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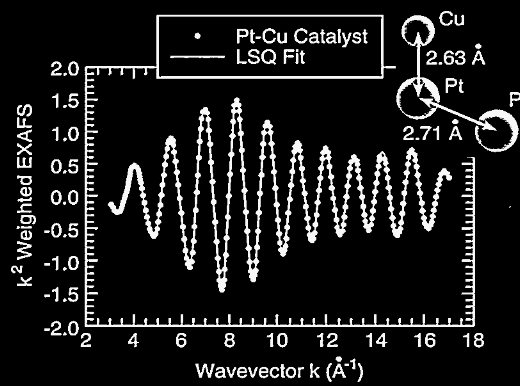

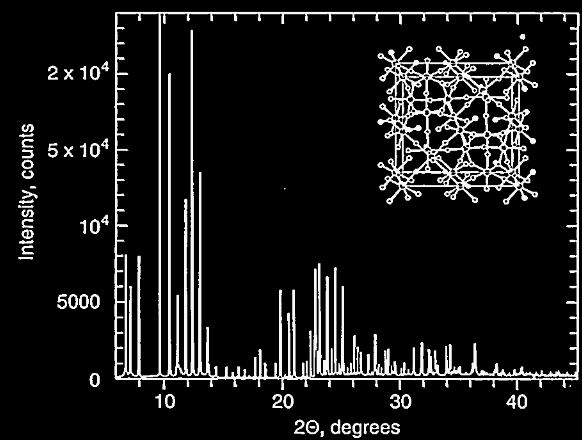

RESEARCH BY INDUSTRY AT THE NATIONAL SYNCHROTRON LIGHT SOURCE

MAY 1995

BROOKHAVEN NATIONAL LABORATORY

Associated Universities, Inc.
Upton, New York 11973-5000

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UNITED STATES DEPARTMENT OF ENERGY

The world's foremost facility for scientific research using x-rays and ultraviolet and infrared radiation is operated by the National Synchrotron Light Source Department. In a single year, a total of about 2,750 researchers from almost 425 institutions perform experiments at the world's largest source of synchrotron light. Guest researchers often work in collaboration with staff scientists at the Light Source, conducting a wide range of innovative experiments in physics, chemistry, biology, materials science and various technologies.

National Synchrotron Light Source Department

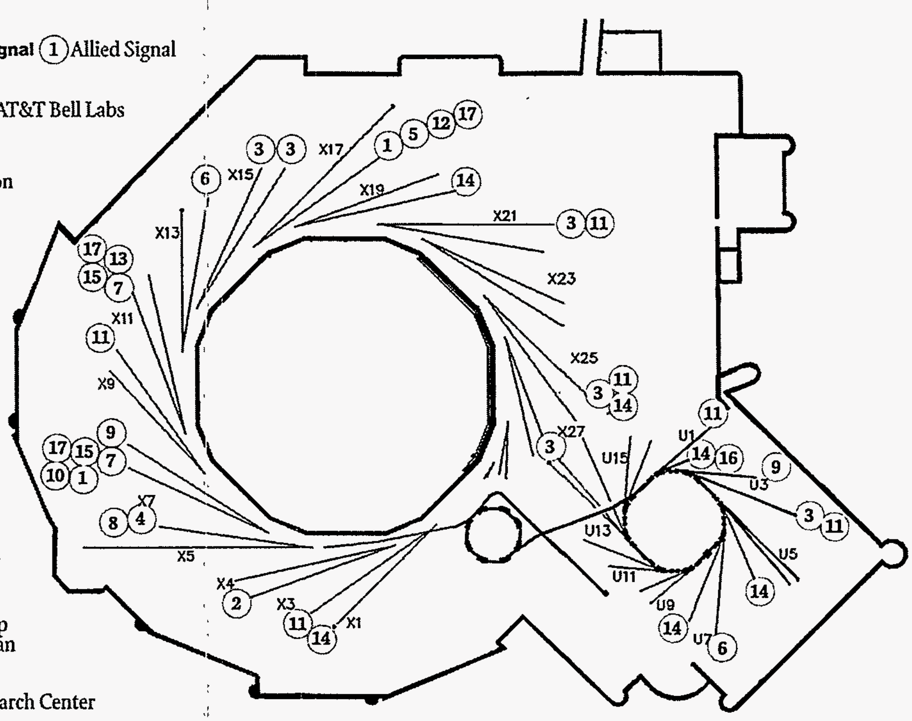
A Beacon for Industry

Since its commissioning in 1982, the National Synchrotron Light Source (NSLS) has been a beacon for industrial researchers and an important tool for facilitating technology transfer.

A source of light that is as much as 10,000 times brighter than conventional laboratory-generated beams, and a source of intellectual inspiration where hundreds of

Corporations that contributed to PRTs at Brookhaven National Laboratory's National Synchrotron Light Source

1. Allied Signal
2. Amoco
3. AT&T Bell Labs
4. BP America
5. Chevron
6. Dow Chemical
7. Du Pont
8. Eastman Kodak
9. EG&G Energy Measurements
10. Engelhard
11. Exxon Research and Engineering Co.
12. GTE
13. Hoechst Celanese
14. IBM
15. Mobil Research and Development Corp.
16. Northrop Grumman
17. UOP Research Center



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researchers from a range of institutions exchange ideas each working day, the NSLS is a powerful agent for transforming both basic and applied research into technological progress.

From AT&T Bell Laboratories to Upjohn Company, industrial giants as well as smaller entrepreneurial companies have been attracted to the Light Source to perform research on the 85 beam lines that surround its vacuum ultraviolet and x-ray rings. Today, some 350 industrial scientists from 70 companies are pursuing research that may lead to future innovative technologies.

Industry's Investment

Most of the beam lines are built by consortia of scientists from industry, universities and government laboratories called Participating Research Teams (PRTs). These scientists can use up to 75 percent of the available beam time to further their own research, with the research programs reviewed by panels of experts every three years. The remaining 25 percent is available for individual investigators through a peer-reviewed general user program. Beam time at the NSLS is free for all users unless their work is proprietary. In that case, a full-cost recovery fee is charged.

PRTs design the instrumentation at their beam lines to fit their individual needs. PRTs to which industry has contributed are listed on the previous page. Collectively, these 17 companies have invested more than $40 million and roughly 400 person-years of labor to design and install their own experimental equipment at the NSLS.

Pharmaceutical companies that use the National Synchrotron Light Source:

Bristol-Meyers Squibb

DuPont Merck Pharmaceutical Co.

Eli Lilly and Company

Genencor International, Inc.

Genentech, Inc.

Glaxo Inc.

Hoffmann-LaRoche Inc.

Merck & Co., Inc.

Miles, Inc.

Monsanto Company

Procter & Gamble Company

SmithKline Beecham Pharmaceuticals

Sterling Winthrop Inc.

The Upjohn Company

Advanced Analysis

For research that ranges from designing catalysts to developing computer chips, industrial researchers are drawn to the NSLS because it provides advanced analytical capabilities that are not available at their home laboratories. They study the absorption and scattering of x-rays at the NSLS to determine such properties as crystalline structure and magnetic characteristics of various materials. Such basic research may lead to practical outcomes, such as new pharmaceuticals and improved recording devices, or it may provide useful clues for solving problems in industrial processes.

For example, industrial researchers use a technique called x-ray diffraction, in which they measure the way an x-ray beam is deflected away from a sample, to determine the structure of crystals and monitor how that structure changes during industrial processing. The technique also can be used to detect defects in semiconductors. In addition, pharmaceutical companies use x-ray diffraction to understand how the arrangement of atoms in a drug molecule influences the way it works, so that they can design new drugs.

To keep up with the demand for beam time from structural biologists from pharmaceutical companies and academia, construction has started on an NSLS structural biology addition. When complete, it will house eight labs and a conference room. A summary of companies that have used the NSLS for pharmaceutical research is at left.

A Variety of Techniques

For materials that are difficult to study using x-ray diffraction, researchers can investigate local structural information using absorption spectroscopy, a technique in which they measure the absorption of x-rays by a sample. For instance, the local environment around the active species in a catalyst, which might affect its functioning, can be detected in this way.

Another technique that uses x-ray absorption, called microtomography, allows researchers to obtain three-dimensional images of the internal structure of visually opaque, heterogeneous materials. With the aid of this technique, for example, the oil industry can gain crucial information on oil flow through sedimentary rocks.

When a sample absorbs x-rays, electrons are emitted from its surface. Analyzing the behavior of these electrons can provide data on a sample's electronic structure, which is valuable in many industrial fields, most notably, the semiconductor industry.

New Discoveries

Also, synchrotron x-rays can change the structural properties of materials to make them industrially valuable. The best example is IBM's use of x-ray lithography at the NSLS to irradiate and chemically etch plastic-coated semiconductor wafers, thus producing some of the world's most advanced computer chips. In a new application of lithographic techniques, BNL and University of Wisconsin researchers are making micromechanical components that hold promise for many industrial applications.

In another recent endeavor, industrial researchers are attempting to make novel materials with the aid of synchrotron x-rays. For example, using synchrotron radiation to assist normally unfavorable chemical reactions in materials synthesis can yield products that are important for industry.

As the NSLS matures, still more innovative techniques for exploring materials are likely to be developed and new experimental capabilities will be discovered, undoubtedly leading to further industrial progress.

Industrial Research — A Sampling

From creating new catalysts to shrinking the size of computer chips, the wide variety of industrial research at the National Synchrotron Light Source (NSLS) reflects the diverse interests of the American marketplace. The following are a few typical examples:

Catalysis

Catalysts are vital to many industrial processes, as they drive the conversion of raw materials into useful products. Even small improvements in a catalyst's efficiency can lead to large savings in production. Thus, several of the nation's largest petroleum and chemical companies are using the NSLS for catalyst development.

For example, Exxon Corporation researchers use x-ray absorption spectroscopy to determine the geometric and electronic structure of catalysts. Solid, liquid or gaseous samples can be studied by this technique.

Recently, Exxon researchers have examined catalysts that contain tiny bimetallic clusters. By varying the surface composition of these catalysts, the researchers can alter the mix of chemical products as well as change the rates at which they are made. Such investigations may lead to more economical or environmentally safer gasoline.

Pharmaceuticals

At present, 14 pharmaceutical companies are working at the NSLS to develop novel drugs for treatment of a broad spectrum of diseases. Their approach, called structure-based, or rational, drug design, often involves designing molecules called inhibitors that attach themselves to enzymes, thereby blocking the action of disease-causing agents. To find effective inhibitors, the researchers use a technique called x-ray crystallography to determine the structure of an enzyme when a candidate inhibitor is attached.

Several companies — among them, SmithKline Beecham, Monsanto and Upjohn — are investigating the structure and functioning of the HIV protease, an enzyme associated with AIDS. Other research runs the gamut between attempting to find a cure for the common cold to designing new drugs for treating arthritis, hypertension and depression.

Environmental Assessments

Several teams at the NSLS are working on solving environmental problems. For example, soils in some regions of the U.S. have been contaminated with lead from mining, paints, leaded gasoline and industrial activities. To assess lead pollution, researchers from Du Pont are using x-ray absorption spectroscopy at the NSLS to determine the various types of lead found in contaminated soils.

One striking finding is the attraction of lead in soil for sulfur. When the two elements are combined, the resulting lead sulfide is highly insoluble, making it extremely unlikely that it would enter drinking water supplies. Such research can be used to make decisions concerning public health.

In another case, BNL researchers, with scientists from the Savannah River Ecology Laboratory and the University of Chicago, are using x-ray absorption spectroscopy to study the specific oxidation states of toxic metals and radioisotopes in contaminated soils at U.S. Department of Energy nuclear-processing facilities. Using an x-ray microprobe to determine how the oxidation varies within the sample, the scientists collect data on the extent of toxicity in the soil and the behavior of atoms in it.

X-Ray Lithography

A dramatic example of technology transfer at the NSLS came in 1988, when IBM announced that it had used x-ray lithography at the NSLS ultraviolet ring to make some of the world's most densely packed computer chips. With only 0.5 microns between components, the test chips were approximately four times denser than any mass-produced chip of the time. Such density promises vast increases in chip power and speed.

Based on this success, IBM has since built its own synchrotron storage ring in East Fishkill, New York, where chips are now consistently made with features as small as 0.25 microns. The company is now working to bring this technology to the marketplace.

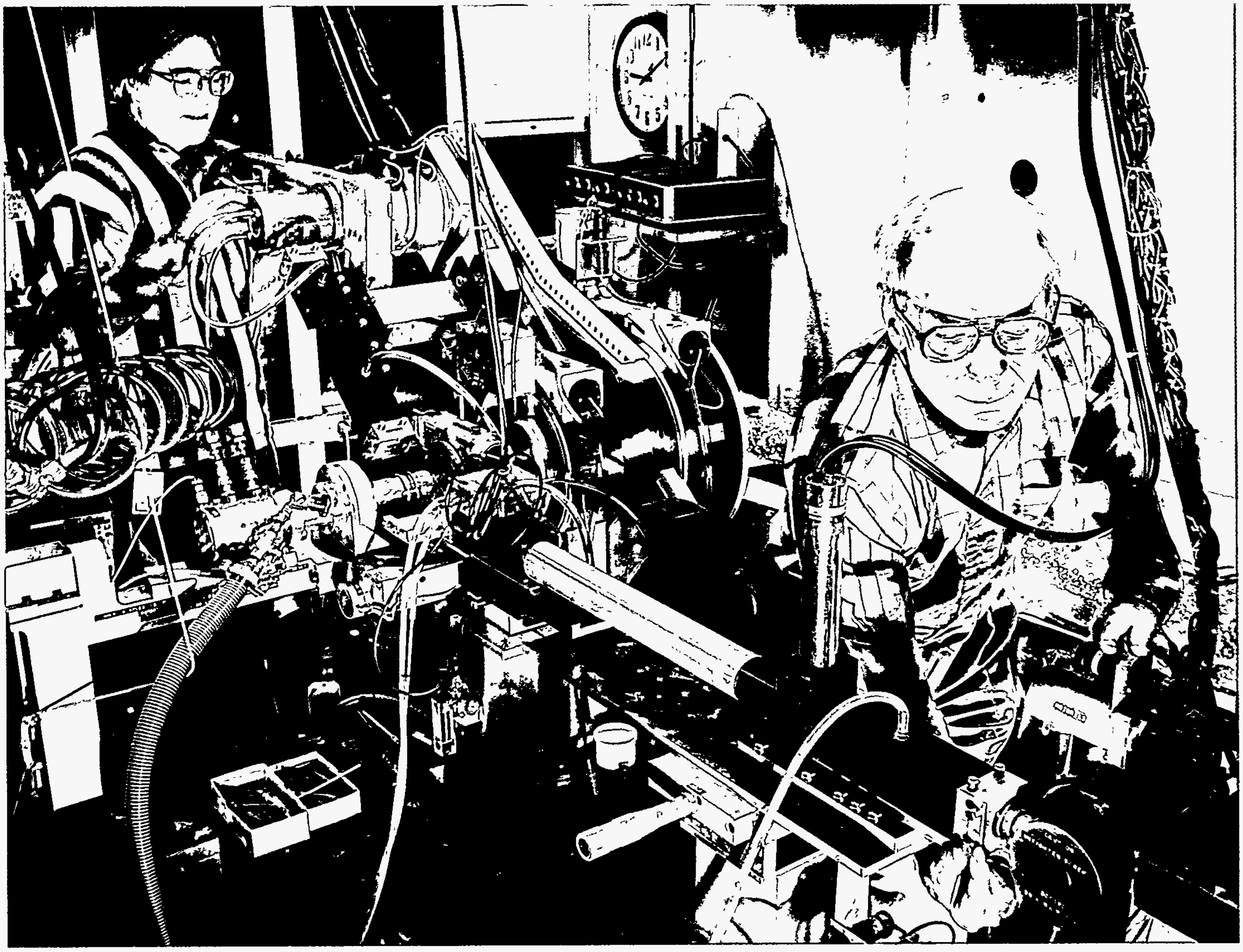
As the technology progresses, the number of circuits on chips is expected to increase approximately one hundredfold, making computers even faster and more powerful.

Elsewhere at the NSLS, AT&T Bell Laboratories is developing a technique called extreme ultraviolet projection lithography. This technique uses longer-wavelength light than IBM's shadow-mask x-ray lithography. In the AT&T method, which is far from the production stage, features as small as 0.05 microns have been imaged.

Another type of lithography — one that uses high-energy x-rays — is being developed by BNL and University of Wisconsin at Madison researchers to produce mechanical components that are several inches in size but match the precision of micromachines. These tiny mechanical components are used, for example, to make fiber-optic devices and electric motors the size of a pinhead. Successful development of this new lithographic technique into a full manufacturing process would expand micromachine applications and thus give the U.S. a competitive edge in the precision-machining area.

Medical Technology

The NSLS is at the forefront of developing promising medical technologies. Among them is transvenous coronary angiography, a technique that produces research-quality images of human coronary arteries. Since this angiography procedure requires catheterizing a vein, rather than a plaque-clogged coronary artery as in the conventional method, risk to the patient is reduced.



Before collecting powder diffraction data on a catalyst, Richard Harlow (left), DuPont Corporation and David Cox, BNL, make adjustments to the beam slits at beam line X7A.

An estimated one million angiographic procedures are performed annually in the U.S. to assess atherosclerotic disease, which is the number-one cause of death in the country. To make transvenous angiography more widely available, BNL scientists are currently working with two industrial partners — Advanced Acoustic Concepts, Inc., and Science Research Laboratory, Inc. — to improve the image-display system and to produce a compact source of high-intensity x-rays for hospitals and clinics.

Polymers

Polymers, chemical compounds made of giant molecules, are widely used in such products as plastics, nylon, rubber and vinyl. A new technique to study polymers at high spatial resolution has been developed at the NSLS by North Carolina State University and the State University of New York at Stony Brook in collaboration with Dow Chemical Company, Du Pont and Exxon.

Called x-ray absorption spectromicroscopy, the technique provides 50-nanometer resolution with little radiation damage to the sample. The new method gives information on the spatial distribution of chemical compounds and the orientation of specific chemical bonds in polymers. These data can help researchers to make new polymeric materials with such desirable properties as biodegradability, strength and durability.

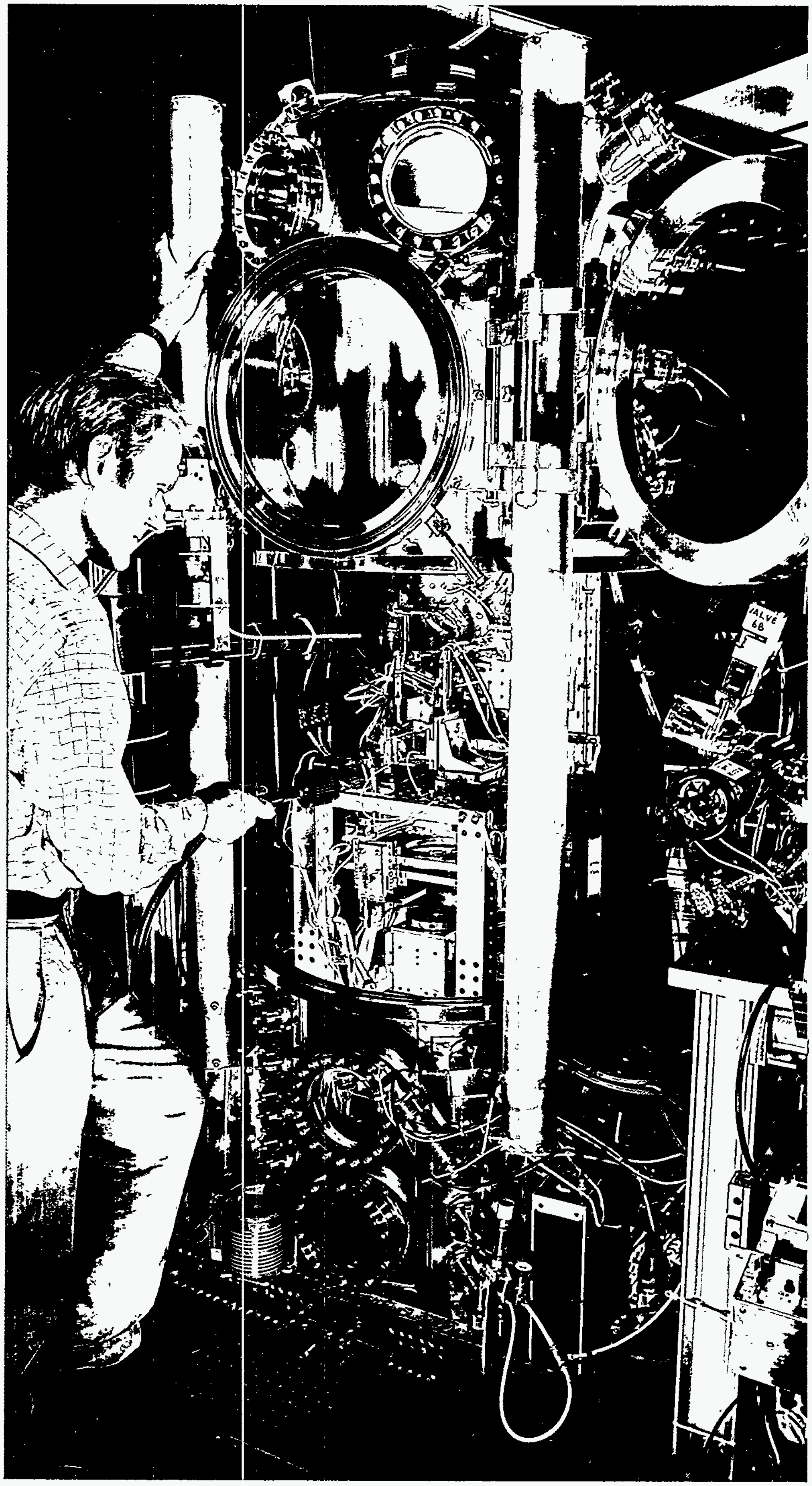
In other polymer-related research, Dow Chemical has used the NSLS to develop a unique nonadhesive coating that requires no cleaning, which may be used on a wide range of surfaces, from kitchen countertops to airplane wings. Instrumental to this discovery were data acquired from spectroscopic techniques used at the NSLS in collaboration with the National Institute of Standards and Technology to study the surface structure and composition of the material's constituent polymers. The new coating is made of both fluorocarbon and hydrocarbon molecules, which occur as parts of long polymer chains.

Microelectronics

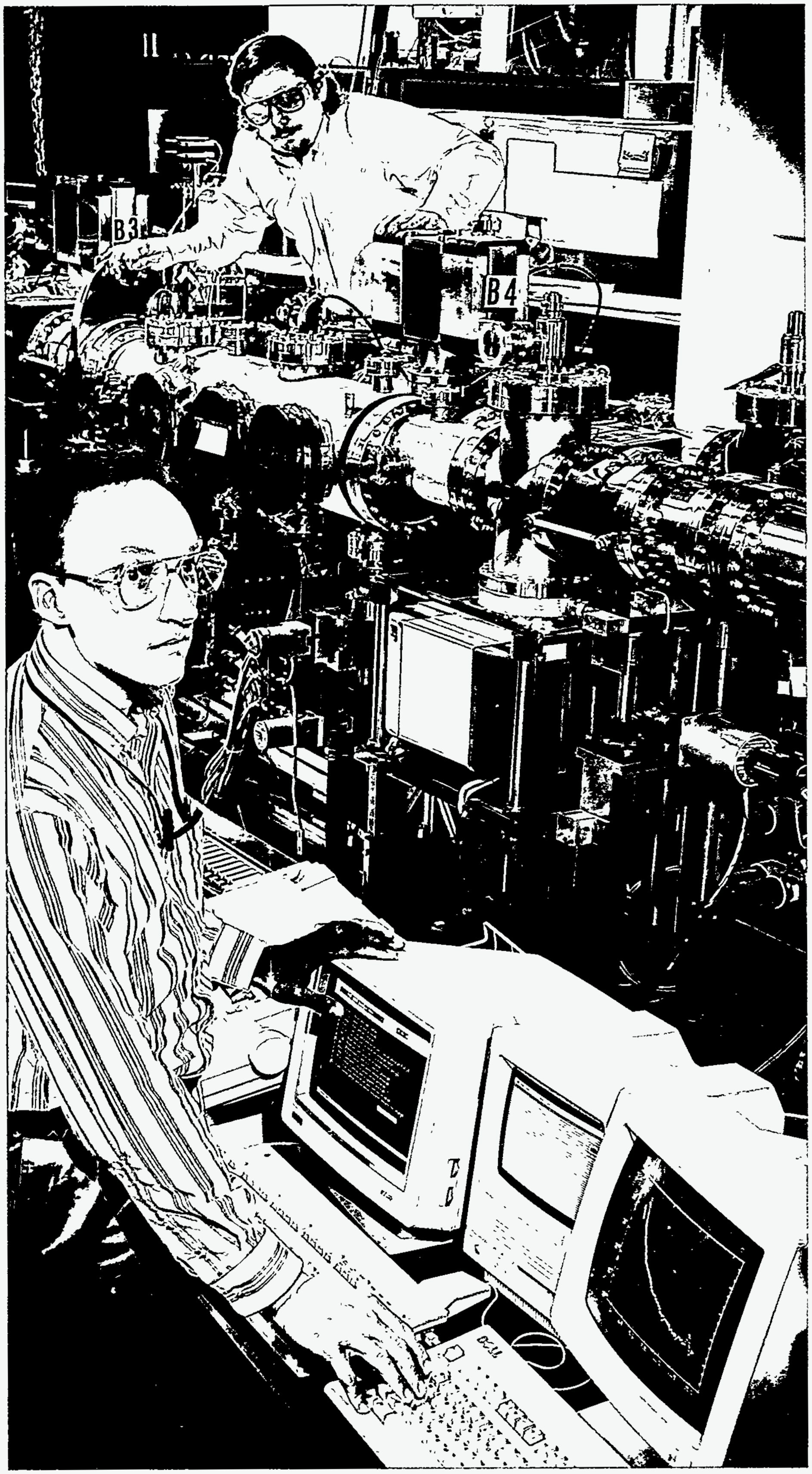
Just as the microelectronics industry continues to build computer chips with increasing densities, the communications industry designs more and more sophisticated fiber-optics systems to carry information from one computer or telephone to another.

Companies like AT&T Bell Laboratories and IBM come to the NSLS to study the atomic and electronic structures of semiconductors and semiconductor devices. As the feature sizes become smaller on computer chips, the required power densities increase. This in turn leads to new effects, which must be understood in order to design the next generation of chips.

Scientists from IBM study the dynamics of electrons moving through semiconductors at these high-power densities and compare the results with model calculations.



Alastair MacDowell, AT&T Bell Laboratories, characterizes optics needed for extreme ultraviolet projection lithography on beam line X13UB.



AT&T scientists investigate how thin films of aluminum that connect semiconductor devices deform under conditions of high-power density by a process called electromigration.

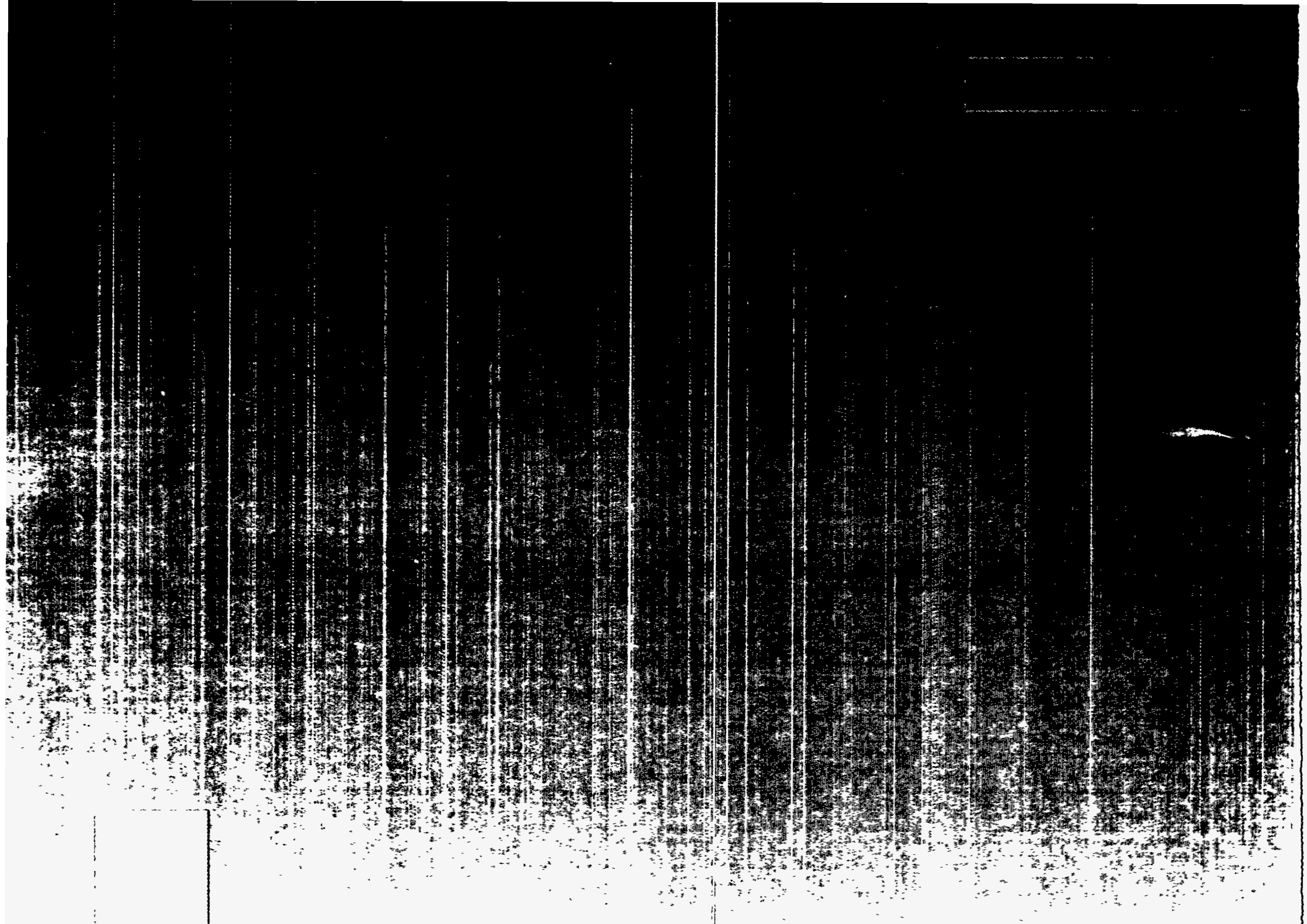
Defects and impurities play a critical role in semiconductor device performance. Scientists from the State University of New York at Stony Brook in separate collaborations with Northrop Grumman Corporation and the U.S. Army's Harry Diamond Laboratory are using a technique called x-ray topography at the NSLS to determine the types and densities of dislocations in semiconductor crystals.

Magnetic Recording

At least 25 institutions conduct research at the NSLS on magnetic materials, as they are important for developing state-of-the-art recording devices. Techniques developed at the NSLS since the 1980s have enabled researchers to probe magnetic thin films with better accuracy and precision than ever before.

Researchers from the Naval Research Laboratory are using polarized x-rays at the NSLS to probe the structure of thin magnetic films of terbium iron, the mainstay of certain computer storage devices. The direction of magnetization in these films can be made to align perpendicularly to the film's plane, which permits more efficient data storage. Studies at the NSLS revealed that the perpendicular magnetization is related to subtleties in the arrangement of the iron and terbium atoms. Such research is essential for the design and fabrication of more powerful storage devices for computers.

At beam lines X10B and X10C are Exxon researchers Brian DeVries (front) and Michael Sansone.



Cover Figure Left
EXAFS Study of Bimetallic Pt-Cu Catalysts
Pt L_3 edge EXAFS oscillations and non-linear least squares refined fit of Pt-Cu bimetallic catalyst on an activated carbon support. This catalyst system is being investigated for possible use in chlorofluorocarbon degradation by The Dow Chemical Company on beamlines X19A and X23A2. The quantitative results indicate that true bimetallic Cu-Pt particles are formed with each Pt metal atom surrounded by approximately five Pt atoms at a 2.71Å distance and five Cu atoms at a 2.63Å distance.

Cover Figure Right
The Crystal Structure of a New Form of AlF_3 as Determined from its Synchrotron Powder-Diffraction Pattern
Aluminum fluoride phases are established catalysts in many classes of fluorocarbon transformations. A new form of AlF_3, the *theta* phase, was prepared by a synthetic route (thermal decomposition) which strongly suggests that no single crystals of this phase would be forthcoming. Thus, its structure had to be determined using powder diffraction data obtained by a team from DuPont and the BNL Physics Department at beamline X7A. The structure is depicted in the inset and consists of corner-shared AlF_6 octahedra assembled into 3-, 4-, and 5-rings. The 5-rings form an undulating 3-D interconnected channel system around tetrahedral clusters consisting of 4-Al and 6-F atoms.