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ON THE COVER

Part of the 913-detector array of the new Disk Chopper Spectrometer (DCS) that will be available to users in 2000. To view the detectors as found in the instrument, the picture should be rotated 90°. Information about the DCS and other instruments at NCNR is available at http://www.ncnr.nist.gov/.

Photo: Mark Helfer

NCNR 1999

NIST CENTER FOR NEUTRON RESEARCH ACCOMPLISHMENTS AND OPPORTUNITIES

NIST Special Publication 944

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January 2000

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National Institute of Standards and Technology Special Publication 944 Natl. Inst. Stand. Technol. Spec. Publ. 944, 80 pages (January 2000) CODEN: NSPUE2

U.S. GOVERNMENT PRINTING OFFICE - WASHINGTON: 2000 For Sale by the Superintendent of Documents, U.S. Government Printing Office Washington, DC 20402-9325

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FOREWORD

Once again, it is a pleasure to be able to reflect on the accomplishments of the NIST Center for Neutron Research over the past year. In the reactor operations area, 1999 was another outstanding year. In spite of an unsheduled maintenance shutdown, the reactor operated 250 days, with a reliability factor of better than 90%. The cold source availability for the period was 98%; i.e., the cold source held the reactor from operation 4 days during the year. The remaining spent fuel in the storage pool was shipped, providing space for at least five years of operation. Also, an order has been placed for a new cooling tower which will not only provide needed capability for the next 25 years, but will also reduce the plume visible during cold weather. Last, steady progress has been made in preparing for a license renewal application to the Nuclear Regulatory Commission, in order to extend the period of operation beyond 2004.

We have also made great progress in instrumentation, with the back scattering spectrometer operational; with the spin echo spectrometer now being used for real measurements; and with the disk chopper spectrometer being commissioned. All three of these instruments will be available to "friendly" users in the next proposal cycle. Other work is also advancing well–the perfect crystal small angle scattering spectrometer (part of the NSF/NIST CHRNS) is being installed at the reactor; the first phase of the high intensity filter analyzer spectrometer is ready to begin installation; and the design and manufacture of new thermal neutron spectrometers is underway. This simultaneous development program has put severe strains on our resources, but we can now look forward to many years of benefit from the results.

Finally, as always, the results are seen in the output of the researchers who use the facility. As was done last year, we are presenting highlights of this work in the following chapters of this report. I think all can agree that the results truly speak for themselves.



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INTRODUCTION TO THE NIST CENTER FOR NEUTRON RESEARCH (NCNR)

Modern technological society is dependent upon increasingly sophisticated use of materials, many of whose attributes are dictated by their sub-microscopic structural and dynamical properties. Our knowledge of these properties is provided by a wide range of scientific techniques of which the many types of scattering (for example, X-rays, light, electrons, neutrons) are arguably the most important. Of these probes, neutrons are perhaps least familiar, but they provide important advantages for many types of measurements.

Neutrons, as prepared for use at modern sources, are moving at speeds comparable to those of atoms moving at room temperature, thus providing the ability to probe dynamical behavior. At the same time, neutrons are well matched to measurements at length scales ranging from the distances between atoms to the size of biological or polymer macromolecules. Neutrons are sensitive to the magnetic properties of atoms and molecules, allowing study of the underlying magnetic properties of materials. They also scatter quite differently from normal hydrogen atoms than they do from heavy hydrogen (deuterium), allowing selective study of individual regions of molecular systems. Finally, neutrons interact only weakly with materials, providing the opportunity to study samples in different environments more easily (at high pressures, in shear, in reaction vessels, etc.), and making them a non-destructive probe. These favorable properties are offset by the relative weakness of the best neutron sources compared to X-ray or electron sources, and by the relatively large facilities required to produce neutrons. As a result, major neutron sources are operated as national user facilities to which researchers come from all over the United States (and abroad) to perform small scale science using the special measurement capabilities provided.

In addition to scattering measurements, neutrons can be used to probe the atomic composition of materials by means of capture and resultant radioactive decay. The characteristics of the decay act as "fingerprints" for particular atomic nuclei, allowing studies of environmental samples for pollutants (e.g., heavy metals), characterization of Standard Reference Materials, and many other essential measurements. While the scattering and capture users of neutrons are little concerned with understanding the inherent properties of the neutron, there are important areas in physics that can be explored by carefully measuring fundamental neutron behavior. Examples include the lifetime of the free neutron, an important quantity in the theory of astrophysics; the beta decay process of the neutron, the details of which are stringent tests of nuclear theory; and the effects of various external influences such as gravity or magnetic fields on neutrons.

The NCNR utilizes neutrons produced by the 20 MW NIST Research Reactor to provide facilities, including the nation's only internationally competitive cold neutron facility, for all of the above types of measurements to a national user community. There are approximately 35 stations in the reactor and its associated beams that can provide neutrons for experiments. At the present time 26 of these are in active use, of which 6 provide high neutron flux positions in the reactor for irradiation, and 20 are beam facilities. A schematic layout of the beam facilities and brief descriptions of available instrumentation are given below. More complete descriptions can be found at http://www.ncnr.nist.gov/.

These facilities are operated both to serve NIST mission needs and as a national facility, with many different modes of access. Some instrumentation was built years ago, and is not suited to general user access; however, time is available for collaborative research. NIST has recently built new instrumentation, and reserves 33 % of available time for mission needs with the balance available to general users. In other cases, instrumentation was built and is operated by Participating Research Teams (PRT). PRT members have access to 75 % of available time, with the balance available to general users. In a special case, NIST and the National Science Foundation established the Center for High Resolution Neutron Scattering at the NCNR, with a 30 m Small Angle Scattering (SANS) instrument, a cold neutron triple axis spectrometer, and a perfect crystal SANS under construction. For these facilities, most time is available for general users. While most access is for research, whose results are freely available to the general public, proprietary research can be performed under full cost recovery. Each year, approximately 1600 researchers (persons who participated in experiments at the facility, but did not necessarily come here) from all areas of the country, from industry, academe, and government use the facility for measurements not otherwise possible. The research covers a broad spectrum of disciplines, including chemistry, physics, biology, materials science, and engineering.

NIST CENTER FOR NEUTRON RESEARCH LAYOUT



1 Horizontal Sample Reflectometer

Horizontal surface of sample allows reflectivity measurements of free surfaces, liquid vapor interfaces, as well as polymer coatings.

2 Neutron Interferometry and Optics Station

Includes perfect silicon interferometer; vibration isolation system provides exceptional phase stability and fringe visibility.

3 Prompt Gamma Activation Analysis

Cold neutron fluxes allow detection limit for H of 1 μ g to 10 μ g. Focused beams are available for profiling.

- 4 NG-7 30 m SANS Small Angle Neutron Scattering instrument for microstructure measurement sponsored by NIST, the ExxonMobil Research and Engineering Co., the University of Minnesota, and Texaco R&D.
- 5 Neutron Physics Station A cold neutron beam 150 mm x 60 mm, available for fundamental neutron physics experiments.

6 Fermi Chopper TOF Spectrometer

A hybrid time-of-flight spectrometer for inelastic scattering, with wavelengths between 0.23 nm and 0.61 nm. The wavelength is chosen by a focusing pyrolytic graphite crystal, while the beam is pulsed by a simple Fermi chopper.

7 Spin Echo Spectrometer A neutron spin echo spectrometer offering neV energy resolution, based upon Jülich design, sponsored by NIST, Jülich and ExxonMobil.

8 SPINS Spectrometer

Spin Polarized Inelastic Scattering, a cold neutron triple axis spectrometer with spin polarization capabilities for high resolution studies, and position sensitive detector capability, sponsored by the National Science Foundation and NIST; part of Center for High Resolution Neutron Scattering (CHRNS).

9 Disk Chopper TOF Spectrometer

Versatile time-of-flight spectrometer, with beam pulsing and monochromatization effected by 7 disk choppers. Used for studies of dynamics in condensed matter, including macromolecular systems.



10 NG-3 30 m SANS

Instrument for microstructure measurement sponsored by the National Science Foundation and NIST; part of CHRNS.

11 Backscattering Spectrometer

High intensity, very high resolution backscattering spectrometer, with many innovative features, and energy resolution of approximately 1 µeV.

12 8 m SANS

Instrument for polymer characterization, sponsored by Polymers Division.

13 Vertical Sample Reflectometer Instrument for measuring subsurface structure with polarization analysis capability. Reflectivities down to 10⁻⁸ can be measured.

14 BT-8 Residual Stress Diffractometer

Diffractometer optimized for depth profiling of residual stress in large components.

15 BT-9 Triple Axis Spectrometer

Triple axis crystal spectrometer for measurements of excitations and structure.

16 BT-1 Powder Diffractometer

Powder diffractometer with 32 detectors; incident wavelengths of 0.208 nm, 0.154 nm, and 0.159 nm, with highest resolution of $\delta d/d = 8 \times 10^{-4}$.

17 BT-2 Triple Axis Spectrometer

Triple axis crystal spectrometer with polarized beam capability for measurement of magnetic dynamics and structure.

18 BT-4 Filter Spectrometer

A triple axis crystal spectrometer with a Be or Be/Graphite filter analyzer option for chemical spectroscopy.

19 Cold Neutron Depth Profiling

A station for quantitative profiling of subsurface impurities and coatings, based on neutron capture and emission of a charged particle.

20 Thermal Column

A very well-thermalized beam of neutrons used for radiography, tomography, dosimetry, and other experiments.

NCNR IMAGES 1999

1. An incoming SANS image excites the interest of Martin Vigild (center) as Frank Bates (standing), Newell Washburn (right) and Ken Hanley, all of the University of Minnesota, look on.

2. NCNR's Peter Gehring describes the analyzer of the spin polarized triple axis spectrometer (SPINS) to NCNR Summer School participants.







3. The Spin Echo Spectrometer (NSE) at NCNR, commissioned this year, is described in a Research Highlight in this issue.

4. Stephen FitzGerald (Oberlin College) loads a sample at the HFBS.

5. Gudrun Schmidt (NIST, Polymers Division) awaits the display of an updated SANS image.

4







6. NCNR Summer School participants interacting at the recently commissioned high-flux backscattering spectrometer (HFBS).

7. Silke Rathgeber and So Hyun Park (both at NCNR), ready to load a sample at the NSE.

8. The science/engineering/technical team which built and put the HFBS into operation.









9. John Copley (NCNR) in front of the soon-to-be commissioned disk chopper time-of-flight spectrometer (DCS).

10. Dave Clem and Mike Rinehart (both of NCNR) installing biological shielding at the DCS.

SUPERLATTICE MAGNONS

powerful technique for optimizing material properties is to deposit alternating layers of different materials to form a thin film superlattice. In particular, magnetic and non-magnetic materials grown as a superlattice exhibit a variety of tunable couplings between the magnetic layers. Control of these couplings by varying the layer materials and thicknesses has lead to dramatic increases in performance in certain device applications. A prime example is the recent use of layered magneto-resistive films in high-performance magnetic recording heads and sensors. The inter-layer magnetic coupling can survive across as many as 30 non-magnetic atomic planes due to its one-dimensional nature, but the coupling strength is always much weaker than bulk material magnetic interactions.

The behavior of the magnetic fluctuations (magnons) in these weakly coupled layer systems is of interest because it provides a

direct measure of the magnetic interactions responsible for the magnetic structure, and leads to a better understanding of the unique layer to layer couplings. Until now, the only measurements that have probed these fluctuations have used Brillouin light scattering [1], or ferromagnetic-resonance techniques [2], both of which measure only the longest wavelength dynamics. These measurements have found interesting resonances associated with the superlattice structure.

It is important to directly measure the magnons at shorter wavelengths in order to determine the dispersion, which directly relates to the magnetic interactions, both within and between layers, and whether or not the magnetic fluctuation waves propagate between layers. Only inelastic magnetic scattering techniques can provide this information, but with current neutron scattering sources the intensities from such measurements can be prohibitively low, because the amount of magnetic material in the films is so small.

In order to overcome this difficulty we have made neutron inelastic scattering measurements on a very large superlattice of alternating layers of dysprosium and yttrium. Dysprosium is the magnetic constituent, and it has the strongest neutron magnetic-scattering of all the elements. In order to maximize the amount of Dy, 350 bilayers composed of 43 Å of Dy and 28 Å of Y (designated $[Dy_{43}/Y_{28}]_{350}$) were grown by MBE techniques on a 2.5 cm x 1.3 cm substrate resulting in 3 mg of Dy in the sample.

The magnetic structure of this superlattice has previously been determined to be the basal-plane helical structure of bulk



FIGURE 1. Inelastic magnetic scattering from a $[Dy_{43}/Y_{28}]_{350}$ superlattice, obtained by subtracting 10 K data from 75 K data, shown as an intensity map in Q-energy space. The highest intensity is 300 counts/ 30 min and decreases by 30 counts for each level. A magnetic Bragg peak is just off the graph at Q = 1.97 Å⁻¹. A. Schreyer Institut für Experimentalphysik Festkörperphysik Ruhr-Universität Bochum D-44780 Bochum, Germany

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Dy with a coherence length greater than 4 bilayers. The helix progresses remarkably undisturbed through the conduction electrons of the non-magnetic yttrium, but with a turn-angle that is different than in the dysprosium. The coupling strength across the yttrium has been measured by applying a uniform external magnetic field. The coupling breaks down when the external field provides 0.2 meV per Dy atom in the basal-plane, while the equivalent zone-boundary magnetic fluctuation in bulk Dy is greater than 5 meV per Dy atom.

The inelastic neutron scattering measurements were performed at the NIST Center for Neutron Research on the coldneutron spectrometer SPINS. A multi-component crystal was used to analyze the energy of the scattered neutrons in order to enhance the measured intensity while sacrificing wave-vector (Q) resolution. Measurements were performed at 75 K as a compromise between the size of the ordered magnetic moment and the thermal population of magnons. The magnons of interest are those propagating along the superlattice growth direction or the c-axis of the hcp rare-earth structure. Lower magnon energies produce higher thermal populations, but become contaminated with elastic background scattering, so it is necessary to subtract scans taken at low temperatures where the magnons have become depopulated in order to remove this



FIGURE 2. The dispersion along the growth direction (c-axis) measured for the $[Dy_{48}Y_{22}]_{350}$ superlattice is plotted against the energy scale on the left side. It is compared to the measured dispersion in bulk dysprosium (solid lines) expected to originate from each of the superlattice magnetic Bragg peaks. (Only the negative Q branches from the two low Q Bragg peaks are shown). On the right side the diffraction scan for this sample showing the superlattice peaks is displayed. This film is so large that the sample diffraction peaks are as strong as the substrate peak shown in blue on the right.

background. The resulting magnetic signal is shown in Fig. 1 as a color-coded intensity map in Q-energy space. There is a clear ridge of intensity which moves to higher energies as Q moves away from the magnetic Bragg peak at 1.97 Å⁻¹. We have concentrated on the magnon branches that extend towards smaller Q since they move away from the intense magnetic and nuclear Bragg peaks that produce a large elastic background. This measured dispersion is compared with bulk Dy in Fig. 2, which also shows the diffraction pattern along the growth direction (c-axis) of the superlattice. The bulk Dy dispersion is shown as lines originating from the magnetic Bragg peaks at Q = 1.97 Å⁻¹ and Q = 2.06 Å⁻¹. The measured magnons are shown as bars centered on the measured peak positions and with lengths representing the full-width-at-half-maximumintensity. The agreement with the bulk dispersion is quite good. There is no evidence in these data of the influence of the yttrium layers other than possibly the splitting of the dispersion into two branches because of the superlattice structure as shown by separate bars at both Q = 1.8 Å⁻¹ and at Q = 1.75 Å⁻¹. The observed modes are not over-damped, but we cannot measure the damping under the current experimental conditions. Also, these data would have to be extended to smaller Q, in order to approach the interface thickness. We are currently designing additional focusing configurations so that the instrumental resolution will be better optimized for measurements of this dispersion surface.

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ANTIFERROMAGNETIC SPIN REORIENTATION IN EXCHANGE-BIASED Fe₃O₄/NiO SUPERLATTICES

The magnetic hysteresis loop of a ferromagnetic material can be displaced along the field axis as a result of magnetic exchange coupling to an antiferromagnet. This "exchange-biasing" phenomenon was discovered over 40 years ago in oxidized Co particles [1] and has been observed in a variety of thin films and multilayers. Artificial spin-valve structures with exchange-biased layers show great promise for applications as magnetoresistive sensors in read heads. Recent theories [2-4] make specific predictions about the origin of exchange biasing and the response of the antiferromagnetic layer.

High-angle neutron diffraction is an ideal probe as the antiferromagnet gives rise to distinct reflections of magnetic origin. We have performed neutron diffraction studies of exchange-biased [001] Fe₃O₄/NiO superlattices on the SPINS and BT-9 triple-axis spectrometers. These measurements confirm that the NiO layers retain their bulk antiferromagnetic structure in which ferromagnetic {111} planes alternate direction along each <111> axis. Our data show that exchange biasing is associated with domain walls that form and "freeze" within the antiferromagnetic NiO layer. Upon field cooling into the exchange-biased state, magnetic domains lock within the NiO layers and do not change with subsequent application of magnetic fields. In contrast, the antiferromagnetic domain sizes in unbiased samples prepared by cooling in zero field depend sensitively on the strength of the applied field.

We focus here on a $\text{Fe}_3\text{O}_4(6 \text{ nm})/\text{NiO}(11 \text{ nm})$ superlattice deposited using oxygen plasma-assisted molecular beam epitaxy. Measurements of the magnetic hysteresis loop show little evidence of exchange biasing after cooling in zero field. Strong exchange biasing is induced upon field cooling the superlattices from high temperatures. For our superlattice, the biasing field at 30 K is 0.043 T after field cooling from 550 K.

For the diffraction experiments the sample was oriented as shown in the inset of Fig. 1. Vertical magnetic fields, H, were applied in the sample plane. In this configuration, the (1 1 1) and $(\bar{1} \ \bar{1} \ l)$ NiO antiferromagnetic reflections lie in the scattering plane. Using a horizontal-field magnet, the (1 $\bar{1} \ l)$ and ($\bar{1} \ l$ 1) reflections could be accessed by rotating the sample 90° about the growth axis. Figure 1 shows a typical growth-axis (00*I*) scan through the (111) reflection for the superlattice. Structural stacking faults [5] at the NiO/Fe₃O₄ interfaces limit the coherence of the Fe₃O₄ structural and magnetic order to a single Fe₃O₄ layer. As a result, we can easily separate the broad Fe₃O₄ and narrow NiO contributions to the reflection and track the latter as a function of field.

The neutron scattering data reveal that the magnetic domain sizes in the antiferromagnetic NiO depend on the presence or absence of exchange biasing. Figure 2 shows the full-width-at-halfmaximum (FWHM) for the (111) NiO peak scanned along the (00*l*) and (*ll*O) directions after cooling in a 6 T field (i.e., exchange-biased state) and cooling in zero field (i.e., unbiased state). After field cooling, the FWHM of the NiO reflection scanned along the (00*l*) growth direction shows no dependence on field.



FIGURE 1. Zero-field growth-axis (00/) scan through the (111) reflection for the $Fe_3O_4(6 \text{ nm})/NiO(11 \text{ nm})$ superlattice after cooling to 78 K in a 6 T field parallel to [110]. The broad Fe_3O_4 peak is shown in red, and the remaining scattering is from the NiO. The green arrow marks the FWHM of the NiO peak. The inset shows the scattering diagram.

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The antiferromagnetic domain size along the growth direction, determined from the inverse of the FWHM, remains constant near 750 Å after field cooling. This contrasts with the pronounced field-dependence of the FWHM observed after cooling in zero field (Fig. 2). In this state, the FWHM is smallest near zero field, but approaches the constant field-cooled value when the magnitude of the field is greater than 2 T. The corresponding domain size varies from approximately 1200 Å in zero field to 800 Å in high fields. Growth-plane (*ll*0) scans through the NiO reflection show a similar difference between the field dependence of the FWHM after cooling in zero field and cooling in a 6 T field (Fig. 2).

Upon field cooling, domain walls both parallel and perpendicular to the growth direction lock into the antiferromagnetic layer, presumably due to the exchange coupling between the NiO antiferromagnetic moments and the Fe_3O_4 moments that are aligned parallel to the cooling field. After cooling in zero field, the domains are



FIGURE 2. Full-width-at-half-maximum (FWHM) of the (111) NiO reflection as a function of field at 78 K after cooling in a 6 T field and after cooling in zero field. The top plots show the FWHM of the peak along the (00/) direction. The bottom plots show the FWHM from (//O) scans. The black, blue and red data are from each field cycle.

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larger, but reversibly decrease in size in high fields (Fig. 2) as the magnetic frustration increases. Consistent with several theoretical predictions [2,3] we believe that exchange biasing may originate from magnetic frustration that leads to "frozen" domain walls in the field-cooled state.

We also observe that the magnitude of the ordered NiO moments in all four of the {111} domains in the Fe₃O₄/NiO superlattices depends upon applied field. After cooling in a large vertical field, the intensity of the antiferromagnetic (111) NiO reflection reversibly decreases as the field is increased. (The behavior is qualitatively similar after cooling in zero field.) Analogous fieldcooled measurements of the (111) NiO peak were performed in a horizontal field and surprisingly show a comparable intensity decrease. Some of the NiO moments thus seem to disappear out of all four {111} domains. We speculate that these NiO spins may become disordered as a result of the high internal fields. Simultaneously, some of the NiO moments reorient perpendicular to the Fe_3O_4 magnetization direction in high fields [6]. While this "spin-flop" response is favored by some theoretical models [4], it does not appear to be directly responsible for the exchange biasing for these samples.

Future studies will focus on the characteristics and origin of the "spin-flop" response of the NiO spin structure to large magnetic fields. In addition, we will further explore the differences between the antiferromagnetic domains for the exchange-biased and unbiased conditions.

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SPIN DENSITY WAVE ORDER IN THE SUPERCONDUCTING STATE OF La₂CuO_{4+Y}

In the high temperature (high- T_c) superconductors, the multiple roles played by the electrons continue to defy theoretical understanding. It appears that all high- T_c superconductors are based on structures with CuO₂ planes, in which the electrons on neighboring copper ions are strongly coupled magnetically. In systems based on La₂CuO₄, antiferromagnetism is a dominant feature of the phase diagram at low doping levels, and conventional itinerant-electron behavior dominates in the high doping regime. Intermediate doping levels are described by neither, but this is where the superconducting properties are optimal. In our experiments, we focus on the evolution of magnetic properties from the insulating antiferromagnet to the superconductor.

It is becoming increasingly apparent that incommensurate spin structures are universal to the high- T_c superconductors. Especially noteworthy is the observation of static incommensurate magnetic ordering coexisting with superconductivity in $La_{1.6-x}Nd_{0.4}Sr_xCuO_4$ and recently in $La_{2-x}Sr_xCuO_4$ [1, 2]. Further experiments are needed to understand the coexistence of the incommensurate spin density waves (SDW) and superconductivity, the details of the spin structure, and the influence of pinning potentials.

A crystal of La₂CuO_{4+y} with a superconducting transition at ≈ 42 K was produced by doping pure La₂CuO₄ electrochemically with a large quantity of excess oxygen (y ≈ 0.12). The superconducting shielding signal measured after cooling in zero field is shown in Fig. 1a. The transition is very sharp with an onset T_c ≈ 42 K. Our initial characterization suggests that the crystal is a bulk superconductor with a hole concentration similar in density and homogeneity to that of La_{2-x}Sr_xCuO₄ crystals with x ≈ 0.15 .

At temperatures below the superconducting T_c , we observe elastic magnetic scattering at a quartet of incommensurate positions centered around (100), which is the Bragg position for the antiferromagnetism in the undoped insulator. Surprisingly, we find that the incommensurate wavevectors are not precisely along the Cu-O-Cu bond direction, but are rotated by about 3[°].

In Fig. 2a we show elastic scans along the in-plane h direction through an incommensurate position for various temperatures using 5 meV neutrons. Below 42 K, the observed peaks are extremely sharp and are resolution-limited, while above 42 K



FIGURE 1. (a) Magnetic susceptibility measured after cooling in zero field. (b) Peak intensity of the incommensurate elastic scattering as a function of temperature. The measurement was performed with two different neutron energies of 13.7 meV and 5 meV. The solid line denotes the BCS superconducting order parameter squared, with a T_c of \approx 41 K.

the peaks disappear. The solid lines in the figure are Gaussians convolved with the instrumental resolution which indicate that the in-plane static magnetic order is correlated over distances larger than 400 Å. From this, we conclude that static long-range magnetic order exists in the superconducting state of La_2CuO_{4+y} . Also, the SDW order is not specific to a tetragonal crystal structure as previously believed since this crystal is orthorhombic; it is a more general phenomenon.

We then investigated how the static spin arrangement is correlated between CuO_2 planes. The *l*-dependence of the incommensurate scattering is shown in Fig. 2b. The intensity modulation of both the (10*l*)- and (01*l*)-centered scattering is reminiscent of the spin structure of the undoped parent compound La_2CuO_4 . The solid lines in both panels represent fits to Gaussian lineshapes convolved with the instrumental resolution, assuming a model for the stacking arrangement and spin direction identical to that of pure La_2CuO_4 . Y. S. Lee, R. J. Birgeneau, and M. A. Kastner Department of Physics Massachusetts Institute of Technology Cambridge, MA 02139

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FIGURE 2. (a) Scans along the in-plane H direction over one of the incommensurate SDW peaks for various temperatures. (b) The top panel shows the *l*-dependence of the SDW scattering centered about the (10/) position. The bottom panel shows scattering centered about the (01/) position.

Here, the only free parameters are the width and a single overall intensity scale factor. The agreement is clearly satisfactory, with the fit indicating that the spins are correlated across $\approx 3 \text{ CuO}_2$ planes. We conclude that the stacking arrangement of the magnetically ordered planes in our La₂CuO_{4+y} sample follows that of undoped insulating La₂CuO₄, even though the magnetic order in the CuO₂ planes is incommensurate. This is the first direct evidence that the magnetism of the doped superconductor mimics the magnetism in the undoped insulator in such a specific way.

We show in Fig. 1b the peak intensity of the elastic signal as a function of temperature measured using both 13.7 meV and 5 meV neutrons. The fact that one obtains identical results for the temperature dependences of the intensities with these two different neutron energies and, concomitantly, energy resolutions indicates that the scattering is truly elastic. The intensity of the elastic scattering turns on at approximately the same transition temperature as superconductivity. Noting that the intensity of the magnetic scattering is proportional to the square of the magnetic order parameter, we plot the square of the BCS order parameter curve over the data using a T_c of \approx 41 K. The agreement indicates that the magnetism exhibits mean field behavior just like conventional superconductivity. This is very surprising given the two dimensionality of the ordered magnetism. The size of the ordered moment is $0.15 \mu_{\text{B}}$, which is 25 % of the ordered moment in pure La₂CuO₄. Our results argue against an itinerant electron description of the incommensurate magnetism since it is difficult to see how a delocalized model can support interplanar spin correlations and choose the same preferred spin direction as in insulating La,CuO₄. It appears that the spins are localized and ordered in this high temperature superconductor.

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LOCAL SPIN RESONANCE AND SPIN-PEIERLS-LIKE PHASE TRANSITION IN ZnCr₂O₄

ost magnets order when the thermal energy drops below a microscopic energy scale for magnetic interactions. The topology of certain lattices can, however, reduce the energy gain associated with long range ordering thus favoring more entropic phases. Figure 1 shows one such lattice, the tetravalent site in the pyrochlore structure, that forms a network of corner-sharing tetrahedra. As shown in the figure, antiferromagnetically (AFM) interacting spins on the lattice cannot satisfy all their exchange interactions simultaneously. This phenomenon, called geometrical frustration, can lead to macroscopic classical ground state degeneracy and offers the possibility of qualitatively new states of matter. Theoretical studies have in fact shown that spins with nearest neighbor antiferromagnetic Heisenberg interactions on the pyrochlore lattice do not have a long range ordered phase at all.

Pyrochlore magnets studied experimentally so far exhibit a coninuous phase transition at a finite temperature, T_f , into a glassy phase with static short range correlations [1]. Spinel antiferromagnets, AB_2O_4 , in which the octahedral B site forms the same magnetic lattice as in the pyrochlore structure, however behave quite differently. For instance, $ZnCr_2O_4$ exhibits a first order phase transition to a long-range ordered Néel phase at $T_c = 12.5$ K, much less than the Curie-Weiss temperature $|\theta_{CW}| = 393$ K. We have explored this ordered phase and the corresponding phase transition through inelastic neutron scattering [2].

Figure 2 provides an overview of our neutron scattering results as a color image of $I(Q, \omega)$ at three temperatures. For $T > T_{a}$, Figures 2a and 2b show a constant-Q ridge centered at $Q \approx 1.5$ Å⁻¹ and extending beyond 10 meV. The ridge indicates quantum critical fluctuations of small AFM clusters, most likely antiferromagnetically correlated tetrahedra, and closely resembles those obtained in similar experiments on other frustrated AFM. For $T < T_c$, however, the low energy spectral weight concentrates into a sharp constant-energy mode centered at $\hbar \omega = 4.5 \text{ meV}$ \approx |J| >> k_BT_c. The wave vector dependence of this resonance intensity reveals that it is an excitation among antiferromagnetically correlated nearest neighbor spins. Though they can not be seen in Fig. 2c, there are in fact magnetic Bragg peaks in the elastic scattering channel (see Fig. 3b), which provide evidence for longrange order for $T < T_c$. It is unusual that excitations of such localized character exist in a long-range ordered phase. The resonance





indicates the presence of weakly interacting spin clusters within the ordered phase, which is a key feature of geometrically frustrated magnets.

Theoretical work indicates that magnetic order cannot develop in an isotropic spin system with nearest neighbor antiferromagnetic Heisenberg interactions on the pyrochlore lattice. It is natural to ask what deviation from this model causes order to develop in $ZnCr_{2}O_{4}$? To answer the question we probed the temperature dependence of static and dynamic features of this system in the vicinity of the phase transition. Figure 3 shows that long range antiferromagnetic order (blue squares in frame (b)) and the local spin resonance (frame (a)) appear simultaneously in a spectacular first order transition. It also shows that magnetic ordering is accompanied by a cubic to tetragonal lattice distortion (red circles in frame (b)). The lattice distortion plays a crucial role in relieving frustration and allowing long-range order to develop. It is well known that exchange interaction between Cr3+ ions whose oxygen coordination octahedra share an edge are strongly dependent on the oxygen bond angles and hence the metal ion spacing. As a consequence the tetragonal strain $\epsilon_s > 0$ and $\epsilon_s < 0$ yields weaker AFM interactions between spins occupying the same basal plane and stronger AFM interactions between all other spin pairs. This reduces S.-H. Lee

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the energy of a particular long range ordered spin configuration with respect to the low energy degenerate manifold thus allowing the system to achieve long range order. The overall picture that emerges is that of two distinct phases in competition: a cubic cooperative paramagnet and a tetragonal long-range ordered antiferromagnet. Though the spin Hamiltonian has a lower expectation value in the latter phase, the lattice energy is greater and the entropy is lower in the tetragonal phase. The phase transition occurs when the free energy of the tetragonal low entropy phase drops below that of the disordered cubic paramagnet.

There are strong analogies between the phase transition in $ZnCr_2O_4$ and the spin-Peierls (SP) transition. In both cases the high T phase is nearly quantum critical and can lower its energy through a lattice distortion. In both cases the transition occurs from



FIGURE 2. Contour maps of the magnetic neutron scattering intensity at temperatures spanning the phase transition at $T_c = 12.5(5)$ K. The data were taken by utilizing a flat analyzer and two-dimensional position-sensitive detector at the SPINS spectrometer.

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FIGURE 3. (a) Image of inelastic neutron scattering for Q = 1.5 Å¹. (b) T-dependence of magnetic Bragg scattering from a powder, σ_m (blue squares), and of lattice strains measured n a single crystal (red circles).

a strongly correlated paramagnet: $T_c \ll \theta_{CW}$, and in both cases low energy spectral weight is moved into a finite energy peak.

There are also important differences between the two transitions. The low T phases are qualitatively different, the transition in ZnCr_2O_4 is a first order one, while the SP transition is second order, and the change in entropy at T_c plays an important role in ZnCr_2O_4 , but not in a SP transition. The central idea that finite lattice rigidity can preclude a spin liquid at T = 0 however does carry over and should be relevant for any frustrated magnet when other symmetry breaking interactions are sufficiently weak.

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COMPOUND REFRACTIVE OPTICS IMPROVE RESOLUTION OF 30 m SANS INSTRUMENT

n the most favorable cases, cold neutrons can be deflected through an angle of a degree or two by grazing incidence reflection, but by only an arc second or two by refraction. Hence grazing incidence reflection optics has long been considered the most promising means for focusing neutrons for applications such as smallangle neutron scattering (SANS). Numerous attempts over more than 30 years to produce highly reflective surfaces for neutrons have been vitiated, however, by SANS from the mirror surfaces themselves, which blurs the focus. The best mirrors produced thus far are only marginally better for SANS than pinhole collimation, i.e., circular apertures separated by long distances.

Scientists at Bell Laboratories recently took a fresh look at this problem and proposed that multiple refraction from relatively high index, low absorbing material could be superior to reflection optics or conventional pinhole collimation for SANS. Initial measurements [1] at Risø National Laboratory, Denmark, demonstrated the proposed focusing effect, but did not make quantitative comparisons with reflection optics or pinhole collimation for application in SANS instruments. Measurements made recently at the NCNR in collaboration with the Bell Labs scientists [2] have addressed these issues and have demonstrated and quantified the significant



FIGURE 1. Now installed in the pre-sample flight path of the NG-7 30 m SANS instrument are two sets of MgF_2 biconcave lenses that can be inserted into the beam under computer control. The 28-lens array in the foreground focuses 8.44 Å neutrons at a distance of 15 m from the lenses, and the 6-lens set focuses 18 Å neutrons at the same distance. Each lens is 25 mm in diameter, has a radius of curvature of 25 mm, and is 1 mm thick in the center.



FIGURE 2. Upper panel, conventional SANS pinhole collimation. The source and sample apertures, A_{1p} and A_{2p} , respectively, determine the shape and extent of the beam profile, I(x), at the detector plane. Lower panel, focusing lens geometry. Ideally, the source aperture, A_{11} , alone determines the beam profile.

improvement in resolution that can be achieved with compound refractive optics.

Our tests were made with the same set of cylindrical biconcave MgF_2 (magnesium fluoride) lenses used in the Risø study. Up to 30 lenses were placed end-to-end near the sample position of the 30 m SANS instrument to focus neutrons, emanating from a circular source aperture 15 m upstream, onto the plane of instrument's twodimensional detector. Figure 1 shows an array of 28 lenses for focusing 8.44 Å neutrons at a distance of 15 m from the sample, next to a set of 6 lenses for focusing 18 Å neutrons at the same distance.

For this geometry, as depicted in Fig. 2, the lenses ideally produce a 1:1 image of the source aperture at the detector independent of the size of the sample. Since the scattering signal is proportional to sample size, the lens system can, in principle, be used to improve resolution more efficiently, by reducing the size of the source aperture, than is possible with pinhole collimation where both the source aperture and sample size must be reduced proportionally to improve angular resolution. Aberrations and small-angle scattering by the lenses could, however, blur the image to such S.-M. Choi, J. G. Barker and C. J. Glinka NIST Center for Neutron Research National Institute of Standards and Technology Gaithersburg, MD 20899-8562

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FIGURE 3. Red dots are measured points along the vertical profile of the image formed by the 28 lens array shown in Fig. 1 of a 9.5 mm diameter source of neutrons 15 m upstream from the lenses. The blue dots are a Monte Carlo calculation of the profile that includes the effects of spherical and chromatic aberrations as well as the broadening caused by gravity. The shoulder in the measured profile at Y = 0 is due to a residual fast neutron component in the beam.

a degree that any advantage over pinhole collimation, which does produce a sharply defined beam spot at the detector, would be lost.

To accurately measure the intensity profile produced by the lenses, a dysprosium foil was positioned at the focal plane and exposed to the focused beam for approximately 2 h. The activated foil was then placed in contact with a high resolution image plate which stored the image produced by the emitted gamma rays with a spatial resolution of better than 0.1 mm. A typical profile obtained from reading out the image plate is shown in Fig. 3. Also plotted in the figure is a Monte Carlo calculation of the profile that includes the effects of spherical and chromatic aberrations as well as the broadening caused by gravity. The measured profile agrees with the simulation down to intensity levels of 10⁻³ of the peak intensity and has an overall signal-to-background ratio in the wings approaching 10⁵, which is highly satisfactory for most SANS measurements.

The practical benefit provided by the lenses is demonstrated by the SANS data from voids in a single crystal (2.5 cm in diameter and 1 cm thick) of fast-neutron-irradiated aluminum shown in



FIGURE 4. Small-angle scattering from voids in a single crystal (2.5 cm in diameter and 1 cm thick) of fast-neutron-irradiated aluminum. The measurements were made under equivalent resolution conditions (i.e. nearly identical beam spot size at the detector) using both simple pinhole collimation and the 28 biconcave lens array shown in Fig. 1. The integrated gain in intensity due to the lenses is approximately 26.

Fig. 4. The measurements were made under equivalent resolution conditions (i.e., nearly identical beam spot size at the detector) using both simple pinhole collimation and the 28 biconcave lens array shown in Fig. 1. The first data point unaffected by the beam stop in both data sets occurs at $Q \approx 0.001$ Å⁻¹, but the scattered intensity at the detector (counts/cm²/s) is more than 10 times higher by using the lenses to illuminate a much larger area of the sample.

The focusing lenses shown in Fig. 1 are now installed for routine use in the NCNR's 30 m SANS instrument on guide NG-7. Further testing is planned to understand, and hopefully eliminate. the sources of parasitic scattering that contribute to the tails of the beam profile seen in Fig. 3, prior to installing a lens system in the NIST/NSF 30 m SANS instrument on guide NG-3.

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POLYMER BRUSH RESPONSE TO SOLVENT FLOW

The presence of polymer chains grafted or adsorbed onto a surface can dramatically alter the forces that affect interactions between surfaces. The equilibrium properties of such polymer brush systems have been studied for the past two decades, yielding general agreement between theory and experiment. Conversely, the nonequilibrium properties of polymer brushes are still under intense theoretical and experimental investigation. Of particular interest is the response of a brush to the frictional forces imposed by solvent flow. The behavior of polymer brushes subjected to flow has important technological implications for the rheology of colloidal dispersions stabilized by polymer layers, for the lubrication properties of polymer coated interfaces, for biocompatibility of medical implant devices, and for permeation flow through polymercontaining porous media [1].

The height of a polymer brush is determined by the equilibrium conformation of the tethered chains, which depends on both the grafting density and quality of solvent. The basic physics governing the static behavior of a polymer brush result from a competition between two opposing tendencies: 1) elastic contraction, as the chains attempts to maximize their entropy by adopting random walk configurations, and 2) monomer-monomer interactions, such as polymer-polymer repulsions, and polymer-solvent wetting [2, 3].

Polymer chain stretching in densely grafted brushes has been studied by many different techniques including surface forces apparatus [4], neutron reflectivity [5], and small angle neutron scattering (SANS) [6]. In general, there is good agreement with results from experiment, simulations and analytical calculations [3].

Oscillatory shear measurements performed with a surface forces apparatus suggest that the normal forces between a pair of brush surfaces are altered when sheared. However, these measurements do not give the actual brush profile either with or without shear. Effective hydrodynamic thickness measurements of polymer brushes under shear indicate a thickening of the brush; whereas neutron reflectivity experiments on adsorbed PS-PEO block copolymer brushes on a silica surface show no effect of shear on the brush density profile in good solvent, and a slight increase in poor solvent. These earlier reflectivity measurements were limited to shear rates of $\approx 10,000 \text{ s}^{-1}$ [7] since the adsorbed PS-PEO block copolymer





FIGURE 1. Schematic illustrating the effect of shear on grafted brushes predicted by Miao *et al.* [8] Such an effect would be consistent with the present data.



FIGURE 2. Shear cell. Arrows denote solvent flow direction. The polystyrene brush is grafted onto the Si crystal.

brushes have a tendency to come off the surface at higher shear rates.

Predictions from theoretical calculations of brush profiles under shear span the gamut of possibilities, ranging from brush thickening to brush compression, including no effect of shear flow on the density profile [3]. Miao *et al.* [8] predict that the response of a brush to the solvent shear flow is displayed as chain tilting toward and chain stretching along the direction of flow. However, the overall conformational properties such as brush thickness remain essentially unaffected (Fig. 1).

We have performed neutron reflectivity measurements on a chemically grafted polymer in both good and poor solvents at shear rates over an order of magnitude greater than previously reported. Our neutron reflectivity experiments measure the segment density profile of the polymer brushes under shear in an experimental cell similar to the one used by Baker *et al.* [9], (Fig. 2). We use deuterated polystyrene (d-PS), 83 kg/mol, with a trichlorosilane end group to bind the d-PS brush chemically onto a single crystal Si surface [5]. We used a good solvent, toluene, and a poor

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FIGURE 3. Representative shear data for deuterated polystyrene brush under shear in cyclohexane (a), and toluene (b). In both cases, black circles represent shear of 0 s⁻¹, whereas red squares represent data taken at 30,000 s⁻¹.

solvent, cyclohexane, as the solvent media. The dry brush height was measured by X-ray reflectivity to be 17.5 nm. Without shear the brush extends to 31 nm in cyclohexane and 75 nm in toluene. We measured the brush profile at several shear rates, up to 130,000 s⁻¹, yet we see no effect of shear on the brush density profiles in either solvent (Fig. 3). No desorption of the polymer brush was ever observed. In fact, the neutron reflectivity profiles at 0 s⁻¹ and 130,000 s⁻¹ look identical, indicating less than a 2 % to 3 % change in the brush density profile.

We have also been able to establish that the slight shear induced swelling reported by Baker *et al.* [7] for a poor solvent (cyclohexane), was probably due to frictional heating of the solvent. In high shear fields, heat generated from friction between the solvent and interior surfaces of the apparatus does not readily dissipate, causing a $\approx 2 \,^{\circ}$ C to 3 $^{\circ}$ C rise in the temperature of the shear cell. We were able to demonstrate that the brush height in cyclohexane is unaffected by shear when the cell temperature is carefully controlled. An elevated cyclohexane temperature swells the brush as the solvent quality improves. Naturally, these effects were not observed in toluene.

Thus, our neutron reflectivity data represent the first comprehensive measurements of shear effects on the density profile of a grafted polymer brush into regimes that are predicted by some to display an effect. We cannot at this time determine if the brush responds as predicted by Miao *et al.* [8], or if there is insufficient solvent penetration into the brush to exert enough force on the chains to induce conformational changes. Future measurements will distinguish between these cases.

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MACROMOLECULAR CONFORMATION IN ULTRATHIN POLYMER FILMS

Since thin polymeric films are ubiquitous in technological applications such as paints, lubricants, and adhesives, a critical characterization of their thermophysical properties is essential. A central premise in the development of theories for predicting the properties of polymer melts in confined geometries is that chains maintain their unperturbed Gaussian conformation, which they adopt in the bulk, in the direction parallel to the surfaces under all conditions [1,2]. These assumptions, which form the foundations of the important field of polymer thin films, have been questioned on the basis of indirect experimental findings [3,4]. We have utilized the power of small angle neutron scattering, especially the high neutron flux at NCNR, to unequivocally characterize the chain structure and conformation in ultrathin polymer films, and thus have resolved this important fundamental question.

The experimental measurements of chain conformations and system thermodynamics in thin films have remained elusive due to the small amounts of sample material involved. To illustrate this point, a thin film of 10 nm incurs a decrease in signal by $\approx 1 \times 10^4$ from a typical bulk polymer sample. In this case, the noise is comparable to the signal, complicating the experiments. Prior to upgrades of the cold neutron source at the NCNR, data collection times were prohibitive. Improvements in sample preparation, which are discussed in detail elsewhere [5,6], have allowed us to measure molecular size and conformation of an isotopically labeled blend of polystyrene (25 wt% d-PS/75 wt% h-PS) in films as thin as 12 nm. Two blends of nominally matched molecular weight, Mn, of 270,000 and 650,000, respectively, were utilized. These were labeled 270k and 650k, respectively. Solutions of the blends were spin cast on silicon substrates (Semiconductor Processing) and annealed at 120 °C ($T_{a} \approx 105$ °C).

The scattered intensity for a 15 nm thick film of the 270k blend is compared to an analogous bulk sample in Fig. 1. It is clear from the figure that, on a unit volume basis, the thin film scattering is higher than that of the bulk. We postulated that this difference is attributable to the scattering from the imperfections at both the air and the substrate interfaces, which is driven by the relatively high neutron contrast at these boundaries. To evaluate this component, films of pure d-PS were spin cast under identical conditions and their scattering measured.

The pure d-PS film data were fit with a simple Debye-Bueche form factor to obtain parameters for a roughness term. The blend film data were then fit by scaling this roughness term and adding a component obtained from the Random Phase Approximation (RPA) model. In the fitting, two RPA model parameters were also varied: the blend chain radius of gyration (R_g) and the Flory interaction parameter. The combined model is illustrated as the solid line in Fig. 1 along with the roughness term (long dashed line) and RPA term (short dashed line). The fact that the RPA term is very close to the data for the bulk blend illustrates that the film and bulk



FIGURE 1. Plots of I(Q) as a function of Q for dPS/hPS blends of Mn = 270k. Filled symbols, bulk sample; open symbols, data for a film of thickness D = 18 nm. The fit to these data (solid line) was obtained by utilizing both a roughness term (long dashed line) as well as the standard RPA form (short dashed line).

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samples have nearly the same R_g . Using this combined model, R_g was determined for films ranging in thickness over two decades (0.5 $R_g < D < 50 R_g$). The molecular size was found to be independent of film thickness (Fig. 2). Since the scattering vector is primarily in the surface plane, this conclusion is consistent with theoretical assumptions and suggests that chain conformation in the direction parallel to the surfaces are unaffected by confinement.



FIGURE 2. Plots of ratios of R_g and I(0), derived from the RPA component (short dashed line in Fig. 1) of the fits to the blend thin film data, to their corresponding bulk values. The ratios are plotted versus the ratio of the film thickness to the bulk R_g. Data are displayed for blends with Mn = 270k (filled circles) and Mn = 650k (open circles).

Our results clearly show that, in the thinnest films, the volume pervaded by a coil is decreased as compared to the bulk. This is because the R_g in the direction parallel to the surfaces is unaffected, while the corresponding quantity in the third direction is strongly reduced. In conjunction with other studies, which indicate a thickness-independent density in ultrathin polymer films, these conclusions indicate decreased intermolecular entanglement in thin polymer films. Since entanglement density directly affects the dynamic properties of polymeric systems, we contend that unusual thin film properties, such as the anomalous thickness dependence of diffusion coefficients and glass transition temperatures, are caused by this reduced entanglement density near a surface.

With thin films as a model system, and continuing increases in cold neutron flux, SANS at the NCNR is now an appropriate tool to study a host of problems involving interfacial structure, finite size phase behavior, and nano-patterning in systems as far ranging as engineering thermoplastics to biological systems. These, and related problems, are the focus of investigation in our research groups.

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PROBING THE LOCATION OF THE TERMINAL GROUPS OF A DENDRIMER

Dendrimers represent a new class of macromolecules developed in recent years. Typically, a dendrimer structure has a tri- or tetrafunctional core, which is surrounded by several "generations" of stepwise added trifunctional monomers, leaving the last generation with a large number of terminal units as shown in Fig. 1. The molecular weight doubles with each generation, leading to high molecular weights, and causing the dendrimer to become very compact and crowded.

Many of the potential technological applications of dendrimers depend on their segment density distribution. Previous scattering studies have shown that dendrimers have uniform interiors and are quite spherelike in their shape [1]. The location of the terminal groups is also of importance, since they are usually different chemically from the rest of the dendrimer. This invites a number of applications such as the support of catalysts or drugs or their use as hyperfunctional crosslink sites. The accessibility of these terminal groups depends on their location compared to the other dendrimer units. The location of the terminal units can be determined by labeling the last generation of the dendrimer with deuterium and using contrast matching techniques. Figure 1 shows the labeled groups in red and the rest of the dendrimer in blue. By choosing the proper mix of h- and d- solvents, the interior of the dendrimer will be matched, making only the labeled end groups visible in small angle neutron scattering (SANS).

A sixth generation polyamidoamine (PAMAM) dendrimer was reacted with acrylonitrile (vinyl-d3) to give the deuterium labeling for the SANS. Ethylene diamine was reacted with the dendrimer to give a labeled seventh generation dendrimer. A similar reaction was used to make a seventh and eighth generation dendrimer without labeling. Solutions of unlabeled eighth generation dendrimer were made in mixtures of CH₃OH and CD₃OH for determination of the match point. Three samples were analyzed, an unlabeled dendrimer in CD₃OH (high contrast), an unlabeled dendrimer in the match mixture (dendrimer matched), and the labeled dendrimer in the match mixture (interior matched).



FIGURE 1. Dendrimer structure with labeled terminal units.



FIGURE 2. SANS from G8 dendrimer in CD₃OH/CH₃OH mixtures.

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SANS was performed at the 30 m facilities at NIST. The spectrometers were operated at a wavelength of $\lambda = 6$ Å, and a wavelength spread of $\Delta\lambda/\lambda = 0.15$.

Figure 2 is a plot of SANS of dendrimer solutions with different CD₃OH contents. The intensity is the strongest in pure CD₃OH, weakens as CH₃OH is added and increases again when pure CH₃OH is used. The coherent scattering intensity varies as $I \approx (b_s - b_p)^2$ where b_p is the contrast of the dendrimer and b_s is the average contrast of the solvent mixture.

Figure 3 is a plot of the square root of the scattered intensity versus solvent composition with the values to the right made negative so that a straight line can be put through all of the data points. The zero intersection is at a mass fraction of $60.5 \text{ CH}_3\text{OH}$ which was the composition used in the matching experiments.

Figure 4 is a plot of the SANS of the three G7 samples. The circles give the scattering from the high contrast sample, showing strong scattering typical of large spherical dendrimers. The diamonds show the SANS of the same dendrimer, but under match conditions. This sample has no measurable coherent scattering sig-

1.5 1 0.5 0 $I(0=0)^{1/2}$ -0.5 -1 ϕ_{M} -1.5 -2 0.2 0.4 0.6 0.8 0 1 ϕ_{D}

nal, demonstrating that the match conditions have been achieved. The labeled dendrimer SANS is given by the squares. The scattering is weak because only the labeled terminal groups scatter.

A Guinier analysis of the scattering of the high contrast sample gives the radius of gyration (R_g) of the whole dendrimer, and the labeled - contrast match sample gives the R_g of only the terminal groups. The R_g of the whole dendrimer is (34.2 ± 0.2) Å, while the R_g of the terminal groups is (39.3 ± 1.0) Å.

The terminal groups of a seventh generation PAMAM dendrimer are 15 % larger than the average of all of the units. Therefore, the terminal units of a dendrimer are concentrated in the outer shell of a dendrimer.

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FIGURE 4. SANS of labeled and unlabeled G7 dendrimer in match and high contrast solvents.

FIGURE 3. Location of the match point.

SELF-ASSEMBLIES OF MEMBRANE ACTIVE PEPTIDES

n recent years we have made progress by neutron diffraction in a major structural challenge, namely in detecting and analyzing the structures of peptide assemblies in fluid membranes [1,2]. These experiments were performed with membranes in the form of oriented multilayers. Originally the samples were investigated in full hydration so that the physical properties of the lipid bilayers were close to those at physiological conditions. However, it was soon realized that new phenomena involving peptide-lipid interactions occur when the sample hydration is varied. In general, in full hydration, the peptide organization in each membrane is uncorrelated to the neighboring membranes. As the hydration level decreases, the peptides become correlated between bilayers, even though the membranes are still fluid. In many cases, further dehydration strengthens the correlation such that the peptide organization in the multilayers crystallizes [2]. The crystallization provides the possibility for high-resolution diffractional studies. Investigations along this line might also lead to useful information for crystallization of membrane proteins.

Antimicrobial peptides are inducible innate host defense molecules found in all multicellular organisms, including humans and plants. These peptides have a folded size comparable to the membrane thickness. All evidence indicates that antimicrobial peptides act by permeabilizing the cell membranes of microorganisms. But the molecular mechanisms of their actions are still not clear. We have found that all peptides, when they are bound to lipid bilayers, exhibit two distinct oriented circular dichroism spectra, one at low peptide-to-lipid ratios (P/L) and another at high P/L. This indicates that each peptide has two different physical states of binding to a membrane.

The transition from the low to the high P/L spectrum occurs over a narrow range of P/L as if there is a threshold concentration, called P/L*. At concentrations below P/L*, the peptides are embedded in the headgroup region, as suggested by the peptide orientation and the membrane thinning effect. At concentrations above P/L*, neutron in-plane scattering showed that the peptides form pores in the membranes, while no pores were detected below P/L*. The detection was achieved by exploiting the sensitivity of neutrons to D₂O, which had replaced the water in the membrane pores. As an example, Fig. 1 shows neutron in-plane scattering taken at





FIGURE 1. In-piane SANS from alamethicin in DLPC lipid bilayers at a high peptide-to-lipid ratio where the peptides self-assemble to form channels through the lipid bilayer as depicted in Fig. 2. In the upper panel, the channels were filled with either $D_2O(+)$, giving strong scattering contrast, or $H_2O(o)$. In the lower panel the lipid was deuterated, providing stronger scattering contrast with H_2O in the channels (+) compared with $D_2O(o)$. These data were taken at ANL.

Argonne National Laboratory (ANL) of alamethicin in protonated lipid bilayers (Fig. 1a) and in deuterated lipid bilayers (Fig. 1b), with D_2O or H_2O filling the pore channels. The peak in these data is due to the fairly regular pore spacing and is most pronounced when there is a strong contrast, either between D_2O and protonated lipid, or between H_2O and deuterated lipid. All four sets of data are consistent with the model shown in Fig. 2 once the differences in contrast are taken into account (solid curves). Thus we concluded



FIGURE 2. Model for the channels formed by octamers of alamethicin in DLPC bilayers that is consistent with the in-plane SANS data shown in Fig. 1.



FIGURE 3. Examples of SANS patterns from channel-forming peptides in lipid bilayers at various temperatures and stages of dehydration. The upper left panel is for magainin pores in fully hydrated fluid bilayers. As this and other peptide/lipid systems studied are dehydrated, the pores in adjacent bilayers become correlated and eventually crystallize.

that alamethic in DLPC bilayers forms octameric pores in the barrel-stave fashion.

Interestingly, the barrel-stave model is not the only possible pore formation. We have detected another type, called toroidal pores, in which the lipid monolayer bends continuously from one leaflet to another like the inside of a torus [1]. However, while the evidence for the pores is clear by the detection of the water (D_2O) channels through the lipid bilayers, the evidence for the pore structures is indirect. Thus the discovery of the crystalline phases is an important new development for the field of antimicrobial peptides.

We developed a method of off-plane scattering [2] to record the diffraction pattern on a SANS instrument that includes both the in-plane and out-of-plane momentum components. Figure 3 exhibits some typical diffraction patterns as recorded on the NG-3 30 m SANS instrument's detector by this method. The top left panel shows the diffraction pattern of magainin pores in fully hydrated fluid bilayers. When the sample was slightly dehydrated, the pattern changed to the top middle panel. Our analysis [2] showed that the positions of the magainin pores in each bilayer become correlated with the pores in adjacent bilayers, even though the bilayers are still in the fluid phase. The cause of this correlation was hypothesized to be due to the hydration force. Upon further dehydration or cooling, the pore arrangement crystallized into a lattice (the left panel of the middle row) having ABCABC stacking of hexagonally ordered planes.

The other crstalline patterns shown in Fig. 3 were from magainin and other peptides in various lipids at different hydration levels and temperatures. These crystalline phases offer a new way of studying peptide-lipid interactions that will help us to understand the molecular mechanisms of membrane active peptides in cell biology.

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SHEAR ORIENTATION OF VISCOELASTIC POLYMER-CLAY SOLUTIONS

Shear-induced structural changes in complex fluids of anisotropic species are very general phenomena, occurring in polymer solutions, liquid crystalline materials and block copolymer melts. The purpose of our work is to investigate the influence of shear on the structure of a highly viscoelastic, aqueous clay-polymer solution. Many structural models have been proposed for such solutions [1-3], but little is definitively known about mesoscopic properties or shear behavior. This information is important in the production of nanocomposite materials [4].

In our work, we use small-angle neutron scattering (SANS) to study a solution of the synthetic hectorite type clay, Laponite LRD (Laporte Industries Ltd.), and poly(ethylene-oxide) (PEO) ($M_w = 10^6$ g/mol). The results reported here are for a highly viscoelastic solution containing a mass fraction of 3 % LRD and 2 % PEO at room temperature. The clay particles produce transparent dispersions of disk shaped particles ca. 300 Å in diameter and ca. 10 Å thick [5,6]. The pH and ionic strength of the solutions were controlled by the addition of NaOH and NaCl, respectively.

Figure 1 shows the shear rate dependence of the birefringence of the clay-polymer solution. A distinct minimum in the birefringence is observed at a critical shear rate of approximately 40 s⁻¹. The source of the shear dependence of the birefringence is due to the alignment of the clay particles and the PEO. Previous work demonstrated that the sign of the birefringence of the clay particles oriented along a flow field is negative, therefore at low shear rates, the orientation of the clay dominates the birefringence. Above the critical shear rate, the birefringence due to the orientation of the polymer chains dominates.

A double logarithmic plot of viscosity, η , versus shear rate shows that the solution is shear-thinning over the entire range according to a power law with exponent m = -0.65. No signature of the critical shear rate is observed in the viscosity behavior.

The SANS shear cell utilized has been described previously [2]. It consists of a cylinder that rotates within an outer cylinder with the sample in the gap between them. The instrument was configured in both "radial" (incident beam parallel to the shear gradient along the cylinder diameter) and "tangential" (incident beam passing between the cylinders, parallel to the flow direction) geometries. Using 9 Å wavelength neutrons gives a Q range between 0.0027 Å⁻¹ and 0.0199 Å⁻¹. The primary contrast in the



FIGURE 1. Optical birefringence as a function of shear rate. The arrow indicates the shear rate where the minimum in the birefringence occurs.

SANS experiment used to detect the orientation of the clay platelets and polymer chains under shear is between D_2O and the other solution components.

The results obtained from the polymer-clay solutions in the "radial" and "tangential" beam configurations are summarized in Fig. 2. At low shear rates, a diffuse isotropic ring of SANS intensity is observed (Fig. 2a). The diffuse ring corresponds to an average spacing between platelets of 800 Å to 1100 Å. With increasing shear rate, the ring becomes more diffuse (Fig. 2b) and an anisotropic streak develops parallel to the vorticity axis of the flow field (the cylinder axis). If we neglect the main reflected beam which appears as a background streak in the gradient direction for tangential beam measurements (Fig. 2d), the anisotropic streak becomes the dominant feature in both scattering geometries with increasing shear rate After cessation of shear, the streaks relaxed to an isotropic state in less than 2 min.

To account for the SANS and birefringence results, our current understanding is that the polymer chains are in a dynamic adsorption/desorption equilibrium with the clay particles to form a network. The peak position in the quiescent scattering pattern in Fig. 2a is an indication of the mesh size of this network (≈ 1000 Å). A 2 % solution of only PEO, at the same pH, polymer and salt concentration showed no anisotropic SANS scattering at

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shear rates up to 100 s⁻¹. Similarly a 3 % aqueous clay solution shows no evidence of an anisotropic SANS pattern. Therefore, we can conclude that the anisotropic SANS pattern observed in the clay-polymer solutions is due to this coupling between clay platelets and polymer, allowing a higher orientation than either single component in solution can produce. From the birefringence data, the clay particles orient at low shear rates, while strong orientation of the PEO does not occur until the critical shear rate is exceeded. Since the clay platelets and the PEO chains are of comparable size (both about 300 Å), the lack of internal flexibility of the rigid clay particles makes them much easier to align than the flexible polymer chains.

According to SANS patterns from both beam configurations (Fig. 2) the shear flow results in an alignment of clay platelets orienting with their surface normals in the vorticity direction. One would expect the surface normals to orient along the gradient direc-

b) radial beam

a) radial beam



c) radial beam



FIGURE 2. SANS patterns of the clay-polymer solutions as a function of shear rate in the radial geometry (a-c) and tangential geometry (d) at shear rates of a) 0.5 s⁻¹, b) 20 s⁻¹, c) 90 s⁻¹, d) 90 s⁻¹.

tion of the flow field, however, the type of orientation observed in these clay-polymer solutions is also observed in some liquid crystalline lamellar phases, block copolymer solutions, and melts. The critical shear rate is the shear rate at which the rate of chain desorption is slower than the terminal relaxation time of the chain, hence chain extension is observed in the birefringence.

On cessation of shear, the stress on the network decays almost immediately, and the recovery of the isotropic structure is controlled by the relaxation of the stretched chains. As the chains retract, the coupling of the chains to the clay allows the platelets to randomize in orientation in the local viscous environment. The recovery from anisotropy is much faster than expected from simple Brownian motion of only the clay particles in a medium of the same viscosity as the clay-polymer solution exhibited macroscopically, and is indicative of the dynamic coupling of the polymer chains to the clay. Future work will compare the relative rates of the relaxation in the PEO and clay with the cooperative adsorption/ desorption kinetics which occur during deformation.

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UNIQUE DETERMINATION OF BIOMIMETIC MEMBRANE PROFILES BY NEUTRON REFLECTIVITY

ew biomimetic membrane materials, of fundamental importance in understanding such key biological processes as molecular recognition, conformational changes, and molecular selfassembly, can be characterized using neutron reflectometry. In particular, scattering length density (SLD) depth profiles along the normal to the surface of a model biological bilayer, which mimics the structure and function of a genuine cell membrane, can be deduced from specular neutron reflectivity data collected as a function of wavevector transfer Q. Specifically, this depth profile can be obtained by numerically fitting a computed to a measured reflectivity. The profile generating the best fitting reflectivity curve can then be compared to cross-sectional slices of the film's chemical composition predicted, for example, by molecular dynamics simulations [1]. However, the uniqueness of a profile obtained by conventional analysis of the film's reflectivity alone cannot be established definitively without additional information. In practice, significantly different SLD profiles have been shown to yield calculated reflectivity curves with essentially equivalent goodness-of-fit to measured data [2], as illustrated in Fig. 1.

The existence of multiple solutions, only one of which can be physical, is especially problematic in cases where a key additional piece of structural or compositional information is lacking as can happen in the investigation of these biological membrane systems.

Why this inherent uncertainty? The neutron specular reflection amplitude for a model SLD can be computed exactly from first principles; the square of its modulus gives the measurable reflectivity. It is firmly established, however, that the complex amplitude is necessary and sufficient for a unique solution of the inverse problem, that of recovering the SLD from reflection measurements. Unambiguous inversion requires both the magnitude and phase of reflection. Once these are known, practical methods [3] exist for extracting the desired SLD.

In fact, considerable efforts were made about a quarter century ago to solve the analogous "phase problem" in X-ray crystallography using known constraints on the scattering electron density [4] and by the technique of isomorphic substitution [5]. Variations of the latter approach have been applied to reflectivity, using a known reference layer in a composite film in place of atomic substitutions. These





FIGURE 1. Family of scattering length density profiles obtained by modelindependent fitting of the reflectivity data in the inset. The profile represented, by the blue dashed line is unphysical for this Ti/TiO film system yet generates a reflectivity curve that fits the data with essentially equivalent goodness-of-fit (all the reflectivity curves corresponding to the SLD's shown are plotted in the inset but are practically indistinguishable from one another).

FIGURE 2. Reflectivity curves for the thin film system depicted schematically in the inset, one for a Si fronting (red triangles), the other for Al_2O_3 (black circles). The curve in the lower part of the figure (blue squares) is the real part of the complex reflection amplitude for the films obtained from the reflectivity curves by the method described in the text.

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solution methods, however, were tied to the Born approximation, which generally is valid in crystal structure determination but which fails catastrophically at low Q (low glancing angles) in reflection from slab-shaped samples such as thin films. Exact inversion requires accurate knowledge of the reflection amplitude over the entire Q-range, especially at low Q.

In this decade the reflection phase problem has been exactly solved using a protocol of three reflectivity measurements on composite films consisting of the film of interest in intimate contact with each of three known reference layers [6, 7]. Subsequently, variations using only two measurements have been shown to partially solve the phase problem, an additional procedure being required to choose between two solution branches, only one of which is physical [8, 9]. In the past year [10], an exact solution has been found for a two measurement strategy in which the film surround, either the fronting (incident) or backing (transmitting) medium, is varied. This new approach is simpler to apply than reference layer methods and is adaptable to many experiments. Surround variation neutron



FIGURE 3. SLD profile (red line) resulting from a direct inversion of the Re r of Fig. 2 compared with that predicted by a molecular dynamics simulation (white line) as discussed in the text. The headgroup for the Self-Assembled-Monolayer (SAM) at the Au surface in the actual experiment was ethylene oxide and was not included in the simulation but, rather, modelled separately as part of the Au. Also, the Cr-Au layer used in the model happened to be 20 Å thicker than that actually measured in the experiment.

reflectometry has been successfully applied to the challenging type of biological membrane depth profiling described earlier.

In Fig. 2 are plotted a pair of neutron reflectivity curves measured for the layered film structure schematically depicted in the upper right inset, one with Si and the other with Al₂O₃ as the fronting medium. The lower part of Fig. 2 shows the real part of the complex reflection amplitude for the multilayer as extracted from the reflectivity data, according to the method described above, and which was subsequently used to perform the inversion to obtain the SLD shown in Fig. 3. For comparison, the SLD predicted by a molecular dynamics simulation is also shown in Fig. 3, in a slightly distorted version, corresponding to a truncated reflectivity data set, which indicates the spatial resolution of an SLD obtainable in practice. This latter SLD was obtained by inversion of the reflection amplitude computed for the exact model SLD, but using values only up to the same maximum Q value (0.3 Å⁻¹) over which the actual reflectivity data sets were collected. Overall, agreement between the experimentally determined profile and the theoretical prediction is remarkable, essentially limited only by the Q-range of the measurement. Surround variation neutron reflectivity thus makes it possible to measure complicated thin film structures without the ambiguity associated with curve fitting. The veridical SLD profile is obtained directly by a first principles inversion.

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MULTI-TECHNIQUE STUDIES OF ULTRATHIN SIO₂ FILMS

urrent gate dielectrics in silicon based devices are only a few nm thick. Optical techniques such as ellipsometry are used to monitor film thicknesses and optical properties in production. However, for the current integrated circuit (IC) generation the accuracy of ellipsometry degrades because parameters such as thickness and index of refraction (which reflects the composition) become strongly correlated. Thus, it is difficult to unambiguously determine these parameters simultaneously, and the accuracy of ellipsometry would benefit from an independent calibration. In reflectometry techniques, on the other hand, these parameters are nearly decoupled. The thickness of a layer is approximately inversely proportional to the oscillation period of the reflected intensity, whereas the differences in scattering length density SLD (also an indicator of composition) between the layers is related to the amplitude of the oscillations. Neutron reflectometry (NR) is better suited than X-ray reflectometry (XR) for the study of the SiO₂/Si system because there is a relatively large contrast (or difference in SLD) between the scattering length densities of the two materials: 65 %, vs. 7.6 % for X-rays.

Consider as an example a sample with a nominally 10 nm thick thermal oxide film on silicon. This moderate thickness was chosen to increase our confidence in the results of the various



FIGURE 1. Comparison of spectroscopic ellipsometry experimental data, Ψ and Δ , to the fits (solid line) for the clean and surface contaminated sample. The inset shows < ε_1 > and < ε_2 > as a function of depth determined by the fit.

characterization methods, while remaining thin enough that the results are relevant to film of technological interest. Figure 1 shows spectroscopic ellipsometry (SE) data and corresponding best fits for the sample with surface contamination and after an organic cleansing. Nominally, the only change is a decrease in the thickness of the contamination layer [1].

In XR data (Fig. 2) two oscillation periods are observed for the contaminated sample. The high frequency oscillation corresponds to the SiO_2 film, whereas the low frequency modulation is due to the thinner contamination layer (which is not present after cleaning, indicating removal of the contamination.)

The NR measurements (Fig. 3) were done in a vacuum to reduce the air scattering background. This allowed us to achieve a very large range in reflectivity, over 10^8 , which is among the best examples in NR measurements to date. A slightly thinner contamination layer in NR is consistent with the fact that the XR was done in air, during which the contamination was growing. This was confirmed by changes in XR scans immediately following those in Fig. 2.

The average of the 5 measurements of the SiO₂ film thickness was 10.27 ± 0.13 nm. The excellent agreement among the results for the three different techniques increases our confidence in the



FIGURE 2. X-ray reflectivity and best fits for the clean and surface contaminated sample. The inset shows the scattering length density profile determined by the fits.

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FIGURE 3. Neutron Reflectivity data and best fit for a sample with surface contamination. The inset is the scattering length density profile determined by the fit.

parameters extracted via these models. Thus the XR and particularly the NR corroborate the correct analysis required in SE (which is the technique most practical in monitoring production).

To further investigate the applicability of these techniques to thinner SiO, layers, we simulated the SE and the XR and NR curves for 6 nm, 4 nm, and 2 nm thick layers of SiO₂. For a realistic and consistent set of roughness parameters in the models, we used the average values obtained from the actual measurements previously discussed. The SE simulation, Fig. 4a, shows distinct differences in both the magnitude and shape of Δ among the three thicknesses shown. In models of XR, shown in Fig. 4b, only very weak oscillations are seen for even the thickest of the SiO, layers because of the low contrast between SiO₂ and Si. However, in NR, for SiO₂ layers as thin as 2 nm strong oscillations are clearly seen above the 10⁻⁸ lower limit, demonstrated in Fig. 3. Therefore both NR and SE are well suited for the study of SiO, films as thin as 2 nm. Encouraged by these models, we obtained NR data for a thinner, 2.4 nm, sample. While these data are not yet fit to a model curve, we note that both the reflected intensities and oscillation amplitude are similar to those of the 2.0 nm model, indicating similar interface widths.





We have shown that three different techniques can offer complimentary information on the structure of thin SiO_2 films on Si. All offer a significant degree of sub-monolayer thickness sensitivity, although in NR there is a much higher contrast between SiO_2 and Si than in XR.

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CERTIFICATION OF AN ION-IMPLANTED ARSENIC IN SILICON STANDARD REFERENCE MATERIAL

he secondary-ion mass spectrometry (SIMS) community in the United States recently undertook a round-robin study to calibrate the implanted dose of arsenic in silicon by consensus. Variations in dose determination among laboratories were as high as 30 %, reflecting primarily the errors of the respective in-house standards. By contrast, in an international round-robin exercise sponsored by the International Standards Organization (ISO) the U.S. laboratories achieved a relative standard deviation of 4 % for nine independent determinations of the boron content in an unknown boron-doped silicon sample. This level of agreement was only possible because all the laboratories used SRM 2137 Boron Implant in Silicon as a common reference material. These results demonstrate the need for a common arsenic reference material to improve interlaboratory reproducibility. Furthermore, SEMATECH (a consortium of semiconductor manufacturers) recently listed SRM implants of phosphorus and arsenic in silicon as high priority industrial needs. Consequently, a Standard Reference Material (SRM 2134 Ion-Implanted Arsenic in Silicon) was produced at NIST, using a wafer from the SIMS intercomparison. The material for this SRM was provided by a major ion implanter manufacturer who supplied three 200 mm diameter wafers that had been implanted with arsenic at an energy of 100 keV (in the same batch). One of the wafers was diced and distributed to 12 participating laboratories for the arsenic round-robin study described above. Each of the remaining wafers could provide 221 SRM units, enough for an estimated 10 year supply. One of the remaining wafers was therefore diced into 1 cm x 1 cm pieces with a wafer saw for use as SRM 2134. Because of the specificity and matrix independence of instrumental neutron activation analysis (INAA), this technique was chosen as the primary method for certification of the arsenic implanted dose.

Ten analytical specimens of silicon were selected from among the 221 pieces cut from the wafer to determine arsenic content and test for homogeneity. Two blank chips of silicon were analyzed in parallel. Pre-irradiation handling was done on a class 100 clean bench. In a Teflon TFE beaker, the specimens and blanks were rinsed together in ultrapure (UP) water, rinsed with 95 % ethanol, cleaned ultrasonically for one minute in ethanol, and rinsedtwice with UP water. The samples and blanks were then agitated for one minute in UP water, blotted on Whatman 41 filter paper, and



FIGURE 1. Region of the gamma-ray spectrum for analysis of As in SRM 2134. Limits of peak and baseline areas are indicated by vertical lines.

air-dried. Each was weighed and heat-sealed in a polyethylene bag that had been cleaned in high purity nitric acid.

A dilute solution standard was prepared by gravimetrically diluting a 10 mg/g solution of As_2O_3 in dilute ammonia to about 2 mg/kg. Another solution was prepared similarly from SRM 3103a Arsenic Spectrometric Solution. Aliquants of both solutions were weighed from a polyethylene pipette onto 1-cm disks of acid-leached Whatman 41 filter paper and dried to produce standards with about 200 ng arsenic each. Two standards from each solution were irradiated with the samples. A blank filter paper was shown in a separate experiment to contain no significant arsenic (<< 0.01 % of the amount in the standards).

Samples, standards, and blanks were stacked in the irradiation container in the following sequence: standard, blank, five samples, two standards, five samples, blank, and standard. The stack was irradiated for four hours in the RT-1 pneumatic tube of the NIST research reactor, at a thermal fluence rate of 7.7×10^{13} cm⁻²s⁻¹. The container was inverted halfway through the irradiation to equalize to the first order for the axial fluence rate gradient along the container.

After irradiation the silicon samples were cleaned ultrasonically for one minute in de-ionized water, blotted dry, and heat-sealed in clean conventional polyethylene bags for counting.
Gamma-ray activity was assayed at 10 cm from a germanium detector with a 1.71 keV resolution and 40 % relative efficiency at 1333 keV. Each sample and standard was counted at least twice, after the 2.62 h ³¹Si matrix activity had substantially decayed. The 559 + 563 keV doublet of ⁷⁶As in each spectrum was integrated with the SUM computer program [1]. See Fig. 1. No evidence indicating significant heterogeneity among samples could be seen when the observed sample-to-sample precision was compared to what was expected from counting statistics combined with other sources of analytical variability.

NIST currently certifies elemental concentrations in SRMs using one of three modes: (1) a primary method at NIST with confirmation by other method(s); (2) two independent critically evaluated methods at NIST, and (3) one method at NIST and different methods by outside collaborating laboratories [2].

Table 1. Individual Uncertainty Components for Determination of Arsenic in SRM 2134.

Source of Uncertainty	Individual Uncert. (1s) %	
Basis Measure - Sample Area / Micrometer Accura	cy 0.012	
Basis Measure - Sample Area / Precision $(n = 26)^{1}$	0.0089	
Concentrations of Comparators (Standards)	0.106	
Mass Determination - Comparators	0.041	
Isotopic Variability	0	
Blank and Blank Correction $(n = 10^2)^1$	0.013	
Irradiation Geometry Differences	0.039	
Neutron Self - Shielding/Scattering Differences	0.024	
Timing	0	
Irradiation Interferences	Negligible	
Measurement Replication $(n = 9)^1$	0.081	
Counting Statistics - Standards (n = 10 ⁵) ¹	0.073	
Counting Geometry Differences	0.009	
Pulse-Pileup	0.066	
Dead-Time Effects (inadequacy of LT extension)	Negligible	
Decay Timing Effects (Half life related)	0.053	
Gamma-Ray Self Shielding	0.004	
Gamma-Ray Interferences	0.00004	
Peak Integration Method	0.033	
Overall	0.189	

Certification using a primary method is only possible when all potentially significant sources of uncertainty have been evaluated explicitly for the application of the method and the matrix under investigation. In addition, confirmation of measurements by a primary NIST method may be accomplished by one or more of the following: determination of certified constituents in other SRM(s) or CRM(s) of similar matrix and constituent concentration range; a second NIST technique with appropriate controls; or results of measurements from selected outside collaborating laboratories with appropriate experience.

In order to certify the arsenic concentration in SRM 2134 using INAA as a primary method it is therefore necessary to evaluate all significant sources of uncertainty explicitly. For this set of measurements, we considered sources of uncertainty greater than 0.01 % relative to be significant. The results of a complete evaluation of all sources of uncertainty are listed in Table 1. This evaluation yielded an expanded relative uncertainty of 0.38 % (as defined by ISO and NIST) and gives an approximate level of confidence of 95 %. The arsenic concentration observed was 91.20 ng/cm² \pm 0.35 ng/cm².

In conclusion, we have successfully applied INAA as a primary method for the certification of this new SRM. The observed relative expanded uncertainty of 0.38 % is considerably smaller than the 1 % value desired by the semiconductor industry. This new SRM should greatly enhance the U.S. semiconductor industry's ability to achieve accurate and reproducible analytical results for this key dopant in silicon.

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"n" indicates degrees of freedom which are listed for Type A sources of uncertainty.

THE ROTATIONAL DYNAMICS OF H₂ IN POROUS VYCOR GLASS

orous materials play an increasingly important role in the exploration of fundamental scientific issues related to diverse technological applications including adhesion, lubrication, tribology and the engineering of materials. Surface interactions and finite size effects both play a key role in the qualitative modification of the properties of the materials contained within the porous host. Due to its light mass and weak electronic interactions, molecular hydrogen is an ideal system to probe the effects of surface interactions. H, is well described as a quantum rigid rotor with discrete energy levels labeled by the rotational quantum number, J, and energies given by $E_{I} = BJ(J + 1)$ where B is the rotational constant which is equal to 7.35 meV for H₂. These energy levels can be studied by using neutrons to stimulate the J = 1 to J = 0 transition of the H, molecule, a process that is normally doubly forbidden due to the quantum statistics obeyed by H₂. When neutron stimulated conversion occurs, the neutron gains an amount of energy equal to the rotational energy of the molecule. This results in an extremely clean signal since all other processes are frozen out at the low temperatures of these measurements.

Studies of the rotational levels of H_2 adsorbed in Vy cor, a commercially available porous glass, were carried out for pore fillings ranging from 0.10 (corresponding to less than a single monolayer on the pore surface) to 0.92 (corresponding to nearly full pores). As can be seen in Fig. 1, at low filling fractions only a single broad peak at 10 meV is present which can be attributed to the scattering from an adsorbed layer of H_2 strongly bound to the pore surface. The appearance of scattering at 14 meV, corresponding to the free molecule transition, for fillings above 0.45 is associated with the appearance of "bulk"-like material in the pore center. The clear separation of the scattering from molecules adsorbed on the walls and in the pore center suggests that these are quite distinct states which we refer to as the bound and bulk-like states, respectively.

The shift in the energy of the rotational transition at low fillings can be directly related to the interaction of the molecules with the surface [2]. Figure 2 shows the shift of the J = 1 to J = 0 transition as a function of the rotational energy barrier for the molecule. From Fig. 2 one finds that the observed rotational energy of 10 meV corresponds to an orientational potential with a barrier height of $V_B/B_H = 2.7$ ($V_B = 19.8$ meV). The magnitude



FIGURE 1. The top panel shows a contour plot of the inelastic scattering from H_2 in Vycor at various pore fillings at 6 K. The bottom panel is a cut through the contour plot at f = 0.65 (indicated by the line).

of this potential is quite large, comparable to that seen in alpha alumina, a catalyst used for ortho- H_2 enrichment [3], and on leached glass [4].

Careful inspection of the data reveals that the peak due to the bound state actually shifts from 9.1 meV to 10.1 meV between filling fractions, f, of 0.20 and 0.27. Thus for f between 0.27 and 0.45, the bound state may be separated into a layer in direct contact with the pore wall and a layer that feels a weaker orientational potential. The scattering from these two layers can be separately determined assuming the intensity from the first layer varies linearly with filling.

The scattering from the second surface layer can be obtained from the difference of the monolayer and bilayer spectra. The scattering from the second layer is centered at E = 11.3 meV, compared D. W. Brown Manual Lujan Jr. Neutron Scattering Center Los Alamos National Laboratory Los Alamos, NM 87545 P. E. Sokol Department of Physics The Pennsylvania State University University Park, PA 16802 **S. A. FitzGerald** Department of Physics Oberlin College Oberlin, OH 44074



FIGURE 2. Shift of the rotational energy levels of H₂ as a function of barrier height.

to the first layer which is centered at 9.1 meV, indicating that the second layer interacts less strongly with the wall.

The width of the peak we associate with the bulk-like molecules is due entirely to instrumental resolution. This is consistent with the view of these molecules as being free rotors with a single well defined value for B. On the other hand, the width of the peak which we attribute to the bound layer is much broader than the instrumental resolution. A single well defined orientational potential would yield a shift in the peak location, as observed, but no additional broadening, since the energy levels would still be well defined. Thus, we attribute this additional width to a distribution of orientational potentials which can also be directly extracted using the model of White and Lassettre [2] (Fig. 2). After correcting for instrumental effects, the strength of the scattering at a given energy is directly proportional to the number of molecules which feel the corresponding orientational potential. Thus, plotting the scattering as a function of V/B_{H} , directly yields the distribution of orientational potentials shown in Fig. 3. As can be seen, the



FIGURE 3. Distribution of potential barriers felt by the molecules in the first (red) and second (blue) layers.

orientational potential for the first layer is broad, asymmetric, and centered at $V_B = 3.5B_H$, whereas the potential of the second layer is relatively narrow and centered at $V_B = 2.0B_H$, indicating a weaker interaction with the surface.

The data allow us to draw the following picture of the adsorption of molecular hydrogen on a porous glass surface. The first monolayer is bound tightly to the rough pore surface. The rough surface prevents free rotation of the molecule, altering the rotational energy states. The second layer perceives the surface roughness smoothed by the presence of the first layer, and accordingly, the rotational transitions are affected to a lesser degree. Subsequent to the completion of the second monolayer, the H₂ molecules sense no significant orientational interaction from the glass surface. This is in agreement with the picture presented by Katsaros *et al.* [5], in which surface roughness is created by dangling bonds on the pore surface.

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FIRST-PRINCIPLES COMPUTATIONAL AND NEUTRON SCATTERING STUDY OF PROTONIC CONDUCTORS

uel cells, which produce electricity via hydrogen oxidation to water, have emerged in the last decade as one of the key technologies for meeting the world's energy needs well into the 21st century. Solid-oxide fuel cells (SOFCs) are the most promising among the many different types of fuel cells being developed. However, the required high operating temperature of an SOFC places stringent requirements on its component materials. Potential candidates to replace the oxygen-conducting electrolyte used in current SOFCs include the perovskite-based high-temperature protonic conductors (HTPCs), since switching from anionic to protonic conduction can lower the practical operating temperature from 1300 K to around 1000 K.

We undertook neutron vibrational spectroscopy (NVS) measurements of various HTPCs (namely, SrCeO₃ and SrZrO₃ aliovalently doped with different rare earth cations) in order to probe the bonding potentials experienced by the residual protons incorporated as OH⁻ species via steaming. At high temperatures, these protons migrate from site to site, giving rise to protonic conduction. The NVS measurements revealed the existence of dopant-related perturbations to the bending-mode energies of OH⁻ in SrCe_{0.95}M_{0.05}H_xO₃₋₈ (M = Sc, Ho, and Nd), confirming the trapping effects of the dopants. The bending-mode energy could be correlated with the size of the dopant cation, generally increasing for smaller cations. NVS measurements of $\text{SrZr}_{0.95}\text{M}_{0.05}\text{H}_{x}\text{O}_{3-\delta}$ (M = Sc, Y, and Nd) indicated differences in the OH⁻ bending-mode energies between the cerates and zirconates. These differences reflect changes in the lattice potential experienced by the protons, which ultimately effects the proton jump rates and therefore the performance of these materials for use in fuel cells.

An understanding of the dynamics of the undoped perovskites is the first step towards a comprehensive picture of the protonated doped materials. To explore this in more detail, we performed *ab initio* total-energy calculations using the CAmbridge Serial Total Energy Package (CASTEP). We calculated the Q = 0 phonons in the primitive unit cell (i.e., $Zr_4Sr_4O_{12}$) of undoped strontium zirconate and compared with the experimental density of states (DOS) at room temperature, as shown in Fig. 1. While at low energies, the calculated modes are almost at the same energies as observed modes, at high energies, we overestimate the energies of the modes by about 10 %. However there is still a one-to-one correspondence between the main experimental and calculated spectral



FIGURE 1. LEFT: A view of the structure of undoped $SrZrO_3$ in the [001] direction. RIGHT: Vibrational spectra for $SrZrO_3$ measured by neutron time-of-flight spectroscopy. The space group and the lattice parameters (in Å) are also shown. Numbers in parentheses are the theoretical values. The lower solid line is the DOS of the Q = 0 phonons calculated from first principles.

features, allowing us to identify the modes observed by our NVS measurements.

In order to model the dynamics of protons trapped in Sc-doped SrZrO₃, we replaced one of the Zr atoms in the $\sqrt{2} \times \sqrt{2} \times 1$ supercell by (Sc + H), which yields a cell formula Sr₈Zr₇ScHO₂₄ (See Fig. 2). We performed calculations for protons at either the "undoped" (U) or "doped" (D) sites. Even though the MO₆ octahedra are quite rigid, the distortions due to the presence of the proton at these sites are quite large.

These distortions are also reflected by the vibrational spectrum of the proton. The two tangential OH- bending modes depend strongly on the proton siting. At the Sc site, the lowest tangential mode is found at 122.9 meV. However at the Zr site, the mode is much softer at 88.5 meV. Interestingly, we observe new features in our NVS spectra at these energies upon proton addition. To further investigate this effect, we performed "embedded cluster" calculations in which the vibrational spectrum of the H-MO₆ cluster is calculated while all other atoms are kept at their equilibrium positions. The similarity between experiment and the calculated spectrum from H-ZrO₆ + H-ScO₆ clusters suggests that the mode observed near 120 meV is due to protons trapped at the Sc sites





FIGURE 2. LEFT: Optimized structures when the proton is trapped at the undoped site (top) and at the doped site (bottom). MIDDLE: Comparison of the neutron vibrational spectrum of SrCe_{1.2}Sc₁H_.O₃ (top) and SrZr_{1.2}Sc₁H_.O₃ (calculated). Dashed and dotted lines shown at the bottom are the contributions from the H-MO₅ clusters, where M = Zr (undoped site) and Sc (doped site), respectively. RIGHT: Potential energy of the crystal as the proton migrates from the undoped site (U) to the doped site (D).

while some portion of the peak near 80 meV is due to protons at the undoped sites. Thus NVS can be used to determine the hydrogen occupancy of the various sites.

For a better understanding of the protonic conduction in these materials, we calculated the total energy of the system as the proton migrates from Zr to Sc sites. We found that the doped site has a much lower energy (-1.13 eV) than the undoped site. The two sites are separated by an energy barrier of 1.5 eV. Hence, we expect that most of the protons are trapped at the dopant site. This is primarily because the proton prefers to be closer to the Sc cation, which has a charge of +3 compared to +4 for Zr. Yet, one also has to consider steric effects; i.e., if the dopant cation has a larger radius, then the proton may prefer to occupy the Zr site, despite the larger Coulomb interaction. Thus these calculations indicate that the protonic transport is sensitive to the competition between short-range repulsive and Coulomb interactions, and suggest that the use of a large dopant cation is one important step in developing a HTPC with increased protonic conductivity. We are currently testing the usefulness of the calculated proton potentials for predicting the protonic diffusional motions observed experimentally via quasielastic scattering measurements.



MAGNETIC TRAPPING OF ULTRACOLD NEUTRONS

The demonstration of three-dimensional magnetic trapping of neutrons was performed this past year at NIST [1]. The techniques developed should lead to improved precision in the measurement of the neutron beta-decay lifetime, thereby expanding our knowledge of the weak nuclear force and our understanding of the creation of matter during the Big Bang.

Magnetic traps are formed by creating a magnetic field minimum in free space. The confining potential depth is determined by the magnetic moment of the neutron and the difference between the magnitude of the field at the edge of the trap and at the minimum. A neutron in a low-field-seeking state (one with its magnetic moment anti-aligned with the local magnetic field vector) feels a force pushing it towards the trap minimum and will remain confined within the trapping region.

In order to load a neutron into a static conservative trap, its energy must be lowered while it is in the potential well. We rely on a loading technique that employs the "superthermal process" [2]. Superfluid helium fills the trapping region and serves as a neutron scattering medium. A neutron with kinetic energy near 11 K (where the free neutron and Landau-Feynman dispersion curves cross) that passes through the helium can lose nearly all of its energy in a single scattering event. Neutrons that scatter to energies less than the trap depth (1 mK) and in the appropriate spin state are trapped. Neutrons in this energy range are called ultracold neutrons (UCN).

Isotopically pure superfluid helium is contained in a tube located inside the superconducting magnet and centered axially within its trapping field (see Fig. 1). The superconducting magnet, trapping region, and other key parts of the apparatus reside within a cryogenic dewar. The incident neutron beam is collimated, passes through the trapping region, and is absorbed by the beam stop. As the beam traverses the trapping region, about 1 % of the neutrons scatter in the helium. Some of these neutrons are trapped and the remainder are absorbed by shielding materials that surround the helium. Low-field-seeking UCN are trapped and remain in the trapping region until they decay.



FIGURE 1. Half-section view of the neutron trapping apparatus. A beam of cold neutrons enters from the left, is collimated, passes through the trapping region and is absorbed at the rear. Scattered neutrons (yellow) in the low-field-seeking spin state and with energy below the trap depth are magnetically confined. Electrons from neutron beta-decay create EUV scintillations in the superfluid helium which are wavelength shifted to the visible and transported to the photomultiplier (to the right).

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The trapped neutrons are detected when they decay. When a trapped neutron decays (into an electron, proton and anti-neutrino), the resulting high-energy electron travels through the helium leaving a trail of ionized helium atoms. These ions quickly combine with neutral helium atoms to form metastable diatomic molecules. Most of these molecules decay within 10 ns, emitting a pulse of light in the extreme ultraviolet (EUV), $\lambda \approx 70$ nm to 90 nm. This pulse of scintillation light is the signal of a neutron decaying in the trap.

In an experimental run, the cold neutron beam is allowed to pass through the trapping region, after which the beam is blocked and pulses of light are counted. A background signal, with both constant and time-varying components, obscures the trapped neutron signal. These backgrounds are subtracted by collecting data



FIGURE 2. Counting rate as a function of time after the neutron beam is turned off (pooled background subtracted data). (a) Trapping data set I, $N_1 = 560 \pm 160$, (b) Trapping data set II, $N_1 = 240 \pm 65$, (c) Combined trapping signal, $\tau = 750^{+200}_{-200}$ s, (d) Combined ³He data, $N_{au} = 53 \pm 63$.

where the magnetic field is on during the initial loading phase, so that UCN are confined by the trap and subtracting data where the magnetic field is off initially, so that no neutrons are trapped. Equal numbers of each data set were taken, pooled and subtracted to give the background subtracted data.

Two sets of background subtracted data were collected: set I with a trap depth of 0.76 mK (Fig. 2a) and set II with a lower trap depth of 0.50 mK (Fig. 2b) due to problems with the magnet. For each set, the pooled background subtracted data are modeled to extract the amplitude and lifetime of the decaying neutron signal. The best fit values for the initial counting rates combined with the measured detection efficiency gives $N_I = 560 \pm 160$ and $N_{II} = 240 \pm 65$. Calculations using the known beam flux, trap geometry and the theory of the superthermal process predict $N_I = 480 \pm 100$ and $N_{II} = 255 \pm 50$, in good agreement with the measured values. The best fit value for the trap lifetime, $\tau = 750^{+330}_{-200}$ s, is consistent with the presently accepted value of the neutron beta-decay lifetime of 886.7 \pm 1.9 s [3].

To verify that our signal is in fact due to trapped neutrons, we doped the isotopically pure ⁴He with ³He at a concentration of 2 x 10^{-7} ³He/⁴He. This amount of ³He absorbs the trapped neutrons in less than 1 s without affecting anything else in the experiment (less than 1 % of the cold neutron beam is absorbed by ³He). The data (Fig. 2d) is modeled with the lifetime fixed at \equiv 750 s, yielding N_{3He} = 53 ± 63, consistent with zero. This confirms that our signal is due to trapped neutrons.

Magnetic trapping of neutrons is a new technique that should allow a higher precision measurement of the neutron lifetime, and offers the prospect of precision much greater than the current limit of one part in 10³. In order to realize the potential of this technique, we are in the process of improving our apparatus in many ways, including increasing the size and depth of the trap. We expect to make a competitive neutron lifetime measurement in the near future.

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CHARGE DISPROPORTIONATION AND MAGNETIC ORDERING IN CaFeO₃

Transition metal oxides adopting the perovskite crystal structure (or one of its relatives) have occupied a place in the scientific limelight for nearly fifty years now. Beginning with the discovery of ferroelectricity in BaTiO₃, following World War II, the dielectric properties of perovskites have been extensively studied over this entire period. Today perovskite dielectrics, due to their widespread usage in telecommunication, are still extensively studied. The discovery of superconductivity first in doped BaBiO₃, and later in the cuprates, such as YBa₂Cu₃O_{7-X}, triggered an unprecedented avalanche of scientific activity in the late 1980's and early 1990's. More recently, phenomena such as collosal magnetoresitance (CMR), charge, orbital and spin ordering, and phase separation in the manganate perovskites, $(Ln_{1-x}A_x)MnO_3$ (Ln = lanthanide ion, A = alkaline earth ion), have captured the imagination of the condensed matter scientific community.

Ultimately the electronic and magnetic properties of a material depend upon the behavior of the outermost or valence electrons. In a first row transition metal oxide the valence electrons are shared, though not equally, between the 3d orbitals of the transition metal ion and the 2p orbitals of oxygen. In compounds with the perovskite structure the oxygen atoms are arranged in an octahedral geometry about the transitional metal ion; each oxygen is then shared by two transition metal ions to form a three dimensional network of corner sharing MO₆ octahedra. The octahedral coordination removes the energetic degeneracy of the 3d orbitals, forming the familiar triply degenerate t_{2g} and doubly degenerate e_{g} set of orbitals. The e_{g} orbitals point directly at the oxygen ligands to form a strongly antibonding σ^* bond. In contrast, the $t_{_{2g}}$ orbitals have a smaller overlap with the oxygen 2p orbitals, which leads to a weakly antibonding π^* band. If this covalent bonding interaction between the transition metal and oxygen is weak, the valence electrons are localized and a magnetic insulator is typically observed. If we increase the strength of the interaction sufficiently, either by increasing the electronegativity of the transition metal ion or the spatial overlap of the metal 3d and oxygen 2p orbitals, partial delocalization of the valence electrons can occur. Frequently, this leads to metallic conductivity. In a number of transition metal oxides the metal-oxygen interaction strength is such that delocalized



FIGURE 1. The low temperature structure of CaFeO₃. The green spheres depict the calcium ions. The other shaded objects represent iron centered octahedra, where an oxygen atom can be found at each vertex and an iron atom at the center of each octahedron. The purple and blue objects represent Fe³⁺ centered and Fe⁵⁺ centered octahedra respectively.

electrons and metallic conductivity are seen at higher temperatures but give way to a magnetically ordered, insulating ground state upon cooling.

LaMnO₃, SrFeO₃ and CaFeO₃ are isolectronic; they each have an electronic configuration of $t_{2g}^{3} e_{g}^{1}$. According to the Jahn-Teller theorem, the presence of a single localized electron in the doubly degenerate e_{g} set of orbitals is not a stable situation. LaMnO₃ responds to this instability by undergoing a cooperative Jahn-Teller distortion of the MnO₆ octahedra, producing two long and four short Mn-O bonds, Thereby, removing the degeneracy of the e_{g} orbitals. SrFeO₃ takes a different approach, by delocalizing its e_{g} electrons to form a σ^{*} band. The contrasting behavior of these two materials can be understood in terms of the strength of the metal-oxygen Patrick M. Woodward Department of Chemistry Ohio State University Columbus, OH 43210 David E. Cox and Evagalia Moshopoulou Department of Physics Brookhaven National Laboratory Upton, NY 11973 Arthur W. Sleight Department of Chemistry Oregon State University Corvallis, OR 97331-4003

interaction. Contrasting LaMnO₂ to SrFeO₂, the oxidation state of the transition metal increases from +3 to +4, which leads to an increase in the metal-oxygen interaction strength, increasing the width of the σ^* band and stabilizing a metallic ground state. Since they both contain Fe⁴⁺, one might expect that CaFeO₂ and SrFeO₂ would behave very much alike. However, Ca2+ is smaller than Sr2+, which causes a tilting of the FeO₆ octahedra to satisfy the valence requirements of calcium. This seemingly subtle distortion has several important consequences. The Fe-O-Fe bond is distorted away from the linear geometry observed in SrFeO₂ (the Fe-O-Fe angle is 158° in CaFeO₃). This reduces the spatial overlap of the Fe e_{a} and O 2p orbitals, and the width of the σ^* band decreases. The reduction in bandwidth triggers an electron localization that occurs just below room temperature (290 K). Once electron localization occurs, a cooperative Jahn-Teller distortion to the LaMnO₃ crystal structure is expected.

However, CaFeO₂ refuses to conform with expectations. Instead CaFeO₃ undergoes a charge disproportionation (CD), 2Fe⁴⁺ $(t_{\sigma}^{3}e_{\sigma}^{1}) \rightarrow Fe^{3+} (t_{\sigma}^{3}e_{\sigma}^{2}) + Fe^{5+} (t_{\sigma}^{3}e_{\sigma}^{0})$. Evidence for a CD in CaFeO₃ was first proposed over 20 years ago, based on Mössbauer studies [1]. However, crystallographic confirmation of this rare phenomenon has proven elusive. Through the combined use of synchrotron x-ray (X7a-NSLS) and neutron powder diffraction (BT-1 at NCNR), we have elucidated the crystal structure of CaFeO₃ in its CD state for the first time. The resulting structure (Fig. 1) clearly shows the presence of two chemically and crystallographically distinct Fe sites. The average Fe-O bond length about the "Fe⁵⁺" site is 1.872(6) Å, while the same distance about the "Fe³⁺" site is 1.974(6) Å. The ordered arrangement of Fe³⁺/Fe⁵⁺ is such that each Fe³⁺ is surrounded by six Fe⁵⁺ ions, and vice versa (NaCl or G-type ordering), optimizing the Coulomb stabilization of the CD state. Alternately, the CD process can be viewed as the condensation of a breathing phonon mode.

In addition to electronic properties, there has long been both technological and fundamental scientific interest in the magnetic properties and interactions in perovskites containing transition metal ions. Goodenough's study of the various antiferromagnetic structures observed in the $(La_{1,x}Ca_x)MnO_3$ system was vital in the development of the Goodenough-Kanamori rules of superexchange [2]. These rules correctly predict an A-type antiferromagnetic structure for LaMnO₃. Using the same rules, we would expect CaFeO₃ to have a simple ferromagnetic ground state. Perhaps not surprisingly, our investigation showed that again CaFeO₃ defies expectations. Low temperature neutron powder diffraction data reveals instead an incommensurate antiferromagnetic ground state ($T_N \approx 120$ K). Analysis of the data shows the magnetic structure to be either a screw spiral or a sinusoidal amplitude-modulated structure. In either case, it would appear that a long range AFM interaction (probably between next-neighbors) is present in addition to the nearest neighbor FM superexchange interaction.

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BOND VALENCE ANALYSIS OF RUTHENATES

Bond valence analysis allows us to evaluate, for any compound whose bonding scheme is known or assumed, the bond distances that the atoms would form in an ideal structure in which all atomic valences are exactly balanced. In a significant number of cases this information is sufficient to make accurate predictions about the real crystal structure of the compound.

The bond valence method is based on two concepts that can be stated in the following way.

(*i*) A bond valence $v_{ij} = v_{ji}$ is assigned to a bond between atoms *i* and *j*, of valences V(i) and V(j), so that

$$\sum_{i=1}^{n(i)} v_{ij} = V(i) \quad \text{and} \quad \sum_{i=1}^{n(j)} v_{ji} = V(j) \tag{1}$$

where n(i) and n(j) are the number of atoms in the coordination spheres of *i* and *j*, respectively. This principle of local valence balance is a generalization of Pauling's principle of local charge balance in ionic crystals, and is known as the *valence sum rule* [1].

(*ii*) The sum of the bond valences around any loop in the structure, taken with alternating signs, is equal to zero

$$\sum_{loop} v_{ij} = 0.$$
 (2)

Equation (2) expresses the mathematical conditions that result in the most regular distribution of the valences among the bonds in a structure and is known as the *equal valence rule* [1]. The system of equations (1) and (2) allows us to evaluate the valences of all the individual bonds if we know how the atoms are bonded together in a structure [2]. The description of atomic bonding in terms of bond valences is useful because the length d_{ij} of a bond between atoms *i* and *j* is a function only of the bond valence v_{ij} . The relationship between these two quantities is expressed by the empirical formula

$$d_{ii} = R_{ii} - 0.37 \ln v_{ii} \tag{3}$$

where the bond valence parameter R_{ij} depends on the nature and the oxidation states of atoms *i* and *j* forming the bond, and expresses the length of a bond of unit valence. Values of R_{ij} can be evaluated from the bond distances of known structures and are tabulated for most chemical species [3,4]. By means of equations (1-3) we may evaluate the expected bond lengths for any known atomic configu-



FIGURE 1. Schematic representation of (a) the 4-layer structure of TRuO₃ (T = 0.875 Ba + 0.125 Sr); and (b) the 9-layer structure of BaRuO₃. For clarity only the Ru and O atoms are shown in the figure. The symbols *c* and *f* indicate the layers on which the RuO₆ octahedra share corners and faces, respectively.

ration. These distances satisfy exactly the valence requirements of the atoms and, in general, differ significantly from those determined experimentally. The discrepancies are in some cases due to the electronic behavior of particular cations, which may cause distortions not accounted for by the bond valence model (for example, lone pair distortions around cations such as Bi3+ and Pb2+, or Jahn-Teller distortions around Mn³⁺ and Cu²⁺). In the majority of cases, however, the bond lengths calculated with equations (1-3) are incommensurate under the constraints imposed by the crystal geometry, and have to be stretched or compressed in order to fit them into a particular configuration. Since these changes introduce strains into the structure, the process of adapting the theoretical model to the requirements of a space group symmetry must be carried out in such a way that the violations of the bond valence sum rule and of the equal valence rule are kept as small as possible. We have recently applied the concepts discussed above to the determination of the crystal structures of $TRuO_{3}$ (T = 0.875Ba + 0.125Sr) [5] and BaRuO₂ [6], using initially only the information obtained from the indexing of the neutron diffraction patterns of these materials (i.e., crystal system symmetry and lattice parameters), and ignoring any

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other structural details obtained in the experimental work. Since T^{2+} and Ba^{2+} have ionic radii similar to that of O^{2-} , we may expect that the structures of $TRuO_3$ and $BaRuO_3$ are built with some sphere packing mechanism. This assumption is corroborated by the fact that in both cases the *a*-parameter calculated from the average *r* of the ionic radii of *A* and O (A = T, Ba) for the composition AO_3 is in good agreement with the experimental values, and by the fact that the ionic radius of Ru^{4+} is quite close to the radius of the octahedral void formed by the close packing of oxygen anions. The periodicity *n* of the stacking sequences of the AO_3 layers in the vertical direction of the *c*-axis, evaluated with the formula $n = c/(2r\sqrt{2/3})$, shows that $TRuO_3$ and $BaRuO_3$ have 4- and 9-layer structures, respectively, in which the RuO_6 octahedra are related to one another as indicated in Fig. 1a and 1b.

In order to fit this configuration, the theoretical bond lengths calculated with equations (1-3) have to be changed, and in particular the *A*-O bonds have to be compressed, on the average, and the

Table 1. Models of the Structure of BaRuO, (R 3 m)

	1	0	2	4
		2	3	4
Lattice parameters (Å) and atomic positions				
a	5.754	5.754	5.747(1)	0.007
C	21.142	21.626	21.602(1)	0.024
×	1/6	0.1769	0.1769(1)	0.0000
Z ₁	1/9	0.1087	0.1082(1)	0.0005
Z ₂	2/9	0.2185	0.2175(1)	0.0010
Z ₃	7/18	0.3844	0.3829(1)	0.0015
Bond distances (Å)				
Ba(1)-O(1)	2.877	2.877	2.8733(1)	0.004
-0(2)	2.877	2.938	2.926(2)	0.012
Ba(2)-O(1)	2.877	2.988	3.002(2)	-0.014
-0(2)	2.877	2.882	2.880(2)	0.002
-0(2')	2.877	2.957	2.945(3)	0.012
Ru(1)-O(1)	2.034	2.001	2.005(2)	-0.004
Ru(2)-O(1)	2.034	1.995	1.974(1)	0.021
-0(2)	2.034	1.995	2.007(2)	-0.012

Note. 1, commensurate structure derived from sphere packing geometry; 2, model obtained with the relaxation process discussed in the text; 3, experimental results; 4, difference between calculated and observed values. The theoretical bond distances calculated with equations (1-3) are: T-0 = 2.932 Å, Ba-0 = 2.948 Å and Ru-0 = 1.984 Å. Ru-O bonds have to be stretched. This process of relaxation of the initial model is carried out by modifying the structural parameters obtained from sphere packing geometry in such way that the violations of equations (1) and (2) are contained within reasonable bounds [7]. The results of the bond valence analysis of BaRuO, are reported in Table 1, where they are compared with the corresponding values determined experimentally. Similar results, reported in reference [5], were obtained for TRuO₂. The agreement between the observed and calculated structures is quite good for both compounds, and the differences between bond distances are well within 0.02 Å. This result proves that, at least in favorable cases, the bond valence method may yield an accurate model of the structure without requiring more information than that needed to index a powder pattern. More importantly, however, it shows that the need to satisfy the valence requirements of the atoms with an acceptably regular distribution of the bond valences is the driving force in determining the magnitude and the direction of the atomic shifts allowed by the symmetry, and that, as a consequence, non-bonded metal-metal and oxygen-oxygen interactions do not play an important role in the way in which the structures of these ruthenates are built. In particular, the shifts that pull together the oxygen atoms forming the shared faces of the RuO₆ octahedra (thus providing a shielding effect to Ru-Ru interactions) are specifically designed to improve the local valence balance of the Ru and O atoms involved in the Ru-O bonds. Application of the method to structural types other than perovskites is now under consideration.

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RESIDUAL STRESSES IN COLD-COILED HELICAL AUTOMOMOTIVE SPRINGS

ost front-wheel-drive automotive suspension systems use helical springs. The process chosen to produce these is, like any other engineering dilemma, determined by quality, performance, price, environmental issues, etc. Ford Motor Company has developed a potentially cost saving cold-coiling process in which less time is spent treating spring metal at elevated temperatures. The pronounced residual stress pattern within the as-cold-coiled spring is undesirable for its unpredictable effect on fatigue and corrosion behavior, and Ford evaluates these stresses by X-ray measurements of the surface stress field along with modeling of the internal stresses. The success of these at-home procedures requires an independent verification of the actual residual stress field over the cross-section of the original wire stock. The only available wellestablished method for this is neutron diffraction, and that is where NCNR expertise comes into play.

Generally, there are two ways to coil a spring: hot coiling and cold-coiling. Hot coiling implies that the spring is wound from stock at or above the recrystallization temperature. The strength and fatigue resistance are controlled afterwards by an appropriate heat treatment. Cold-coiling means that the helical winding takes place at a low temperature after the spring has been hardened and tempered. Cold-coiling allows the high temperature heat treatments to take place on the bar stock, which is easier to handle than the coiled end-product. The resulting residual stresses can be essentially eliminated by a relatively low temperature tempering treatment following the cold coiling.

The idea is to measure the residual stress field in a number of specimens that represent various stages of the production process. Using neutron diffraction one can determine the effect of the prior processing on the residual stress state of that particular stage in the process. Of equal importance, these measurements can serve to verify well established elasto-plastic models that are being used to predict the formation of residual stress. Finally one can look for a way to correlate the residual stress at the surface to the stress field as a whole.

We have looked at three cold-coiled springs. The first spring is an as-cold coiled spring. The second one is cold-coiled followed by a relatively low temper. The third one is identical to the second one, but in addition to being tempered the spring has been compressed to the point where the length of the spring is equal to the



FIGURE 1. Experimental setup. The three arrows indicate three directions of measured lattice spacing with respect to the specimen: A = axial, R = radial, T = tangential. In the current configuration, the spacing of planes whose normal is parallel to T is being measured. The sampling volume is defined by the primary aperture P and secondary aperture S, respectively.

number of windings times the wire thickness. After this the spring was allowed to relax. A small part of this torsion strain is in the plastic region, so this spring is slightly shorter than all the others. In the automotive industry this process is known as "bulldozing".

The measurements were carried out on the Double Axis system for Residual stress, Texture and Single crystal analysis (DARTS) at beam tube 8 (BT-8) in the NIST Center for Neutron Research. This instrument is specifically designed for residual stress measurements, and to that effect is equipped with very accurately positioned apertures as is shown in Fig. 1.

For these experiments the apertures were chosen to allow a sampling volume of $2 \times 2 \times 2$ mm³. The residual stress in the three springs across the cross-section of the originally 14 mm thick wire stock was determined by detecting the diffracted monochromatic neutron intensity with a position sensitive detector. The neutron wavelength was chosen such that the [211] Fe planes would scatter diffracted intensity over approximately 90°. The specimen was rotated and translated in this geometry, such that the sampling volume was scanned across this cross-section allowing the elastic (residual) strain to be determined from the small shifts in scattering Jiri Matejicek and Paul C. Brand NIST Center for Neutron Research National Institute of Standards and Technology Gaithersburg, MD 20899-8563 A. R. Drews Ford Research Laboratory Dearborn MI 48121



FIGURE 2. Contour map of the residual stress in the direction of the length of the coiled bar stock, plotted on the bar stock cross-section. With reference to Fig. 1, this is the tangential direction. This map represents the as-coldcoiled spring. The left and right side of the map represent the convex and concave sides respectively.

angle. This was done in three mutually perpendicular directions. These strain measurements allowed us to calculate the residual stress in three perpendicular directions from the equations published by Allen *et al.* [1] in each of the three specimens. For every specimen, the stress free lattice parameter was determined on the basis that the net force on the cross-section under investigation had to be zero.

From these experiments a set of interesting observations can be made. First is the notion that the residual stress pattern across the wire stock in the as-coiled spring is very pronounced and exactly matches what one would expect when a cylindrical bar is plastically bent into a hoop, a process much resembling helical coiling. With reference to Fig. 2 we note essentially uniaxial residual stress in the length direction of the original wire stock. Through the diameter of the stock, the stress goes from highly compressive at the convex side to highly tensile at the concave side. On its way through the cross-section the stress level changes sign three times, while the maximum compressive and tensile stresses are -600 MPa and +800 MPa respectively. For the tempered and bulldozed specimens



FIGURE 3. Same as Fig. 2, these data representing the as-tempered spring. The data for the as-bulldozed spring, though not given here, look essentially the same.

the pattern is roughly the same, albeit at a much reduced level. The stress range being from -170 to +160 MPa as depicted in Fig. 3.

The uncertainties in these stress levels are around \pm 30 MPa. This means that the bulldozing process does not introduce additional residual stresses, a fact that can be well understood considering that the plastic deformation under pure torsion is essentially uniform and thus cannot contribute to the residual stress state.

These results will allow Ford to correlate their model predictions and X-ray residual stress measurements to the complete residual stress field. This constitutes a powerful tool in optimizing the parameters of the spring manufacturing process.

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THE NCNR NEUTRON SPIN ECHO SPECTROMETER

The first experiments have been performed on the NCNR's Neutron Spin Echo (NSE) Spectrometer, which is the only spectrometer of its kind in the United States. This cold neutron spectrometer allows studies of dynamic processes in macromolecular systems that are relevant to, among others, polymer [1] and biomedical [2] sciences. It covers a time-scale of 0.01 ns to 100 ns and a length-scale of 2 Å to 200 Å with the equivalent of extremely high energy resolution and moderate Q-resolution. Unlike the other high-resolution inelastic instruments at the NCNR, which measure in the energy domain, the NSE spectrometer measures, in the time domain, the real part of the intermediate scattering function, I(Q,t). This is done by using the neutron's spin precession in a magnetic field as a clock to determine the energy transfer in the scattering process.

The NSE spectrometer, developed in partnership with Exxon Research and Engineering and the Forschungszentrum Jülich in Germany [3], is located at the end position of the NG-5 guide. The guide is tapered horizontally and then deviated so that the spectrometer is out of the direct line of sight of the reactor core, thereby reducing the background and the radiation load on the sample region [4]. The taper is followed by a Neutron Velocity Selector (NVS), which transmits a $\Delta\lambda/\lambda = 10$ % FWHM band of neutrons to the spectrometer. The last element of the guide is a transmission polarizer [5], which produces a polarized beam of neutrons with spin anti-parallel to the beam direction for wave-

lengths greater than 5 Å. The neutrons precess, from the first $\pi/2$ flipper (1) to the sample, through a phase angle that is determined by the time that the neutron spends in the solenoidal magnetic field (4) and the field integral along the neutron path. Near the sample, a π flipper (2) rotates the spin by 180° around the vertical axis. If the solenoid on the second arm of the spectrometer provides the same field integral for the scattered neutron and if the scattering is elastic, the neutron will precess through the same phase angle as along the first arm and will end up with the original spin orientation at the final $\pi/2$ flipper (3). This $\pi/2$ flipper rotates the spin back into the horizontal plane. If the neutron is scattered quasielastically, it will precess a slightly different number of times in the second arm of the spectrometer and end up at the second $\pi/2$ flipper rotated by some angle that is proportional to the wavelength shift. The analyzer (7) projects the component of the neutron spin that is parallel to the field direction onto the detector.

Figure 2 shows the detector count rate as a function of current in the phase coil (5), which changes the field integral on one arm of the spectrometer. The field integrals for the first and second arms of the spectrometer are equal at the echo point where the amplitude is at a maximum, since for an elastic scatterer the neutrons all arrive at the analyzer in phase. As the phase current is changed, the neutron spins rotate away from the polarization axis of the analyzer (7) and the count rate is changed. The period of this oscillation is proportional to the mean wavelength. The envelope of the



FIGURE 1. (Top) Schematic plan view of the NSE spectrometer. The numbers refer to spectrometer elements as described in the text. (Bottom) Changes of neutron spin orientation passing through the spectrometer elements for elastically (black) and inelastically (blue) scattered neutrons.

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FIGURE 2. Phase scan of an echo for an elastic scatterer (Grafoil). A is the amplitude at the echo point; N_{on} and N_{orr} are counting rates with the π flipper on and off, respectively, and the $\pi/2$ flippers off.



FIGURE 3. Normalized and resolution-corrected measurements of the intermediate scattering function at the indicated Q-values for 10 % SDS in D_2O at room temperature. The resolution curve is a normalized measurement of an elastic scatterer (Grafoil) at 10 Å.

oscillations gives the wavelength distribution. The amplitude A is proportional to I(Q,t), which can be normalized by measuring the difference in intensity with the π flipper on and off, N_{oN} – N_{oFF}. For the normalizing measurement, which is proportional to I(Q,t = 0), the $\pi/2$ flippers are off since there is no precession of the neutrons in this configuration. Instrumental resolution effects are removed by dividing the normalized sample signal by the normalized values from an elastic scatterer.

We have also verified the operation of the correction elements (6) which allow non-axial and divergent neutrons to satisfy the echo condition. These are essential for operation at high fields. The value of the resolution amplitude at 25 ns in Fig. 3 would be at least a factor of ten smaller without the contribution of the correction elements.

As an example of the science that is available through NSE, we have reproduced the measurements of Hayter and Penfold [6] on a micellar solution of 10 % sodium dodecylsulfate (SDS) in D_2O . Fig. 3 shows the normalized intermediate scattering function versus time at several Q values at, well below, and well above the interaction peak in the structure factor. The effective diffusion constant $D_{eff}(Q)$, obtained from I(Q,t) ; exp($-D_{eff}(Q)Q^2t$), varies inversely with the structure factor, reaching a minimum at the structure factor peak, an effect analogous to DeGennes narrowing. As a result, in a dilute solution of interacting micelles, one may unambiguously separate the structure factor from the form factor, which may not be done with only a SANS measurement.

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SERVING THE SCIENTIFIC AND TECHNOLOGICAL COMMUNITIES

roviding neutron beam methods to the U.S. research com-Ð munity is a central purpose of the NCNR. Intense neutron sources are scarce, as are the advanced instruments needed to fully exploit them. Expert scientists will always recognize opportunities to perform research with the most powerful tools available, so that the existence of the NCNR as the Nation's premier reactorbased neutron facility stimulates considerable demand from the community. However, in order to foster the best possible science, many of our procedures and activities are designed to encourage researchers to learn more about our capabilities, and eventually to obtain instrument time through proposals and collaborations. The first instruments became operational in the NCNR guide hall in 1991, and a formal proposal system was started to accommodate users from universities, industry, and government laboratories. Since then, the quantity and the diversity of research carried out at the NCNR has grown steadily, and the number of research participants (Fig. 1) is now several times larger than it was just a few years ago.



FIGURE 1. Research participants at the NCNR.

THE USER PROGRAM

NIST has always relied on advice from accomplished scientists to assist in formulating policy. The Program Advisory Committee (PAC) is the body primarily responsible for proposal review and recommending user policies for the NCNR, working closely with the Center's Director and staff. Its current membership includes Sanat Kumar (Penn State University, chair), Robert M. Briber (University of Maryland), Michael K. Crawford (DuPont), Dieter K. Schneider (Brookhaven National Laboratory), Thomas P. Russell (University of Massachusetts), Sunil K. Sinha (Argonne National Laboratory), Emile A. Schweikert (Texas A&M University), Laurence Passell (Brookhaven National Laboratory), and Gabrielle G. Long (NIST).

At their meeting in May 1999, the PAC made several recommendations for improving the current system. They felt that program proposals for longer-term projects had not had their intended effect of reducing the burden for external reviewers, and should be discontinued for the time being. Noting a potential for large increases in instrument time for biology-related proposals in the next few years, the addition of another PAC member with expertise in that area was recommended. The PAC discussed the results of a recent independent survey of user satisfaction, coordinated by Anne Mayes of MIT. Ideas for obtaining more feedback from users were also considered. Incidentally, Professor Mayes, the head of the NCNR User's Group, recently won the American Physical Society's Dillon Medal, partly for work carried out using NCNR instruments.

Most of the PAC meeting was devoted to proposal review. With the help of several written reviews for each proposal from selected experts, the PAC considered 151 proposals requesting 1138 instrument-days, and allocated 514 instrument-days to 95 proposals for SANS, reflectometry, SPINS, and time-of-flight instruments. The PAC usually meets twice a year, but because of a planned shutdown for cold source and reactor upgrades, only one meeting was held in 1999.



FIGURE 2. Nick Rosov explains the operation of the Neutron Spin Echo Spectrometer to summer school participants.

SUMMER SCHOOL ON METHODS AND APPLICATIONS OF NEUTRON SPECTROSCOPY

Each summer for the past five years, the NCNR has held a oneweek school to introduce researchers to the methods of neutron scattering. Organized by senior staff members John Copley and Peter Gehring, the 1999 summer school was devoted to spectroscopy with neutrons, with an emphasis on the opportunities afforded by new instruments in the guide hall. Most of the 32 participants, chosen from almost 60 who applied, were graduate students and postdoctoral fellows. The curriculum consisted of lectures by NCNR staff and experts from other institutions, tours of the facility, and four three-hour, hands-on, sessions at instruments in the guide hall. Informal discussions among students and the resident staff were also an important part of the school's activities.

Beginning with lectures on the fundamental concepts of nuclear and magnetic scattering, the agenda turned to talks on applications of neutron spectroscopy to a wide range of research topics. Subsequent presentations covered the reactor and cold source, specific scattering techniques, computer modeling and ab initio calculations, and the operating principles of various spectrometers used in the experimental sessions. In the latter, students and staff used time-of-flight, backscattering, triple-axis, and neutron spinecho spectrometers to measure, respectively, quasielastic scattering from water, rotational tunneling in solid methyl iodide, magnetic excitations in a geometrically frustrated antiferromagnet, and coherent scattering from the time domain in a system of spherical micelles. (The last example is illustrated in the article on the Neutron Spin Echo Spectrometer.) The lecture materials will be placed on the NCNR website, in order to reach as wide an audience as possible.

THE CENTER FOR HIGH-RESOLUTION NEUTRON SCATTERING (CHRNS)

Supported by the National Science Foundation (NSF), CHRNS is a very important component of the user program. It operates a suite of three instruments, including a 30 m SANS machine, the SPINS triple-axis spectrometer, and a double-crystal, high-resolution SANS. The last of these is under construction. Approximately 40 % of the instrument time allocated by the PAC goes to experiments carried out on CHRNS instruments. The NSF is currently reviewing the CHRNS renewal proposal, which requests support for upgrading existing instruments and including an improved 9 m SANS instrument within CHRNS. Approval would mean an increase of more than 50 % in CHRNS' capacity to serve users.

COLLABORATIONS

Direct collaborations on specific experiments remain a common way for users to pursue their ideas using NCNR facilities, accounting for approximately 60 % of the number of instrument-days. The thermal-neutron triple-axis spectrometers are mainly scheduled in this way. Most of the time reserved for NIST on these and all other NCNR instruments is also devoted to experiments that are collaborations with non-NIST users.

Another mode of access to the NCNR is through Participating Research Teams (PRTs). In this case, groups of researchers from various institutions join forces to build and operate an instrument. Typically, 50 % to 75 % of the time on the instrument is then reserved for the PRT, and the remaining time is allocated to general user proposals. For example, a PRT involving ExxonMobil, the University of Minnesota, Texaco R&D and NIST cooperates on the NG-7 30 m SANS instrument. Similar arrangements involving other PRTs apply to the horizontal-sample reflectometer, the highresolution powder diffractometer, and the neutron spin-echo spectrometer.

INDEPENDENT PROGRAMS

There are a number of programs of long standing located at the NCNR which involve other parts of NIST, universities, industrial laboratories, or other government agencies.

The Polymers Division of the Materials Science and Engineering Laboratory has two major program elements at the NCNR. In the first, the purpose is to help the U.S. microelectronics industry in addressing their most pressing materials measurement and standards issues. In today's ICs and packages, the feature size on a chip is ever shrinking, approaching 250 nm, while the size of a polymer molecule is typically 5 nm to10 nm. As feature size shrinks, the structure and properties of interfaces play an increasingly important role in controlling the properties of the polymer layers used in interconnects and packages. NIST scientists use both neutron reflectivity and other neutron scattering methods to characterize polymer/metal interfaces with regard to local chain mobility, moisture absorption, glass transition temperature, and crystalline structure. In the second program element, the objective is to understand underlying principles of phase behavior and phase separation kinetics of polymer blends, both in the bulk and on surfaces, in order to help control morphology and structure during processing. SANS and reflectivity measurements in equilibrium, in transient conditions, and under external fields provide essential information for general understanding as well as for specific application of polymer blend/alloy systems. Customers include material producers and users, ranging from chemical, rubber, tire, and automotive companies, to small molding and compounding companies. The focus of research on polymeric materials includes commodity, engineering and specialty plastic resins, elastomers, coatings, adhesives, films, foams, and fibers.

The ExxonMobil Research and Engineering Company is a member of the Participating Research Team (PRT) that operates, maintains, and conducts research at the NG-7 30 m SANS instrument and the recently commissioned NG-5 Neutron Spin Echo Spectrometer. The mission is to use those instruments, as well as other neutron scattering techniques, in activities that complement research at ExxonMobil's main laboratories as well as at its affiliates' laboratories around the world. The aim of these activities is to deepen understanding of the nature of ExxonMobil's products and processes, so as to improve customer service and to improve the return on shareholders' investment. Accordingly, and taking full advantage of the unique properties of neutrons, most of the experiments use SANS or other neutron techniques to study the structure and dynamics of hydrocarbon materials, especially in the fields of polymers, complex fluids, and petroleum mixtures. ExxonMobil regards its participation in the NCNR and collaborations with NIST and other PRT members not only as an excellent investment for the company, but also as a good way to contribute to the scientific health of the Nation.

The Nuclear Methods Group (Analytical Chemistry Division, Chemical Science and Technology Laboratory) has as its principal goals the development and application of nuclear analytical techniques for the determination of elemental compositions with greater accuracy, higher sensitivity, and better selectivity. A high level of competence has been developed in both instrumental and radiochemical neutron activation analysis (INAA and RNAA). In addition, the group has pioneered the use of cold neutron beams as analytical probes with both prompt gamma activation analysis (PGAA) and neutron depth profiling (NDP). PGAA measures the total amount of a particular analyte present throughout a sample by the analysis of the prompt gamma-rays emitted during neutron capture. NDP, on the other hand, determines concentrations of several important elements (isotopes) as a function of depth within the first few micrometers of a surface by energy analysis of the prompt charged-particles emitted during neutron bombardment. These techniques (INAA, RNAA, PGAA, and NDP) provide a powerful combination of complementary tools to address a wide variety of analytical problems of great importance in science and technology, and are used to help certify a large number of NIST Standard Reference Materials. During the past several years, a large

part of the Group's efforts has been directed towards the exploitation of the analytical applications of the guided cold-neutron beams available at the NIST Center for Neutron Research. The Group's involvement has been to design and construct state-of-the-art cold neutron instruments for both PGAA and NDP and provide facilities and measurements for outside users, while retaining and utilizing our existing expertise in INAA and RNAA.

The Center for Food Safety and Applied Nutrition of the U. S. Food and Drug Administration (FDA) directs and maintains a neutron activation analysis (NAA) facility at the NCNR. This facility provides agency-wide analytical support for special investigations and applications research, complementing other analytical techniques used at FDA with instrumental (INAA), neutron-capture prompt-gamma (PGAA), and Radiochemical Neutron Activation Analysis (RNAA) procedures, radioisotope X-ray fluorescence spectrometry (RXRFS), and low-level gamma-ray detection. This combination of analytical techniques enables diverse multi-element and radiological information to be obtained for foods and related materials. The NAA facility supports agency quality assurance programs by developing in-house reference materials, by characterizing foodrelated reference materials with NIST and other agencies, and by verifying analyses for FDA's Total Diet Study Program annually. Other studies include the development of RXRFS methods for screening foodware for the presence of Pb, Cd, and other potentially toxic elements, use of INAA to investigate bromate residues in bread products, and use of PGAA to investigate boron nutrition and its relation to bone strength. The FDA's NAA laboratory personnel frequently provide intra-agency technical assistance, the most recent example being participation in the production of the document "Accidental Radioactive Contamination of Human Food and Animal Feeds: Recommendations for State and Local Agencies" by the Center for Devices and Radiological Health.

The Neutron Interactions and Dosimetry Group (Physics Laboratory) provides measurement services, standards, and fundamental research in support of NIST's mission as it relates

to neutron technology and neutron physics. The national and industrial interests served include scientific instrument calibration, electric power production, radiation protection, defense nuclear energy systems, radiation therapy, neutron radiography, and magnetic resonance imaging. The Group's activities may be represented as three major activities. The first is Fundamental Neutron Physics - including operation of a neutron interferometry and optics facility, development of neutron spin filters based on laser polarization of ³He, measurement of the beta decay lifetime of the neutron, and investigations of other coupling constants and symmetries of the weak interaction. This project involves a large number of collaborators from universities and national laboratories. The second is Standard Neutron Fields and Applications - utilizing both thermal and fast neutron fields for materials dosimetry in nuclear reactor applications and for personnel dosimetry in radiation protection. These neutron fields include thermal neutron beams, "white" and monochromatic cold neutron beams, a thermal-neutron-induced ²³⁵U fission neutron field, and ²⁵²Cf fission neutron fields, both moderated and unmoderated. The third is Neutron Cross Section Standards - including experimental advancement of the accuracy of neutron cross section standards, as well as evaluation, compilation and dissemination of these standards.

Several universities have also established long term programs at the NCNR. The **University of Maryland** is heavily involved in the use of the NCNR, and maintains several researchers at the facility. **Johns Hopkins University** participates in research programs in solid state physics and in instrument development at the NCNR. The **University of Pennsylvania** is working to help develop biological applications of neutron scattering. It is also participating in the construction of a new filter analyzer neutron spectrometer, along with the **University of California at Santa Barbara**, DuPont, Hughes, and Allied Signal. The **University of Minnesota** participates in two PRTs, the NG-7 30 m SANS and the NG-7 reflectometer. The **University of Massachusetts** also participates in the latter PRT.

REACTOR OPERATIONS AND ENGINEERING

The reactor operated for 250 full power (20 MW) days during the past year, or 68 % of real time. This meets our goal, and was achieved in spite of a shutdown for maintenance early in the fiscal year. The reactor operated with better than 90 % predictability–i.e., days operated on the day originally scheduled. This is substantially better operation than is specified in national goal of 85 % reliability for user facilities. Just after the end of the fiscal year, another fuel shipment was made, thus clearing additional space in the spent fuel storage pool. This completes our current round of scheduled spent fuel shipments. There is now ample storage space for at least 5 years of continuous 20 MW operation.



TRTR Chairman-Elect, Pedro Perez, presents NIST Director, Raymond G. Kammer, with a plaque stating:

"The National Organization of Test, Research and Training Reactors recognizes the National Institute of Standards and Technology on the occasion of its centennial as the foremost measurements and standards laboratory in the world, and honors NIST for 100 years of distinguished service to the nation and to all the segments of science and technology, and particularly for its pioneering efforts in nuclear research and radiation standards and in appreciation of its leadership in the formation of TRTR." As part of preparing for another 20 years of operation past 2004, we have been assessing the present cooling tower. During the past year, a contract for construction of a hybrid wet/dry tower was signed. This new tower will not only meet our cooling needs; it will also reduce the visible plume given off by the tower during the winter months, a substantial additional benefit. The new tower will be erected adjacent to the current one, and will not require an outage until the switch is made. This outage will be scheduled when the new tower is fully ready for use, so as to minimize down time. Extensive planning for the piping changes that will be needed is now underway. It is expected that the new second-generation cold source will be installed at the same time, so as to maximize operating time.

During the coming year, we will take advantage of the normal Christmas shutdown to replace the shim control arms. This operation is required every 4 to 5 years, since the cadmium in the control arms is burned up in service. By utilizing the time period of the holiday shutdown, the entire operation will be done with only 3 weeks of additional shutdown time. Since this period will also be used for required training and requalification of operations personnel, there will be little lost operating time compared to our best possible schedule.

A major highlight of the year was the annual conference of the National Organization of Test, Research and Training Reactors (TRTR) held at NIST, chaired and organized by the group. By all accounts, it was highly successful with the largest number of participants ever. Many distinguished speakers addressed the conference including the Chairman of the Nuclear Regulatory Commission and the Under Secretary of Energy. Also, TRTR became the first professional organization to honor NIST on its forthcoming centennial.

Finally, preparations for a license renewal application are proceeding as planned. We are fortunate to have attracted Dr. Seymour H. (Sy) Weiss to our staff, as Deputy to NCNR Director. He will be leading the relicensing effort and associated reactor upgrades for the coming years, and has already started several tasks related to this effort.

INSTRUMENTATION DEVELOPMENTS

IMPROVEMENTS TO THE PERFORMANCE OF HFBS

The recent commissioning of the high flux backscattering spectrometer (HFBS) has opened up opportunities to a variety of users from around the country to study the dynamics of condensed matter systems with time scales on the order of nanoseconds (10⁻⁹ s) and length scales up to 10 Å. A careful design which incorporates stateof-the-art neutron optics has resulted in a flux on sample as high as that of any other backscattering spectrometer in the world with comparable energy resolution. The first call for proposals for the HFBS was made in FY 1999 and the response was outstanding. Several successful user experiments have already been performed (see Fig. 1) with more scheduled in the near future. Although the initial performance of the instrument was impressive, improvements continue to be made.

One of the recent changes to the instrument has been a modification to the data acquisition system. Initially the data were binned to the velocity of the reciprocating silicon monochromator. However the method in which the velocity was determined overemphasized local details of the monochromator motion which were not observed



FIGURE 1. Measurement of quasi-elastic scattering from monolayer coverage of alkanes on grafoil performed on the HFBS by H. Taub, D. Fuhrmann, L. Criswell, and K. Herwig. The low temperature data (blue) is resolution limited with a full-width at half maximum of 0.96 μ eV. The higher temperature data (red) clearly displays broadening indicative of the diffusive motion of the alkanes.

in the neutron data. A detailed analysis of the performance of the monochromator system indicated that the quality of the data could be improved by making a rather simple change in the way that data is collected and stored, namely by binning data versus time during a period of the Doppler monochromator motion. The data are later re-binned to velocity using the average monochromator motion profile. This straightforward change to the way that the data are collected has improved the quality of the data, suppressing the spurious effects observed in the velocity-binning mode.

Improvements have also been made to the Doppler monochromator system which have improved its reliability and performance. One of the problems encountered is associated with the fact that the monochromator is being driven to higher energy transfers than any other Doppler monochromator system in the world. The monochromator, which is vibrated over a distance of 9 cm at high frequencies, can experience accelerations in excess of 100 g's. Prolonged vibration of the monochromator resulted in the silicon wafers coming off of the monochromator surface. Recent changes in the gluing method have dramatically increased the lifetime of the silicon wafers on the monochromator.

Efforts to optimize shielding in various parts of the instrument have resulted in improvements in the signal to noise ratio in the detectors. During normal operation the instrument vessel (which is made up of the sample, analyzer crystals, and the detectors) is evacuated in order to reduce the background due to air scattering. Additional shielding has been installed on the detector assembly and on parts of the analyzer system in the vessel. Improvements have also been made to the shielding on the phase space transform chopper to handle the highly divergent beam from the converging neutron guide. This has resulted in a substantial increase of the signal to noise ratio to almost 600 to 1.

THE DISK CHOPPER SPECTROMETER

The Disk Chopper Spectrometer (DCS) is an extremely versatile time-of-flight instrument, designed to achieve a broad range of energy resolution full widths, from 12 µeV to 1 meV, by changing chopper speed, wavelength, and/or beam width. Once commissioned, the DCS will be in high demand for experiments on a variety of systems such as proteins, molecular crystals, disordered materials, and metal-hydrogen systems. The instrument is to be



FIGURE 2. Photograph of the DCS detector bank and flight chamber.

included, on a limited basis, in the first Call for Proposals in FY2000.

Over the past twelve months the DCS has seen major progress in a number of important areas. The sample chamber and flight chamber were equipped with beam-handling components, and the insides of both chambers were lined with cadmium. Flow tests were performed in order to complete the design of the gas handling system for the sample and flight chambers; the system had been fabricated and had largely been assembled by the end of the fiscal year. An overhead platform, to be used for sample environment preparation and installation into the sample chamber, has also been designed.

Problems with some of the amplifiers for the DCS detectors prompted us to send the complete inventory to the manufacturer for mutually agreed modifications. On their return discriminator thresholds were individually set, and the full complement of 913 detectors and amplifiers was installed on the detector racks, which were then installed and aligned at the spectrometer. The detectors and voltage distribution assemblies were cabled and fully tested prior to installation of the outer shields. Much work was devoted to troubleshooting and greatly improving the reliability and performance of the VME data acquisition electronics, and to the development of data acquisition software.

PERFECT CRYSTAL SANS DIFFRACTOMETER

A perfect crystal diffractometer (PCD) used for very high resolution small angle neutron scattering (SANS) is being developed jointly by the NCNR and NSF as part of the CHRNS facility. The higher resolution obtained by using perfect silicon crystals increases the maximum size of features that can be measured from 0.1 μ m obtained using the current NCNR's two 30 m, pinhole geometry SANS instruments, to 10 μ m with the new instrument. The monochromator and analyzer use triple reflections before and after the sample. Using large channel-cut silicon crystals suppresses the "wings" of the beam profile, improving the signal-to-noise ratio to values comparable to that obtained from the pinhole instruments. The PCD will cover a Q-range from 0.0004 nm⁻¹ to 0.1 nm⁻¹ with an expected beam current of 50,000 s⁻¹.

Located on the BT-5 beam tube in the Confinement Building, the layout of the instrument is shown in Fig. 3. A vertically and horizontally focusing graphite premonochromator directs the neutron beam away from the main reactor beam towards the perfect crystal monochromator. The monochromator reflects a highly collimated beam to the sample. After the sample, a high precision rotation stage rotates the analyzer crystal to scan the small angle scattering obtained from the sample, which is then collected by the detector. The shields supporting the premonochromator are set on kinematic mounts for accurate repositioning. The monochromator and analyzer are isolated from room vibrations using a heavy pneumatic vibration isolation table.



FIGURE 3. Schematic layout of the BT-5 perfect crystal diffractometer.

In 1999, all detailed design work was finished and all purchased parts received. The beam shutter and premonochromator shielding have been installed. Major components yet to be installed are the detector housing, sample position table, and the small monochromator shield. Preliminary characterization measurements leading to instrument commissioning are planned for early 2000.

THE FILTER ANALYZER NEUTRON SPECTROMETER

The new Filter Analyzer Neutron Spectrometer (FANS) is designed to give U.S. researchers access to unprecedented sensitivity for measuring the vibrational spectra of a wide variety of materials. This spectrometer, which will replace the current BT-4 instrument, uses cryogenically cooled polycrystals as low energy band-pass filters for neutrons scattered from the sample. A dramatic gain in signal over the existing filter analyzer is achieved primarily through a large increase in the solid angle covered by the secondary spectrometer. The FANS instrument is being developed in two phases. Phase I includes the first of two new filter assemblies, and Phase II includes the second filter analyzer and a new monochromator and monochromator drum system. During the past year, the detailed design of the first filter wedge was completed, the vacuum chamber which encloses the Be and graphite filters was delivered, and the Be and graphite filter wedges were assembled (see Fig. 4). Recent tests demonstrated that these filters can be cooled to cryogenic temperatures which is necessary to maximize the performance of the instrument. Furthermore all of the parts for the undercarriage for Phase I have been received, assembled, and operated successfully under load. Most of the shielding and data acquisition system have also been received. Major assembly and installation of Phase I will begin early in 2000.

DEVELOPMENT OF AN ACTIVE DOUBLE Focussing Monochromator system

The use of vertical or horizontal focussing monochromators to increase the intensity of selected neutrons incident on the sample is well known and widespread. Less common are systems combining these features. Often the machinery employed to do this is cumbersome and results in extraneous material in the beam, increasing the background from unwanted neutrons. For horizontal focussing it is desirable to have maximum flexibility by adjusting many



FIGURE 4. The FANS filter assembly. The first Be filter is in the foreground with the PG filter second and the final Be filter in the background just before the detector bank. A radial collimator is visible in the middle of the assembly.



FIGURE 5. A drawing of the active double focussing monochromator system.

monochromator elements while keeping the corresponding mechanical components away from the beam area. By contrast, vertical focussing can be confined to the problem of adjusting only one parameter, the radius of curvature of the array, and admits a correspondingly simpler mechanical solution.

We are currently developing a system that combines completely flexible horizontal focussing with vertical focussing that is achieved by buckling the entire system of separately controllable vertical crystal arrays. Cylindrical curvature is to be accomplished by compressing the system of specially shaped strips upon which the individual monochromating crystals are mounted. This design eliminates extraneous material in the beam, which should result in greatly reduced background.

AN ADVANCED LIQUID HYDROGEN COLD SOURCE

The NIST liquid hydrogen cold source has completed over four years of service. It was installed with three goals: at least double the cold neutron intensity with respect to its predecessor (D_2O ice); operate simply and reliably; and pose no safety threat to the reactor or personnel. It has successfully met or exceeded all these goals. The cold neutron flux increased by a factor of 4 to 6, for wavelengths in the range of 0.2 nm to 2 nm. The availability of the source has been nearly 99 % of the time that the reactor was available (the reactor is shut down if the source is inoperable). And there have been no hydrogen leaks, nor have any of the insulating vacuums or helium containments been compromised.

Even as Unit 1 was being installed in 1995, however, improvements in the MCNP model (used for Monte Carlo simulations) of the NIST reactor were pointing toward a new, but more complicated cryostat assembly, with a possible additional gain of a factor of two. Better coupling between the reactor fuel and the cold source can be achieved by expanding the cooling jacket into the volume now occupied by the insulating vacuum, so that it partially surrounds the moderator vessel. In this way, the D₂O coolant also serves as an extension of the reactor reflector. Additional changes in the new source are based on our operating experience with the existing LH₂ source and extensive MCNP calculations. Unit 2 will be an ellipsoidal shell rather than spherical, it will have an evacuated inner vessel, rather than vapor-filled, and the LH₂ layer will be up



FIGURE 6. NCNR Mechanical Engineering Technician Scott Slifer welds the aluminum moderator vessel for the advanced hydrogen cold source.

to 30 mm thick, rather than 20 mm, without increasing the LH_2 volume.

The advanced liquid hydrogen cold source is currently being fabricated and will be installed in the NIST reactor next year. Enhanced mechanical design and manufacturing tools are exploited in the fabrication of the advanced source. Components of the hydrogen, insulating vacuum, helium containment, and D_2O vessels are cut from solid blocks of Al 6061 on a computer-controlled, high-speed mill at the NIST Instrumentation shop, and are then welded and thoroughly tested by NCNR staff (see Fig. 6). It is expected that the flux of cold neutrons will increase by a factor of 1.8.

IMPROVEMENTS TO NCNR SAMPLE ENVIRONMENT EQUIPMENT

The sample environment equipment has seen a number of changes in FY1999 at the NCNR. One of the most visible accomplishments has been the development of informative webpages that detail sample environment resources, specifications, and even the current operating condition and location for specific devices. This information is easily accessible through the NCNR homepage (http://www.ncnr.nist.gov/), allowing guest researchers to plan their experiments and design appropriate sample holders.

Commissioning has begun on a new high magnetic field/ low temperature superconducting magnet system which is financed through a joint collaboration of Johns Hopkins University, NCNR, NEC Research Institute, Princeton, the University of Maryland, and a National Science Foundation IMR grant. The 0.022 K dilution refrigerator of this new system has already been tested and used on triple axis spectrometers. A 7 T magnet has been temporarily outfitted in the system at this time, with replacement by a much stronger superconducting magnet of 10 T to 12 T in the coming months. The magnet is capable of operating with either a homogeneous magnetic field at the sample position or with a field gradient, a great aid for polarized neutron beam experiments. This system is

Table 1 Recent Sample Environment Acquisitions

Helium flow cryostat	1.5 K to 300 K, dedicated to
	Dackscattering spectrometer
Helium flow cryostat	1.5 K to 300 K, dedicated to disk chopper
	time-of-flight spectrometer
Helium flow cryostat	1.5 K to 300 K
Pumped helium-3 cryostat insert	0.30 K to 300 K, for use with 7 T
	vertical field magnet
Closed-cycle helium refrigerator	7 K to 320 K
Closed-cycle helium refrigerator	10 K to 320 K, modified for
, ,	backscattering spectrometer
Poiseuille flow shear cell	shear rate up to 130,000 s ⁻¹ near surface
Light scattering particle sizer	2 nm to 100 nm diameter
Bheometer	1.7 x 10 ⁻³ Pa s to 2.7 x 10 ⁸ Pa s.
	-60 °C to 600 °C, for in-situ measurement
	during SANS experiments
Lyophilizer	freeze drver for sample preparation
Kare Fisher titrator	quantitative analysis of water in a sample
Erootor	20.0C for longer term storage of
Freezer	-20 °C, for longer term storage of
· · · · · · · · · · · · · · · · · · ·	Diological samples
Superconducting magnet	0 A to 120 A, bi-directional
power supply	
Dual channel lock-in amplifier	1 mHz to 102 kHz frequency range
Four temperature controllers	programmable, remote operation
Dual channel lock-in amplifier	0 A to 120 A, bi-directional
Four temperature controllers	programmable, remote operation
Single-crystal windowed tail	for SANS low-temperature
	electromagnet experiments
Sensitive equipment	custom-designed to protect
transport carts	valuable equipment
indisport carts	valuable equipment

top loading, which provides an important capability for experiments requiring multiple sample changes or for quick changeover between consecutive experiments.

From new capabilities to improved sample preparation and screening, the sample environment resources have been significantly expanded during the past year. Listed in Table 1 are further acquisitions, notable modifications, and improvements that combine to offer the researcher more control over the sample conditions. Fig. 7 displays a typical cryorefrigerator sample environment. More details are available at the web site mentioned above.



FIGURE 7. Schematic diagram of the sample environment in a cryorefrigerator in the neutron beam.

RESEARCH TITLES

MATERIALS SCIENCE/ CRYSTALLOGRAPHY

Acid Sites in Zeolite ZSM-5 A. Peters³⁴, R.-H. Hu²¹⁴, S. Purnell²¹⁴, B. H. Toby¹⁰², R. Senesi¹⁵⁰, D. A. Neumann¹⁰² and D. H. Olson¹⁹⁷ Composition Aerogels Investigated by SANS with **Contrast Masking Solutions** C. Merzbacher⁹⁹. M. Anderson⁹⁹ V. Cepak⁹⁹ and J. G. Barker¹⁰² E. Ku⁸³ **Amorphous Content Determination** Methodology J. K. Stalick¹⁰², R. Senesi¹⁵⁰ and B. H. Toby102 **Cation Location in Ion Exchanged** (Li,Na) Chabazite L. J. Smith¹⁷⁵ and A. K. Cheetham¹⁷⁵ Characterization of Ag and Mixed Li/Ag Faujasite Zeolites Using Neutron Diffraction Nano-Fibers N. Hutson¹⁹⁰, R. Yang¹⁹⁰, B. A. Reisner¹⁰² and B. H. Toby¹⁰² Characterization of Microcracking in Iron Titanate by SANS A. J. Allen¹⁰⁵ and E. Fuller¹⁰⁵ Chemical Ordering in Ni-Mn-Ga **Heusler Alloys** Structure R. Overholser188, M. Wuttig188 and D. A. Neumann¹⁰² **Comparison of Residual Stress** Type **Measurement Techniques** T. Gnäupel-Herold^{188, 102}. P. C. Brand¹⁰², H. J. Prask¹⁰², S. Spooner¹²³, B. Pardue¹⁵², D. Nelson¹⁴⁹ and S. Foss⁷⁰ **Complex Oxide Structures** A. Sleight¹²⁸, I. Radosavljevic¹²⁸ and T. Amos¹²⁸ 'CONV' and 'STRESS': New Codes for Shear **Data Reduction in Neutron Diffraction Residual Stress Measurements** P. C. Brand¹⁰² and T. Gnäupel-Herold^{188, 102} CPD Round Robin on Ouantitative Phase Analysis J. K. Stalick¹⁰² **Crystal Chemistry of Protonic Centers** in Zeolites D. H. Olson¹⁹⁷ and B. H. Toby¹⁰² Crystal Structure of Ba,CaCu₂O₆(CO₂) Q. Z. Huang^{102, 188} and F. Izumi⁹ **Crystal Structure of Dehydrated** LaD-LSX D. H. Olson¹⁹⁷, B. A. Reisner¹⁰² and B. H. Toby102 **Crystal Structure of Ferroelectric Tetragonal Tungsten Bronze Lead Barium Niobate** Q. Z. Huang^{102, 188} and R. Guo¹³³

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