NIST

U.S. DEPARTMENT OF COMMERCE
Technology Administration

RESEARCH. SERVICES. FACILITIES.

NATIONAL INSTITUTE OF STANDARDS AND TECHNOLOGY

GAINING THE COMPETITIVE EDGE

Sales of U.S. exports are booming. Business and innovation are flourishing, and the United States is an industrial power to be reckoned with. The year is 1901 and the U.S. Congress has just created the National Bureau of Standards to help the nation's industries reach their full potential.

Ninety years and a new name later, the National Institute of Standards and Technology continues its mission with renewed purpose. After more than a decade of trade deficits and losses in global market share in key industries, U.S. industry's prospects are again on the rise. Exports are up. Productivity is rising. While competition remains fierce, a ground swell of new determination among U.S. business leaders to produce high-quality, low-cost products makes me optimistic about the future.

Here at NIST we have a similar determination. We have made it our top priority to help U.S. industry improve the quality and international competitiveness of its products. In the last few years, we have added new services and outreach efforts to complement long-standing research programs of proven practical value to business and industry. We have expanded the range of topics available for research collaborations and the mechanisms for doing so—from one-on-one joint studies to industry/government consortia with dozens of members. And we've worked through NIST's management of the Malcolm Baldrige National Quality Award to help U.S. companies adopt management practices that improve product quality and customer satisfaction.

Each year more than 1,000 researchers from industry, universities, or other government agencies come to NIST to conduct cooperative research projects lasting from a few weeks to several years. What they find here is a capable, motivated staff and many first-class laboratory facilities—like a 20-megawatt research reactor with a cold neutron source, a video supercomputer, and an automated manufacturing research facility.

In years past, some U.S. companies have been reluctant to conduct joint research with federal agencies for fear their competitors could gain access to proprietary information through Freedom of Information Act (FOIA) requests. Other firms have been unwilling to invest in cooperative research when the results of that research would be co-owned with the government and thus available to competitors.

Since the passage of the Federal Technology Transfer Act in 1986 these fears have been laid to rest. The act explicitly protects proprietary information provided by a cooperative research and development partner from FOIA requests. It also allows federal laboratories to grant exclusive licenses to cooperative research partners for the intellectual property developed during a given project.

In today's fast-paced business environment, major scientific advances are commercialized in a year or two rather than a decade, and product life cycles are dramatically shorter. Business survival demands that companies continuously improve their products and fine-tune their processes. In such an environment, NIST research, services, and facilities can help provide companies with that critical competitive edge needed for success in the marketplace.

The pages that follow describe the full spectrum of NIST programs and facilities available for industry participation and use. Each item includes the name and phone number of a NIST program manager or researcher to contact for more information. Take time now to find the NIST resources and research projects of greatest interest to you. Then give us a call. Chances are there's a NIST program or service that can help your company meet its research and product quality goals.

John W. Lyons
Director

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SERVING THE CUSTOMER

MEETING CHALLENGES

In business circles these days, the concept of "total quality management" is rapidly taking hold. Its main tenets are quite simple. Involve everyone in the organization from production-line workers to the CEO in producing quality products and never lose sight of one thing—your customers.

At the National Institute of Standards and Technology, U.S. businesses, large and small, are our most important customers. Our full range of programs—from direct grants for development of generic technologies to frontier studies of physical laws to calibrations of quality control instruments—is designed to help U.S.-based companies compete in the marketplace.

NIST is a strategic national resource. The Institute offers a "critical mass" of resources, research personnel, and facilities that can help firms leverage their research and development investment and speed progress toward meeting technology goals for new products or improved processes. It does so through several major avenues:

- direct technical or financial assistance to U.S.-based companies;
- measurement and standards services, such as Standard Reference Materials or equipment calibrations;
- joint research with industry, university, or other government scientists and engineers;
- standards activities with industry; local, state, and federal governments; and international organizations; and
- availability of selected research facilities for cooperative research or for proprietary work on a cost-recovery basis.

Within each of these areas, many different levels of interactions are possible. Technical assistance provided by NIST or its regional Manufacturing Technology Centers may mean answering a single question over the phone or offering guidance for a complete overhaul of a company's shop floor operations. Joint research may involve exchanging a few samples for measurements or several years in which an industry researcher works side-by-side with a NIST researcher on a project of mutual benefit. In fact, cooperative efforts may involve any number of different combinations of personnel, equipment, funds, or facilities resources.

TANGIBLE BENEFITS

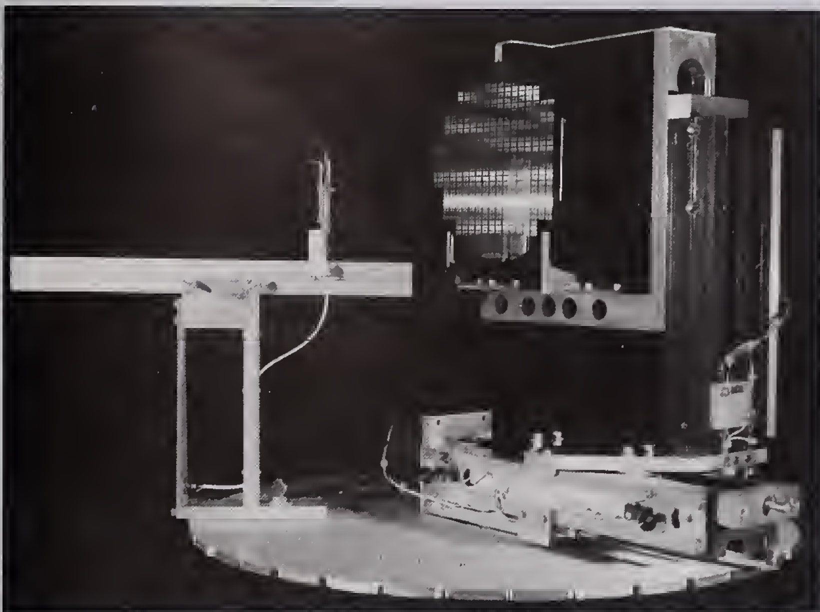
Companies that take advantage of NIST programs reap tangible, lasting benefits. Bell Helmets recently cooperated with NIST's "Shop of the 90s" researchers, six computer software and hardware companies, and the United States Performance Engineering Program to produce an aerodynamic helmet for the U.S. Olympic speed-skiing team. In about 1 month, the cooperative research and development project resulted in a computer-designed and manufactured (CAD/CAM) mold for producing the futuristic-looking helmets.

Prior to the project, Bell Helmets made all its new helmet molds by hand. With its new CAD/CAM expertise, the company now plans to convert all of its research and development design work to computer technology by 1993. The changeover should save the company up to 9 months of design time for each new helmet.

Soft X-ray "mirrors" made from a sample material, held here by physicist Richard Watts, could dramatically improve the performance of certain telescopes, lasers, microscopes, and semiconductor integrated circuits.

In a different type of cooperative effort, NIST physicists have provided critical measurements to AT&T Bell Laboratories, the Lockheed Corp., Ovonics Inc., and several government and university laboratories involved in the new field of soft X-ray optics. With a special characterization facility—the only one of its kind in the United States available to the general scientific community—the NIST researchers evaluate multilayer "mirrors" provided by the companies and used for focusing X-ray beams like a lens focuses light.

Measurements of properties like homogeneity, reflectivity, and surface roughness made at the NIST facility allow the cooperating companies to accelerate their research by matching precise property data with processing parameters. A lucrative market awaits commercial production of the new X-ray "mirrors," which could dramatically improve the performance of certain telescopes, lasers, and microscopes and allow semiconductor manufac-



Using the neutron depth profiling apparatus with the new cold neutron source at the NIST 20-megawatt research reactor, NIST researchers have collaborated with the Intel Corp. to map precisely boron concentrations in silicon wafers.

turers to further shrink the size of integrated circuits.

An example of NIST working with industry on a large scale is the North American ISDN Users' Forum. The group addresses the protocol standards and other user needs for the emerging technology called Integrated Services Digital Network (ISDN). The technology promises to revolutionize telecommunications by making it possible to send voice, data, and images simultaneously over telephone lines. Working cooperatively, NIST computer researchers and forum committees have developed a series of test specifications for these systems designed to ensure that ISDN products by different manufacturers work compatibly. The recent acceptance of these test specifications by international standards organizations governing such standards will enhance the ability of U.S.-based manufacturers to compete in international markets.

BETTER "CHIPS"

A project carried out over the last 7 years between NIST chemists and researchers from Intel Corp. has helped the company reduce waste in production of its advanced semiconductor "chips." Working at the Institute's 20-megawatt research reactor, Intel and NIST researchers use the unique capabilities of two neutron depth profiling facilities to map precisely the chemical composition of silicon wafers used in making integrated circuits. The non-destructive method allows the researchers to pinpoint the location and concentration of otherwise difficult-to-measure elements like boron in the surface of the wafer and then to test the same sample with optical or chemical analysis techniques. Precise chemical composition data are critical for quality production of the boron-containing glasses used as insulating layers in such circuits.

In the same field of semiconductor manufacturing, a senior research fellowship program coordinated by the American Statistical Association with funds from the National Science Foundation placed an industrial research consultant on a 1-year appointment at NIST to study statistical methods for improving quality control. The consultant and NIST statisticians and electronics engineers applied an alternative model to one widely used in

NIST AT A GLANCE

The National Institute of Standards and Technology was established by Congress to "assist industry in the development of technology . . . needed to improve product quality, to modernize manufacturing processes, to ensure product reliability . . . and to facilitate rapid commercialization . . . of products based on new scientific discoveries."

An agency of the U.S. Commerce Department's Technology Administration, NIST's main goals are to strengthen U.S. industry's competitiveness, advance science, and improve public health, safety, and the environment.

NIST conducts basic and applied research in the physical sciences and engineering, developing measurement techniques, test methods, standards, and related services. The Institute does generic and precompetitive research and development work on new advanced technologies.

Budget	$354 million (FY 91 estimated resources from all sources)
Staff	More than 3,000 scientists, engineers, technicians, and support personnel, plus some 1,000 visiting researchers each year
Sites	Gaithersburg, Md. (headquarters — 234-hectare campus) Boulder, Colo. (84-hectare campus)
Main Research Areas	Electronics and electrical engineering Manufacturing engineering Chemical science and technology Physics Materials science and engineering Building and fire research Computer systems Computing and applied mathematics

the industry to predict when integrated circuits will fail due to a phenomenon called "electromigration."

As manufacturers make integrated circuits smaller and smaller, high densities of electrical current running through microscopic "wires" in the circuits are more likely to cause the aluminum in the "wires" to move or migrate out of position. When enough metal in the "wire" moves, a void forms in the circuit and the entire device fails. The NIST cooperative research team has suggested an improved model that better accounts for circuits of varying lengths. Once the model is validated with laboratory experiments using special test structures, the industry as a whole should benefit through more accurate predictions of lifetimes for these complex devices.

INDUSTRIAL CONSORTIA

Other research programs incorporate formal consortia in which a group of companies exchange data, ideas, researchers, and/or materials with NIST to meet agreed-upon research goals. For example:

■ A NIST consortium consisting of Cascade Microtech Inc., ITT Defense and the Gallium Arsenide Center, TRW Electronic System Group, and several U.S. Department of Defense laboratories contributes funding to Institute research on standards and test methods for a new type of miniaturized microwave device. The devices incorporate components for generating or processing microwave and millimeter-wave signals on a single integrated "chip," just like current integrated circuits used in computers. The new technology could cut the cost of some microwave systems by tens of thousands of dollars. In return for their support of NIST research in this area, members of the consortium receive new standards and software developed through the program 1 year in advance of the general technical community.

■ A cooperative project with the Automotive Composites Consortium (ACC) aims to improve the manufacture of new structural polymer composites for use in future automobile frames. ACC members include Chrysler Corp., Ford Motor Co., and General Motors Corp. NIST materials science researchers have created a computer model that describes in three dimensions the flow of a polymer into a mold containing reinforcing fibers. The polymer hardens around the fibers to create these lightweight, yet strong, automotive frames. The participating companies will use the NIST flow model to improve the efficiency of their companies' fabrication of such components.

■ A consortium of manufacturers and trade associations provided $1 million through the National Fire Protection Research Foundation to fund NIST research on a new method for estimating fire risks. Working collaboratively with scientists and engineers from the National Fire Protection Association and Benjamin/Clarke Associates, a fire protection consulting firm, NIST researchers developed a way to estimate on a national scale fire death rates associated with new product designs.

The method uses fire incidence data to produce descriptions of tens of thousands of fires in different settings, such as single-family homes, offices, and hotels. This information is used with a NIST software program called HAZARD I to predict how new compositions or designs for furniture, building materials, or other products would affect the number of fire deaths associated with each scenario. Combining the expected outcomes from all of the fire simulations allows manufacturers to determine whether new product designs will increase or decrease fire risks compared with current products.

MALCOLM BALDRIGE NATIONAL QUALITY AWARD

First presented in 1988, the Malcolm Baldrige National Quality Award has quickly become both the U.S. standard of quality achievement in industry and a comprehensive guide to quality improvement. Tens of thousands of U.S. companies are using the application guidelines to evaluate their operations in seven key areas of quality management and performance: leadership, information and analysis, planning, human resource use, quality assurance of products and services, quality results, and customer satisfaction. The results of these internal evaluations provide firms with a clear view of where they stand and of how far they must go to achieve world-class levels of quality—and to compete for the national quality award.

The award program, developed and managed by NIST with the cooperation and financial support of the private sector, recognizes quality achievements in three categories: manufacturing, service, and small business. Up to two awards can be made in each category. Applications for the award undergo a rigorous evaluation by an independent review board composed of quality experts from the private and public sectors. Examiners conduct on-site reviews at firms that receive high scores after an initial screening. All applicants receive a written summary that identifies their strengths and points out areas for improvement.

Firms entering the competition pay a fee that covers the cost of the evaluation and feedback reports. Profiles on the quality programs of past award winners are available upon request. Copies of application guidelines also are available.

Contact: Malcolm Baldrige National Quality Award Office
A537 Administration
(301) 975-2036

PROJECT CONTACTS

To help you identify which NIST programs, services, or facilities best fit the needs of your particular organization, the remainder of this booklet provides detailed descriptions of individual activities. The cooperative research opportunities and facilities listed are representative of what is available within each major NIST laboratory. Unless otherwise noted, all addresses listed are at NIST, Gaithersburg, Md. 20899.

If you are not sure which NIST research area or facility matches best with your organization's needs, the NIST Technology Development Program staff can probably help you. This office handles NIST patents, licensing, cooperative research and development agreements, and other formal cooperative research contracts.

CONTACT: Bruce E. Mattson
A345 Physics
(301) 975-3084

ADVANCED TECHNOLOGY PROGRAM

Many of tomorrow's commercial success stories will start with today's basic research discoveries and technological innovations. Perhaps understood in only scant detail, today's laboratory curiosities provide a glimpse of potentially far-reaching future applications in products and processes. But in the foreground stand tremendous obstacles—high development costs and technical challenges that may take many years to resolve.

NIST's Advanced Technology Program (ATP) provides partial funding to single firms or industry-led joint ventures that undertake high-risk, high-return research projects to develop generic, precompetitive technologies. The aim of the cost-sharing program is to reduce some of the risk inherent in pursuing applications of emerging technologies. Once technical feasibility is established, companies can undertake on their own development of a prototype product and the other steps needed for commercialization.

The ATP embraces all areas of technology but emphasizes those with a broad range of potential applications. Grants to individual firms are limited to $2 million over 3 years; awards to joint ventures can be up to 5 years, with the total grant determined on a case-by-case basis. The private sector must share the costs of all ATP projects. Awards are made yearly, and the program budget is set annually by Congress.

Contact: Advanced Technology Program
A430 Administration
(301) 975-2636

Senior research fellow Wayne Nelson (left) poses with NIST collaborators (from left to right) electronics engineers Harry Schafft and John Suehle and statistician James Lechner. The team developed an alternative model to predict failure of integrated circuits due to a phenomenon called "electromigration."

TECHNOLOGY SERVICES

NIST provides a wide variety of services and programs to help U.S. industry get on with its most pressing tasks: innovation, rapid commercialization of new technology, and achieving total quality in all facets of business operations. The programs and services described below complement NIST's basic and applied research programs. New outreach efforts like the regional Manufacturing Technology Centers build on NIST's long-standing experience serving industry's needs for reference materials and standards development.

The measurement services work performed by Technology Services offices has applications in every area of manufacturing and technology development. If a process cannot be measured, then it cannot be fully understood. Incomplete understanding, in turn, increases the likelihood of process errors, product flaws, and other problems that lead to inefficiency and variations in quality. Companies spanning nearly all industrial sectors depend on the precision and reliability of NIST measurement services to keep their production processes running smoothly and efficiently.

Similarly, standards information and development are critical elements for every industry sector in the global marketplace. As national economies become more interdependent and as foreign sales account for an ever-larger share of many firms' bottom lines, domestic and international standards-setting activities have grown in economic and strategic significance. NIST staff members are active in more than 800 voluntary standards committees. In addition, the Institute provides a variety of informational and advisory services that can help businesses and other interested observers stay abreast of developments in the nation's and world's standards bodies.

Contact: Donald R. Johnson
A363 Physics
(301) 975-4500

MANUFACTURING TECHNOLOGY CENTERS

Hands-on technical assistance for small and mid-sized manufacturers—that's the mission of the five Manufacturing Technology Centers (MTCs) jointly funded by NIST, business, and state and local governments. Programs are tailored to the needs of local industry, but each MTC emphasizes technology transfer, helping manufacturers make effective use of the advanced technology most appropriate for their operations. The other common emphasis is educating companies on the concepts and practices of total quality management.

For many of the nation's more than 350,000 small and mid-sized manufacturers, moving up the technological ladder from manual operations to flexible computer-integrated manufacturing systems can be a risky and difficult task. Using in-house demonstration facilities, MTC teams of business, manufacturing, marketing, and training experts help companies identify the best combination of equipment and software for particular business needs. They also follow up with worker training programs and other measures to ensure that the new technology delivers all of its anticipated benefits.

CONTACTS:

General Information: Philip Nanzetta, MTC program director, B124 Metrology, NIST, Gaithersburg, Md. 20899, (301) 975-3414

Great Lakes MTC: George Sutherland, director, Great Lakes MTC, Cleveland Advanced Manufacturing Program, 2415 Woodland Ave., Cleveland, Ohio 44115, (216) 987-3200

Northeast MTC: Gene Simons, director, Northeast MTC, Rensselaer Polytechnic Institute, CII-9009, Troy, N.Y. 12180, (518) 276-6682

Southeast MTC: Steven Eisele, assistant director, Southeast MTC, University of South Carolina, Swearingen Engineering Center, Columbia, S.C. 29208, (803) 777-9595

Mid-American MTC: William Brundage, acting director, Kansas Technology Enterprise Corp., 112 S.W. 6th Ave., Suite 400, Topeka, Kan. 66603, (913) 296-5272

Mid-West MTC: Jack Russell, director, Industrial Technology Institute, 2901 Hubbard Rd., Ann Arbor, Mich. 48106, (313) 769-4690

STANDARDS AND CERTIFICATION INFORMATION

The NIST National Center for Standards and Certification Information is the U.S. focal point for information on standardization programs and related activities at home and abroad. Center staff provide information on U.S., foreign, regional, and international voluntary standards bodies, as well as mandatory government regulations and conformity assessment procedures for non-agricultural products. As the U.S. member of the International Organization for Standardization Information Network (ISONET), NIST has access to foreign national standards information through approximately 60 other ISONET members and the

ISO information center in Geneva, Switzerland. NIST also serves as the U.S. inquiry point under the General Agreement on Tariffs and Trade (GATT) Agreement on Technical Barriers to Trade.

At the center, NIST maintains an extensive collection of reference materials, including U.S. military and other federal government specifications, U.S. industry and national standards, international standards, and selected foreign national standards. Staff members respond to requests for information either directly or by identifying the most appropriate source of information. They also prepare directories and indexes of specialized standards information, arrange for translations of foreign standards, and issue periodic publications explaining ongoing developments in domestic and international standards activities.

Two telephone hotlines located within the center offer weekly updates on draft European standards [(301) 975-4164] and on proposed foreign regulations that may significantly affect trade [(301) 921-4041]. Information for the latter hotline is supplied by the GATT Secretariat in Geneva.

CONTACT: JoAnne Overman
A163 TRF
(301) 975-4037

The Great Lakes Manufacturing Technology Center helped AccuSpray Inc. redesign its product and production method. Here, AccuSpray employee Kevin Rowell demonstrates the special paint spraying equipment, which uses half the paint of conventional methods.

STANDARDS MANAGEMENT

NIST manages U.S. technical representation and participation in the International Organization of Legal Metrology (OIML) and administers the U.S. Department of Commerce's Voluntary Product Standards (VPS) program.

OIML is a treaty organization with a membership of 49 voting and 34 non-voting nations. It works to enhance trade by harmonizing national regulations governing performance requirements for measuring instruments used in commerce and for monitoring and maintaining public health and safety. NIST standards management staff solicit technical advice and support from U.S. trade associations, instrument manufacturers, academia, and federal and state regulatory agencies in developing and approving OIML draft documents.

Staff members also serve as the secretariat for the development of voluntary standards for selected products, with the costs being paid by proponent trade associations or other groups. Current standards published and maintained under this VPS program include softwood lumber, construction and industrial plywood, and glass bottles for soft drinks.

CONTACT: Samuel E. Chappell
A625 Administration
(301) 975-4023

WEIGHTS AND MEASURES

Helping state and local governments ensure the equity of weights and measures in the marketplace is one of NIST's longest running and best known programs. It includes a certification program for state weights and measures laboratories in the areas of mass, length, and volume, as well as providing test protocols, certification training, and ongoing laboratory assistance.

NIST sponsors the National Conference on Weights and Measures, which involves over 3,000 industry and regulatory agency representatives, and program staff produce numerous training manuals, handbooks, and other publications. The staff also operate an electronic bulletin board to provide the weights and measures community with a mechanism for rapidly exchanging information.

CONTACT: Carroll S. Brickenkamp
A617 Administration
(301) 975-4004

STATE TECHNOLOGY OUTREACH

The State Technology Extension Program staff work with state and local technology outreach programs to improve the competitiveness of small and mid-sized businesses through the application of science and technology. Assistance offered by program staff includes:

- stimulating cooperation and communication between and within states to enhance their capability to meet the needs of local businesses;
- collecting and disseminating information about successful technology assistance activities, such as best practices, model programs, and common tools; and
- providing matching grants for development and coordination of technology assistance activities.

CONTACT: Gale R. Morse
A343 Physics
(301) 975-4520

RESEARCH AND TECHNOLOGY APPLICATIONS

Facilitating the transfer of NIST-developed technologies to business and industry is the function of the NIST Research and Technology Applications Program. The office staff identify NIST research staff, publications, and technologies to respond to technical questions from individual businesses. Staff members organize or participate in workshops and seminars on specific technical topics and new technology transfer methods. Program staff also arrange visits to NIST for business and industry researchers and managers interested in learning more about Institute research programs.

The office staff serve as the NIST point of contact for the Federal Laboratory Consortium.

CONTACT: Joseph G. Berke
A343 Physics
(301) 975-5017

TECHNOLOGY DEVELOPMENT AND SMALL BUSINESS INNOVATION

A variety of outreach efforts managed under the Technology Development Program aim to increase the number of NIST cooperative research projects and to improve the transfer of NIST technologies to U.S.-based industries. Program staff members help industrial, academic, or government researchers locate NIST personnel and facilities in their fields of interest for either collaborative or proprietary research and they facilitate preparation of formal cooperative research and development agreements. They also handle the licensing and administration of NIST's more than 120 patents and other intellectual properties.

In a separate program, a percentage of NIST's extramural research budget is earmarked for funding of innovative research and development by small businesses—those with fewer than 500 employees. Each year, the Department of Commerce, NIST's parent organization, issues a list of recommended research and development topics. NIST personnel evaluate the proposals submitted by small businesses for research in these areas. Winners of Phase I awards receive $35,000 to support studies of technological feasibility. In Phase II, applicants may receive up to $200,000 to support development of promising technologies.

CONTACT: Bruce E. Mattson
A343 Physics
(301) 975-3084

To help ensure the accuracy of environmental quality measurements, NIST sells a wide range of Standard Reference Materials from "Estuarian Sediment" to "Trace Elements in Coal."

STANDARD REFERENCE DATA

Working closely with industry, NIST provides well-documented numeric data to scientists and engineers for use in technical problem-solving, research, and development. These recommended values are based on data which have been extracted from the world's literature, assessed for reliability, and then evaluated to select the preferred value. The evaluations are carried out through a network of data centers, projects, grants, and cooperative programs.

Standard Reference Data (SRD) have a variety of uses in industrial applications and are available as databases for personal computers, as well as in other computerized forms, and as publications. Common applications include use for calibration points (such as spectral wavelengths and transition energies) and as input to the design of new processes and materials. Among the many subjects covered by the data are analytical chemistry, atomic and molecular physics, chemical kinetics, fluid mixtures, thermochemistry, materials properties, and phase equilibria.

To increase the usefulness and accessibility of data, NIST has developed a series of personal computer databases with interactive programs, search routines, and other calculational and graphical software features. Most databases are updated yearly, adding more data and more software capabilities.

A free catalog of NIST SRD data products and services is available. Program staff also issue related publications, respond to telephone inquiries on data sources, and sponsor seminars on the program and selected data centers.

CONTACT: Malcolm W. Chase
A323 Physics
(301) 975-2200

STANDARD REFERENCE MATERIALS

Now numbering more than 1,200 and steadily growing to meet the needs of industry, Standard Reference Materials (SRMs) are a diverse collection of solids, liquids, and gases certified for their chemical composition or physical properties. With an SRM, companies can verify the accuracy of analytical or monitoring methods under development or calibrate established measurement systems to ensure consistently accurate performance of equipment and operators. Specific types of SRMs range from linewidth standards for producing integrated circuits to metal alloys for checking quality control in steel production, to radiopharmaceuticals for calibrating medical equipment.

NIST's SRM catalog provides a complete listing of available reference materials along with a description of their certified properties. In addition, the *Handbook for SRM Users* offers practical guidance on the use of the materials, describes the fundamental elements and concepts of quality control and measurement processes, provides advice on using control charts and statistical tools to evaluate measurement quality and uncertainty, and includes articles on quality assurance, sampling, and validation of analytical instruments.

The SRM program staff also publish timely information on reference materials, offer telephone consultations on SRM uses, and organize seminars to advise industry on SRM applications.

CONTACT: Lee T. Best
204 Engineering Mechanics
(301) 975-6776

CALIBRATION AND RELATED MEASUREMENT SERVICES

NIST provides more than 500 different services to ensure that manufacturers and other users of precision instruments achieve measurements of the highest possible quality. These services, which satisfy the most demanding and explicit requirements, link a customer's precision equipment or in-house standards to national standards. For calibrations and special tests, NIST personnel check, adjust, or characterize an instrument, device, or set of in-house, or transfer, standards. Customers are assured that measurements are consistent with national standards and adequate for their intended use. Besides individual equipment items, NIST measurement assurance programs calibrate entire measurement systems.

The full range of NIST calibration services ensures the accuracy and compatibility of measurements used in day-to-day quality-control applications. The services, available for a fee, encompass seven major areas: dimensional measurements; mechanics, including flow, acoustic, and ultrasonic; thermodynamics; optical radiation; ionizing radiation; electromagnetics, including direct current, alternating current, radio frequency, and microwave; and time and frequency.

The program staff publish a regularly updated catalog describing available services, fee schedules, and detailed descriptions of calibration protocols. Staff members offer telephone consultations on the use of services and on the importance of traceability to national standards, and they make presentations to business groups and other organizations.

CONTACT: Joe D. Simmons
B362 Physics
(301) 975-2002

VOLUNTARY LABORATORY ACCREDITATION

Staff of the NIST National Voluntary Laboratory Accreditation Program (NVLAP) rigorously evaluate the competencies and technical qualifications of public and private laboratories for conducting specific tests or types of tests in key areas of commerce, health, and safety. Among other benefits, certification of proficiency by the NIST program provides laboratory managers with a quality-assurance check on laboratory performance, concrete advice for improving performance, and national recognition of competency. At the same time, program certification helps users—from industry, government, and elsewhere—identify providers of high-quality testing services.

For a fee, NVLAP currently accredits laboratories offering services in the following areas: acoustical testing, asbestos fiber analysis, product testing (carpets, paints, papers, plastics, plumbing, seals, and sealants), testing of interface protocols for computer networks, construction testing services, electromagnetic compatibility and telecommunications, personnel radiation dosimetry, wood stoves, and thermal insulation materials. Any interested laboratory, organization, or agency can request accreditation in these and other areas. Requests for expanded program services will be evaluated on a case-by-case basis.

The program staff publish an annual directory of NVLAP-accredited laboratories.

CONTACT: Albert Tholen
A146 TRF
(301) 975-4016

ENERGY-RELATED INVENTIONS

In cooperation with the U.S. Department of Energy (DOE), NIST evaluates new product or process ideas for their potential to improve energy efficiency, reduce energy costs, or increase energy supply. Besides their energy-related merits, inventions are evaluated on the basis of technical and commercial feasibility. Inventions may be submitted to the program at any stage of development—conceptual, prototype, or actual production. All inventors are informed, in detail, of the results of the free evaluations.

Inventions that satisfy NIST criteria are recommended to DOE, which may choose whether to support development and commercialization. Since 1975, DOE has awarded more than $24 million to move NIST-recommended inventions closer to commercialization. In addition, inventors may use the NIST recommendation report to help attract private investment capital.

CONTACT: George Lewett
A115 TRF
(301) 975-5500

INFORMATION SERVICES

The Office of Information Services maintains a comprehensive international collection of information in scientific disciplines such as metrology, mathematics, computer science, and materials science. The staff serve the technical information needs of NIST scientists and engineers and communicate the results of NIST research to scientific and engineering communities worldwide.

The office staff participate in national and international publications and technical information networks and consortia, as well as a document exchange program to ensure that NIST publications are available to scholars, scientists, engineers, industry researchers, and others. An inquiries service assists the public in obtaining information about past and present NIST programs, publications, and special projects.

CONTACT: Peggy M. Saunders
E128 Administration
(301) 975-3058

ELECTRONICS AND ELECTRICAL ENGINEERING LABORATORY

Vying in fiercely competitive world markets, U.S. manufacturers of electronic products continually push the limits of current technology, presenting new and ever-more-stringent demands for measurement accuracy and precision. NIST's Electronics and Electrical Engineering Laboratory works to meet these demands, consulting closely with industry to identify the most critical measurement needs in the manufacture of semiconductor, magnetic, radio frequency, microwave, optical, and optoelectronics devices and products, as well as electrical power systems.

In the area of lightwave technology, for example, the laboratory is developing methods for evaluating the efficiencies of interconnections in optical-fiber networks. In another project, researchers use a newly installed video supercomputer to develop improved circuit designs and test methods for advanced imaging technologies. Other new or stepped-up research programs are addressing the measurement needs of companies employing advanced magnetic or microwave technologies.

While working to improve manufacturing methods for silicon-based electronic devices, NIST researchers are also looking beyond the current technological horizon to future generations of devices that will embody new materials and create new market opportunities. The laboratory's fundamental studies on the properties and characteristics of high-temperature superconductors support American industry's efforts to develop commercial applications for the highly promising, yet technically challenging materials. Other basic research is paving the way for the manufacture and use of devices that transmit and process both electronic and lightwave signals.

This laboratory provides the fundamental basis for all electrical measurements in the United States.

Contact: Judson C. French
B358 Metrology
(301) 975-2220

COOPERATIVE RESEARCH OPPORTUNITIES

ELECTRICITY

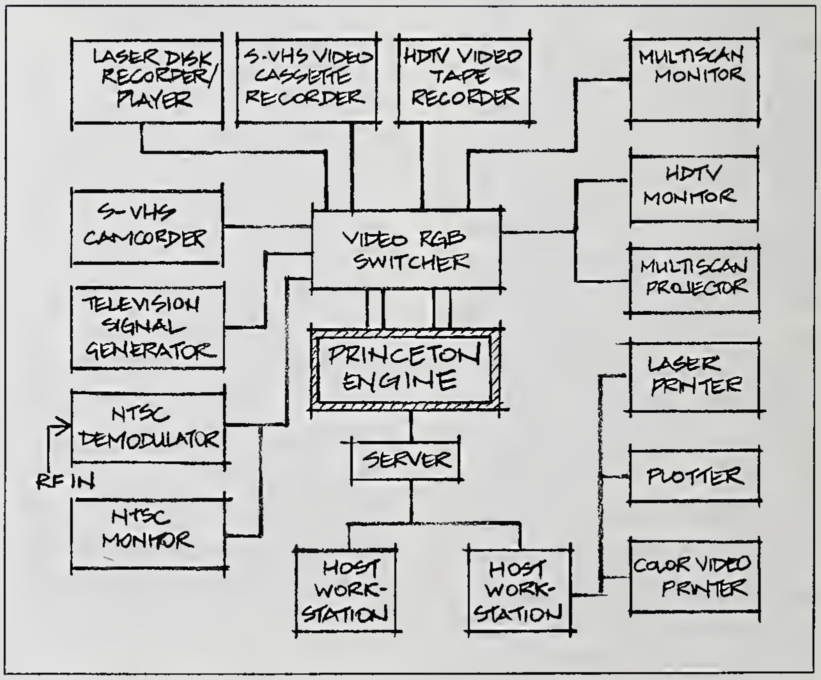
VIDEO PROCESSING

Data compression, motion encoding, scan-rate conversion, scientific visualization, image analysis, and other video processing research topics are being explored at NIST using a massively parallel video supercomputer, the Princeton Engine. The Princeton Engine, developed by the David Sarnoff Research Center, Princeton, N.J., is a 14-giga-instruction-per-second image-processing system capable of simulating video rate signals, including conventional National Television Standard Code and high-definition video, in real time. Because the engine is programmable, it can be used to evaluate software prototypes of image-processing components rapidly and at a cost below that of building hardware.

The Princeton Engine consists of 1,024 parallel processors, where each 16-bit processor has its own ALU, multiplier, and 128 kilobytes of local memory. Each processor operates on one picture element per video scan line, and all processors execute the "same" instructions. Wideband input and output channels accept and produce a number of analog and digital video formats. For many applications, video data can be processed and output at the same rate as they are input, that is, in real-time. For longer algorithms up to 7 seconds of video data may be acquired in real time, stored in local memory, and, once processed, displayed at the output at the original data rate.

Programming is accomplished using a proprietary graphical programming system intended to

Below. Schematic of the Princeton Engine and associated equipment. Right. With images of the ozone hole over Antarctica, electronics engineer Bruce Field demonstrates how the Princeton Engine video supercomputer can be used to design circuits for compressing high-definition television signals.



simulate a circuit design of video-processing circuits. Video recorders, multiscan monitors, high-definition monitors, and additional video support equipment are available.

NIST researchers are interested in using the Princeton Engine laboratory for a wide variety of collaborative research projects. Such collaborations would focus on precompetitive research with broad applications in advancing the state of the art in high-definition systems.

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ELECTRICAL METROLOGY WITH OPTICAL SENSORS

Researchers at NIST are developing electro-optical methods to measure electrical quantities and phenomena as part of a program to develop theory, methods, and physical standards for measuring electrical quantities in advanced high-voltage/high-power systems. Theoretical studies are focused on the use of finite-element code for electric-field computation and computer-aided data acquisition and analysis. Experimental research includes high-voltage ac, dc, and impulse measurements; high-speed camera techniques; optical multichannel analyzers; and lasers and detectors.

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GASEOUS ELECTRONICS

NIST scientists are developing measurement methods to characterize gaseous dielectrics for high-voltage power systems. Emphasis is on investigation of phenomena that affect reliability and safety associated with operation of gas-insulated systems, such as production of toxic byproducts in electrical discharges. Theoretical work addresses Boltzmann equilibrium statistics, chemical kinetics code, and computer-aided data acquisition and analysis. Experimental work focuses on high-voltage ac and dc tests, gas chromatograph and mass spectrometer techniques for chemical characterization, and partial discharge measurements. Researchers are investigating rf discharges used for processing electronic materials, including the study of plasma diagnostics and kinetics as they apply to the plasma processing of semiconductor materials.

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AC VOLTAGE STANDARDS

NIST is conducting both theoretical and experimental research on the synthesis of precision ac waveforms for use in ac voltage standards operating nominally below 10 MHz. The theoretical work includes Walsh functions and Fourier analysis, time-domain analysis, and precision RMS-to-dc conversion techniques. Experimental work involves high-speed, high-accuracy digital-to-analog conversion; precision, high-speed switching; assembly and interpretive-level programming for hardware control; and wideband, fast-settling amplifiers.

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TESTING ELECTRONIC SYSTEMS

New strategies are needed to evaluate the performance of complex electronic circuits using the fewest possible tests. A program is under way at NIST that includes theoretical studies of modeling for non-linear systems, optimization techniques using matrices, statistical and random processes, and artificial intelligence. In addition, experimental work will address test strategies for component and instrument testing; fault diagnosis, functional testing, and calibration; and computer analysis using both desktop computers and supercomputers.

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WAVEFORM RECORDER STANDARDS

As part of a program aimed at meeting the metrological needs involved in improving signal acquisition and processing systems, NIST researchers are developing theory, methods, and standards for waveform metrology of conducted signals. The scientists are conducting the theoretical and experimental research necessary to develop standards for determining the performance of waveform recorders operating nominally below 1 GHz and will develop techniques for synthesis of precision waveforms and for characterization of those waveforms. Theoretical studies will be conducted on Fourier analysis, deconvolution techniques, and time-domain analysis. Program plans include experimental work in precision pulse generation, static and dynamic testing, and assembly and interpretive-level programming for hardware control.

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ADVANCED AC VOLTAGE AND CURRENT MEASUREMENTS

Thermal voltage and current converters offer the most accurate and broadband method for measuring ac voltage and current for applications in communications, power generation, aerospace, and defense. Thermal transfer standards are calibrated by NIST in terms of reference converters, which have themselves been characterized by reference to the NIST primary standards—special multijunction thermal converters whose performance is known. These primary and working standards in common use throughout the metrology community employ thermal converters fabricated from wire elements. Researchers at NIST are studying new methods for the manufacture of film thermal converter structures made by the use of photolithography on silicon substrates. The application of this new technology may result in improved performance and reduction in the cost of thermal converters.

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JOSEPHSON-EFFECT VOLTAGE STANDARDS

High-accuracy voltage-standard systems have proliferated among many industrial, government, and international standards laboratories with the advent of the Josephson-array device. Within this laboratory, there are three array voltage-standard systems in operation, including a fully automated 10-V array system. Guest researchers can gain hands-on experience with array system operation and verification, as well as cooperate on studies into both precision voltage metrology and Josephson array physics. The metrology goals are to improve measurement precision to better than one part in 10^8 in applications of direct system-to-system intercomparisons and lab-to-lab volt transfers, achieve greater reliability in automation algorithms, and further the development of solid-state reference standards and precision digital voltmeters. The physics research addresses the effects of electromagnetic noise on the stability and accuracy of the Josephson quantized-voltage steps, studies the boundaries of chaotic behavior in junction-junction interactions, and explores other possible array device applications, such as the generation of ac signals through frequency modulation of the millimeter-wave drive frequency.

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RESISTANCE STANDARDS AND MATERIALS

Component precision resistors of both film and wire construction have found widespread use as references and dividers in precision instrumentation, such as digital multimeters and calibrators. The quality of these resistors and their level of immunity to the effects of environmental parameters, such as temperature and mechanical shock, have enabled the 3-month performance of these instruments to begin to approach that of the standards most commonly used to calibrate them. This fact and the desirability of calibrating such instruments where they will be used has engendered the need to develop the next generation of resistance standards—standards whose performance in adverse conditions would eclipse that of existing standards in a laboratory environment.

NIST scientists are beginning a program to develop new standards with sub-ppm performance, both short- and long-term, under field conditions. To do so, the electrical/physical properties of a number of alloys are being investigated, and new resistor designs are being formulated and tested. Future efforts will investigate metallurgical techniques such as rapid quenching, ion implantation in glasses and semiconductors for resistors $>10^7\ \Omega$, and Evanohm or Nichrome film deposition on Si substrates. The desired output will be fixed-value standards in the range from 1 Ω to $10^{12}\ \Omega$ with sub-ppm per year drift rates, temperature coefficients less than 0.1 ppm/°C, and low power and voltage coefficients. A metallurgical facility with the capability of monitoring the electrical properties of materials during annealing and a silicon processing facility are available along with access to precision resistance measurement systems and the national resistance standards.

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QUANTUM HALL EFFECT

When an electrical current is passed through the microscopically thin layers of a GaAs/AlGaAs heterostructure at low temperatures in very high magnetic fields, plateaus of constant resistance for certain values of the magnetic field are observed. These values of the device resistance are equal to h/ie^2 where h is Planck's constant, i is an integer, and e is the electronic charge. On Jan. 1, 1990, this effect, which is known as the quantum Hall effect, became the basis for the new world standard of resistance. The adoption of this standard was in large part due to the development of measurement systems of unprecedented accuracy developed at NIST. While the quantum Hall effect provides a standard of resistance that does not drift with time and is independent of the specific device used, many features of the effect and its use as a basis for a standard are not well understood.

Researchers at NIST are attacking these problems on three broad fronts, including understanding the physical principles underlying the effect, understanding sample-specific artifacts, and improving the measurement systems. A 15.5-T magnet and dilution refrigerator facility is being used to conduct research on the range of parameters over which the quantum Hall effect provides the most accurate and reproducible standard of resistance. Work at NIST has already shown that the quantum Hall resistance may deviate from its ideal value of h/ie^2 above certain temperatures and above certain currents.

Recent research at NIST has opened up the possibility that quantum Hall devices may be a source of very-high-frequency phonons when large current densities are passed through the devices. Using GaAs heterostructures grown at NIST, researchers are using a dedicated class 10 clean room facility to investigate different methods of making contacts to the devices that will have very low resistances (in the milli-ohm range) even at temperatures below 4.2 K and in high magnetic fields.

Research is also being conducted on determining the degree to which the device resistance is independent of its material by comparing the resistances of devices made from silicon and GaAs/AlGaAs heterostructures.

NIST researchers are also working to improve and simplify the measurement systems used to calibrate resistors; a new He-3 refrigerator and 16-T magnet facility are being developed for use with a new cryogenic current comparator. This should enable the accuracy of NIST calibrations to be increased severalfold.

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APPLYING HIGH-T_c SUPERCONDUCTORS TO PRECISION ELECTRICAL MEASUREMENTS

NIST and other national standards laboratories for years have used cryogenic current comparators (CCCs) to make ratio measurements of voltage, current, and resistance with accuracies of 0.01 ppm or better. This approach has not found widespread commercial use because the cryogenic current comparator must be operated at liquid-helium temperatures, which presents a variety of operational difficulties. Moreover, existing comparators are working prototypes rather than completed instrumentation systems and, accordingly, are difficult to use.

Recent advances in superconductivity technology have made the application of high-temperature superconductors practical. In at least one case, an integrated circuit SQUID magnetometer, similar to devices used in CCCs to detect low levels of magnetic flux, has been built that operates at liquid-nitrogen temperatures. Thus, the possibility for developing a commercial instrument based on a CCC has now been opened. Such an instrument might be run with a refrigerator at liquid-nitrogen temperatures.

This development and the possibility of commercialization make it feasible to automate the basic CCC design, which then will be more attractive for general calibration use. NIST scientists are now engaged in designing, building, and testing CCCs to support measurements of the new national resistance standards based on the quantized Hall effect. This involves establishing a few select ratios that range from 1:1 to 100:1. Future efforts will include the development of CCCs with selectable ratios over a somewhat larger range.

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SEMICONDUCTOR ELECTRONICS

SILICON CHARACTERIZATION

NIST conducts research in semiconductor materials, processes, devices, and integrated circuits to provide the necessary basis for understanding measurement-related requirements in semiconductor technology. As part of this program, NIST scientists are using electrical, optical, and X-ray methods to study the resistivity, dopant distribution, and concentration of electrically inactive impurities, such as carbon and oxygen, in silicon. They are developing new or improved techniques by two- and three-dimensional mapping of these properties, refining the quantitative aspects of existing methods, and developing nondestructive methods. Measurement techniques include four-probe, spreading resistance, and capacitance-voltage; Fourier transform infrared spectroscopy; deep-level transient spectroscopy; X-ray topography; and synchrotron radiation studies.

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COMPOUND SEMICONDUCTORS AND SEMICONDUCTOR MICROSTRUCTURE

The electronics industry now requires light-emitting and detection devices and ultrahigh-speed structures that cannot be fabricated from silicon. Compound semiconductors including III-V binaries and alloys, such as GaAs and AlGaAs, and II-VI materials, such as CdTe and HgCdTe, are employed to complement the functions of the Si circuitry. In addition, special and unique electronic and structural properties can be obtained using artificially structured materials, such as quantum wells and superlattices. Efficient exploitation of these novel materials and structures in the production of useful electronic devices requires detailed studies to understand the fundamental physics involved as well as characterization of possible device structures.

Scientists and engineers at NIST currently are involved in producing and characterizing III-V binary and alloy materials and device structures using optical techniques, such as ellipsometry; electrical procedures, such as variable temperature Hall effect; resistivity measurements; capacitance-voltage profiling; deep-level transient spectroscopy; photoconductivity and X-ray diffraction; and rocking curve studies with conventional and synchrotron radiation sources.

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MOLECULAR BEAM EPITAXY

The controlled growth capabilities of molecular beam epitaxy (MBE) have resulted in the fabrication of structures that represent a new class of semiconductors with properties that do not exist in bulk materials. The MBE program at NIST includes the growth and characterization of GaAs and AlGaAs layers, as well as the growth of heterostructures for superlattice and quantum confinement studies. Scientists examine fundamental properties of the MBE layers using photoluminescence, deep-level transient spectroscopy, Hall effect, secondary-ion mass spectroscopy, and in-situ reflection high-energy electron diffraction (RHEED). Studies are under way to correlate RHEED oscillation intensity measurements with material quality and growth parameters. The MBE program is an interactive effort, and cooperative research opportunities exist in a variety of materials characterization and device-related areas.

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SEMICONDUCTOR SPECTROSCOPY

Spectroscopic studies in the visible and near visible regions of the electromagnetic spectrum are invaluable in investigating both materials and device-related properties. The ability to couple to electronic states of interest in device applications as well as the non-destructive nature of the spectroscopic analyses makes them attractive research and analytical tools. Studies currently under way at NIST focus on the electronic and structural behavior of semiconductor materials, such as Si, GaAs, and HgCdTe; microstructures, including quantum well and superlattices; and photonic and electronic devices. Excellent spectroscopic facilities are available to perform high-resolution photoluminescence, Raman scattering, reflection, absorption, spectroscopic ellipsometry and modulation spectroscopic measurements, such as electroreflectance and photoreflectance. The equipment in use includes one of the highest resolution ellipsometers in the world, excitation lasers, spectrometers, cryostats, and associated optical and electronic instruments.

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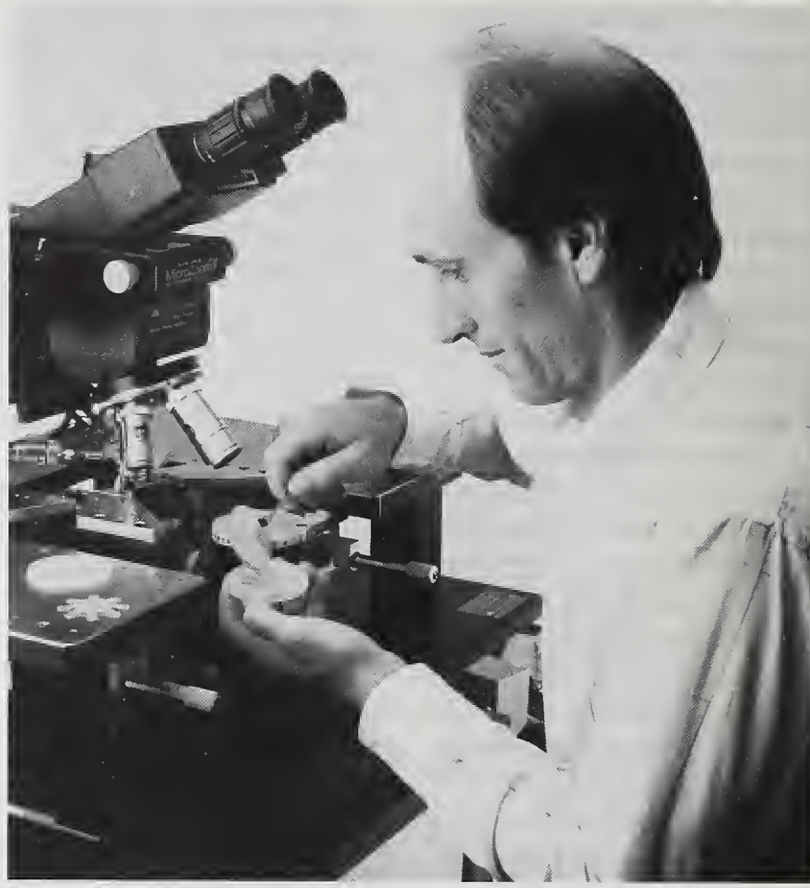

Electrical engineer Dylan Williams examines a microwave chip at a NIST wafer-probe workstation.

SEMICONDUCTOR DEVICES

To develop physically sound techniques for characterizing, analyzing, and predicting the operation and performance of semiconductor devices, NIST researchers are designing and improving measurement methods to determine critical device parameters for both VLSI-scale and power devices.

Research in device modeling includes two-dimensional silicon MOSFET and GaAs MESFET model development and investigations into the validity of the physical assumptions typically employed in silicon bipolar and GaAs device models. Theoretical research is carried out on the transport of ions and electrons in semiconductors for improved process modeling, and experimental research on the nature and characterization of electronic states in oxides and at oxide/semiconductor interfaces is under way. NIST scientists are developing methods for physical and electrical measurements of device and material parameters that are critical for verifying the accuracy and validity of device models. In addition, they are researching the electrical and thermal properties of power semiconductor devices.

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ADVANCED INTEGRATED-CIRCUIT TEST STRUCTURE METROLOGY

Integrated-circuit (IC) test structures and test methods developed by NIST are used widely by the semiconductor industry and other government agencies. These devices can be used to characterize integrated-circuit manufacturing processes, to evaluate the effectiveness of semiconductor processing equipment, to obtain crucial parameters for process or circuit simulators, to perform product acceptance tests, and to determine the reliability of the products manufactured. NIST work involves test structure design, modeling, data acquisition, and data analysis. Institute engineers are investigating pattern recognition techniques for the rapid diagnosis of IC manufacturing processes and for establishing methods to determine the reliability of thin films used in state-of-the-art microcircuits.

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ELECTROMAGNETIC FIELDS

ADVANCED MICROWAVE/ MILLIMETER-WAVE METROLOGY

Rapidly developing and expanding microwave technology requires research in advanced microwave measurements and standards. The microwave industry and the Department of Defense depend on NIST for calibrations of transfer standards to provide the measurement traceability required for quality assurance and performance evaluations. NIST researchers have developed highly accurate six-port techniques for automated measurements of microwave power, attenuation, impedance, scattering parameters, and noise. They currently are developing greatly improved power and impedance standards and extending measurement services to cover millimeter waves and subminiature coaxial connectors.

Collaborative work is particularly desirable in microwave and millimeter-wave circuit theory as applied to both tradiional and advanced circuits and systems, such as MMIC (monolithic microwave/millimeter-wave integrated circuit) devices. Applications include development of advanced wideband automated network analyzers using six ports and coplanar and microstrip-on-wafer MMIC standards.

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DIELECTRIC PROPERTIES OF MATERIALS

Inadequate knowledge of the electromagnetic properties of materials inhibits development of new technologies, drives up the cost of systems and components, and may prevent achievement of optimal performance levels. A relatively new NIST program in materials is aimed at developing primary standards and accurate techniques for measuring the dielectric properties of materials used in electromagnetic applications. Scientists at NIST have developed precision measurement techniques for complex permittivity based on improved theory and new cavity and transmission line sample holders. The researchers currently are performing the necessary error analyses and, in the future, plan to cover a variety of important materials and temperature ranges over the frequency range of approximately 10 MHz to 100 GHz. Reliable nondestructive methods are needed to measure complex permittivity and the reflectivity of large sheets of material or structures.

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ELECTROMAGNETIC INTERFERENCE AND COMPATIBILITY

NIST researchers are engaged in a wide range of projects aimed at quantifying electromagnetic interference (EMI) and electromagnetic compatibility (EMC). One thrust of the NIST work is to develop measurement techniques and methodologies for measuring emission of unintentional radiation from electronic devices. Another aspect under active investigation is the susceptibility of electronic equipment to such radiation. The researchers are identifying and defining quantities that characterize the susceptibility of a device and then developing methods to measure those quantities. Successful completion of this research should result in the development of standards and measurement techniques for EMI and EMC that are meaningful, technically practical, and reliable. These techniques could then be incorporated into voluntary standards by both U.S. and international standards organizations.

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ANTENNA MEASUREMENTS

Researchers at NIST are developing reliable techniques and standards for measuring key performance parameters of antennas and components used with satellites, Earth terminals, radars, and communications systems. Near-field scanning is now used routinely to characterize microwave and millimeter-wave antennas. NIST researchers are now focusing on developing and implementing techniques to correct for errors in the scan surfaces and applying all near-field techniques to higher frequencies. Software for the analysis of spherical near-field data recently has been rewritten and improved. Other research areas include spacecraft and phased-array antenna measurements, antenna diagnostics using near-field techniques, and antenna systems measurements using celestial radio sources.

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ELECTROMAGNETIC TECHNOLOGY

MAGNETICS

NIST researchers characterize magnetic materials, such as ferromagnetic and magnetoresistive films, recording tapes and disks, ferromagnetic steels, very weakly magnetic alloys, amorphous ribbons, spin glasses, ferrites, and permanent magnets, as a function of magnetic field and temperature. Traditional and improved magnetometer techniques are used, including vibrating-sample, SQUID, and Hall-probe. Alternating-field techniques (ac susceptometry, B-H loops, rf permeability) and magnetic-force microscopy also are used. Attention is given to calibration accuracy, measurement precision, and instrument development. The research has applications in basic physics and in applied engineering. Theoretical work is done in particulate recording media and mixtures and demagnetizing factors. Eddy-current probes for non-destructive evaluation are designed and used to map flaws in metals.

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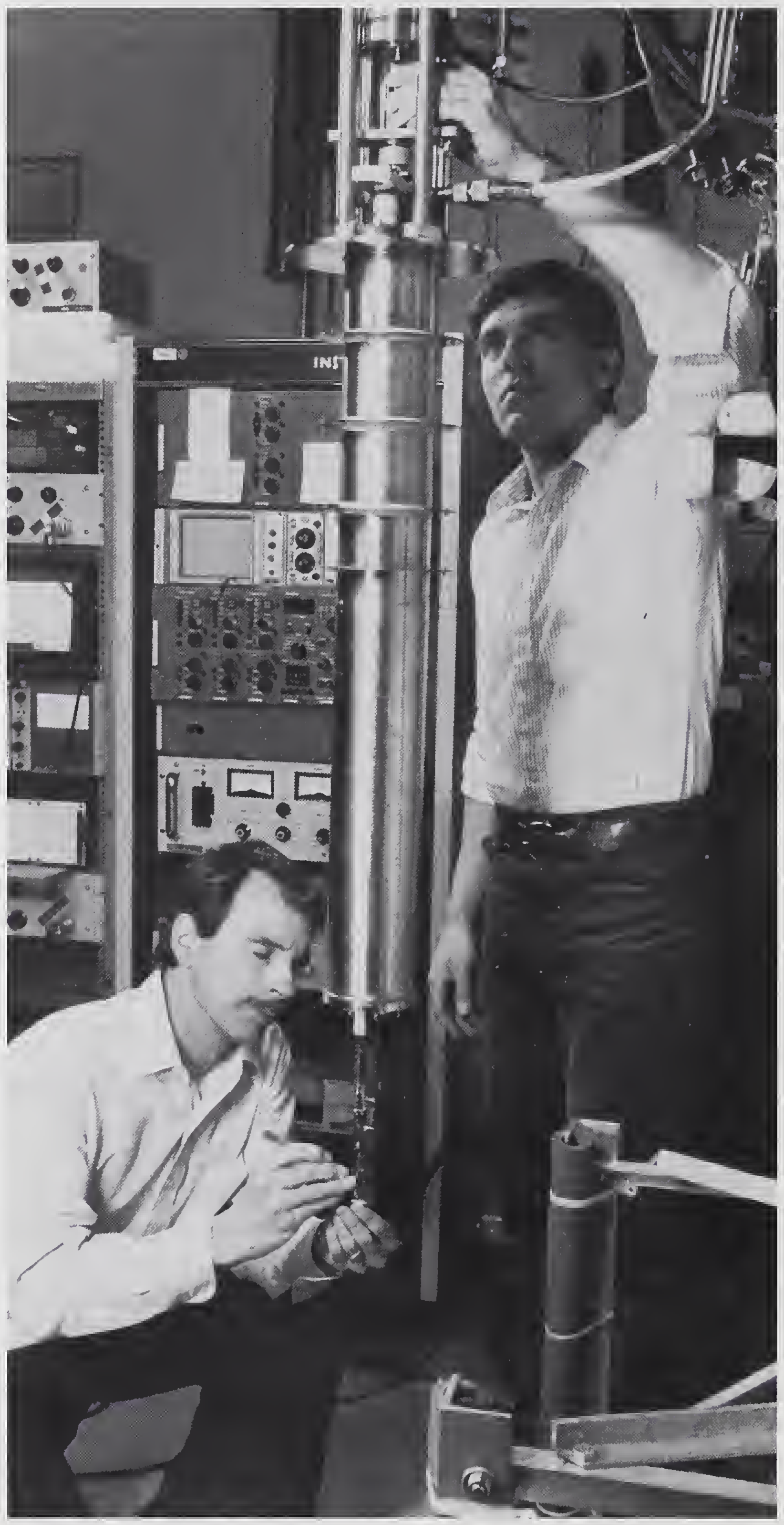
Physicist John W. Ekin (left) and electronics engineer Steven L. Bray measure the effect of transverse stress on the electrical properties of a superconducting material sample.



SUPERCONDUCTOR MEASUREMENTS

Recent advances in superconductivity have resulted in a critical need for measurement technology to characterize the different types of superconductors, which now range from very fine filament alloy conductors used in the Superconducting Super Collider cables to a variety of high-temperature ceramic superconductors. Active research programs at NIST involve measurement techniques for critical current, critical magnetic field, ac losses, magnetic hysteresis, and electron tunneling. In addition, specialized experimental work is being done to determine the effect of strain on the superconducting properties of low-temperature commercial conductors and the high-temperature materials.

In a recent collaboration with industry, the resistance of contacts between normal metals and high-temperature superconductors was decreased by eight orders of magnitude, allowing accurate measurements of the magnetic-field dependence of the critical current. A standard for measuring critical current in low-temperature superconductors has been published through ASTM, and a Standard Reference Material has been produced for use in calibrating critical current measurement apparatus. A new reference material for large currents is being developed.

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Engineer Paul Rice positions the piezoelectric scanner of a scanning tunneling microscope designed to study magnetic features in materials with submicron resolution.

OPTICAL ELECTRONICS

NIST researchers are studying optical fiber measurements, optical communication device metrology, laser measurements, and optical fiber sensors. These activities range from the growth of optical and electronic materials, through the fabrication of integrated optic devices, to the development and evaluation of new measurement systems. Materials are grown by chemical beam epitaxy, chemical vapor deposition, and sputtering. Devices are created with optical and electron-beam lithography. The measurement systems may incorporate new, in-house microstructures or an assembly of commercially available components. This research results in publication of new technology and measurement procedures and calibration services to support the laser and optical communications industries.

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LOW-TEMPERATURE ELECTRONICS

Cryogenic, and especially superconducting, electronics provide remarkably high speed and sensitivity, coupled with exceptionally low-power dissipation. NIST has a complete facility for fabricating superconducting integrated circuits from conventional low-temperature superconductors and is developing a similar capability for high-temperature ceramic superconductors.

NIST research spans a range from very basic studies of ultrasmall tunnel junctions to a superconducting series array voltage standard in operation at more than two dozen laboratories around the world. The work has established numerous world performance records over the years with such devices as analog-to-digital converters, samplers, electrometers, microwave and infrared detectors, lithographed antennas, and magnetic flux detectors using SQUIDs. NIST efforts support private industry through cooperative research and assistance with measurement techniques.

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RESEARCH FACILITIES

SEMICONDUCTOR PROCESSING RESEARCH LABORATORY

As integrated circuit sizes increase to more than 1 cm^2 and feature sizes within the circuits decrease to less than 1 μm, critical demands are placed on the measurement capability required to control and monitor their fabrication successfully. To meet the demand, NIST researchers are developing state-of-the-art measurement procedures for microelectronics manufacturing.

The semiconductor processing research laboratory provides a quality physical environment for a broad variety of research in semiconductor microelectronics as well as in other areas of physics, chemistry, and materials research. The laboratory facilities are used for projects addressing many areas of semiconductor materials and processes, including process control and metrology, materials characterization, and the use of integrated circuit materials and processes for novel applications.

The laboratory complex occupies about 372 square meters, approximately half of which is composed of clean rooms. Within the clean rooms, work areas are maintained at class 10 or better. The facility is designed so that the work areas can be easily modified to accommodate the frequent equipment and other changes required by research.

CAPABILITIES: The laboratory has a complete capability for integrated circuit fabrication. Principal processing and analytical equipment are listed below. The capabilities are expanded and improved continuously to meet the technological challenges.

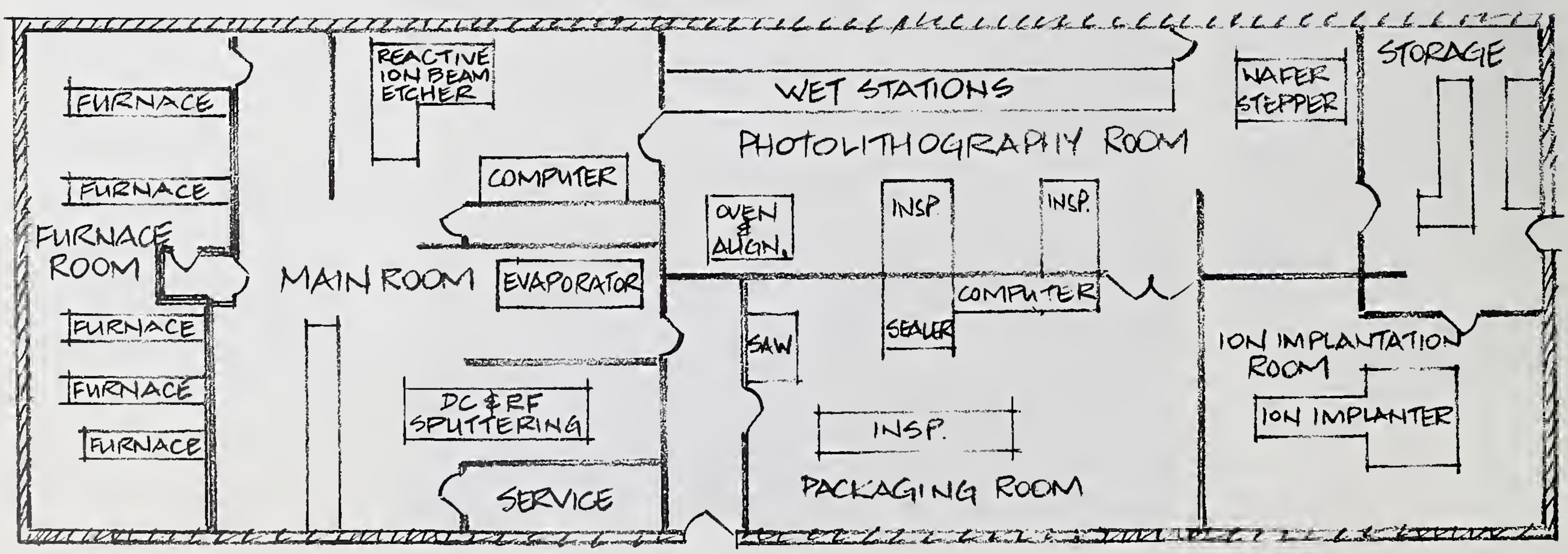
- Diffusion, Oxidation, and Annealing. Six furnace tubes for up to 75-mm-diameter wafers and nine tubes for up to 100-mm-diameter wafers.
- Photolithography. Research mask aligner (proximity and contact) for wafers up to 100 mm in diameter and irregularly shaped samples and 10× direct-step-on wafer system for 75-mm-diameter wafers. Photoresist spin coating and developing and related chemical processing, including oxygen plasma stripping.
- Film Deposition. Low-pressure chemical vapor deposition systems for depositing silicon nitride, polysilicon, and low-temperature silicon dioxide. Rf and dc vacuum sputtering of metals and dielectrics. Electron beam and hot filament vacuum evaporation of metals.
- Etching. Wet and dry etching processes. Reactive ion beam etcher capable of ion milling and chemical etching with gases such as freon, sulfur hexafluoride, oxygen, and chlorine.
- Ion Implantation. Multipurpose 200-KeV ion implanter.
- Analytical Measurements. Thin-film reflectometry and other thickness measurements, optical microscopy, and grooving and staining. Automated and manual probe stations for current-voltage measurements and capacitance measurements as a function of voltage, frequency, and time.

APPLICATIONS: Small quantities of specialized semiconductor test specimens, experimental samples, prototype devices, and processed materials can be produced. The processes and processing equipment can be monitored during operation to study the process chemistry and physics. The effects of variations in operating conditions and process gases and chemical purities can be investigated. Research is performed under well-controlled conditions.

Because the work is of a research nature, the laboratory is not designed to produce large-scale integrated circuits or similar complex structures. The breadth and flexibility of the laboratory is primarily to support the varied types of projects performed.

Currently, active research projects address many aspects of microelectronic processing steps and materials as well as silicon micro-machining. Examples include: metal-oxide-semiconductor measurements; metal-semiconductor specific contact resistivity; uniformity of resistivity, ion implanted dopant density, surface potential, and interface state density; characterization of deposited insulating films on silicon carbide; ionization and activation of ion-implanted species in semiconductors as a function of

Below. Schematic of the Semiconductor Processing Research Laboratory. Right. At the NIST High-Voltage Measurement Facility, researchers study how electrical equipment and insulation materials perform if subjected to power surges or struck by lightning.



annealing temperature; electrical techniques for dopant profiling and leakage current measurements; and processing effects on silicon-on-insulator materials. A simple CMOS process has been established.

AVAILABILITY: Laboratory staff welcome collaborative research projects consistent with the research goals of the NIST semiconductor program. Work is performed in cooperation with the technical staff of the laboratory.

The most productive arrangement begins with the development of a research plan with specific goals. The commitment of knowledgeable researchers to work closely with NIST staff and the provision of equipment and other needed resources are required. Because hazardous materials are present, laboratory staff must supervise all research activities.

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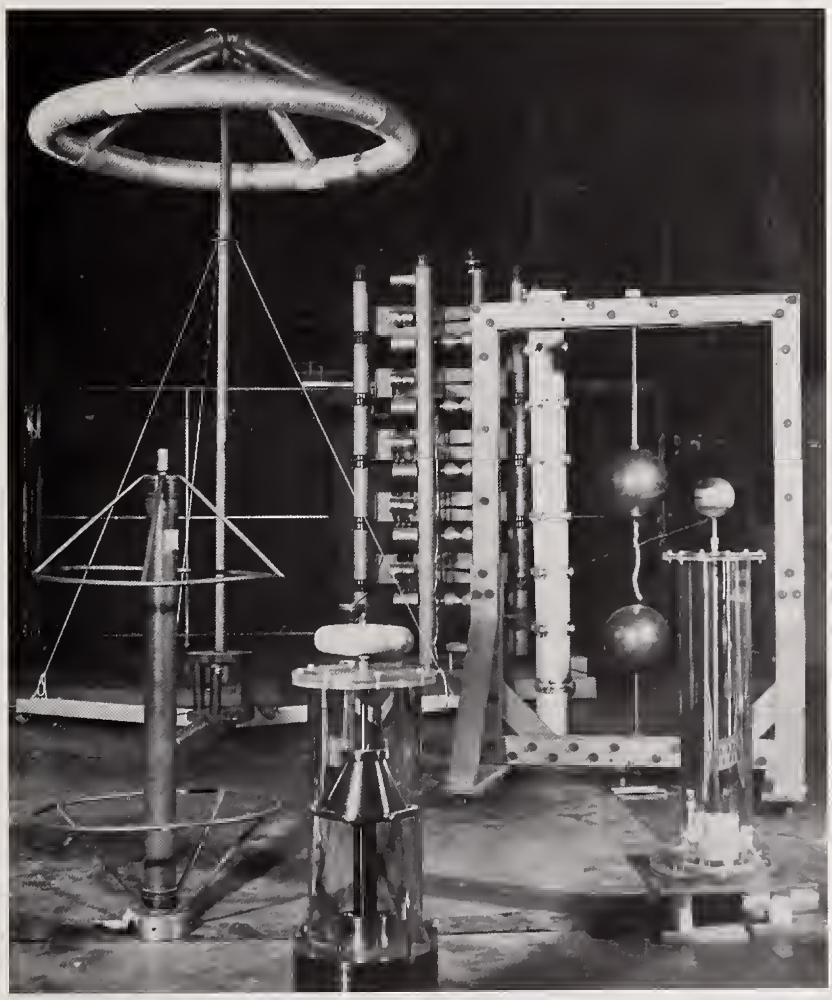

HIGH-VOLTAGE MEASUREMENT FACILITY

NIST maintains a high-voltage measurement facility in which researchers develop and evaluate the measurement techniques needed for the efficient, reliable transmission and distribution of electric power (including for defense purposes). Major programs now being pursued using this facility are the measurement of transient voltages and currents; the development of techniques to quantify pre-breakdown and breakdown phenomena in liquid and gaseous dielectrics; and the measurement of low-frequency electric and magnetic fields.

CAPABILITIES: With existing power sources, direct voltages of 300 kV, 60-Hz alternating voltages of 175 kV, and standard lightning impulses of 500 kV can be produced. Selected waveforms, such as microsecond duration trapezoidal waveforms up to 300 kV and gated 60-Hz waveforms up to 100 kV, are also available. Supporting equipment includes high-voltage standard capacitors rated at 200 kV; high-accuracy, current-comparator bridges for 60-Hz measurements; a precision dc divider rated at 200 kV; dividers to measure standard lightning impulses up to 1-MV peak; partial discharge measurement systems; high-speed cameras and supporting optical equipment (including an image-preserving optical delay); a computer-controlled system to measure the electric field in transformer oil; a gas-chromatograph/mass-spectrometer system; and a system to produce a known electric field and current density in air at atmospheric pressure.

Measurement systems are available to measure pulsed voltages and currents with characteristic times ranging from nanoseconds to milliseconds. These systems include Rogowski coils and capacitive probes as well as the necessary recording equipment to acquire and store digital records. These conventional measurement systems are supplemented by optical sensors and couplers.

APPLICATIONS:

- Instrumentation and Component Evaluation. Impulse, ac, and dc dividers; electric and magnetic field meters; capacitors; transformers; lightning arresters; and ion counters.
- Dielectrics Research and Development. Chemical degradation studies; measurement of the fundamental processes of discharge initiation; onset and magnitude of partial discharges; space charge measurement; and streamer propagation studies.

AVAILABILITY: The high-voltage facility is used by NIST staff and by guest researchers from industry, universities, and other federal agencies. Use of the facilities must be scheduled in advance. Because of the complexity of the system, typical use of the facility is in the form of a collaborative investigation with NIST staff.

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NEAR-FIELD SCANNING FACILITY FOR ANTENNA MEASUREMENTS

The automated facility is designed to measure the near-zone phase and amplitude distributions of the fields radiated from an antenna under test. Mathematical transformations are used to calculate the desired antenna characteristics.

CAPABILITIES: Near-field data can be obtained over planar, cylindrical, and spherical surfaces; the planar technique is the most popular. Efficient computer programs are available for processing the large quantities of data required.

When operated in the planar mode, the facility is capable of measuring over a 4.5-m × 4.5-m area with probe position errors of less than ±0.01 cm. Improved position accuracy is possible with further alignment, especially over smaller areas. Antennas with apertures up to about 3 m in diameter can be measured with a single scan. The facility has been used successfully over the frequency range 750 MHz to 75 GHz. It incorporates provisions for scanning larger antennas in segments.

APPLICATIONS:

■ Antenna Characteristics. The facility is used primarily for determining the gain, pattern, and polarization of antennas. Accuracies are typically ±0.15 dB for absolute gain and ±0.10 dB/dB for polarization axial ratio. Patterns can be obtained down to the −50 dB to −60 dB levels with side lobe accuracy typically about ±1.0 dB at the 40-dB level. (The exact uncertainties depend on the frequency, type, size of antenna, and other factors.) Near-field data also can be used to compute near-field interactions (such as mutual coupling) of antennas and radiated field distributions in the near zone.

■ Antenna Diagnostics. Near-field scanning is also a valuable tool for identifying problems and for achieving optimal performance of various types of antenna systems. It has been used to advantage in locating faulty elements in phased array antennas and for adjusting feed systems to obtain the proper illumination function at the main reflector. Phase contour plots of the near-field data also can be used to determine surface imperfections in reflectors used for antennas or compact ranges.

■ Probe Calibrations. A spherical probe calibration facility is used as a far-field range for measuring the receiving characteristics of probes used to obtain near-field data. These measurements are required to determine the probe coefficients, which, in turn, are used to calculate accurate, probe-corrected, far-field gain and pattern characteristics of an antenna.

AVAILABILITY: Two kinds of arrangements can be made to use this facility. NIST staff can perform specified tests or measurements on a reimbursable basis. In this case, the customer has no direct use of the facility; all measurements are performed by NIST staff and the customer is issued a test report. As an alternative, work may be performed on a cooperative basis with NIST staff. This arrangement permits the user the advantage of developing firsthand knowledge of the measurement processes, and the user is responsible in large part for the accuracy of test results. In either case, arrangements need to be made well in advance, and reimbursement is required for the facility use and time of NIST staff involved.

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GROUND SCREEN ANTENNA RANGE

The ground screen antenna range is an open area test site.

CAPABILITIES: The ground screen consists of 6.35-mm mesh galvanized hardware cloth stretched over a level concrete slab. The screen is 30.5 m wide × 61 m long and is spring-loaded around the perimeter to ensure uniform tension, a flat surface, and adequate compensation for thermal expansion. The overall size of the ground screen permits far-field measurements in the high-frequency portion of the spectrum, and the mesh dimension provides for an efficient ground plane well into the ultrahigh frequency region.

APPLICATIONS: The range can be used for the following:

■ antenna calibrations;
■ antenna patterns at any polarization;
■ electromagnetic susceptibility measurements;
■ electromagnetic radiated emission measurements;
■ calibration of field intensity meters; and
■ wave propagation studies in frequency or time domains.

AVAILABILITY: This facility is used heavily in performing calibrations for industry and other government agencies. The facilities are available for independent or collaborative work.

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MODE-STIRRED CHAMBERS

NIST researchers have designed and constructed mode-stirred (reverberating) chambers to measure radiated electromagnetic emissions and susceptibility of electronic equipment and shielding effectiveness of materials and cable/connector assemblies. A mode-stirred chamber is an electrically large (in terms of wavelength), high-quality cavity whose boundary conditions are varied by means of a rotating conductive tuner or stirrer. The time-averaged field inside such a cavity, when a sufficient number of modes are excited, is formed by uniformly distributed plane waves coming from all directions. This causes the polarization of the field to vary randomly, hence eliminating the need for, or the utility of, physical rotation of test objects in the field. A microwave oven is a simple example of a mode-stirred chamber without measurement support instrumentation.

CAPABILITIES: The mode-stirred chamber simulates near-field conditions for tests at frequencies from 200 MHz to 40 GHz. High-level test fields (up to 1000 V/m) can be generated efficiently over a large test volume in the chamber, or the chamber can be used to measure low-level radiated emissions (total radiated power down to −100 dBm) from equipment under test with minor instrumentation changes. Equipment as large as 1.5 m × 2.0 m × 3.0 m can be tested.

APPLICATIONS: In addition to performing radiated emission or susceptibility measurements of electronic equipment, the mode-stirred chambers can measure the shielding effectiveness of gasketing, composites, and other materials used for radio frequency shielding applications. The chambers also can be used to measure the shielding effectiveness of wiring harnesses and electrical cables, connectors, and assemblies.

LIMITATIONS: The usable lower frequency is limited by insufficient mode density, tuner effectiveness, and ability to uniformly excite all modes in the chamber. These factors are a function of both chamber geometry and size. Measurement uncertainties vary from ±10 dB at 200 MHz decreasing to ±4 dB from approximately 1.0 GHz to 18 GHz. Directional characteristics of an antenna or test equipment placed inside a mode-stirred chamber are lost, resulting in the need to estimate their free-space maximum gain as a function of frequency in order to correlate results obtained by open-field tests. However, tests can be performed cost effectively in a shielded environment, with sufficient accuracy to make these facilities very attractive for diagnostic testing and for minimizing the need for expensive testing in facilities such as anechoic chambers.

AVAILABILITY: Two mode-stirred chambers are available: one that is 2.74 m × 3.05 m × 4.57 m and a smaller version, 1.16 m × 1.425 m × 1.47 m. NIST staff are available for collaborative programs or to advise and interpret measurement results. Independent testing also can be arranged.

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TRANSVERSE ELECTROMAGNETIC CELLS

NIST researchers have designed and constructed several transverse electromagnetic (TEM) cells that are available for use. A TEM cell is a device for performing radiated electromagnetic emission and susceptibility measurements of electronic equipment. Its design is based on the concept of an expanded transmission line operated in a TEM mode. The cell is a two-conductor system with the region between the inner and outer conductors used as the test zone. The tapered sections at both ends are required to match the cell to standard 50 Ω coaxial-cable connectors.

CAPABILITIES: The cell provides a shielded environment for testing without introducing multiple reflections experienced with the conventional shielded enclosure. It simulates very closely a planar far field in free space and has constant amplitude and linear phase characteristics. The external electromagnetic signals will not affect the measurement of low-level radiated emission from the device under test. The high-level test field generated inside a cell for radiated susceptibility tests will not interfere with external electronic systems.

APPLICATIONS: In addition to radiated electromagnetic compatibility/interference testing, other applications of the TEM cells include the calibration of antennas and the study of biological effects of radio-frequency radiation.

LIMITATIONS: The usable frequency range is limited by an upper bound determined by the appearance of the lowest high-order mode. The volume available for testing purposes is inversely proportional to this upper frequency limit. The size of the device to be placed inside a TEM cell for testing should be small relative to the available test volume in order that the field structure associated with the ideal TEM mode existing in an empty cell not be significantly perturbed.

AVAILABILITY: Several TEM cells with five different sizes and five upper frequency limits in the range 100 MHz to 1 GHz are available. In collaborative programs, NIST staff are available to advise and interpret measurement results. Independent testing also can be arranged.

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ELECTROMAGNETIC ANECHOIC CHAMBER

The electromagnetic (EM) anechoic chamber at NIST is a facility for generating standard (known) electromagnetic fields. Such fields are fundamental to the research, development, and evaluation of antennas, field probes, and EM material properties.

CAPABILITIES: EM fields up to 100 V/m can be established in the chamber over the broad frequency range from 200 MHz to 18 GHz, and up to 200 V/m for certain frequency bands above 1 GHz. A majority of the individual systems composing the measurement system are under computer control, thus enhancing statistical control of the measurements. Work is under way to extend the frequency range to 40 GHz and to improve the computer control of the chamber systems. The chamber dimensions are 8.5 m × 6.7 m × 4.9 m.

APPLICATIONS: The EM chamber is used in areas such as:

- research, development, and evaluation of new EM-field-generation and measurement methods;
- antenna and field-probe development and evaluation;
- calibration of field measurement instruments;
- susceptibility testing of electronic equipment;
- shielding effectiveness and material parameter studies; and
- special tests for government agencies, industry, and universities.

AVAILABILITY: This facility is used heavily in performing calibrations for industry and other government agencies. The facilities are available for independent or collaborative work with NIST.

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MANUFACTURING ENGINEERING LABORATORY

NIST engineers and scientists are developing many of the essential elements of automated intelligent processing systems that will soon be at the core of all world-class manufacturing operations. These components include robotics; advanced sensors for real-time in-process measurements; software for precision control of machine tools; and information technology for integrating all elements of a product's life cycle, from planning and design through marketing and customer support. Building on its in-house research and development, the laboratory provides technical support for industry groups that are developing standards for measurements, measurement techniques, hardware, software, and data interfaces.

Two projects illustrate the broad scope of the laboratory's research activities. In one major initiative, staff members are refining the laboratory-designed and -built molecular measuring machine for precision engineering. Intended to support the fabrication and characterization of future generations of integrated circuits and other "nanotechnologies," the instrument will be able to measure atomic and molecular structures over an area of 25 square centimeters.

In NIST's "Shop of the 90s," technical experts from the Institute are helping small metal fabrication plants modernize their operations. The majority of the nation's 125,000 job shops, which turn out 75 percent of the fabricated metal parts used in construction and manufacturing, use decades-old technology. To help these companies become more efficient and competitive without incurring prohibitively high expenses, the "Shop of the 90s" uses off-the-shelf computer technology to help integrate and automate manufacturing and other business operations.

Technology transfer is part and parcel of the Manufacturing Engineering Laboratory's activities. In their laboratories, at the Automated Manufacturing Research Facility, and on the floors of U.S. manufacturing plants, the Manufacturing Engineering Laboratory staff work closely with their counterparts from industry.

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COOPERATIVE RESEARCH OPPORTUNITIES

PRECISION ENGINEERING

ATOMIC-SCALE MEASURING MACHINE

By the year 2001, uncertainty requirements for dimensional metrology of step heights, surface roughness, linewidth, and line spacing for the integrated circuit and optics industries will be 0.1 nm to 1 nm. Furthermore, these uncertainties must be held over areas ranging from several square millimeters to fractions of a square meter. To address these needs, NIST is building the molecular measuring machine (M^3). M^3 will be capable of positioning and measuring to atomic-scale accuracies over an area of 25 cm^2. It incorporates a scanning tunneling microscope into a unique system design that includes a very stiff core structure, carriages for moving the probe over the sample, interferometry for measuring probe and sample position, and two stages of isolation from seismic and acoustic perturbations. Construction and testing are under way.

After construction is completed, M^3 will serve as an independent means for characterizing distances, geometries, and distortions of highly ordered arrangements of atoms on single-crystal surfaces. M^3 also will serve as an exploratory tool for building mechanical and electrical structures in the nanometer-size range. Among the organizations collaborating on the construction of M^3 are several major universities and national laboratories, as well as Watson Research Center, AT&T Bell Laboratories, and Zygo Corp.

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Within the spherical copper frame of the molecular measuring machine, NIST scientists plan to use a scanning tunneling microscope and lasers to measure the positions of individual atoms over distances hundreds of millions of atoms wide.

ADVANCED OPTICAL SYSTEMS

Advanced optical systems increasingly are designed around high-accuracy, aspheric optical elements. Measuring the figure error of generalized aspheres to the required accuracy is a complex and unsolved problem. NIST is embarking on a program to characterize the systematic errors of commercial, phase-measuring interferometers used for surface figure metrology and to develop techniques to use these instruments for aspheric metrology. The goal of the program is to close the gap between the resolution and the accuracy of phase-measuring interferometers. NIST researchers are working with personnel at

Wyko Corp. and Zygo Corp., which manufacture these interferometry systems.

NIST researchers already have demonstrated a significant sensitivity to fringe density of the measured figure error for several commercial phase-measuring interferometer systems. They are working to build a dedicated test facility for future systematic investigations. The results of this work will make it possible to manufacture lightweight, high-performance optical systems for space-based applications and multilayer mirror systems for projection X-ray lithography.

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MEASURING PATTERNED LAYERS ON INTEGRATED CIRCUITS

NIST researchers are developing techniques for measuring the critical dimensions of patterned layers on integrated circuits. The work involves theoretical projects on the formation of images in the optical and scanning electron microscopes, as well as experimental and design projects on the construction of new metrology instruments for the calibration of standards. The project was initiated about 15 years ago at the request of the semiconductor industry.

The ever-smaller dimensions on integrated circuits have created a demand for new and improved techniques of measurement and their related standards, especially as feature sizes approach and become smaller than the wavelength of light used in conventional optical metrology instruments. The dimensions of present interest range from about 0.5 μm to 30 μm and will extend to smaller dimensions in the future. A series of three linewidth/pitch Standard Reference Materials for photomasks are presently in production. Research is in progress to develop new and improved standards for use in instruments utilizing optical or scanning electron microscopy.

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VISION-BASED MEASURING SYSTEMS

NIST has extended its study of coordinate measuring machines to equipment with computer vision sensors. The use of video sensors is a much more complicated process than the traditional touch-trigger probes. Much work remains to be done to develop suitable standards for characterizing the performance of these types of measurement systems. Even procurement is a problem if there is no standard way to specify, characterize, and test the accuracy of the equipment. The goal is to produce a knowledge base that can be used to develop standardized methods for both testing and using vision-based measuring machines.

NIST researchers currently are using a Precis 3000 vision-based measuring machine on loan from View Engineering of Simi Valley, Calif. It is being used to test different methods for characterizing the precision and accuracy of vision-based measuring systems. NIST researchers are introducing the concept of error enhancement into video-based coordinate measuring systems. NIST pioneered this technique, and it has been used with touch-trigger and analog probes for several years.

The NIST researchers are active members of standards committees for dimensional measurement and will use the results of these studies to assist the committees in developing appropriate standards and guidelines.

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MEASURING SURFACE ROUGHNESS

The need for effective, on-line control of surface texture is increasing. In fact, many U.S. automobile companies are beginning to see superior coatings as a potential competitive advantage over foreign competition. One of the keys to achieving this advantage is better on-line surface measurement methods. NIST is using contacting and optical profiling techniques as well as optical scattering to develop such methods (which will be sensitive to the functionality of the components). The researchers already have used an experimental light-scattering instrument and a long-range scanning tunneling microscope to generate accurate descriptions of light scattering from rough metal parts and from glossy paper. They currently are studying the effects of using a light-scattering system to inspect glossy paper during manufacturing. The researchers plan to adapt this system for other materials and manufacturing processes. They also intend to build a remotely controlled profiling instrument.

These advances will provide a better understanding of the relationship between process parameters and surface finish. NIST researchers currently are working closely with academia, the automotive and painting industries, and standards committees.

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CAD-DIRECTED INSPECTION

NIST researchers are implementing a CAD-directed inspection system for manufactured parts that utilizes the ANSI/CAM-I Dimensional Measuring Interface Standard (DMIS) and the Initial Graphics Exchange Specification. The goal is to produce a closed-loop, integrated inspection system completely from commercially available products. Six companies have loaned products to NIST to be used in this project. They are Automation Software, CADKEY Inc., CMX Systems Inc., ICAMP Inc., Renishaw Inc., and Sheffield Measurement.

NIST researchers already have built the first prototype of such a system for simple parts on a single personal computer platform. Currently, the inspection plan is input manually using a mouse. Research continues in several areas: expanding probing capabilities, adding more complex part inspections, expanding and refining DMIS, and automating the actual inspection planning. The research results of this project are being used to assist the committees—ANSI/CAM-I DMIS, ANSI/ASME B89.3, and ANSI/ASME B89.4—in the development of the appropriate standards and guidelines. NIST researchers also are working with the University of North Carolina-Charlotte to make use of process information in developing the inspection plan.

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AUTOMATED PRODUCTION TECHNOLOGY

QUALITY IN AUTOMATION PROGRAM

This program is an effort to achieve higher part accuracy from existing discrete-parts manufacturing equipment. A four-layer, closed-loop, control architecture has been proposed. Three layers are being implemented: real-time (RT), process-intermittent (PI), and post-process (PP) control loops. The RT and PI loops implement algorithms to predict and/or measure machine- and process-related systematic errors and compensate for them via real-time tool path modification and NC program modification. The PP loop is used to verify the cutting process and to tune the other two control loops by detecting residual systematic errors measured on the finished parts, correlating these errors to the uncompensated machine- and process-related errors, and modifying the control parameters of the other loops accordingly.

Feature-based error analysis techniques are being developed to identify the residual systematic errors of the system. NIST researchers are working with industry and academia to use DMIS-defined features in their analyses. They are developing tools for feature segmentation of any computer-aided design for manufacture and analysis point of view. They are also developing a quality database using feature types and the errors measured on these features as key fields.

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Mechanical engineer Nicholas Dagalakis monitors the performance of the Advanced Deburring and Chamfering System, an industrial robot and cutting tool that produces beveled edges in metals, such as titanium, at high speeds.

MACHINE-TOOL PERFORMANCE EVALUATION AND ACCURACY ENHANCEMENT

Deterministic manufacturing is a concept based on the premise that most errors in manufacturing are repeatable, thus predictable. Errors that are predictable can be measured and corrected, thereby improving quality through better control of the existing manufacturing equipment. Machine tools are computer-controlled, multi-degree-of-freedom structures that have inherent quasistatic and thermally induced geometric errors. Complete characterization of these errors is being investigated to evaluate and enhance performance under changing thermal conditions. This process is complicated and time consuming.

NIST researchers are working with industry and academia to optimize this process to make it feasible for even small manufacturers. NIST, industry, and academia worked together to develop the industry standard for machining center performance evaluation (ASME B5.54). Compensation of machine-tool errors is an area of continuing interest. Researchers are developing generic electronic hardware to implement error compensation without any intrusion into the existing machine-tool controllers. They also are working to speed up the on-machine part inspection by introducing fast probing and powerful data analysis capability at the machine-tool level.

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PORTSMOUTH WORKSTATION

The Fastener Quality Act, Public Law 101-592, recently passed by Congress, requires the inspection and certification of industrial fasteners used in critical applications. The fabrication of high-tolerance fasteners, such as hex head bolts, screws, and studs, is critical to the maintenance of submarines. With tolerances as small as 12.7 μm, these fasteners often require frequent inspection at various stages of production. In addition, they are typically made in small lots from hard-to-cut materials such as k-monel. These three facts—tight tolerances, small lots, and exotic materials—make them difficult and costly to procure.

NIST is working with personnel from the Portsmouth Naval Shipyard and several private companies to build an advanced, computer-controlled, machining workstation capable of automatically manufacturing, inspecting, and engraving these fasteners. The hardware and software have been integrated, and the first certified parts have been produced. Two techniques pioneered at NIST, error compensation and in-process control, are now being incorporated. In addition, NIST researchers expect to develop new statistical process control techniques to be used in this and other small batch production environments. This will maximize the production rate and minimize scrap.

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ROBOT SYSTEMS

INTELLIGENT MACHINE CONTROLS

The concept of advanced real-time, sensory-based control of machines has been a goal of researchers and system developers for over a decade. However, the lack of a structured, theoretical approach for designing and developing such systems has severely limited the number and sophistication of applications seen in operation today. Based on years of research and development in real-time, sensory control of robots and manufacturing systems, NIST is conducting the research necessary to develop a standardized architecture for implementing intelligent machine control. This architecture has been adopted by such organizations as the National Aeronautics and Space Administration and the U.S. Air Force.

NIST's current research seeks to expand and document a formal mathematical theory for the control of intelligent machines and to verify these theories using testbeds involving both simulation and actual controller hardware. Advanced control concepts, involving planning, sensory processing, world modeling, and knowledge representation, are being investigated. Several independent development projects are under way to demonstrate implementations of these control principles on functioning machines. They involve automated manufacturing, robotic deburring, space robotics, construction, remotely operated land vehicles, underground mining, and undersea vehicles.

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SENSORY PROCESSING AND WORLD MODELING FOR INTELLIGENT CONTROL

Intelligent control of machines requires a detailed knowledge of both the machine and its operating environment. Since these are both dynamic in nature, it is necessary to measure, analyze, and comprehend changes in real time to achieve intelligent, sensory-based control of machines. NIST has a number of projects focused on developing advanced sensory processing and world modeling capabilities. Sensory processing research includes work on specialized vision systems, real-time image processing, and sensor data fusion. Vision systems being investigated include non-uniform resolution, active scanning of the fovea over points of interest, and active control of multiple cameras.

Under investigation are image-processing techniques for obtaining dense range maps from image flow and from stereo images. World modeling research is concentrating on data structures that can represent both spatial and symbolic information about surfaces, objects, and regions of space. Methods are being developed for comparing world model and sensory data so that one could, for example, generate a map overlay that is registered with and tracks camera images in real time. Also being developed are Kalman filtering techniques for updating the world model based upon sensory data.

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OFF-LINE PROGRAMMING OF ROBOTIC SYSTEMS

Off-line programming (OLP) systems are used to generate robot programs without the use of the actual robot. OLP has two advantages over the traditional teaching-dependant programming method: It improves safety because the robot is not run and robot/operator interactions are reduced, and the system downtime is reduced because the robot is not involved in developing and debugging programs. But most commercially available OLP systems rarely produce the final robot control programs.

Research at NIST has focused on the use of OLP to generate directly robot control programs to determine robot trajectories for part handling and deburring. Since the models of the robot and the workstation layout never exactly match the actual equipment, there can be significant errors between the OLP-generated robot positions and the actual points through which the robot will move. To compensate for this, NIST has developed several sensor-based approaches for automatically calibrating or registering the OLP model of the robot and workstation with the real systems. This permits automatic generation of reliable robot programs in a matter of seconds or minutes instead of the hours or days required for manual teaching.

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This computerized turning workstation operated by numerical control programmer Richard Beaulieu (left) of the Portsmouth Naval Shipyard and NIST mechanical engineer Mahn Hee Hahn can produce precision machined fasteners in less than one-tenth the time required with conventional equipment.

ROBOT METROLOGY

The characterization of the performance of a robotic system is important in understanding the limitations of the system and in optimizing its operation. Although several U.S. and international standards committees are working on measuring robot performance, no standard robot acceptance and characterization tests currently exist. Research is ongoing at NIST to develop and validate standard test procedures for measuring robot performance. NIST has been working closely with the Robot Industries Association subcommittee on developing such tests.

Currently, a laser tracking interferometer system, originally designed and developed by NIST and now available commercially, is used to make these measurements. This system provides very accurate position measurements of 1 to 2 parts in 100,000 and an angular accuracy of 1 to 2 seconds of arc. Also under development is a low-cost robot calibration system that consists of a series of string encoders mounted to a fixture. This system provides a relatively inexpensive robot calibration system with an accuracy of ±0.127 cm. Currently, research is being conducted to characterize the performance of this system and to expand its capabilities from being able to measure three degrees-of-freedom to a full six degrees-of-freedom.

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MOBILE ROBOTIC SYSTEMS

Current applications of robotic systems are typically limited to industrial operations such as part handling, assembly, welding, and painting. A number of applications are being investigated by the research community that place a different emphasis on the requirements for a mobile robotic system.

The U.S. Olympic speed-skiing team races at speeds of up to 190 km/h using a special computer-designed helmet produced through a cooperative project involving NIST researchers, Bell Helmets, and several other industrial partners.

NIST has been conducting a joint research program with the U.S. Army on the development of a mobile robotic platform for military applications. NIST is involved in specifying the basic control system architecture for the mobile vehicles. This control system handles mobility functions on the vehicle—both teleoperation and autonomous-subsystem integration—and communication with the remote operator station.

NIST has demonstrated remote operation of the vehicle over an rf radio link and "retro-traverse"—an autonomous operation where the vehicle retraces a previously traveled path based upon data from an onboard inertial navigation system (INS). Other research aspects include video data compression for remote driving, a low-cost INS, and obstacle-detection sensor systems. The goal is to have a standard architecture that could be adopted by the military for all of its robotics applications, thereby significantly reducing integration costs and allowing subcomponents to be transferred between different robotics systems.

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ROBOTIC DEBURRING OF MACHINED PARTS

Engineers at NIST are developing an automated system that produces precision chamfers on aircraft engine components fabricated from titanium and inconel. The chamfers, or beveled edges, must be placed on parts such as turbine engine blades and hubs to relieve potential areas of stress concentration and ease assembly. The system, called the advanced deburring and chamfering system (ADACS), consists of a six-axis electric robot fitted with an actuated chamfering tool. This tool is capable of high-speed force control within a small work area.

The approach is to use the industrial robot as a coarse positioning device, while relying on the actuators and force sensors of the tool to provide control of cutting force and stiffness at the part edges. The tool also compensates for robot inaccuracies and other factors, such as part misalignment and large tolerances. The system has maintained chamfers of 38 μm on titanium while traversing corners, in spite of part positioning errors on the order of several millimeters. The ADACS builds on two techniques pioneered in the Automated Manufacturing Research Facility: a technique to generate robot trajectories off-line from CAD data and a technique to integrate a variety of sensors into the robot-control system.

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COMPOSITE-PART MANUFACTURING USING ROBOTIC FIBER PLACEMENT

The manufacture of high-performance composite parts remains very time and labor intensive. NIST has initiated the development of an advanced manufacturing workstation for the fabrication of composite parts. The project will apply sensor-based, hierarchically controlled robotic systems to automated fiber placement of thermoplastic composite materials. In such a system, continuous, fiber-reinforced thermoplastic polymer is fed from a spool onto a mandrel or part form. The orientation of the fiber is based on the part's geometry and structural design. As the composite material is applied, it is heated to melt the polymer matrix and then consolidated in place as it cools.

The project will demonstrate that fiber placement via two cooperating robot manipulators and in-situ consolidation can produce composite parts with complex shapes. Finite-element thermal modeling of the process is being conducted to gain insight into the relationships between the various process parameters and other system variables. Sensor systems are integrated into the process to provide necessary feedback data. The finished parts are inspected using various mechanical testing and non-destructive evaluation techniques to determine the relationships between the process parameters and the resulting part integrity.

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FACTORY AUTOMATION SYSTEMS

PRODUCT DATA EXCHANGE STANDARDS FOR THE APPAREL INDUSTRY

Numerous vendors supply computer-aided apparel design and pattern-making equipment. Each vendor's equipment represents the design and pattern data in a unique or proprietary file structure, preventing the direct electronic exchange of data among different organizations. NIST is helping the apparel industry to develop standards for product data exchange. NIST researchers have developed a prototype computer program for translating apparel pattern data between different file storage formats. The program demonstrates the feasibility of using a single, standard, interchange format. The program implements an information model also developed at NIST.

The software was developed as part of an ongoing project at NIST to extend the emerging international Standard for the Exchange of Product Model Data to apparel applications. The work is partially sponsored by the Defense Logistics Agency and is being carried out in cooperation with the Computer Integrated Manufacturing Committee of the American Apparel Manufacturing Association. In the short term, the goal is to develop a neutral data format for exchanging two-dimensional pattern data between apparel CAD systems. In the longer term, researchers plan to develop an information model that can be used to encompass the entire apparel life cycle.

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INSPECTION AND TOLERANCES RESEARCH

To establish a unified framework of U.S. and international standards dealing with mechanical tolerance issues, NIST scientists are carrying out a program of research and technology development in inspection and tolerances. These activities are aimed at developing a common technical basis for tolerance standards. The efforts involve support of the development of emerging standards on dimensional measurement methods, particularly regarding coordinate measuring machine (CMM) methods.

The standards of interest include standards for tolerance models, inspection performance, mathematics of tolerances, and statistical tolerance methods. Examples of critical emerging and draft standards are the Standard for the Exchange of Product Model Data, the Mathematical Definitions of Dimensions and Tolerances, and Performance Testing of Coordinate Measuring Machine Software. The work also involves participation on standards committees, research on measurement methods, and interaction with other government agencies to assess and improve the performance of CMMs used in their laboratories and production facilities.

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NATIONAL PDES TESTBED

Product data is an integral part of the information shared across computer applications and organizations. That is, it is a crucial part of any integration scheme. Currently, no commercial products exist that allow systems to share information in a standard way and, consequently, be integrated together. STEP (the Standard for the Exchange of Product Model Data), when implemented, will simplify this integration problem. However, STEP is still in the definition/testing phase. NIST is developing the National PDES Testbed to provide technical leadership and a testing-based foundation for the rapid and complete development of STEP. (PDES stands for "Product Data Exchange using STEP.")

Major objectives of the testbed project include: the identification of computer software applications that will use STEP, the specification of technical requirements for these applications, the evaluation of the proposed STEP standard with respect to application requirements, the design and implementation of prototype STEP applications, the establishment of configuration management for STEP specifications and certain supporting software, and improved interactions between organizations working to develop STEP.

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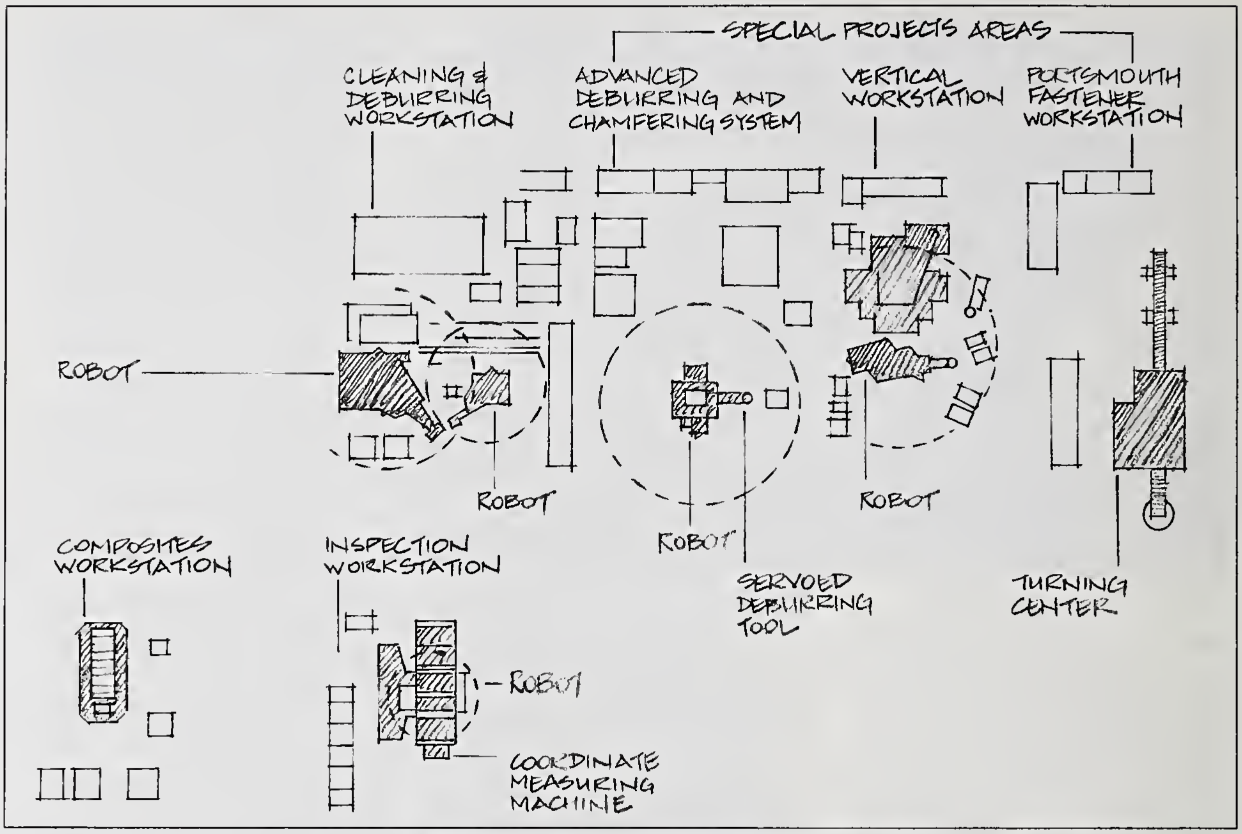
MANUFACTURING SYSTEMS INTEGRATION

Despite years of product development, truly modular, flexible, integrated manufacturing systems are still not prevalent in U.S. industry. In particular, information sharing across engineering, production management, and control systems is still not possible. Furthermore, no standards specify the precise interactions among these systems. To address these problems, NIST has embarked on the manufacturing systems integration (MSI) project. The goal is to build a testbed environment for conducting integration experiments with a variety of commer-



Schematic of the Automated Manufacturing Research Facility.

cial, university, and in-house software/hardware.

The MSI research team has designed, and is in the process of implementing, a systems architecture to serve as the foundation for this experimentation. The MSI architecture is a step in the continuing evolution and refinement of hierarchical control principles developed in the NIST Automated Manufacturing Research Facility. Ongoing research topics include: distributed, real-time planning, scheduling, and control; running the systems with both real and animated equipment; separate but integrated administrative control and task control; information models for all subsystems; and production management error recovery. NIST researchers are working with several major universities, auto companies, aerospace companies, and vendors. Team members are active in the international standards arena.

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DESIGN RESEARCH LABORATORY

As automated design becomes more and more prevalent, researchers are, of necessity, becoming more interested in the "process" of design. Furthermore, the emphasis on concurrent engineering is making this an iterative and integrated process, rather than a one-shot, stand-alone one. Several research questions remain. Can software tools be developed to create and maintain candidate designs in digital form? Can data structures for representing and updating the knowledge contained in these designs be developed? Can formats for exchanging that knowledge across manufacturing functions be devised? To address these and other interesting questions, NIST is building an engineering design laboratory equipped with state-of-the-art computers and software.

NIST researchers are investigating the issues involved in integrating a variety of commercial and university "design" tools. They also have begun a project aimed at capturing and representing "design knowledge and intent" in a manner that is compatible with the emerging Standard for the Exchange of Product Model Data. The work currently is focused on rigid mechanical parts, including assemblies. NIST researchers are working with several major universities through the Defense Advanced Research Projects Agency.

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ROBOTIC CRANE TECHNOLOGY

Shipbuilding, aircraft manufacturing, and building construction operations rely on cranes with a single cable for lifting heavy loads. The problem with this approach is that the load is free to rotate and swing with the slightest side force. NIST has designed a crane that provides a very stiff load platform that can be used to lift heavy loads or as a base for mounting a conventional industrial robot. The design consists of an equilateral, triangular platform suspended from six wire cables. By adjusting the length of each cable from independently controlled winches it is possible to position the platform accurately. As long as the cables remain in tension, this configuration provides a significant improvement in stiffness and positioning capability over conventional cranes.

NIST is building five testbed systems to investigate the performance of this crane design. These consist of:

- a full-scale model with a 9,072-kg capacity;
- a ¼-scale model with a small industrial robot mounted upside down on the lower platform;
- an ⅛-scale model for performing stiffness, damping, and stability measurements;
- a single-cable, 1-D testbed for developing real-time control algorithms; and
- a ¼-scale model bridge crane with a servoed X-Y carriage.

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RESEARCH FACILITIES

AUTOMATED MANUFACTURING RESEARCH FACILITY

The Automated Manufacturing Research Facility (AMRF) is a major national laboratory for technical work related to interfaces and standards for the next generation of computer-automated manufacturing. The facility, begun in 1981, was put into full operation in 1986. A companion installation, located in the adjacent instrument shop, serves as a state-of-the-art production facility, making use of the best commercially available technology.

The workstations of the AMRF are being used in active research programs by NIST researchers, industrial research associates, guest researchers, university personnel, and scientists and engineers from other government agencies.

CAPABILITIES: The facility currently supports research in machine tool and robot metrology, sensors and sensory processing, robot safety, robot control, software accuracy enhancement of machine tools, process planning and data preparation for machine tools and robots, parts routing and handling, real-time control of robots and aggregations of devices, workstation control, cell control, and materials-handling control. It is particularly valuable for studying interfaces between control modules and among data users. The AMRF is unique in the opportunities it provides for studies of an integrated system of significant size.

The facility consists of a two-robot cleaning and deburring workstation, inspection workstation, vertical machining workstation, and two special project areas, which are occupied by applications-oriented projects. Currently in the special projects areas are an advanced deburring and chamfering system, which will perform production deburring of jet engine blades and discs, and an integrated turning workstation, which machines threaded fasteners from difficult alloys. It will be sent to the Portsmouth Naval Shipyard upon completion.

Nearby are a manufacturing systems integration (MSI) system and an early-stage composites manufacturing workstation. Using the MSI system, researchers continue the study of AMRF control and data architectures, making use of the shop floor facility for validation of simulated results.

AVAILABILITY: Due to the nature of the problems addressed, the AMRF is generally best suited for research projects of an extended nature. Most successful work to date has involved a close working relationship between NIST and a collaborator for periods of 6 months to 1 year.

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ACOUSTIC ANECHOIC CHAMBER

This facility is used to perform acoustical measurements under free-field conditions, usually at moderate to very small sound pressures. These measurements include calibrating the free-field amplitude and phase responses of sound transducers, such as microphones and loudspeakers, and determining acoustic intensity in well-defined sound fields.

The facility is a vibration-isolated shell-within-shell structure. All interior surfaces of the inner shell are covered with a sound-absorptive treatment consisting of custom-designed, triple-density wedge modules. The chamber dimensions, measured wedge tip to wedge tip, are 6.7 m × 10 m × 6.7 m, defining a volume of 450 m^3. A wire-mesh floor permits access to instrumentation within the chamber.

Accessories include signal, control, and power lines as well as instrumentation supports on all six interior surfaces. Air-conditioning ducts are acoustically treated and vibration-isolated. Temperature and humidity within the chamber are independently controlled by the heating, ventilating, and air-conditioning system (HVAC).

CAPABILITIES: The chamber provides a highly anechoic sound field and a very small mechanical ambient noise level. The wedge modules are designed to absorb 99 percent or more of the normally incident sound energy at frequencies above 45 Hz. If increased acoustical isolation from the HVAC system and a uniform temperature within the chamber are required simultaneously, the air flow to the chamber interior can be turned off while controlled air flow continues between the inner and outer shells.

APPLICATIONS: The chamber is used to calibrate and to characterize acoustical instruments and their directionality under free-field conditions at moderate to very small sound pressures, to measure non-linear distortions, and to determine self-noise equivalent free-field sound pressures. The chamber is used for basic research aimed at developing methods for calibrating microphones of standard and non-standard geometries, for calibrating sound intensity probes and other transducer arrays and instruments, and for determining acoustic intensity.

AVAILABILITY: This facility has substantial potential for use by researchers in industry, universities, and other government agencies. Scheduling arrangements can be made for collaborative programs and individual research.

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CHEMICAL SCIENCE AND TECHNOLOGY LABORATORY

The chemical-manufacturing, energy, health-care, biotechnology, food-processing, and materials-processing industries all require accurate, high-precision measurements for characterizing chemicals, controlling production processes, and monitoring compliance with health, safety, and environmental regulations. Collectively, firms in these U.S. industries perform about 250 million measurements daily. The Chemical Science and Technology Laboratory (CSTL) develops the calibration and measurement standards for a wide range of instruments used in measuring pressure, temperature, gas and liquid flow rates, leak rates, and humidity. It also produces Standard Reference Materials and Standard Reference Data needed to achieve ever-lower detection limits and to improve quality, productivity, and efficiency.

The laboratory maintains the national system of chemical measurement and coordinates the system with measurement systems of other nations. It provides advisory and research services to other government agencies; conducts basic and applied research in analytical chemistry, biotechnology, chemical engineering, and physical chemistry; and conducts interdisciplinary research efforts with other NIST laboratories in these areas.

Recent CSTL projects have explored new chemical separation and purification methods and automated techniques for preparing protein crystals, DNA fragments, and other samples essential to determining the three-dimensional atomic structure of biological compounds. Another major initiative aims to develop reliable computer models and simulations for studying and describing chemical processes.

A newly formed consortium of U.S. businesses and government agencies is developing a prototype automated analytical chemistry laboratory. The modular system, which will prepare samples, perform separations, and assay the resulting compounds, is part of a larger effort to develop industry-wide standards for automated laboratory equipment.

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COOPERATIVE RESEARCH OPPORTUNITIES

BIOTECHNOLOGY

PROTEIN CHARACTERIZATION BY TWO-DIMENSIONAL ELECTROPHORESIS

NIST researchers are using a two-dimensional electrophoresis system to characterize proteins and peptides. They are probing the influence of size, shape, and charge on migration characteristics in the electrophoretic medium. Although well-defined protein "markers," especially of high molecular weight, are required to allow standardization of polyacrylamide gel electrophoretic systems, charged polymeric materials other than proteins may be considered for markers. Because staining and detection of such markers is of special interest, the researchers plan to examine the mechanisms of silver stains using neutron activation techniques.

Additional studies will be directed toward understanding the interactions of proteins with metal ions.The researchers will use image processing by state-of-the-art instrumentation to form meaningful databases. As part of this program, NIST plans to issue well-characterized mixtures of proteins as Standard Reference Materials that will be used to assess the abilities of existing and new electrophoretic techniques to separate and detect proteins.

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DNA CHEMISTRY

Working in several areas of DNA chemistry, NIST scientists are actively manipulating DNA to produce proteins, developing methods for measuring DNA damage on the molecular level, and developing methods for characterizing DNA, including profiling.

NIST scientists are developing experimental methods to measure DNA damage in mammalian cells exposed to free-radical-generating systems, such as ionizing radiation, elevated oxygen pressure, redox-cycling drugs, and a number of carcinogenic compounds. Free radicals produced in-vivo are thought to be mutagenic and carcinogenic. Measurement of DNA damage at the molecular level in mammalian cells is a prerequisite to understanding the chemical mechanisms of damage by free radicals. Techniques used for measuring DNA damage include gas chromatography, mass spectrometry, HPLC, and NMR spectroscopy.

NIST scientists are working on new methods for DNA profiling, ranging from developing well-characterized DNA fragment standards for restriction fragment length polymorphisms to performing research for rapid determination of DNA profiles by polymerase chain reaction amplification and automated detection of fragments. In addition, cooperative development of STR (short term repeat) technology and attendant standards is sought.

Research chemist Kristy Richie uses an electrophoresis gel system to analyze Standard Reference Materials that will help improve quality control for DNA "fingerprinting" techniques.

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NUCLEAR MAGNETIC RESONANCE

Programs are under way in which NIST scientists are using nuclear magnetic resonance (NMR) spectroscopy to study simple peptides and proteins, aminoglycoside and macrocyclic antibiotics, carbohydrates, glycoproteins, and DNA. The studies are facilitated by research materials isotopically labeled with carbon-13 or nitrogen-15, which are prepared by synthetic or biosynthetic methods.

The researchers are making extensive use of two-dimensional NMR methods, including homonuclear and heteronuclear chemical shift correlation and J-resolved techniques, nuclear Overhauser measurements in the rotating frame, and indirect detection. Measurements of chemical shifts and coupling constants provide information on the structures, stereochemistry, and conformations of biomolecules. Determination of nuclear Overhauser effects and relaxation times allows the imposition of distance constraints for three-dimensional structure analysis and characterization of molecular dynamics, respectively.

In the future, researchers plan to use three- and four-dimensional NMR techniques to investigate higher molecular weight biomolecules. Detailed knowledge of the three-dimensional structures and conformations of the biomolecules is important to an increased understanding of biochemical reaction mechanisms, enzyme specificity, drug-protein/DNA binding, and molecular-recognition processes.

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BIOPROCESS ENGINEERING MEASUREMENTS

NIST scientists are actively involved in the development of theories, measurement methods, models, and databases for upstream and downstream bioprocessing.

To accurately measure cell properties and product amounts during fermentation for bioreactors—upstream processing—researchers are studying precision thermodynamic measurements and the development of sensor technology. Downstream processing research includes the development of fundamental transport data and experimental methods and models for downstream separation of proteins and other biomolecules from complex mixtures. Among the methods under study are aqueous two-phase partitioning systems, chromatography, and electrokinetic demixing technologies.

In the area of biothermodynamics, NIST researchers have developed accurate and precise microcalorimeters to measure the heat released in enzyme-catalyzed biochemical reactions of interest to biotechnology. When coupled with equilibrium measurements, these measurements enable the reliable modeling of the thermodynamics of these processes. These data are used to predict reliably the efficiency of biochemical processes outside the normal measurement ranges for temperature, pH, and ionic strength.

Light-scattering studies are being made on biomacromolecular solutions to characterize their transport and thermodynamic properties. Models and experimental methods, needed to obtain the necessary data, are being developed to characterize aqueous two-phase separation techniques. NIST scientists are using small-angle neutron scattering to characterize chromatographic media in concentrated polymer solutions.

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ELECTRICAL MEASUREMENTS IN BIOREACTORS

Biological cells are known to be electrically active as demonstrated by the membrane potential present in all living cells. To provide fundamental data for sensor development in bioreactors, NIST researchers are developing measurement techniques for observing electrical activity of cell suspensions. Both linear and non-linear electrical responses are under investigation. The non-linear properties are especially interesting since they are expected to originate from the cell membrane.

Theoretical investigations are in progress to relate the observed electrical behavior to the activity of membrane transport proteins. The researchers are working to achieve a better understanding of the coupling of applied electric fields to membrane proteins, which can lead to practical schemes to influence the activity of cells. Research areas include electrical impedance measurements, study of microwave propagation in cell suspensions, electrostimulation of membrane processes, and observation of electrophoretic mobility with dynamic light scattering. Additional work is planned to measure the modulation of fluorescence from membrane proteins by applied electric fields.

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BIOSENSOR TECHNOLOGY

Biosensors use a biocomponent for the rapid, sensitive, and specific determination of an organic or biological compound. These devices, which can be utilized for on-line measurements in real time, have potential for widespread use in biomedical, clinical, environmental, and industrial monitoring.

Basic problems associated with biosensor technologies must be overcome, however, to ensure successful commercialization. NIST scientists are examining the mechanisms of protein immobilization and stabilization on surfaces. Optical fiber techniques are being developed for making non-intrusive, rapid and selective measurements. Fluorescence techniques, as well as Raman and resonance Raman spectroscopy, are used to determine the characteristics of amino acids, small peptides, and small organic molecules as an aid to eventually providing improved sensor specificity and selectivity. Molecular diffusion, rotational mobility, complexation, and photochemical interaction also are being examined.

NIST scientists are developing amplification technologies, including liposome-encapsulated molecules, multiturnover enzyme reactions, and ion-exchange polymer-modified electrodes. Such methods and devices provide the potential for extremely sensitive devices.

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CENTER FOR ADVANCED RESEARCH IN BIOTECHNOLOGY

At the Center for Advanced Research in Biotechnology (CARB), jointly established by NIST, the University of Maryland, and Montgomery County, Md., researchers study protein structure/function relationships. They are focusing on the measurement of protein structure by X-ray crystallography and nuclear magnetic resonance spectroscopy, and the manipulation of structure by molecular biological techniques including site-directed mutagenesis. Protein modeling, molecular dynamics, and computational chemistry are used to understand protein structure and

to predict the effects of specific structural modifications on the properties of proteins and enzymes. A variety of physical chemistry methods are used to measure and analyze structural changes, activities, and thermodynamic behavior of proteins under investigation. CARB maintains state-of-the-art facilities for crystallography, NMR spectroscopy, molecular biology, and physical biochemistry. Its computer facilities include a mini-supercomputer, several high-resolution graphics workstations, and access to the NIST Cyber 205 supercomputer.

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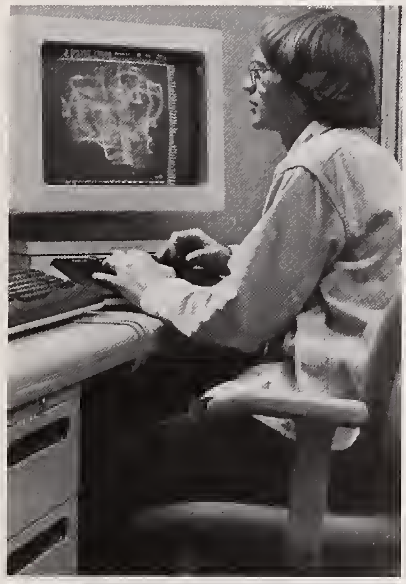

Top left. Research chemists Anne Plant and Steven Choquette investigate the optical characteristics of fluorescent molecules in order to increase the specificity and sensitivity of biosensing devices. Bottom left. Student Jennifer Schwarz grows protein crystals at the Center for Advanced Research in Biotechnology (CARB).

Left. Travis Gallagher, NIST/National Research Council post-doctoral fellow, displays the three-dimensional image of a protein molecule on a computer screen at one of the interactive color graphics workstations at CARB.

CHEMICAL ENGINEERING

SEPARATIONS

Separation and purification are critical steps in the manufacture of chemical products using existing and emerging process technologies, such as energy production or environmental protection. Separation processes affect both the economies of production and the fundamental ability to produce a product of desired form or purity. NIST is creating an engineering science base in membrane-based separations.

In the membrane area, NIST scientists are studying gas separations, biomolecule transport and adsorption, and reactive membranes; in the area of gas separations, researchers are investigating novel facilitated transport membranes for use in acid gas removal and air separations. Reactive membrane systems are being studied for trace component removal and for gas cleanup/recycle applications. In an interdisciplinary project, researchers are examining the adsorption of biological macromolecules to ultrafiltration membranes, with the objective of developing better predictive models for fouling, flux decline, and chromatographic applications. Other study areas include pervaporation membranes for close boiling mixtures and composite and polysaccharide membranes for small biomolecule separations.

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REFRIGERATION

Cooling superconducting electronics, magnetic devices, and highly sensitive infrared detectors requires specialized refrigerators capable of reaching cryogenic temperatures. Other applications, such as satellite cooling or gas liquefaction, demand long-term reliability and maintenance-free operation. Advances in the 1980s led to widespread interest in many regenerative-cycle refrigeration systems for these applications, including the use of pulse-tube refrigerators. NIST is the world leader in pulse-tube refrigeration research and is developing extensive engineering databases for these devices and other regenerator applications.

In pulse-tube studies, NIST scientists have developed the orifice pulse-tube refrigerator (OPTR), which has reached temperatures of 60 K in a single stage. Using thermoacoustic drivers (TADs) in place of mechanical compressors, an OPTR was designed with no moving parts. A patent, Strategic Defense Initiative Office (SDIO) innovative technology award, and an R&D 100 award have been received for this device, called a TADOPTR. Recently, the TADOPTR was proposed for liquefaction of natural gas at remote well sites, where about 30 percent of the gas produced would be burned to run the TAD, providing cooling to liquefy the remaining 70 percent.

Substantial research efforts have been applied to the development of improved regenerators for refrigeration systems. NIST researchers have assembled the world's largest database for adsorption of low-temperature gases on carbon. NIST computer models of regenerator performance are the only optimization tools available to designers of regenerators.

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CHEMICAL KINETICS AND THERMODYNAMICS

CHEMICAL AND BIOCHEMICAL THERMODYNAMICS

NIST researchers have developed precision oxygen combustion calorimeters to measure the enthalpy of combustion, from which enthalpy of formation can be derived, for samples varying in mass from 10 mg to 2.5 kg. These instruments are used to characterize thermodynamic properties for species of interest in biochemistry, organic chemistry (including strained species), phosphorous chemistry, and heterogeneous fuel technologies. The measurements also serve as the basis for Standard Reference Materials and Standard Reference Data for a variety of technologies, including energy.

The researchers maintain several data centers that evaluate thermodynamic data in inorganic, organic, and aqueous chemistry. They examine existing data, extrapolate these data to temperatures outside the range of measurement, create algorithms for data evaluation and manipulation, and produce reliable estimation schemes for predicting the properties of species for which measurements are not available. Current interests include the technological problems of combustion, nuclear waste disposal, and atmospheric ozone depletion and the prediction of the formation properties of organic species in the gaseous, liquid, solid, and aqueous phases.

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CHEMICAL KINETICS

The chemical kinetics program at NIST provides reliable chemical kinetics data, measurement methods, and theoretical models. Applications of this research include combustion, new chemical technologies, the chemistry of the upper atmosphere and other planetary atmospheres, effects of ionizing radiation on materials, solar energy conversion, biotechnology, flue-gas cleanup chemistry, acid rain, toxic waste incineration, coal conversion, and analytical applications of kinetics.

Among the experimental projects under way at NIST are pulse radiolysis of aqueous solutions and kinetic mass spectrometric studies (Fourier transform ion cyclotron resonance, high-pressure mass spectrometry, tandem mass spectrometry) of the kinetics and thermochemistry of ion/molecule reactions and ion/molecule clustering processes.

Researchers also are studying free-radical kinetics using heated single-pulse shock tubes, flash photolysis kinetic absorption spectroscopy and a flash photolysis resonance fluorescence technique, vacuum ultraviolet laser photolysis with kinetic absorption detection, and high-temperature reactors. Resonance enhanced multiphoton ionization (REMPI) spectroscopy is used to provide new, previously unobtainable data about the electronic structures of a wide variety of free radicals. REMPI procedures also lead to very sensitive and selective schemes for the optical detection of the radicals. An important focus of the kinetics program is the production of databases of evaluated chemical kinetic data, as well as the design of databases and relevant software.

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INORGANIC ANALYTICAL RESEARCH

ATOMIC SPECTROSCOPY

Atomic spectroscopy methods are probably the most widely used analytical techniques in industry today. Considerable research is required, however, to keep up with the changing needs of industry. At NIST, research in atomic spectrometry is focused on several different areas. For instance, researchers are working to improve the analytical capabilities of direct current plasma and inductively coupled plasma, as well as experimenting with the glow discharge as an atom reservoir.

Additional research concerns the use of spark sampling for the direct analysis of solids, the continued development of laser-enhanced ionization in flames, and the use of uv-Vis Fourier transform spectroscopy for the purpose of optical line assignments of atomic transitions in commonly available atom sources. X-ray fluorescence is used for homogeneity testing and certification of a wide variety of metals, ceramics, and other materials.

NIST scientists also are developing a series of neutral density filters that can be issued as Standard Reference Materials for verifying the accuracy of the transmittance and absorbance scales of ultraviolet, visible, and near infrared absorption spectrophotometers.

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ANALYTICAL MASS SPECTROMETRY

Analytical mass spectrometry has played a key role in industries, such as the semiconductor industry, that require accurate measurements of trace elements in raw materials, products, and product containers. The NIST inorganic mass spectrometry program is concerned with developing analytical capabilities for making highly accurate determinations of trace inorganics using stable isotope dilution and highly precise measurements of isotopic compositions, as well as highly accurate measurements of absolute isotopic compositions to redetermine atomic weights.

Areas of research include instrumentation in thermal source, inductively coupled plasma source, and ionization and chemical separations at the trace level using chromatography and other techniques. Methods are developed for generating and maintaining pure reagents and a clean environment to decrease limits of quantitation.

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INORGANIC ELECTROANALYTICAL RESEARCH

Electroanalytical research is vital to the development of methods and materials for environmental and clinical determinations. A broad range of electroanalytical and chromatographic techniques, such as voltammetry, coulometry, and ion chromatography, are used by researchers at NIST for analysis and research. Research on standardization of pH and electrolytic conductance leads to the certification of new classes of Standard Reference Materials.

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ACTIVATION ANALYSIS

Nuclear methods of analysis provide unique approaches to quantitative analysis. In this research, methods of nuclear analysis are investigated utilizing the 20-MW NIST research reactor. All areas of the technique are researched, including the capabilities of cold-neutron activation, the use of monitor activation, radiochemical separations, the determination of new mathematical procedures for the resolution of gamma spectra, the development of prompt gamma activation techniques, the use of charged-particle activation techniques, and neutron depth profiling.

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ORGANIC ANALYTICAL RESEARCH

LIQUID CHROMATOGRAPHY ELECTROCHEMISTRY

Although liquid chromatography (LC) is one of the most widely used analytical techniques in industrial laboratories, its use can be made even more widespread by improvements in detection methods. NIST researchers are working to develop novel approaches for the electrochemical detection of organic analytes separated by liquid chromatography. For this technique to be applied most effectively, the electrode reaction mechanisms of the compounds to be detected must be investigated using techniques such as cyclic and reverse-pulse voltammetry, coulometry, and LC/ultraviolet spectroscopy.

Ongoing NIST work focuses on the use of differential pulse and dual-electrode detection, as well as the development of single- and array-microelectrode detectors. Researchers also are developing new liquid chromatographic separations employing specific chemical interactions, including chelation, ion-pairing, charge transfer complexation, and acid-base equilibria.

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CAPILLARY ELECTROPHORESIS (CE)

The exciting new technique of electrophoresis in small-diameter capillaries provides tremendous opportunity for basic research into the fundamental properties and interactions of ions and neutral molecules. The association of analyte ions via simple electrostatic attraction, or more stable complexation, may be directly studied by the effect on the electrophoretic mobility of the ions. Hydrophobic interactions between neutral molecules and charged micellar surfactants can be investigated using micellar electrokinetic capillary electrophoresis (MECE).

Through the use of electrolyte modifiers such as urea and methanol, MECE can be extended to very non-polar molecules. Surfactants with more specific interactive character, such as aromaticity and chirality, need further exploration. Other "physical" additives also promise to increase the applicability of CE. Soluble polymeric gels can be used to provide molecular weight discrimination for biomolecules, where differences in charge may be too small to allow direct CE separation.

Detection in CE is also a hot research area. The small sample size and capillary dimensions make sensitive detection difficult. Electrochemical detection in CE by the use of microelectrodes does not suffer from the dimensional liabilities normally associated with the use of small capillaries. Using knowledge gained from the recent exploration of oxidative detection via carbon fiber and platinum electrodes, researchers should also be able to carry out reductive detection at mercury film electrodes. Sensitive optical CE detection using axial absorbance, as well as laser-source thermal lens and fluorescence modes are of current interest at NIST.

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BIOANALYTICAL SENSORS

Biosensors are a new generation of analytical devices with the potential for widespread use in biomedical and industrial monitoring applications. Biosensors will incorporate the latest advances in biotechnology to provide high specificity and sensitivity. Biologically derived substances have great value as components of sensing devices because of their binding specificity, the strength of their interactions, and their potential for use in conjunction with a wide variety of amplification schemes. Immunological, enzymatic, and receptor-ligand interactions are being explored as the basis for analytical measurement.

NIST researchers are using a variety of optical techniques for detection. Detection can be based on changes in size and rotational mobility of analytes or binding agents upon interaction, or it can be the result of enzymatic activity that occurs due to analyte binding, causing enhanced fluorescence or absorbance signals. Amplification can be achieved with release to liposome-encapsulated molecules or multi-turnover enzyme reactions. Combining high specificity for analytes with amplification provides the potential for extremely sensitive devices.

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SUPERCRITICAL FLUID EXTRACTION (SFE)

NIST researchers are studying the elucidation of the basic mechanisms responsible for analyte release in supercritical fluid extraction processes. They are designing experiments to determine the role of pressure, temperature, and extractant fluid composition on the equilibrium and kinetic processes of analyte release from both simple model and complex environmental matrices.

Research in this area also encompasses the practical application of analytical SFE to a wide variety of sample matrices; the modeling of the analyte-trapping process and concurrent design/optimization of trapping media; hardware and software development for automated extraction systems for on-line chromatographic analysis; investigations of intermediate analyte class separations based on solid phase adsorbents, following the extraction; and the design of expert systems for the optimization of SFE system performance. Analyte/matrix combinations being studied include polycyclic aromatic hydrocarbons and polychlorinated biphenyls in such matrices as sediments, soils, and air particulate matter; fat-soluble vitamins in foods and serum; and drugs of abuse in body fluids and hair.

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LABORATORY AUTOMATION AND ROBOTICS FOR ORGANIC ANALYSIS

NIST has joined with industry and other government agencies in a cooperative project to develop automated analytical devices for organic analysis based on new chemistries and apparatus, as well as on laboratory robotic systems.

One project focuses on the development of an on-line system for extraction of solid samples (using supercritical fluids and volatile liquids) with deposition onto a short chromatographic column, followed by selective elution for conventional chromatographic analysis. In a second project, researchers are studying the use of a state-of-the-art laboratory robotic system for automated sample preparation of samples for clinical and environmental analysis.

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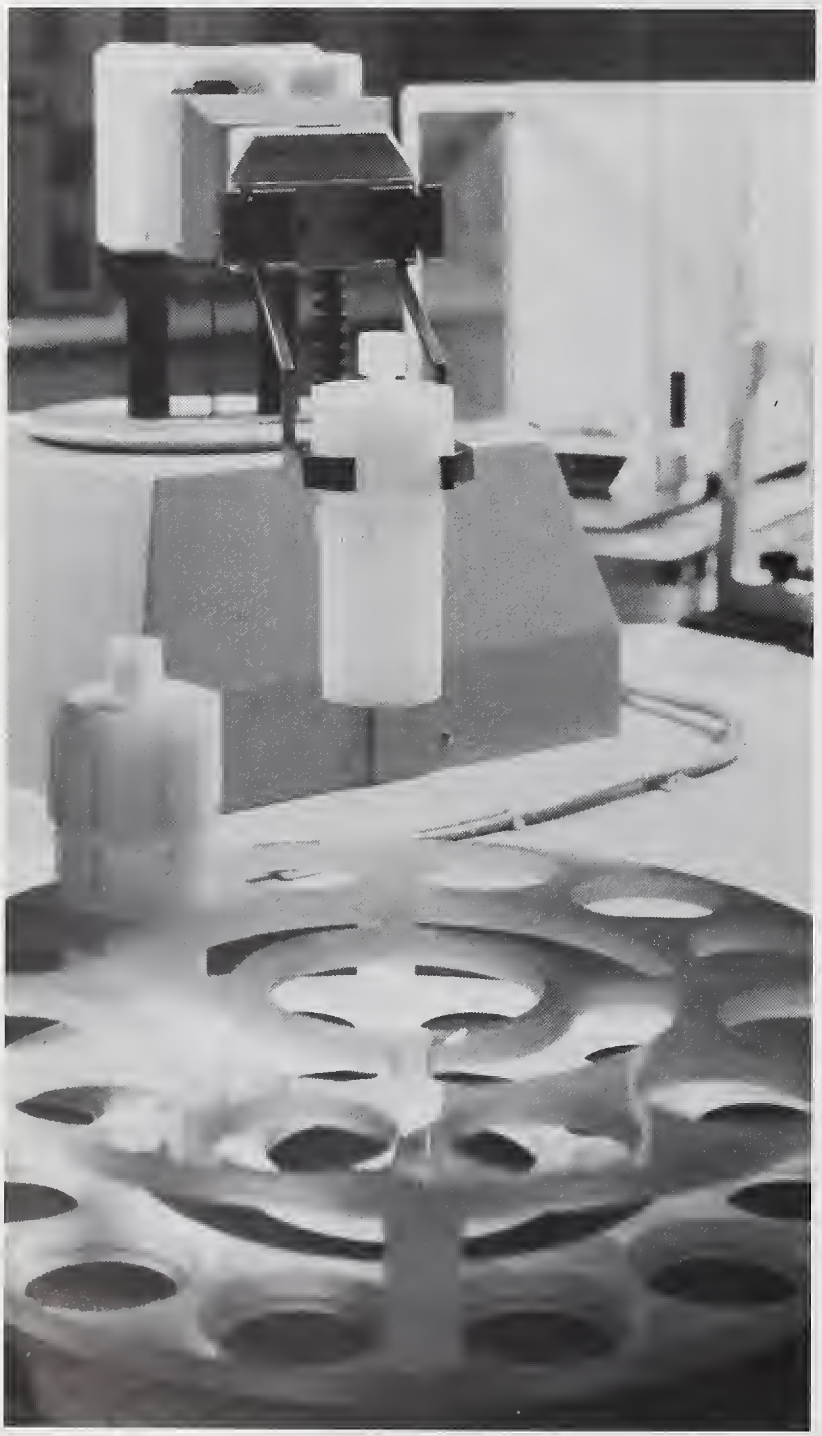

NIST has organized a consortium of industry and government researchers to develop a prototype automated analytical chemistry laboratory. When completed, the modular system will perform the major steps of a chemical analysis: sample preparation (dissolving by microwaves, as shown in photo), separation, and detection.

SUPERCRITICAL FLUID CHROMATOGRAPHY

Supercritical fluid chromatography (SFC) in both capillary and packed columns has been shown to offer several advantages when compared with liquid and gas chromatography for high-efficiency separations of non-volatile, reactive, or thermally labile molecules. NIST scientists are investigating the variables that influence retention, selectivity, recovery, and efficiency of SFC separations; the use of SFC as an analytical technique for providing high-efficiency separations and accurate quantitative analysis of complex mixtures; and the use of supercritical fluids for extraction of solid and liquid samples emphasizing on-line extraction and analysis of micro samples.

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FUNDAMENTAL CHEMICAL AND PHYSICAL PROCESSES IN CHROMATOGRAPHY

Solute retention in chromatographic systems is the result of a complex assortment of molecular interactions between the solute, the stationary phase, and the mobile phase. The diversity of these interactions can be used to optimize separations for different classes of compounds by varying separation parameters, such as stationary phase and/or mobile phase composition, and column temperature. An understanding of these fundamental retention mechanisms facilitates the optimization of separations in gas chromatography (GC), liquid chromatography (LC), and supercritical fluid chromatography (SFC).

Recent LC research has focused on the design of chemically bonded stationary phases (such as monomeric and polymeric C_{18} phases and charge transfer phases), which offer unique capabilities for the separation of isomeric compounds and compound classes. Polycyclic aromatic hydrocarbons have been used as model solutes for investigating retention mechanisms. Since these compounds are highly isomeric, retention effects resulting from differences in solute shape can be isolated by studying isomer sets.

Molecular modeling of solutes and stationary phase species holds the potential to provide further insight into retention mechanisms. Similar approaches can be taken to characterize retention processes in GC and SFC. A comparison of the retention behavior for isomer sets in GC, LC, and SFC may prove useful in developing a unified understanding of chromatographic retention processes.

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TRACE ORGANIC ANALYSIS USING HIGH-RESOLUTION CHROMATOGRAPHY

NIST research in high-resolution chromatography is focused on the development of gas chromatography (GC) and liquid chromatography (LC) methods for the determination of individual organic compounds in complex mixtures. Research areas in GC include:

- development of systematic chromatographic approaches for sample preparation/cleanup and analyte preconcentration prior to GC analysis;
- development of multidimensional chromatographic procedures using stationary phases of differing selectivity;
- use of simultaneous multiple and/or selective detection systems (such as mass spectrometric, electron capture, and flame photometric detectors); and
- development of on-line extraction and analysis of solid microsamples.

Recent activities have emphasized the measurement of trace levels of environmentally significant compounds, including polychlorinated biphenyls and polycyclic aromatic hydrocarbons, in natural matrices such as sediment, tissue, and airborne particulate matter.

Using LC, researchers will focus on the following approaches to analyzing complex mixtures: solid-phase extraction on short columns as an isolation/cleanup/preconcentration step; development of multidimensional LC procedures (multidimensional) to isolate and quantify selected compounds; selective detection systems (such as uv, fluorescence, electrochemical, mass spectrometric, chemical derivatization); and the development of microcolumn LC procedures.

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METHODS AND STANDARDS FOR DRUGS OF ABUSE TESTING

To reduce the use of illegal drugs, particularly in work-related activities, many employers are now performing drug tests on employees and prospective employees. Most of these tests involve analysis of urine samples for traces of drugs or metabolites. Because tests used to screen urine samples for drugs are subject to cross-reactivity from legal substances, it is strongly recommended, and in many cases mandated, that all positives from the screening procedure be subjected to confirmatory analysis by gas chromatography/mass spectrometry (GC/MS). The GC/MS methods are used not only to confirm the presence of drugs but also to quantitatively determine if concentrations of the drugs are above specified cutoff levels. Laboratories performing these confirmatory analyses must ensure that their methods produce accurate results.

To support accuracy in drug testing, NIST scientists are developing a series of urine-based Standard Reference Materials with appropriate concentrations of drugs of abuse. Drug levels are certified by a combination of two independent methods, one of which is generally GC/MS. Research is ongoing to develop alternate methods to complement GC/MS.

Considerable attention is now focused on an alternate matrix for drug testing. Human hair provides a long-term record of drug use, in contrast to urine which provides a record of only the previous 48 hours to 72 hours. NIST scientists are investigating analytical aspects of hair analysis for drugs of abuse. Research areas include the extraction of drugs from hair, measurement methods, the effects of hair treatments on drug levels, and differentiation between hair externally contaminated with drugs and hair with drug levels acquired through internal use.

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MASS SPECTROMETRY OF BIOMOLECULES

Major advances in mass spectrometry have improved its capabilities for characterizing biomolecules. Techniques such as fast-atom bombardment, electrospray, matrix-assisted laser desorption, and others permit the accurate determination of molecular weights of peptides and small proteins previously unmeasurable by mass spectrometry. Molecular weights greater than 100k daltons are now being determined with accuracies far exceeding those of other techniques. Advances in structural determinations of biomolecules include the use of collision-induced dissociation and photodissociation for determining sequence information for peptides and oligonucleotides. Considerable attention now is focused on coupling these new mass spectrometric techniques with developments in capillary electrophoresis and microscale liquid chromatography.

Scientists at NIST are investigating these new techniques to understand their capabilities and apply them to biomolecule characterization. They are particularly interested in applying these techniques to the quantitative analysis of species not previously amenable to mass spectrometric analysis. These capabilities should result in the development of reference materials to support industries involved in biotechnology and biomedical research.

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TRACE GAS MEASUREMENT TECHNIQUES

Accurate measurement of gaseous species is of great importance to many industries for applications ranging from quantification of pollutant and toxic gas emissions to the quality control of products. The validity of data derived from such measurements is tied directly to the availability of useful gas measurement techniques and to the degree of understanding of their capabilities and limitations. Although a variety of techniques have been applied to trace gas analysis, more research is needed to improve the present state of the art. This research is particularly important because of the growing need for the analysis of specific gas species in multicomponent gaseous mixtures; it also is necessary to extend accurate analyses to below the parts-per-million and parts-per-billion levels.

Current NIST research focuses on new detection systems using chemiluminescence, electrochemistry, infrared diode laser systems, capillary gas-liquid and gas-solid chromatography coupled to mass spectrometry, and isotope-dilution mass spectrometry. Institute scientists also are examining the use of class-specific detectors for gas chromatography and evaluating electronic circuitry to optimize signals and reduce instrumentation noise and drift.

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INSTABILITY OF COMPRESSED GAS MIXTURES

Inorganic and organic compressed gas mixtures are employed extensively throughout industry to calibrate equipment used to assess the quality of products and the effectiveness of emission controls. Although the stability of these mixtures is critical to their successful use, a number of instances have been noted in which instability has been observed, particularly in mixtures containing low levels of reactive gaseous species, such as nitrogen oxides, sulfur dioxide, and hydrocarbons.

The reasons for instability may differ somewhat for various gaseous species, but they are related to at least two possible phenomena: gas-phase reactions and gas-metal interactions with the internal surface of the cylinder. NIST researchers are working to improve the understanding and predictability of these phenomena. Their techniques involve those employed in surface science studies and other approaches, such as Fourier-transform infrared, diode laser, and other spectroscopies; mass spectrometry; metal analysis; trace water and oxygen analysis; chemiluminescence analysis; and the use of specifically doped mixtures and homogeneous gas phase kinetics.

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PROCESS MEASUREMENTS

FLOW MEASUREMENT RESEARCH AND STANDARDS

The accelerating costs of scarce fluid resources—particularly petrochemical fluids—are causing increased concerns about the performance levels of flow meters. Improved flow measurement traceability needs to be established and maintained so that realistic, quantified data are generated on a continuing basis to assure practical fluid measurements at satisfactory, specified levels of performance. To achieve the desired flow measurement traceability, NIST is designing transfer standards to link the performance of calibration facilities to appropriate national reference standards.

Because of the importance of these measurements, transfer standards need to be designed so that high levels of confidence can be placed in them and their performance. The new transfer standards will be rigorously evaluated against NIST fluid flow calibration standards. As part of the evaluation, the appropriate range of calibrations will be done on the developed standards so that performance levels can be assured at specified levels. Current fluid-metering research programs use laser Doppler velocimetry (LDV) techniques to focus on the pipeflows produced by conventional pipeline elements and by standard flow conditioning elements. New experimental programs are feasible using LDV or other anemometry or flow-visualization techniques to study other flows. NIST also has computational capabilities to model numerically a number of closed conduit flow fields.

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HIGH-TEMPERATURE REACTORS

The chemical and associated industries produce enormous amounts of byproducts. To avoid wasting industrial resources and polluting the environment, the impact of these byproducts must be minimized. The solution of the problem lies in: minimizing waste at the source by modifying industrial processes; recovering energy and chemicals for reuse; and converting pollutants to acceptable species. For all of these solutions, thermal treatments offer the most promising approach. Hence, there is urgent need for detailed mechanistic elucidation of fundamental thermal processes, such as pyrolysis and oxidation, that govern chemical conversion and destruction of both model compounds and actual industrial byproducts.

NIST maintains two instrumented facilities that are exceptionally promising for efficient pyrolytic and oxidative destruction of a wide variety of chemical compounds: a fluidized bed reactor and a plug-flow reactor designed for long residence times (seconds) at elevated temperatures. The research centers on kinetic studies, both global and mechanistic, of destruction of model compounds representative of major industrial byproducts, the most prominent of which are chlorinated hydrocarbons and nitrogen-containing species (amines and nitrocompounds, for example). The scientists are investigating the effects of temperature, pressure, residence time, and composition of reactor fluids on destruction efficiency of the model compound, as well as formation and disappearance of intermediate products. Diagnostic instrumentation includes both on-line sampling with subsequent analysis by several analytical techniques (GC, MS, and FTIR) and in-situ optical systems.

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CHEMICAL SENSOR RESEARCH

To understand the performance of thin-film and chemical sensors, NIST scientists are conducting research aimed at improving their accuracy, stability, selectivity, and response, as well as developing concepts for new measurement techniques. Activities incorporate adhesion, oxidation, interfacial diffusion, surface adsorption and desorption, and phase morphology to investigate mechanisms of chemical sensing. Analytical methods are used to relate the structure and composition of sensing devices to fabricating parameters and performance, and for development of mechanistic performance models. Research areas include thin-film thermocouples and resistance devices, moisture and pH sensors, and gas-phase chemical detectors based on tin and other metal oxides. Fabrication facilities available for this research are rf and dc sputtering for both alloy and reactive film deposition and gas reactors.

The researchers also are investigating the feasibility of thin-film systems as improved chemical sensors. Their research activities employ an array of surface characterization techniques to determine the electronic properties of films and single crystals for dielectric strength and surface conductance. Among the techniques used are X-ray and uv photoemission and thermal desorption spectroscopies, SIMS, and in-situ electrical measurements. Recent research activities have focused on the use of spontaneously organized monomolecular films for sensing of chemical and biochemical materials.

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CHEMISTRY OF SUPERCRITICAL WATER OXIDATION PROCESSES

Supercritical water oxidation (SCWO) is a new technology with high promise as a safe, efficient process for the minimization and destruction of hazardous materials. It is a totally contained process that takes place in water above its critical point, that is, at pressures greater than 22 MPa and temperatures above 374 °C. Under these conditions, many reactions proceed unusually rapidly producing benign end-products carried in the output water stream. Industrial implementation of SCWO has lagged, in part because of an inability to prescribe the process variables, such as the temperature, pressure, flow rates, and concentration of oxidants, required for safe, efficient waste destruction. These, in turn, demand understanding of fundamental thermodynamic and kinetic processes in supercritical media.

NIST researchers are seeking to supply both the understanding and data necessary to support reliable process modeling for SCWO. The experimental work centers around reactors for studies of chemical kinetics and durability of materials, as well as a unique flow reactor, which provides optical access for in-situ measurements of density, chemical species, temperature, and product/reactant concentrations. The researchers currently are studying the ammonia destruction reactions as functions of temperature, pressure, and added oxidant.

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CHEMISTRY OF HIGH-TEMPERATURE, GAS-PHASE SYNTHESIS OF MATERIALS

The semiconductor, coatings, and ceramics industries require high-performance thin films, particles, and fibers. This demand for materials with micro-engineered mechanical, electrical, and optical properties has placed greater emphasis on the development of new processing methods and on the need for increased fundamental understanding of materials-fabrication processes.

NIST researchers are seeking an understanding of high-temperature materials synthesis in a fast-flow reactor that provides reaction temperatures in excess of 1500 K and residence times as short as 10 ms, allowing extension of conventional chemical kinetics to temperature regimes representative of the chemistry relevant to materials processing. The reactor has provision for molecular-beam sampling of gas-phase species, including clusters, with subsequent mass spectrometric analysis. It also is equipped with optical excitation and diagnostic probe beams.

Currently, NIST scientists are researching the homogeneous (gas-phase) and heterogeneous (thin-film and cluster/aerosol) chemistry of silicon oxide. They are comparing measurements of species and growth kinetics to the predictions of chemical reaction and fluid flow models developed for the process. This approach identifies rate, purity, or defect-limiting steps in the process.

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DYNAMIC PRESSURE AND TEMPERATURE RESEARCH

U.S. industry increasingly relies on real-time monitoring of process parameters, particularly temperature and pressure, to produce efficiently a desired end-product, to warrant safe operation, and to assure equity in commerce. This trend is pervasive in the chemical and materials-processing industries, as well as in energy and raw materials production and transfer. NIST has a research program and is developing a test facility to provide a reliable basis for the evaluation and calibration of transducer dynamical response functions. The research seeks to develop a primary standard for dynamic temperature and pressure based on the fundamental properties of the molecular constituents of a dynamical system. Information about the molecules is accessed via laser-optical diagnostic techniques, and measurement times of the order of 10 ns at accuracy levels of 5 percent appear feasible. Through the use of these measurement techniques, an accurately characterized dynamical source will be developed. This reference source and its associated measurement system will provide industry with a means for assuring the accuracy of transducers used to measure time-varying temperature and pressure.

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CHEMICAL-VAPOR DEPOSITION REACTION KINETICS AND FLOW MODELING

Chemical-vapor deposition (CVD) is an important process used to grow thin films in the manufacture of microelectronics devices, as well as to produce high-performance coatings. CVD involves complex gas-phase chemistry, surface chemistry, and mass and heat transport processes. These highly coupled processes control the quality, uniformity, and yield of the deposited layers. NIST researchers are developing and testing numerical models that can be used to design and control CVD processing reactors. These models account for gas-phase chemistry, particle formation, heat and mass transport, and surface deposition leading to the formation of thin films.

The modeling effort is coordinated with a measurement program to supply the required chemical kinetics and materials-growth information and to validate the modeling results in a materials-synthesis flow reactor. The measurement program includes optical and mass spectrometric diagnostics of the gas phase and post-production evaluation of particle and film growth.

An interactive, graphics-based chemical kinetics program has been developed. Researchers are constructing a supporting thermochemical and kinetics numerical database, and they are developing two-dimensional heat and mass transport models, which simulate reactor geometries relevant to practical devices.

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PARTICULATE AND DROPLET DIAGNOSTICS IN SPRAY FLAMES

To minimize the cost of high-priced fuel, U.S. industry needs to obtain maximum energy output from fuel combustion. NIST researchers are tackling this problem by attempting to improve combustion efficiency. The researchers are studying the dynamics of spray flames to investigate droplet vaporization, pyrolysis, combustion, and particulate formation processes and to delineate the effect of chemical and physical properties of fuels on the above processes. The research results will provide an experimental database, with well-defined boundary conditions, for developing and validating spray combustion models.

The experiments are being carried out in a spray combustion facility, with a moveable-vane swirl burner, which simulates operating conditions found in practical combustion systems. A combination of non-intrusive probing techniques is used to obtain comprehensive data on spray combustion characteristics, including soot particle and droplet size, number density and volume fraction, gas composition, and velocity and temperature fields.

Currently NIST scientists are focusing their efforts on laser scattering and laser Doppler velocimetry measurements to determine the correlation between droplet size and velocity distributions, respectively, in both low-temperature and burning sprays.

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TEMPERATURE SENSOR RESEARCH

Improved industrial processes and sophisticated scientific research require temperature sensors that cover wider temperature ranges with better accuracy and precision than previously required. For example, the degradation of thermocouples exposed to high temperatures for extended periods of time represents a serious impediment to temperature measurements in jet engines, furnaces, and so forth.

NIST has several projects under way to test and improve the performance of currently available sensors, including thermocouples, and use resistance thermometers of various types. The temperature range covered by these projects extends from about 0.2 K to 2100 °C. NIST has excellent temperature calibration facilities, an automated laboratory equipped to evaluate thermocouples at high temperatures, several laboratories equipped for work on resistance thermometers, and a laboratory to evaluate industrial grade thermometers. NIST is planning a series of new materials and techniques to provide highly precise and accurate temperature measurements.

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SURFACE AND MICROANALYSIS SCIENCE

MICROBEAM COMPOSITIONAL MAPPING

Interpreting the relationship between the physical and chemical microstructure of materials is important in understanding their macroscopic behavior and in extending their in-service performance. Conventional microbeam techniques for elemental/molecular compositional analysis on the micrometer scale, such as the electron microprobe and ion microscope, have been restricted to quantitative analysis at individual locations. Mapping of the distribution of constituents has been possible only at the qualitative or semiquantitative level. However, recent NIST research developments have led to the production of the first truly quantitative elemental compositional maps. Quantitative compositional mapping with the electron microprobe has been demonstrated down to levels of 0.1 weight percent, while quantitative isotope ratio measurement in images has been demonstrated with the ion microscope.

Current research activities at NIST include extending compositional mapping to analytical electron microscopy, laser Raman microanalysis, and laser microprobe mass analysis. Potential projects could involve applying the compositional mapping instruments to materials characterization problems, developing new techniques for compositional mapping on other microanalysis instruments, and investigating basic topics in elemental and molecular quantitative analysis with microbeam instrumentation. Among the equipment available at NIST is an electron microprobe, an analytical scanning electron microscope, 200- and 300-kV analytical electron microscopes, an ion microscope (secondary ion mass spectrometry, SIMS), a time-of-flight SIMS, a laser microprobe mass analyzer, a laser Raman microprobe of NIST design, and extensive computer facilities, including image processing.

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ATMOSPHERIC AND CHEMOMETRIC RESEARCH

Current regional and global concerns with the impact of human activities on environmental contamination and wastes, atmospheric pollution, and potential effects on health and climate make it imperative to determine with a high degree of accuracy the individual sources of the noxious species. State-of-the art research, pioneered at NIST, makes possible unique source identification by application of the most advanced microchemical and isotopic analytical techniques, including accelerator mass spectrometry and high-precision gas isotope ratio mass spectrometry.

Complementing advanced isotopic-chemical characterization of atmospheric gases and particles is basic research in chemometrics, which represents the synthesis of chemical knowledge and measurement with modern statistical and computational methods. Work in this area is directed toward improving the quality of chemical measurements generally through advanced design, measurement and data analysis quality assurance, and graphical multivariate data exploration.

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Top. Chemical engineer Joe Magee prepares a simulated natural gas mixture for a Standard Reference Material to be used for precise density measurements. Bottom. Studies conducted by research engineer Cary Presser in the spray combustion facility are designed to provide more efficient use of both conventional and alternative fuels.

THERMOPHYSICS

PROPERTIES OF FLUIDS

Thermophysical properties data are essential for the design and operation of many chemical processes, such as supercritical extraction. To obtain these data, NIST scientists are using three new phase equilibria apparatus for studies at elevated temperatures: one makes VLE measurements on carbon dioxide-hydrocarbon systems and refrigerant-hydrocarbon systems; the second is a dew/bubble point apparatus extending to 800 K; and the third employs a palladium-silver membrane to measure the fugacity of hydrogen-containing mixtures.

Five exceptional instruments are available for making PVT, PVT_x, and heat capacity measurements on pure fluids and fluid mixtures: an isochoric PVT apparatus, a Burnett apparatus, a combined Burnett/isochoric PVT apparatus, a magnetic suspension densimeter for PVT and PVT_x measurements, and an instrument to measure constant volume heat capacity. Facilities also exist to determine properties along the two-phase coexistence line of pure fluids and mixtures and to make sound speed measurements.

NIST researchers also can devise techniques to characterize fluids and fluid mixtures when the temperatures, pressures, and times involved can result in reactions during the measurement process.

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THERMOPHYSICAL AND SUPERCRITICAL PROPERTIES OF MIXTURES

Chemical process technology requires accurate knowledge of various thermophysical properties of pure, polyfunctional chemicals and their mixtures. NIST research focuses on the most important of these properties—equilibrium phase composition, density, and enthalpy. NIST researchers are developing predictive methods for the properties of chemically dissimilar compounds, especially complex mixtures and aqueous solutions. Another project is aimed at developing accurate predictive models for the thermo-dynamic and transport properties of near critical and supercritical mixtures. This work includes PVT_x and VLE measurements on mixtures containing carbon dioxide, halogenated hydrocarbons, and similar supercritical solvents. Other experimental work involves using supercritical chromatography to measure diffusion coefficients in supercritical mixtures and theoretical studies to focus on applying extended corresponding states to supercritical systems.

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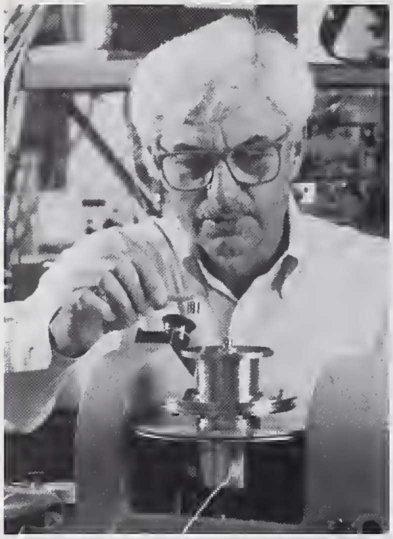
PROPERTIES OF ATMOSPHERICALLY SAFE REFRIGERANTS

Chlorofluorocarbons (CFCs) have been used widely for the past 50 years as refrigerants; as foam in building insulation, furniture, and car seats; and in many other applications. Recent evidence has shown, however, that CFCs are breaking down the ozone layer that protects the Earth from harmful levels of ultraviolet radiation.



Chemist Lloyd Weber measures chemical and physical properties of alternate refrigerants. The goal is to help industry find effective replacements for chlorofluorocarbons or CFCs, which break down the Earth's ozone layer.

Alternative chemicals must be found to replace the existing fluids within the next decade. For this to be achieved, an accurate knowledge of their thermophysical properties is required. NIST has a research program designed to provide these data to industry, ultimately in the form of interactive computer codes. The research includes extensive experimental measurements on pure fluids and mixtures, including PVT, PVT_x, vapor pressure, saturation density, heat capacity, thermal conductivity, viscosity, sound speed, and surface tension. The program also includes substantial effort in modeling fluid properties and in developing equations of state.

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PRESSURE STANDARDS

Achieving better understanding and control of the pressure measurement process will lead to better quality control in the manufacture of new materials and better design and performance of transducers for thousands of applications in modern technology. The measurement of pressure from subatmospheric to 689 MPa and above with uncertainties of tens to hundreds of parts per million is essential in modern technology.

The most fundamental instrument at these high pressures is the deadweight piston gauge. In the piston gauge, a force generated in a working fluid acts against the surfaces of a piston and against the walls of a cylinder that confines the piston. At high pressures, the materials undergo significant distortion, which leads to limitations and uncertainties in the area on which fluid is acting. The fluid interacts with the side of the piston, creating upward forces that are difficult to interpret. NIST scientists are using various tools—finite-element analysis to characterize distortion, better hydrodynamic measurement and modeling of the annular region through which the fluid flows, and improved piston gage designs—to understand these effects.

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VACUUM AND LEAK STANDARDS

Many industries depend on accurate vacuum (pressure) and leak measurements for research and development and for process and quality control. NIST develops and maintains pressure and vacuum standards from above atmospheric pressure to ultrahigh vacuum; leak or flow standards are operated from 10^{-3} to below 10^{-9} std cc/s. Facilities include five ultrahigh vacuum systems; two low-range flowmeters; high-accuracy mercury manometers; pressure and vacuum control systems; and vacuum, electronic, data acquisition, and data analysis equipment.

These facilities and measurement capabilities enable researchers to develop improved measurement techniques and equipment and to investigate the performance of vacuum and pressure equipment, specifically mechanical pressure gauges, momentum transfer gauges, ionization gauges, standard leaks, and residual gas analyzers. In addition, NIST plans to use this measurement capability to investigate properties of materials and physical phenomena of fundamental interest. Among the planned projects is the development of reference standards for the transition regime between low pressures measured by mechanical or electromechanical gauges and pressures measured by vacuum technology devices. The development of accurate methods to measure and characterize ultrahigh vacuum systems is also under investigation.

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RESEARCH FACILITIES

WATER FLOW MEASUREMENT FACILITY

The NIST water flow measurement facilities are used to establish, maintain, and disseminate flowrate measurements, standards, and data for the wide range of conditions needed by U.S. industry. Industry requests include flow meter calibrations, round robin testing programs to establish realistic traceability chains in the form of flow measurement assurance programs, data-generation programs for industrial groups and trade associations, and testbeds for carrying out industrial research programs focused on flow measurement topics.

CAPABILITIES: Water flow facilities enable flowrates up to 40,000 liters/min in pipe sizes that range up to 500 mm in diameter. Maximum operating pressure is 1 MPa. Flowrate determination schemes use static and dynamic gravimetric systems.

APPLICATION: These facilities are used to establish and maintain the national bases for the liquid flowrate measurement systems. The end result is orderliness in the marketplace, both domestically and internationally, for U.S. industries involved in the custody transfer and/or process control of valuable fluid resources and products. Potential users include the National Aeronautics and Space Administration, the Department of Defense, the oil and gas industries, the chemical and related industries, and the power and energy-generation industries.

AVAILABILITY: The facilities are available upon request to U.S. industry, other government agencies, and academia for collaborative research projects and calibrations.

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FLUID METERING RESEARCH FACILITY

The NIST Fluid Metering Research Facility combines primary calibration techniques with the capability to conduct detailed surveys of fluid velocity profiles in temperature-controlled water flows using laser Doppler velocimetry (LDV).

LDV is a non-intrusive technique for determining the fluid velocity at a particular location in the flow. It enables pipeflows to be surveyed for a wide range of piping configurations in pipe sizes up to 20 cm in diameter. Temperature control enables stable fluid and flow conditions to be established and maintained for extended test periods. The primary calibration technique uses dynamic gravimetric methods to determine accurately the bulk liquid flowrate.

CAPABILITIES: Water flowrates up to 240 liters/min in a range of pipe sizes up to 20 cm in diameter can be produced. Maximum operating pressure is 0.5 MPa, and temperature can be stabilized in the range from 16 °C to 30 °C.

APPLICATIONS: This facility is used to conduct calibrations, special fluid measurement tests, or selected research programs on flow topics. An industry-government consortium currently supports a research program on flowmeter installation effects, and the U.S. Navy is sponsoring a series of tests to assess the performance of selected flow transfer standards in a range of non-ideal installation conditions.

The facility is used to generate critical databases used to initiate or update the national standards on generic fluid metering topics. These standards are used for accurate custody transfer of fluid resources or products in the domestic and international marketplace or for process control in chemical manufacturing processes. Among the potential users of the facility are the Department of Defense, the National Aeronautics and Space Administration, the oil and gas industries, the engine manufacturing and testing industries, and the energy-generation industries.

AVAILABILITY: These facilities are available upon request to U.S. industry, other government agencies, and academia for collaborative research projects or calibrations.

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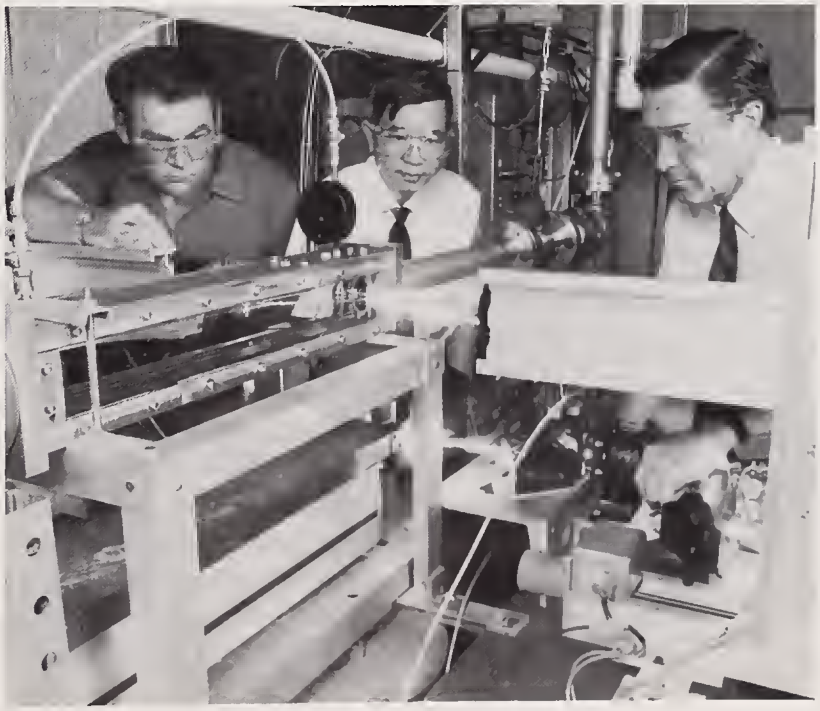
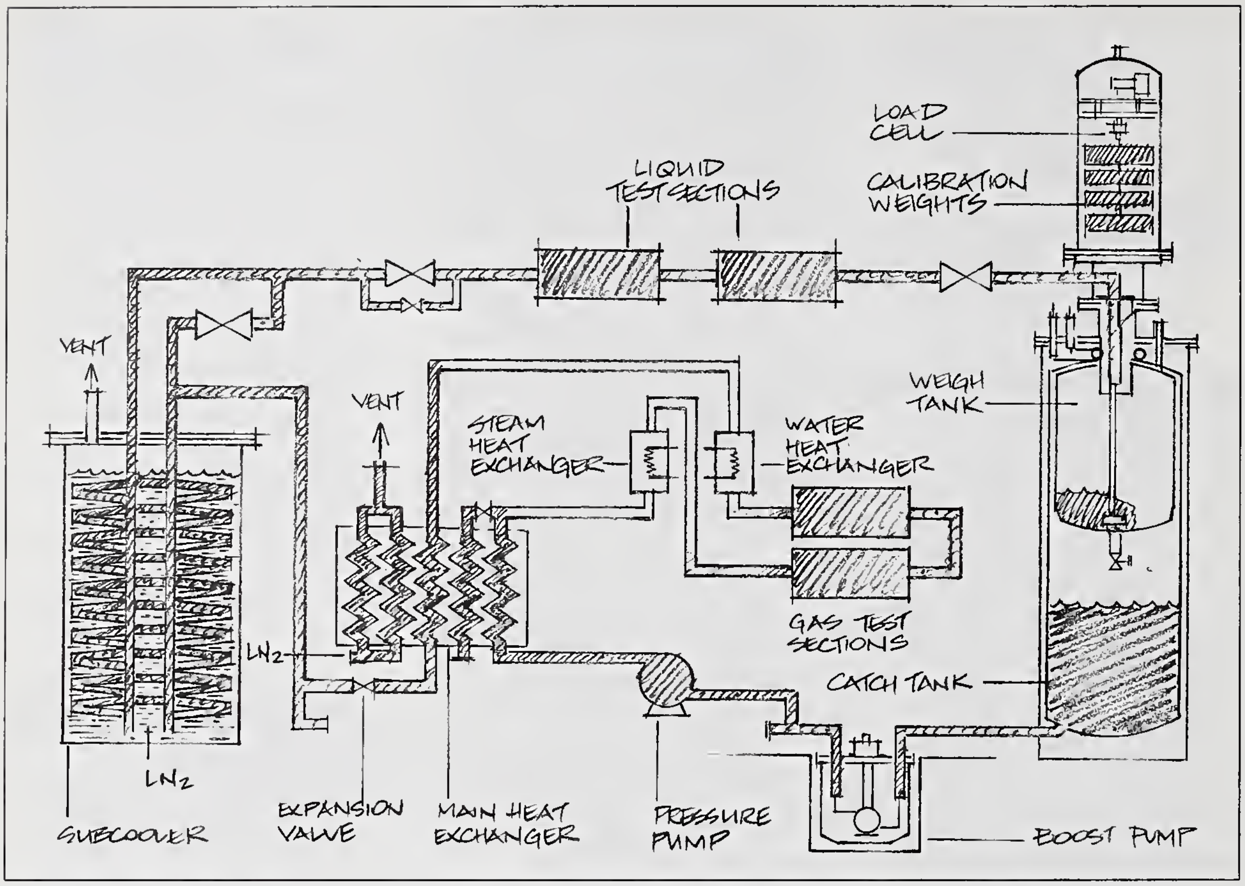
NITROGEN FLOW MEASUREMENT FACILITY

The nitrogen flow measurement facility is a mass-based reference system capable of both liquid and gas flow measurement. Well instrumented for temperature and pressure, the facility is adaptable and capable of a variety of piping arrangements. Located completely indoors, it is not subject to environmental changes.

CAPABILITIES: When configured for gas flow measurements, the facility has a flow rate range of 0.5 kg/s to 2.4 kg/s. In this mode, the nitrogen gas is at pressures of approximately 4 MPa and temperatures of approximately 288 K. The facility has a flow rate range of 0.05 kg/s to 10 kg/s when set up



Mechanical engineers George Mattingly (right) and T.T. Yeh (center), with technician Boyd Shomaker, study flow-measuring devices using laser Doppler velocimetry at the NIST fluid metering research facility.



Schematic of the Nitrogen Flow Measurement Facility.

for liquid flow measurements. Liquid nitrogen can be at pressures up to 0.7 MPa and temperatures between 75 K and 90 K in this configuration. This continuous flow facility permits dynamic mass flow measurements. For volume flow measurements, density is determined by making pressure and temperature measurements and calculating density from an equation of state.

APPLICATIONS: The nitrogen flow facility can be used for testing a variety of flow measurement instrumentation, including flowmeters, temperature sensors, pressure sensors, and densimeters. The ability to operate the facility at stable conditions for long periods of time permits testing of instrumentation stability. The ability to vary system parameters permits testing of instrument sensitivity.

AVAILABILITY: Collaborative or independent programs for this test facility can be arranged. The facility must be operated by NIST staff.

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NEUTRON DEPTH PROFILING FACILITY

The neutron depth profiling (NDP) facility uses a neutron beam for non-destructive evaluation of elemental depth distributions in materials. Working with the Institute's 20-MW nuclear reactor, researchers use the technique to provide concentration profiles for characterizing the near-surface regime of semiconductors, metals, glasses, and polymers to depths of several micrometers. The facility uses filters and collimators to produce a high-quality neutron beam with good thermal neutron intensity and minimum contamination with fast neutrons and gamma rays. An aluminum target chamber is used to contain the samples in a vacuum, and a full array of electronic components is available for data acquisition and analysis.

CAPABILITIES: With the thermal neutron beam provided by the reactor, depth profiling can be carried out with sensitivities approaching 10^{13} atoms/cm^2. A single analysis produces a profile typically 5 μm to 20 μm deep with a resolution of better than 30 nm. Once calibrated with the appropriate elemental standard, the concentration scale is fixed independently of the sample composition. Elements that do not produce charged particles under thermal neutron irradiation contribute no interference.

APPLICATIONS: Applications of NDP include range measurements for boron implanted in Si, GaAs, and MCT; observations on near-surface boron in glasses; lithium concentration profiles in lithium niobates and aluminum alloys; measurement of helium release in single-crystal nickel to investigate the He-trapping phenomena; and measurement of high-dose nitrogen implants in steels.

AVAILABILITY: The facility is available to qualified researchers on an independent or collaborative basis with NIST staff.

CONTACT: R. Greg Downing
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PHYSICS LABORATORY

Attending to the long-term needs of many U.S. high-technology industries, NIST's Physics Laboratory conducts basic research in the areas of quantum, electron, optical, atomic, molecular, and radiation physics. This research is complemented by work in quantum metrology and efforts to improve the accuracy and precision of time and frequency standards.

Much of the laboratory's research is devoted to overcoming the barriers to the next technological revolution, in which individual atoms and molecules will serve as the fundamental building blocks of electronic and optical devices. Manufacturing products based on ultrasmall, atomic-scale systems will require entirely new measurement techniques. To develop the necessary measurement capabilities, laboratory scientists are using highly specialized equipment, such as polarized electron microscopes, scanning tunneling microscopes, and synchrotron radiation sources, to study and manipulate the behavior of individual atoms and molecules.

They are investigating how atoms and molecules aggregate into clusters and microstructures and how these microstructures behave on surfaces and in solids. With lasers and other advanced spectroscopic methods, scientists have trapped atoms and ions, induced unusual quantum phenomena, and measured chemical reactions on material surfaces over time spans as short as a few hundred quadrillionths of a second, the time frame for a single molecular vibration.

In support of industries ranging from photography to aerospace to lighting, the laboratory is working to improve optical measurement techniques for a diverse range of applications, including remote sensing, advanced colorgraphic imaging systems, and optically pumped atomic clocks. Complementing research fosters the development of emerging X-ray technologies, such as X-ray lithography for the semiconductor industry and X-ray imaging.

Contact: Katherine B. Gebbie
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COOPERATIVE RESEARCH OPPORTUNITIES

ELECTRON AND OPTICAL PHYSICS

UV OPTICS TESTBED

The emerging field of high reflectance, normal incidence, soft X-ray/extreme ultraviolet (xuv) optics has a wide range of applications. The ability to produce high-quality images at wavelengths below 40 nm has allowed construction of xuv solar telescopes with unprecedented resolution; xuv microscopes able to study living biological samples with sub-micron resolution; and xuv photolithographic systems that will produce the next generation of integrated circuits.

NIST has initiated an xuv multilayer characterization facility at the SURF II electron storage ring, which is available to all researchers on a cooperative basis. The present facility is capable of measuring the reflectance or transmission of xuv optics (such as mirrors, filters, and gratings) as a function of wavelength, angle of incidence, and position on the optic from 8 nm to 60 nm. NIST is constructing a new facility that will extend measurement capabilities to shorter wavelengths, larger substrates, and more highly curved optics and at the same time increase resolution and accuracy. An optical testbed also is being constructed to measure the imaging properties of individual optics and entire optical systems. The first element of this testbed will be a quasi-real-time imaging device with a resolution goal of 25 nm.

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ATOMIC PHYSICS

VACUUM ULTRAVIOLET RADIOMETRY

As part of its research to understand and measure various forms of radiation, NIST is conducting vacuum ultraviolet studies involving radiation damage, polymerization of organic molecules, and solar simulation. NIST scientists have worked with researchers from industry to develop and test vacuum ultraviolet instrumentation, new spectrometer designs, and detector systems, especially for flight in space. They also have collaborated on special sources, narrow-band filters, and lasers. NIST scientists are interested in doing cooperative research in several other areas, including studying hollow cathode lamps, laser plasmas, and spark-discharge light sources as secondary standards. State-of-the-art radiometric facilities and advanced optical equipment are available at NIST for these studies.

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CHARACTERIZATION OF LOW-TEMPERATURE PLASMAS

The properties of low-temperature plasmas play a key role in the processing of materials such as semiconductors. Proper characterization of these plasmas is essential to develop accurate plasma diagnostics and useful plasma models for specific applications. At

NIST, plasma discharges are characterized utilizing optical emission spectroscopy, laser-induced fluorescence, laser scattering, and optogalvanic methods. Modeling of the plasma is also an integral part of this characterization. Discharge sources include low-pressure rf plasmas, stabilized arcs, glow discharges, heat pipes with laser resonance ionization, and inductively coupled plasmas.

An extensive array of laboratory equipment is available to accomplish this characterization, including several Nd:YAG pumped, high-resolution ($<0.1\ cm^{-1}$) dye lasers; Ar ion-pumped dye and ring dye lasers; a Fizeau wavemeter; a high-throughput (f/4), high-resolution (<.001 nm) spectrometer with an automated intensified diode array detector; a 80-MHz quadrature He-Ne laser interferometer; uv spectrometers; grazing incidence spectrometers; laboratory computers; and miscellaneous optics. The principal quantities measured are particle density distributions—both spatially and temporally—for electrons, atoms, ions, and molecules. Also included are electric-field distributions, electron and ion temperatures, and non-equilibrium phenomena.

CONTACT: James R. Roberts
A167 Physics
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EBIT FACILITY FOR RESEARCH ON HIGHLY IONIZED ATOMS

NIST's new electron beam ion trap (EBIT) source provides many opportunities for definitive measurements aimed at a basic understanding of plasma processes and atomic structure. Ions can be generated over a wide range of species and charge states (ultimately up to fully stripped uranium). Ions are radially trapped and probed with a monoenergetic electron beam. Electrostatic end caps confine the ions axially. A large magnetic field is applied by a superconducting magnet to pinch the electron beam to high density and provide additional radial trapping. The carefully controlled conditions in EBIT allow scientists to unravel complex collision processes and measure spectra to very high accuracy. Highly charged ions can be produced at low temperatures and observed in fluorescence with adequate brightness. The ion temperature can be further lowered by evaporative cooling techniques. A variety of instruments are available to characterize and probe the trapped ions, including X-ray spectrometers and a new laser system.

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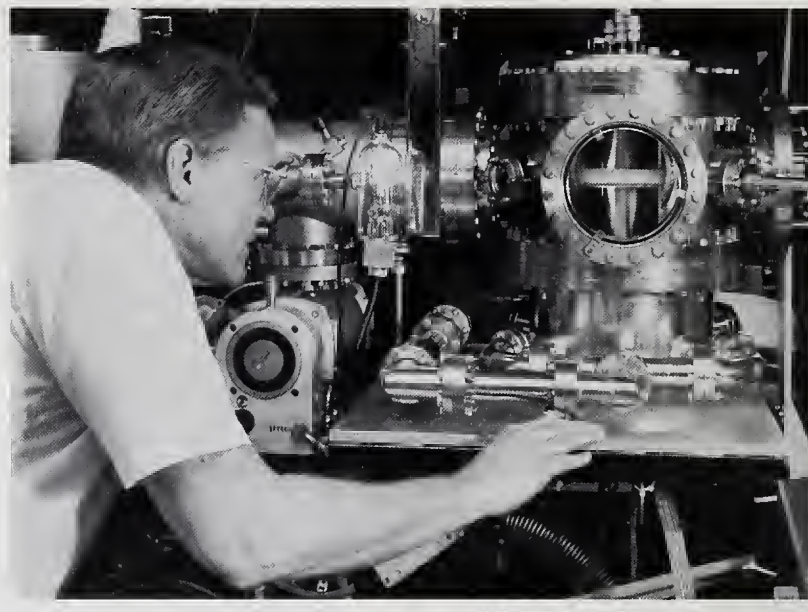
A167 Physics

RADIOMETRIC PHYSICS

LUMINESCENCE SPECTRAL RADIOMETRY

Luminescence techniques have broad application in virtually every scientific field, including radiation measurement, remote sensing, quantitation of biomolecules by intrinsic luminescence and immunoassay techniques, and characterization of laser, semiconductor, and superconductor materials. The accurate spectral radiometric quantitation



Above. Physicist James Roberts observes the optical emission from a plasma discharge generated in a radio-frequency reference cell. Right. Physical scientist Stephen Ebner analyzes data from the NIST Low Background Infrared Radiation Facility where blackbody, infrared sources are calibrated. These sources, in turn, are used to calibrate infrared detectors.

of light emission is an exacting task requiring painstaking radiometric measurements and knowledge of the fundamental chemical and physical processes represented by these radiative transitions. Standard lamps, both radiance and irradiance, and silicon detector radiometry provide the accuracy base for the spectral and quantum efficiency measurements. Luminescent phenomena under investigation at NIST are photo-, chemi-, thermo-, electro-, and bio-luminescences. NIST researchers are conducting luminescence radiometric research in the near-ultraviolet, visible, and near-infrared spectral regions and are developing accurate standards and measurement procedures for these regions. Facilities available for this research include various laser and lamp sources, the NIST reference spectrofluorimeter, and a low-light-level spectroradiometer now under construction.

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THERMAL RADIOMETRY

NIST researchers are investigating the use of thermal imaging cameras as a temperature-measuring tool. These devices may prove to be very useful in determining the quality of products and in investigating changes in different processes. Research projects involve the development of large-area blackbodies, use of Pt-Si as detector standards, and the characterization of thermal imaging cameras. Equipment available includes several heat-pipe blackbodies, a Pt-Si camera, and an infrared radiometer.

CONTACT: Robert Saunders
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UV RADIOMETRY

The measurement of terrestrial solar irradiance in the uv-b spectral region is being investigated by NIST researchers in order to provide improved techniques and standards in this region. Their work is of importance not only to scientists studying biological effects but to researchers investigating the aging of materials by uv light. Specific projects include the development of a reference spectral radiometer, broadband detectors, and source standards in the region. A high-accuracy spectrometer, standard detectors, and standard sources are available.

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SPECTRAL RADIOMETRY

Research and development programs at NIST span a broad spectrum of activities associated with the measurement of optical radiation, including spectral radiance measurements and new techniques for spectrophotometric measurement of dense optical media. These activities cover the ultraviolet spectral region from 200 nm to the far infrared region and include the development of appropriate detector methodology to perform the measurements and relate them to the U.S. radiometric measurement base. Specific research and development projects involve low-background infrared calibrations in a cryogenic environment, solid-state photodiode metrology, applications of detector metrology to all areas of radiometry, development of an absolute cryogenic radiometer, and application of laser heterodyne technology to optical density measurement.

Several well-equipped laboratories for optical measurements in the uv and visible spectral region are available for use, and new facilities are being developed that will enable scientists to research both detector development and optical properties of materials in the infrared spectral region.

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INFRARED SPECTRAL RADIOMETRY

NIST researchers are developing devices and techniques for high-precision measurements of radiant power in the 2 µm to 30 µm spectral region to enable characterization of infrared spectral sources, optical components and detectors. Novel experiments using these capabilities will investigate physical and chemical processes in materials and molecular structures. State-of-the-art radiometers, a cryogenic blackbody with multiple apertures, lead salt lasers, spectral instrumentation, and solid-state infrared detectors are being acquired for experiments. A unique low background infrared radiation (LBIR) facility is dedicated for this research and development effort.

Ancillary research projects in progress are measurements of optical density of filters using laser heterodyne technology and determination of spatial uniformity and linearity in the response of infrared detectors. Collaborative research opportunities exist in optical properties of materials and molecular structures in the infrared spectral region and in detector development.

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BIDIRECTIONAL SCATTERING METROLOGY

The bidirectional characterization of optical scatter from surfaces is a useful diagnostic in evaluating elements contained within large optical systems that require the minimization of scattered light. This information is needed for the development of ring-laser gyroscopes, telescopes, and superpolished mirrors. It is also used for the characterization of materials for use in stray light reduction in thermal control and inspection processes in optical manufacturing settings. NIST research projects involve the development of a multiangle scattering reference instrument and the development of measurement methodologies and Standard Reference Materials for the spectral range from the ultraviolet to the infrared region.

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QUANTUM METROLOGY

STUDY OF ATOMIC STRUCTURE OF MATTER WITH X-RAYS

X-ray spectroscopy provides information on electronic structure and on the local atomic structure of atoms in matter. A synchrotron radiation beamline has been constructed by NIST scientists at the National Synchrotron Light Source, providing the highest flux, intensity, and energy-resolving power of any existing beamline in the X-ray energy range from 500 eV to 5000 eV. NIST equipment complements the synchrotron radiation instrumentation. X-ray absorption spectroscopy techniques, such as X-ray absorption near-edge structure and extended X-ray absorption fine structure, have been used to determine the atomic structure of metals, semiconductors, polymers, catalysts, biological molecules, and other materials of interest to industry. Researchers also use X-ray emission spectroscopy, X-ray photoelectron spectroscopy, and Auger electron spectroscopy to probe the electronic structure of solids, liquids, or gases.

The X-ray standing-wave technique uses interference between incident and diffracted X-rays to determine the precise location of impurities or imperfections within a crystal or at its interfaces. The technique can be used with semiconductors or optical crystals, growth of overlayers on crystals, and the structure of catalysts supported on crystal substrates. In addition, evanescent X-rays, which penetrate only a few nanometers from an interface, can be controlled to study chemical composition in the vicinity of an interface.

NIST scientists recently pioneered a new technique, diffraction of evanescent X-rays, which combines and extends the capabilities of the X-ray standing-wave method and experiments based on evanescent X-rays. The synchrotron radiation beamline provides an ideal facility for applying these techniques.

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IONIZING RADIATION

RADIATION PROCESSING

To enhance quality-control methods used in industrial radiation processing of foods and in the production and use of medical devices, electronic components, and polymers, NIST researchers are developing standardization and measurement assurance methods related to industrial high-dose applications of ionizing radiation. As part of this program, Institute scientists are investigating radiation chemical mechanisms and kinetic studies applied to chemical dosimetry systems in the condensed phase, including liquids, gels, thin films, and solid-state detectors.

They also plan to examine sensor materials, such as doped plastics, solid-state matrices, fiber optics, organic dye solutions, semiconductors, scintillators, amino acids, metalloporphyrins, and organic or inorganic radiochromic and luminescent aqueous solutions and gels. A number of analytical methods will be used, including transmission and fluorescence spectrophotometry, electron spin resonance spectrometry, and chemiluminescence spectrophotometry, as well as optical waveguide analysis, microcalorimetry, pulse radiolysis, laser-induced photochemistry, and conductivity measurements.

Various X-ray and gamma-ray sources and electron accelerators with energies in the 0.1-MeV to 10-MeV range are used in this work. Conventional ultraviolet, visible, and infrared spectrophotometers and spectrofluorimeters, high-intensity gamma-ray sources, pulsed and continuous beam electron accelerators, and organic-chemical analytical equipment are also available.

CONTACT: William L. McLaughlin
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Physicist Charles Dick aligns an X-ray detector for characterizing a multiple-focal-spot tomographic X-ray source. Such sources can be used to produce three-dimensional dental images that may soon improve selection of appropriate treatments for periodontal disease.

INDUSTRIAL RADIOLOGIC IMAGING

The use of penetrating radiation for imaging is one of the most powerful investigative techniques available to industry for maintaining or improving the quality of products. Designers, aware of this, are creating components that facilitate such non-destructive testing. Research is under way at NIST to allow better quantification of radiographic images. Particularly relevant to image evaluation are computer-based systems that permit pseudo three-dimensional images and the implementation of image processing on these or traditional images in real time or near real time.

NIST research focuses on image processing for improved imaging of low contrast for noisy images; adaption of tomographic equipment to industrial needs and measurement of the performance characteristics of such systems; and development of reliable techniques for image storage and retrieval. Available equipment includes X-ray sources, low-energy electron accelerators, gamma-ray sources, and state-of-the-art radiologic imaging devices.

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NEUTRON FLUENCE MEASUREMENT AND NEUTRON PHYSICS

NIST researchers are studying industrial applications of neutron fluence and dose determination in the neutron energy region from thermal to 20 MeV. They are developing effective methods to transfer personnel protection technology to the private sector. This research provides a basis for standardizing personnel protection control procedures in nuclear reactor and high-energy accelerator operations. Specific research involves the measurement of reference standard neutron reaction cross sections; characterization of reference fission deposits; development of neutron detectors with fast timing; and calibrations using standard neutron and gamma-ray fields. Equipment available includes a 100-kV ion generator-based 2.5-MeV neutron source, a 3-MV pulsed positive-ion accelerator, and a 20-MW nuclear reactor.

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TIME AND FREQUENCY

FREQUENCY, TIME, AND PHASE NOISE MEASUREMENT

Advancements in communication and navigation systems require atomic oscillators with increased performance and reliability. NIST has several programs aimed at providing advanced frequency standards with the potential for benefiting commercial atomic standards. NIST scientists currently are working on an optically pumped cesium-beam standard that should significantly surpass the performance of standards based on magnetic-state selection and detection. Their studies on ion storage and radiative cooling are exploring the potential for standards operating at accuracy levels of 1 part in 10^{15} and beyond.

Aerospace systems often require extreme phase stability, which has led to a need for high-quality phase noise characterization of amplifiers, frequency multipliers, oscillators, and other electronic components. NIST has initiated a program to develop methods for measuring phase noise in such components over a broad frequency range (into the millimeter range). The work will involve primarily the two-oscillator technique, but other techniques will be studied.

Requirements for synchronization (time) and syntonization (frequency) of broadly dispersed sets of nodes for communication, navigation, and other electronic systems are increasing significantly. Because of the inherent reliability, simplicity, and low cost of using satellite transfer, NIST is studying several possible approaches for using this technique. The NIST time scale and reliable ties to many other international timing centers provide the basis for performance analysis of these time-transfer techniques. In addition, the Institute is equipped with Global Positioning System receivers, Earth communication terminals, and automated systems for statistical analysis of system performance.

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QUANTUM PHYSICS

LASER STUDIES OF SEMICONDUCTOR MATERIALS

Optical/laser probing of the gas phase species involved in the molecular beam epitaxial (MBE) growth of III-V semiconductor materials is an area of high potential relevance for standards and technology. Work under way has demonstrated sensitive, direct laser detection methods for study of Ga and In atoms and arsenic species during GaAs growth. It will soon be possible to consider minor dopant species such as phosphorous and impurities, for example, carbon monoxide. With the incorporation of non-invasive laser probes into advanced generations of MBE machines, researchers will be able to carry out in-situ diagnostics to quantify and characterize the growth process, to provide optical feedback for adjustment of species concentrations, and to determine the purities of materials used during semiconductor fabrication.

In related experimentation, laser vaporization of thin films is used to produce sources of translationally energetic species for etching and deposition studies. Thermal chlorine molecules produce little or no etching of silicon materials, whereas etch rates increase dramatically with increasing kinetic energy, with an apparent threshold. New research will also explore the kinetic energy dependence of film growth by using velocity selected beams of refractory materials and insulators.

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LASER STABILIZATION

Many sensitive and sophisticated applications of lasers depend on the laser's spectral coherence, frequency stability, and low-intensity noise. NIST scientists have been working on laser intensity stabilization, laser frequency linewidth reduction with active control techniques, and several methods for producing quantitative laser frequency scans.

Two new systems have been developed. One, based on optical

Electrical engineer John Lowe adjusts a laser system for NIST-7, the latest generation in a series of extremely accurate atomic clocks developed at NIST.

sideband production by broadband microwave phase modulation of the laser, allows scans over a GHz range, with inaccuracy below 10 kHz. The other scan technique utilizes a novel interferometer/ phase-locked rf system. The system maps optical frequency change into the corresponding phase change of an rf signal suitable for control, stabilization, and scanning. Another NIST system under development works entirely externally to a continuous-wave laser to shift the output laser frequency and reduce the intensity in a controlled manner.

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SILICON THIN FILMS

Thin films of amorphous silicon are used in photosensitive devices, displays, and in photovoltaic cells. Scientists are examining physical and chemical mechanisms involved in discharge and thermal chemical vapor deposition (CVD) production of such films. A scanning tunneling microscope is used to examine in situ the morphology and chemical character of as-deposited films. All electronics, vacuum, and gas-handling apparatus necessary for producing and diagnosing discharge and thermal CVD films under controlled conditions are available.

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RESEARCH FACILITIES

SYNCHROTRON ULTRAVIOLET RADIATION FACILITY-II

The Synchrotron Ultraviolet Radiation Facility-II (SURF-II) is a 300-MeV electron storage ring that radiates synchrotron radiation which is highly collimated, nearly linearly polarized, and of calculable intensity. Seven beamlines are available, and a users' program is in operation. SURF-II is well-suited for studies in radiometry; atomic, molecular, biomolecular, and solid-state physics; surface and materials science; electro-optics; and surface chemistry and radiation effects on matter. Special facilities are available for developing and testing soft X-ray optics and optical devices.

CAPABILITIES: The typical storage ring electron beam current is 200 mA at 284 MeV. The photon intensity in the region 60 nm to 120 nm is about 3×10^{12} photons per second per milliradian of orbit for an instrumental resolution of 0.1 nm. Experiments can be conducted conveniently throughout the wavelength range 4 nm to 1000 nm, from the soft X-ray region to the infrared. A normal incidence, a grazing incidence, and several toroidal grating monochromators are available to disperse the radiation. A large, ultrahigh vacuum spectrometer calibration chamber, 1.2 m × 1.2 m × 2.5 m and accessible through a clean room, is available for radiometric applications in a clean, vacuum environment. A 6.65-m, normal-incidence vacuum spectrometer, with resolving power of about 300,000, is available on a beamline dedicated to high-resolution vacuum ultraviolet radiation research.

APPLICATIONS: The continuous radiation from SURF-II is used as a national standard of spectral irradiance for radiometric applications and for fundamental research in the following areas:
- atomic and molecular absorption spectroscopy;
- optical properties of materials;
- electron density of states in solids;
- surface characterization;
- photoelectron spectroscopy;
- molecular kinetics and excitation and ionization dynamics; and
- radiation interactions with matter (such as lithography, radiation damage, dosimetry, photobiology).

AVAILABILITY: Beam time on SURF-II is available to any qualified scientist if beamline vacuum requirements are met and scheduling arrangements can be made. Proposals should be submitted for NIST review before use of the facility is desired. Informal contact is also encouraged.

CONTACT: Robert P. Madden
B119 Radiation Physics
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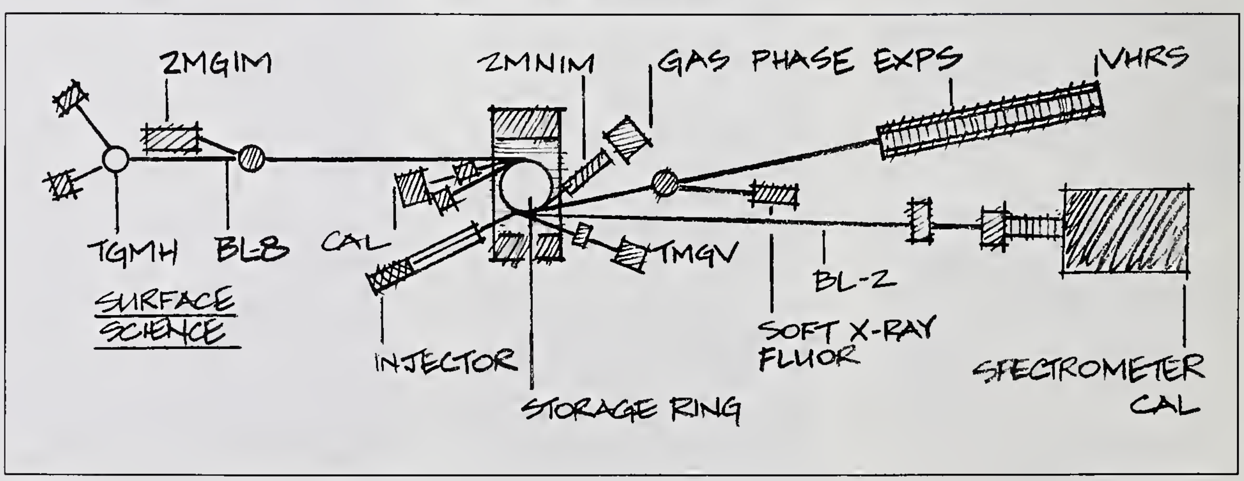
LOW-BACKGROUND INFRARED RADIATION FACILITY

In the Low-Background Infrared Radiation Facility (LBIR), radiant background noise levels less than a few nanowatts are attained in a large (60-cm-diameter × 152-cm-long) vacuum chamber by cooling internal cryoshields to temperatures less than 20 K using a closed-cycle helium refrigerator system. An absolute cryogenic radiometer (ACR) of the electrical substitution type that operates at 2 K to 4 K is housed in the chamber.

CAPABILITIES: The ACR is a broadband detector with a flat response from the visible to the long wavelength infrared spectral

A schematic of the Synchrotron Ultraviolet Radiation Facility-II.



region. It can measure power levels of 20 nw to 100 μw at its 3-cm-diameter aperture within an uncertainty of less than 1 percent. The ACR has a resolution of 1 nanowatt, and its time constant is about 20 s.

APPLICATIONS: This unique facility can be used to measure total radiant power from sources such as cryogenic blackbodies. Ongoing improvements will allow measurement of the spectral distribution of radiation from sources and characterization of infrared detectors and optical components.

AVAILABILITY: The facility is operated by NIST staff in support of user infrared calibrations. It is available for collaborative research by NIST and outside scientists in areas of mutual interest.

CONTACT: Raju Datla
A221 Physics
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ELECTRON PARAMAGNETIC RESONANCE FACILITY

NIST is leading a national and international effort in electron paramagnetic resonance (EPR) dosimetry for measuring ionizing radiation. Paramagnetic centers (molecules/atoms with unpaired electrons) are produced by the action of radiation on materials. In the EPR experiment, irradiated materials are placed in a magnetic field and electron spin transitions are induced by an electromagnetic field of the appropriate frequency (typically GHz). EPR is used as a nondestructive probe of the structure and concentration of paramagnetic centers. The centers created by ionizing radiation are proportional to the absorbed dose and provide a sensitive and versatile measurement method.

Top right. Physical scientist Jacqueline Calhoun makes an activity measurement of a gamma-ray-emitting sample by placing it in the NIST "4π"γ Ionization Chamber." Bottom right. Precise measurements of absorbed radiation dose for industrial and medical applications can be made at the NIST Electron Paramagnetic Resonance Facility. Here, physical science trainee Francoise Le inserts a bone fragment into the sample chamber.

CAPABILITIES: The EPR dosimetry facility is supported by a state-of-the-art X-band EPR spectrometer capable of measuring radiation effects on a wide range of materials from inorganic semiconductors to biological tissues. The recently upgraded data acquisition system provides full computer control of all spectrometer functions, including real-time spectral display and rapid acquisition scan to analyze rapidly decaying signals. The data acquisition system is interfaced with an advanced data analysis station for data manipulation and is capable of simulating and deconvoluting multicomponent spectra.

APPLICATIONS: EPR dosimetry is operable over several orders of magnitude in absorbed dose (10^{-2} Gy – 10^{5} Gy) and impacts many facets of society and industry. Areas of impact include:

- Radiation Protection/Accident Dosimetry. Using biological tissues (bone, tooth enamel) or inanimate materials (clothing), retrospective dose assessment and mapping can be accomplished.
- Clinical Radiology. Ionizing radiation doses administered in cancer therapy can be measured for external beam therapy using dosimeters of crystalline alanine (an amino acid) or validated for internally delivered bone-seeking radiopharmaceuticals using bone biopsies.
- Industrial Radiation Processing. Routine and transfer dosimetry for industrial radiation facilities can be performed using alanine dosimeters, as well as post-irradiation monitoring of radiation-processed meats, shellfish, and fruits using bone, shell, or seed.

The EPR facility also serves as a fully functional materials research facility for analyzing radiation effects on semiconductors, optical fibers, functional polymers, and composites.

AVAILABILITY: The EPR facility is available for collaborative research by researchers from industry, academia, and other government agencies under the supervision of NIST staff.

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RADIOPHARMACEUTICAL STANDARDIZATION LABORATORY

Radioactivity measurements for diagnostic and therapeutic nuclear medicine in the United States are based on measurements at NIST. Activity measurements for the gamma-ray-emitting radionuclides are made using the NIST "4π" γ ionization chamber. The calibration process also includes identification of radionuclidic impurities by germanium spectrometry. Recent development work has focused on therapeutic nuclides for nuclear medicine, radioimmunotherapy, and bone palliation.

CAPABILITIES: The radiopharmaceutical standardization laboratory provides calibration service for the gamma-ray-emitting radionuclides and is available for technical users who must make measurements consistent with national standards or who require higher accuracy calibrations than are available with commercial standards.

NIST also undertakes basic research to develop new methods of standardizing radionuclides for diagnostic and therapeutic applications. These studies include measurements of decay-scheme parameters, such as half lives and gamma-ray emission probabilities, and identification of radionuclidic impurities.

AVAILABILITY: The customer has no direct use of the facility. NIST staff can provide calibration services for the gamma-ray-emitting radionuclides that comply with the specifications as stated in NIST Special Publication 250-10. Research associates of the United States Council for Energy Awareness distribute radiopharmaceutical Standard Reference Materials.

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MAGNETIC MICROSTRUCTURE MEASUREMENT FACILITY

The magnetic microstructure of materials can be measured with very high spatial resolution by a technique called scanning electron microscopy with polarization analysis (SEMPA). An ultrahigh-vacuum electron microscope has been modified so secondary electrons from the sample can be analyzed for their electron spin polarization. This allows for a measurement of the surface magnetism with moments both in the plane and perpendicular to the plane of the sample.

CAPABILITIES: SEMPA allows the simultaneous observation of surface microstructure and surface magnetic domains at a resolution of 0.05 μm, using an innovative, extremely compact electron spin detector invented at NIST.

APPLICATIONS: This unique measurement facility can be used for research in magnetic thin films, high-coercivity magnetic materials, high-density magnetic storage media, and other advanced magnetic materials.

AVAILABILITY: These facilities are available for collaborative research by NIST and outside scientists in areas of mutual interest on a time-available basis.

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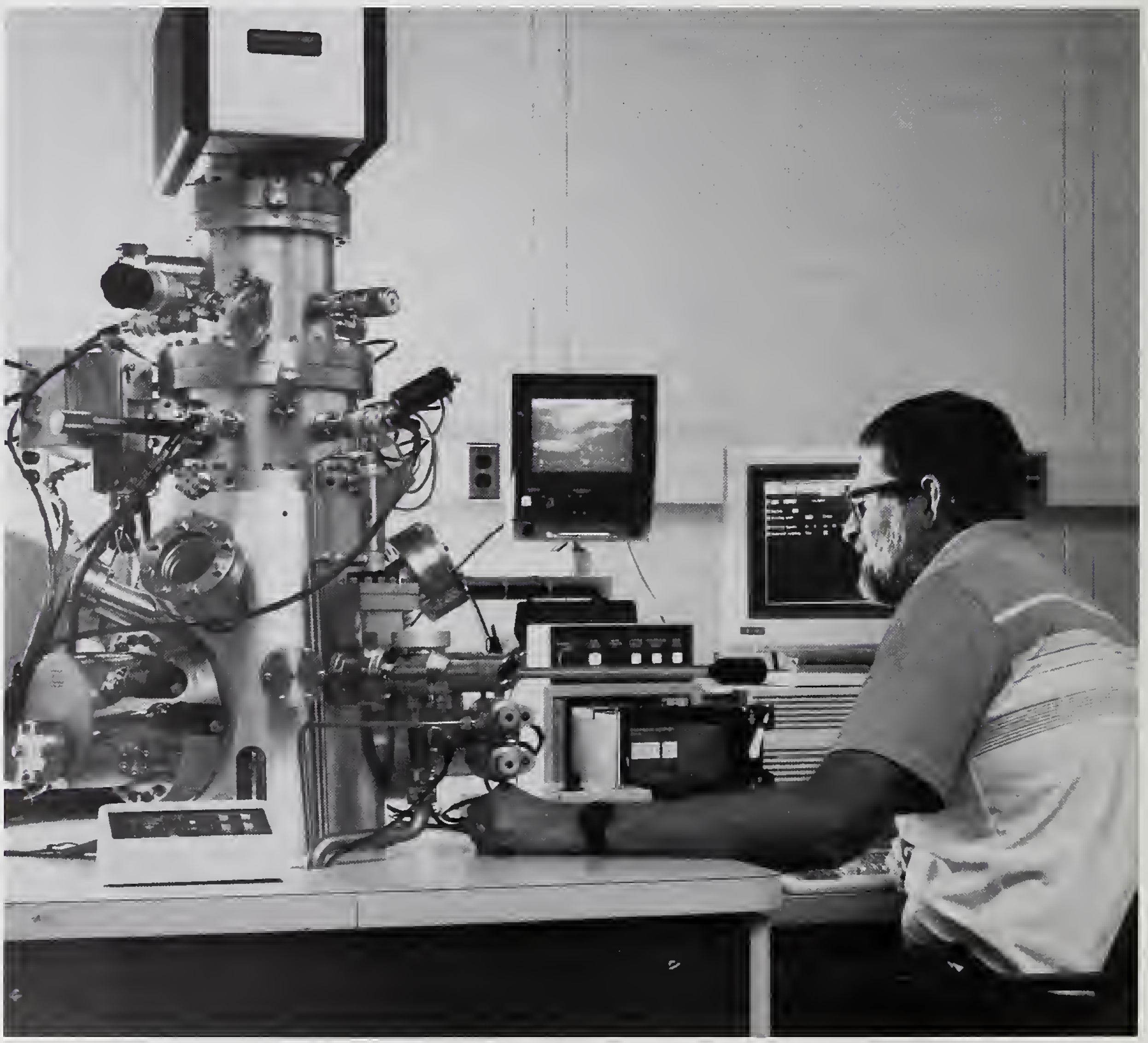
Using a scanning electron microscope with polarization analysis, physicist Mike Kelley examines magnetic microstructures of magnetic materials such as the recording media used in computer disks.



MATERIALS SCIENCE AND ENGINEERING LABORATORY

Covering all classes of advanced materials, as well as conventional structural materials, the laboratory's research programs address the full range of issues in materials science and engineering—those related to design, synthesis, processing, performance, instrumentation, and analysis and modeling. A unifying aim is to acquire the knowledge and tools needed for intelligent manufacturing methods with real-time automated process controls.

Separate research initiatives address ceramics, metals, polymers, composites, and superconductors. This research supports U.S. industry efforts to develop reliable, low-cost manufacturing methods for producing tailor-made materials and products with superior properties.

Through laboratory-organized consortia and one-to-one collaborations, NIST's materials scientists and engineers work closely with their counterparts from U.S. industry. For example, one consortium is developing a computer-controlled system for intelligently processing rapidly solidified metal powders. The research involves development of an integrated system to control particle size in real time. Other collaborations are developing non-destructive evaluation sensors for aluminum and steel processing.

The Materials Science and Engineering Laboratory is also strengthening its research relationships with manufacturers of high-technology products, the major users of advanced materials. On the basis of discussions with U.S. microelectronics firms, it has launched new programs to improve materials for semiconductor packaging.

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COOPERATIVE RESEARCH OPPORTUNITIES

INTELLIGENT PROCESSING OF MATERIALS

INTELLIGENT SENSORS, CONTROLS, AND PROCESS MODELS

Advanced materials are capable of providing outstanding properties, but they generally require unusual processing operations and tend to be expensive. Intelligent processing offers the potential to design and produce materials with substantially improved quality, reduced lead time, and increased flexibility in production.

Intelligent processing incorporates four principal elements including materials processors, non-destructive evaluation sensors that can measure physical and mechanical characteristics of materials in real time during processing, rigorous models to describe the materials production process, and expert systems to control the production process in real time through integration of sensors and process models.

Research sponsored by the Office of Intelligent Processing of Materials is directed to process models, sensors, and intelligent control systems. Research topics include processing modeling, sensing and control of the production of metal powders by high-pressure gas atomization, measurement and control of steel sheet texture, magnetic methods for measuring

An ultrasonic sensor developed by physicist Ray Schramm can be embedded in train tracks to detect cracks in railroad wheels as a train rolls by.

mechanical properties, ultrasonic measurement of interfaces, powder-particle-size sensing, inspection of electronic components, X-ray radioscopy standards, fluorescence spectroscopy in polymers processing, magnetic resonance imaging and ESA for ceramic processing, eddy-current sensing in metals and composites, and laser ultrasonic methods.

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CERAMICS

CERAMIC PROCESSING

Sintering of ceramics is a complex process that involves the interaction of many different processing variables. The influence of these processing variables on sintering cannot be determined simply by measuring final density or some other end-point property. Chemical composition of the ceramic powders is known to be of major importance and, under certain conditions, can mask effects of most other processing variables. NIST scientists are investigating the effect of trace levels of impurities using clean-room processing to produce, compact, and sinter ultrahigh purity ceramics.

Results from this research will enhance investigations of other processing variables, including particle size, shape, agglomeration, compaction method, and atmosphere under controlled composition conditions. The data can be used to design better models for microstructure evolution during sintering. Use of predictive models, in conjunction with other ongoing efforts to produce unique compositions and phases, can lead to new advanced ceramic materials with unique microstructures and properties.

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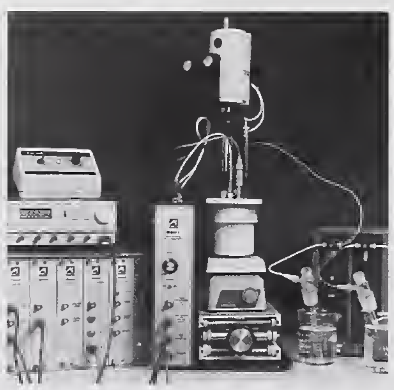

TRIBOLOGY OF CERAMICS

Researchers in the NIST ceramics tribology program are investigating the fundamental mechanisms of friction and wear of advanced materials and lubricants and are developing test methods for evaluating the performance of these materials. The program's primary focus is on the characterization of the interfacial phenomena that occur when two solid surfaces interact. Research activities include analysis of chemical reactions and formation of tribochemical films, physical and mechanical behavior of surface films, and deformation and fracture processes leading to wear and failure. Tribology laboratories consist of state-of-the-art tribometers, such as high-temperature (1000 °C) wear testers, and analytical instruments, such as time-resolved micro-Raman laser systems, which can be used for cooperative research with industry and other federal agencies.

NIST researchers are compiling data in the form of "wear maps" and computerized data, and are studying the mechanisms for wear of structural ceramics at elevated temperatures, self-lubricating metal-matrix and ceramic-matrix composites, and wear-resistant coatings.

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Top left. Research chemist Pu Sen Wang uses a nuclear magnetic resonance instrument to determine the distribution of water and other chemicals in a silicon nitride slurry, a ceramic material used to form advanced automotive engine components. Top right. This electrokinetic sonic amplitude instrument has been adapted by NIST scientists to measure the homogeneity of ceramic slurries. Bottom. Materials research engineer Mark Austin uses an X-ray diffraction apparatus at cryogenic temperatures to study phase transitions in a high-temperature superconductor.

ELECTRO-OPTIC CRYSTALS

Imperfections in highly perfect crystals typically limit their performance in high-technology applications, such as optical communications and optical signal processing. In particular, limitations in the perfection of electro-optic and photorefractive materials, such as lithium niobate and bismuth silicon oxide, have inhibited the development of optical switches and modulators.

NIST researchers are investigating crystal perfection at a unique, monochromatic X-ray topography facility at the NIST/Naval Research Laboratory beamlines on the high-energy ring at Brookhaven National Laboratory's National Synchrotron Light Source in New York. Conducted jointly with growers of high-quality crystals, current studies are expected to improve substantially the quality, and hence the performance, of these crystals.

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MECHANICAL PROPERTIES OF CERAMICS

Several long-term programs are being conducted at NIST on the fracture, creep, and creep rupture of monolithic and composite ceramics. Researchers, for example, are examining the effect of very near surface forces on the fracture properties of monolithic ceramics. These forces have been measured for mica, sapphire, and fused silica surfaces, using a crossed cylinders apparatus.

A second program uses an indentation strength procedure to determine the fracture toughness (R-curve) behavior of monolithic alumina ceramics and to relate the R-curve to the specific microstructure of the material. In-situ microscopic observations of crack growth demonstrate the presence of grain bridging and frictional sliding in the crack wake as a significant source of material toughening.

A program in composites is aimed at determining the effects of the fiber/matrix interface on fracture behavior of these materials. An instrumented micro-indenter is available to measure the fiber/matrix interface strengths. Researchers also are investigating the stresses developed in composites during conventional sintering processes to determine methods of reducing these stresses through the use of fiber coatings—thus minimizing or eliminating cracking of the matrix as densification proceeds.

High-temperature deformation and fracture behavior of ceramics are other areas of research. Creep and creep rupture of several varieties of siliconized SiC have been investigated and their behavior related to differences in microstructure, particularly grain size. Research continues on whisker-reinforced Si_3N_4 and Al_2O_3, SiC, and other high-temperature materials. Facilities exist for performing creep measurements in tension, compression, and flexure at temperatures up to 1800 °C. The tensile creep apparatus is almost completely automated, and measurements are made using a laser-imaging technique. Displacement measurements are accurate to $\pm 1\ \mu m$ at 1500 °C.

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CERAMIC PHASE EQUILIBRIA

Ceramic phase equilibria studies at NIST involve complementary research activities in experimental, theoretical, data evaluation, and compilation aspects of ceramic phase equilibria. The data-evaluation and compilation work is carried out under the joint American Ceramic Society/NIST program to provide industry and others with a comprehensive database of up-to-date, critically evaluated phase-diagram information. Ceramic phase diagrams are also being determined experimentally for systems of technical importance, such as high-transition-temperature ceramic superconductors.

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CERAMIC CHEMISTRY

NIST scientists are investigating low-temperature synthetic approaches to both oxide and non-oxide ceramic powders and coatings. They are using novel chemistry and techniques to solve the problems of generating homogeneous, multicomponent materials with predictable and optimal properties. The researchers are studying the following materials: high T_c ceramic superconductors; ultrafine ceramic powders with high-electrical conductivity; ceramic coatings with selected optical, electronic, or structural properties; ultrafine magnetic composites; ceramic whiskers-ceramic matrix composites; and ultrafine, pure oxide powders. Depending upon requirements of the study and the powder, synthesis is carried out either in small bulk reactors or in a flow reactor.

The researchers are examining the relationship of process conditions to surface chemistry of the powder, particle size and shape, the phases formed during thermal treatment of the powder, and properties of the powder or sintered ceramic, such as electrical conductivity. Coupled with the powder synthesis is the development of characterization methods involving nuclear magnetic resonance spectroscopy.

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MATERIALS RELIABILITY

NON-DESTRUCTIVE EVALUATION

NIST scientists are developing acoustic and electromagnetic sensors for material characterization, including electromagnetic-acoustic transducers (EMATs), arrays of piezoelectric polymers, and eddy-current and capacitive probes.

EMATs are used for non-contacting applications, where it is not possible to have an intervening material couple sound from transducer to specimen. For example, an EMAT embedded in a rail is used to inspect railroad wheels for cracks in a roll-by mode. Researchers are investigating the possible use of EMATs at the input to an automated press shop, where automobile body parts are formed. Formability measurements could be made on moving sheet, and materials with improper formability would be rejected from the production line.

Acoustic arrays are useful in medical imaging, where their unique advantages allow suppression of artifacts to give better image quality. Research is being conducted by NIST to use these techniques to characterize composites. Various types of arrays are being constructed and interfaced with signal processing devices.

NIST scientists have developed eddy-current probes and techniques for detecting and sizing small defects that could prove to be critical in aircraft and other structures. Capacitive probes are being investigated as a non-contacting means to monitor sintering of ceramics, with possible use for quality control of substrates for semiconductor devices.

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THERMOMECHANICAL PROCESSING

Metals producers and manufacturers rely heavily on pilot-scale or production facilities for the development of new alloys or new manufacturing schedules. This is not only expensive but can also yield unreliable results. These processing problems can be alleviated by using a more economical, versatile, and reproducible laboratory facility. Controlled thermomechanical processing offers the opportunity to produce superior steels at lower cost due to the elimination of post-heat treatment—an energy-intensive process.

NIST scientists and engineers have designed and built a computerized, laboratory-scale, hot-deformation apparatus that can simulate manufacturing processes, such as forging and plate rolling, and measure important properties incurred during processing. The apparatus can be used to study static and dynamic recrystallization, high-temperature deformation resistance in terms of a true stress-strain curve at high-strain rates, and phase transformation characteristics. The apparatus is used to characterize directly cooled forging steel during simulated forging operations in an attempt to develop optimized steels and forging procedures.

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CRYOGENIC MATERIALS

Comprehensive low-temperature facilities are available to conduct both characterization testing and fundamental studies on the mechanical and physical properties of high-strength structural alloys, high-conductivity metals and superconductors, metal and polymer-based composites, and polymer foams.

Properties studied include tensile strength, compression, fatigue, creep, fracture toughness, stress relaxation, elasticity (ultrasonic), and thermal expansion. Strain sensitivities of 10^{-7} at liquid helium temperature (4 K) are now possible, permitting precise low-temperature microstrain measurements. Researchers are assembling equipment to permit load capacities of 5 MN for testing at 4.2 K.

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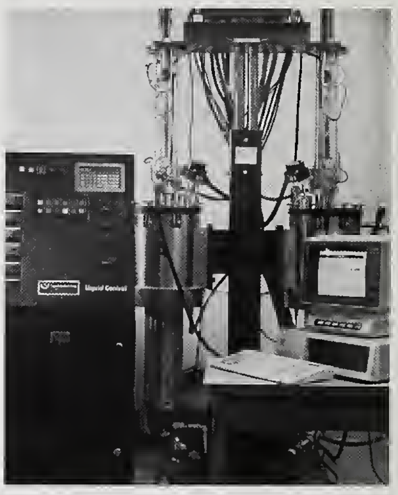
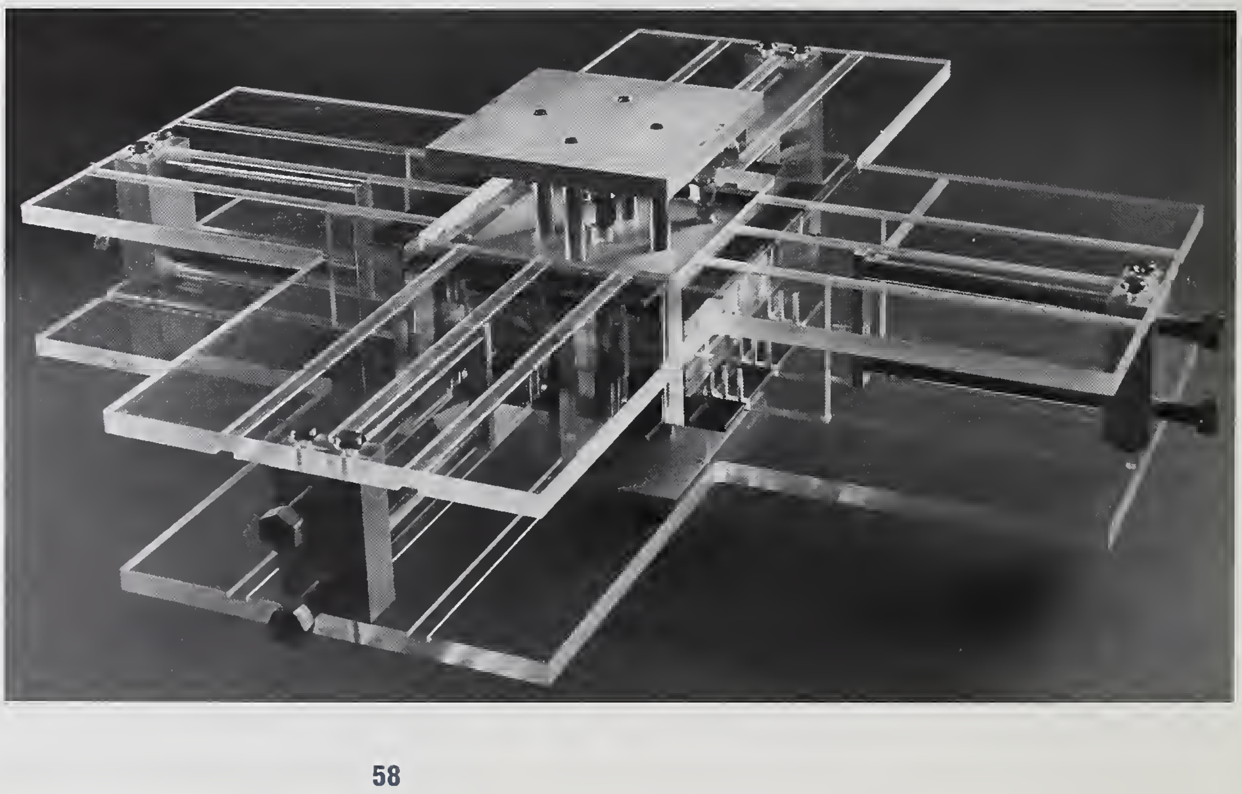
WELDING QUALITY RESEARCH

The integrity of welded joints is a primary concern in the design and fabrication of engineering structures. Researchers at NIST are investigating ways to improve the fracture resistance of weldments and are assessing weld quality by non-destructive evaluation (NDE) techniques. Specific goals of the research are to improve fracture toughness in the heat-affected zone of steel weldments and in the weld metal of materials for low-temperature service and to im-





Above right. A computer model created by NIST materials scientists describes in three-dimensions the flow of a polymer into a mold containing reinforcing fibers. Right. Improved understanding of flow properties in molds like this one will help manufacturers produce strong, lightweight automobile frames more efficiently.

prove the soundness of welds through increased understanding of the metal transfer process in arc welding. In addition, the researchers are applying fine-resolution NDE techniques to evaluate solder joints in printed circuit boards for the electronics industry. NIST scientists have developed techniques to reduce porosity in aluminum weldments, to reduce spatter in gas metal arc welding of steels, and to predict accurately the ferrite number in stainless steel welds.

The NIST welding and NDE laboratories house equipment for shielded metal arc, gas metal arc, and gas tungsten arc welding; radiographic and penetrant techniques for non-destructive evaluation of weldments; and a precision power supply, high-speed photography, and laser-shadow techniques for metal-transfer studies.

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POLYMERS

PROCESSING SCIENCE FOR POLYMER COMPOSITES

Fiber-reinforced composites offer, along with other advantages, versatility in processing combined with high strength and stiffness at low weight. For the current growth in the production of these materials to continue, however, more rapid and reliable processing is needed.

NIST researchers are developing new measurement tools to characterize the starting materials for composite processing and to study in the laboratory the chemical and physical changes that occur during processing. The scientific understanding that can be generated with these tools will facilitate both advances in processing methods and implementation of on-line control and automation.

The infiltration of a resin into a fiber preform during processing is the critical step in composite fabrication by liquid molding. Test methods to characterize the resistance of the preform to resin infiltration have been developed. NIST is working with several companies including the Automotive Composites Consortium (Ford, General Motors, and Chrysler) to characterize this resistance in a variety of materials and use the results in computer simulation models to optimize the liquid molding process.

In addition, NIST is developing measurement techniques to monitor the chemical, morphological, and molecular changes that occur during processing. The program currently has 10 different spectroscopic, dielectric, thermal, and mechanical techniques available for process monitoring, and others are under development. This diversity of tools provides a unique capability for evaluating and calibrating new measurement methods, for developing process models, and for analyzing model resin systems.

For example, through cooperative projects with NIST, several industries have selected and developed monitoring methods for their particular problems.

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POLYMER STRUCTURE

Knowledge of the relationships between mechanical properties of polymers and polymer structure is important to the design and processing of materials for optimal performance. Researchers at NIST are studying semicrystalline polymers, polymer glasses, elastomers, molecular composites, and fibers to develop improved models of mechanical behavior, characterize structure from the atomic- to fine-texture level, and elucidate relationships between mechanical performance and structure.

NIST scientists use a variety of techniques to characterize the structure of polymers in the solid state. Nuclear magnetic resonance spectroscopy is used to determine molecular orientation, molecular dynamics, and microstructure on the 1-nm to 10-nm scale. Microstructural information is deduced from C-13 lineshapes obtained with magic angle spinning or by proton "spin diffusion" experiments in which domain-size information is inferred from the rates at which proton magnetization diffuses in the presence of magnetization gradients. Fourier transform infrared spectroscopy helps to determine molecular architecture, orientation, and molecular processes, such as measurement of the amount of chain scission associated with mechanical deformation of polymers. Microstructural features, including spherulytic morphologies, lamellar texture, fiber structures, and crystallization habits, are elucidated through optical and electron microscopic studies of polymers.

NIST scientists are examining the relationships between mechanical performance of polymers and fine structures by investigating the morphological changes that polymers undergo when they are deformed. Wide- and small-angle X-ray diffraction techniques, which include the use of position-sensitive detectors, are also used in these investigations.

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DENTAL AND MEDICAL MATERIALS

Modern dental and medical materials utilize numerous substances in an array of combinations. NIST has a comprehensive program aimed at increasing basic understanding of the causes for failure or poor performance of these materials, proposing and testing new material systems, and transferring the resultant science and technology to industry. The program has the active participation of researchers from the American Dental Association, the National Institute of Dental Research, dental industries, and universities.

Researchers are working in a number of areas. For example, in a tribology study, scientists are examining wear and degradation of materials in various environments. In addition, improved resins are being synthesized to produce polymers that have improved properties, such as wear resistance, higher strength, resistance to oral fluids, or X-ray opacity. Researchers have developed an in-mouth shield for cancer patients undergoing radiation therapy to protect healthy tissues from secondary radiation emitted from metallic restorations.

In an effort to improve the strength of dental systems, different combinations of materials are being designed and tested, including resin-matrix composites and ceramic-metal, metal-cement, cement-tooth, and composite-adhesive-tooth materials. New monomers are being synthesized, which are designed to reduce polymerization shrinkage and/or improve the degree of wear. Unique methods for determining the effectiveness of coupling agents and curing efficiency are being explored. Weibull statistical analysis is employed to identify the weakest links, and finite-element analysis is applied to define stress states within systems.

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ELECTRICAL AND OPTICAL PROPERTIES

NIST is conducting a number of studies on the electrical properties of polymers. The research focuses on dielectric measurements, fundamentals and applications of piezoelectric and pyroelectric polymers, measurement of space charge distribution within polymer films, and electro-optic properties of polymers.

Scientists have developed instrumentation and data analysis techniques to measure the dielectric constant and loss of polymer films over a frequency range of 10^{-3} Hz to 10^{5} Hz in less than 30 minutes. These developments make it possible to follow changes in the dielectric spectrum as a function of time, processing conditions, or other parameters.

The toughness, flexibility, low-dielectric constant, and an acoustic impedance close to water make piezoelectric polymers ideal for many transducer applications. NIST researchers have considerable experience with the fundamental properties of polymers such as polyvinylidene fluoride and its copolymers and can work with industry to develop transducers for novel applications or to assist in measuring the properties of new piezoelectric polymers or composites.

The scientists also have designed instrumentation and data analysis techniques to measure the charge or polarization distribution across the thickness of a polymer film by analyzing the transient charge response following a pulse of energy on one surface of the film. This technique has been used to detect the presence of non-uniform electric fields in the poling of piezoelectric polymers and currently is being used to investigate the role of space charge in the dielectric breakdown of polymer insulation.

Organic molecules with highly delocalized π-conjugated electrons offer great potential in the field of non-linear optics due to their relatively large second-order susceptibilities and their picosecond response times. The orientation and stability of orientation of polar groups in polymers containing these molecules are being investigated by measurements of dielectric properties and polarization distributions.

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MOLECULAR NETWORKS

Thermoset resins are used extensively in practical applications, especially polymer-based composites. However, knowledge of the basic structural entity of these resins, for example, their molecular network structure, is very limited. NIST researchers have developed a neutron-scattering technique that can be used to determine quantitatively characteristics of the network, including the average distance between crosslinks, the rigidity of the network, the molecular weight distribution between crosslinks, and the topological heterogeneity of the network structure.

Current NIST research focuses on developing correlations among chemical compositions, processing conditions, and resulting network structure for epoxy-type polymers. To establish processing-structure-property relationships, these materials are then evaluated to determine performance properties, such as fracture behavior.

The neutron-scattering technique also is being used to study how the molecular network deforms when the material is placed under load. These studies help industry by providing guidelines for designing and processing polymers for optimal performance.

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POLYMER BLENDS

Blending polymers has become an effective method for producing high-performance engineering polymers. The fundamental data required to design a manufacturing process include the equilibrium phase diagram, the energetic interaction parameter (compatibility) between the blend components, diffusion coefficients, and the interfacial tension. At NIST, small-angle neutron scattering has been used to measure the interaction parameter and phase diagram of polymer blends. NIST scientists have developed forced Rayleigh scattering and temperature-jump light-scattering techniques to measure polymer-polymer diffusion and other parameters that control phase separation kinetics and morphology.

Currently, researchers are testing various kinetic theories of phase separation and late-stage coarsening and are studying the control of morphology and mechanical properties of microphase-separated polymer blends. As part of this program, they are working with industry to study the homogenization and phase coarsening of rubber blends.

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FLUORESCENCE MONITORING

NIST researchers are using fluorescence spectroscopy to characterize structural and dynamic properties of polymers and to monitor the behavior of polymer flows during processing.

Fluorescence quenching is employed, for example, to monitor the uniformity of mixing in polymer blends and in particle-filled polymer melts. Excimer fluorescence is used to monitor the cure of thermoset resins, to detect crystallization and glass transition temperatures, and to measure the temperature of polymers during processing. Fluorescence anisotropy is used to measure molecular orientation and to monitor the non-Newtonian flows of polymer solutions and melts.

For polymer processing and composites monitoring, optical fibers are used to transmit fluorescence and excitation light energy between the processed polymer and light source and detection equipment. Well-equipped laboratories containing laser light sources, spectrofluorimeters and a nanosecond spectrofluorimeter are available for carrying out this research.

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METALLURGY

METALLURGICAL PROCESS CONTROL SENSORS

Special facilities at NIST enable researchers to develop advanced measurement methods and standards for application in process modeling and control for intelligent processing of materials. Measurement methods available include ultrasound, eddy currents, and acoustic emission. In particular, non-contact ultrasonic facilities featuring high-intensity pulsed lasers and electromagnetic acoustic transducers have been designed.

Coupled with state-of-the-art materials-processing equipment and expertise, these facilities offer a unique opportunity to ascertain feasibility and develop prototype specifications for a wide spectrum of sensor needs, including the measurement of internal temperature, phase transformations, surface-modified layers, porosity, grain size, and inclusion/segregate distributions.

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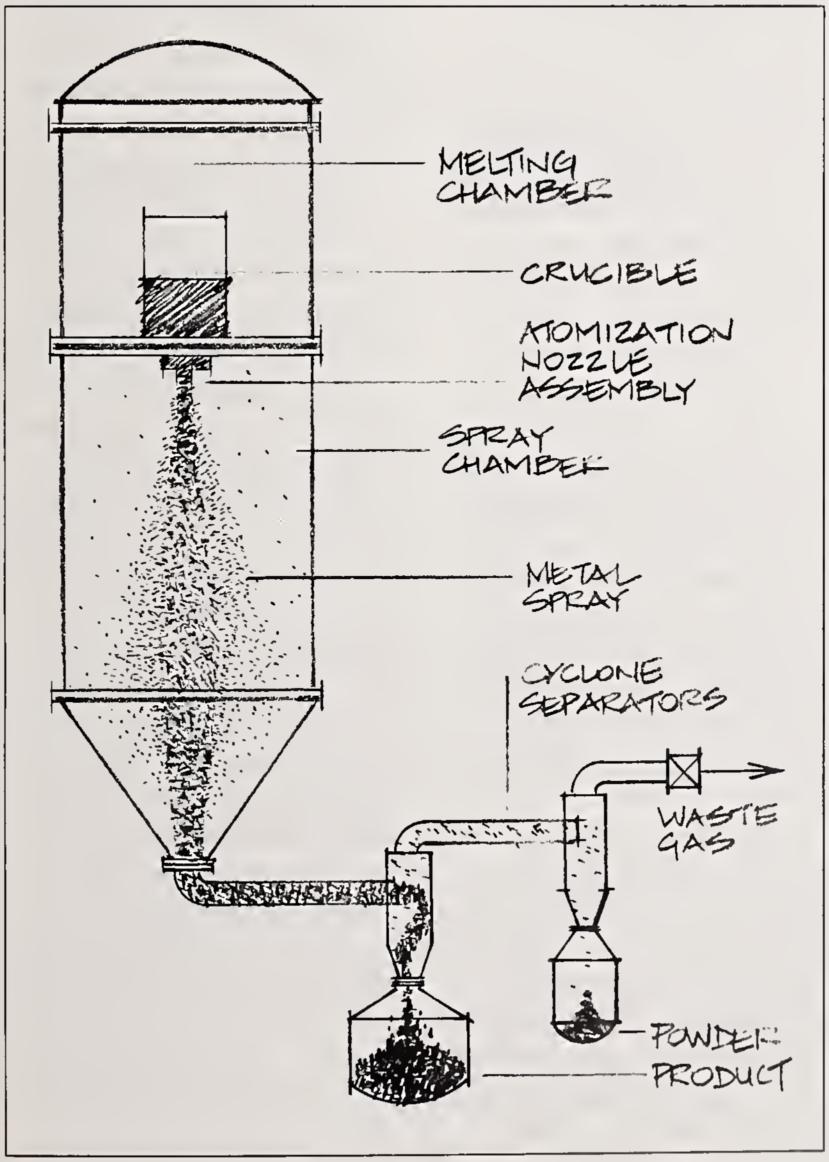

Schematic of the Supersonic Gas Metal Atomizer.

CORROSION DATA PROGRAM

Corrosion is a major limiting factor in the service life and cost of many products, plants, and equipment. The costs of metallic corrosion to the U.S. economy are estimated to be $218 billion in 1989 dollars, $33 billion of which could be saved by using existing technology.

The National Association of Corrosion Engineers (NACE) and NIST have joined together in a collaborative NACE/NIST Corrosion Data Program to reduce the enormous costs of corrosion. The focus of this program is to establish an evaluated corrosion database that can be accessed easily by computer using intelligent interfaces to obtain data in a number of graphical, tabular, or textual formats.

The researchers are developing evaluation methodologies and a prototype database for areas covering thermodynamic and kinetic corrosion data. NIST scientists are emphasizing interactions with industry both to help set priorities for data projects and as a source of corrosion data for the database.

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CORROSION MEASUREMENTS

Electrochemical measurements can give important information about the chemical processes causing the corrosion and the rate of corrosion as well as its forms, whether uniform or localized. Researchers at NIST are determining current and potential and their variations with time, measuring the frequency spectrum of the ac impedance of corroding electrodes, and detecting and analyzing random fluctuations of electrical parameters.

Electrochemical methods for measuring corrosion rates offer the possibility of tracking the process in a non-destructive way and assessing the effect of various environmental variables. Other electrochemical techniques can shed light on the kinetics of the corrosion reactions and on the critical factors that may contribute either to catastrophic failures or corrosion prevention.

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FIELD CORROSION OF METALS

The corrosion of metals in natural and manmade environments is very costly to industry and government. For several years, NIST researchers have been involved in developing methods for measuring the corrosion of metals in field conditions, such as steel piling in soil and seawater, electric utility lines in soil, and most recently, steel in concrete.

To measure the corrosion of reinforcing steel in concrete bridge decks, NIST scientists have developed a small, portable computer system. With this system, scientists are able to use electrochemical techniques normally limited to the laboratory to measure the corrosion. This new approach, which allows faster, more accurate corrosion measurements, can be used to evaluate the effectiveness of protection systems in place. The system has been applied to several bridges in Maryland and Washington state.

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STEEL SLAG THERMOCHEMISTRY

Thermochemical databases and models are needed by the U.S. steel industry for the design of new or improved steel-making processes. To design such processes, NIST researchers are involved in three programs: measuring refractory and slag thermochemical equilibria; evaluating the scattered literature data; and developing computer models that relate experimental data to industrial conditions. Unique experimental facilities are available at NIST for measuring key phase equilibria and kinetics.

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ENVIRONMENTALLY INDUCED FRACTURE OF ADVANCED MATERIALS

Corrosive reactions between the environment and advanced materials can dramatically alter the fracture resistance of the material. To assist in the development of advanced materials, NIST scientists are studying the mechanisms by which environments can induce crack propagation at stresses well below those which cause normal fracture.

Researchers are developing experimental techniques to evaluate and quantify the susceptibility of different types of advanced materials. Experiments are being conducted to investigate the mechanisms of hydrogen absorption, dissolution, and environmentally induced cleavage on different types of materials, specifically low-density, high-strength Al-Li alloys; intermetallic compounds; and composites as well as simple model systems.

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MAGNETIC MATERIALS

As part of the NIST program in magnetic materials, researchers are studying the magnetic properties of alloys and their relationship to metallurgical structure. Nanocomposites, composition-modulated alloys, high-temperature superconductors, metallic glasses, and icosahedral crystals are among the materials being investigated. The magnetic properties are characterized by ac and dc (VSM and SQUID) magnetic susceptibility measurements, magnetocaloric properties, Barkhausen noise, magneto-optic Kerr effect, ferromagnetic resonance and Mössbauer effect observations. Applications are made to nondestructive evaluation.

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VAPOR-DEPOSITED THIN FILMS

Obtaining new fundamental understanding of the detailed physics and chemistry of thin-film formation from high-temperature vapor sources is an objective of NIST researchers. This effort is currently developing a laser-produced vapor deposition facility, with measurement access for a variety of existing vapor diagnostics facilities.

These in-situ facilities include high-pressure sampling molecular beam mass spectrometry, optical multichannel emission spectroscopy, laser-induced fluorescence spectroscopy, and optogalvanic effect spectroscopy. These diagnostic techniques provide time-resolved, species-specific gas-phase compositions during the gas transport process.

In addition, film characterization facilities, including magneto-optical Kerr effect, SEM and TEM-based X-ray analysis, and Auger/ ESCA are available. Current plans are to extend measurements on film-producing environments to conventional sputtering conditions as well. Films of current interest include electronic, magnetic, and high T_c materials.

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REACTOR RADIATION

NEUTRON NON-DESTRUCTIVE EVALUATION

Neutron radiography has been a well-established tool for non-destructive evaluation (NDE) for some time. More recent neutron applications in NDE include neutron diffraction for texture and residual stress determination. All three of these specialties are available for cooperative research and development at the NIST reactor.

In general, these techniques parallel their X-ray counterparts; however, in certain cases (such as where hydrogenous components are critical, or subsurface texture or residual stress distributions are sought) X-rays do not provide the needed sensitivity or penetration.

NIST scientists are collaborating with scientists from the Smithsonian Institution to continue the examination of art works by neutron autoradiography. The technique has also been applied to industry-related problems, and efforts are being made to extend its capabilities to, for example, cold neutron radiography. NIST and Department of Defense (DOD) scientists are continuing a cooperative development and application of neutron diffraction to texture and residual stress characterization. Texture studies have centered on copper, tantalum, and uranium-alloy components for a variety of DOD end-items; recent work has included ceramic superconductors. Neutron residual stress studies have included components made of the above metals, ceramics, fiber-reinforced ceramic composites, and steel.

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A100 Reactor

NEUTRON SPECTROSCOPY

The vibrational dynamics and diffusion of hydrogen in metals and molecular species in heterogeneous catalysts, clays, and other layered materials are studied at NIST with inelastic and quasi-elastic neutron scattering. The results of this research reveal the bonding states and atomic-scale interactions and diffusion paths in such materials. Recent progress has allowed in-situ spectroscopic studies of hydrogen and molecular species down to 0.1 of an atomic percent.

These measurements can provide direct information, for example, on the molecular processes that affect reaction and selective release of chemicals in industrial catalysts and on the local trapping and clustering of hydrogen in metals and semiconductors, which cause embrittlement, corrosion, or changes in electronic properties. Collaborations with researchers from outside laboratories also include studies of the dynamics of atoms and molecules in molecular solids, alloys, and zeolites.

For the above research, the equipment available includes three-axis neutron crystal spectrometers and time-of-flight spectrometers for inelastic scattering, which along with neutron diffractometers, measure structural and dynamic processes in the time regime from 10^{-10} s to 10^{-14} s.

In the Cold Neutron Research Facility, two new very-high-resolution inelastic scattering spectrometers will be available. Controlled temperature (0.3 K to 1300 K) and pressure devices are available for changing sample environments.

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SMALL-ANGLE NEUTRON SCATTERING

Small-angle neutron scattering (SANS) is used to characterize sub-micron structural and magnetic properties of materials in the size regime from 1 nm to 100 nm. The SANS diffraction patterns produced by structural features in this size regime—for example, by small precipitates or cavities in metal alloys, by micropores or cracks in ceramics, by colloidal suspensions and microemulsions, or by polymers and biological macromolecules—can be analyzed to give information on the size and shape of the scattering centers as well as their size distribution, surface area, and number density. This detailed microstructure information is often a key to the prediction or understanding of the performance or failure modes of structural materials and materials processing conditions.

This five-detector, high-resolution thermal neutron diffractometer allows for accelerated data collection on the crystal structures of high-temperature superconductors, metals, and other materials in powdered form.

A number of scientists from the chemical, communications, advanced materials, and aerospace industries are already engaged in SANS research at NIST.

In the Cold Neutron Research Facility, 30-m and 8-m SANS spectrometers are operational, with a second 30-m nearing completion. These world-class SANS instruments will significantly enhance resolution and sample throughput. Computer-automated equipment is available for maintaining samples at temperatures from 4 K to 700 K and in magnetic fields up to 20 kilogauss. To extract structural information from the data, the researchers analyze SANS patterns with an interactive color graphics system and related programs.

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NEUTRON DIFFRACTION

Precise information on crystal structures in solids is often a key to understanding or improving the properties of modern materials and creating new materials with specific properties. Many important materials, for example, ceramics, catalysts, and rapidly solidified alloys, often can be obtained only in powdered form. In addition to single-crystal diffraction, state-of-the-art capabilities are in place at NIST for measuring and analyzing the crystal and magnetic structure of polycrystalline materials by neutron diffraction. Both single-crystal and multidetector, high-resolution powder diffractometers are available.

NIST researchers continue to develop improved methods for accurate structure refinement. A major effort currently is under way in relating atomic arrangement, including defects, to superconducting properties in high T_c ceramic superconductors. In addition, a number of industrial scientists are collaborating with NIST staff in neutron diffraction studies of inorganic catalysts, new kinds of ionic conductors for small batteries and fuel cells, improved ceramics for microcircuit substrates and engine components, and high-performance lightweight alloys for advanced aircraft.

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NEUTRON REFLECTOMETRY

The importance of thin films, layer structures, and interfaces has grown significantly in the last few years in a number of applications areas. Analogous to electromagnetic radiation, neutrons can undergo specular reflection at surfaces, and because neutrons can distinguish between different isotopes of the same element (such as hydrogen and deuterium), can penetrate to focus on subsurface interfaces, and are sensitive to magnetic properties of materials, they provide an important complement to other surface probes.

Thermal neutron reflectometry and grazing-angle diffraction are currently available at the NIST reactor. For the former, reflectivities as low as 2×10^{-7} have been measured, which is competitive with any neutron reflectometer in the world. Reflectometry studies at NIST and elsewhere include superconducting and magnetic layers, magnetic multilayers, polymer interfaces, and solid-liquid interfaces.

An even more sensitive instrument is being constructed in the Cold Neutron Research Facility. Neutron grazing-angle diffraction is a very new technique—first demonstrated at NIST. With this approach, lattice spacings of planes with normals parallel to the sample surface are determined to depths of ~10 nm.

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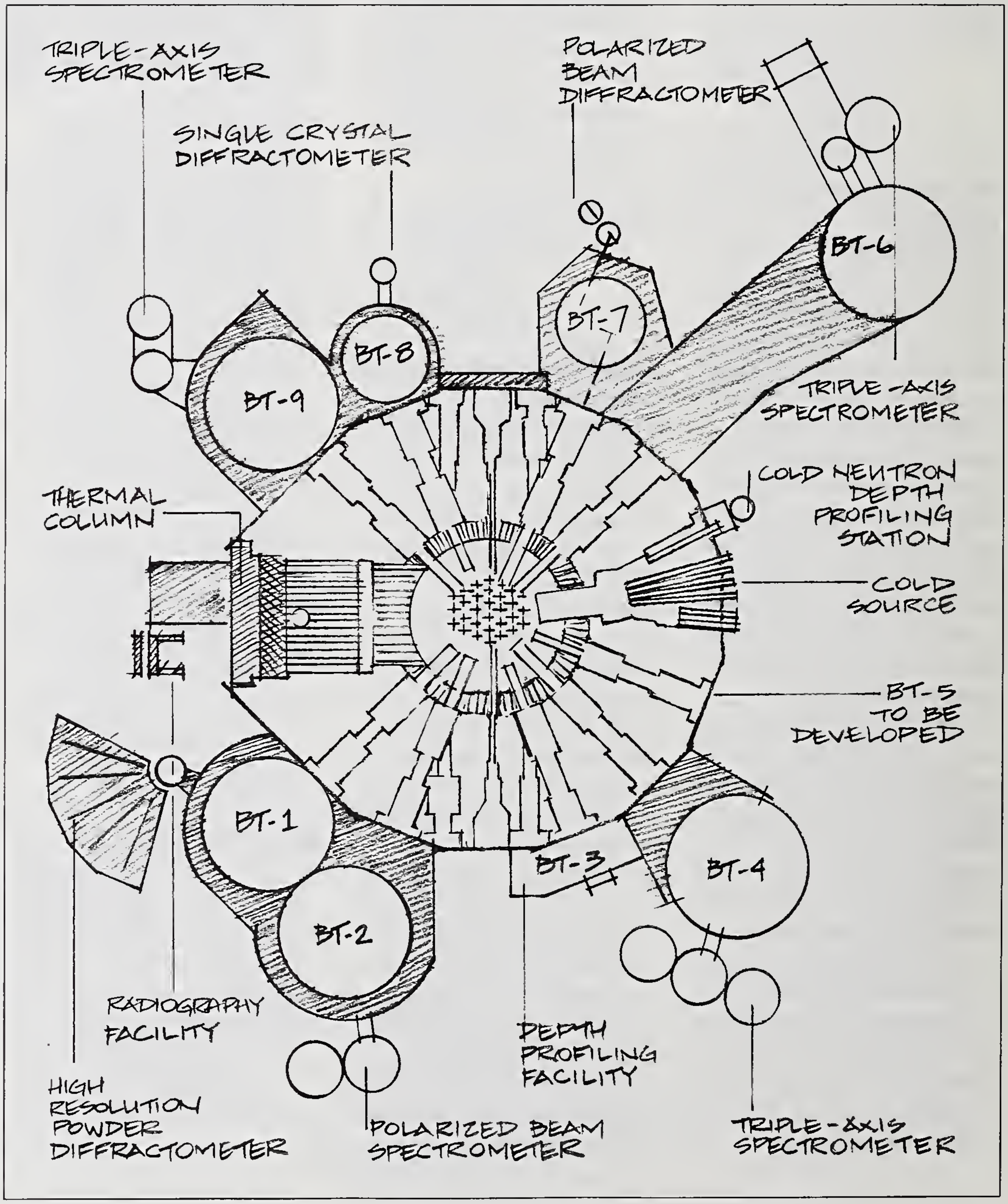
NIST RESEARCH REACTOR

The NIST research reactor (NBSR) is a national center for the application of reactor radiation to a variety of problems of national concern. A wide variety of internal and external programs have benefited from the broad range of capabilities available to researchers from industry, universities, and government laboratories.

CAPABILITIES: The NIST reactor is an enriched-uranium, D_2O cooled and moderated reactor, with a peak thermal core flux of 4×10^{14} neutrons/cm^2-s. The core comprises 30 fuel elements of a unique, split-core design, in which nine radial beam tubes "look" at a 17-cm gap between fuel-element halves. A large-volume cold source, two tangential and one vertical beam tube, a thermal column, several vertical thimbles, and four "rabbit" tubes complete the configuration. The reactor operates continuously, 24 hours a



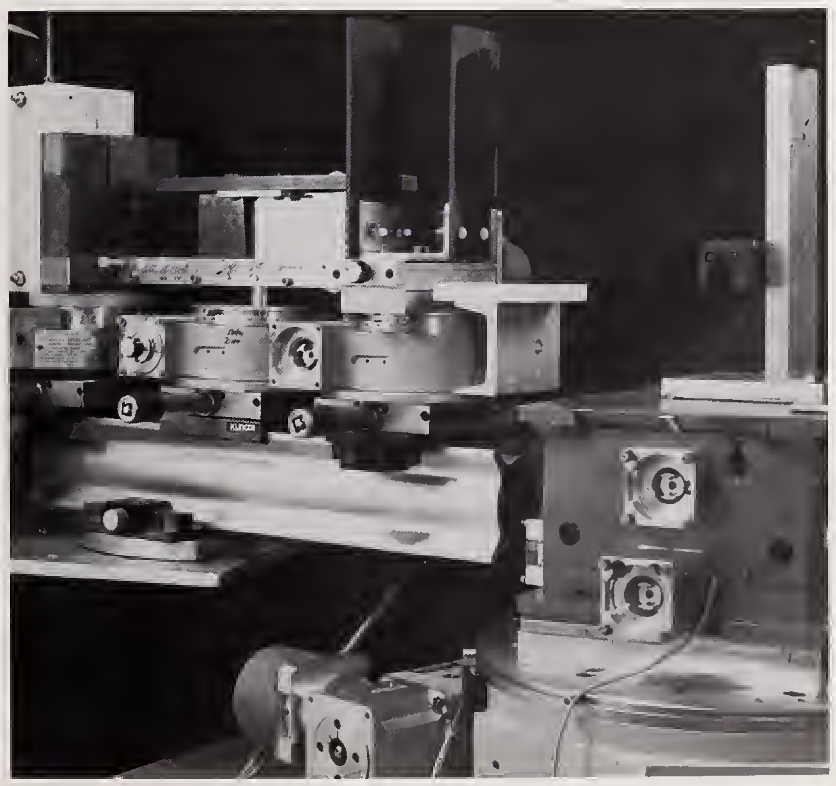
day on monthly cycles, followed by approximately a week of shutdown for refueling and maintenance.

The NBSR utilizes the flux available in a very efficient manner through relatively short core-to-instrument distances and large-diameter beam tubes. As a result, the flux on the sample for certain instruments is comparable to that at other major research reactors of higher power and peak core flux.

The experimental facilities in the reactor hall are used for:

- neutron scattering and diffraction;
- neutron radiography;
- trace analysis and depth profiling;
- neutron dosimetry and standards development;
- fundamental neutron physics;
- irradiations; and
- isotope production.

Left. Schematic of the NIST 20-megawatt research reactor and its associated thermal neutron instruments. Below. The thermal neutron reflectometer shown here produces high-resolution information on structures of multilayer coatings, magnetic films, liquid-solid interfaces, and other surfaces.



Sample environments for low and high temperature, low and high pressure, and high magnetic fields are routinely available. On-line graphics, plotting, and data fitting are available via personal computers, Macintoshes, MicroVAXs, and a DEC 5810.

■ Elastic Scattering. Four beam ports are dedicated to elastic scattering studies. A high-resolution powder diffractometer is installed at beam tube 1 (BT-1). The existing diffractometer is a five-detector system arranged with 20° spacing between detectors so that a complete diffraction scan (~10° to 110°) is made by incrementing the detector system through 20°. The wavelength is set at 0.154 nm, and collimations as low as 10′-10′-10′ are available. In late 1991 or early 1992, a 32-detector diffractometer will replace the current instrument. It will provide three monochromator positions, each set at 0.154-nm wavelength but at different take-off angles to allow maximum flexibility in selecting the optimal resolution function for a given measurement. Collimations down to 7′-20′-7′ are planned.

A triple-axis spectrometer with a four-circle goniometer is installed at BT-6 and is used primarily for residual stress and texture measurements, although inelastic scattering experiments also can be performed. This spectrometer is typically operated in the elastic scattering mode without an analyzer for texture studies and with the analyzer for residual stress measurements. Incident energies are continuously variable with $2\theta_M$'s from 20° to 75° available. Beam sizes up to 30×50 mm^2 and down to 2×2 mm^2 (for residual stress studies) are utilized. Single-crystal structure determinations are performed on this instrument while the new diffractometer is being completed.

A new thermal-neutron reflectometer, in which the incident beam can be polarized and the spin state of the reflected beam analyzed, is operational on BT-7. In this reflectometer, the polychromatic beam first passes through a pyrolytic graphite (PG) filter to suppress the higher-order wavelengths that would also be reflected by the vertically focusing PG monochromator set for a principal wavelength of 0.235 nm. For collimations of ~1° preceding and 1′ following the monochromator, respectively, the wavelength resolution $\Delta\lambda/\lambda \sim 0.01$, and the actual flux on the sample is $\sim 5 \times 10^5$ neutrons/cm^2-s. Two ^3He detectors are incorporated: one for reflectivity measurements, the other for grazing-angle-diffraction experiments. Polarization of the incident beam is accomplished in transmission by one or more polarizing Fe-Si supermirrors deposited on single-crystal Si substrates.

Because the reflectivities of the polarizing supermirrors are not unity for the unwanted spin state, two or more supermirrors are used in series. Reflectivities down to about 2×10^{-7} with a signal-to-noise ratio of about 1 have been measured.

■ Inelastic Scattering. Three triple-axis inelastic scattering spectrometers are operational. The BT-4 instrument employs two remotely selectable, vertical-focusing monochromator crystals (pyrolytic graphite and Cu), which provide incident energies from 3 meV to 300 meV continuously. It is capable of elastic-peak resolution down to 0.04 meV and also can be used in the "inverted-filter" mode for energy-loss spectroscopy of vibrations in the 15 meV to 280 meV range. The BT-9 spectrometer offers variable incident energy in the 5 meV to 50 meV range and collimations down to 10′-12′-12′-16′.

The BT-2 instrument has both triple-axis and polarized-beam (up to 96 percent) capabilities. Either a pyrolytic graphite or a Heusler-alloy monochromator, each vertically focused, are available, with remotely insertable cold beryllium or pyrolytic graphite filters. Built-in guide fields and spin rotators and collimation as low as 10′-5′-5′-20′ are available.

■ Elemental Analysis. Neutron activation analysis is performed utilizing the clean room for sample preparation, NBSR irradiation facilities, semi-hot and warm radiochemistry labs, and state-of-the-art radiation-counting labs. Development of methodology has aimed at accuracies and sensitivities over concentrations of 10^{-10} percent to 100 percent.

Radiochemical separations for specific elements and multi-element analysis at the ultratrace level are available.

A thermal neutron-capture prompt-gamma activation analysis facility is operational, with a neutron fluence of 5×10^8 n/cm^2-s in a 2-cm-diameter beam.

A thermal neutron depth-profiling facility is installed on BT-3 for non-destructive determination of near-surface elemental depth distributions in a variety of materials. Sensitivities approaching 10^{13} atoms/cm^2 with a spatial resolution of better than 30 nm for depths of 5 μm -20 μm are achievable.

■ Neutron Standards and Dosimetry. A number of neutron fields for standards and dosimetry are available. These include a Cf fission source, a ^{235}U cavity fission source, the thermal column beam, an intermediate energy standard neutron field, and 2-, 24.5-, and 144-keV filtered beams.

■ Irradiation Facilities. Four pneumatic tubes for 40-cm^3 "rabbits" with fluence ranges of 3×10^{11} to 2×10^{14} n/cm^2-s for irradiations of seconds up to hours are available. The cadmium ratio range for these facilities is 4 to 3000 (Au). For long irradiations 6- and 9-cm-diameter in-core thimbles are used. These are D_2O filled with fluences of $2\text{-}4 \times 10^{14}$ n/cm^2-s.

■ Neutron Radiography. Radiography facilities are available at a highly thermalized beam of the thermal column. Fluences range from 10^5 to 10^7 n/cm^2-s, depending on resolution, with a Cd ratio of 500:1, and an L/D ratio adjustable from 20:1 to 500:1. The diverging beam is 25 cm in diameter at the image plane 2 m from the reactor face. Facilities for autoradiography have also been developed.

APPLICATIONS:

■ Structural and Spectroscopic Studies of Magnetic Materials. Recent efforts have focused on rare-earth compounds, amorphous magnets and spin glasses, superconductors, magnetic bilayers, and hydrogen in metals.

■ Molecular-System Studies. These range from determination of crystal dynamics in condensed systems as complex as catalysts and military "energetic" materials, to characterization of phase transitions and translation-rotation interactions in ionic crystals.

■ Crystallographic Studies. Such studies have ranged from development of analytical techniques in single-crystal structure refinement and (powder) total-profile refinement to a host of structure determinations. The latter include ceramics (such as high-T_c superconductors and ionic conductors), alloys, zeolites, minerals, energetic, biological materials; and structural studies focused on the elucidation of phase transition mechanisms.

■ Thermal Neutron Reflectometry. This is a relatively new capability which already has produced very important results in studies of supermirrors, thin films, and interfacial structure characterization.

■ Elemental Analysis Programs. Examples include: studies in environmental chemistry, nutrition, biomedicine, energy and electronic devices—with emphasis on Standard Reference Materials for these applications.

■ Neutron Dosimetry Studies. Specific projects include: neutron fluence standards for power reactor pressure vessel irradiation surveillance, ultralight mass assay for commercial track recorder detectors, absolute fission-rate measurements, and development of thermal neutron beam monitors.

■ Non-Destructive Evaluation (NDE). Neutron radiography capability has proven to be a very important complement to X-ray radiography for applications where light-element localization is necessary, particularly in the presence of heavy elements. Neutron-induced autoradiography has been applied primarily to examining works of art. Diffraction facilities are being used for texture characterization and residual stress determination for a variety of metallurgical and ceramic hardware.

AVAILABILITY: There are 25 thermal-neutron facilities at the NBSR, which provide about 125,000 instrument hours per year. In 1990 over 450 researchers from 15 NIST divisions, 19 other federal organizations, 90 U.S. industrial and university laboratories, and 34 foreign laboratories utilized the facilities, either collaboratively with NIST staff or independently.

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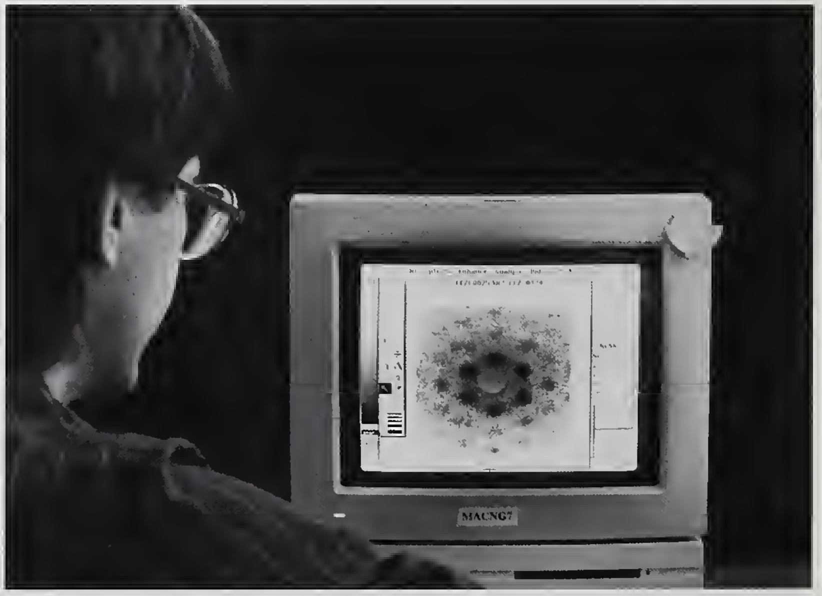
COLD NEUTRON RESEARCH FACILITY

The NIST Cold Neutron Research Facility (CNRF) is the first in the United States devoted exclusively to cold neutron research. The facility provides U.S. researchers prime access to a key tool of modern materials science. When fully complete in 1993, the $30-million project will provide 15 new experimental stations for use by U.S. researchers, with

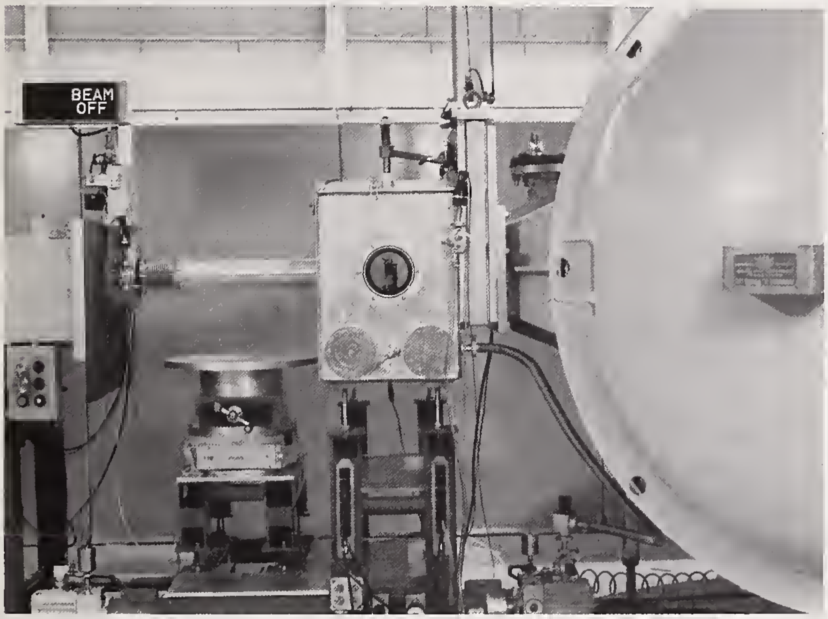


capabilities currently unavailable in this country.

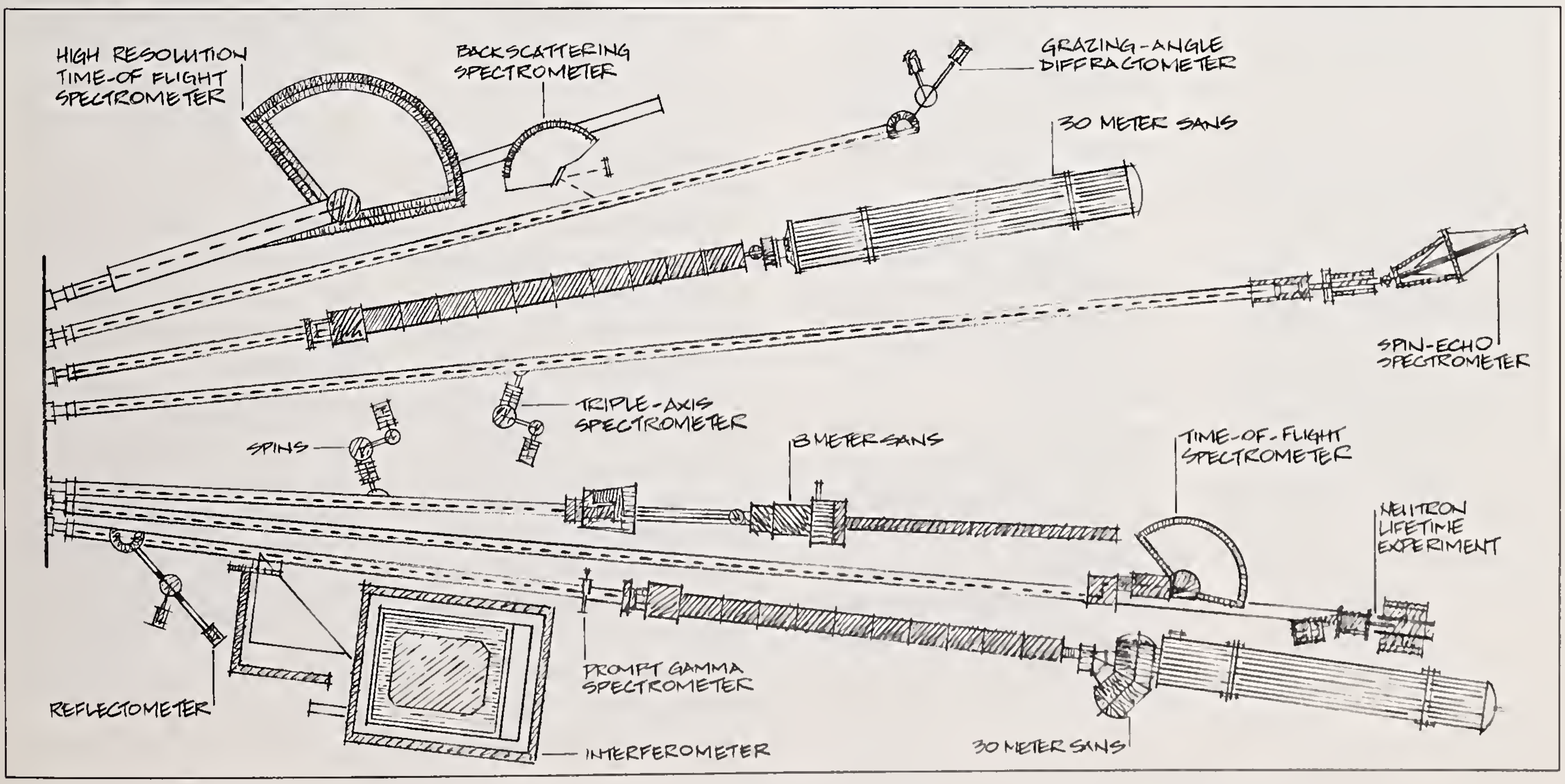
The facility is focused on the cold source, a block of D_2O ice (with 8 percent H_2O added) cooled to 30 K to 40 K by recirculating helium gas. The cold source is viewed by one port inside the reactor hall and seven neutron guide tubes. The guide hall and associated office and laboratory space has more than tripled the workspace available at NIST for neutron beam researchers.

CAPABILITIES: Instruments currently in operation include:

- Small-Angle Neutron Scattering (SANS). The NIST/Exxon/University of Minnesota 30-meter SANS will be the first to use a doubly curved mirror as a focal element in a long flight path to provide angular resolution and beam intensities that compare favorably with any SANS instrument in the world. Two large-area position-sensitive detectors will provide exceptional sensitivity to materials structures ranging from roughly 1 nm to 500 nm. A second 30-meter SANS—scheduled for completion by early 1992—will be one of two instruments in the NSF/NIST Center for High-Resolution Neutron Scattering (CHRNS). Polarized neutron capabilities are planned for both instruments.
- Depth Profiling Instrument. With a measured chemical sensitivity 20 times that of the existing NIST thermal-beam instrument, this station features automated sample handling; near real-time spectral processing; goniometer positioning of sample and detectors; and sample temperature control. Increased signal intensity will permit multidimensional imaging of elemental distributions and profiling of nuclides with absorption cross-sections for charged particle emission of less than 1 barn.
- Prompt-gamma-ray activation analysis. Providing greater sensitivity for this method than any other existing instrument in the world, this equipment provides unique measurements of chemical elements, such as hydrogen, that are difficult to detect by other means.
- Fundamental Physics Station. Available for use only in collaboration with the NIST fundamental physics research group, an end



Far left. An interactive color graphics program used by materials scientist John Barker displays 30-m SANS data in real time. Left. The 30-m small-angle neutron scattering spectrometer at the CNRF is operated jointly by NIST, Exxon, and the University of Minnesota. Below. Schematic of current and planned instrumentation at the Cold Neutron Research Facility.



guide position physics station currently is instrumented for a new measurement of the lifetime of the neutron.

Instruments planned for completion between 1992 and 1993 include:

- neutron reflectometer;
- grazing-incidence diffractometer;
- triple-axis spectrometer;
- spin-polarized inelastic neutron spectrometer (SPINS);
- time-of-flight spectrometer;
- back-reflection spectrometer;
- spin-echo spectrometer; and
- neutron interferometer.

APPLICATIONS: The unusual sensitivity and range of measurements possible at the NIST CNRF provide applications in materials structures, materials dynamics, chemical analysis, and neutron physics. Currently operational instruments are used to study microstructures in metals, ceramics and colloidal mixtures; molecular geometry of polymer and biological macromolecules; chemical composition of semiconductors, high-tech materials, air-pollution filters, and other samples; and neutron decay rates.

AVAILABILITY: The CNRF operates as a national facility open to all qualified researchers. NIST develops experimental stations for use by the general U.S. research community. Two-thirds of the available time on these stations will be allocated by a Program Advisory Committee (PAC) on the basis of scientific merit of written proposals. The PAC is appointed by NIST with a majority of members chosen from outside NIST.

The second mode involves Participating Research Teams (PRTs), which are developing the remaining stations. The PRTs are responsible for design, construction, and maintenance of the facilities in return for three-fourths of the available time. The remaining time will be allocated by the PAC. The NSF/NIST (CHRNS) instruments constitute a special PRT in which all allocatable time will be available to the research community through the proposal review/PAC system. For all instrumental stations, instrument-responsible scientists will be designated to assist users in performing their experiments.

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METALS PROCESSING LABORATORY

The metals processing laboratory at NIST contains special facilities for the production of rapidly solidified alloys, including equipment for gas atomization and electrohydrodynamic atomization to produce rapidly solidified alloy powders, melt spinning to produce rapidly solidified alloy ribbons, and electron beam surface melting to produce rapidly solidified surface layers. Hot isostatic pressing equipment is available for consolidating alloy powders.

CAPABILITIES:

- Gas Atomization. The gas atomization system can be used to produce up to 25 kg of rapidly solidified alloy powder per batch while maintaining a controlled atmosphere throughout the atomizing and powder handling process. High-energy gas (Ar, He, or N) impinging on a liquid metal stream breaks up the liquid into small droplets, which solidify rapidly. Cooling rates are up to 10^5 K/s. The atomized powder, entrained in the gas flow, is collected in removable, vacuum-tight canisters.
- Electrohydrodynamic Atomization. In the electrohydrodynamic atomization system, a liquid metal stream is injected into a strong electric field. The field causes the stream to disintegrate into droplets, which solidify rapidly to produce extremely fine (<1 μm diameter) alloy powder. Powder produced by this process is well-suited for studying solidification dynamics. As the system is presently configured, small quantities for microscopic examination can be produced from alloys with melting points up to 900 °C.
- Melt Spinning. Melt-spinning techniques can be used to produce rapidly solidified alloys in ribbon form. Because of the high cooling rates (up to 10^6 K/s) with this method, amorphous alloys as well as crystalline alloys can be produced. Ribbons up to 3-mm wide and 0.05-mm thick can be produced in quantities of several grams per batch. Materials with a wide range of melting points, from aluminum alloys to super-alloys, have been produced.
- Electron-Beam Surface Melting. An electron-beam system, which provides surface melting and subsequent rapid resolidification of surface layers, can be operated in either a pulsed or continuous mode. The electron beam can be focused to a spot less than 1 mm in diameter and deflected at frequencies up to 5 kHz.
- Hot Isostatic Press. A hot isostatic press (HIP) with microprocessor control of the temperature-pressure-time cycle is available for consolidation of powder or compacted powder shapes. The HIP has a cylindrical working volume 15 cm in diameter and 30 cm high. The maximum working pressure is 207 MPa. The molybdenum furnace has a maximum heating rate of 35 K/min and is capable of maintaining 1500 °C.

AVAILABILITY: These facilities are designed to produce alloy research samples that otherwise are difficult for users to obtain. Typically, industrial companies or universities send workers to NIST to participate in preparing alloys of special industrial and scientific interest for further analyses in their home laboratories and to collaborate with NIST scientists in investigations of generic relationships between processing conditions and resulting alloy microstructures and properties.

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POLYMER COMPOSITE FABRICATION FACILITY

The composite fabrication facility permits the preparation of well-controlled polymer composite samples for scientific studies and the evaluation of results from NIST's processing science program in a realistic fabrication environment.

CAPABILITIES: The facility involves five major components and associated support equipment: a prepregger to prepare sheets (up to 180 cm × 30 cm) of unidirectional fiber coated with resin; a fully computerized autoclave capable of temperatures up to 535 °C and pressures up to 3.6 MPa; an automated hydraulic press (temperatures up to 535 °C) with water-cooled platens for rapid, controlled cooling; a unidirectional flow facility for characterizing the permeability of fiber preforms in all three directions; and a specially designed two-component pumping system for fabrication of samples by resin transfer molding and for conducting flow visualization experiments.

APPLICATIONS: The facility can be used to study a variety of questions, such as the details of resin flow and void formation during processing and the influence of processing conditions, fiber orientation, and fiber-surface treatments on performance.

AVAILABILITY: Used by NIST staff in an active research program on polymer composites, the facility is available for collaborative studies with industry, academic institutions, and other government agencies.

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POWDER CHARACTERIZATION AND PROCESSING LABORATORY

Advanced ceramics are manufactured by the consolidation of fine powders. Researchers at NIST are working to develop the scientific foundation needed for improving reproducibility and reducing the cost of producing ceramic components. The powder characterization facility offers specialized instrumentation for measuring physical properties, phase composition, and surface chemical properties. In addition, facilities exist for processing and synthesizing ultrapure powders.

CAPABILITIES:

■ Physical Properties Measurement. Particle size distribution, specific surface area, specific gravity, and porosity are some of the major physical properties for which instrumentation is available. Powder size distribution can be determined by gravity sedimentation followed by X-ray absorption, centrifugal sedimentation, light diffraction, and photon correlation spectroscopy. The size range of these instruments covers 0.03 μm to 200 μm. Each instrument works in a specific range and provides the data in the form of a discrete size range. The particles are examined directly by the application of scanning and transmission electron microscopy of particles as small as 0.001 μm.

■ Surface and Interface Chemistry Measurements. The surface and interface characterization of powders contributes to the knowledge base of the surface interactions of particles in contact with solvents. As the particles become smaller, their surface characteristics become more significant. Micro-Raman and Fourier transformed infrared spectroscopy are used to study surface composition, and electrophoretic mobility and acoustophoretic mobility are used to study modification to the powder surface as a result of an interaction with a solvent.

■ Phase Composition. Since most of the ceramic powders undergo phase transformation during densification, understanding the phase changes in specific densification environments is an active part of NIST research. The Siemens high-temperature X-ray diffractometer has a temperature range from room temperature to 3000 K and a position sensitive detector. In addition, sintering can be carried out in oxygen-free argon or nitrogen environments.

■ Solid-State Imaging. The solid-state nuclear magnetic resonance (NMR) spectrometer/imager carries out measurements in chemical shift, nuclear spin density, relaxation times, and imaging of NMR active nuclear distribution. Ceramic powder slurries, green bodies, and dense ceramics can be analyzed for identification of impurities, chemical state, and composition distribution by nondestructive evaluation. In addition, the NMR can determine amorphous phase content of the powders.

■ Powder Synthesis. The ability to synthesize powders of controlled characteristics is needed to develop a powder-processing knowledge base. The fine-powder synthesis facility consists of a chemical flow reactor for controlled synthesis and inert atmosphere chambers. A solution atomizer is available to generate polydisperse aerosols in the range 0.01 μm to 1.0 μm, as is a spray dryer to form monodisperse agglomerates in the range 0.5 μm to 100 μm.

■ Colloidal Suspensions Preparation. Colloidal processing of ceramic powders has emerged as an attractive technology for producing defect-free ceramics. However, some major issues still remain to be addressed, including the lack of characterization techniques for slurries that contain high concentrations of solids and scientific understanding of limitations in the preparation of such slurries. The powder processing laboratory consists of an acoustophoresis instrument, a rheometer, a high-energy agitation ball mill, and slurry consolidation equipment. These techniques are used to study interface chemistry, flow behavior, size reduction, morphology modification, and densification of polydisperse particles and similar processes resulting from interactions between the particles and their environment.

AVAILABILITY: These facilities are used primarily to support NIST research programs using ceramic powders. They are available for collaborative or independent work by qualified government, industry, or university personnel with preference given to projects related to ceramics research.

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MECHANICAL BEHAVIOR LABORATORIES

Mechanical property measurement facilities at NIST permit characterization of all mechanical properties over a wide range of force levels and temperatures.

CAPABILITIES: Conventional mechanical property measurements, such as yield and ultimate strengths, elongation, and reduction of area, can be made at temperatures ranging from 4 K (produced by immersion of the specimen in liquid helium in a cryostatted test apparatus specially designed for this purpose) to 2800 K (produced by testing in a vacuum furnace heated by tungsten elements). Equipment for measuring fracture toughness over the same temperature range is used. Material fatigue resistance can be characterized in a variety of ways—including fatigue crack growth, load- and strain-controlled, random-load, or conventional rotating beam—at temperatures from 4 K to 600 K.

Narrow and wide-plate behavior characteristics, such as in crack arrest testing, are studied using computer-assisted data acquisition and reduction equipment. Up to 100 strain gage channels are available, as well as capabilities for capacitance and strain-gage extensometers and full-field video optical strain data measurement (photoelastic and moire interferometry). High-speed digital oscilloscopes are used to record strain patterns produced by rapidly moving cracks (1000 m/s) in crack arrest tests. Special test requirements, including dynamic tear, drop weight, torsion, and impact, can be met using existing facilities. Complete metallographic and hardness characterizations of specimen materials are available.

The high-load mechanical testing machine has a force capacity of 50 MN in compression and 25 MN in tension. Servo-hydraulic tension-compression machines are available in a range of load capacities and actuator travel rates; the maximum load capacity is 4 MN and the maximum displacement rate is 50 cm/s. These smaller machines are used routinely for measuring fracture and fatigue properties in liquid helium. Two screw-driven tensile machines have capacities of up to 100 kN for tensile testing in liquid helium.

AVAILABILITY: These facilities are available to guest researchers for collaborative programs. NIST routinely performs research on these facilities for outside sponsors.

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The mechanical properties of structural alloys, superconductors, polymer composites, and other materials are measured at cryogenic temperatures by physical scientist David McColskey.

SMALL-ANGLE X-RAY SCATTERING FACILITY

Small-angle X-ray scattering (SAXS) is a technique in which a highly collimated beam of X-rays (wavelength range 0.7 angstrom to 3 angstroms) is used to probe the structure of materials on the size scale of 0.1 nm to 100 nm. Materials exhibiting structure in this size range include polymers, biological macromolecules, ceramics, metals, and alloys. The small-angle scattering patterns can be analyzed to characterize the size and shape of the scattering centers as well as their spatial distribution and surface area. Data obtained from these experiments are complementary to data obtained from other diffraction studies and from morphological studies using electron microscopy.

CAPABILITIES: The NIST 10-m SAXS camera uses a 12-kW rotating anode X-ray generator as a source of X-rays. The target is usually copper $\lambda(K\alpha) = 1.54$, but additional wavelengths are available. The collimation path is defined so as to permit a 10^{-2} nm^{-1} resolution in reciprocal space at the surface of a two-dimensional position-sensitive proportional detector. The collimation path and the scattered beam path are evacuated, and all elements of vacuum operation, X-ray optical configuration, sample selection, and calibration are computer controlled. Image data are collected by a minicomputer, and an associated VAX/730 computer supports a complete repertory of software for displaying, analyzing, and modeling the results.

Sample chambers are being developed for measurements at high temperatures and to deform specimens during measurement.

APPLICATIONS: The SAXS technique is used to study molecular conformation, microphase domain structures, crystallization phenomena, network formation, craze initiation, void distribution, and similar phenomena resulting from fluctuations in electron density within a material. Such studies are currently of broad interest in all areas of materials science.

AVAILABILITY: This facility is operated by members of the NIST staff in support of their active research program in polymeric materials. It is available for use by researchers from industry, academia, and other government agencies on either a collaborative or independent basis.

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MATERIALS SCIENCE X-RAY BEAMLINES

NIST operates two beamstations on the X23A port at the National Synchrotron Light Source (NSLS) at Brookhaven National Laboratory in New York. The light source provides an intense continuous-spectrum beam of collimated and polarized X-rays many orders of magnitude brighter than the beams that conventional laboratory sources provide. These two beam stations offer access to dedicated instrumentation for small-angle X-ray scattering, X-ray diffraction imaging (topography) and extended X-ray absorption fine structure.

CAPABILITIES: Small-angle X-ray scattering can be carried out in the photon energy range from 5 keV to 11 keV. The minimum wavevector is 4×10^{-3} nm^{-1} and the wavelength resolution is $\Delta\lambda/\lambda = 10^{-4}$, enabling anomalous small-angle scattering with excellent resolution. Diffraction imaging of single crystals and powders is carried out with monochromatic photons between 5 keV and 30 keV. An energy-tunable X-ray image magnifier enables imaging of microstructure down to less than 1 mm. The energy-scanning experiments, primarily EXAFS, also are performed over an energy range from 5 keV to 30 keV.

APPLICATIONS: Small-angle scattering measurements on ceramic and metallurgical materials are used to characterize the microstructure in the 2-nm to 1-µm size range as a function of starting chemistry and processing parameters. Scattering from a particular entity can be separated from other scatterers in a complex material using anomalous small-angle X-ray scattering. Diffraction imaging is used to study imperfections and strains in single crystals and powder compacts. The structure of strained semiconductor interfaces and metal multilayers can be studied using EXAFS. A combination of EXAFS and diffraction will provide a capability for site-specific local structure determination in crystals.

AVAILABILITY: Beam time is available to qualified scientists provided safety requirements are met and scheduling arrangements can be made. Proposals for collaborative use of the facility are reviewed at NIST; proposals for independent use of the facility should be submitted to the NSLS.

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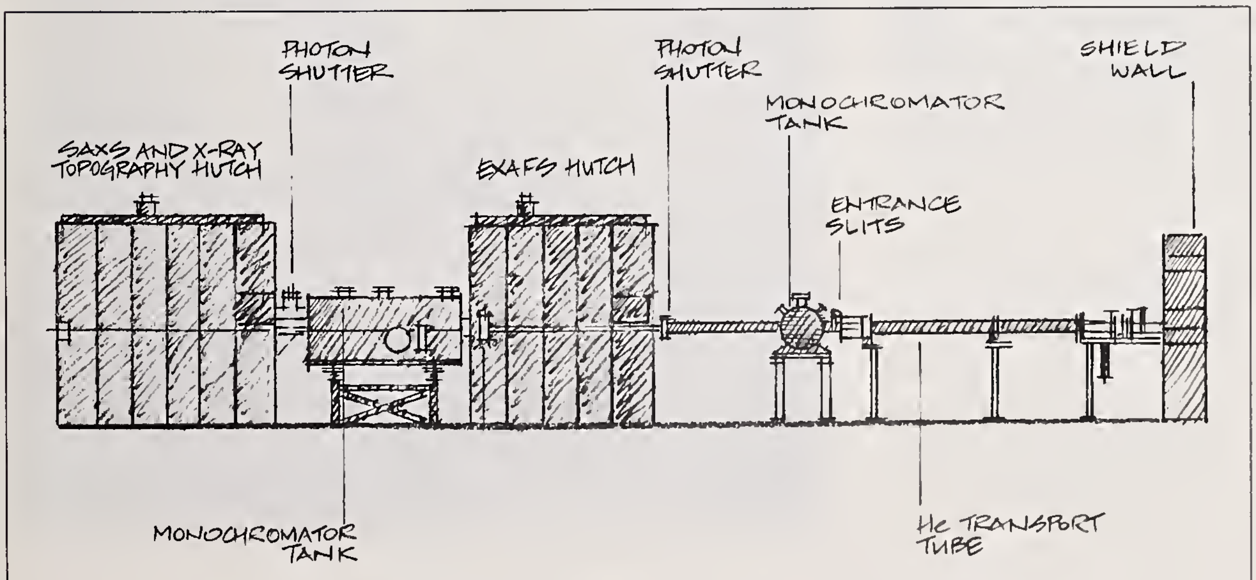
Schematic of the NIST X-Ray Beamline at the Brookhaven National Synchrotron Light Source.



BUILDING AND FIRE RESEARCH LABORATORY

Major goals of the Building and Fire Research Laboratory (BFRL) are to improve the productivity of the U.S. construction industry, which now faces stiff competition from overseas firms, and to reduce the human and economic costs resulting from fire, earthquakes, and other hazards.

Products of the laboratory's research include measurements and test methods, performance criteria, and technical data that are often incorporated into building and fire standards and codes. Staff members are involved in more than 100 activities to develop voluntary standards. In addition, studies on fire science and engineering, building materials, computer-integrated construction practices, and structural, mechanical, and environmental engineering yield results presented in a form that industry can use.

For example, HAZARD I, a BFRL-developed computer program, models the behavior of building fires, helping engineers, architects, and others to anticipate and reduce the potential for hazards. Another computer model simulates how microstructural defects can undermine the structural integrity of concrete.

Laboratory personnel also conduct investigations at the scene of structural failures due to earthquakes, hurricanes, or other causes. The knowledge gained from these investigations is applied to the laboratory's recommendations for construction practices to reduce hazards.

NIST scientists and engineers also use investigations of major fires to advance their research and guide their recommendations to policy-makers. Following the Persian Gulf War, these researchers made measurements of combustion products released by the hundreds of oil well fires raging in Kuwait. Such measurements will help determine whether the burning of oil spills poses less environmental damage than other clean-up methods.

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COOPERATIVE RESEARCH OPPORTUNITIES

STRUCTURES

STRUCTURAL ENGINEERING

Under the National Earthquake Hazards Reduction Program, NIST does research and development work that is used in standards for the seismic safety of structures. Experimental and analytical research is under way at NIST to develop knowledge for design and construction standards for new and existing buildings and lifeline structures. The work involves identifying mechanisms of failure and establishing criteria to ensure structural safety.

Current research addresses:

- a rational procedure to determine the ultimate shear strength of partially reinforced masonry walls;
- strength and ductility of connections in precast concrete structures;
- drift control criteria for flexible frames;
- evaluation criteria for base-isolation systems and test procedures for evaluating the response of structures subjected to seismic loading;
- strengthening methodologies for concrete frame structures; and
- effects of subsurface conditions on ground motion.

Other structural research includes:

- studies of structural performance for development of standards and test methods for high-performance concrete in major construction applications;
- techniques and instrumentation for assessing the properties of existing structures and for developing technical criteria and methodologies for strengthening and repairing existing structural members and systems; and
- analytical and experimental methods for identification of dynamic response characteristics of flexible members and structural networks.

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BUILDING MATERIALS

CEMENT AND CONCRETE

NIST researchers are undertaking research to develop a fundamental understanding of the relationship between chemistry, microstructure, and service life of concrete and other inorganic building materials. The goal of the program is to be able to predict the behavior of these materials and their service lifetimes. The service life of concrete largely depends on the rate of moisture ingress and of transport of dissolved salts and gases in the pore system of concrete.

Models are being developed to consider service conditions, including composition of the environment; the transport rate of reactants by diffusion, convection, and capillary forces; and reaction mechanisms. Researchers are developing mathematical and simulation models to predict the relationships between pore structure and diffusion and permeability of concrete.

Research projects include development and validation of mathematical models of microstructure development in cement pastes as the cement hydrates, the effects of microstructure on permeability and fracture of concrete, and mechanisms of degradation of concrete. Artificial intelligence systems are being developed for optimizing the selection of materials and for diagnosing the causes of material degradation. The research is performed using a variety of techniques, including scanning electron microscopy, computerized image analysis, X-ray diffraction, and thermal analysis.

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Physicist Edward Garboczi works on a computer model that will simulate how the microstructure of concrete develops during the setting process.

ORGANIC BUILDING MATERIALS

NIST is conducting basic and applied research to develop methodologies to aid in predicting the service life of organic building materials, such as protective coatings for steel, roofing materials, and asphalt. The researchers are investigating degradation mechanisms, improving characterization methods, and developing mathematical models of the degradation processes. Stochastic models, which are based in reliability theory and life-testing analysis, are included in the modeling efforts for coatings and roofing materials.

To help in understanding the mechanisms of degradation and provide data for models, materials are characterized in many ways, including Fourier transform infrared spectroscopy, thermal analyses methods, scanning electron microscopy/energy dispersive X-ray, and visual and infrared imaging. Researchers also are developing improved ways to characterize atmospheric environments in which these materials often are used. Characterization of environmental parameters that cause degradation is needed to link inventory data with service life prediction.

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BUILDING ENVIRONMENT

LIGHTING

The NIST lighting program focuses on fundamental measurements of interior luminance and illuminance distributions, light fixture output, and the interaction between lighting and HVAC systems. A continuing challenge is to develop metrics for lighting quality that evaluate the combined effects of illuminance, luminance, contrast, color rendering, lighting geometry, and configuration.

Current research includes a major project on the measurement and modeling of the interaction of lighting and HVAC in a simulated interior office module. This research will result in the development of a detailed computer model for predicting the effects of different types of lighting and HVAC systems. Modifications will be made to the facility in the near future to study the effects of dimming, thermal mass, and external solar conditions.

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INDOOR AIR QUALITY

Measurement and testing procedures, technical data, and comprehensive indoor air-quality models are being developed by NIST researchers as part of a multiyear program to improve indoor air quality in buildings. Experiments are being conducted in several large buildings to develop test methods to evaluate how air moves into and within large commercial buildings. The researchers are developing comprehensive computer models to predict pollutant levels from sources introduced into buildings from outdoor air and those generated inside from sources as combustion equipment and floor and wall coverings.

The first phase of the research is complete. Models exist that can predict indoor contaminant levels as functions of emission, dilution, and intrabuilding air movement. The models currently are being extended to model reactive contaminants. Test procedures also are being developed to evaluate the effectiveness of various filter media for removing gaseous pollutants. A test facility for this purpose has been designed and assembled. Test methods will be developed for commercial gaseous air filters and for those used in residences.

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Using sophisticated equipment such as this Fourier transform infrared microscope, NIST research chemist Eric Byrd develops ways to predict the service life of organic building materials, such as protective coatings for steel, roofing materials, and asphalt.

REFRIGERATION MACHINERY

NIST researchers are exploring the use of refrigerant mixtures to improve the efficiency of refrigeration cycles and replace harmful chlorofluorocarbon refrigerants that are damaging the ozone layer of the upper atmosphere. The researchers evaluate a wide variety of refrigeration cycles by using a breadboard heat pump to "plug" and "unplug" circuits and components. The breadboard heat pump will be altered to test new refrigeration cycles. This is based on the results of a theoretical study being conducted to find the optimal combination of mixtures and appropriate refrigeration cycles for the best and most versatile performance.

The "best" of the advanced cycles will be selected and equipment built and evaluated. A mini-breadboard that will use a very small charge will also be built so that rare refrigerants of limited production can be studied.

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THERMAL INSULATION

Researchers at NIST are developing basic data and simulation models for heat, air, and moisture transfer through building envelope components. They are developing and validating a theoretical model for moisture transfer, completing research required to develop a low-conductivity Standard Reference Material (SRM), developing a thermal conductivity database for chlorofluorocarbon (CFC) blown thermal insulation products, and refining a dynamic test procedure for building components.

A 1-m guarded hot plate is used to develop SRMs; to determine thermal conductivity values for various materials, such as CFC-blown insulation; and to provide measurement services to manufacturers and researchers. A calibrated hot box is used to measure the steady-state and dynamic performance of full-scale wall systems.

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BUILDING CONTROLS

NIST research is fostering the development of more intelligent, integrated, and optimized building mechanical systems. A dynamic building/heating, ventilating, and air-conditioning control system simulation program is used to study HVAC/control system dynamics and interactions. An expanded building management and controls laboratory is used to assist the building controls industry in the development, evaluation, and testing of communication protocol standards for the open exchange of information.

The application of knowledge-based systems to buildings is a new area of research. Plans call for exploring how real-time models, "tuning" techniques, forecasting, optimal control theory, and a rule-based expert system can be combined to evaluate control system performance, make control strategy decisions that optimize building performance, and perform diagnostics to advise the building operator or manager on building operations, equipment problems, or maintenance requirements. Work has begun for the Department of Energy to develop an HVAC/building emulator/EMCS tester, which will provide a method for evaluating application algorithms.

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COMPUTER-INTEGRATED CONSTRUCTION

NIST researchers are developing rational techniques for defining and testing computer representations of information needed throughout the building process, from the conception of a building to its demolition. The goal of the program is to develop validated neutral data representations for use in standards for accessing, exchanging, and archiving information. As a corollary, the program seeks to develop testing methods that assure consistency, completeness, and correctness of information. The research draws on evolving information technologies, including knowledge engineering and semantic modeling. Subject areas for research include standards and codes, engineering drawings, and product data.

Past research has resulted in methodologies for representing, analyzing, and expressing standards, and for interfacing standards to computer-aided design systems. Recent research results are being applied in the development and validation of national and international standards for product data, such as the Initial Graphics Exchange Specification and the Standard for the Exchange of Product Model Data.

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Using a small unmanned blimp, NIST researchers measure the combustion products and the smoke plume flow from crude oil spill fires. This study was conducted by NIST at the U.S. Coast Guard Fire and Safety Test Detachment in Mobile, Ala.

FIRE SCIENCE AND ENGINEERING

FIRE SIMULATION

This project is designed to provide the expedient transfer of scientifically based technology from NIST to the professional user community and to create a link between NIST computer-based activities and others doing similar or complementary work. Over the past decade, NIST researchers have developed many computer models of various aspects of fire. Researchers develop engineering systems for design application as well as expert systems, collect supporting data and programs, and operate a working and training laboratory dedicated to fire-safety computations.

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FIRE DYNAMICS

Predictive fire models need accurate representations of burning objects. The most important of these for residences are upholstered furniture and wall coverings. NIST researchers are using both laboratory experiments and computer modeling to understand the processes that govern these types of combustion. Algorithms are being developed and validated for use in hazard analysis.

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FIRE MEASUREMENT AND RESEARCH

HAZARD ANALYSIS

The United States has one of the worst fire records in the industrialized world. NIST researchers are helping to reduce the losses and the cost of fire protection by providing scientific and engineering bases needed by manufacturers and the fire protection community. One project involves the development of predictive, analytical methods that permit the quantitative assessment of hazard and risk from fires. Researchers base these methods on numerical modeling but also include hand-calculation methods for estimating hazards and design curves/tables to be used by architects and engineers. To ensure widespread use, the necessary data must be readily available, and data input and presentation must be in terms readily understandable by the average professional. Thus, the projects include a strong emphasis on state-of-the-art computer graphics and computer-aided design techniques.

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LARGE FIRES

Large fires, which result from industrial and transportation accidents or natural disasters, present a hazard to the surrounding area. While these fires attract media and public attention, very little is known about their characteristics, including the chemical compounds that are released into the atmosphere. NIST is researching and measuring large fires to understand and predict the effect of these fires in terms of fuel burning rate, chemical compounds in the smoke plume, and the downwind movement of the smoke plume, including particulate settling. As a first step, NIST is measuring smoke plumes from large, open-air fires to help develop computer models which can predict their size and movement. Further fire dynamics research and advanced computational fluid dynamics will lead to methods capable of assessing the local impact of large fires. These methods can be used by industry in preplanning analysis for potential accidents or as technical support for emergency response.

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ADVANCED FIRE SENSING

Fires that are detected and suppressed quickly do little damage. NIST research is studying new "fire signatures" that would enable a new generation of detectors. The signals from these detectors would be electronically analyzed to alert occupants or suppression devices, perhaps even before flames exist. The research also is intended to understand and provide technology for avoiding the high false alarm rate of current sensors.

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SMOKE AND TOXIC GAS PREDICTION

Most people who perish in fires die from smoke inhalation. Many are exposed to this smoke for longer periods because the blackness of the smoke impedes their escape from the burning building. NIST research is developing the scientific base and the predictive methods for the yields of carbon monoxide (the predominant toxicant) and soot. These require improved knowledge of the chemical and physical processes in flames, combined with the fluid mechanical processes that dominate air entrainment into fire plumes and the flow fields within a compartment.

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POLYMER COMBUSTION RESEARCH

NIST is conducting research to develop a scientific base for the gasification and combustion of natural and synthetic polymers, particularly for the more applied fire research activities. For example, NIST scientists are working on the kinetics and heats of combustion for wood, theoretical modeling of thermal degradation of polymers, smoldering research, and detailed degradation mechanisms of polymers.

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RESEARCH FACILITIES

LARGE-SCALE STRUCTURES TESTING FACILITY

The large-scale structures testing facility consists of a universal testing machine (UTM) that may be used with a 13.7-m-high reaction buttress equipped with a horizontal hydraulic ram of 4.5-MN capacity. A combination of horizontal and vertical forces may be applied to large-scale specimens.

CAPABILITIES: The UTM portion of the facility is a hydraulically operated machine of 53.4-MN capacity and is one of the largest in the world. It tests large structural components and applies the forces needed to calibrate very large capacity force-measuring devices. It can apply compression forces to column sections or fabricated members up to 18 m in height. The reaction buttress will resist horizontal forces to 4.5 MN from floor level to 12.2 m high. Flexure and tension specimens may be subjected to forces up to 18 MN and 26 MN, respectively. Two-m-thick test floors east and west of the machine may be used to tie specimens in place.

A servo system has been added to the original manual controls of the UTM. It may be programmed by function generator or computer to create any desired loading function using force, strain, or displacement as the variable.

Loads may be applied to a specimen by both the UTM and horizontal ram. A four-rail track system equipped with low-friction rollers has been used for concrete column tests. An "A" frame was used to resist horizontal reaction forces generated by the ram and was attached to the buttress at the desired height.

APPLICATIONS: A testing program was conducted to evaluate the performance of concrete columns 1.5 m in diameter and up to 9.1 m high. Another test series evaluated fracture propagation in steel plates 1 m wide and 0.1 m and 0.15 m thick. A third experiment used the servo-control system programming repeated loads applied to composite specimens.

Low-cycle fatigue tests, destructive or ultimate loads, earthquake simulation in two dimensions, and complex loading of components may all be accomplished in this facility. Servo operation of this machine creates a unique potential for precisely controlled very large forces applied to test components.

AVAILABILITY: This facility, which must be operated by NIST staff, is available for cooperative or independent research. Tests should be arranged as far in advance as possible as special hardware may be needed for specimen attachment.

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TRI-DIRECTIONAL TEST FACILITY

The tri-directional test facility is a computer-controlled apparatus capable of applying cyclic loads simultaneously in three directions. It is used to examine the strength of structural components or assemblages under the application of a variety of loading phenomena, such as an earthquake or wind. This is one of the largest such facilities in the world, both in terms of its high load capacity and its capability to handle large, full-scale specimens.

CAPABILITIES: The facility can apply forces and/or displacements in six directions at one end of a specimen. The other end of a specimen is fixed. Specimens up to 3.3 m high × 3 m in length or width may be tested. The six degrees of freedom are translations and rotations in and about three orthogonal axes. The forces are applied by six closed-loop, servo-controlled hydraulic actuators that receive instructions from a computer. Operating under computer control, the facility simultaneously maintains control of the load and/or displacements in each of the three orthogonal directions. Loads may be applied up to 2,000 kN in the vertical and about 890 kN in each of the two horizontal directions.

APPLICATIONS: The test facility is limited only by the size of the test specimen. Loads may be cyclic or unidirectional depending on the type of loading condition being simulated. The facility is used to study masonry shear walls subjected to reverse cyclic lateral loading and precast concrete connections also subjected to reversed cyclic lateral loading. This facility supports NIST's role in conducting research for seismic design and construction standards in the National Earthquake Hazards Reduction Program.

AVAILABILITY: The tri-directional test facility is used by NIST staff in a variety of NIST research projects and collaborative projects with other agencies. It also is available for independent research but must be operated by NIST staff.

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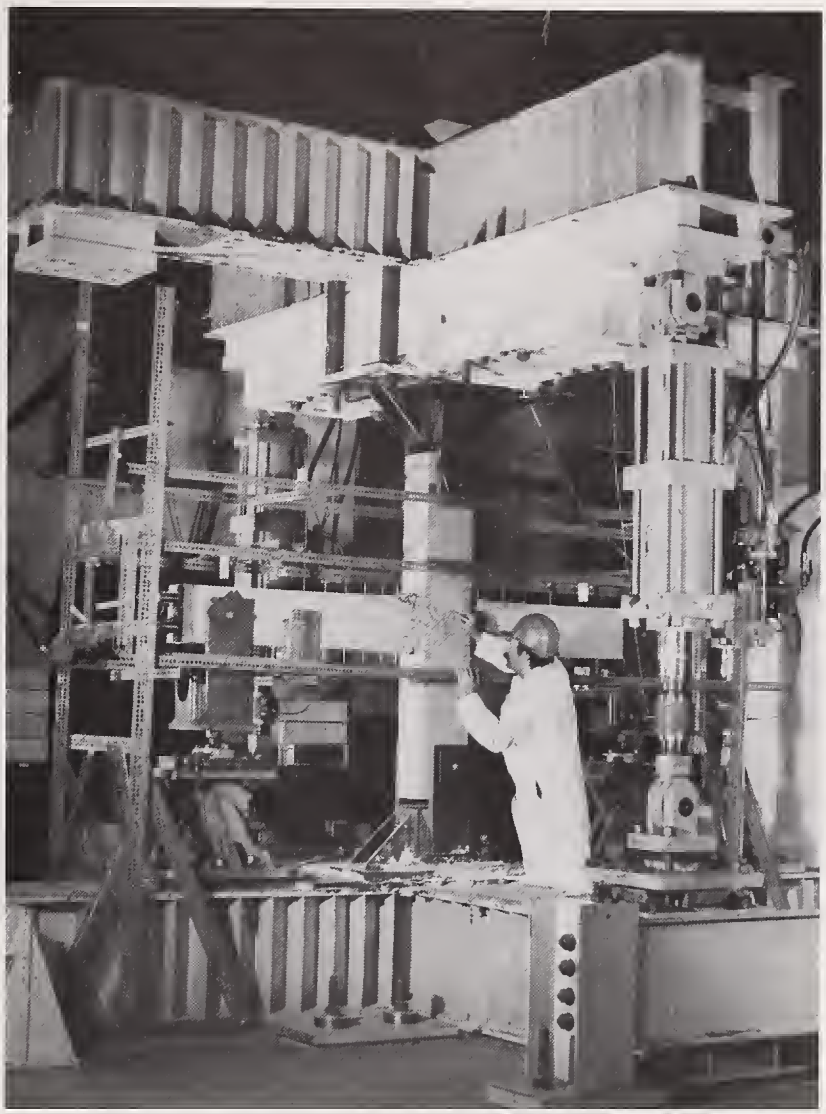

Technician Max Peltz examines cracks on a precast concrete beam-column connection being tested for strength and ductility in the NIST tri-directional test facility.

LARGE ENVIRONMENTAL CHAMBER

The large environmental chamber is 14.9 m × 12.8 m × 9.5 m high. It has an earth floor and may be excavated as needed for building construction. The chamber is one of the largest of its kind, capable of accommodating two-story houses under simulated environmental conditions. This chamber has been used for thermal performance, heating and cooling load measurements, and energy consumption studies of buildings of different kinds.

CAPABILITIES: The chamber is capable of automatically maintaining steady and/or dynamic temperature profiles from –45 °C to 65 °C and humidity from 50 percent relative humidity (rh) at 1.7 °C up to 35 °C dewpoint at 49 °C. A wider range of rh (15 percent to 80 percent) may be obtained manually. Air circulation maintains the temperature variation within the chamber to within ±1 °C. Damper-control return ducts in all four corners of the chamber permit good air distribution. Supply air is furnished by ceiling diffusers.

APPLICATIONS: The chamber is used to measure indoor temperature fluctuation, heat loss and heat gain through building envelopes, energy conservation and moisture condensation studies, and air infiltration under simulated and dynamically fluctuating outdoor temperature cycles. The chamber has been used to test a wide variety of conventional and special structures and equipment, including military hardware (such as inflatable life rafts, relocatable air-inflatable hospital units, and portable walk-in coolers) under extreme climatic conditions.

AVAILABILITY: This facility, along with several smaller chambers (including one designed for vehicles), has substantial potential for use by researchers in industry and universities. Collaborative programs and individual research can be arranged.

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CALIBRATED HOT BOX

The calibrated hot box measures the heat transfer coefficient of full-scale building wall sections. Designed in accordance with ASTM Standard C976, it consists of two large, heavily insulated chambers—an environmental chamber and a climatic chamber—each with one open side. Indoor and outdoor conditions are simulated in the chambers. The open test section measures 3 m × 4.6 m. A well-insulated frame supports the wall specimen that is clamped between the open sides of the two chambers.

CAPABILITIES: This facility is the only one of its kind designed to perform simultaneous dynamic transfer measurements of air, moisture, and heat during simulated winter and summer conditions under steady-state and dynamic climatic conditions. While the environmental chamber temperature and humidity are maintained to simulate a relatively steady and narrow range of indoor conditions, the climatic chamber can attain temperatures ranging from –40 °C to 65 °C. The apparatus measures the performance of homogeneous or composite walls having a range of thermal resistance from 0.35 m^2 to 8.8 $m^2 \cdot$ C/W. It accommodates wall specimens up to 0.6 m thick and up to 700 kg/m^2 in weight per unit area.

APPLICATIONS: NIST researchers use the facility to develop standard test methods to evaluate dynamic thermal performance of full-scale walls under cyclic temperature conditions. The building industry and government agencies are seeking reliable evaluation techniques for wall thermal mass, especially to predict energy consumption of buildings with heavy mass effects in comparison to standard wood-frame buildings.

NIST has participated in round-robin test activities with domestic and overseas thermal insulation laboratories.

AVAILABILITY: The facility provides a unique opportunity to measure simultaneous transfer of air, moisture, and heat through wall and roof specimens with openings for windows and doors. While available for use by those outside NIST, this apparatus must be operated by NIST staff.

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LINE HEAT-SOURCE GUARDED HOT PLATE

The 1-m guarded hot-plate apparatus measures thermal conductivity of building insulation. NIST researchers use the hot plate to provide calibration specimens for guarded hot plates in other laboratories. The hot plate also is used to investigate edge heat loss from thick thermal insulation materials. This facility is the only one of its kind in the world that will permit low-density thick insulation to be measured with an uncertainty of less than 0.5 percent.

CAPABILITIES: This apparatus has a test temperature range of 80 °C for the hot plates and –20 °C for the cold plates. The apparatus permits measurement of vertical and horizontal heat flow to simulate heat transfer through ceilings and walls, respectively. This apparatus operates within its own carefully controlled temperature and humidity environment shielded by an insulated aluminum enclosure. This facility provides for absolute measurement of thermal resistance of thick and low-density test specimens used as transfer standards. These standards are used to calibrate or verify heat flow meter (ASTM C508) or guarded hot-plate (ASTM C177) equipment.

AVAILABILITY: This apparatus is available for use by those outside NIST, but it must be operated by NIST staff. Collaborative programs may be arranged.

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FIRE RESEARCH FACILITIES

As the federal government's principal fire research laboratory, NIST maintains some of the country's best and most extensive fire testing facilities. A substantial portion of NIST's fire tests are performed in a specially equipped fire research building, which measures 27 m × 57 m and is designed for large-scale fire experiments. Smoke abatement equipment permits large fire tests to be conducted safely without polluting the environment.

In addition to several individual burn rooms, which are modified from time-to-time to accommodate special NIST testing requirements, the facility also houses several specially designed calorimeters for measuring the rate of heat release from materials and large samples, a room/corridor facility for studying smoke and toxic gas transport, pilot furnaces, and reduced-scale model enclosures. Also, a two-story "townhouse" is used to study fire spread from a burning room, smoke transport between levels, and sprinkler performance. The townhouse features a living room that is continuously weighed, allowing the burning rate of finish materials to be measured.

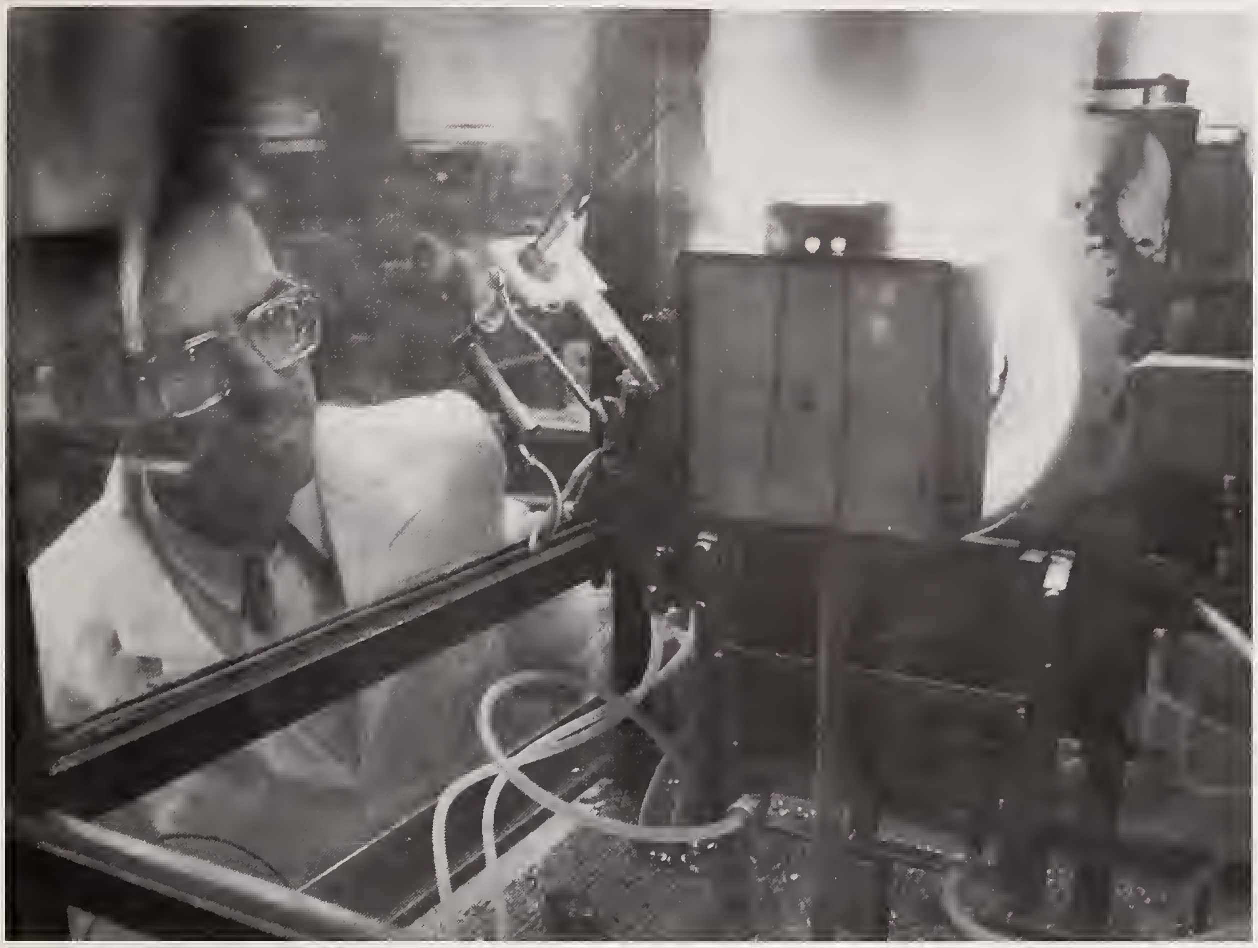
A new computer-based data acquisition system provides state-of-the-art data collection capabilities for all large-scale fire testing. Up to 300 instruments with scanning rates over 100 channels per second can be dedicated to a single test. During an experiment, real-time, full-color graphics present the data as collected, with automatic conversion to engineering units for gas analysis, rate of heat release, temperature, and other measurements.

A fire simulation laboratory, containing personal computers, state-of-the-art graphics, video and reference materials, and associated databases, is used to develop, demonstrate, and apply fire modeling computer programs.

CAPABILITIES:

■ Heat-Release Rate Calorimeters. NIST pioneered and developed the oxygen consumption methodology for measuring the rate of heat release and has used it longer and in more devices than any other laboratory. The major benefit of this technique is the independence of the apparatus in measuring enthalpy responses to changes in heat release rate.

NIST has two calorimeters available for measuring the rate of heat release of freestanding items, such as pieces of furniture. The smaller one has a capacity of ½ MW; the larger, 7 MW. Provisions for measuring smoke production and gas species yield are available with both instruments.



Fire protection engineer Vytenis Babrauskas helped develop the NIST cone calorimeter. It provides data critical to predicting the fire hazard of a product using a small sample of material—replacing time-consuming and expensive full-scale tests.

■ Room/Corridor Facility. NIST researchers have constructed and used a room/corridor facility to evaluate an analytical model that predicts the transport of smoke and toxic gases from the room of fire origin into the corridor and secondary target rooms. The design of this facility makes it possible to measure the hazards associated with the burning of wall linings or room furnishings by evaluating the rate of heat release, smoke production, and toxic gas generation. The facility is available in its present form or with design modifications for evaluating a variety of building contents and furnishings.

■ Burn Rooms. A standard burn room built to ASTM specifications, 2.4 m × 3.7 m × 2.4 m high, adjoins a large overhead hood that collects the exhaust products from the room fires. The exhaust collection system is calibrated to measure the rate of heat release and the generation rates of smoke and other combustion products from the fire. The burn room is available for developmental and validation studies of mathematical models and for studies of fire performance of furnishings and interior finish materials.

The room fire environment can be characterized in terms of temperature and pressure gradients and the spatial distribution of thermal flux, gaseous combustion products, and smoke. Other measurements permit the calculation of thermal losses to the room boundaries and mass and energy flows from the room. Smaller burn rooms also are available.

■ Pilot Furnaces. A pilot furnace for evaluating the fire endurance of wall assemblies or floor/ceiling assemblies is available. This furnace, capable of handling specimens 1 m × 1 m, may be used for research purposes only and cannot be used for code acceptance testing. Typically, fire exposure similar to that specified by ASTM E119, under carefully controlled conditions of furnace pressure and oxygen concentration, can be carried out. Depending on the parameters required, a variety of other exposure conditions can be applied.

■ Reduced-Scale Models. NIST facilities are available for reduced-scale modeling of full-scale fire configurations. Physical models offer an economical means of achieving sufficient variation of physical parameters for a generalized understanding of fire behavior. Based on the results of reduced-scale experiments, limited full-scale verification then can be performed.

■ Fire Simulation Laboratory. The laboratory contains four personal computer workstations, a high-resolution graphics workstation, a digitizing table for entering building design data, a plotter, and a printer. The workstations are connected to NIST's mainframe computers, a minicomputer, and the computerized database in the Fire Research Information Service.

Computer programs available for use and development in the laboratory address room fire growth, fire and smoke transport through buildings, building structural fire protection, escape and rescue, and sprinkler response. Special graphic display programs also are available. A database for use with some of the programs has been assembled.

AVAILABILITY: Industry, university, and government representatives are encouraged to use these fire testing facilities on a collaborative or independent basis, with certain restrictions. For safety reasons, NIST staff must closely supervise all use of the facilities.

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COMPUTER SYSTEMS LABORATORY

Both users and manufacturers of computer and telecommunications technology benefit from the work of the NIST Computer Systems Laboratory (CSL). Its research and testing programs foster the orderly development of an "open systems" environment intended to make all forms of information technology compatible and interoperable. For manufacturers of hardware and software, industry-wide adoption of standards expands marketing opportunities, and users are freed of the constraints and frustrations of incompatible proprietary systems.

Much of the laboratory's work is devoted to advising and assisting industry in developing standards that satisfy user needs and yet accommodate innovations that differentiate the products of competing vendors.

Emerging Integrated Services Digital Network (ISDN) technology will permit simultaneous transmission of digitized voice, data, text, and images over telephone lines. A CSL-created forum for North American manufacturers and users of ISDN is helping define both potential ISDN applications and their underlying technical specifications. Similar efforts are intended to refine and further the adoption of communication protocols supporting the Open Systems Interconnection model and the evolving Portable Operating System Interface for Computer Environments. In addition, laboratory personnel perform conformance tests to evaluate the compatibility of new products with these and other standards.

To help protect information in federal computer systems, laboratory personnel advise and assist federal agencies in planning computer security programs, increasing awareness of the need for computer security, and carrying out computer security training.

The laboratory also has research programs on computer security, parallel processing, automated speech recognition, database and network management, software engineering, and other topics related to the effective use of information technology.

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COOPERATIVE RESEARCH OPPORTUNITIES

INFORMATION SYSTEMS ENGINEERING

DATABASE TESTING

To aid the management of information resources, NIST researchers are developing test methods and techniques for evaluating implementations of the database language SQL and Information Resource Dictionary System (IRDS) for conformance to federal, national, and international standards. The researchers are attempting to derive a general methodology for designing conformance tests, to use this methodology to generate test suites, and to evaluate the test suites for effectiveness. A prototype implementation of the IRDS specifications, which may be suitable for such tasks as modeling the structure of a standard and for recording the parts of a standard specifically tested, will be used in this project.

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GEOGRAPHIC INFORMATION SYSTEMS

Geographic information systems (GIS) technology allows users to collect, manage, and analyze large volumes of spatially referenced and associated data. New research directions are emerging from the interdisciplinary uses of GIS. NIST researchers are exploring future GIS technology through studies into integrating computer graphics standards, database management standards, expert systems technology, and optical disk technology to support GIS applications. Their research is focused on providing GIS compatibility through standards and conformance testing for future GIS standards, such as the Digital Cartographic Data Standard. Because the activities of many governmental and private organizations are land and/or locationally related, GIS technology will be important in integrating existing spatial data for these organizations.

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COMPUTER GRAPHICS TESTING

The development of several graphics standards and work on related conformance testing and measurement techniques for graphics software are under way at NIST. Specifically, NIST researchers are testing implementations of the graphical kernel system (GKS), the computer graphics metafile (CGM), and the programmer's hierarchical interactive graphics system (PHIGS) for conformance to existing and emerging federal, national, and international standards.

Researchers are attempting to derive a general methodology for designing conformance tests, to use this methodology to generate test suites, and to evaluate the test suites for effectiveness. The computer graphics laboratory, which contains various computer graphics hardware and software systems designed to support standard specifications, is used in these efforts. An existing test suite for GKS is available with a FORTRAN interface. Priority is being given to testing methodologies and test suites for PHIGS, CGM, and the conversion of FORTRAN tests for all computer graphics standards to other languages (C, Pascal, and Ada).

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SYSTEMS AND SOFTWARE TECHNOLOGY

INTERFACES FOR COMPUTER ENVIRONMENTS

For the past several years, NIST has collaborated with vendors, users, and voluntary standards organizations to advance the implementation and use of the standard on Portable Operating System Interface for Computer Environments (POSIX). POSIX promotes the portability of computer applications at the source code level. NIST researchers continue to work with voluntary standards committees to develop additional standards needed for interfaces to operating systems, including commands and utilities, system administration, and operating system security. Research efforts focus on the operating systems functional areas of NIST's Application Portability Profile for Open System Environments, which integrates federal, national, and international standards to provide functionality needed for a broad range of government information technology applications.

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INTERACTIVE MEDIA

Portable multimedia courseware (computer-based interactive training software) provides a viable alternative to the current practice of distributing courseware with proprietary interfaces to system services. NIST and other federal agencies are working together to develop a strategy for multimedia courseware that would create an environment in which high-quality portable courseware is available as commercial, off-the-shelf products competitively supplied by vendors. Researchers are developing a computer-based interactive training applications profile that would identify needed standards in response to user needs.

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SOFTWARE QUALITY

Growing dependence on computers requires assurance that critical systems will operate reliably and exactly as intended without adverse effects, even when outside circumstances cause other systems to fail. NIST researchers are studying problems and potential solutions in building and operating high-integrity systems and are looking at life-cycle methodology, risk management, formal methods, object-oriented design, software reliability, clean-room techniques, and formal verification.

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COMPUTER SECURITY

COMPUTER SYSTEMS SECURITY

Computer systems are vital elements of today's business and scientific environments. However, as sensitive and critical information is processed and stored on computer systems often interconnected by local area networks, there is an increasing need for methods to protect that information from unauthorized access or modification. But selection of information security measures should be based on cost analysis of such measures and the resulting reduction in losses. NIST researchers are investigating various technologies that can be used to achieve additional control and security of information on computer systems. Their research involves the identification, analysis, development, and application of these technologies.

Although it is desirable to have security mechanisms as an integral part of computer systems and networks, this is not always possible or economical because such mechanisms often are not part of the original system design. NIST researchers also are examining the technology available to enhance the security of existing systems. This research involves identifying, analyzing, and comparing security mechanisms used in isolation or combination.

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SECURITY IN ISDN AND OSI NETWORKS

NIST has initiated a laboratory-based program to bring together government organizations and contractors interested in interoperability and security in the Open Systems Interconnection (OSI) computer network architecture and the Integrated Services Digital Network (ISDN) communications architecture. Researchers develop prototype systems to demonstrate the interoperability of proposed standards for OSI and ISDN using a selected set of security services. These standards are expected to be implemented in commercial applications with a broad market.

Researchers plan to develop demonstration prototypes of applications and equipment, including hardware and software, that provide one or more levels of security in an OSI and/or ISDN environment. Specifications will be developed for data formats, protocols, interfaces, and support systems for security in an OSI/ISDN environment that can be used as a basis for Federal Information Processing Standards. Users, developers, and vendors jointly can define, develop, and test systems that will provide a range of telecommunications, network management, and security services in a distributed information processing environment.

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MALICIOUS CODE AND RELATED THREATS

Operators and users of computer systems and networks are experiencing an increased number of incidents of malicious code (such as computer viruses and "worms"), unauthorized access ("hackers," for example), and similar threats. Protection from such threats requires a combination of management awareness, user involvement, and technical protection. NIST researchers are actively involved in improving each of these areas. Their research involves an understanding of both the potential technical vulnerabilities of systems and of how systems are used and administered. Advanced methods of system protection, anomaly detection, system self-audit, and related techniques are being developed. In addition, methods of rapid reaction and response to computer security incidents are being developed and coordinated throughout the federal government.

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NETWORK SECURITY ARCHITECTURE

To protect computer systems and networks, NIST is developing a comprehensive security architecture consistent with the Open Systems Interconnection (OSI) Reference Model.

Cryptographic functions are being implemented in certain OSI layers to provide data secrecy, data integrity, and peer entity authentication. The research will combine the security standards for individual OSI layers into a unified security framework. As part of this project, NIST researchers will define a common interface for cryptographic algorithms and develop a key management methodology capable of providing keys to the cryptographic functions of any layer.

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The cryptographic adapter held here by computer specialist Shu Jen Chang is one of several being evaluated for various applications by NIST computer security researchers.

SYSTEMS AND NETWORK ARCHITECTURE

NETWORK MANAGEMENT

NIST researchers are working with industry to establish a set of standards for exchanging network management information between heterogeneous management systems. Their goal is to establish a standard enabling integrated, interoperable, automated management of multivendor computer systems, routers, bridges, switches, multiplexors, modems, and provider services.

The researchers are defining protocols for exchanging management information; identifying, collecting, and specifying the format of managed objects; defining protocols to support management functions in the areas of fault, configuration, performance, security, and accounting; and implementing prototype management systems reflecting the protocols and specifications. Other research areas include network management user displays, network management applications software development, and application of expert systems to network management.

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DIRECTORY SERVICES AND DYNAMIC ROUTING

In cooperation with industry, NIST is pursuing development of commercial off-the-shelf products implementing standard distributed directory services and dynamic routing. The distributed directory services require research work in several areas, including access control, replication of information, extension of directory schemas, and distributed update. Proposed solutions to these directory problems are implemented and tested in a NIST prototype directory implementation conforming to the international directory standard (X.500).

The aim of NIST dynamic routing research is development of an international standard for the exchange of routing information between autonomous domains. NIST researchers propose and analyze a variety of mechanisms to accommodate such exchanges, and they investigate proposed algorithms through simulation models.

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AUTOMATED PROTOCOL METHODS

NIST researchers are designing tools for editing, compiling, and interpreting computer communications protocol specifications. The goal of the research is to advance the state of the art in using such tools to realize automatically executable implementation of the protocols. As part of this project, NIST researchers are developing a syntax-directed editor for Estelle and, using the same grammar, devising a portable compiler for Estelle and the supporting runtime libraries.

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ADVANCED SYSTEMS

INTEGRATED SERVICES DIGITAL NETWORKS

In cooperation with industrial and other users, NIST advances the development of standards for Integrated Services Digital Networks (ISDN), which combine voice, data, text, and image communications over a single network connection. Research in this area focuses on the measurement capabilities and testbed facilities required to develop conformance tests and performance metrics for emerging ISDN standards. Activities include support for standards writing, development of the technical foundation for implementation agreements on protocol options, and testing ISDN implementations for interoperability. NIST researchers also are involved in the research and standardization of B-ISDN-based protocols as well as FDDI conformance tests.

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PERFORMANCE OF MODERN-ARCHITECTURE COMPUTERS

NIST researchers in the area of computer-systems performance are promoting the effective evaluation and efficient use of advanced computers by the federal government. Their areas of interest include: characterization of new computer architectures to identify improved ADP technology for applications; exploration of economical programming methods that standardize across classes of architecture; and design of coherent evaluations that economically and reliably characterize the machines. Two dedicated, instrumented multiprocessors serve as special project resources. One has 16 nodes loosely coupled as a hyper-cube; the other has 16 processors in a shared-memory configuration.

These NIST researchers also are involved in the development of instrumentation and related management techniques for gigabit networks used for visualization-based applications.

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OPTICAL DISK

As industrial standards for optical disk media evolve, test methods will be needed for conformance testing of the media. NIST researchers are setting up a laboratory to develop and demonstrate data/media interchange tests and to verify conformance to established or planned national and international standards for rewritable and write once, read many (WORM) type optical disks. The program will be coordinated with voluntary standards committees and interested federal agencies.

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DISTRIBUTED SYSTEMS

NIST researchers are developing application profiles that will promote integrated platforms for video, imagery, computer data, and voice, thus defining multimedia applications in a distributed processing environment. This research focuses on prototype development and demonstration using ISDN technology.

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AUTOMATIC SPEECH RECOGNITION

Recent advances in automatic speech recognition technology have resulted in computers able to recognize correctly continuous speech with lexicon sizes of at least 1,000 words. Speech databases are typically large in size (gigabytes) and too costly for any one organization to develop. To improve the technology, the research community relies heavily on shared use of the databases and standard test methodologies.

With support from the Defense Advanced Research Projects Agency and other agencies, NIST has collected speech database material and, using CD-ROM technology, distributed this material to more than 100 research organizations. Researchers at NIST also have developed CD-ROM format speech databases that are used for speech recognition research in large vocabulary speech (5,000-word office correspondence), word spotting, speaker identification/verification, and goal-directed spontaneous speech. Among the research facilities used are a VAX 11/780, a Sun workstation, and speech-processing peripherals and software tools. Areas of interest include characterization of the speech database materials (such as acoustic-phonetic locators and classifiers), artificial neural nets, performance measurement, and natural languages.

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IMAGE RECOGNITION

Image-recognition research at NIST focuses on developing methods for evaluating image quality, compression efficiency, and image systems used in optical-character recognition. These evaluation methods are designed to include highly parallel computers and special-purpose chips as well as conventional computer architectures. The methods being developed are used for automated fingerprint recognition, automation of data entry from images of forms, and measurement of recognition systems on realistic applications. A general model of the recognition process in parallel computers is being implemented to provide better methods to analyze the performance of SIMD (single-instruction, multiple-data) computers for image compression, image quality evaluation, and recognition accuracy.

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RESEARCH FACILITIES

INFORMATION SYSTEMS ENGINEERING FACILITY

The information systems engineering facility consists of laboratories with the computer hardware and software needed for research and development of standards, guidance to federal agencies, and validation tests. The following areas are included: graphics; programming languages; database management systems; distributed database management systems; object database management systems; data dictionary systems; data administration, especially database design; data interchange; knowledge-based and expert systems; and geographic information systems (GIS).

CAPABILITIES: The facility contains a variety of computers. Among them are a VAX 11/785, Symbolics 3650 LISP machine, 386 and 486 microcomputers, Macintosh II, Silicon Graphics IRIS workstation, and a SUN SPARC II workstation. Other computer systems, such as the NIST Cray Y-MP supercomputer, are accessible. Also available are a variety of hard-copy output devices, such as laser printers, camera output systems, and color thermal transfer printers.

Software used in the facility includes:

- graphical kernel system (GKS) and programmer's hierarchical interactive graphics system (PHIGS) implementations, which allow graphics programmers to design a wide variety of graphics programs, ranging from simple passive graphics to complex real-time systems;
- computer graphics metafile (CGM) implementations that permit transfer of graphical pictures among heterogeneous graphic devices;
- a variety of programming language processors and system software;
- database management systems for the VAX, workstations, and microcomputers supporting SQL and object technologies;
- a prototype implementation of the Information Resource Dictionary System (IRDS) standard;
- LISP and Prolog;
- microcomputer expert system shells; and
- GIS systems.

Validation suites are available for testing conformance to Federal Information Processing Standards (FIPS) for COBOL, FORTRAN, Pascal, Ada, GKS, CGM, and database language SQL. Testing services are provided for COBOL, FORTRAN, Pascal, Ada, GKS, CGM, and SQL.

APPLICATIONS: An active area of cooperative work is the development and evaluation of tests to validate the conformance of language processors and other system software to FIPS. Major programs under way are: validation of the programming language processors COBOL, FORTRAN, Pascal, and Ada; validation of the GKS, PHIGS, and CGM graphics systems; validation of database language SQL; and validation of IRDS.

Possible future work includes the development of tests and procedures for validating additional programming language systems, such as VHSIC Hardware Description Language, MUMPS, C, LISP, and Prolog; for validating the computer graphics interface; and for validating the following data management and data interchange software: SQL2, SQL3, data description file for information interchange, and abstract syntax notation one (ASN.1).

AVAILABILITY: The facility, which must be used under the guidance of NIST staff, is available for collaborative projects in test development and application research.

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COMPUTER AND NETWORK SECURITY FACILITY

The NIST computer and network security facility is used to improve the current security posture of federal computer and telecommunication systems and to provide security for these systems as they migrate toward open system environments. Research done in the facility is aimed at applying methods to protect the secrecy and integrity of information in computer systems and data networks; evaluating personal identification and authentication techniques to control access to information resources; and developing computer and network security architectures to determine proper implementation of controls for integrity and confidentiality of information and authentication of users.

CAPABILITIES: The facility is equipped with mini- and microcomputers, security devices, terminals, personal identification systems, and access to large mainframes and supercomputers through local area, national, and global networks. A variety of communications technologies and applications environments are available for research efforts, including Open Systems Interconnection and Integrated Services Digital Network for developing and testing security protocol standards.

Operational capabilities include a computer emergency response team to facilitate identification and response to acute

Using an Integrated Services Digital Network software package, computer scientists Douglas White and Camie Roberts demonstrate how this technology will allow remotely located computer users to participate in voice and video conferencing.

computer and telecommunications security incidents involving self-replicating computer viruses. Test and evaluation capabilities range from specific functionality tests of cryptographic modules to test methodologies for network security protocols to the specific criteria used to evaluate the trustworthiness of systems that handle unclassified, but sensitive, data.

A risk management laboratory, utilizing a Dell System 325 (AT clone), is available for research in risk management techniques and methodologies and evaluation of risk management software to determine applicability to different agency environments. Three Sun computers support the development of advanced computer access control systems based on smart token technology. The virus laboratory uses two IBM PS/2 Model 60s, a Sun workstation for research in multiuser environments; and a Macintosh SE for research in the MacOS environment. A small systems security laboratory completes the computer and network security facility.

APPLICATIONS: The facility is used primarily to develop and test federal standards for computer and network security. Support is provided to other federal agencies and industry where the protection of unclassified data is required.

AVAILABILITY: Collaborative research programs can be arranged.

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ISDN AND DISTRIBUTED SYSTEMS FACILITY

The NIST Integrated Services Digital Network (ISDN) and distributed systems facility provides laboratories for research and development, standards, and conformance testing in distributed computer systems and advanced computer communications, including ISDN and the fiber distributed data interface (FDDI). Significant research and development programs include open distributed systems, transaction processing, distributed multimedia, ISDN applications, ISDN conformance testing, and broadband ISDN.

CAPABILITIES: The facility is equipped with microcomputers, workstations, minicomputers, multimedia display units, ISDN and Ethernet communications, and laser printers. Access to mainframes and supercomputers is provided via local, national, and global networks. The laboratories have eight basic rate interface (BRI) ISDN lines, three Sequent computers (S27, S81), five Sun workstations, four 386-class microcomputers, four ISDN terminal adapters, four ISDN telephones, two ISDN protocol analyzer/emulators, and two Macintosh microcomputers. In the near future, researchers anticipate access to primary rate interface ISDN as well as the BRI. The laboratories also feature software for compiling formal descriptions of distributed systems into implementations, tool kits for programming distributed systems, and user interfaces as well as advanced operating systems such as MACH.

APPLICATIONS: Facilities are used primarily for developing and testing federal, national, and international standards for advanced communications and distributed multimedia systems.

Research activities support the North American ISDN Users' Forum, chaired by NIST. The forum was established by NIST with industry in 1988 to create a strong user voice in the implementation of ISDN applications. Through the forum, users and manufacturers concur on ISDN applications, the selection of options from standards, and conformance tests. NIST works with the forum to develop tests that determine whether the agreed-to specifications will result in compatible products and services.

A principal focus is standards conformance test design for ISDN implementations based on CCITT recommendations. NIST has developed reference implementations for the Q.921 and Q.931 recommendations, conformance test suites for both recommendations, and a conformance test system based on these items. In addition, conformance research on broadband ISDN is conducted.

Also under development are application profiles for ISDN, research in distributed computing environments, and prototype distributed multimedia information systems based on ISDN and other communications media. Researchers have produced a distributed reference implementation for the International Standard Transaction Processing Protocol directly from a formal specification and are investigating how transactions support multimedia information access and cooperative processing.

AVAILABILITY: Collaborative research programs with government, industry, and academia can be arranged and are encouraged.

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COMPUTING AND APPLIED MATHEMATICS LABORATORY

The NIST Computing and Applied Mathematics Laboratory develops mathematical, statistical, and state-of-the-art scientific computing tools that help other NIST researchers and collaborators accomplish their research objectives.

In new initiatives focusing specifically on the needs of U.S. industry, the laboratory is developing algorithms, databases, and software for computer-aided modeling of complex phenomena, such as charge distribution in semiconductors, protein folding, and phase changes in structural materials. In the ongoing Design for Quality program, laboratory scientists and collaborators from industry and academia are developing and refining new statistical, mathematical, and computer-based models for improving and integrating the design, engineering, and manufacturing of new materials and products. NIST materials scientists and engineers are using these methods to improve the fabrication of advanced ceramics.

Other research programs focus on advanced computer graphics programs that produce two- and three-dimensional visualizations of complex problems or create images integrating data from scientific experiments. NIST-pioneered methods for displaying, manipulating, analyzing, and transmitting large volumes of data are providing researchers with new perspectives and new experimental capabilities. The laboratory also is developing software applications for harnessing the problem-solving power of parallel processors, an effort carried out in conjunction with the federal government's multiagency program on high-performance computing and communications.

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COOPERATIVE RESEARCH OPPORTUNITIES

APPLIED AND COMPUTATIONAL MATHEMATICS

SCALABLE PARALLEL ALGORITHMS

A major problem for users of massively parallel computers is the lack of libraries of commonly used subroutines. Algorithms and computer codes developed for single-processor machines rarely give massive speedups when transferred to massively parallel computers.

NIST researchers concentrating on algorithm research for massively parallel computations are making substantial progress in algorithms for important problems in computational geometry, such as triangulation in two and three dimensions. Additional research focuses on random processes, including Monte Carlo simulations, and randomizing algorithms.

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NON-LINEAR MECHANICS

Mathematical analysis, used with symbolic computation, leads to efficient analytical approximations by computers. Perturbation algorithms applied to non-linear differential equations, especially in celestial mechanics, result in analytical developments where the complexity grows exponentially with the order of the approximation. Several avenues are being explored at NIST to simplify literal developments generated by perturbation algorithms applied to non-linear systems. These include identifying algebraic structures on the domain of the normalization, smoothing transformations to eliminate perturbation terms outside the kernel of the Lie derivative, preparatory transformations of a geometric nature, and creating natural intermediaries.

Problems being examined include resonances at an equilibrium, perturbed pendulums, and the major theories of celestial mechanics. NIST researchers, using a LISP computer, are focusing their studies on algorithms amenable to computer automation through symbolic processors.

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MATHEMATICAL MODELING AND SOFTWARE

Mathematical and computational problems are becoming more elaborate as measurement techniques, physical understanding, and computational capability improve. Solving these problems requires innovative combinations of the methods of modern applied and computational mathematics. With scientists and engineers, NIST mathematicians develop and analyze mathematical models of phenomena; design and analyze computational methods and experiments; transform these methods into efficient numerical algorithms for modern, high-performance computers; and implement them in high-quality mathematical software. Active areas of interest include crystal growth, fluid flow, electromagnetic waves, magnetic materials, molecular dynamics simulations, partial differential equations, and several kinds of inverse problems.

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OPTIMIZATION AND COMPUTATIONAL GEOMETRY

In many aspects of NIST's research, the need arises for computational procedures, such as curve fitting, parameter estimation, and maximum entropy calculations. These activities require solving various types of optimization problems. NIST researchers investigate many aspects of numerical optimization, including large-scale linear and quadratic programming problems by interior-point methods, and the extension of these procedures to general large-scale non-linear programming problems via sequential quadratic programming methods. Researchers also have developed procedures for nonlinear least squares and orthogonal distance regression problems, and they produce software for these methods as needed.

Computational geometry is a rapidly emerging field with applications in robotics, statistical mechanics, cartography, computer graphics, materials science, and molecular dynamics. Researchers at NIST have developed robust and efficient computational schemes to compute triangulations and shellings of point sets, Voronoi diagrams, and other geometrical calculations. Algorithms for two- and three-dimensional triangulation have been implemented for sequential and parallel machines and are used at NIST and other scientific centers.

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STATISTICAL ENGINEERING

DESIGN FOR QUALITY

Through its Design for Quality (DFQ) program, NIST supports collaborative research within NIST and with industry, academia, and technical societies to develop technology for quality improvement. Such collaborations are aimed at meeting critical industrial product and process design needs. A typical collaboration involves industrial partners, NIST and academic subject matter experts, and NIST statisticians, mathematicians, and computer scientists. The technology developed is disseminated through industrial collaborations, tutorials, workshops, conferences, and publications. Current DFQ projects include technology development for advanced materials characterization, formulation and processing, and computer-integrated flexible piece parts manufacturing.

Materials being investigated include: silicon nitride, reinforced structural ceramics, superconducting ceramics, atomized metals, polymer composites, dental and medical materials, and artificial diamond films. The techniques of statistically planned experiments are used to understand the cause and effect relations among measurement, testing, and processing parameters; microstructural features; mechanical, electrical, and chemical properties; and performance attributes.

In the area of computer-controlled high-precision machining, statistical methods and other quality techniques are used to characterize and model those factors affecting dynamic friction, derive correction factors to compensate for backlash and temperature bias, and determine adaptive servo-control process parameters for optimal operation.

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MEASUREMENT ASSURANCE

Quality assurance for physical and chemical measurements requires assessment of uncertainties for highly precise measurement systems and development of on-line quality control strategies. NIST statisticians, confronted with difficult uncertainty problems, develop and refine methods, such as the bootstrap, for application to experiments like the recent redetermination of the atomic weight of nickel or determining confidence intervals for polarized beam measurements. Methods for developing and validating models are explored for dealing with complex error structures.

In the process control area, change-point algorithms and computer-intensive software have been applied to a materials reliability problem where a noisy sequence of welding voltages contains peaks indicating points of metal detachment. The programs were originally developed by NIST researchers to detect changes in chemical processes. Research is still needed for electronic systems where autocorrelative structure and limited knowledge of the underlying distribution of in-control measurements are at issue.

Calibrations, where instrument response depends on stimuli over a regime, is of ongoing interest. Research is focused on optimal procedures for real-life models and on improving uncertainty estimation for Scheffe-type bounds.

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SCIENTIFIC COMPUTING ENVIRONMENTS

SCIENTIFIC VISUALIZATION

Researchers at NIST are developing and applying advanced methods for using state-of-the-art, computer-based scientific graphics for rendering complex experimental, computational, and analytical results in physics and chemistry. Researchers use a collection of vector and raster workstations, photographic and video hardware, high-speed networking for transmitting large graphics data sets between computers and graphics devices, and computational geometry algorithms and software for the analysis of two- and three-dimensional data sets. Techniques also have been developed to manipulate dynamic objects in automated design and manufacturing systems; to display quasicrystal structures with icosahedral symmetries and scanning electron tunneling microscopy data with polarization analysis; and to study models of turbulent combustion showing the dependence of solutions on time and fuel-oxidizer mixture parameters.

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