide *quantum amplification*.

One of the strategies being pursued involves alkali-atom rare gas optical pumping. Modern optical pumping methods[19,20] can prepare liter-atm quantities of these gases at polarization levels of 30-40%. The phenomenon was first observed by Bouchiat, Carver, and Varnum in 1960[6] and was subsequently studied in great detail by, most notably, C. Cohen-Tannoudji (ENS, Paris) and W. Happer (Princeton).[1,3] More recently, the research groups of A. Pines (U.C. Berkeley), M. Mehring (U. Stuttgart), and G. Cates[4,5] have exploited the enhanced nuclear polarization in applications ranging from physics, chemistry, biology, and materials science. Presently, there is rapidly growing interest in this method in the NMR and *in-vivo* MRI communities. New optical pumping research programs are being initiated at the highest rate ever. Indeed, the excellent prospects for application of optically pumped MRI to medical diagnosis in lung, heart, brain and other organs has even led to commercialization of the technology. This article will review some of the successful methodologies for imaging and spectroscopy of optically pumped ^{129}Xe and ^{3}He.

Optical Pumping Basics. The physics of optical pumping can be thought of as a two step process. Firstly, excitation with circularly polarized light of the D1 line of atomic rubidium (sodium, cesium of potassium can also be used) vapor leads to polarization of the hyperfine levels. In the second step, the electronic spin order of the rubidium is transferred to the rare gas nucleus during spin-exchange collisions. In the case of xenon, short lived Rb-Xe van der-Waals complexes form, increasing the efficiency of the spin exchange. In the two spin-1/2 rare gas types, ^{129}Xe and ^{3}He, the cross relaxation achieved through spin-exchange dominates over other relaxation processes. The T_1 relaxation of the rare gas atoms by other mechanisms is relatively long in the gas phase, allowing the polarization to build up, under favorable conditions, to values of several tens of percent. Thermodynamic speaking, the spin temperature of the xenon atoms, which is essentially infinite before optical pumping begins, approaches the very low spin temperature of the rubidium atoms. In comparison with the typical parts-per-million polarization obtained even at the highest available fields, where B = 33 T, the nuclear polarization of ^{129}Xe at T = 300 K is

$$P - 2.8 \times 10^{-5} \qquad (2)$$

It is evident that optical polarization can yield an improvement in signal by a factor of > 10^4 compared to thermal polarization techniques. The lower limit on the number of ^{129}Xe nuclei that can be detected is commensurately reduced. This dramatic sensitivity increase opens the door to a plethora of new NMR and MRI applications, a few of which are mentioned here.

An Exemplary Application of Xenon Optical Pumping. Once the ^{129}Xe has been polarized, it may be easily separated from the rubidium and then transported to a high field magnet for NMR spectroscopy. Polymers, zeolites, and nanocrystalline materials have been studied in this way. In one of the more interesting chemical applications, the feasibility of forming an optically pumped xenon hydrate clathrate was demonstrated.[8] As shown in Fig. 1, the formation of the type-I and type-II xenon-hydrate clathrate cages can be monitored in time, allowing kinetic study of the formation. This shows the excellent potential for the use of optically pumped xenon for characterization of microcages, vacancies and defects in other non-stoichiometric clathrate and non-clathrate solids containing spin polarized ^{129}Xe atoms. Using the dramatic signal enhancement afforded by optical pumping, it should be possible to monitor in real time slight structural changes in the cages of known clathrates as a function of temperature and annealing time. The discovery of new clathrates systems might also be facilitated.

Since interpretation of ^{129}Xe chemical shifts and line shapes can sometimes be difficult if not impossible, polarization transfer from the optically pumped ^{129}Xe to the spins comprising the cage or internal surface could serve to selectively intensify the signals from these sites, providing the additional information needed to make spectral assignments. Several ways of accomplishing this are described below.

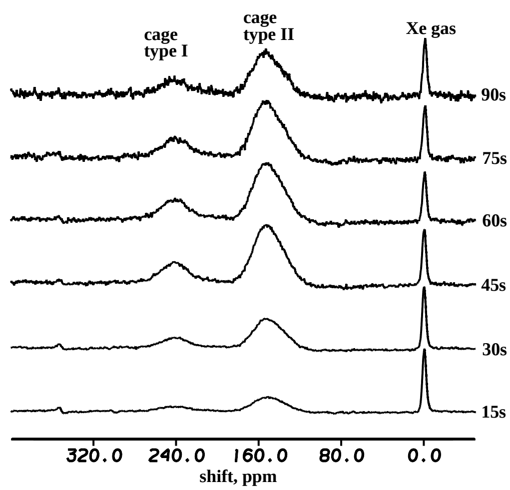

Figure 1. By taking advantage of the dramatic signal enhancement afforded by optical pumping, the formation of laser polarized xenon hydrate clathrate can be monitored *in situ* and in real time by ^{129}Xe NMR. (adapted from Ref. 8)

Optically Pumped Nuclear Polarization Transfer Methods. The transfer of polarization from optically ^{129}Xe or ^{3}He to the spins on a molecule or surface of interest has great potential to develop into a general method for NMR sensitivity enhancement. Certain conditions need to be obtained, however, in order to extract an appreciable fraction of the rare gas polarization. The rare gas atom must be brought into dipolar contact with the spin system of interest, and it must remain immobilized during the cross-polarization mixing time. To date, three distinct mechanisms for polarization transfer have been reported in the literature. These are briefly described as follows:

Thermal mixing:[9] the timing sequence for this scheme is shown in Fig. 2. The molecular species of interest is condensed into a laser polarized solid matrix. Guest-host polarization transfer can be facilitated by a brief exposure to low field. At fields low enough to achieve energy overlap of the transitions, heteronuclear polarization transfer can efficiently occur by thermal mixing.[10] Thermal mixing with a molecular species was first demonstrated using $^{13}CO_2$ trapped in the optically pumped xenon matrix. The enhanced ^{13}C spectrum shown in Fig. 3 exhibits the typical chemical shift powder pattern, and the overall phase is determined by the helicity of the circularly polarized pumping light. The enhancement factor of ≈ 200 is the largest factor to be obtained in an optically pumped xenon cross-polarization experiment.

Hartmann-Hahn Cross Polarization:[11] polarization transfer between physisorbed ^{129}Xe spins and ^{1}H nuclei on a surface at low temperature was first demonstrated by the Pines group.[12] Optically pumped xenon gas was physisorbed onto a microporous polymer, polytriarylcarbinol, followed by $^{129}Xe \rightarrow {}^{1}H$ cross polarization.

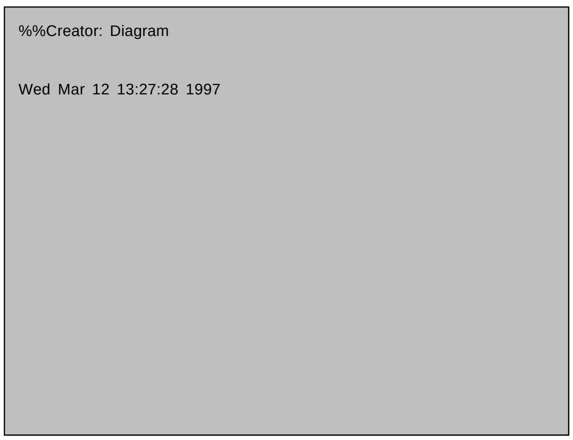

Figure 2. Timing sequence for thermal mixing with a laser polarized host matrix. After optical pumping, the xenon is combined with CO_2 gas and condensed into a precooled NMR tube. Following polarization transfer during τ_{mix}, the sample is rapidly transported to high magnetic field for detection of the enhanced ^{13}C signal. (adapted from Ref. 10)

The Nuclear Overhauser Effect:[13] the enormous initial polarization of the optically pumped ^{129}Xe made it possible to observe a heteronuclear, intermolecular nuclear Overhauser enhancement on the slowly relaxing protons of d5-benzene in d6-benzene solution.[14] The flip-flop transitions in the heteronuclear case do not conserve energy in the isolated spin system and therefore occur with a slow rate. Though it is remarkable that any cross polarization can be observed via such an inefficient process, an enhancement factor of only 1.5 was reported.[14]

In summary, cross-polarization has been achieved in several specific cases by a variety of mechanisms, but none of these techniques has fully capitalized on the potential for dramatic 10,000 fold signal enhancements. In the Bowers lab, new methodologies are being developed in specific systems to increase the efficiency of polarization transfer from optically pumped ^{129}Xe, either by driven radio frequency double resonance, or by the natural tendency for spin temperature equilibration as a result of cross relaxation.

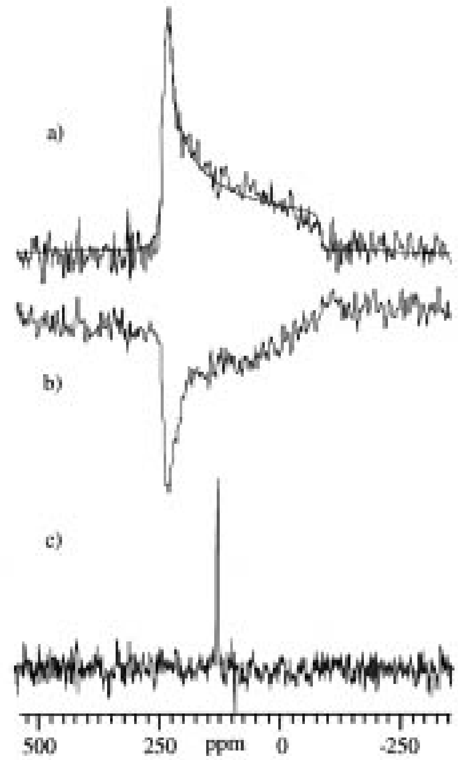

Figure 3. (a) The enhanced ^{13}C spectrum of CO_2 obtained after thermal mixing with xenon which has been optically pumped with σ- light. The signal was obtained in a single scan. (b) The spectrum obtained under the same conditions as in (a) but with σ+ optical pumping. (c) The spectrum of pure $^{13}CO_2$ gas. The optical pumping signal enhancement factor is ≈ 200. (adapted from Ref. 10)

This concludes our review of the spectroscopic applications of optically pumped xenon and helium. In the next issue of *NHMFL Reports*, the focus will be on the potential impact of this technique for magnetic resonance imaging.

References:

1 N.D. Bhaskar, *et al.*, *Phys. Rev. Lett.* **49**, 25 (1982).
2 W. Happer, *et al.*, *Phys. Rev. A* **29**, 3092 (1984).
3 X. Zeng, *et al.*, *Phys. Rev. A* **31**, 260 (1985).
4 G.D. Cates, *et al.*, *Phys. Rev. A* **45**, 4631 (1992).
5 M. Gatzke, *et al.*, *Phys. Rev. Lett.* **70**, 690 (1993).
6 M. A. Bouchiat, *et al.*, *Phys. Rev. Lett.* **5**, 373 (1960).
7 B. Black, personal communication.
8 T. Pietrass, *et al.*, *Abstract #242, 35th Experimental Nuclear Magnetic Resonance Conference*, Pacific Grove, CA (1994).
9 M. Goldman, Spin Temperature and Nuclear Magnetic Resonance in Solids, Oxford (1970).
10 C.R. Bowers, *et al.*, *Chem. Phys. Lett*. **205**, 168 (1993).
11 S.R. Hartmann and E.L. Hahn, *Phys. Rev*. **128**, 2042 (1962).
12 H.W. Long, *et al.*, *J. Am. Chem. Soc.* **115**, 8491 (1993).
13 J.H. Noggle and R.E. Schirmer, The Nuclear Overhauser Effect, Chemical Applications, Academic Press, New York (1971).
14 G. Navon, *et al., Science* 271, 1846 (1996).
15 B. Driehuys, *Pt. Photonics News* **6**, 38 (1995).
16 J.P. Mugler, *et al., Magn. Reson. Med.*, preprint.
17 A. Haase, *et al., J. Mag. Res*. **67**, 258 (1986).
18 B.A. Inglis, *et al.*, unpublished.
19 B. Driehuys, *et al., Phys. Rev. Lett*.. **74**, 4943, (1995).
20 N.R. Newbury, *et al., Phys. Rev. Lett*. **67**, 3219 (1991).
21 W.J. Cummings, *et al., Phys. Rev. A* **51**, 4842 (1995).

LANL cont. from page 1

a load pulse, resulting in a final speed of about 1260 rpm. The converters are designed to function over this speed range, i.e., from 60 Hz to 42 Hz. Each 12-pulse power supply consists of two 43 MVA (rated pulsed power) step-down transformers, one in a delta-delta and the other in a delta-wye configuration. The transformers, each with a steady-state rating of 4.3 MVA, are of the cast coil type, ideal for indoor installation. This coil type with proper coil bracing is quite well suited for repetitive pulsed operation at 43 MVA. The secondary no-load voltage of each transformer is 3.1 kV. The two 6-pulse rectifiers of the converters are connected in parallel, without an interphase reactor. The "high" impedance of each transformer (30% based on rated pulsed power) allows for the direct parallel connection without excessive circulating currents. Each leg of a 6-pulse bridge has two 94 mm, 5200 V, 3000 A thyristors connected in series, with no parallel thyristors being used. The thyristors are triggered via a fiber optic system with the power for the trigger pulses derived from the forward blocking voltage of each semiconductor device.

Control and Protection Philosophy

Each group of converters is controlled by a digital programmable high speed controller.[3] These controllers, besides performing regular control and protection tasks, control the current of each coil group to follow the predetermined current pattern (up to 200 points, i.e. 199 linear segments), which is downloaded prior to each pulse. Other control parameters needed to run the pattern with the required speed and accuracy are also downloaded.

Protection is given major attention because the magnet will contain 90 MJ of energy at full field. The processor-based protection system is backed by a hard wired protection system. Considerable effort was put into making the power supplies capable of surviving the most severe faults in the system. If one magnet section shorts across its terminals, both bridges of the converter will be immediately switched into the free-wheeling mode. As a back-up and to minimize heating of the free-wheeling thyristors, a fast acting closing switch was installed in parallel with each coil or power supply group. This crowbar switch closes in 12 to 14 ms. The voltage drop across the switch is considerably smaller than the forward voltage drop across the free-wheeling thyristors and the connecting cables, and the free-wheeling current quickly transfers out of the rectifiers into the switch. Should the crowbar switch fail, the thyristors of the free-wheeling path (internal crowbar) can assume the full crowbar current of 25 kA with a decay time constant of 3 s at the end of a full 20 kA / 2 s pulse without damage. Low impedance faults on the 24 kV bus or on the converter transformer primary side are interrupted by a fast, current limiting, explosively triggered fuse system installed at the generator terminals. Higher impedance primary side faults and converter side short circuits, where the short circuit current is limited by the transformer impedance, are interrupted by a 72 kV, 40 kA, 5 cycle, SF-6 breaker. Any breaker opening command is accompanied by a free-wheel command to the converters and a close command to the crowbar switch to minimize overvoltages.

Power Supply Testing

One transformer was subjected to six full voltage, sudden short circuit tests (300 ms) and passed with no signs of deformation or damage, proving the soundness of the final design.

At Los Alamos each converter was tested initially with a 13.4 kV power feed and currents up to 1300 A, to check its basic functionality. The circulating current was found to agree well with simulations. Then each converter was connected to the generator terminals and energized with up to 21 kV at 50 to 60 Hz. A special load, consisting of a variable, 160 MJ resistor bank and high current coils, was assembled to provide the correct resistance to test each converter at rated conditions (140 mΩ cold and about 162 mΩ hot, 2.2 mH). At 20 kA a maximum voltage of about 3.25 kV could be reached, satisfying design specifications.

References:

1. J. B. Schillig, *et al*., "Design of a 400 MW Power Supply for a 60 T Pulsed Magnet," IEEE, Transactions on Magnetics, Vol. 30, No. 4, July 1994.
2. H.J. Boenig, *et al*., "Design, Installation and Commissioning of the Los Alamos National Laboratory Pulsed Power Generator," IEEE, Transactions on Energy Conversion, Vol. 7, No. 2, June 1992.
3. ABB Drives, P. K. Steimer, *et al*., "PSR - the world's fastest programmable controller with function block language," ABB CH-U 93003 E.

This report is based on a paper, "Design and Testing of a 320 MW Pulsed Power Supply," by J.B. Schillig, H.J. Boenig, J.A. Ferner, F. Bogdan, M. Eichler, G. Kasper, R.P. Marek, and J.R. Martin, to be published in IEEE / IAS.

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