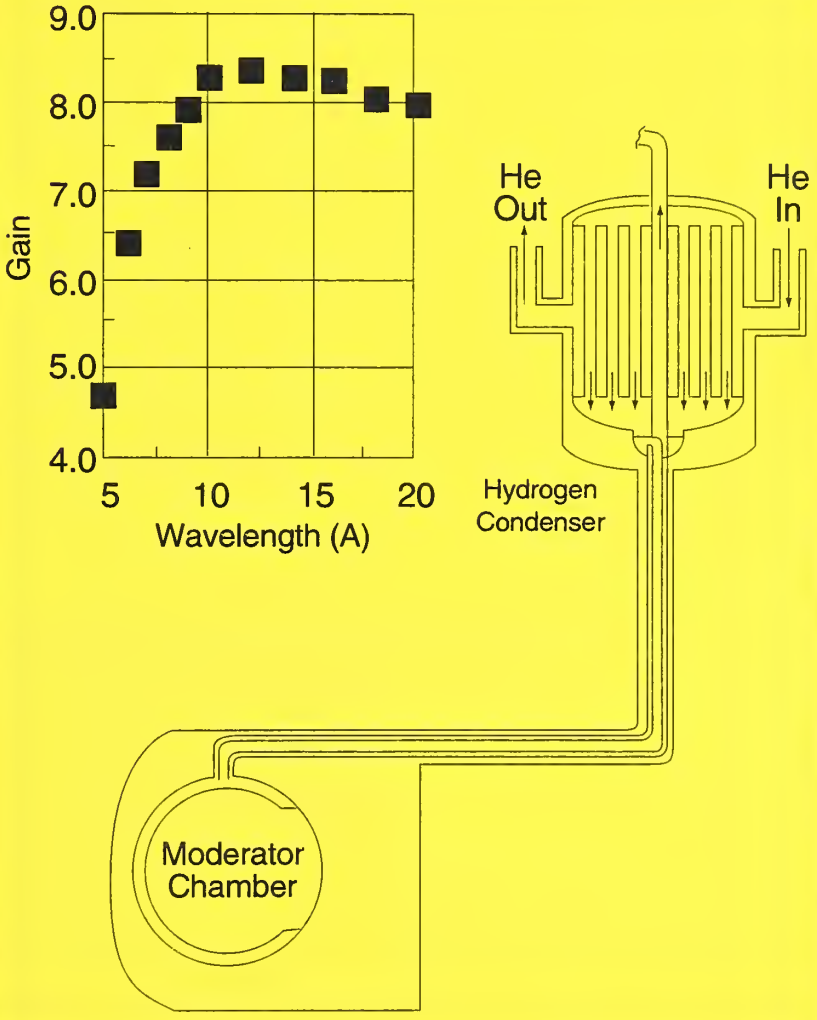
MSEL

Materials Science and Engineering Laboratory

REACTOR RADIATION



NISTIR 5751
U.S. Department of Commerce
Technology Administration
National Institute of Standards
and Technology

Technical Activities 1995

Reactor Radiation

Schematic of the new liquid hydrogen cold source system installed at the NIST Reactor and the measured gain in cold neutron intensity after installation of the new cold source and other reactor improvements. The cold neutron intensities now available at the Cold Neutron Research Facility are competitive with those at the Institute Laue'-Langevin, the premier neutron research facility in the world.

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REACTOR RADIATION

J. M. Rowe, Chief
T. M. Raby, Deputy

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U.S. DEPARTMENT OF COMMERCE
Ronald H. Brown, Secretary

TECHNOLOGY ADMINISTRATION
Mary L. Good, Under Secretary for Technology

NATIONAL INSTITUTE OF STANDARDS
AND TECHNOLOGY
Arati Prabhakar, Director

ABSTRACT

This report summarizes all the programs that use the NIST reactor. It covers the period for October 1994 through September 1995. The programs range from the use of neutron beams to study the structure and dynamics of materials through nuclear physics and neutron standards to sample irradiations for activation analysis, isotope production, neutron radiography, and nondestructive evaluation.

KEY WORDS: activation analysis; cold neutrons; crystal structure; diffraction; isotopes; molecular dynamics; neutron; neutron radiography; nondestructive evaluation; nuclear reactor; radiation.

DISCLAIMER

Certain trade names and company products are identified in order to adequately specify the experimental procedure. In no case does such identification imply recommendation or endorsement by the National Institute of Standards and Technology, nor does it imply that the products are necessarily the best available for the purpose.

Contents

REACTOR RADIATION DIVISION (856)

J. Michael Rowe, Chief
Tawfik M. Raby, Deputy Chief

The responsibilities of the Reactor Radiation Division are threefold: to operate the research reactor (NBSR) as a NIST and national resource in a cost-effective manner while assuring the public safety; to conduct a program of materials research using neutron methods, while developing and maintaining state-of-the-art instrumentation to ensure the best utilization of the NBSR neutron scattering facilities; and to develop and operate the Cold Neutron Research Facility (CNRF) as a national center, providing unique measurement capabilities to U.S. researchers.

In order to fulfill these responsibilities, the Reactor Radiation Division (RRD), in collaboration with researchers in the Materials Science and Engineering Laboratory (MSEL), the Physics Laboratory (PL), the Chemical Science and Technology Laboratory (CSTL), and other NIST organizations, as well as many outside organizations (industrial, university and other government agency), develops and applies neutron methods to a broad range of problems of national concern. The techniques include nuclear activation methods for chemical analysis; neutron scattering methods for the characterization of atomic and molecular arrangements and dynamics in all classes of materials; neutron diffraction methods for determination of residual stress and texture; neutron autoradiography for art history and restoration; neutron radiography; and various techniques for neutron flux calibrations, personnel radiation monitoring, and fundamental neutron physics.

The sections that follow are a summary of the technical activities of the Reactor Radiation Division over the past year. A detailed report on work performed at the NIST reactor is available in the NIST Internal Report 5829 entitled "NIST Reactor: Summary of Activities, October 1994 through September 1995."

Major Activities:
The Reactor Operations and Engineering group is primarily responsible for the safe and efficient operation of the reactor in order to provide a cost-effective and productive unique national resource. This group also performs sample irradiations (for both activation analysis and isotope production), helps users to design and install new experiments, and is responsible for monitoring many experimental systems (e.g. the helium refrigerator for cold source cooling). In addition to operation of the reactor, group staff perform periodic maintenance, complete necessary surveillance tests, design and oversee major reactor improvements and upgrades, and are responsible for ensuring compliance with all regulatory requirements.

The Neutron Condensed Matter Science group, in collaboration with hundreds of other researchers from NIST, universities, industry, and other government organizations carries out an active program of research, applying neutron methods to a broad variety of problems in the general areas of physics, chemistry and material science. Of particular interest are new materials for science and technology, such as polymer blends, fullerenes, oxide superconductors, ceramics, chemical catalysts, novel magnetic systems, thin films and interfaces, biological materials, and composites. They also apply neutron methods to nondestructive testing, in areas such as residual stress determination, texture measurements, and neutron radiography. This work involves more than 700 researchers from outside the division, working both collaboratively and independently. In order to maintain the NBSR at the forefront of neutron research, the group also develops new methods, applications, and instrumentation to serve broad national needs.

The Cold Neutron Project group has responsibility for the development of the Cold Neutron Research Facility (CNRF) as a national resource to provide cold neutron measurement capability to a broad research and development community, including materials science, physics, chemistry and biology, which is drawn from industry, universities, and government agencies. They are responsible for the development, operation and maintenance of new cold neutron sources, a network of eight neutron guides, fifteen experimental stations, and a full complement of ancillary equipment such as cryostats, furnaces and magnets. The CNRF, as a national facility, provides measurement capability to outside researchers, on the basis of scientific or technological merit of long-term programs or individual experimental proposals. The RRD staff support these researchers, as well as maintaining a NIST cold neutron research program. Many groups from outside the division are participants in the CNRF, including the Physics (PL) and Chemical Science and Technology (CSTL) Laboratories, Exxon Research and Engineering Company, IBM, Eastman Kodak, the University of Minnesota Center for Interfacial Engineering, the National Science Foundation, Sandia National Laboratory, the University of Maryland, the University of Missouri, and others.

There are a number of Independent Programs based at the NIST reactor. These programs are long term, make use of the unique capabilities of the NBSR, and are not directed by RRD. For example, the Nuclear Methods group (CSTL), develops and applies nuclear methods to problems in analytical chemistry. This group is responsible for most of the activation analysis work performed at the NBSR, including, materials analysis, characterization of SRM's, environmental studies, and other areas. They interact strongly with outside users, such as the EPA, FDA, Smithsonian, FBI, and Treasury, and are responsible for the development of the depth profiling, prompt gamma activation analysis and focussed neutron stations in the CNRF. The Neutron Standards and Dosimetry group (PL) carries out a program in neutron metrology, radiation standards, and calibration, in cooperation with a number of outside organizations, such as the NRC, Westinghouse, and EPRI. This group is also responsible for the development, operation and maintenance of the fundamental physics and neutron interferometry stations in the CNRF. The Polymers Division (MSEL) operates the 8 m SANS for advanced polymer research, primarily in the area of polymer blends. This program interacts with many other organizations, and serves a broad variety of industrial users. Other groups using the reactor for mission activities include the

Smithsonian Institution, the U.S. Army, FDA, and the University of Maryland. Participating Research Teams (PRTs), which are groups formed to develop specific instruments, are also independent programs. These programs are discussed in the appendix entitled, NIST **REACTOR: Summary of Activities, October 1994 through September 1995.**

Reactor Utilization:
During the first 11 months of FY95, the reactor was shut down in order to complete an extensive maintenance and upgrade program, as discussed in last year's report. The scheduled work included replacement of the main heat exchangers; replacement of other auxiliary heat exchangers; replacement of the control shim arms; replacement of the heavy water; refurbishment of the cooling tower; removal of the old cold source and bismuth tip; installation of the new liquid hydrogen cold source; and numerous smaller projects. In addition, visual inspection of the reactor vessel and reactor internals revealed that some BORAL shielding on the bottom of the refueling head was failing. This required complete removal of the top head, for the first time since the reactor went into service. While this head was removed, the entire refueling mechanism was overhauled, a difficult and unscheduled task that will improve the efficiency of the reactor refueling for many years to come. Another unscheduled task involved discovery of a cold vacuum leak in one portion of the hydrogen source after installation. Although the entire system had been tested at low temperatures, this leak did not show up until this test (the last before hydrogen was to be added). Repair of this leak caused additional down time. However, all of this work was completed successfully and the reactor was ready for operation by mid September, 1995. On September 25, the reactor power was increased to 10 MW after a careful approach to criticality, and extensive operator retraining and requalification. On September 26, liquid hydrogen was condensed into the cold source, and power was returned to 10 MW. Power was increased to 20 MW over the next week, and all systems were observed to function well (the hydrogen source performance is featured on the cover of this report). As a result of the work performed during the outage, all experiments were increased in performance by at least 4/3; experiments using cold neutrons improved by over a factor of 8. The reactor and associated equipment is now ready for steady uninterrupted operation for at least 5 years, until a major (3 month) outage will be required for control shim arm replacement.

As a result of the shut down, reactor utilization during this period was virtually zero. However, with the increased performance obtained from the enhancements, the lost time will be recovered quickly (in two months for cold neutron experiments!). In addition, all of the work performed directly serves the goal of extending reactor life beyond the current license expiration in 2004.

Virtually all of the experimental programs normally carried out at the reactor involve collaboration or cooperation with outside researchers from universities, industry or other government laboratories. This trend has continued and accelerated in the more recent past, both within the thermal neutron program and in the construction and operation of the CNRF (which was planned from the beginning as a national user facility). The number of participants using the reactor each year is considerably larger than those of any other major neutron facility in the United States, and accounts for more than half of all US research using neutron methods. At the

same time, reactor operation costs have been tightly controlled, making the NIST facility the most cost-effective in the world.

A formal user proposal system is successfully operating for the facilities of the CNRF, and scheduled experiments are underway, in response to the Call for Proposals issued late in FY95. In this mode, prospective users submit a formal written proposal for use of one of the available instruments. These proposals are mailed out for peer review, and the final decision on time allocations for the proposals are made by the Program Advisory Committee (PAC), which meets at NIST at least twice per year.

In addition to the formal outside user program for the CNRF, other modes of access to these facilities and to the other NBSR facilities are in place. Several of the CNRF instruments were built and are operated by Participating Research Teams (PRT's), which share in the cost of instrument development. In return, the PRT members receive 75 % of the available time on such instruments, while providing 25 % to the general community through the user program described above.

Most of the non-CNRF facilities at the NBSR are not made available through the formal user program, but—as previously stated—virtually all of the research done in the division involves collaboration with outside researchers. Many of the instruments were developed in a manner analogous to the PRT mode described above, and the experimental program is operated in the same manner. Several researchers are long term associates—for example, the group from the U.S. Army, which participated in the development of the BT-4 and BT-6 thermal neutron spectrometers has been stationed at NIST for more than 20 years. Other long term cooperative programs have been formed at various times. At present, these include the University of Maryland, Johns Hopkins University, and MIT among others. Similar arrangements with other universities and industrial laboratories are under discussion.

Several non-NIST programs, as well as non-division NIST mission activities are also served by the NBSR facilities in an as-needed mode for services such as irradiation, radiography, or materials characterization. In such cases, the research may be either collaborative or independent, and arrangements are made for each specific use on a case by case basis. Examples include the program with the Smithsonian Institution for autoradiography of paintings; the program with the FDA for characterization of foods or drugs; and activation analysis services for the FBI. In some cases, non-NIST researchers perform proprietary research using the facilities of the NBSR. In these cases, full cost recovery is required.

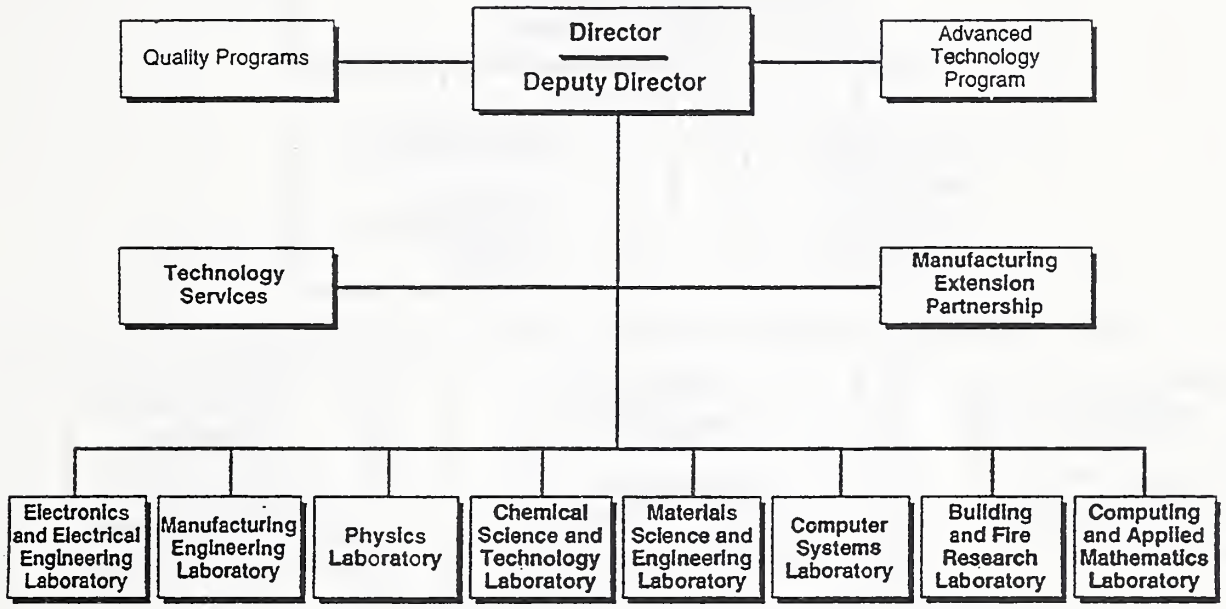
Thus, the interactions of the Reactor Radiation Division with the research community are extensive, varied, and growing. One element of change which should be noted is the growing trend towards university based research programs which directly serve industrial R&D needs. In the past year, at least 1/3 of university based research was of this general character. Examples include the University of Delaware (DuPont), MIT (Texaco), the University of Minnesota Center for Interfacial Engineering (over fifty affiliated companies), and Princeton (Boeing). This trend is expected to grow in the coming years, as more of the university based research effort is directed

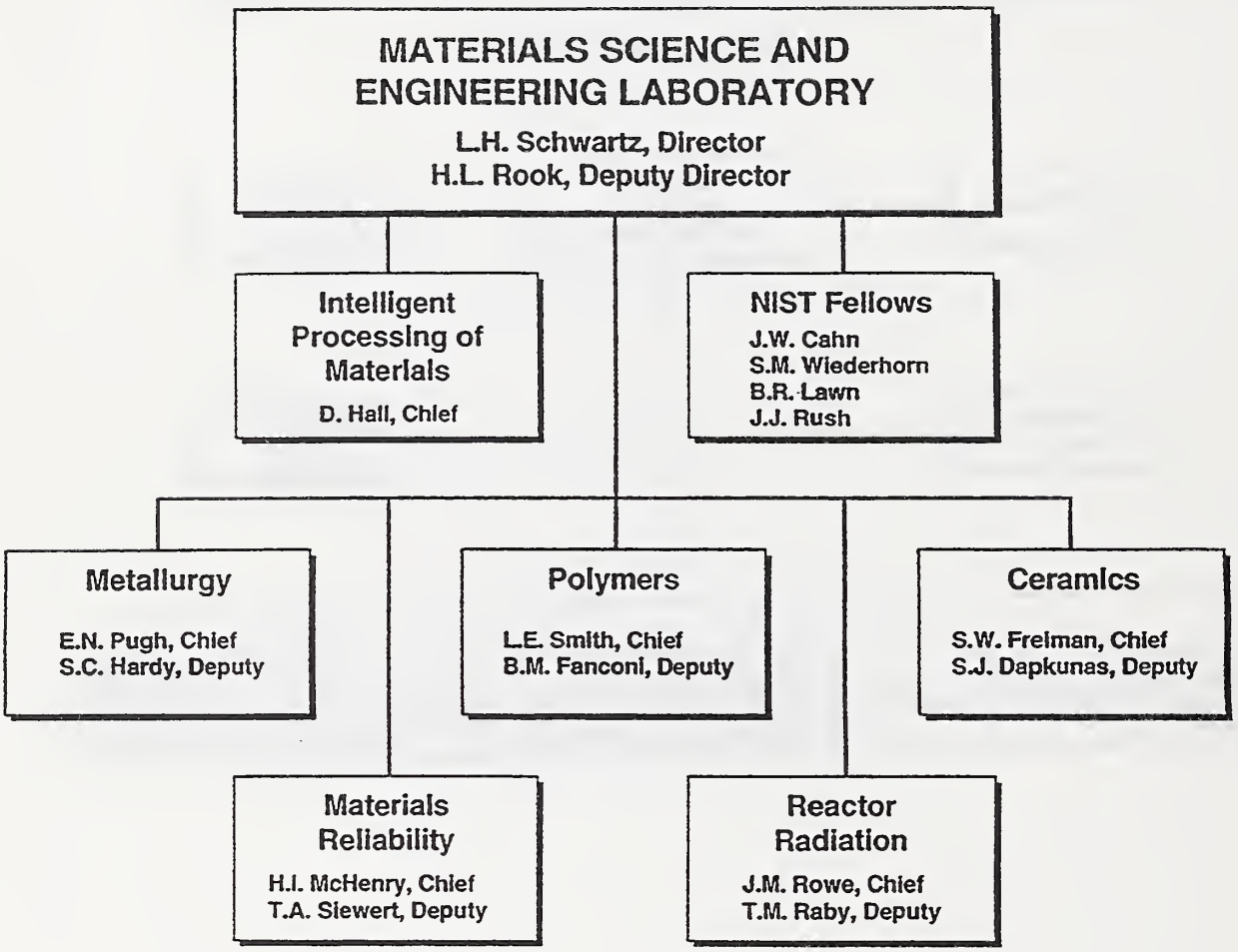
towards strategic national goals. The Division considers the development of new applications of neutron measurement technologies, and encouragement of their use by the broadest possible community in the United States, as one of its primary goals. In the future, additional mechanisms for interaction will undoubtedly arise, as will new uses of the unique resources provided by the NBSR and its associated experimental facilities.

Organization Chart and Research Staff

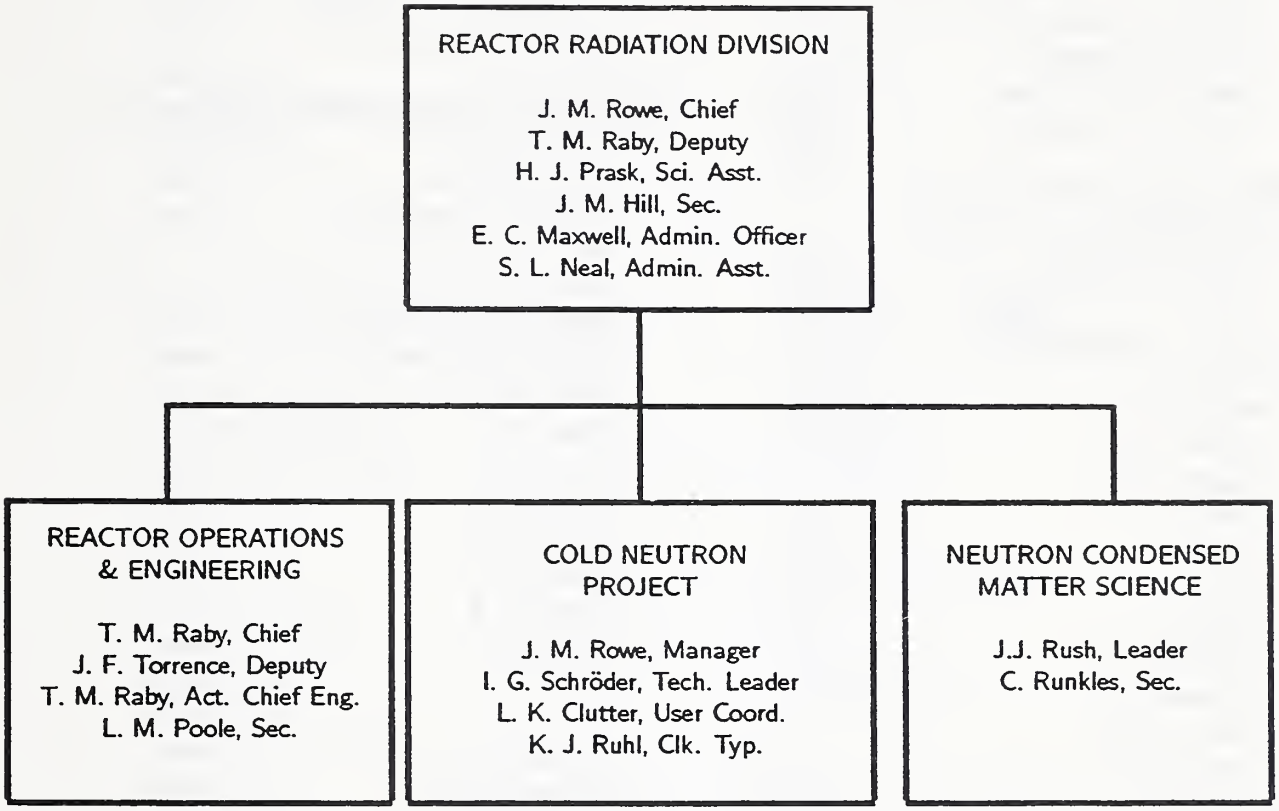
National Institute of Standards and Technology

Organizational Chart




MATERIALS SCIENCE AND ENGINEERING LABORATORY
L.H. Schwartz, Director
H.L. Rook, Deputy Director
Intelligent Processing of Materials
D. Hall, Chief
NIST Fellows
J.W. Cahn
S.M. Wiederhorn
B.R. Lawn
J.J. Rush
Metallurgy
E.N. Pugh, Chief
S.C. Hardy, Deputy
Polymers
L.E. Smith, Chief
B.M. Fanconi, Deputy
Ceramics
S.W. Freiman, Chief
S.J. Dapkunas, Deputy
Materials Reliability
H.I. McHenry, Chief
T.A. Siewert, Deputy
Reactor Radiation
J.M. Rowe, Chief
T.M. Raby, Deputy

Personnel Roster



The Division staff is organized formally into three groups, as shown in the chart above; however, staff are utilized where necessary, irrespective of group. Below the group level of organization, personnel are grouped into research teams according to their predominant interest. Once again, these groupings are not hard and fast; there are many overlapping interests. These teams, including long-term guest researchers, are shown in table 1. A number of "non-resident" Ph.D. students and continuing collaborators from universities and industry are not listed.

It should be noted that members of the Cold Neutron Project are included in the teams by scientific interest, even though in most cases, their predominant responsibility is for CNRF instrument building and/or operation. In fact, they have only 30% of their time given to the conduct of research, while 70% is dedicated to the facility. Likewise, many members of the Neutron Condensed Matter Science Group have sizable CNRF responsibilities, which may amount to as much as 1/2 time when needed.

Table 1. NBSR and CNRF Resident Staff

ADMINISTRATIVE - DIVISION 856

Rowe, J. M., Chief
Maxwell, E. C., Admin. Off.
Neal, S. L., Admin. Asst.
Hill, J. M., Secretary
Prask, H. J., Sci. Asst.
Barker, J. G., Matls. Sci.

REACTOR OPERATIONS & ENGINEERING

Raby, T. M., Chief
Torrence, J. F., Deputy
Poole, L. M., Secretary

Operations

Beasley, R.D.
Bickford, N. A.
Cassells, M. G.
Clark, F. C.
Dilks, H. W.
Flynn, D. J.
Guarin, E. L.
Lindstrom, L. T.
McDonald, M. J.
Mueller, W. W.
Myers, T. J.
Ring, J. H.
Sprow, R. P.
Stiber, R. F.
Toth, A. L.
Wilkison, D. P.
Wright, K. D.

Engineering

Raby, T. M., Act. Chief
Poole, L. M., Secretary
Beatty, J. A.
Brady, D. E.
Hall, K. D.
Heine, C. J.
Liposky, P. J.
Shuman, L.A.
Suthar, M.A.
Thompson, R. G.

Research Associates

Anderson, D. A. (FDA)
Billos, J. (Montg. College)
Cunningham, W. C. (FDA)
Olin, J.(Smithsonian)

NEUTRON CONDENSED MATTER SCIENCE GROUP

Rush, J. J., Leader
Runkles, C. L., Secretary
Berk, N. F.
Borchers, J. A.
Casella, R. C. (retired)
Copley, J.R.D.
Dura, J. A.
Drews. A. R.
Erwin, R. W.
Fulford, D. B.
Gehring, P. M.
Glinka, C. J.
Hammouda, B
Karen, V. L.
Krueger, S. T.
Lynn, J. W.
Majkrzak, C. F.
Maliszewskyj, N. C.
Mighell, A. D.
Neumann, D. A.
Orts, W. J.
Reznik, D.
Robeson, L. A.
Santodonato, L. J.
Santoro, A.
Satija, S. K.
Slawecki, T. M.
Stalick, J. K.
Toby, B. H.
Tobias, D. J.
Udovic, T. J.

Guest Scientists

Altorfer, F. B.
Choi, C. S. (retired)

Chang, Y.-T.
Clinton, T. W.
Huang, Q. Z.
Klosowski, P.
Mrose, M.
Nunez, V.
Prince, E.
Tobias, D. J.
Trevino, S. F.
Watanabe, T.
Yildirim, T.
Zhang. H.

COLD NEUTRON PROJECT
Rowe, J. M., Leader
Clutter, L. K., User Coord.
O'Connor, C. L., User Coord. (retired)
Hill, J. M., Secretary
Ruhl, K. J., Clerk Typist
Baltic, G. M.
Bostian, C. D.
Clem, D. L.
Clow, W. R.
Dickerson, W. E.
Fravel, D. H.
Gallagher, P. D.
Green, T. A.
Greene, G. C.
Heald, A. E.
Kamitakahara, W. A.
Knill, W. C.
Kopetka, P. H.
Kulp, D. L.
LaRock, J. G.
Layer, H. P.
Pierce, D. J.
Rinehart, M. J.
Rosov, N. S.
Rymes, W. H. (retired)
Schroder, I. G.
Thai, T. T.
Tobin, P. J.
Williams, R. E.

Guest Scientists
Allen, A. J.
Brand, P.C.
Brocker, C. W.
Christman, R.
Desrosier, F. L.
Lin, M. Y.
Moyer, J. J.
Nunes, A. C.
Wrenn, C. W.

NEUTRON INTERACTIONS - 846
Gilliam, D. M., Leader
Rhodes, S. E., Secretary
Mattiello, R., Secretary (retired)
Arif, M.
Boswell, E. W.
Dewey, M. S.
Eisenhauer, C. M.
Greene, G. L.
Grundl, J. A.(retired)
McGarry, E. D.
Nico, J. S.
Schwartz, R. B.
Thompson, A. K.

HEALTH PHYSICS -DIVISION 354
Slaback, L. A., Leader
Thomas, C. L., Secretary
Brown, D. R.
Campbell, C. D.
Cassells, L. H.
Clark, J. S.
Deardorff, G. E.
Fink, L. E.
Mengers, T. F.
Shubiak, J. J.

NUCLEAR METHODS GROUP-DIVISION 839
Greenberg, R. R., Leader
Wilson, J. M., Secretary
Becker, D. A.
Benenson, R. E.
Bishop, R. L.
Blackman, M. J.

Chen-Mayer, H. H.
Ciurczak, D. M.
Demiralp, R.
Downing, R. G.
Fitzpatrick, K. A.
Garrity, K. M.
Iyengar, G. V.
Koster, B. J.
Lamaze, G. P.
Langland, J. K.
Lindstrom, R. M.
Mackey, E. A.
Mildner, D.F.R.
Norman, B. R.
Paul, R. L.
Sharov, V. A.
Welsh, J. F.

Research and Engineering Staff

J. G. Barker	SANS instrumentation and research Microstructure of materials
N. F. Berk	Condensed matter theory Scattering theory for microstructure analysis Computer software for graphics and data analysis
N. A. Bickford	Reactor operations Reactor irradiations Reactor utilization
J. A. Borchers	Thin-film analysis Artificially modulated materials Magnetism
D. E. Brady	Electrical/electronic engineering Nuclear reactor instrumentation
J. R. D. Copley	Time-of-flight spectrometer development Neutron instrumentation conceptual design Condensed matter physics
W. E. Dickerson	Neutron scattering instrumentation Microcomputer interfacing Nuclear and engineering physics
A. R. Drews	Perfect Crystal Diffractometer Development Condensed Matter Physics
J. A. Dura	Combined molecular beam epitaxy and neutron reflectivity/ diffraction instrumentation Surface, interfacial, and epitaxial physics Metastable phases in artificial materials
R. W. Erwin	Magnetic materials Phase transformations Cryogenics
D. B. Fulford	SANS equipment development and maintenance Mechanical engineering
P. D. Gallagher	Neutron reflectometry instrumentation Interfacial phenomena in polymer systems and complex fluids Phase transitions and critical phenomena

P. M. Gehring	Neutron backscattering instrumentation Magnetic and structural phase transitions in disordered systems Dynamics of high T_c materials
C. J. Glinka	SANS microstructure of metals and porous media CHRNS project director Cold neutron instrument development
G. C. Greene	System and user software for cold neutron instrumentation Spectrometer and data acquisition systems interfaces
B. Hammouda	SANS from polymers, liquid crystals, and colloids Dynamics of polymers in solution Scattering from sheared fluids
A. E. Heald	Design engineering Neutron instrumentation Shielding
W. A. Kamitakahara	The CNRF guest researcher program Dynamics of disordered solids Condensed matter physics
V. L. Karen	Crystallographic database development Theory of lattices and symmetry Neutron and x-ray diffraction
P. Klosowski	Scientific data visualization Numerical computer modeling Data acquisition software and hardware
P. A. Kopetka	Mechanical engineering Cold source design Electro-mechanical systems
S. T. Krueger	SANS instrumentation Microstructure of materials Biological problems
J. G. LaRock	Mechanical engineering Neutron instrumentation design
H. P. Layer	Electronics and data processing Advanced instrumentation Fundamental physics
P. J. Liposky	Design engineering Nuclear systems and components

J. W. Lynn	Condensed matter physics Magnetic and superconducting materials Neutron scattering methods
C. F. Majkrzak	Condensed matter physics Polarized neutron scattering and instrumentation development Neutron reflectivity measurements
A. D. Mighell	Crystallographic database development Single crystal and powder diffraction Theory of lattices
D. A. Neumann	Molecular and layered materials Condensed matter physics Neutron and x-ray scattering instrumentation
W. J. Orts	SANS instrumentation Neutron scattering and reflectivity from polymers Polarized neutron small angle scattering
D. J. Pierce	Mechanical engineering Neutron instrumentation design
H. J. Prask	Residual stress measurement methodology Neutron NDE applications Neutron NDE instrumentation
T. M. Raby	Reactor operations Nuclear engineering Reactor standards
G. Reilly	Design engineering Nuclear systems and components
D. Reznik	Condensed matter physics Dynamics of High T_c materials Dynamics of fullerenes
N. Rosov	Spin echo techniques Phase transformations Magnetic materials
J. M. Rowe	Orientationally disordered solids Cold source development Cold neutron research and instrumentation
J. J. Rush	Catalysts and molecular materials Hydrogen in metals Inelastic scattering methods

L. Santodonato	Condensed matter physics Cryogenics
A. Santoro	Structure of electronic and structured ceramics Theory of crystal lattices Powder diffraction methods
S. K. Satija	Low-dimensional molecular systems Fractal aspects of microporous media Neutron reflectometry
I. G. Schröder	Cold neutron instrumentation development Nuclear and engineering physics Optical devices for neutron transport
T. M. Slawecki	SANS and reflectometry from polymers Complex fluid microstructure SANS instrumentation
J. K. Stalick	Neutron and x-ray diffraction Inorganic chemistry Crystal database development
M. A. Suthar	Design engineering Nuclear systems and components
J. F. Torrence	Reactor supervision Reactor maintenance Nuclear engineering
T. J. Udovic	Neutron time-of-flight instrumentation Properties of catalysts and adsorbates Hydrogen in metals
R. E. Williams	Cold neutron source development Nuclear engineering

Technical Staff

Reactor Operations

Richard D. Beasley
Mark G. Cassells
Forrest C. Clark
Howard W. Dilks
Daniel J. Flynn
Enrique L. Guarin
Larry T. Lindstrom
Michael J. McDonald
William W. Mueller
Thomas J. Myers
John H. Ring
Ricky P. Sprow
Robert F. Stiber
Attila L. Toth
Daniel P. Wilkison
Kevin D. Wright

Reactor Engineering

James A. Beatty
Keith Hall
Lynn A. Shuman

Neutron Condensed Matter Research

George M. Baltic
C. Douglas Bostian
David Clem
William R. Clow, Jr.
Donald H. Fravel
Thomas A. Green
Wayne C. Knill
Doris Kulp
Michael J. Rinehart
Lewis P. Robeson
William H. Rymes (retired)
Thuan T. Thai
Patrick J. Tobin
Robert H. Williams (retired)

Program: Neutron Facility Operation

Project Title: Reactor Engineering

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Technical Description:
This project provides mechanical and electrical engineering support for the operation, maintenance, and upgrade of the NIST Research Reactor.

Technical Objectives/Goals:
To ensure that the systems which make up the NIST Research Reactor are maintained in good operating condition in order to maximize utilization and efficiency, while protecting public safety.

Outcomes:
The facility will operate safely and reliably, minimizing routine maintenance downtime as well as unscheduled shutdowns. The reactor systems will support operation through 2004 (current license expiration) and beyond.

Accomplishments:
During FY95, the main reactor heat exchangers were replaced, along with much of the primary and secondary piping system. The new systems are designed to last through 2024. As a result of the extensive planning done by this project, the system replacement was done quickly and efficiently, mostly by in-house staff.

In the course of the shutdown for heat exchanger replacement, problems were discovered in the top refuelling plug of the reactor. The Reactor Engineering project staff analysed the problem alongside of Reactor Operations, and designed effective fixes and replacements. As a result, the refuelling system is now functioning better than it has for many years, and is in excellent condition for a further 30 years of operation.

The upgrade of reactor control room electronics continued during the period, with replacement of all annunciator panels. While the period, with replacement of all annunciator panels. While the prior systems continued to perform reliably, maintenance was becoming difficult as a result of component obsolescence.

For their outstanding performance during the shutdown, the members of this project received the Department of Commerce Silver Medal.

Outputs:
Main reactor heat exchangers replaced.
Reactor primary and secondary piping replaced where necessary.
Continued progress in reactor control panel upgrade.
All systems performing at or above expectations after reactor restart

Impact:
The cost of operation of the NIST Research Reactor remains below that of any comparable facility worldwide, primarily as a result of our excellent maintenance and upgrade system. The reactor has operated at better than 99 % reliability since restarting in September 1995, with no shutdowns attributable to our systems.

Highlights:
The Reactor Engineering Team received the Silver Medal of the Department of Commerce for their contributions to the cost-effective operation of the NIST Research Reactor as a major national facility. Among other accomplishments, the award recognizes their qualification of a new type of heat exchanger for use in the reactor, which resulted in savings of over $1 M in heat exchanger cost.

Project Title: Reactor Improvement

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Technical Description:
This project is designed to implement an ongoing modernization of the reactor and its experimental facilities, including reactor systems and components, reactor analyses, and experimental facilities.

Technical Objectives/Goals:
This project is designed to maximize the utilization of the reactor experimental facilities, and to prepare for a renewal of the reactor license for a further 20 years beyond its current expiration date of 2004.

Outcomes:
Two major outcomes are anticipated for this project: (1) the case for a license extension will be prepared and submitted to the Nuclear Regulatory Commission no later than 2003, and (2) the experimental facilities inside the reactor confinement building will be upgraded to state-of-the-art, providing enhanced capabilities and improved personnel radiation protection.

Accomplishments:
An interagency agreement for the design of new shielding drums at Oak Ridge National laboratory has been concluded, and design work has begun. This design will be the basis for four improved neutron spectrometers.

A new design for radiation control shutters has been completed, and is being implemented. A new remotely operated monochromator crystal system has been designed and built which will decrease experimenter radiation exposures in accordance with ALARA.

During the reactor shutdown, several improvements to critical systems have been made, several changes to simplify maintenance have been incorporated, and the beginning of data collection necessary for preparation of a new safety analysis report, essential to relicensing has begun.

Outputs:
Several new designs have been completed, and some have been fabricated. One of these, the

monochromator changer system, has been installed at three instruments, and will result in lower radiation exposures to experimenters in the coming year. Several major upgrades to reactor plant systems have been implemented, which are all designed to last at least thirty years.

Impact:
It is too early in this project to have demonstrated impacts, as this is a long range activity.

Project Title: Reactor Operations

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Technical Description:
This project is responsible for the safe, cost-effective operation of the NIST Research Reactor.

Technical Objectives/Goals:
To protect the safety of the general public and NIST staff while operating the NIST Research Reactor in a cost-effective manner in order to serve national neutron measurement goals.

Outcomes:
Except during large scheduled maintenance outages, the reactor will operate at least 70 % of real time, and will operate at 99 % reliability (Percentage of scheduled time actually operated). The operation of the reactor will satisfy all regulatory requirements, and in all cases, shall attempt to exceed such requirements.

Accomplishments:
During FY95, the reactor underwent major maintenance as part of a scheduled outage. Most of the actual work (excepting rigging and welding) was performed by Reactor Operations personnel, thus resulting in direct savings of funds. However, beyond this obvious savings, there were additional benefits which derived from the use of operating staff for these maintenance and upgrade operations. These include better knowledge by operating staff of plant configuration; better knowledge by those performing upgrades of operations needs (a good example of this benefit is the observation of the origin of a long existing problem which could and did lead to dropped fuel elements during overhaul of the refuelling head); and better morale of operations staff, who are involved in all parts of the facility.

The reactor operations staff completed a wide range of facility improvements, including upgrades to the thermal shield system, improved operations of the refuelling system, needed maintenance of the cooling tower, improved filtration capabilities for secondary water, and many other small improvements.

Outputs:
During September, 1995 the reactor was successfully returned to full 20 MW power operation. As a result, all facilities improved by a factor of 4/3. In addition, those instruments served by the

cold neutron source gained a further factor of between 4 and 6 in intensity.

Impact:
The NIST Research Reactor continues to have the lowest operating cost of any comparable reactor worldwide. This is universally recognized, and was explicitly acknowledged by a Department of Energy study submitted to the Secretary of Energy during 1995. This study showed that the NIST reactor is operated safely and highly cost-effectively, providing the basis for the foremost neutron research facility in the United States.

Highlights:
The NIST Research Reactor resumed operation in September, 1995, reaching 10 MW on September 25. Following a week of testing at 10 MW, power was increased to 20 MW on October 5, 1995.

Project Title: Cold Neutron Research Facility Operations

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Technical Description:
This project encompasses the maintenance and operation of the cold neutron measurement instruments located in the Cold Neutron Research Facility (CNRF), as well as provision of scientific and technical assistance to researchers using the facility.

Technical Objectives/Goals:
To operate the CNRF as a major national resource for researchers from industries, universities and other government agencies, providing unique measurement capabilities relevant to materials science, chemistry, physics, biology, and engineering.

Outcomes:
The facility will be at the forefront of neutron measurement capabilities worldwide, and will serve over 600 participants each year of operation. It will provide the research tools needed by the national research community, as indicated by the continuing high utilization of the facilities.

Accomplishments:
In the last full year of operation, more than 150 formal proposals to use the facility were received, in addition to a large number of interactions based upon the activities of the CNRF staff in collaboration with outside researchers, and the activities of participating research teams. The experiments performed involved more than 600 researchers. The CNRF was broadly recognized as the foremost U. S. facility for cold neutron research.

During this operating period, the cold source, guides and instruments were fully available for more than 95 % of scheduled time. During the extended reactor shutdown for maintenance, a new cold source was installed, three additional neutron guides were installed, the entire guide network was reworked and improved, the SPINS instrument was completely rebuilt, and the SANS instruments were upgraded.

Project researchers have developed a new standard for data interchange among facilities, have upgraded data acquisition software, and have organized several workshops, meetings, and a summer school.

Outputs:
Details are given in measures of performance.

Impact:
The NIST CNRF alone (excluding other reactor facilities) served more researchers in its last full year of operation than any other U. S. neutron facility. CNRF facilities were used in a CRADA with an ATP award winner to develop a new neutron and X-ray focussing device in a shorter time than could have been accomplished otherwise. The NSF supported CHRNS facility was provided with the neutrons needed to meet their goals, as evidenced by the recent renewal of the operating grant for a further five years. The participating research teams using the facility (including Exxon Research and Development, Texaco, IBM, University of Minnesota, Johns Hopkins University and others) have continued their active support of the facility as a result of the importance to them of the capabilities provided.

Project Title: The Center for High Resolution Neutron Scattering (CHRNS)

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Technical Description:
The Center for High Resolution Neutron Scattering (CHRNS) was established by joint agreement between the National Science Foundation and NIST in March, 1989, to provide state-of-the-art cold neutron scattering instrumentation for use by the general scientific community at NIST's Cold Neutron Research Facility (CNRF). CHRNS presently comprises a 30 meter, high resolution, Small Angle Neutron Scattering (SANS) instrument and a Spin Polarized Inelastic Neutron Scattering (SPINS) spectrometer. The SANS instrument has been in operation since September, 1992, and has become the most heavily utilized instrument at the CNRF by visiting researchers. The instrument is used by university, government and industrial researchers in materials science, chemistry, biology and condensed matter physics to obtain microstructural information (on a size scale from 1 to nearly 500 nm) on materials such as synthetic and biopolymers, metal alloys, ceramics, porous media, structured fluids and gels, biological macromolecules and magnetic materials that is characteristic of the bulk materials and, in many cases, cannot be obtained by other techniques. The SPINS instrument has recently been completed (August, 1995) and will provide condensed matter physicists and physical chemists with a powerful probe of dynamical processes in materials, including spin-dependent cross sections, with energy resolution from 0.02 to 2 meV (time scales from 3×10^{-11} to 3×10^{-13} sec). Through the use of innovative neutron optical components, SPINS will provide enhanced sensitivity for measuring low energy magnetic, vibrational and rotational excitations in ordered and disordered systems. The NSF support for CHRNS was renewed for five years in March, 1995, through agreement No. DMR-9423101.

Technical Objectives/Goals:
Some of the principal technical objectives and goals for the 5-year period of the current NIST/NSF cooperative agreement are:

- To maintain the high level of use and reliability that has been achieved with the CHRNS SANS and to provide support for users from a scientific staff with interests and expertise across a broad range of disciplines.

- To continue to improve the measurement capabilities and ease of use of the CHRNS SANS instrument. Specifically, SANS measurements with polarized neutrons will be made routinely

available. This capability is presently not available at any high resolution SANS instrument in the U.S.

- To expand the variety of sample environment equipment that is available to users of both the CHRNS SANS and SPINS instruments. For SANS we would provide the added in-situ capability to make simultaneous rheological measurements on complex fluids and soft solids under shear, to mechanically deform elastomers, to apply steady or pulsed electric and magnetic fields, and to measure polymer melts, solutions, and complex fluids under pressure up to 1 kbar for temperatures up to 200 C, for example.

- To develop, over a two year period, a Multiple Perfect Crystal Diffractometer (MPC-SANS) as a new user oriented facility for very high resolution SANS.

- To hold an annual one week "Summer School on SANS and Neutron Reflectometry from Submicron Structures" at the CNRF, jointly supported by the CNRF and CHRNS, that would acquaint students and scientists with the capabilities of these techniques and provide actual hands-on experience using the CNRF and CHRNS facilities.

- To implement for routine use on the SPINS spectrometer the supermirror transmission polarizers and energy-dependent Drabkin-type spin flipper that will give this instrument its unique capabilities. In addition, a horizontally focusing, multi-crystal energy analyzer, that is interchangeable with the Drabkin spin-dependent analyzer, will also be available that will significantly enhance the sensitivity and efficiency of the instrument for many types of low energy dynamical studies of condensed matter systems.

- To establish an active users' program on the SPINS spectrometer.

Accomplishments:

- The SPINS spectrometer became fully operational as a cold neutron triple-axis spectrometer in FY95. The first full scale supermirror transmission polarizer, that will provide the spectrometer with full polarization analysis capability, has also been completed.

- Through a collaboration with the Physics Department of Johns Hopkins University, a focusing multi-crystal energy analyzer has been built and successfully tested on the SPINS spectrometer. This device will provide high energy resolution with high intensity, through relaxed momentum resolution, for measurements of low energy excitations in many types of condensed matter systems.

- The first annual, NIST/NSF sponsored, one week summer school on neutron scattering techniques was held at the CNRF on August 28-31, 1995. This year's course, entitled Small Angle Scattering and Reflectometry from Submicron Structures consisted of morning lectures by CNRF staff and afternoon training sessions on the use of the CNRF's SANS and reflectometry instruments. The course was attended by 27 students and junior faculty.

- A polarizing neutron guide, to enable SANS measurements with polarized neutrons, has been constructed for the CHRNS SANS instrument. The device is designed to polarize a wide beam (4 x 5 cm^2) over a range of wavelengths (5-15 Å) by preferentially reflecting neutrons of one spin state out of the beam. The polarizing guide will provide greatly enhanced sensitivity to magnetic microstructure; a capability that is not available at any other SANS facility in the U.S.

- A hardened steel cell with sapphire beam windows has been constructed for *in-situ* SANS measurements on "soft" materials, such as polymers, gels and complex fluids, at pressures up to 1.2 x 10^5 kPa (1.2 kbar) and temperatures up to 200 °C. The cell's hydraulic pressure pump and heating element are interfaced to closed-loop controllers that are programmable via menu-driven software running on a PC linked to the SANS instrument's data acquisition computer. With this system, pressure and temperature are continuously monitored and can be changed automatically between scattering measurements. The cell has been used successfully to observe compressibility effects at the molecular level in polymer blends and block copolymers and is expected to become increasing utilized to explore pressure-dependent changes in the morphology and phase behavior of a wide variety of systems including membranes, colloids and liquid crystals.

- Since the CNRF resumed operation in October, 1995, the user program on the CHRNS SANS instrument has been back in full swing, with demand for beam time exceeding capacity by more than a factor of two. The March 1995 Call for Proposals produced 52 SANS proposals requesting 235 days of beam time. The first inelastic scattering experiments have been done on the SPINS spectrometer and a small but growing user community is developing for that instrument as well.

Outcomes:
CHRNS will continue to be an integral part of the CNRF at NIST's research reactor. The addition of a Multiple Perfect Crystal (MPC) Diffractometer for very high resolution SANS will enable CHRNS users to use neutrons to characterize materials microstructure over an unprecedented 3 1/2 orders of magnitude in Q-range (corresponding to feature sizes from 1 nm to nearly 10 microns). Similarly, the SPINS spectrometer, with its high resolution and full polarization analysis capability, will provide a capability for measuring low energy magnetic vibrational and rotational excitations in ordered and disordered condense matter systems that exists at only a few centers in the world and nowhere else in the U.S.

Outputs:
There have been over 95 technical publications by users of the CHRNS instruments from the commencement of operation of the CHRNS SANS instrument in September, 1992; 44 technical publications in FY95.

Impacts:
Since becoming operation in late 1992, the CHRNS SANS instrument has become the most

heavily utilized instrument by visiting groups of any instrument at the CNRF. Over 160 visiting scientists from universities, government and industry used the CHRNS SANS instrument from September, 1992, until May, 1994.

Highlights:

The NSF recently completed a thorough review of the progress of CHRNS and as a result has agreed to continue and expand its support for CHRNS. Under the terms of a new, five year cooperative agreement (commencing March, 1995), the NSF will continue to provide support for the staff and equipment needed to operate the SANS and SPINS instruments as user-oriented facilities, and will, in addition, provide one-half of the support required to build and operate a new multiple perfect crystal diffractometer (MPC) for very high resolution SANS. The MPC-SANS will extend the upper size limit of the 30 m SANS instrument by more than one order of magnitude, to nearly 10 microns, to provide overlap and complementarily with optical techniques. Up to 75% of the beam time on the CHRNS instruments is allocated based solely on the scientific merit of submitted proposals that undergo external peer review.

Three out of four planned modes of operation are now available for the CHRNS Spin Polarized Inelastic Neutron Scattering (SPINS) spectrometer. SPINS can now operate as a conventional cold neutron triple-axis spectometer with variable incident and final energy; as a cold neutron spectrometer with an innovative multi-crystal focusing energy analyzer which increases intensity while preserving energy resolution; and, as a cold neutron polarized beam spectrometer with full polarization analysis as a result of the successful development of wide beam, transmission supermirror polarizers whose combined transmission and polarizing efficiencies are the best in the world.

Project Title: CNRF Engineering

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Technical Description:
This project provides detailed design, analysis, and engineering support for the development, construction, and installation of advanced neutron scattering instrumentation.

Technical Objectives/Goals:
In collaboration with the CNRF scientific staff, this project will provide detailed designs for neutron scattering instrumentation and provide engineering support during all phases its development, construction, and installation.

Accomplishments:
A large number of engineering projects were underway in FY95 due to the continued, planned outage of the reactor for the installation of the new cold source and for the upgrades to the reactor cooling system. Especially noteworthy, of course, was the completion of the new, liquid hydrogen cold source and its installation into the reactor beam tube. This major effort drew heavy support from the CNRF engineering project. Paralleling the improvements to the cold source was the design and installation of the remainder of the cold neutron guide network which transmits the intense neutron beams to the Guide Hall: All seven neutron guides were installed in the confinement building and through the confinement penetration casings into the Guide Hall. This installation featured a new vacuum casing design which allows the guides to be kept under vacuum without mechanical stress on the glass walls of the guides. Substantial improvements were made in the design of the biological radiation shielding around the guides.

Work on specific neutron instrumentation included substantial progress on the design and installation of five inelastic spectrometers in the CNRF. The final stages of the installation of the re-designed SPINS triple axis spectrometer was completed on NG-5. The final design stage of the High-Flux Backscattering Spectrometer (HFBS) was completed and major progress was made on the fabrication of most of its components. Work was begin on the design for the precision epoxy floor for the neutron Spin Echo spectrometer. This instrument will utilize an air pad system to move the 1 ton magnetic guide field coils. The Disc Chopper Spectrometer (DCS) sample chamber design was completed and fabrication begun. The neutron guide optical filter for this instrument was installed as part of the guide installation effort. Other CNRF projects this year included: modification of the NG7 SANS detector

vessel, automation of the presample flight path for the NG7 SANS, and redesign of the NG7 Reflectometer.

Outputs:
The demonstrated outputs from this project include:

1. The new liquid hydrogen cold source has been installed and awaits only final testing to evaluate its performance.

2. The remaining three neutron guides (NG-1, NG-2, and NG-4) have been installed in the confinement building. This project includes installation of the in-pile glass, vacuum casings, shutter mechanisms and biological shielding.

3. The design stage for the backscattering spectrometer is complete. Most of the major components are now either ready or being fabricated.

4. The design for the DCS sample chamber is in the final stages.

5. The neutron spin echo spectrometer is in the final fabrication stage. Design and testing has started on the "tanzboden", the special floor required for the instrument air pads.

6. Final installation of the re-designed SPINS triple axis spectrometer at NG-5.

7. Final installation of several modifications to the two 30m SANS machines, including: modification of the NG-7 detector vessel, relocation of the NG-3 filter into the guide hall, and several improvements aimed at automating various functions of the instrument.

8. Installation of a redesigned elevator on the NG-7 Reflectometer allowing for complete, automated motion over the entire design range.

Outcome:
The completion of the design, installation, and testing of the described engineering efforts will provide the CNRF with neutron scattering measurement capabilities that will exceed those available anywhere in the United States and be competitive with those available anywhere else in the world in terms of available flux on sample, resolution, or maximized signal to background ratios.

Program: Neutron Characterization

Project Title: Surface and Interfacial Studies

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Technical Description:
The principal purpose of this project is to characterize the microscopic chemical and magnetic structures of thin films and multilayers from both specular and nonspecular neutron reflectivity and diffraction measurements. The systems studied are of interest to physicists, chemists, materials scientists, and biologists.

Technical Objectives/Goals:
This research project is designed to assist in industrial as well as academic efforts to more completely understand those properties of matter which are correlated to microscopic structure at interfacial boundaries.

Outcomes:
Significant structural information can and has been obtained for a number of specific thin film and multilayer systems including;

1. polymers (especially relating to diffusion and adhesion);
2. magnetic superlattices (particularly those displaying a "giant" magneto-resistance (GMR) effect which have more immediate potential technological applications);
3. electrode materials (with applications to batteries and corrosion resistance);
4. high-temperature superconductors (measurement of magnetic penetration depth, an important property for applications involving the generation of magnetic fields);
5. biological membranes (lipid bilayers and protein adsorption);
6. Langmuir-Blodgett films (for a variety of scientific and technological uses spanning a variety of different disciplines).

Accomplishments:
Two state-of-the-art neutron reflectometers are now in operation contributing to the solution of interfacial structural problems at the forefront of research in a variety of scientific disciplines.

The BT-7 thermal neutron reflectometer is capable of providing polarized incident neutron beams with polarization analysis of the scattered radiation. The NG-7 reflectometer located in the CNRF or guide hall utilizes longer wavelength neutrons emanating from the liquid hydrogen moderator. The horizontal sample geometry of this instrument also makes possible the

measurement of reflectivities from gas-liquid boundaries. Both instuments can be equipped with position sensitive detectors for more efficient nonspecular scattering measurements. The NG-7 instrument is run by a participating research team which includes IBM and the University of Minnesota in collaboration with NIST MSEL staff. A fraction of time is also reserved for general users whose proposals are approved by a panel of referees.

Outputs:
A total of 47 technical papers were published in the open scientific literature, and both invited and contributed presentations were given at international conferences.

Impact:
Neutron reflectivity studies performed at NIST continue to provide essential microscopic structural information about interfacial regions in numerous materials of interest to a diverse community of scientists in both academic and industrial research, from polymers and biological membranes to synthetic magnetic superlattices and thin film superconductors. For example, neutron reflectivity studies of commercially produced polarizing and nonpolarizing "supermirror" reflecting devices for neutrons have already contributed to patents awarded for their manufacture (Osmic Company, Troy Michigan).

Highlights:
Although the reactor was not in operation this past year, considerable progress was made in understanding and reporting on experiments performed prior to the shutdown and in developing data analysis methods. Some of the highlights regarding this work are described here.

Observation of Two Length Scales Above T_N Holmium Thin Film
The last several years have seen a renewed interest in high-resolution x-ray and neutron scattering studies of the critical behavior exhibited at magnetic and structural phase transitions. A surprising result has been the discovery of additional scattering, distinct from the normal critical scattering, at temperatures above the critical temperature in various perovskites, rare earths, and actinides [1-4]. This additional scattering can be characterized by a single, temperature-dependent length scale that is about ten times larger than that of the normal critical fluctuations[1,2]. The scattering appears to be located in the near-surface volume, or `skin', of the sample[3], but to a depth that varies from at least several thousand Angstroms in the actinides, to as much as 100-200 ~ μm in terbium [3,5]. High-resolution measurements on terbium and $SrTiO_3$ have shown that the additional scattering is absent in the crystal interior [6], whereas recent studies of UO_2 have shown that the additional scattering may be induced when the surface is mechanically roughened [4]. These results indicate that the additional scattering is not necessarily a bulk property, and that it might originate with defects or random strains which occur at the surface when the samples are grown and/or polished.

The physical origin of the additional scattering remains unclear. Early studies suggested that the second length scale might be associated with critical fluctuations because the linewidth went to

zero at T_c . This behavior was interpreted in terms of a heuristic argument due to Imry and Wortis who showed that for a first-order transition it is possible to have large-scale local fluctuations from a lower temperature phase into a higher temperature phase occurring around defect sites, provided that the energy gained balanced the cost of producing a domain wall [7]. However, inelastic neutron experiments on terbium and holmium, employing energy resolutions down to 2 μeV full width at half maximum (FWHM), have yet to resolve a finite energy width for the additional scattering observed in these systems [5]. It is therefore unclear whether the additional scattering can be identified with critical fluctuations. Consistent with these findings, the measurements of the discontinuous magnetic ordering transition of UO_2 have raised the possibility that the additional scattering might correspond to static magnetic order within finite-size domains [4]. Much more recently, Altarelli and coworkers have shown that long-range random strains can induce a cross-over to a ``disordered" fixed point in second-order transformations [8].

The present experiments were undertaken to determine whether or not the additional scattering exists in thin rare-earth films. These systems make interesting candidates for studies of the two length scale problem because a significant strain can exist in the film as a result of the lattice mismatch at the film substrate. Moreover, the strain can be tuned continuously by varying the size of the lattice mismatch. In light of the previous work, it seemed reasonable to wonder whether the second length scale could exist alone in a rare-earth film that is thin compared to the ~100 μm thick skin seen in terbium, or whether the two length scales would coexist.

Our neutron scattering measurements were performed on the NBSR BT-7 reflectometer located at the National Institute of Standards and Technology. The sample used in our measurements is a 1 μ thick (10,000 Å) Ho(001) film grown on a sapphire (Al_2O_3) substrate. A 2,000 Å Nb(110) buffer layer was deposited first onto the substrate, followed by a 2,400 Å Y(001) seed layer, then the holmium film, and finally a 30 Å Ycap layer. The sample measures 2 cm on a side. We note that this film has a holmium thickness that is two orders of magnitude smaller than the thickness associated with the second length scale measured in two different single crystals of terbium [3,5,6].

The Neutron scattering profile obtained by scanning the transverse-momentum transfer q_T across the $(0,0,\tau)$ position at $T_N + 0.24$ K cannot be described by a single line shape such as a Lorentzian or Gaussian function of q_T. Instead, the magnetic scattering exhibits both a narrow and a broad component. This is the first such observation in a thin film system, and clearly indicates the presence of two length scales.

That the two components coexist in this thin film sample is interesting in light of the recent x-ray and neutron measurements of Hirota *et al.* on several different samples of $SrTiO_3$ [10]. This study concluded that, depending on the particular sample studied, the amplitude of the broad component is either weakened or vanishes in the so-called `skin' region, this being at least 3 μm thick. By comparison, the holmium thin film used in the present study is only 1 μm thick, some two orders of magnitude smaller than the 100 -- 200 μm thickness measured in two different

samples of bulk terbium [5,6]. Assuming a model in which the narrow component occupies an increasingly higher fraction of the near-surface volume the nearer one probes the surface, one would expect the scattering intensity from the broad component to diminish. In fact, the q_T-integrated intensity of the broad component is nearly twice that of the narrow component. These results are significant because they suggest that, at least over a thickness of 1 μm, the two length scales coexist in roughly equal proportion.

The linewidths of both the broad (Lorentzian) and narrow (Lorentzian squared) component were determined at several temperatures after deconvolving the instrumental resolution function. The limited number of data points prevents an accurate determination of the exponent ν, which governs the temperature dependence of the respective correlation lengths. However, the widths of the broad component are of order 0.008 $Å^{-1}$, and agree well with those measured in bulk holmium by Thurston t *et al.* in this temperature range [2]. We therefore have some confidence in associating this scattering with that from the normal critical fluctuations. The widths for the narrow component were difficult to determine precisely because of the rather large mosaic spread of the holmium thin film (relative to that of typical bulk samples.) Nevertheless we can state that they are at least 10 to 20 times smaller than those of the broad component, indicating a much longer length scale. These values are also in agreement with those reported by Thurston *et al.* in bulk holmium [2]. This indicates that the normal critical fluctuations of the order parameter do coexist with the additional scattering responsible for the second length scale.

Many researchers now associate the presence of a second length scale with strain in the near-surface volume of the sample. In the present case, the strain is different from that in bulk crystals as the film is clamped by the epitaxial growth on the yttrium substrate. The observation of two length scales in a 1 μm thick holmium film places constraints on the nature of the skin referred to in the terbium studies as well as on the actual morphology of the coexistence of the two length scales. Since strain/defects represent quenched disorder, they have a static distribution. It then seems reasonable to speculate that only *one* length scale will exist locally, regardless of what microscopic mechanism produces the second length scale. In this context, the present results are interesting because they imply that the volume fractions that carry the two respective length scales are in some sense mixed together, at least over a 1 μm scale, and that the concept of a skin that carries just one component is not applicable to holmium (or if so, then it is much smaller than 1 μm in extent). It would be interesting to study holmium thin films sandwiched between a series of identical Y/Lu alloy seed and cap layers, thick enough to clamp the thin film basal plane lattice spacing. This would allow one to tune the holmium lattice strain in a continuous fashion, possibly through a point in which the basal plane lattice mismatch between the holmium film and the Y/Lu layers is zero. This would result in a nominally zero strain environment and could provide an extremely interesting test of the effect of strain in producing the unusual second length scale. We plan to pursue such studies in the near future.

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Single Lipid Bilayers and Biominetic Membranes Supported on Planar Substrates

In a continuing collaboration with scientists from the NIH, the structure of single phosphatidylcholine bilayers is being studied using neutron reflectivity and atomic force microscopy (AFM). AFM measurements performed by NIH scientists provided an independent measure of total lipid coverage on the substrate and also confirmed that the lipid covers the substrate and also confirmed that the lipid covers the substrate in uniformly-distributed patches 100-500 Å in diameter. This additional information made it possible for us to refine our scattering length density profiles, which were initially represented by model-independent parametric B-splines and then more quantitatively by histogram functions based on the theoretical lipid composition. We were able to conclude that our measurements were sensitive to 1-2 Å changes in total bilayer thickness. However, details of structural changes within the bilayer were limited to 5-10 Å resolutionun der the available experimental conditions (1). Plans to move the reflectometer from the reactor confinement building to the guidehall will allow us to take advantage of the documented higher flux of the new cold neutron as well as a lower overall background. Thus, signal-to-noise is expected to improve by at least a factor of 10, allowing us to measure details within the bilayer at the 1-2 Å level.

Preliminary x-ray reflectivity measurements have been made on self-assembling phopholipid/alkanethiol biomimetic membranes supported on planar substrates as a result of a new collaboration with scientists from NIST's Biotechnology Division. These hybrid bilayers membranes (HBMs) are more robust than lipid bilayer membranes and they can support functional membrane proteins to emulate biological functions. Thus, HBMs are commercially significant for a number of applications including biosensors, tissue engineering, bioelectronics and biocatalysis. Since HBMs can be easily fabricated on substrates such as quartz or silicon which have been coated with a layer of gold, they have practical applicability to industrial-scale use. Initial x-ray reflectivity experiments have been aimed at confirming that sufficiently smooth gold layer can be deposited on large (> 10 cm diameter) silicon substrates and that uniform HBMs can be formed on these larger surfaces with a high degree of surface coverage.

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Capillary Condensation in Thin Polymer Blend Films
Neutron reflectivity (NR) experiments were conducted to study concentration profiles in thin polymer blend films of a classic lower critical solution temperature system (LCST), deuterated Polystyrene (dPS, Mw = 443k) Polyvinylmethylether (PVME, Mw = 84k) in the vicinity of the bulk two-phase boundary. These studies are aimed at mapping out phase boundaries in thin films of polymer blends, and the effects of surface interactions and finite size on the phase boundary. For these studies, thin films of dPS/PVME were spun-cast from toluene solution of the blends onto passivated Si wafers, and the NR measurements were performed in-situ at elevated temperatures under high vacuum. Temperature jumps of 10 °C or less allowed for short equilibration times between scans. The films were allowed to equilibrate for an hour at each temperature (repeated NR scans were performed in this interval to ensure that the reflectivity was stable after an hour) before measurements were taken. The film thicknesses ranged between 100 to 2500 Å and encompassed several different dPS compositions ranging between 0 to 100% dPS.

It has been observed that the critical edge for total reflection shifts to a significantly higher wavevector value at higher temperatures, indicating an aggregation of dPS within the film, as one might expect in a phase separation process.

Neutron Reflection Study of Polymer Brushe in Contact with a Mixture of Solvents
Further insights about the temperature dependence of the transverse concentration profile within the film were obtained by fitting the reflectivity to model profiles subject to constraints of mass conservation. In general, it can be seen that the lower surface tension component PVME strongly segregates preferentially to both the air and the silicon surfaces (capillary condensation), while the dPS is enriched in the middle of the film. As the two phase boundary is approached by increasing the temperature, these features accentuate, i.e. further enrichment of dPS occurs within the film (which tends to shift the critical edge to progressively larger values) accompanied by more condensation of PVME to the boundary walls of the film. Quantitatively however, at the highest temperature only, the density profile obtained from neutron reflectivity measurements shows very different behavior of PVME at the air/film interface as compared to the back Si/film interface. While the PVME/Si interface remains sharp, the relatively unconstrained film/air boundary has broadened enormously. Complementary X-ray reflectivity measurements performed under similar conditions indicate an abrupt physical roughening of the air/film interface at these temperatures. That this phenomena was a consequence of phase separation was verified through optical microscopy measurements which clearly reveal a laterally phase separated structure for annealed films at and above these temperatures, consistent with the NR and X-ray measurements. A more complete study of capillary condensation and phase separation induced surface roughening phenomena in dPS/PVME blend films of other compositions and thicknesses is currently in progress.

Neutron reflection was used to study the swelling of densely end-grafted polymer chains ("brush") in contact with a binary mixture of low-molecular weight solvents. These studies are motivated by theoretical considerations that predict an interesting variety of brush density profiles depending on the solubility of the brush polymer in the two solvents. One may even expect a partitioning of

different solvents (preferential solvation) within the brush thus providing novel chromatographic prospects for practical applications. In addition, these studies offer the possibility of studying cosolvancy effects, i.e. a mixture of solvents at certain compositions has the synergetic effect of being a better solvent than either components.

For this study, the brush layer comprised of densely grafted monodisperse trichlorosilane end-functionalised deuterated polystyrene (dPS) chains of molecular weight 85,000, end-tethered to the surface of a polished 10 cm diameter thick silicon wafer utilizing silanol chemistry. As prepared, the dry brush in air had a nominal thickness of approximately 160 Å ,measured by X-ray and neutron reflectivity. Neutron reflectivity measurements were performed on the NG-7 reflectometer utilizing a wavelength of 4.1 Å with the incident neutron beam penetrating through the silicon slab at grazing angles. The specularly reflected beam from the Si/dPS-Solvent mixture interface was picked up by a shielded He_3 single detector. A series of neutron reflectivity curves from the dPS brush in contact with regular solvent mixtures of cyclohexane/toluene, with measurements taken over the full range of compositions, ranging from pure cyclohexane to pure toluene are characterized by decaying oscillations corresponding to the brush layer height, h $\sim 2\pi/q^*$ where q^* is the position of the first minima in the oscillations corrected for refraction effects. The relative damping of these oscillations reveal details about the analytical form of the brush density profile as the solvent mixture quality is varied. In general, the poorer the solvent quality, the more collapsed is the brush and the oscillations are less attenuated. While cyclohexane is a marginal solvent for polystyrene, toluene is an athermal good solvent that strongly swells each polymer chain within the brush layer. As a consequence of the strong intra and inter chain "excluded volume" interactions, the brush stretches out to several times its unperturbed length in toluene.

Similar measurements were repeated for two other binary solvent mixtures with the same dPS brush film. These binary mixtures comprised of methanol/dichloromethane and hexane/dichloromethane of varying compositions between pure solvent end-points. While dichloromethane is a moderately good solvent for polystyrene, hexane is a poor solvent while methanol is nearly a non-solvent. It has been concluded from neutron reflectivity data that the methanol/dichloromethane mixture is clearly not as good as the cyclohexane/toluene mixture, and the hexane/dichloromethane is intermediate between the two. These results are consistent with the basic expectation for binary solvent mixtures of poor and good solvents, based on their individual ranking of solvent qualities described earlier. A closer look at the swelling data indicates possible cosolvancy effect for the cyclohexane/toluene and the hexane/dichloromethane mixtures at compositions between 60-70% good solvent volume fraction. Further analysis and fitting of the reflectivity data is in progress along with comparison with self-consistent field (SCF) calculations. In addition,preferential solvation issues are being systematically explored by selectively deuterating individual component solvents.

Diffraction of Neutron Standing Waves in Thin Films with Resonance Enhancement
In conventional grazing-angle diffraction, an evanescent wave is created when a plane wave is incident upon a sample surface at an angle below the critical angle for total externalreflection.

While traveling parallel to the surface, the evanescent wave has a non-uniform amplitude distribution perpendicular to the surface: its amplitude decreases exponentially into the bulk. One may take advantage of the spatial non-uniformity of the evanescent waves to achieve surface diffraction.

In the present experiment we exploit spatial non-uniformity of a sinusoidal type in neutron waves. In contrast to using the neutron or x-ray waves *below* a total reflecting surface as inevanescent scattering, we propose to use what is *above* a total reflecting surface. Above the reflecting surface, the totally reflected wave interferes with the incident wave near the surface; the superposed wave travels parallel to the surface with a sinusoidally varying amplitude in the direction normal to the surface: it forms a standing wave in the normal direction. If a thin film has been grown on top of the total reflecting surface, the near-surface standing wave (traveling parallel to the surface) may undergo diffraction, which can be used to study the in-plane lattice structures of the thin film.

To demonstrate this diffraction method, we used an epitaxially grown Y/Gd/Y/Nb/Al_2O_3 thin-film sample and studied diffraction from the Y/Gd/Y(1100) plane; the reciprocal vector Q(1100) was parallel to the sample surface within a small surface miscut angle. Both Nb and Al_2O_3 have higher neutron SLD than Gd and Y, and thus can serve as the total reflection mirror.

Specular x-ray reflectivity was measured to characterize the sample. Neutron specular reflectivity and diffraction measurements were carried out at the NG-7 reflectometer. A linear position sensitive detector (PSD) was used to measure the diffraction intensity.

We have experimentally demonstrated the diffraction of neutron standing waves and resonance enhancement via a Y/Gd/Y/Nb/Al_2O_3 sample, and applied a quantitative calculation which describes the diffraction pattern very well. We anticipate that further skillful applications of the new diffraction geometry will shine new light in the study of 2D magnetic and crystal structures and phase transitions in thin films.

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Inversion of Neutron Reflectivity Data and Determinatin of the Phase
In the past year progress has been made in the problem of determining the phase of neutron reflectivity, thereby opening new possibilities for the analysis of specular reflectometry, including the determination of scattering length densities by direct inversion of data. Two theoretical methods of phase determination have been found by division researchers for potentials having compact support, i.e., potentials corresponding to freely supported films. These new methods are effectively restricted in application to neutrons because of an express requirement that the scattering length density profile be only real-valued, which is well met by most material for neutron scattering but not for x-ray scattering.

The first approach makes explicit use of the transfer matrix representation for the complex reflection coefficient as a function of wavevector transfer. In this representation the reflection coefficient depends on four real functions, the elements of the 2 X 2 transfer matrix, but its norm---the measured reflectivity---depends on three distinct combinations of these, which can be exactly determined by obtaining the reflectivity spectra for a system comprising three samples, each consisting of the unknown film and one of three distinct reference layers whose scattering length density profile is known. While the method requires the possible inconvenience of three measurements, it leads to a relatively simple algebraic extraction of the desired reflection amplitude for the unknown film. Moreover, the phase determination at each value of wavevector transfer (i.e., angle of incidence) depends only on data at that angle, without the need to produce intermediary functions that depend implicitly on all the data. The use of reference layers does entail concerns for ensuring that their introduction onto the film of interest is as unintrusive as possible, but for sufficiently robust films, this should not be difficult. Magnetic layers on nonmagnetic films, for example, offer a way of changing the scattering properties of the reference layers by changing the magnetization of a single sample. When polarized neutron beams are used, two distinct references of the needed three are obtained simply by rotating the sample by 180 degrees relative to the polarization direction.

The second method of phase determination is restricted to films that are mirror symmetric, i.e., which present the same density profile from either direction, or equivalently, when flipped over. In fact, many films of interest can be made symmetric by the simple device of putting two similar samples back-to-back. When a film is flipped over, the only effect is to change the phase of the reflection coefficient, so that the measured reflectivity is unaffected. For symmetric films, this phase change depends only on the film thickness. On the other hand, for arbitrary potentials, the transmission coefficient is totally unaffected by flipping the film, while the phases of the transmission coefficient and of the flipped and unflipped reflection coefficients are connected by the unitarity of the scattering matrix. As a result of these relationships, the phase of the reflectioncoefficient is determined for a symmetric film of known thickness by the phase of the transmission coefficient. The latter, in turn, can be determined from a Hilbert transform of the logarithm of the transmission, which is known at once from the reflectivity, since at every angle the reflectivity and transmission add to unity in the absence of absorption. Thus, the phase of refection from a mirror symmetric film is determined by its reflectivity spectrum. This method has a potential advantage over the first in not requiring three distinct samples, but it can only be

applied to films which are known to be symmetric by construction. Moreover, its use requires the difficult computational step of producing an integral transform of the measured spectrum. It turns out that the need for this step also limits consideration to potentials that do not support bound states, i.e., to compactly supported scattering length density profiles that are nowhere negative. On the other hand the method does shed some interesting light on the underlying reflection theory. For example, the contribution of transmission phase to the reflection phase is absent within the Born approximation.

The knowledge of the complex reflection coefficient provides a means of directly inverting neutron reflectometry using the Gelfand-Levitan-Marchenko integral equation and other related methods. This practical aspects of the inversion problem are currently being explored.

A User-Friendly Software, *spec*R, for Model Fitting Neutron/X-ray Reflectivity Data
We are currently creating a user-friendly computer software which model-fits neutron and x-ray reflectivity data. A draft version of *spec*R hasbeen installed on *jazz* for taffs to test it and give feedbacks. The first release version will also be ported to *strad* for routine use by guest researchers to fit data taken at the BT-7 and NG-7 neutron reflectometers as well as the x-ray reflectometer.

*spec*R is born out of frustration experienced by users of the existing software, *mlayer*, who wants to adjust the model scattering length density (SLD) profile easily and see the fitted curves right away on the screen without having to wait for a hard copy coming out of a printer. *spec*R provides a graphic user interface (GUI) using X-window widgets. Besides the GUI, the underlaying structure of sample stratification in *spec*R is different from that in *mlayer*. Instead of dividing a sample into multiple layers of constant SLD connected with an interfacial smoothing function such as *erfc* or *tanh*, *spec*R allows users to divide the sample into a pile of strata and to assign an arbitrary SLD function to each stratum. This flexibility allows users to easily input nearly and theoretically predicted or physically intuitive SLD functions. In addition to the improved user-interface and sample-stratification scheme, *spec*R is designed in a modular fashion to avoid a long spaghetti program and to reduce interconnections between various parts of the program, so that the program will be easily managed and modified for future changes, and the codes will be reusable for further software development (for example, a program which fits both specular and nonspecular reflectivity simultaneously). *spec*R will be complementary to the existing parametric B-spline (PBS) program, in particular when prior knowledge of a sample can suggest a sound physical model of the SLD profile and only certain parameters need to be fitted.

Oxidation of Grain Boundaries in Annealed Symmetric Spin Valves
An important consideration in the development of devices using spin valve multilayer materials is the thermal stability of the spin valve structure. Recent experiments on NiO biased symmetric Cu/Co spin valves have shown that while spin valves of this type can e made to exhibit good thermal stability with respect to the giant magneto resistance (GMR) ratio, the coercivity. Hc, of the ``free" film is a very desirable property for high sensitivity in spin valve devices such as read heads for high density magnetic recording. In this paper, we report results of investigations into

the origin of the anneal-induced increase in the coercivity of symmetric spin valves.

Two types of samples were used in the experiments: a spin valve with the structure NiO/Co/Cu/Co/NiO, and a ``pseudo-valve" film with the structure NiO/Co/Cu/Co/NiO. The pseudo-valve was designed to isolate the center Co film of the full spin valve by replacing the top and bottom Co films with Cu. Separate samples of the pseudo-valve film were annealed for 30 min. under vacuum in the 10_{-6} Torr range at annealing temperatures T_{ann}, ranging from 100 to 400 °C. The full spin valve was annealed sequentially for 20 min. and then 20 hrs. at 250 °C.

Hysteresis loops for a few of the annealed pseudo-valves show that the coercivity slightly increases even for T_{ann} as low as 100 °C, a maximum around 250 °C, followed by decreasing coercivities at higher temperatures. Furthermore, the hysteresis loops show reduced squareness, and a significant loss of magnetic moment in the films for the high values of T_{ann}. The reduction in moment indicates that oxidation may be playing a significant role in the increase coercivity of the center Co film.

Ferromagnetic resonance (FMR) fields at 9.67 GHz for normal ($H\perp$) and in-plane ($H//$) orientation of the applied field show that $H\perp$ decreases and $H//$ increases with T_{ann}. The FMR data is not consistent with simple layer-by-layer oxidation of the Co. With the field applied normal to the film, $H\perp = w/g + Meff$. Layer-by-layer would not effect Meff, but oxidation in grain boundaries would effectively reduce Meff, and would be consistent with the observed reduction in $H\perp$ with annealing.

To further investigate this suspected oxidation, low angle x-ray reflectivity measurements on a full spin valve were made in the as-deposited state and after anneals of 20 min. and 20 hr. The x-ray data was fit to a structural model of the spin-valve which included 11 layers, each with independent values of thickness, roughness, x-ray scattering density, Q_c^2, and x-ray absorption, μ. Minimization of Chi-squared consistently led to one of two similar models in which the metallic layer structure was preserved (minimal interdiffusion), but where the top Co layer was heavily oxidized and a small amount of oxidation was observed in the other metallic layers.

The magnetometry and x-ray data contain strong evidence for oxidation of the films during annealing. However, the good thermal stability of the GMR and of the coupling between the center Co film and the outer Co films[1], along with the stability of the layer structure observed by x-ray reflectivity indicates that the Co/Cu interfaces are not strongly affected by annealing. Consistent with the decreases in $H\perp$ (FMR) and with reduction in Q_c^2 (x-ray), we conclude that significant oxidation occurs in the grain boundaries of the films during annealing. Polarized neutron reflectivity experiments will be performed in the future to determine the relation between the oxidation of the films and the reduction in magnetic moment during annealing.

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Molecular Beam Epitaxy Chamber For Neutron Scattering

With the increased flux available from the new cold source, it is becoming practical to investigate reflectivities at higher values of momentum transfer, Q, and with smaller sample volumes. As thinner films are being examined a more significant fraction of the sample volume is contained in the surface contamination layers that occur on all samples exposed to air. Thus it is becoming more important to protect a samples surface using a UHV chamber. The MBE Chamber for In-Situ Neutron Scattering is an ultra high vacuum chamber that can be used to fabricate single crystal thin films and study them in-situ, as a protective UHV environment for both bulk and thin film samples, and as an ultra-clean environment for loading interstitials. Although this chamber was designed to operate on the BT-7/NG-1 reflectometer, it can also be used on other instruments such as triple axis and SANS spectrometers. At present, several experiments are planned for this chamber, as listed below.

Planned Experiments

- Studies of hydrogen absorption and its effects on the structural and magnetic properties of metals
- Use of ion beam etching to study the two length scales phenomenon near the spiral magnetic phase transition temperature at the surface of bulk Tb
- Measurements of the magnetic penetration depth in alkali-doped C_{60}
- Studies of magnetism in ultra-thin films

Other General Fields of Study

- Surface chemistry, catalysis, and oxidation studies
- Epitaxial growth of organic molecules:
  Biological and polymer systems
- Semiconductor materials processing:
  Growth and dry etching of semiconductors,
  Gate oxides and metallization layers
- Surface magnetism and a artificial magnetic structures
- Epitaxial studies
- Diffusion studies
- Studies of hydrophilic and other sensitive materials

Source Flange

- We now have four standard effusion cells which can deposit materials with melting temperatures, $T_m < 1400$ °C. Expected uniformity is ~2% over a 3" diameter substrate.
- There is room for a total of 7 effusion cells, or a combination of 5 effusion cells and 2 electron beam sources (which can deposit high Tm materials, although with less uniformity).
- Other expansion possibilities for this flange are high temperature effusion cells (providing good flux uniformity of materials with Tm < 2000 °C) and an optical pyrometer (for more accurate determination of elevated substrate temperatures).

Main Chamber

- A 3 cm flood ion gun will allow dry etching at a rate of ~1 μm/hr, surface cleaning of substrates and samples grown elsewhere, ion assisted MBE, and reactive growth of oxides, nitrides, etc.
- The chamber will be pumped by a 2600 L/s cryopump and achieve a base pressure of 10^{-10} *torr*.
- An independent sample shutter will provide isolation during source calibration, limited sample pattern-masking, and thermal shielding during low temperature operation.
- A port is available for the addition of a load lock chamber, allowing quick sample exchange without breaking the vacuum (thus eliminating the lengthy bake out process required for recovery of UHV).
- It is also possible to include devices for ellipsometry, SMOKE (surface magneto-optical Kerr effect), low energy electron diffraction with Auger spectroscopy (for in-plane crystallography and surface compositional analysis, respectively), or XPS (x-ray photo-electron spectroscopy) in the port currently earmarked for the flood ion gun. In this case the flood ion gun can be relocated to one of the large effusion cell ports.

Neutron Window

- It consists of 1/8" Al tubulation welded to explosion bonded Al/Stainless Steel flanges. Neutron scattering can be measured up to a Bragg angle of $\theta = 39°$.
- Since the aluminum window material extends over nearly the entire 360° azimuthal range, it allows for grazing angle experiments in addition to neutron reflectivity. Also, if tilt capabilities are added to the sample holder, the chamber may be reconfigured for high angle diffraction $\theta° > \theta > 180^{\wedge}°$).
- Another grazing incidence angle probe, reflection high energy electron diffraction, is available using the diametrically opposed ports in this section.

Sample Flange

- It houses three systems: sample holder, deposition flux monitoring devices, and feedthroughs for an applied magnetic field.
- Sample holder design parameters include the following:
  - Sample temperature range of ~15 K - ~1300 K
  - Sample rotation, continuous or ± 180°
  - Simultaneous 4-point electrical probe
  - 3" diameter samples
- Deposition Fluxes can be monitored using a quartz crystal micro-balance and/or a quadruple mass spectrometer positioned to intercept the beam below the sample.
- Soft iron pole pieces have been welded into the sample flange to provide a ``magnetic field feedthrough" permitting externally controlled applied magnetic fields up to 300 Gauss from permanent magnets, or higher fields if connected to an electromagnet. This facility will soon be available. It is estimated that the basic configuration, consisting of the vacuum system, sample holder, four effusion cells and flood ion gun will be assembled by the end of February 1996.

Reference

[1] J. A. Dura (work in progress)

Project Title: Crystallography

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Technical Description:
This continuing project involves the development and use of the nation's best neutron powder diffraction capability to study the detailed structure and phase analysis of materials which are important in industrial applications. Strong emphasis is given to advances in crystallographic measurement and refinement methods to extend the accuracy and range of neutron diffraction techniques, including powder and protein crystallography.

Technical Objectives/Goals:
The goal is to provide the best overall U.S. capability in neutron powder diffraction and protein crystallography. Some key objectives are:

(1) serve the generic research needs of U.S. industries and universities who require state-of-the art neutron diffraction to provide key information on the structure of new catalysts, sieves, ceramics, alloys, fullerenes, optoelectronic and other technologically important materials.
(2) to provide uniquely sensitive methods for phase analysis of materials and apply them to important NIST programs in ceramic coatings, phase diagrams, and superconductivity.
(3) to develop new U.S. capabilities for quasi-Laue protein crystallography which will greatly enhance biological structure research in this country.
(4) bring on line a state-of-the art single crystal diffraction station in the next 2 years.
(5) provide updated, highly sophisticated, x-ray, electron, and neutron crystallographic databases.

Accomplishments:
The state-of-the art capabilities in high-resolution neutron powder diffraction, single-crystal diffraction, and the development of quasi-Laue techniques will enable the much-needed structural analysis of technologically significant materials.

Outputs:
The results of the structural analyses on the hundreds of materials studies will be presented at the appropriate major scientific and technological conferences. The categories of materials typically include (but are not limited to catalysts, ionic conductors, alloys, fullerenes, and ceramics.

Some 28 technical papers resulted from this project in FY95.

Revised and updated Crystal Data and Electron Diffraction databases will be issued with approximately 15,000 new entries.

Outcome/Impact:
Among outcomes anticipated this year are:
1) complete installation of major components of state-of-the art four-circle neutron diffractometer for structural analysis of single crystals
(2) complete test with European Molecular Biology Laboratory in Grenoble of a new approach for quasi-Laue neutron diffraction studies of protein crystals (could improve current worldwide capabilities by x50).
(3) In our crystal data program, we will investigate prototype software package/CD to allow distribution to U.S. industries and universities of the entire database for use with a modern PC.

Our research in neutron diffraction techniques for structure determination, structure refinement, and phase analysis, coupled with results from data utilizing the newly developed high-resolution powder diffractometer, will have significant impact on the research and development programs of over 30 collaborative institutions.

Highlights:
The following examples are representative of the many areas of materials research impacted by this project:

Neutron Rietveld refinement is used to analyze the phase content of industrially important materials in which phase composition is critical to materials properties, for example, austempered ductile iron and yttria-stabilized zirconia thermal barrier coatings.

Improved understanding of structure-property relationships in zeolite molecular sieves will allow new materials for gas separations to be developed.

The NIST-sponsored international Workshop on Crystallographic Databases will bring together representatives of all crystallographic data centers, journal editor, instrument manufacturers and database users to explore common concerns and goals.

Project Title: Macromolecular and Microstructure Studies

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Technical Description:
In this project, neutron methods, primarily involving small angle neutron scattering (SANS), are developed and applied to the characterization of submicron structure in a wide range of materials including polymers, colloids, microemulsions, microporous media, biological molecules and complexes, nanocrystalline metals and ceramics, and many others.

Technical Objectives/Goals:
The main objectives of this project are: the development of first class neutron scattering instrumentation and methodology for characterizing submicron structure in materials; the development of applications for said instrumentation and methodology to materials of current interest in materials science; and, the development of collaborations with scientists at NIST, in industry and at universities which address current problems in materials science.

Accomplishments:
Several improvements have been made to the 30 meter SANS instrument at the CNRF that is jointly supported and operated by NIST, the Exxon Research and Engineering Co., the University of Minnesota, and Texaco Research and Development. Chief among these were modifications to the sample chamber and post-sample flight path to allow the instrument's large area detector to be positioned as close as 1.0 m from the sample thereby significantly increasing the angular range of the instrument. As a result, the structural scale accessible on this instrument has been increased and now ranges from 1 nm to over 400 nm.

Cooperative agreement between NIST and the Exxon Research and Engineering Company, that has been in existence since 1986, has been renewed for an additional three years. Under this agreement, Exxon will continue its partial support for the operation of one of the CNRF's 30 m SANS instruments (on guide NG7) and will continue to use this instrument for non-proprietary research in areas such as: the analysis of the compatibility and phase behavior of new polyolefin-based polymer blends; the structure and internal organization of particles in heavy oils such as asphaltenes; the behavior of complex fluids in microporous media; and, the organization of surfactants and block copolymers in organic fluids.

A numerical calculation method, called the arc method, for quantitatively evaluating the effects of

instrumental smearing on SANS data that is computational efficient has been developed and made available to researchers via an interactive PC computer program.

Outputs:
Technical Papers:
8 published or accepted for publication in refereed journals.

Outcome/Impact:
SANS measurements are expected to continue to have a major impact on the development of new polymer materials by providing essential data, unobtainable by other techniques, on the polymer conformation and interactions in blends, composites, surfactant-polymer complexes, etc. The improvements made to the NIST/Exxon/U. Minn./Texaco 30 m SANS instrument now enable microstructural features from 1 to over 400 nm to be resolved.

Largely as a result of recent SANS measurements at NIST, several groups are collaborating in the use of SANS to relate the bulk rheological behavior of complex fluids consisting of interacting particles and macromolecules to their underlying microstructure. Both university and industrial scientists have been involved in studies of, for example, shear thinning and thickening in concentrated colloidal systems, the microscopic origin of viscoelastic behavior in concentrated surfactant systems, and the induction of macroscopic orientational order in colloidal crystals and microphase-separated block copolymers. SANS studies of such systems, exploiting the techniques of contrast variation through deuterium labeling, are proving to be among the most effective methods for understanding the microstructural origins for non-Newtonian rheological effects.

Highlights:
The first successful SANS measurements on confined, ultrathin (100 nm) polymer films have been carried out in collaboration with scientists from IBM and MIT. The measurements were made on samples of block copolymers and polymer blends and have provided some of the first direct evidence on the effects of confined geometry and surface interactions on polymer conformation.

Advanced computer modeling and graphics capabilities are essential to perform the complete analysis of SANS data from biological macromolecules in solution which is necessary for the determination of a reasonable model structure. In order to determine how to best visualize and manipulate macromolecular model structures, several sophisticated commercial software packages were researched and evaluated. Recommendations were then made to NIST's Computing and Applied Mathematics Laboratory (CAML), which has now made a number of these advanced software products accessible to high performance computer work stations throughout NIST. Through continued interaction with CAML and software vendors, we are endeavoring to integrate commercial software with specialized SANS modeling software into a useful package for SANS users.

Project Title: Magnetism and Superconductivity

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Technical Description:
Neutron diffraction and inelastic scattering techniques are utilized to investigate the structure and energetics of magnetic materials, superconductors, and systems that are both magnetic and superconducting. Research is often carried out in close collaboration with many industrial and university experts.

Technical Objectives/Goals:
The technical objectives are to use the unique and powerful neutron scattering facilities located at NIST to investigate the fundamental physical properties of systems that are magnetic or superconducting, and to elucidate the physical characteristic that can be tuned in order to facilitate their use in advanced materials applications. Current interest centers on

1) the magnetic ordering, spin dynamics, and lattice excitations of
    a) high T_c cuprate superconductors
    b) nickel-boron-carbide superconductors
2) vortex structures and dynamics, in particular with regard to trapped-flux applications
3) magnetic properties of Invar related ferromagnets
4) properties of nanocrystalline/amorphous ferromagnets
5) magnetic properties of giant magnetoresistance materials, with particular emphasis on magnetic recording applications

Outcomes:
During the current year we anticipate achieving the following results:

1) complete the investigation of the nature of the spin and lattice dynamical excitations as $YBa_2Cu_3O_7$ enters the superconducting state, and map out the magnetic ordering phenomena in the rare-earth nickel boron carbides
2) elucidate the vortex structure and pinning mechanisms in $YBa_2Cu_3O_7$, and the dynamics of the vortex lattice in elemental niobium
3) made an absolute cross section measurement of the spin wave excitations in Invar
4) investigate the magnetic order in a nanocrystallized amorphous ferromagnet
5) initiated investigations of the La-$SrMnO_3$ GMR and related systems

Accomplishments:
Investigation of the microscopic properties of broad classes of materials in the general areas of magnetism and superconductivity. Neutron scattering data have been collected, analyzed, and interpreted on the structure and energetics on high quality specimens of these materials. Numerous publications have been submitted or published based on these efforts, and we have also given a large number of talks at scientific meetings and institutions as detailed elsewhere.

Substantial progress was made in improving our measurements capabilities on the neutron instrumentation. Features were designed, and in some cases have already been installed, to improve the flexibility of the instruments, and to reduce personnel exposures to ionizing radiation. in accord with our ALARA program.

Outputs:
Scientific presentations have been made at several meetings, including the March meeting of the American Physical Society, the International Conference on Magnetism, the Conference on Magnetism and Magnetic Materials, the International Workshop on Itinerant Electron Magnetism, and the International Conference on Strongly Correlated Electron Systems. Some 39 technical publications were published or submitted refereed scientific journals in FY95.

Impact:
The experimental results have elucidated the nature of the spin and lattice dynamical excitations in cuprate superconductors such as $YBa_2Cu_3O_7$ and electron-type materials such as Pr_2CuO_4. In particular the behavior of the dynamics as the superconducting state develops has been determined, and this has allowed the development of theoretical models for the superconductivity. We have also mapped out the magnetic ordering phenomena in the rare-earth nickel boron carbides, and these results have been widely used and cited by the scientific community.

The vortex structure and pinning mechanisms in $YBa_2Cu_3O_7$, and the dynamics of the vortex lattice in elemental niobium has been determined. This has allowed a test of theoretical models concerning the origins of the symmetry and nature of the pinning of the vortex lattice.

Highlights:
Neutron Diffraction Images of the Superconducting Vortex Lattice.
The behavior of vortices in superconductors is of immense practical importance as well as of fundamental scientific interest. RRD scientists, in conjunction with researchers at Princeton University and the University of Maryland, have been studying the behavior of vortex structures in both elemental superconductors such as niobium and in the high T_c materials such as $YBa_2Cu_3O_7$. This program is also supported by Boeing, who has great interest in high T_c crystals for magnet applications. In niobium, evidence has been found for correlated flux motion at high temperatures, a phenomenon first introduced to explain the degradation of the superconducting properties observed in high T_c cuprate superconductors. In $YBa_2Cu_3O_7$ we have discovered that

the twin planes are an important mechanism for flux pinning in bulk materials.

Some Details: The scattering image obtained from a single crystal of niobium, which is a cubic material whose electronic properties are approximately isotropic., is an undistorted hexagonal diffraction pattern. The relatively short magnetic penetration depth in niobium yields strong vortex scattering; typical high-quality scattering patterns can be obtained in two minutes counting time, and higher-order diffraction peaks are easily observed. At elevated temperatures the scattering weakens, and the spots broaden. However, the basic hexagonal pattern remains, signaling that the lattice has "melted" but the vortex motion is highly correlated.

The vortex image for the high temperature superconductor $YBa2Cu_3O_7$, whose electronic properties are very anisotropic, is reflected in the observed elliptical distortion of the diffraction pattern. The pattern is a distorted hexagon, and is strongly pinned by the twin boundaries in this crystal. The metallurgical scattering from the twin boundaries, coupled with the much weaker vortex scattering, makes these data quite difficult to obtain, and the vortex pattern is readily apparent only in background-subtracted data (counted typically for a few hours). The strong pinning of the vortices by the twin boundaries is very important for technological applications of this material, and indeed the success of these experiments depended critically on the growth of large, high quality single crystals in a program devoted to device applications of bulk $YBa_2Cu_3O_7$.

Project Title: Neutron Beam Applications

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Technical Description:
This project will develop and utilize neutron beam facilities for residual stress, texture, and radiographical measurements.

Technical Objectives/Goals:
This project is designed to provide unique measurement capabilities, utilizing neutron diffraction, for the nondestructive characterization of residual stress and texture for engineering applications for industry and other organizations, and state-of-the-art neutron radiography/autoradiography facilities.

Outcomes:
(1) A dedicated, state-of-the-art neutron diffraction instrument for residual stress and texture measurements to be available as a User Facility.
(2) A world-class in-house research group which is in the forefront in developing neutron diffraction residual stress and texture measurement methodology.
(3) Collaborations, CRADAs, and/or Contracts for the solution of engineering problems in the areas of residual stress and texture.
(4) The development and maintenance of neutron radiography and autoradiography facilities for outside users.

Accomplishments:
Analysis and interpretation of neutron residual stress measurements for a number of engineering-related problems was completed. This included a v-notch steel weldment (for ONR), skip welds for railroad tank cars (for Dept. Of Trans.), a spot-welded steel disk (for ONR). The results for the spot-welded disk was particularly noteworthy because it was selected to provide a simple geometry weldment for FEM modeling, which was successful (see publication below).

Significant progress was made in the fabrication of a new diffractometer for residual stress and texture studies, and single-crystal diffraction. Many features--such as a multi-crystal monochromator assembly; computer-controlled retractable pre- and post-sample collimators; a continuously-variable post-sample neutron aperture; a 50 kg capacity, 17 cm range x-y-z translational sample table--are unique.

A policy and organizational structure was established by which the neutron autoradiography of paintings program could continue even without direct Smithsonian Institution support.

Outputs:
In FY95 eight technical presentations were made at international conferences, one of which was invited. These will all appear in Proceedings. Two papers were published in the open literature, and one paper was published as a NIST Technical Report.

Impact:
The characterization of the full residual stress distribution in a simple-geometry specimen (i.e. a spot weld on a steel disk) has provided a test case for modeling efforts at the Oak Ridge National Laboratory. Both the experimental and modeling efforts are being performed under contract with the Office of Naval Research.

The characterization of the residual stresses around skip welds has provided a test of a theoretical model the predictions of which suggested that skip-welded reinforcement of tank cars would lead to high tensile residual stresses and failures. The neutron-determined residual stresses did not confirm the predictions of the model.

Highlights:
State-of-the-Art Neutron Diffraction Residual Stress Instrument
Neutron diffraction is the only inspection technique by which subsurface triaxial stress distributions can be determined nondestructively. It therefore represents the only method by which certain stress fields can be characterized, and the standard reference method against which alternative, portable techniques be tested or calibrated.

Under development at the BT-8 beam port at the NBSR is a state-of-the-art facility for texture, residual stress measurements, and single-crystal diffraction. It will incorporate a new position-sensitive detector system and focusing monochromator, extensive new software, and optimization of a variable collimation system. Successful completion of this new materials research capability will improve overall measurement power by an order of magnitude over the present system, and open up many opportunities of great importance to U.S. industry and defense.

Ongoing programs which will benefit enormously from the improved instrumentation include collaborations with Metallurgy Division, Army Armament RD&E Command, Ceramics Division, Dept. of Transportation, Oak Ridge Nat'l. Lab, General Motors, and M.I.T.

Project Title: Chemical Physics of Materials

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Technical Description:
This continuing project involves the development and application of neutron scattering methods to yield essential information the atomic and molecular interactions and dynamics which control the properties and performance of molecular solids, disordered materials and molecular species in nano- to microporous systems. Much of this research is unique in the United States and involves cooperative research with dozens of industries, universities, and government labs.

Technical Objectives/Goals:
The goal is to provide U.S. science and industry with needed and unique neutron research and measurements of the submicron properties of molecular and inorganic solids, which have potential, e.g., for new or improved chemical processing and purification, applications in electronic devices or construction materials, and refrigeration.

Accomplishments:
A new cooperative research agreement was established with U. Penn. And Hughes to study the structural and dynamic behavior of a new class of C-Li battery materials and to probe phase transformations and subtle bonding states of conducting polymers by inelastic neutron scattering.

A joint research program has been developed with the Department of Transportation on application of neutron hardening and freeze thaw processes in a variety of new mixtures. Results will be correlated with microstructure and will be used to test existing theoretical models and provide needed predictive capability for advanced concrete.

Developed major plan for world's most sensitive neutron spectroscopy facility (with UCSB, Penn., Dupont). This 2M$ instrument will be the central resource in studies of the atomic and molecular scale properties of new materials (e.g. fullerenes, catalysts, energy storage systems, polymer, etc.).

Initiated, with Biotechnology Division, first combined neutron studies of protein dynamics by high resolution neutron spectroscopy and molecular dynamics simulation carried out in U.S.

Outputs:
Fifty-four papers published or accepted for publication and 17 invited lectures.

Outcome/Impact:
Examples of anticipated outcomes are:

- Advances in atomic scale understanding of bonding states and mobility of molecular species in catalysts, molecular seives, and gas and refrigerant storage media directly related to performance.

- Correlation of submicron hydration states and water mobility in concretes with microstructure and admixture with fly ash and other additives to improve performance and durability.

- Provide understanding and prediction of thermal properties of carbide and nitride coating materials which are being examined by industry for use in engine components and electronic devices.

- Reveal key bonding and diffusive properties of C-Li materials being developed for advanced battery applications.

- Create only U.S. central capability for neutron studies of biomolecular dynamics.

- Imroved understanding of new fulleride compounds, including superconductors.

Highlights:
Some highlights of FY95 activities include establishing through combined neutron studies with industry of the bonding and dynamic behavior of molecules in zeolites, which are a key, e.g., to improved catalysis and for efficient storage and separation of environmentally benign refrigerants; successfully arranging a new cooperative research agreement (with 1.5M$ funding) with U. Penn, U. Cal (SB) and several industries to create a vastly improved new U.S. capability for high-sensitivity neutron spectroscopy of new materials and chemicals; and a new joint program with DOT for developing neuron methods to probe molecular scale curing processes in cement.

Program: Biotechnology

Project Title: Reflectivity Studies of Lipid Bilayers

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Technical Description:
This project will develop neutron reflectivity methods to characterize the structure of single lipid bilayers and biomimetic cell membranes supported on planar substrates in an aqueous environment.

Technical Objectives/Goals:
The reflectivity technique is well-suited to the study of lamellar systems such as lipid bilayers and it is important to be able to measure a single lipid bilayer in aqueous solution since it represents a working model for a biological membrane. The incorporation of active membrane proteins into supported lipid bilayers is a crucial step in creating a true biomimetic membrane. Thus, the goals of the project are to develop neutron reflectivity methods to characterize lipid bilayers and biomimetic cell membranes and, concurrently, to develop methods to produce a robust biomimetic cell membrane consisting of a single lipid bilayer with incorporated membrane proteins. This work will be performed in collaboration with both the NIH and NIST's Biotechnology Division. Microscopic, biochemical and spectroscopic techniques are expected to be used to complement the reflectivity measurements.

Accomplishments:
Combined neutron reflectivity and atomic force microscopy measurements of single phosphatidylcholine lipid bilayers provided new insights into the proper interpretation of the reflectivity data. As a direct result of these measurements, improvements to the neutron reflectivity instrument are currently underway so that the sensitivity of the measurements can be increased to equal that of high resolution diffraction techniques on lipid multilayers. Preliminary x-ray reflectivity measurements of biomimetic lipid bilayers on gold-coated silicon substrates provided a new understanding of the minimum requirements for the thickness and smoothness of the gold coating. Thus, efforts are now being concentrated on making coatings appropriate for optimal neutron reflectivity measurements.

(Anticipated) Outcomes:
This project is expected to provide both the biomedical community and industry with the

structural information necessary to develop structure/function relationships. Membrane proteins not only play a crucial role in biological function, but they are also essential for the development of important industrial devices such as biosensors. The correlation between structure and properties is an essential step in the understanding of biological processes (cell fusion events, ion transport, receptor activity) or in the development of final products (drugs, biosensors).

Outputs:
One invited talk and one contributed talk have resulted directly from this project and one technical paper has been accepted for publication.

(Technical) Highlights:
In collaboration with the NIH, neutron reflectivity has been used to probe the structure of single phosphatidylcholine lipid bilayers adsorbed onto a planar silicon surface in an aqueous environment. By measuring the specular reflection of neutrons from the bilayer at the silicon/water interface, the neutron scattering length density profile perpendicular to the bilayer is obtained. A novel experimental setup which significantly decreases the background scattering allowed measured reflectivities as low as 5.0×10^{-7} to be obtained, resulting in an increased resolution of the scattering density profiles compared to previous studies. Thus, changes in bilayer thickness due to lipid phase transitions could be measured with a sensitivity of 1-2 Å. A model-independent fitting method developed at NIST was employed along with the more standard model-dependent method in order to make a less-biased determination of the scattering density profiles. Independent measurements of the size of the lipid domains and the degree of surface coverage, made under the same experimental conditions using atomic force microscopy, help solidify the interpretation of the neutron reflectivity fits.

Program: Superconductivity

Project Title: Structural Studies of High T Superconductors

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Technical Description:
Thermal neutron diffraction techniques and profile refinement analyses are utilized to investigate and determine the crystal and magnetic structures, composition, and crystal chemical properties of high T_c superconductors and related families of materials. Research is often carried out in close collaboration with many industrial and university experts and is closely coupled with phase studies in the Ceramics Division.

Technical Objectives/Goals:
The technical objectives are to use the unique and powerful experimental technique of thermal neutron diffraction to 1) determine the magnetic and chemical structures of newly discovered high T_c superconductors, 2) establish the relationships between the structural properties, composition, and the physical properties, 3) determine the systematic trends between the crystal and magnetic structures and their physical properties, and 4) to establish the relationships between the structural properties and their superconducting properties that are of central importance in utilizing these materials in applications.

Outcomes:
During the current year we anticipate achieving the following outcomes: 1) determine the crystal structure and composition of the mercury-based cuprate layered superconductors, and relate these properties to the superconducting properties of direct relevance for applications; 2) investigate and determine the relation between the structure and composition of the nickel-containing superconductors and their magnetic and superconducting phase transitions.

Accomplishments:
Investigation of the microscopic properties of broad classes of materials in the general areas of magnetism and superconductivity. Neutron scattering data have been collected, analyzed, and interpreted on the structure and energetics on high quality specimens of these materials. Numerous publications have been submitted or published based on these efforts, and we have also given a large number of talks at scientific meetings and institutions as detailed elsewhere.

Outputs:
Scientific presentations have been made at several meetings, including the March meeting of the

American Physical Society, the American Crystallographic Society, and the International Conference on Strongly Correlated Electron Systems. Fourteen papers were published or submitted for publication in referred scientific journals:

Impact:
The experimental results have elucidated the crystallographic and magnetic structures in the high temperature cuprate superconductors. Emphasis has been placed recently on the Hg containing materials, which presently possess the highest superconducting transition temperatures known. The determination of the structure is essential for the development of theoretical models for the superconductivity. We have also mapped out the crystallography and magnetic ordering phenomena in the rare-earth nickel boron carbides, and these results have been widely used and cited by the scientific community.

Highlights:
New Hg Superconductors - RRD scientists, in collaboration with CNRS, AT&T, and the Texas Center for Superconductivity, have solved the structure of a series of the recently discovered mercury-cuprate superconductors and are continuing an extensive series of neutron diffraction measurements on different layered structures of these new materials, using the newly commissioned high resolution powder diffractometer.

Some Details - The superconductivity community has been excited by the recent discovery of a new series of mercury-containing compounds with onset superconducting transition temperatures as high as 140 K at ambient pressure, and up to 164 K under pressure, which are much higher than the 1-2-3 system and substantially higher than the previous record-holding thallium system (125 K). Of critical importance is the determination the crystal structures and phase diagrams of these new materials. The unique power of neutron powder diffraction has played an essential role in determining structural properties of the copper-oxides, in particular with respect to the oxygen stoichiometry which controls all the relevant physical properties.

There are a number of interesting features of these new materials. One aspect concerns the doping, which is accomplished by adding a small amount of extra oxygen rather than substitutional doping. In the present case the extra oxygen ions go into the centered position in the Hg plane. This raises an important question about the possible role that the Hg-O layers might play in enhancing the superconducting transition temperature, and whether other elements can be substituted for Hg; in the single layer material the Cu-O planes are separated by the rather large distance of 9.5 Å, yet the superconducting transition temperature is 94 K, already higher than the triple-layer 1-2-3 ($YBa_2Cu_3O_7$) material.

The general trend in the cuprate superconductors is that increasing the number of Cu layers increases the superconducting transition temperature, and the new Hg compounds are no exception. The two-layer $HgBa_2CaCu_2O_{6+d}$ (1212) material has recently been successfully prepared in bulk form, and our neutron-determined structure shown in the figure. The extra oxygen that dopes the materials and is responsible for the superconductivity ($T_c \approx 126$ K) goes into the Hg layer, and in this case $d = 0.2$-

0.3. The high T_c of course is not the whole story, as this two-layer system also exhibits superior flux pinning properties crucial for applications. It is believed that the higher T_c's occur if additional Cu layers are added, but the difficulty in fabricating them also increases. Hence if the Hg series of layered cuprates follows the same pattern as the Tl- and Bi-based systems, the compound with the most technological promise is likely the 1212 system. The early indications are that the superconducting and mechanical properties of this new material represent a significant improvement over materials presently being used in applications.

The three-layer 1223 structure, with $T_c \approx 133$ K, has also been solved. The basic structure and crystal chemistry is similar to the 1212, but with three Cu layers per unit cell. It has recently been discovered that substituting Pb for the Hg enhances both the stability as well as the superconducting properties of the 1223 material, and additional diffraction studies on this class of materials are underway.

Program: Evaluated Materials Data

Project Title: NIST Crystal and Electron Diffraction Data Center

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Technical Description:
This project will produce scientific databases with critically evaluated data on all classes of crystalline materials for research and analysis in the materials sciences.

Technical Objectives/Goals:
The principal objective of this project is to produce scientific databases on crystalline materials; to produce theory, algorithms and software used in standardizing, evaluating, and searching the data, and to develop theory and methods to enhance the measurement process. The customer is the general scientific community.

Outcomes:
(1) A long term data effort dedicated to providing critically evaluated crystallographic databases as well as related theory, algorithms, and software to the general scientific community.

(2) A world-class data center which is in the forefront in developing theory and software for the collection, evaluation and use of crystallographic data.

(3) An international effort with long term collaborations with a series of affiliated data centers the objective of which is to provide evaluated data to the research and analytical scientist.

(4) A leader in the on-going revolution in scientific information.

Accomplishments:
During the year, the master database has been significantly argumented with respect to all categories of crystalline materials and now contains approximately 227,000 entries. From this central database, two distributions databases are produced:
(1) NIST Crystal Data and (2) the Electron Diffraction Database. Recently, updated versions of both databases were released to the scientific community. These databases are made available through computer-oriented modes of dissemination: PC, scientific instruments, and on-line searching. A major project during the year was the organization of the NIST-sponsored Workshop on Crystallographic Databases, an international meeting of representatives of all crystallographic data centers, journal editors, instrument manufacturers, and database users. Manuscripts from all the

speakers have been collected and edited and will be published this year in the NIST Journal of Research. A number of the manuscripts describe the use of NIST data products including, for example, a manuscript entitled "Using NIST Crystal Data within Siemens' Software for Four-Circle and Smart CCD Diffractometers."Resulting from the Workshop, there was a consensus that NIST should play a leadership role in a 'federation' of crystallographic data centers.

Outputs:
Details of Outputs are provided in the Measures of Performances Section. In summary, two updated versions of our databases with revised search software were released; an international workshop on crystallographic databases was organized by our data center; manuscripts for a special issue of the NIST Journal of Research on the workshop have been collected and edited; and two invited presentations on applications of our data products were given at national meetings.

Impact:
Our databases (NIST Crystal Data and the Electron Diffraction Databases), as they are comprehensive, have a major impact in research and analysis in the materials sciences. In research, they are used in the prediction of materials properties and in the design of new chemicals, and materials. In analysis, the databases and NIST theory provide the basis for an excellent way to characterize crystalline materials as unknowns can be uniquely identified using lattice/formula matching techniques. In addition to the database, our data center develops theory and software that are widely and routinely used by individual scientists, by affiliated data centers, by instrument manufacturers, and by publishers of scientific journals. In fact, NIST theory/software has had multiple impacts on almost every structure solved by single-crystal diffraction techniques -- starting with procedures used to collect the data and ending with techniques related to data dissemination.

Highlight:
NIST Crystal Data with over 227,000 entries is comprehensive as it contains all materials which have been characterized by a unit cell data and elemental analysis. In fact, it is the only database whose objective is to have entries on all crystalline materials. Newly developed mathematical techniques permit one to establish inter- and intralattice relationships between entries in the NIST Crystal Data and any other database containing crystalline materials (e.g.. cross-references can be uniquely determined between entries in different databases). New and powerful analytical tools for materials identification are created by merging NIST Databases and theory with commercial instruments. For example, Siemens Analytical Instruments now markets NIST Crystal Data with their single-crystal x-ray diffractometer and Princeton Gamma Tech markets the Electron Diffraction Database with their analytical electron microscope. Finally, converse-transformation theory and software (recently patented; principal inventor V. L. Karen) will have many applications in the materials sciences including materials design and the improvement of the basic measurement process used in single-crystal x-ray diffraction.

Program: Nanostructured Materials

Project Title: Magnetic Coupling in Nanocomposites

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Technical Description:
This project involves the characterization of the nanocsale magnetic interactions and the microscopic magnetic and chemical structures in both layered and particulate nanocomposite magnetic systems, important from both a fundamental scientific and applications perspective, using neutron scattering techniques. Neutron diffraction, reflectivity and small-angle scattering (SANS) directly provide information about the magnetic ordering that is inaccessible by other measurement techniques, including bulk magnetization and resistivity.

Technical Objectives/Goals:
The goal of this project is to determine the factors which give rise to and influence nanoscale magnetic coupling. This characterization is vital for the optimization of magnetic sensor materials. The emphasis is on magnetic materials which show enhanced magneto-resistivity and anomalous exchange biasing. our objectives also included upgrading our triple-axis spectrometers in order to improve signal-to-noise ratios, and facilitate sensitive measurements of ultra-thin films with limited sample volumes.

Accomplishments:
our expertise in the areas of magnetic characterization of ultrathin films using high- and low-angle neutron diffraction techniques was applied to the study of a variety of metallic, transition-metal oxide and rare-earth nanostructured materials. The dependence of the magnetic structure on the details of the physical structure was established and used to refine and improve the growth process of these composites. We also obtained fundamental information about the origin and nature of interlayer magnetic coupling in superlattice structures with alternating magnetic and nonmagnetic layers. These studies provided insight into the understanding of giant magnetoresistance and anomalous exchange biasing, important to the magnetic sensor and recording industries.

Outputs:
Sixteen articles describing these studies have been published in many refereed journals. These results have also been formally presented at a variety of national and international conferences, and as seminars at NIST and Ohio University. Several invited talks were given by collaborators at neutron scattering and related conferences.

Outcome/Impact:
The most direct outcome of this project is the utilization of observed correlations between chemical microstructure and magnetic structure to choose optimum preparations for device materials. For example, the permalloy-silver study showed that enhanced low field magneto-resistivity is associated with antiferromagnetic interlayer coupling that arises due to structural segregation produced by annealing. Preliminary studies of annealed permalloy-copper multilayers indicate that the antiferromagnetic exchange results from a completely different mechanism. Measurements of exchange-biased oxide nanostructures reveal that the ordering temperatures of the antiferromagnetic components is strongly influenced by their local magnetic environment. Experiments are underway to test theoretical predictions regarding the dependence of the magnetic domain size in these oxide layers on field and temperature. We expect that this research will catalyze the use of these new weak coupling mechanisms in the design of new low--operating field magnetic sensors.

Highlights:
Following are examples of the significant contributions made in various research areas:

Some types of new generation thin film magnetic sensor devices will require biasing by additional magnetically hard layers. Insulating antiferromagnets have been suggested as candidates for these biasing materials. A collaboration between Reactor Radiation Division scientists and scientists from the Florida State University Center for Materials Research and Technology (MARTECH) has shown that there is substantial perturbation of the biasing layer (here NiO) magnetic structure when a sensor layer (such as magnetite) is magnetized. These neutron diffraction measurements provide a means of testing the degree of magnetic hardness required for the biasing layer materials.

Using x-ray and polarized-neutron reflectivity, we are studying a series of Ni/sub 80/Fe/sub 20//Ag multilayers, annealed at different temperatures, in collaboration with a group at IBM. The samples annealed near 335 C show giant magnetoresistive (GMR) effects at anomalously low magnetic fields, making them excellent candidates for recording heads. Neutron measurements reveal that the ferromagnetic moments are antiferromagnetically coupled along the growth-axis with in-plane domains limited to 1 - 5 microns in size, in contrast to related GMR materials such as Fe/Cr. Future experiments are planned to tailor this domain size and thus control the field sensitivity of these devices to expand the range of possible magnetic sensor applications.

TECHNICAL AND PROFESSIONAL COMMITTEE PARTICIPATION AND LEADERSHIP 1995

American Chemical Society
- A. C. Mighell, Division of Chemical Information

American Crystallographic Association
- A. D. Mighell, A.I.P. Crystal Data Committee
- E. Prince, Publication Committee
- J. K. Stalick, Neutron Scattering Special Interest Group, Secretary

American Physical Society
- J. A. Borchers, Session Co-Chairman
- J. W. Lynn, Symposium Chairman

American National Standards Institute
- T. M. Raby, Standards Steering Committee
  - National Chairman, N-17
  - Working Groups, Chairman ANS-15.1, ANS-15.4, ANS-15.11
- J. F. Torrence, Working Groups, ANS-15.1
  - N-17 National Committee Member

Brookhaven National Laboratory
- C. G. Glinka, HFBR Proposal Review Sub-Committee in Materials Science
- C. F. Majkrzak, Chairman, HFBR Program Advisory Committee
- J. M. Rowe, Advisory Committee on HFBR upgrade
  - Physics Visiting Committee
- J. J. Rush, Neutron Scattering Facility Scheduling Committee

Council on Materials Science and Engineering of the Southeastern Universities Research Association
- J. W. Lynn

Department of Commerce
- Silver Medal Award
  - J. R. D. Copley
  - D. A. Neumann
- Bronze Medal Award
  - J. H. Ring
  - S. K. Satija

Department of Energy
T. M. Raby, Member, Basic Energy Sciences Sub-committee
J. M. Rowe, Search Committee for Director of Basic Energy Sciences
Committee on Cost and Operation of Nuclear Reactors
J. J. Rush, Vice Chairman, Basic Energy Sciences Advisory Committee Panel on Neutron Sources

Elastic Scattering Working Group at the Long Pulse Spallation Neutron Source Workshop (Berkeley, CA, April 18-21, 1995)
C. J. Glinka

Fourth Surface X-ray and Neutron Scattering Conference (Lake Geneva, WI, July 1995)
S. K. Satija, Program Committee

International Advisory Committee for Fourth Surface X-ray and Neutron Scattering Conference, Argonne, IL
C. F. Majkrzak

International Advisory Committee for Fifth Conference on Applications of Nuclear Techniques
H. Prask, Organizer

International Advisory Council of the Canadian Journal of Physics
J. M. Rowe

International Conference on Neutron Scattering, Oxford, England
J. W. Lynn, Rapporteur

International Conference on Neutron Scattering, Sendai, Japan
C. J. Majkrzak, International Advisory Committee

International Conference on Neutron Scattering (Toronto, Canada - 1997)
J. W. Lynn, Organizing Committee
Treasurer, ICNS'97

International Conferences on Hydrogen in Metals
J. J. Rush, Advisory Committee

International Symposium on Nondestructive Characterization of Materials (Prague, June 1995)
H. Prask, Session Chairman

International Union of Crystallography
E. Prince, Commission on International Tables
Commission on Neutron Scattering
Commission on Crystallographic Nomenclature
A. Mighell, Data Commission
B. H. Toby, Commission on the Crystallographic Information File

International Workshop on Itinerant Electron Magnetism (Crimea, Ukraine)
J. W. Lynn, Program Committee

JCDDS-International Centre for Diffraction Data
A. D. Mighell, Crystal Data Management Board
Crystal Data Committee
Data Subcommittee
Electron Diffraction Subcommittee
J. K. Stalick, Data Sub-Committee
B. H. Toby, Technical Committee

Los Alamos National Lab
J. M. Rowe, Advisory Board on LANSCE
J. J. Rush, LANSCE Upgrade Review Committee

Magnetism and Magnetic Materials Conference
J. L. Borchers, Program Committee
J. W. Lynn, Symposium Chairman
Advisory Committee

Massachusetts Institute of Technology
J. M. Rowe, Visiting Committee, Nuclear Engineering Dept.

NAS/NRC
J. J. Rush, Solid State Sciences Committee

National Organization for Test, Research and Training Reactors
T. M. Raby, Past Chairman and Member of Executive Committee

National Science Foundation
W. A. Kamitakahara, Member of Evaluation Panel for CAREER Proposals

National Science and Technology Council
J. J. Rush, Subcommittee on Research Infrastructure

Neutron Optical Devices and Applications, SPIE, San Diego, CA
C. F. Majkrzak, Conference Chairman
J.R.D. Copley, Co-Chairman

Neutron Scattering Society of America
W. A. Kamitakahara, Secretary

Materials Research Society Neutron Scattering Symposium
D. Neumann, Co-Chairman

NIST/NSF Center for High Resolution Neutron Scattering (CHRNS)
C. J. Glinka, Project Director

Rare Earth Research Conference
J. W. Lynn, Session Chairman

Second Workshop on Software Development at Neutron Scattering Sources (Softness '95)
J. R. D. Copley, Organizer

Summer School on Neutron Small Angle Scattering and Reflectometry from Submicron Structures
C. J. Glinka, Co-organizer
B. Hammouda, Co-organizer
C. F. Majkrzak, Co-organizer
S. K. Satija, Co-organizer

Surfactancy and Self-Assembly Technical Advisory Committee for the University of Minnesota's Center for Interfacial Engineering
C. J. Glinka

University of Missouri, External Review Committee
J. M. Rowe, Member

TECHNICAL AND PROFESSIONAL
INDUSTRIAL AND ACADEMIC INTERACTIONS

•As a national center for the development and application of neutron methods in condensed matter and materials science, chemical analysis and radiation standards, reactor-based programs include direct interactions and cooperative research efforts with 90 U.S. universities and 52 U.S.industrial laboratories. Of the over 1150 yearly participants in neutron-based research programs, half are from U.S. university or industrial labs.

•An agreement is in place between NIST and Exxon Research and Development Corporation to jointly and operate a world-class small angle neutron scattering (SANS) spectrometer and a develop a spin-echo spectrometer at the NIST Cold Neutron Research Facility (CNRF). The SANS facility is now in full operation. Cooperative research efforts include work on wetting in microporous media and micellar systems and research on new polymers. The spin echo spectrometer, which allows unique measurements of the details of dynamic processes in the 10^{-7} - 10^{-10} second time regime in polymers, colloids, biological molecules, and magnetic systems, and will be the only such instrument in the United States for the next decade, will be installed by the end of 1996.

•The NIST/NSF Center for High Resolution Neutron Scattering (CHRNS) at the CNRF fully operational. It offers U.S. scientists access to world-class capabilities in high resolution small angle neutron scattering studies of materials microstructure and in very high resolution spectroscopy of condensed materials using polarized neutron beams. NSF has also provided funding for a next generation perfect-crystal SANS to extend the range of microstructure measurements available to U.S. researchers.

•A Participating Research Team (PRT) agreement has been implemented with Texaco R&D Co. and M.I.T to join NIST, Exxon and U. Minn. in the further improvement and utilization of the NG-7 30-meter SANS facility.

•A joint research effort is underway with Dupont scientists to combine thermal and cold neutron scattering methods to provide key molecular-scale information of chemical structure and processes in environmentally benign refrigerents and zeolite storage media.

•A collaborative research effort with IBM and NRL is underway to determine the magnetic structure of multilayers comprised of $Ni_{80}Fi_{75}$/Ag and related transition-metals. Because of their anomalous magnetoresistive properties, these materials are ideal candidates for magnetic sensor devices such as computer read heads. Spin-polarized neutron reflectivity elucidates details of the magnetic behavior not accessible by bulk measurement techniques.

•The Neutron Scattering group has a wide-ranging collaborative research program with the University of California (Santa Barbara) involving neutron inelastic scattering, neutron diffraction, and SANS studies of catalysts, non-linear optical materials, and radiation damage.

•The Reactor Radiation Division has a long-term cooperative research program with the Department of Physics of the University of Maryland. Under this program RRD staff are engaged with Maryland scientists in joint research on magnetic materials, superconductors, polymers, and construction materials. Some of this research is carried out jointly with scientists from industrial labs.

•The University of Minnesota, through its Center for Interfacial Engineering (CIE), is a partner with the Reactor Radiation Division in the development and use of two high- resolution instruments at the CNRF: a 30-meter SANS spectrometer and a cold neutron Reflectometer. The CIE, with over fifty affiliate or sponsoring companies, is using these instruments with NIST for the elucidation of the structure and microstructure of polymers and other materials, as well as surface and interfacial structure and interactions. The Reactor Radiation Division is, in turn, a CIE affiliate and member of the Surfactancy and Self Assembly Program and the Polymer Microstructure Program Technical Advisory Committees.

•The Center for Materials Science and Engineering at the MIT is a collaborative partner with the Reactor Radiation Division in the use of the recently completed 32-detector, high resolution powder neutron diffractometer for materials research at the NIST Reactor. The new instrument, has been improved during the 1994-95 reactor shutdown and possesses state-of-the-art capabilities in resolution, versatility, and data collection efficiency. Of particular initial interest in the collaborative program are structural studies of fast-ion conductors, and advanced ceramics.

•A cooperative research effort established is in place with the Physics Department at Johns Hopkins University for joint research on new magnetic materials, including low dimensional magnetic systems is continuing. As part of this agreement JHU has provided is providing funding and concept design support for joint development of a next generation subthermal neutron triple axis spectrometer at the CNRF.

•A collaborative research effort with guest scientists from the Institute of Crystallography, Moscow, the Max Planck Institute for Polymer Research, Germany, and the University of Akron, Ohio, is underway to understand the microscopic structures of organic Langmuir-Blodgett (LB) film systems. The principal probe in these investigations is neutron reflectivity in which the large difference in neutron scattering power for hydrogen and deuterium isotopes, makes it possible to determine the chemical composition profile normal to the plane of the film with near nanometer resolution, not possible with any other technique.

•Collaborative research efforts with the University of California at San Diego and Florida State University are examining NiO/CoO and NiO/Fe_3O_4 films and superlattices as new magnetic-film candidates for low-cost high-density data storage and recording-head materials. Magnetic neutron diffraction has elucidated a number of important properties in these materials.

•Reactor Radiation Division scientists has established a joint effort with the University of Pennsylvania on computer simulation biological interfaces and biomolecular dynamics. This

cooperative effort is part of a joint program with the NIST Biotechnology Division on neutron studies of biological structure and dynamics.

•Newly commissioned at the BT-8 beam port at the NIST Reactor is a state-of-the-art facility for texture, residual stress measurements, and single-crystal diffraction. The new instrument--in addition to those features which facilitate sample handling, measurement continuity, and versatility--is about an order-of-magnitude faster in measurement time than the previously-used BT-6 spectrometer--and equal or superior to other neutron instruments, worldwide, for precise stress measurements. Ongoing and new programs which will benefit enormously from these improved capabilities include collaborations with the Metallurgy Division, Army Armament RD&E Command, John Deere Co., Ceramics Division, Boeing, Alcoa, General Motors, M.I.T., ONR, and the Department of Transportation.

•The NIST Reactor Radiation Division has recently joined with NSF, the University of Pennsylvania and the University of California at Santa Barbara, and their industrial partners Dupont, Hughes Aircraft, and Allied Signal to greatly advance U.S. capabilities in neutron spectroscopy for studies of new materials. Joint efforts will be focused on the development and operation of a new high intensity neutron spectrometer with unparalleled sensitivity to be installed at the NIST reactor. In addition to leading the development of this instrument, NIST scientists will pursue joint generic and strategic research on new materials (e.g. catalysts, light-weight batteries, conducting polymers, fullerenes) with the university/industry research teams. One-quarter of the time on the new facility will be allocated to the general scientific community on the basis of scientific merit through the NIST proposal system.

Associated Activities

During the past year staff and visiting scientists in the Reactor Radiation Division delivered 45 invited lectures in the United States and abroad. Group members were invited participants and organizers in a number of symposia in the neutron and materials fields. Neutron Group scientists also continue to share several NATO grants with French and German colleagues in different areas of condensed matter physics. Workshops on Crystallographic Databases and applications of neutron reflectivity were organized by RRD scientists, and were well-attended and highly successful.

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