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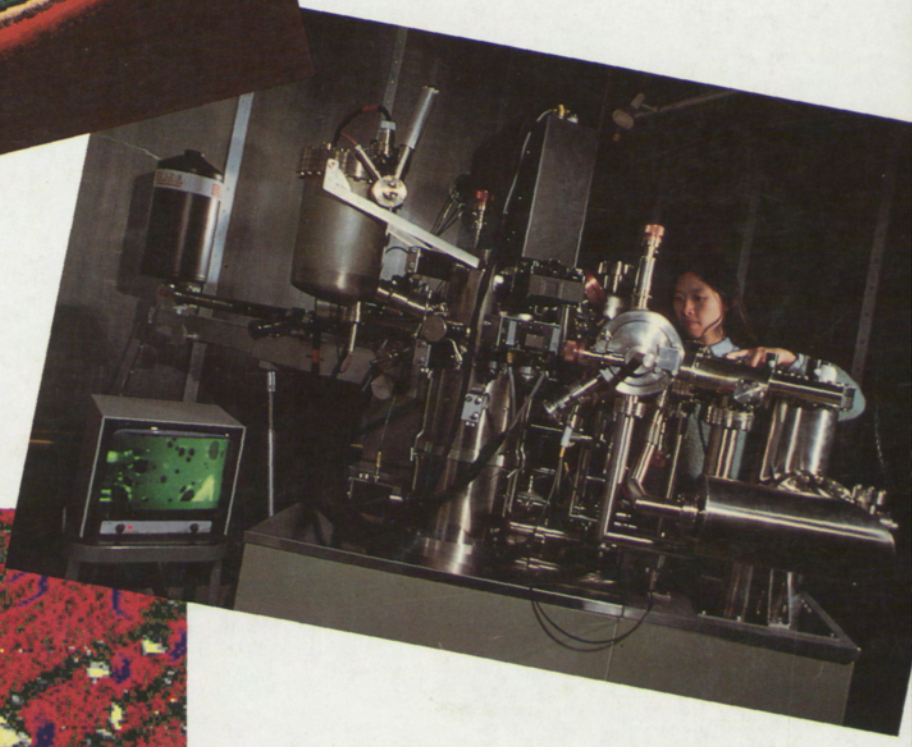

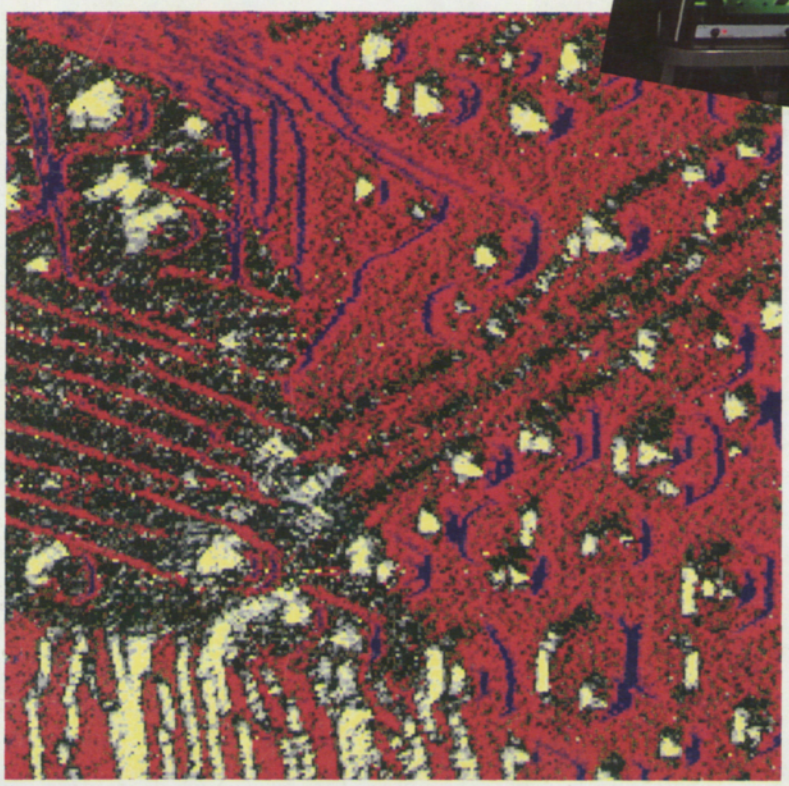

The Materials Science Center

Cornell's Premier Interdisciplinary Laboratory

Cover illustrations:

The image at the top is a false-color contour map of the low-angle electron diffraction pattern from crazes produced in a polystyrene sample deformed at 34°C. The pattern is one of a series that reveals changes with temperature. The patterns were recorded by graduate student Philip Miller using a 200 kV electron microscope in the Electron Microscopy Facility of the Materials Science Center; they were converted to color maps by Donald Buckley, also a graduate student. This research is directed by Professor Edward J. Kramer.

The photograph shows Peirong Xu, who completed her doctoral work this past summer, inserting a silicon sample into the UHV STEM (scanning transmission electron microscope). Xu worked with Professor John Silcox.

The false-color image below shows the surface of a gold specimen. Taken with a scanning tunneling microscope (STM), it is one of a series showing motion of atomic steps on the surface. The area shown is 1,000 angstroms across. This image was recorded by David R. Peale, a graduate student in Professor Barbara Cooper's research group.

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THE MATERIALS SCIENCE CENTER

Cornell's Premier Interdisciplinary Laboratory

by John Silcox

". . . scientists with a strong disciplinary base who are able to communicate and collaborate across disciplinary boundaries have become key to progress in materials research and development."

Properties of materials limit the development of advanced technologies. This became obvious after the launch of the first Sputniks in the late 1950s, when the education of scientists capable of developing improved materials became a national priority. And because many advances in materials require the participation of chemists, physicists, materials scientists, electrical engineers, and other specialists, scientists with a strong disciplinary base who are able to communicate and collaborate across disciplinary boundaries have become key to progress in materials research and development.

At Cornell, the Materials Science Center was established in 1960 as one of a series of federally sponsored interdisciplinary laboratories devoted to the study of materials. These laboratories were provided with the equipment and staff needed for first-class research and graduate education, and as a result, the host universities were able to carry out the primary function of training graduate students to participate effectively in the interdisciplinary environment at leading industrial laboratories.

The Status of Materials Research in the Nation and at Cornell

In the ensuing years, the priority given to the education of materials scientists waned. For example, nondefense federal funding declined in real terms by 21 percent between 1976 and 1987, as documented in the report of a 1989 study carried out for the National Academy of Sciences by Praveen Chaudhuri of IBM Corporation and Merton C. Fleming of the Massachusetts Institute of Technology[1].

This report also documents the outstanding successes of research in extending desirable properties of materials. For example, strength-to-density ratios have improved by a factor of fifty; the operating temperatures of engines have climbed from the 100°C of early steam engines to well over 1200°C for the modern turbojet; the speed of cutting tools has climbed from 10 meters per minute for plain carbon steel to 1,000 meters per minute for diamond-tipped tools; and the magnetic strength of permanent-magnet materials has increased one-hundred-fold. More familiar examples include the relentless shrinkage in the size of electronic components on semiconductor chips, and the development of high optical transparency in optical fibers for communication systems.

At the heart of these developments, the study indicates, is a deepening understanding of materials—an understanding that provides a basis for achieving tighter and tighter control of properties under performance conditions. Intellectual triumphs such as the discovery of the quantum Hall effect, the development of scanning tunneling microscopy and electron microscopy, and the development of new high-temperature superconductors were recognized with Nobel prizes in 1985, 1986, and 1987. Such discoveries fuel further advances in the control of materials properties. The report also points out that the development of materials has a significant impact on a number of industries important to overall economic health and United States competitiveness.

In the wake of this study, interest in materials is again reviving on the national scene. The Federal Coordinating Council for Science, Engineering and Technology (FCCSET) is responsible for establishing federal policies for the development of coherent scientific programs where the need is judged appropriate. Should there be any in materials? If so, what form should they take? Much advice is being sought and given, and senior officials, including the president's science adviser, Allan T. Bromley, are reviewing the situation. A final resolution appears unlikely, however, until the 1993 federal budget becomes public. Never-

[1] *Materials Science and Engineering for the 1990s: Maintaining Competitiveness in the Age of Materials.* Washington, D.C.: National Academy Press, 1989.

theless, the high level of interest and concern attests to the significance that materials research has for the scientific and technological enterprise and shows why the Cornell Materials Science Center (MSC) has flourished as an interdisciplinary unit since 1960.

Through the Division of Materials Research, the National Science Foundation (NSF) currently provides the Cornell center just under $5 million annually (contingent on the results of three-year competitive renewal proposals and site visits). Roughly one-third of these funds goes to support a system of central facilities and the remainder is intended to support faculty research in ways that complement the normal single-investigator mechanisms by providing funding for projects that fit into a broader pattern. Interactive group research that tackles problems requiring a mix of skills and expertise is encouraged. Of course, strong individual investigator programs provide the necessary base for such research.

How the Materials Science Center at Cornell Operates

This issue of *Cornell Engineering Quarterly* provides a picture of how MSC operates. Key features are the provision of well-equipped and well-staffed specialized laboratories, and an organizational structure that promotes the development and carrying out of cooperative and collaborative projects.

How the program of an individual faculty member can fit into a pattern providing a coherent thrust in an important area of materials science is seen in the article by Edward Kramer, who discusses the MSC study group in polymer research. The encouragement and sponsorship of study groups is one of the effective strategies of the MSC.

An alternative approach is the provision of "seed" funding to get a promising research project started. Such funding enabled Chemistry Professor James M. Burlitch to invent methods for preparing silicate ceramics, and as the article by Electrical Engineering Professor Clifford Pollock shows, this is now developing into a coherent program involving groups in several fields. One of these groups is headed by Rüdiger Dieckmann, a professor in the Department of Materials Science and Engineering; he and his associate Roland Geray describe their contribution to the research effort in another article in this issue.

An article by Jean Lee provides a graduate student's view of how the infrastructure provided by the central facility works for those in the front line. Articles describing the facility system are also included.

Decisions on MSC policy questions and on annual allocations of resources to research projects are handled by an executive committee that includes six elected faculty members, all active in research. Chosen by college or departmental constituencies rather than by study groups, these faculty members provide a perspective across a broad range of science. This provides a collective responsibility that is independent of the study groups and it facilitates the adoption of new directions and thrusts. Department heads and directors of the relevant national centers at Cornell also attend the committee meetings, but do not vote.

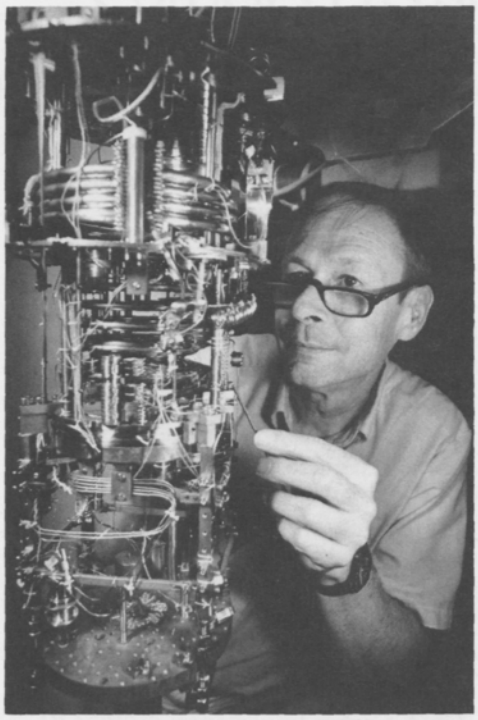
The Growing Importance of Computational Tools

The recent advances and future promise of computational materials science are of special note. With the great increases in computational speed and memory that have occurred over the past two decades and are expected to continue, the customized design of materials (see *Business Week*, July 29, 1991) may be nearing reality. Solid-state physicists have been demonstrating a masterful understanding of perfect-crystal properties for many years. Real materials, used in practical devices, often contain defects, such as dislocations, grain boundaries, and interfaces that must be considered and sometimes even specifically exploited.

Computers are beginning to have enough power to cope with such complications, and suitable software is being developed. Relationships between elastic properties as calculated by specialists in theoretical mechanics may soon be related to the bonding properties of interfaces as observed by electron microscopists, measured by x-ray diffractionists, and calculated by theoretical solid-state physicists.

At Cornell, computer-intensive research is aided by easy access to supercomputing facilities provided through the Cornell Theory Center. It seems likely to become an increasingly important aspect of MSC activity.

"Key features are the provision of well-equipped and well-staffed specialized laboratories, and an organizational structure that promotes... collaborative projects."



In the MicroKelvin Laboratory, the low-temperature apparatus is checked by Robert C. Richardson, professor of physics and director of the university's Laboratory of Atomic and Solid State Physics. The apparatus can reach temperatures of nearly –459.67°F.

Cooperative Programs with Outside Groups

When a clear joint interest can be developed, the MSC fosters collaboration with researchers from industrial and government laboratories.

An example of industry collaboration in interdisciplinary research through the MSC is provided by James P. Sethna in his article in this issue. He describes a collaborative venture in which IBM has furnished Cornell with a number of RS-6000 workstations, and Cornell faculty, staff, and students are developing software to make the workstations valuable and useful in materials research.

Another example is the formation of the Polymer Outreach Program that followed the establishment of the MSC Polymers and Polymer Composites Study Group (discussed in Kramer's article). This outreach program provides the faculty with a collective approach toward developing stronger interactions with outside researchers. Six effective collaborations have been established since the program was started in May 1990, and interest in it is growing. Features include direct interaction between industrial bench scientists and MSC research teams; short-term industrial residencies at Cornell; graduate traineeships and student externships; short courses; and an annual symposium.

The Interdisciplinary Focus and Approaches

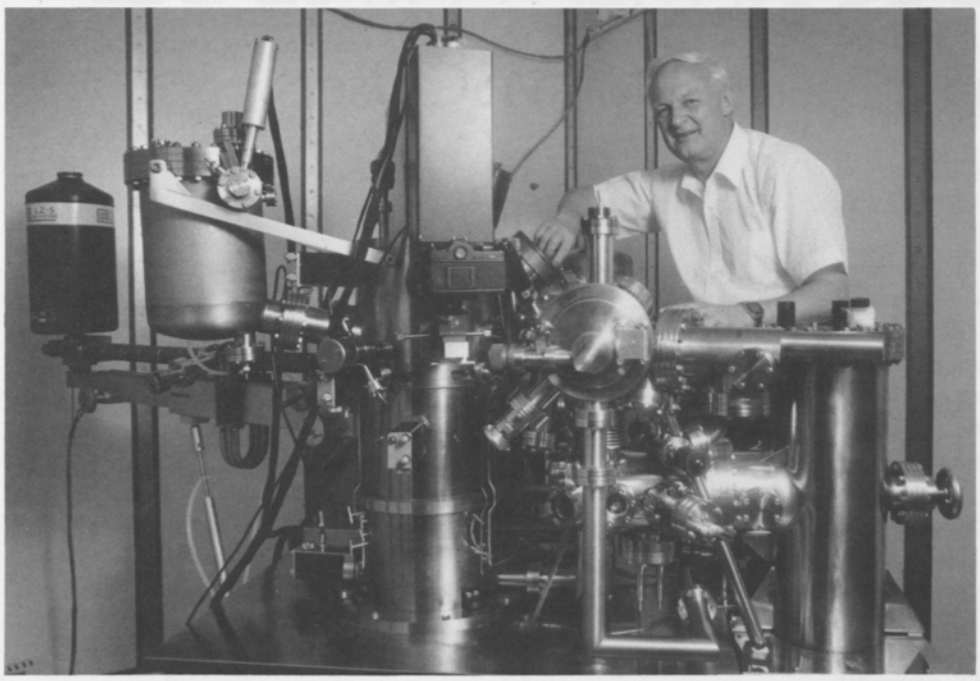
Since all of the center's projects and activities cannot be included in the space available in this issue of *Cornell Engineering Quarterly*, I want to mention some additional examples of interdisciplinary cooperation brought about through the MSC.

Besides the study group on polymers discussed in the article by Kramer, there is an active group in the area of surfaces and interfaces and one in quantum phenomena.

One of the studies by the surfaces and interfaces group is concerned with developing an understanding of the chemical reactions between gases and solid surfaces. On the one hand this involves sophisticated, laser-based work in gas-phase chemistry, and on the other hand it involves superfast electron energy loss studies of the excited states of surface-bound atoms and molecules. Chemistry Professor Paul Houston and Physics Professor Wilson Ho are just beginning these investigations, and equipment and students are going back and forth between Baker Laboratory and Clark Hall.

Another project by members of this study group makes use of the Cornell High Energy Synchrotron Source (CHESS), a national laboratory that provides access to intense x-radiation generated by the Cornell Electron Storage Ring. The spatial locations of atoms at surfaces are a key point of interest in a collaborative project led by Applied and Engineering Physics Professor Boris Batterman, who is an expert in x-ray scattering, and Materials Science and Engineering Professor Jack Blakely, who is an expert in surface science.

The study group in quantum phenomena is exploring one of the frontier areas in solid-state physics—the lowest-lying states of matter. Going to the lowest possible temperature is the only route. Making very, very small samples provides access to the solid state in a form different from the normal, and taking such samples to the lowest possible temperature provides a valuable, different look at the low-lying states. Collaboration between those able to make such samples at the National Nanofabrication Facility and those able to cool the samples down makes such experiments possible.



John Silcox has directed much of his recent research effort to ensuring the development of supercomputer-based modeling of the images and diffraction patterns provided by a dedicated STEM, as well as ensuring that the instrument does work and looks at interesting samples.

Collaborative research is helped by the provision of group laboratories. Currently, the MSC sponsors three: the Laser Group Laboratory (which facilitates the work discussed here by Pollock), the UHV STEM Laboratory, and the MicroKelvin Laboratory. Other central laboratories on campus, such as the National Nanofabrication Facility and the Cornell National Supercomputer Facility (an arm of the Cornell Theory Center) are also important resources.

The MicroKelvin Laboratory is one of two in the United States dedicated to achieving the very low temperatures (100 mK or below) that are needed to explore matter in its least energetic state. Discoveries in this region are likely to provide new insights into the quantum aspects of many particle systems. However, the achievement of these temperatures requires specially designed and custom-built equipment, with special attention to problems such as thermal insulation and vibration.

The invention of complex experimental approaches to studies of materials is a significant outcome of the availability of the special facilities in the group laboratories. For example, my research group has been working to expand the applications of a high-resolution, ultrahigh-vacuum scanning transmission electron microscope (STEM) (see *Engineering: Cornell Quarterly*, Spring 1991).

MSC members are also participating in and will benefit from the development of experimental capability at laboratories that are not directly sponsored by the MSC. For example, Joel D. Brock, an x-ray-diffraction specialist, and Hector D. Abruña, an electrochemist, are overseeing the construction of a high-energy-resolution, high-intensity x-ray beam line that will operate in conjunction with an advanced radiation source (a twenty-five-pole wiggler) to be added to CHESS. The new beam line, jointly sponsored by MSC and CHESS, is expected to be on line within a year—well ahead of comparable capabilities elsewhere.

It is the mission of the Materials Science Center at Cornell to create a context in which interactive research on materials can flourish. In doing this, it must support and complement the academic departments rather than compete with them, and similarly, it must complement rather than duplicate the NSF's individual-investigator program. So far it has managed well, achieving its aims through the encouragement of cooperative research programs and the provision of sophisticated equipment. Of course, new decades bring new problems, but for more than thirty years the MSC has successfully met the challenges in materials science and it will continue to do so in the future. ■

John Silcox, the director of Cornell's Materials Science Center, is the David E. Burr Professor of Engineering in the School of Applied and Engineering Physics.

Educated in England, he received the Ph.D. from Cambridge University in 1961 and joined the Cornell faculty that fall. He has served twice as director of the School of Applied and Engineering Physics. He has spent sabbatical leaves as a Guggenheim fellow in France and England, at Bell Laboratories, and at Arizona State University.

Silcox is a fellow of the American Physical Society and a member and past president of the Electron Microscopy Society of America. He has served on the Solid State Sciences Committee of the National Academy of Sciences/National Research Council, and is currently a member of the Materials Advisory Committee for the National Science Foundation. He is an adviser also to national electron microscopy facilities at Arizona State University and at Argonne National Laboratory.

MAKING EQUIPMENT AVAILABLE

Central Facilities for MSC Users

At the Materials Science Center our aim is to encourage the use of the facilities by the entire materials research community at Cornell—and by people from other universities who have a special need for our sophisticated equipment.

To do this, the MSC operates a complex of eight Central Facilities and, in addition, has cooperative arrangements with several national laboratories located on campus. As described in the annual technical report of the MSC (available on request), the overall use of these facilities has continually increased.

Research Support, Education, and Development of Techniques

The ultimate success of the Central Facilities is measured by their ability to support the full scientific programs of MSC participants. Besides making the equipment available, the facilities offer the help of staff specialists in the preparation of specimens or in the operation of complex apparatus.

Another important function of the MSC Central Facilities is to acquaint students with the capabilities of unfamiliar instruments. Instruction ranges from a formal course on the use of the electron microscopes to videotapes that explain how to use various instruments.

AIMS OF THE MSC CENTRAL FACILITIES

- to provide quality instrumentation
- to encourage hands-on use by students and faculty members
- to develop techniques
- to provide training and consultation
- to promote the exchange of multidisciplinary expertise

New techniques are continually being developed in the course of research at the MSC. Often they are the result of interaction among faculty and staff members, and sometimes corporate partners. For example, Professor James Sethna, a physics theorist, explored with IBM (through the MSC) the feasibility of developing a Physicists Workbench utilizing approximately thirty powerful IBM RS-6000 workstations. (See the article in this issue by Sethna.) This has resulted in a three-year agreement under which $3 million worth of equipment is being made available at the MSC to more than thirty theoretical and experimental research groups.

Consultation and Cooperation: Keys to Successful Operation

At the MSC, decisions are made through consultation and cooperation. Extensive discussions are held before the executive committee makes the key recommendations on financial allocations. This committee has six elected faculty members, and the directors of three national centers at Cornell—the Cornell High Energy Synchrotron Source (CHESS), the National Nanofabrication Facility (NNF), and the National Advanced Scientific Computing Center at the Cornell Theory Center—are nonvoting participants.

This "cross fertilization" promotes collaboration among research groups, enhances the effectiveness of funding decisions, and prevents duplication of equipment and human resources. Opportunities for cost-sharing in the acquisition of major equipment often become evident, and this benefits both the MSC and research groups throughout the physical sciences and engineering communities. For example, during the current three-year grant period, 153 publications included acknowledgment of essential support from MSC facilities for research projects that were not directly supported by the center.

As a result of the system of consultation and cooperation, changes in the thrust of research are followed by shifts in the development and expansion of facilities, and improvements in user services are expedited. For example, last May partial funding of $15,000 was approved for the purchase of a high-reso-

THE MSC CENTRAL FACILITIES

Facility	Manager	Faculty Adviser	Services and/or Equipment
Electron Microscopy *(see page 9)*	Robert Keyse	Stephen Sass	High-resolution electron microscopes. Electron microprobes. Equipment for specimen preparation.
X-Ray *(see page 13)*	Maura S. Weathers	William Bassett	Diffractometers, high-temperature stage. Pole figure device. Real-time Laue. Various cameras.
Technical Operations *(see page 17)*	Gerhard Schmidt	Robert Buhrman	Synthesis and fabrication, including crystal growth and vapor deposition. Characterization of samples by techniques including x-ray spectroscopy and scanning electron microscopy. Sample treatment such as polishing and machining, and brazing, annealing, and sintering under controlled conditions.
Materials Preparation	Bernard Addis	Rüdiger Dieckmann	Ceramic crystal preparation and characterization. Ceramic hot pressing. Metals: crystal growth, casting, purification, heat treatment (tool steels). Thin-film deposition and characterization. Electro-polishing, electrodeposition. Thermal treatment of plastics. Mechanical testing for tensile and compressive strength. Cyclic or programmed testing.
Metallography	Margaret Rich	David Grubb	Specimen preparation: cutting fragile crystals, mounting, grinding, polishing. Microscopy.
Multiuser Computing	Richard Cochran	Carl Franck Michael Thompson	A minicomputer-based system for data acquisition, storage and analysis, and theory modeling. Image processing. Data output facilities, including color plotters. Networks. Media conversion. Software development.
Electronics	Donald Knettles	Robert Cotts	Maintenance, repair, and construction of electronics systems and components. Calibration and standardization.
Liquid Helium	Russell Boettcher	Robert Richardson	Gas recovery, purification, and distribution. Servicing and maintenance of equipment.

Notes:
There are two computing facilities—one in Clark Hall, convenient for physicists and chemists, and one in Bard Hall, convenient for people associated with engineering.

Professors Franck, Cotts, and Richardson are from the Deparment of Physics and the Laboratory of Solid State Physics (of which Richardson is director). Professor Buhrman is director of the School of Applied and Engineering Physics. Professors Sass, Dieckmann, Grubb, and Thompson are from the Department of Materials Science and Engineering. Professor Bassett is a member of the Department of Geological Sciences.

Detailed information about each facility is available in the annual technical report of the MSC. Inquiries may be directed to the MSC office (607/255–4272).

". . . changes in the thrust of research are followed by shifts in the development and expansion of facilities. . . ."

lution x-ray diffractometer. In September $65,000 was obtained from research grants awarded to Professors James Mayer and Michael Thompson; in October an order was placed for a Philips high-resolution instrument, and in May 1991 it was installed in the MSC X-Ray Facility (see page 16).

Cooperation is fostered by the executive committee discussions and the interactions among MSC administrators and facility advisers. Also, the entire administrative and technical staff and the faculty advisers for each of the Central Facilities meet regularly to share ideas and concerns and present new developments. When major new equipment is brought on line, demonstrations or seminars are held to help make everyone familiar with their capabilities and aware of the activities underway throughout the MSC. An electronic bulletin board also helps keep everyone informed.

User Charges as Part of the Operating Procedure

Maintaining modern instrumentation for the Central Facilities is a major responsibility of the MSC. Depending on the circumstances, this may involve direct purchase by the center or a sharing of the cost among a number of sources.

Since the beginning of the MSC in 1960, users have helped support the facilities. The purposes of the charge system are to monitor the usefulness of each facility and obtain a profile of uses and users, to help control the demand for use of the facilities and minimize frivolous requests, and to offset a portion of the operational costs, especially for the servicing of equipment and the purchase of replacement parts. The percentage of operational expenses budgeted for recovery depends on the type of facility. Generally, those that offer a broad range of consulting services and types of equipment are budgeted at lower levels of recovery than those (such as the Electron Microscopy Facility) that provide a more narrow range of specialized equipment.

Managing and Operating the MSC Central Facilities

Each of the Central Facilities has a faculty adviser and a manager who is qualified to advise and instruct users in the capabilities and operation of the equipment. The accompanying table lists these people, and gives a brief summary of what is available in each facility.

Undergraduates are also employed, often through the work-study program at the university. During the academic year, there are usually three or four student technicians, and in the summer more students are employed and they work longer hours. Their duties include operating and cleaning equipment, preparing materials, doing photographic work, and making backups of computer programs.

In the sections that follow, three of the managers discuss some of the highlights of their facilities and the activities that are supported. As these and other articles in this issue show, the MSC Central Facilities are crucial to many programs in the broad area of materials research at Cornell. The Central Facilities present a unique demonstration of the benefits that can be realized through multidisciplinary sharing of equipment, expertise, ideas, and projects.— ***Noel Desch***

Noel Desch became associate director of the Materials Science Center in 1978. Previously, he served for eighteen years in various capacities, including that of director, in the Cornell Physical Plant Division. He holds a B.S.C.E. degree from Norwich University and a M.C.E. degree from Rensselaer Polytechnic Institute, and he is a registered professional engineer in New York State. Locally, he is well known for his role in the development of regional water and sewer agencies.

The Electron Microscopy Facility

Last year nearly two hundred researchers came to the Electron Microscopy Facility in Bard Hall to study problems in materials science. They obtained images or analyses of such small features as the interfaces between ceramics and metals, defects in semiconductors, and the microstructure and composition of new types of polymers and composites. These users were members of some fifty-six research groups from eleven departments at Cornell, and six groups from other universities.

The facility provides critical support to two Materials Science Center (MSC) study groups—in ceramics and in polymers—that include faculty members in both the College of Engineering and the College of Arts and Sciences. Support is also provided to research groups involved in three university-industry cooperative programs: on microscience and technology (sponsored by the Semiconductor Research Corporation), on electronic packaging, and on high-temperature superconductors (sponsored by DARPA). Education is also an important part of the facility's function; graduate students receive training in the basic techniques of practical electron microscopy and microanalysis.

The instrumentation includes a variety of transmission and scanning electron microscopes, specimen-preparation equipment, and sample-coating facilities. The records for 1990 show 6,665 hours of instrument use, involving more than 19,000 pieces of film and 7,600 Polaroid photographs. Also, 6,424 hours of ion-beam milling were clocked up, and more than 3,000 specimens were coated to prevent "charging-up" problems. Imaging accounts for over two-thirds of the time booked, and microanalysis for the rest.

The Facility's Transmission and Scanning Electron Microscopes

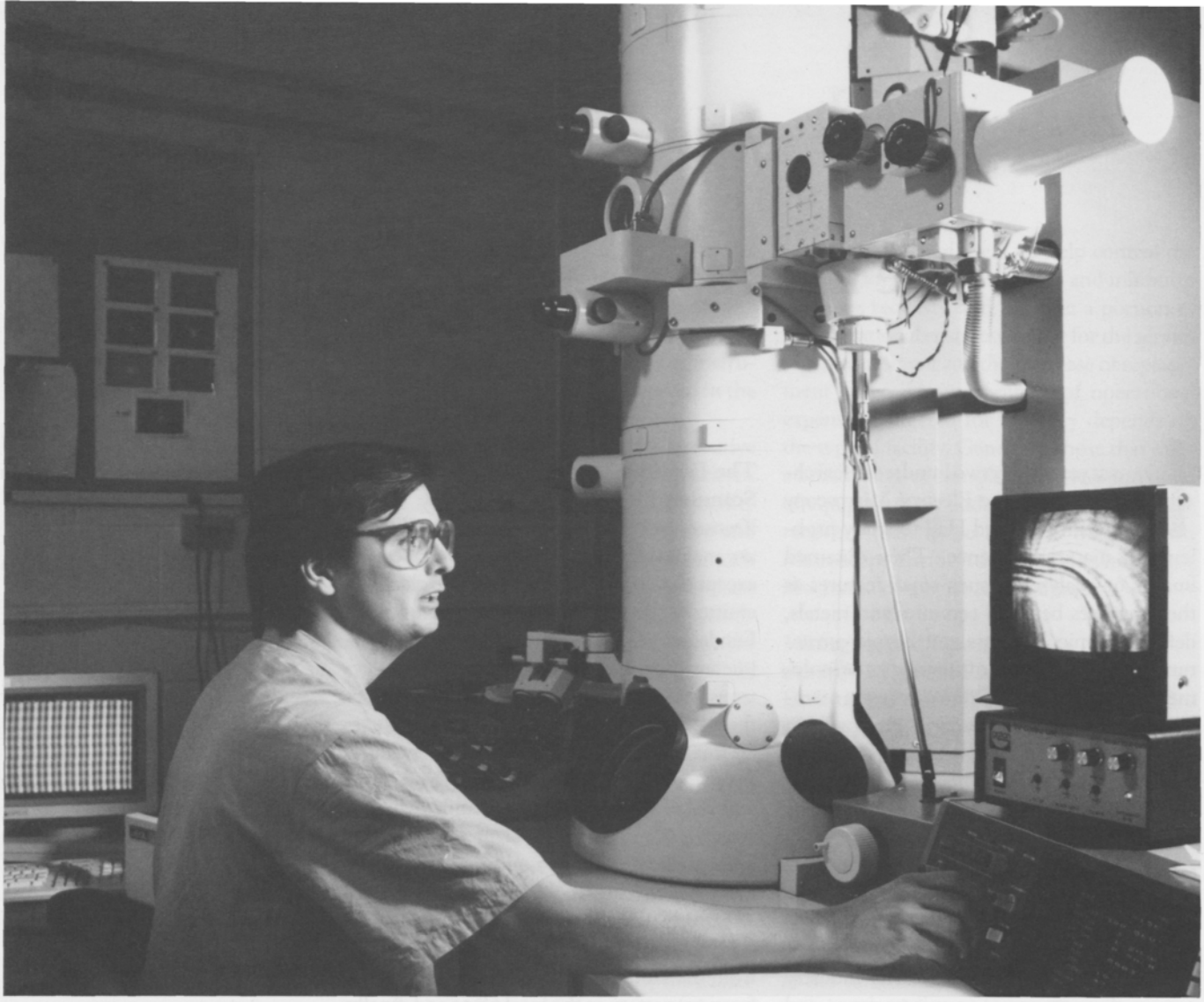
Transmission electron microscopes (TEMs) are analogs of projection optical microscopes, except that instead of light, they use electrons emitted in vacuum from a high-voltage source. Fundamental differences arise from the relativistic and quantum mechanical properties of electrons at moderately high energies, and from their electromagnetic interactions with matter.

Imaging TEMs use shaped magnetic fields as *condenser lenses* to "illuminate" the sample, and as an *objective lens*. The sample, which is typically 3 millimeters in diameter and about one ten-thousandth of a millimeter thick, actually sits inside the objective lens. This lens effectively performs successive Fourier transforms on the electron wavefunction emerging from the sample, to form a scattering (or diffraction) pattern in the focal plane and, finally, a reconstructed image. Subsequently, *projector lenses* build up the image magnification in a similar way, to a maximum value of over a million times the actual size of the object.

The imaging TEMs now available are a 120,000 volt (120 kV) instrument and a 400 kV high-resolution model. The best point-to-point resolution currently attainable is around the size of a platinum ion: 0.16 nanometer (nm), which is 1.6 angstroms (Å) or 1.6×10^{-10} meter. Typically, people use our imaging microscopes to look at structural defects such as dislocations, voids, grain boundaries, and various types of interfaces. Only occasionally is there a need for atomic resolution (available on the high-resolution TEM) and then we attempt to image the projected potential of atom columns.

Scanning electron microscopes (SEMs) use an electron beam magnetically focused into a small probe that moves across the surface of a specimen and forms a TV-like image (using signals from detected secondary electrons or backscattered electrons). Our facility has a high-resolution SEM.

". . . instead of light, [transmission electron microscopes] use electrons emitted in vacuum from a high-voltage source."



Robert Keyse, manager of the Electron Microscopy Facility, is shown at the controls of the high-resolution transmission electron microscope.

The image (in this case, a specimen of gold at a magnification of 200,000) is seen on the screen in front of Keyse. The monitor visible at the far left displays a computer simulation of the crystal structure at a magnification of 10 million. Comparison of experimental and simulated images provides a test of theory.

Images obtained with this instrument are reproduced in the article by Jean Lee (page 42).

Scanning electron microscopy can just as easily be done in transmission, with the resulting STEM instrument also able to use transmitted or diffracted signals to form an "electronic" image; no imaging or projection lenses are necessary (as in the TEM)—instead, the probe itself is an image of the electron source, demagnified to several nanometers in diameter. Our facility has a 200 kV STEM. This medium-resolution microscope, used primarily for microanalysis, is equipped with special specimen holders that enable users to perform in situ experiments while keeping analytical facilities on hand. These holders allow the samples to be tilted about two axes, and they allow heating to over 750 °C and cooling to –180 °C. Also, there is a specimen holder with electrical contacts for measuring induced current and a holder for "bulk" samples.

The resolution of scanning instruments is often taken to be the probe size, and it is possible to generate very small (about 2 Å) probe sizes (although not with the instruments in our facility). The smallest probes that can be used with our equipment are about 4 nm for the 200 kV STEM and 6 nm for the SEM.

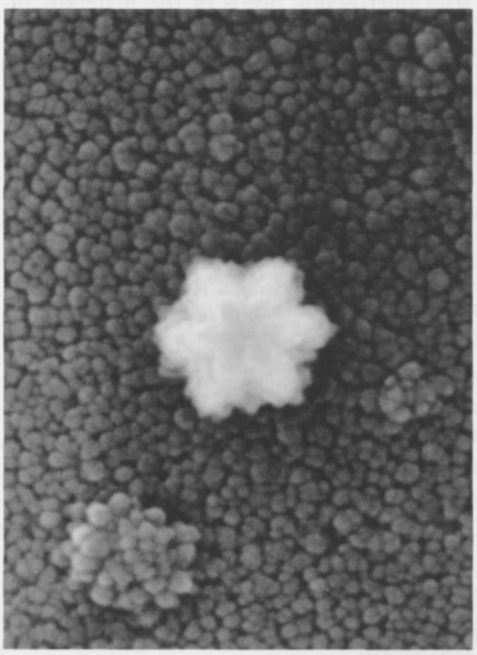
A new accessory for testing the tensile strength of specimens inside the SEM at high temperatures (up to 1000 °C) will soon be available and will provide new information on a variety of materials.

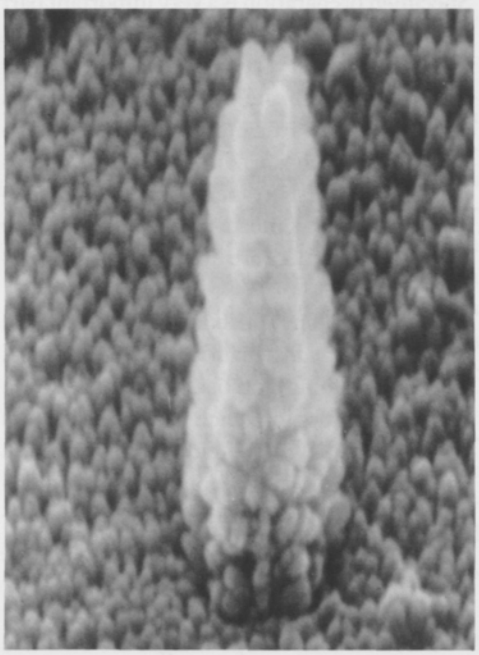
Another possible addition is a recent development in SEM instrumentation that allows specimens to be imaged at high resolution in a partial atmosphere of various gases while heating, cooling, or stretching. Such an "environmental" SEM would be useful to researchers in several departments. For example, dynamic in-situ observations could be made during oxidation at high temperature and simultaneous tensile extension of such materials as intermetallics, ceramics, and composites. The actual ongoing process could be recorded continuously on videotape.

Although all the microscopes currently available at the facility are made by Japan Electron Optics Laboratory (JEOL), this has not always been the case and may change when new instruments are procured.

Above: The electron microprobe was used by Paul Kotula (seated) for senior-thesis research under Professor C. Barry Carter. Conferring with Kotula is John Hunt, research support specialist in the Materials Science Center, who is in charge of the laboratory's microprobes and scanning electron microscopes.

Secondary-electron images of thin films that Kotula obtained were exhibited at the annual national meeting of the American Ceramic Society in May. Entered in the undergraduate student category of the poster competition, Kotula's images (shown below) actually took first place overall.





Left: The images show an (0002)-oriented aluminum nitride (AlN) growth spike on a smooth $(10\bar{1}0)$-oriented AlN film, as deposited by pulsed-laser ablation. At left is a plan-view image showing the hexagonal symmetry of the spike; at right is the same spike imaged at an angle of 60°. The spike, which is about 900 nanometers in diameter, has grown from the surface of the film, which is about 0.5 micrometer thick. This novel (and unexpected) morphology was produced only during deposition at high nitrogen pressure.

"Microanalysis can be carried out by detecting x-rays— either energy-dispersed or wavelength-dispersed—that are emitted as a result of the strong interaction between the incident electrons and the specimen."

The Electron Microprobe and Other Microanalysis Equipment

Microanalysis can be carried out by detecting x-rays—either energy-dispersed or wavelength-dispersed—that are emitted as a result of the strong interaction between the incident electrons and the specimen. This method has several advantages over other microanalytical techniques such as x-ray diffraction and Rutherford backscattering. The chemistry of small-scale regions below the spatial resolution of these other techniques can be determined with accuracy and efficiency.

Our microprobe (a 733 superprobe) can yield a chemical sensitivity of less than one part per thousand from a volume that measures about one ten-thousandth of a millimeter on a side. (The STEM can get 1 percent accuracy on a scale one hundred times smaller.) The microprobe features powerful computer automation for efficient data collection and quantification. It is used by many groups outside as well as within the materials science community.

Other microanalytical techniques that are available include small-area diffraction (either convergent-beam diffraction or microdiffraction), electron energy loss spectroscopy, and cathodoluminescence.

Preparation of Specimens for Electron Microscopy

Specimen preparation is a very important part of electron microscopy, especially for the TEM user, who needs a very thin sample. (A discussion of this from the user's point of view is included in the article, in this issue, by Jean Lee.) At the Electron Microscopy and Microanalysis Facility there is equipment for chemical thinning and mechanical dimpling (fine grinding), and there are many argon-ion milling machines.

The ion milling capability will be improved shortly with the installation of a system with a focused (scanning) ion beam that will allow a precise definition of which part of a sample is being thinned. This system will also have the advantage of allowing secondary electron imaging of the part being milled, and direct transfer between the ion mill and the TEM so that the often mechanically sensitive thin specimen does not have to be disturbed.

Electron microscope images are sensitive to local charging of the sample (resulting from secondary electron emission), and images show this effect as a distortion; coating the sample with a conducting layer of carbon can overcome this problem. Also, the signal can be improved by adding a light coating of a heavy-metal alloy such as gold-palladium. Equipment for vacuum coating both SEM and TEM samples is available at the facility.

Handling Image Data Obtained in the Microscopy Facility

Large quantities of image data—more than 100 giga-bytes per year in photographs—are gathered by users of the facility, and some of this is digitized for further analysis.

One microscope can be connected to the MSC Multiuser Computing Facility, via a TV camera and frame store, for on-line image processing (under SEMPER6) and comparison to simulated images (using TEMPAS). Videotaping dynamic events for later analysis can be done with use of a professional-quality VCR. The SEM and the microprobe have their own digital image acquisition capability, with software for particle sizing and image processing.

Electron Microscopy and Microanalysis: Indispensable for Materials Scientists

When it was established in 1965, the stated purpose of the facility was "to fulfill the electron microscopy needs of the materials research community at Cornell". As these needs have changed and expanded, the facility has kept step. We anticipate further growth within such areas as high-spatial-resolution microanalysis, scanning electron microscopy under controlled environments, and advanced image processing. The Electron Microscopy Facility will continue to provide a service that has become indispensable.—***Robert Keyse***

Robert Keyse, manager of the Materials Science Center's Electron Microscopy Facility, earned a doctorate in physics at Sussex University, England, in 1982. Before coming to Cornell in August 1990, he served as a research fellow in electron microscopy at Leeds University and spent six years at Vacuum Generators' Microscopes Company, working mostly on instrument design and development of field-emission scanning transmission electron microscopes.

The X-Ray Facility

Cooperative effort—the hallmark of Cornell's Materials Science Center—turned a little-used, aging x-ray facility into a well equipped and well staffed laboratory serving hundreds of users every year.

It was in 1987 that the MSC X-Ray Facility was moved from Bard Hall to two new laboratories in Snee Hall, the recently built home of the Department of Geological Sciences. Revitalization of the facility was a joint project of the department, which provides the space, and the MSC, which supports its operation.

At the time the facility was moved, it included several generators and a diffractometer purchased in the 1960s; the newest piece of equipment had been acquired in 1970. Only the most basic types of analysis could be done.

Rebuilding began with the purchase of two new, automated diffractometers from Scintag, Inc. One is a so-called theta-theta diffractometer that has a convenient geometry and an attached chamber capable of maintaining samples at temperatures as high as 2500°C. The other new apparatus is a pole-figure goniometer, which is used for determining the preferred orientation of crystals in polycrystalline samples. Subsequently, attachments for thin-film analysis were added to these two units, and a computer to operate the equipment was purchased. Most recently, a Philips high-resolution diffractometer was installed.

Use of the facility has grown enormously. Last year more than one hundred researchers from some thirty groups on campus analyzed samples in the laboratory. X-ray analysis is an important aspect of many research projects in the Departments of Materials Science and Engineering, Chemistry, Physics, Geological Sciences, and others. And in addition to those from Cornell, researchers from other universities and from industry came to use the facility.

In simple terms, any crystalline material that is zapped by an x-ray beam will produce a pattern that is characteristic of that particular material. The patterns produced by the diffracted x-ray beam can be spots, or lines on a film, or peaks on a scan (see Figure 1). Because each material has a unique diffraction pattern, x-ray techniques can be used to identify unknown samples. Also, information can be obtained about such properties as crystallinity, composition, uniformity, stress, and preferred orientation.

Identifying Unknown Materials with the Theta-Theta Diffractometer

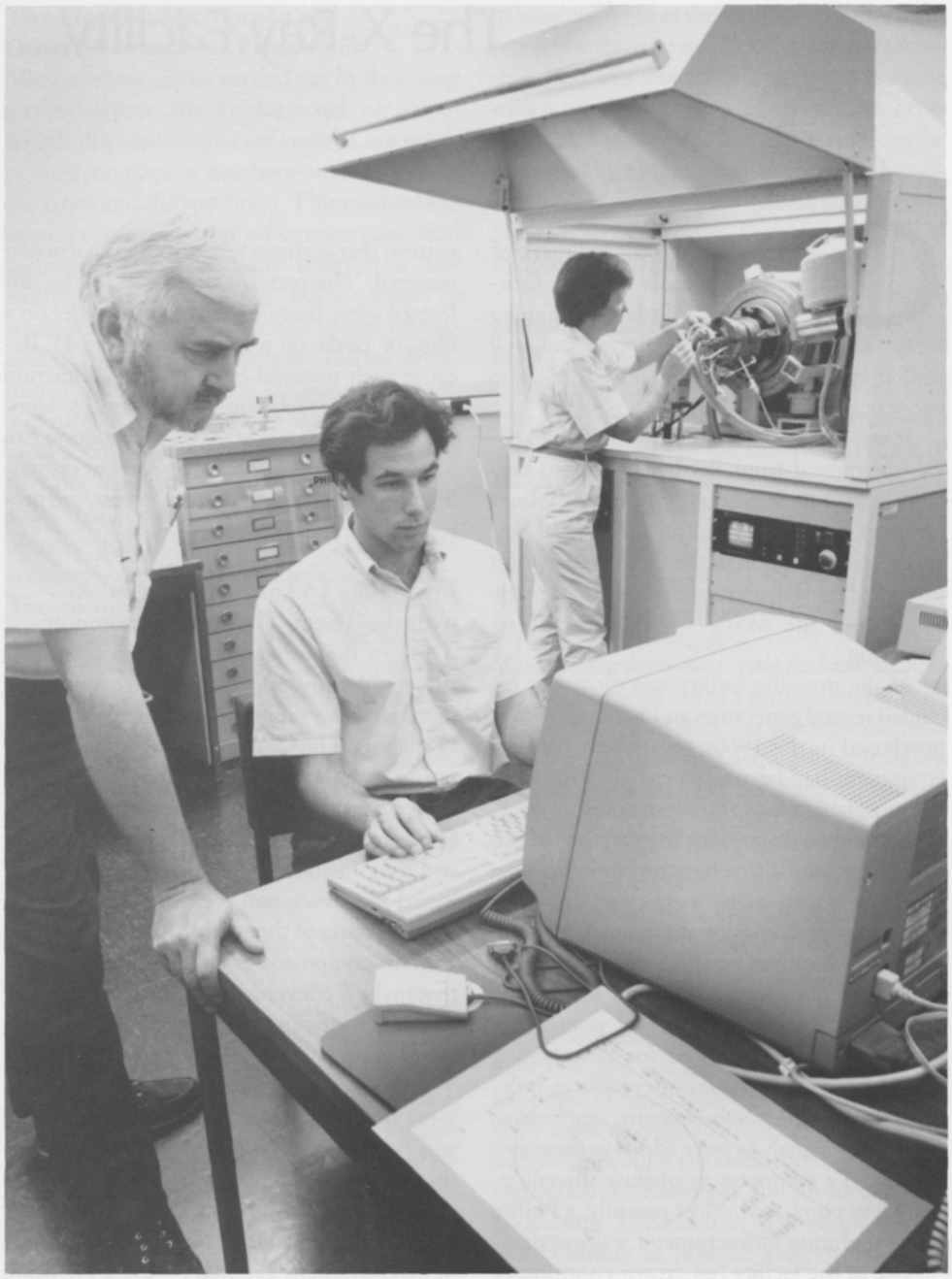
The new theta-theta unit is designed primarily to identify and characterize unknown materials by a rapid, nondestructive method of analysis. The samples analyzed in our laboratory include metals fabricated in the MSC Materials Preparation Facility and ceramics fabricated in the Department of Chemistry; and they include archeological artifacts collected in Greece and rock samples collected from all parts of the world.

This equipment has a number of features that make it convenient and adaptable for use with the wide range of projects the users are working on.

For many years the standard design of x-ray diffractometers included a fixed beam and two parts that moved synchronously—the sample holder, in which the specimen was mounted vertically, and the x-ray detector. Our new theta-theta diffractometer has a much more convenient geometry—the x-ray beam and the detector both move along the theta arc and the sample, mounted horizontally, stays put. This has proven to be a very popular feature.

Another improvement that was incorporated into our theta-theta unit is a solid-state intrinsic germanium detector. This vastly improves the signal-to-noise ratio, as compared with the more usual scintillation counter, monochromator, and filter.

"Use of the facility has grown enormously. Last year more than one hundred researchers from some thirty groups on campus analyzed samples in the laboratory."



The Scintag theta-theta diffractometer in use in the MSC X-Ray Facility. Cory Weiss, a graduate student, is at the computer terminal, where the user usually is stationed during an experiment. With Weiss is William A. Bassett, professor of geological sciences, who is the faculty adviser for the facility. Maura S. Weathers, the facility manager, is working with the instrument.

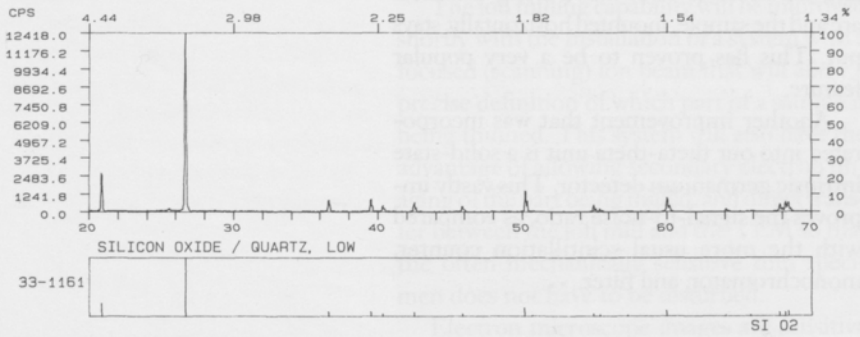


Figure 1 (below). An x-ray diffraction pattern obtained with the theta-theta unit. The sample is a specimen of novaculite, a fine-grained quartz. Here the experimental pattern is compared with the computer-generated pattern of quartz.

The vertical scale represents the intensity of each peak in counts per second (CPS), and the horizontal scale is the angle at which x-rays were reflected from the sample.

Because of the recent increase in thin-film research, the X-Ray Facility purchased a parallel slit attachment for the theta-theta diffractometer. The attachment replaces the standard perpendicular slit block and is used to run a glancing-angle scan of thin-film samples. The x-ray tube is held at a fixed low angle and the scan is accomplished by moving the detector only. The x-ray beam is more efficiently scattered by the thin film at this low glancing angle than it would be in a stan-

Weathers loads a sample into the pole figure device on a Scintag x-ray diffractometer. This instrumentation is used for analyzing the preferred orientation of crystallites in polycrystalline samples.

dard scan because a strong signal from single-crystal substrates is avoided. Thin-film analyses now account for at least half of the use of the unit.

Another attachment that is becoming widely used is the high-temperature chamber capable of maintaining temperatures of up to about 2500°C in a vacuum. This attachment allows investigators to monitor temperature-induced changes in lattice parameter and to look for phase transitions at elevated temperatures.

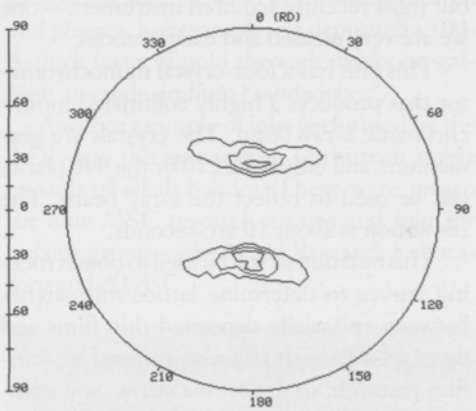


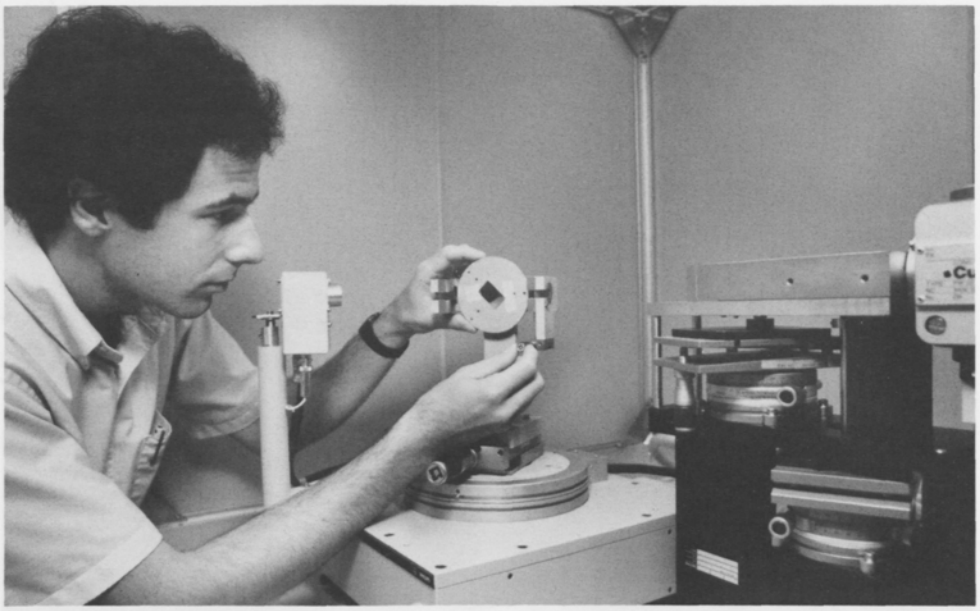
Figure 2. A pole figure of copper foil obtained on the pole figure diffractometer. The tight concentrations of contour lines indicate that the crystallites of copper in this foil have a high degree of orientation.

The Pole Figure Goniometer for Determining Preferred Orientation

The pole figure diffractometer, which was purchased at the same time as the theta-theta unit, functions as a goniometer—an instrument for measuring angles in solid-state samples. An outstanding feature is that samples can be rotated around two axes while the intensity of a peak is measured.

The standard powder diffractometer works on the principle that the grains in a sample are randomly oriented, but this is often not true. If the sample has been stressed, it tends to develop a preferred orientation. The pole figure goniometer can be used to determine what that preferred orientation is, and to quantify it. In the case of naturally deformed rocks, the orientation of the stresses that deformed a sample along a large fault zone can be determined. An example of a pole figure is shown in Figure 2.

At the present time, this instrument is being used to determine fabric in naturally deformed limestone and quartzites from samples collected along fault zones in Wyoming, Greenland, and Scotland, and in sediments deformed in deep ocean trenches. It is also being used to determine the extent of preferred orientation that develops in YBCO (yytrium-barium-copper oxide) superconducting films that have been deposited on different types of substrates.



Graduate student Cory Weiss mounts a sample on the new Philips high-resolution diffractometer. This instrument is significantly expanding the capability of the X-Ray Facility to make detailed analyses of thin films.

The Newest Addition: the High-Resolution Diffractometer

The Philips high-resolution diffractometer is our most recently acquired instrument—one we are very pleased and excited about.

This unit has a four-crystal monochromator that produces a highly collimated monochromatic x-ray beam. The crystals are germanium, and either the 220 or the 440 planes can be used to reflect the x-ray beam. The resolution is about 10 arc-seconds.

This instrument will be used to obtain rocking curves to determine lattice mismatches between epitaxially deposited thin films and their substrates. It can also be used in thin-film research, to determine stress and composition. A computer program will allow the investigator to model the thickness, composition, and number of epitaxial layers to match the actual diffraction scan.

Meeting Diverse Research Needs in the Evolving X-Ray Facility

Not all the equipment in the facility is new. A longstanding and valuable resource is a real-time back-reflection Laue-type apparatus, which was developed in the MSC in the 1970s. Like the standard back-reflection Laue camera, it is used to determine crystal orientation. However, unlike the standard device, which uses film and requires a series of pictures to make a progressive approximation to orient a sample, the real-time apparatus allows the user to observe a spot pattern on an oscilloscope screen while the single-crystal sample is being rotated. This instrument is used for such operations as orienting crystals for epitaxial thin-film deposition, performing single-crystal deformation experiments, and cutting diamond for diamond-anvil cells. This prototype unit will soon be replaced with a new computer-run system.

In addition to the major instruments that have been described, the facility has a variety of x-ray cameras and darkroom equipment. The facility offers individual instruction to all users in operating the instruments and in interpreting the results. Staff members also assist in laboratory instruction for several courses in geological sciences, physics, and materials science and engineering. The X-Ray Facility is a significant arm of the Materials Science Center, contributing to scientific accomplishments and helping to expand the range of research being undertaken across the campus.—***Maura S. Weathers***

Maura S. Weathers, the manager of the X-Ray Facility, is a senior research associate in geological sciences. She holds three Cornell degrees in geological sciences and has held postdoctoral positions in that department since receiving her doctorate in 1978. She has done field work in many parts of the world; in the laboratory, she has developed techniques for using the scanning transmission electron microscope to study geological materials.

The Technical Operations Laboratory

Hands-on use of equipment and close interaction between staff and researchers are key to the way the Technical Operations Laboratory (TOL) works. Even at the expense of equipment wear-and-tear and efficiency, these primary functions are stressed. The belief is that new and imaginative solutions to research problems and effective graduate education require such a direct, hands-on approach.

The services, equipment, and technical support provided by the TOL constitute an essential part of the Materials Science Center (MSC). These operations fall into three main categories:

1. The provision of tools, equipment, space, and technical support such as maintenance, supplies, and consultation. The equipment is used by students in their research and by the laboratory staff to do requested jobs. Much of it is housed in clean-room lab space.

2. The development, in collaboration with researchers, of major new techniques and capabilities. Whereas the main thrust in the 1960s and 1970s was crystal growth, today it is in the preparation of thin-film materials.

3. The performance of many different "job-shop" tasks on a routine basis.

The Wide Variety of Technical Operations

Last year our TOL staff completed a total of 283 job tasks, mostly for members of MSC research groups, but also for researchers at other university laboratories and centers.

Many of these operations involved the use of electron-beam and sputter systems. Substrates such as silicon, sapphire, glass, gallium arsenide, magnesium oxide, sodium chloride, and carbon were thinly coated with metals, semiconductors, or insulators in the form of single-material thin films, multi-layers, alloy films, or compounds such as titanium nitride or tantalum carbide. This kind of work is crucial to research projects of groups throughout the materials science community at Cornell.

Services were also performed for other university groups, including the Cornell High Energy Synchrotron Source (CHESS), the Laboratory of Plasma Studies (LPS), and various departments. For CHESS, for example, we vacuum-brazed sixteen beryllium windows into stainless-steel holders for installation in the beam line at the high-energy x-ray facility. Also for CHESS, we hardened tool-steel parts in hydrogen at 1100 °C. For LPS, aluminum was sputtered on Mylar foil attached to aluminum rings. Other jobs contributed to projects in such fields as electronics research and physics. For example, we deposited a 100-Å-thick layer of gold through masks on gallium arsenide–indium "sandwiches".

Another example of jobs performed by the TOL was the preparation of thirteen single crystals of alkali halides. These were grown for nine MSC research groups and four for outside groups—the Naval Research Laboratory and AT&T.

EQUIPMENT IN HEAVY DEMAND AT THE TOL

- Electron-beam evaporation systems and a bell-jar evaporator
- A sputter deposition system
- Various spectrometers, including an infrared spectrometer with Fourier-transform capability and one with infrared, ultraviolet, and visible light sources
- An ESCA system for surface analysis
- A microdensitometer for analysis of photographic (including x-ray) data
- An optical microscope with magnification up to 2,000 and a color video system and printer
- Mass spectrometer leak detectors for checking vacuum components
- A variety of annealing, vacuum, processing, and crystal-growing furnaces
- A surface profiler
- Equipment for sample polishing, plating, and ultrasonic cleaning
- Analytic balances

Gerhard Schmidt (at left) and graduate student David R. Peale are surrounded by equipment in the Technical Operations Laboratory. At left is a dual electron-beam deposition system; at right is a magnetron sputter system.

". . . new and imaginative solutions to research problems and effective graduate education require such a direct, hands-on approach."

Ion-Solid Interaction: An Example of How Researchers Use the TOL

Thin-film deposition carried out at our facility was part of a major investigation of ion-solid interactions. Examination of nickel-titanium films prepared in different ways showed that ion-induced crystallization affects the intermixing of layers of metals—a finding of significance in achieving a basic understanding of energy dissipation in solids.

The first sample prepared at the TOL was a bilayer of Ni and Ti deposited on a silicon dioxide wafer. After analysis by Rutherford Backscattering Spectrometery (RBS) at the Department of Materials Science and Engineering, the samples were irradiated with energetic ions at the National Nanofabrication Facility and then reexamined by RBS to determine the extent of intermixing of the two metals. Generally, such mixing occurs in local melt zones that are formed as "thermal spikes". The Ni–Ti samples, though, did not exhibit the typical behavior, and subsequent examination in the Electron Microscopy Facility and in the X-Ray Facility indicated a possible reason: they appeared to contain several crystalline phases.

To allow a better test of this possibility, a second set of samples was investigated. Back at the TOL, Ni and Ti were co-deposited in thin films separated by a "marker" layer of bismuth. RBS showed that the initially amorphous Ni–Ti became crystallized under irradiation, but did not interact appreciably with the Bi. Thus the unexpected behavior observed with Ni–Ti samples appeared to be caused by ion-induced crystallization.

The example I have just given demonstrates not only how the TOL contributes to ongoing materials research at Cornell, but also how the various facilities of the MSC and of other research facilities on campus are used cooperatively, efficiently, and effectively. In this particular example, the researchers made use of instruments and expertise in three difference laboratories.

At TOL and throughout the research community at Cornell, collaboration and cooperation produce results.—***Gerhard Schmidt***

Gerhard Schmidt, manager of the Materials Science Center's Technical Operations Laboratory (TOL), was a machinist's apprentice in the physics department at the University of Göttingen, Germany, before he came to Cornell in 1961 as a research technician in physics. He began work here in the crystal-growth facility that became part of the TOL when that was formed in 1978.

HEATING WITH LIGHT:

Growing Ceramic Single Crystals at Very High Temperature

by Roland Geray and Rüdiger Dieckmann

Crystals, which have fascinated people since ancient times, have added significance in our industrial age. Besides being valued as beautiful, sparkling gemstones, they have technical applicability and new scientific interest.

In numerous advanced technical devices, crystals function as components such as powerful lasers, fast transistors, infrared-transparent windows, sensitive radiation detectors, precise frequency references, and super-hard cutting media. In addition, scientists need single crystals of high purity as reference materials to determine how microstructure affects the properties of polycrystalline, multiphase ceramics. By comparing results obtained from both types of samples, rules can be derived for the design of materials with useful properties, designed for specific applications. Properties of interest are, for example, electrical conductivity, ferromagnetism, optical transparency, piezoelectricity, ion diffusivity, and hardness.

Cornell's Facility for Growing Ceramic Single Crystals

Most of the single crystals used in science and technology cannot be found in nature, or at least not in the very pure state that is required, so they must be custom-made in the laboratory. To help satisfy the increasing demand for noncommercial, custom-made single crystals, one of us (Dieckmann) initiated the establishment here at Cornell of a facility for making such samples.

As a first, big step, an image furnace was installed last November in the Bard Hall Materials Preparation Facility of Cornell's Materials Science Center (MSC). Purchase of the furnace (from NEC/Nichiden Machinery Company, Japan) was made possible by an equipment grant from the U.S. Department of Energy and significant matching funds from MSC (which is funded by the National Science Foundation), the Cornell Ceramics Program (sponsored by Corning Incorporated and IBM), and Cornell University.

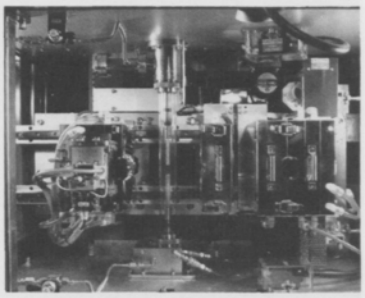
This image furnace, with its particular capabilities, is unique in the United States. Designed for the growth of single crystals from the melt by the so-called *floating-zone method*, it can achieve very high temperatures—up to 2800 °C—with variable gas atmospheres.

The Image Furnace and How It Is Used

Our furnace is essentially a reflective ellipsoid chamber containing a lamp as heat source at one focal point, and two sample drive units at the other focal point (Figure 1). Through the drive units, rods of the ceramic samples can be injected vertically from opposite directions at controlled rates under controlled atmospheric conditions. Gas atmospheres can be reducing or oxidizing.

Figure 2 illustrates how the method works. When the ends of the advancing sample rods reach the region where the radiation is focused, melting occurs and the two rods become connected by a molten zone. Crystal growth subsequently occurs as the two rods are moved slowly downward, initially at different velocities. The speeds at which the rods need to be moved are different for each material; they depend strongly on the individual crystallization behavior, the viscosity of their melt, and the thermal conductivity of the solid sample rods. The drive units can operate at speeds from 0.1 to 200 millimeters per hour, and so can cover the whole spectrum of materials from very slow-growing silicates to fast-growing metals.

This floating-zone method overcomes some technical problems encountered in trying to grow ceramic single crystals with techniques that require crucibles for holding the melt. Crucibles are generally not usable for many of the (transition) metal oxides that are of research interest at Cornell because these sample materials have very aggressive melts



"This image furnace . . . is unique in the United States."

". . . every grown crystal requires custom processing by an experienced operator. . . . Any kind of automatic mass production is practically impossible."

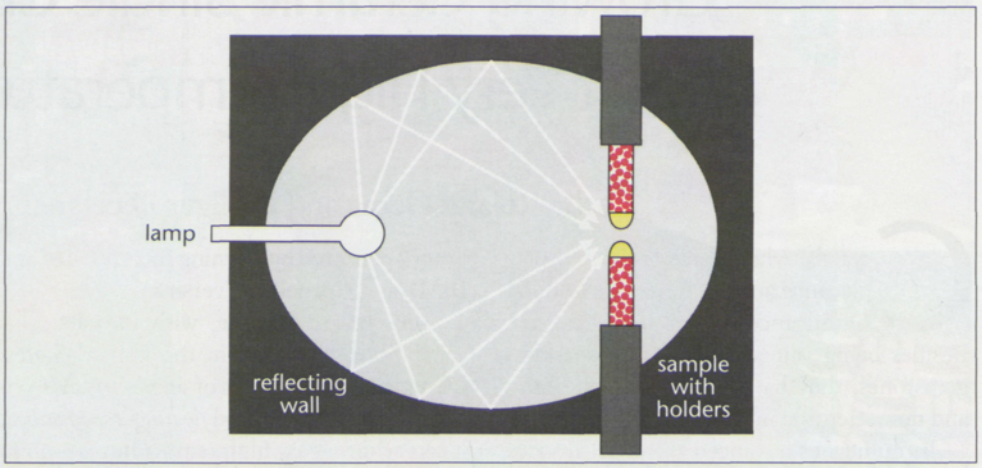


Figure 1.The ellipsoid mirror furnace. A heat lamp is located at one of the two focal points of the ellipsoid, and the radiation is focused at the other focal point. Two holders allow sample rods to be advanced into this second focal-point region, where melting occurs. The sample rods are usually formed from pressed and subsequently sintered powder.

The water-cooled main body of the furnace is made of an aluminum alloy plated with gold for enhanced reflectivity and corrosion resistance. The furnace is equipped with two heat sources—a 3.5 kW halogen lamp that can heat a cubic centimeter of sample as high as 2100°C, and a 5.4 kW xenon high-pressure lamp to achieve temperatures up to 2800°C. A tube of fused silica sealed into two chucks surrounding the sample protects the reflective mirror walls from evaporating sample compounds and allows the use of controlled gas atmospheres, which may be reducing or oxidizing.

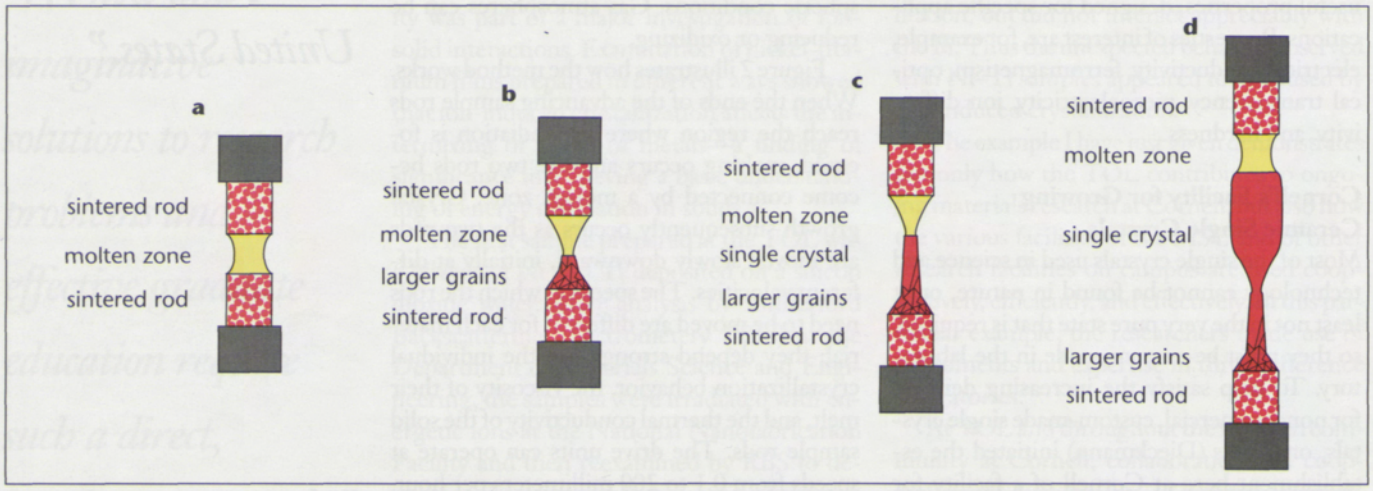


Figure 2. The growth of single crystals by the floating-zone method in the image furnace. The sample material is in the form of rods contained in the movable sample holders shown in Figure 1.

a. The ends of the two sample rods enter the hot zone, melt, and are pushed together to form a connecting section of melt.

b. The two melt-connected rods are lowered slowly, initially at different velocities. As soon as the lower rod moves downward, melt begins to crystallize at the rod's upper surface, with the grains of the sintered powder acting as nuclei. If the lowering rate remains below a certain level (determined by the material), the grains simultaneously increase in size and decrease in number.

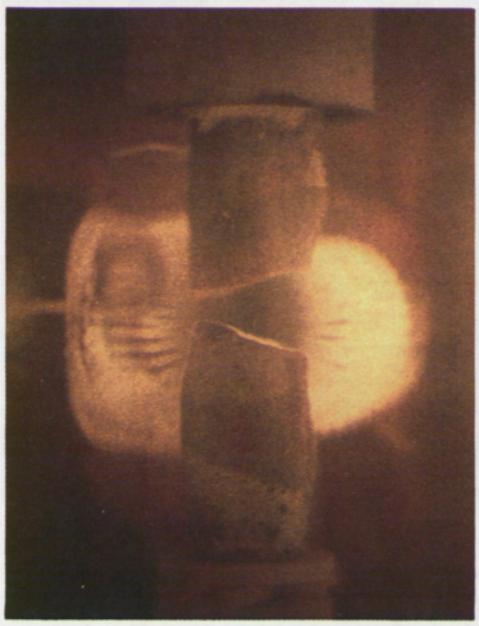
c. As the diameter of the growing rod is reduced to about 1–2 mm (by lowering the lower rod faster than the upper rod), grains are eliminated until only one large grain—the desired single crystal—remains.

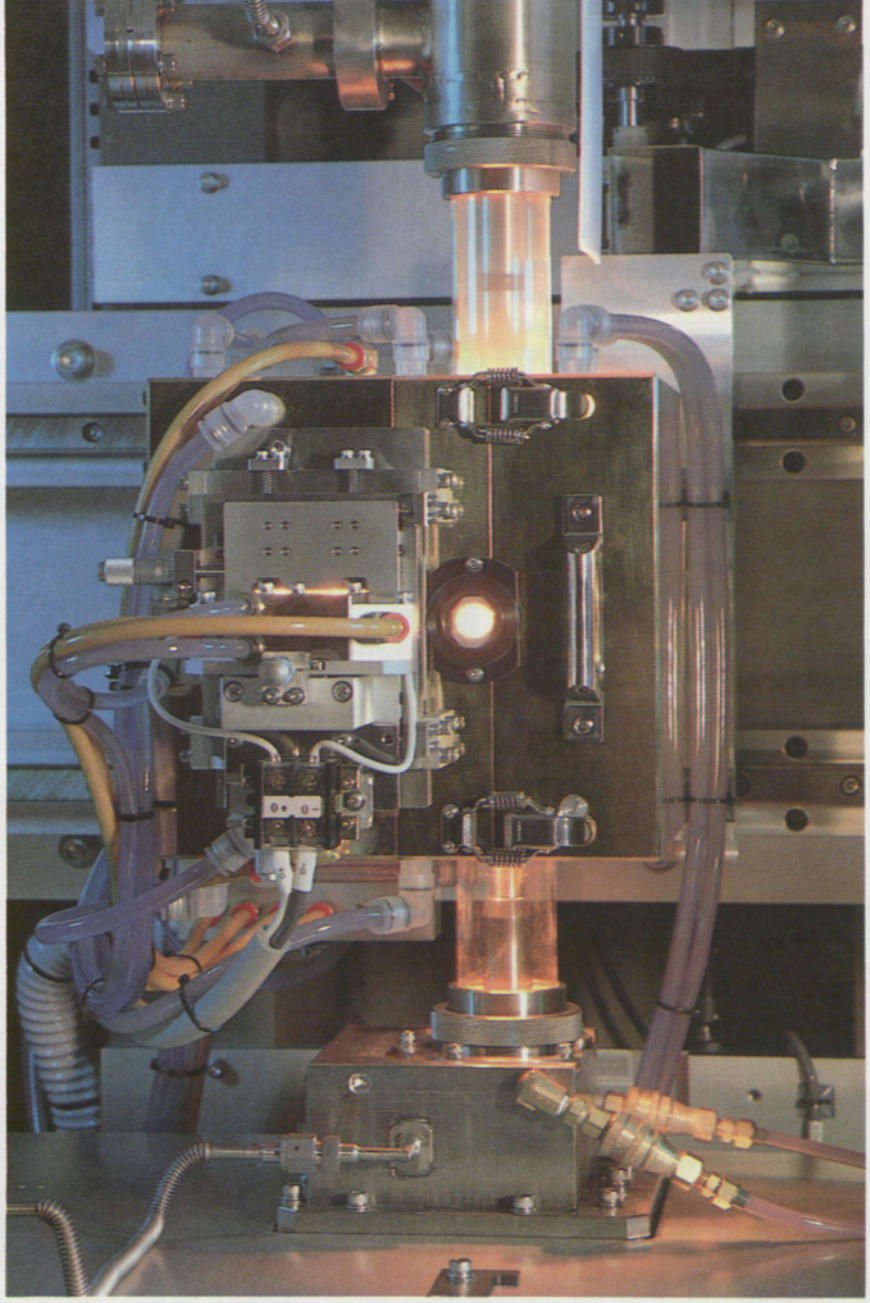
d. After that, varying the rates at which the rods are lowered allows the single crystal to grow in diameter to the final size, typically 5–10 mm; then the crystal of this diameter continues to grow in length as long as the feeding material—the upper rod—lasts.

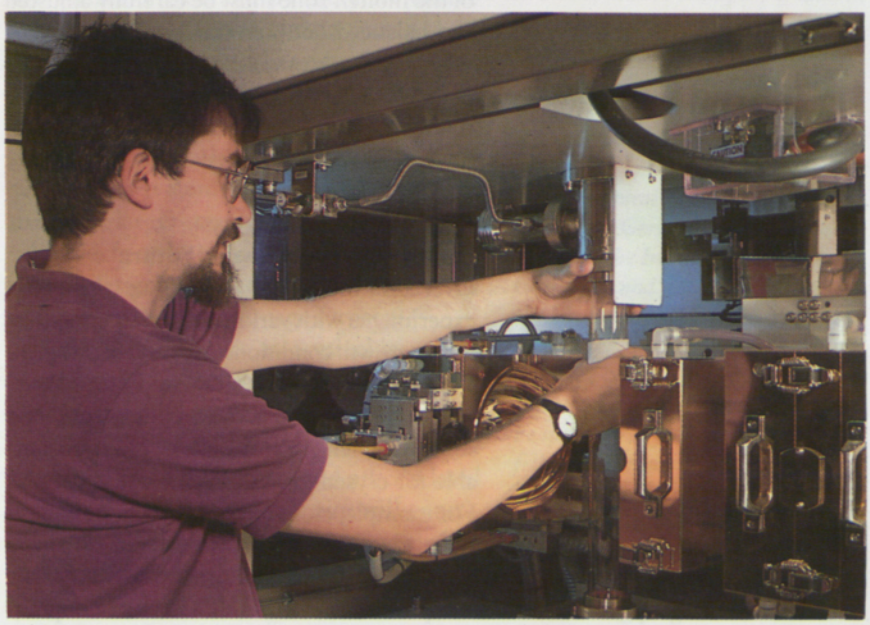
Right: A view into the housing of the gold-coated image furnace. The sample is enclosed in the transparent tube, which extends through the heating chamber.

Right below: Geray inserts a sample. During operation, the two halves of the elliptical heating chamber are closed and the progress of melting and crystal growth is monitored by means of a TV image.

Below: A view of the sample area inside the furnace, showing the ends of the two sample rods about to be melted. This direct view is made possible by a "peephole" in the furnace wall; a magnified projection of the sample area is viewed through a ground-glass window.







that severely attack practically all existing crucible materials, notably noble metals and refractory ceramics. Also, because the floating-zone method uses no crucible, it can accommodate high temperatures, sometimes well above 2000 °C, and the variable gas atmospheres that are required to establish proper melting equilibria.

It should be noted that every grown crystal requires custom processing by an experienced operator. For example, during the crystal growth, the melt zone is kept in position by its surface tension and only a limited amount of weight can be sustained, depending on the surface tension and density of the particular sample. Accordingly, the height and diameter

Figure3. Some of the single crystals grown in the image furnace. The single crystals are shown below the corresponding feeding rods.

From left to right are cobaltous oxide (CoO), ruby ($Cr:Al_2O_3$), rutile (TiO_2), and cobalt-doped spinel ($Co:MgAl_2O_4$).

The diameters of the single crystals were all between 5 and 10 mm and had lengths of up to 10 cm. Current work is with nickel oxide (NiO), olivines ($(Mg,Fe)_2SiO_4$, and chromium-doped forsterite ($Cr:Mg_2SiO_4$).

of the molten zone must be carefully adjusted and balanced between the two rods—a process that is accomplished by watching and manually adjusting the velocities of the rods. Any kind of automatic mass production is practically impossible.

Research at the Facility and the Outlook for the Future

Over a six-month test period, we carried out experiments with various materials under different growth conditions and successfully grew a number of single crystals (see Figure 3).

One aspect of our research is an ongoing study of which gas atmospheres are most suitable for crystal growth in particular systems. For example, close to their melting temperatures, the simple transition metal oxides NiO and CoO are highly nonstoichiometric—their composition varies as a function of temperature and oxygen partial pressure in their environment—and since the equilibria between solid and melt are not yet well established as a function of oxygen partial pressure and temperature, it is difficult to select the appropriate atmospheric conditions for crystal growth. If, for instance, the oxygen partial pressure is too high, bubbles form at the crystallization front and this leads to holes in the growing single crystal. The information we are obtaining from this investigation is useful also for the establishment of phase diagrams. Another benefit is that some of the NiO single crystals to be grown will be used by Professor Stephen L. Sass's group to study the reaction between NiO and platinum and related interfaces.

Our group is also investigating the point defect structure of olivines—$(Mg,Fe)_2SiO_4$—which are a major component of the earth's upper mantle, and the related transport of

Roland Geray (at left) works with Rüdiger Dieckmann (at right) on the growth of single crystals in the image furnace.

ions in them. This information is of great interest to earth scientists because point defects affect not only the transport of ions, but also the mechanical properties and, as a consequence, the mechanism of plate tectonics. We began by using a unique thermobalance to examine the nonstoichiometery of polycrystalline samples prepared in collaboration with James Burlitch in the Department of Chemistry; as soon as single crystals are available, we will begin radioactive tracer diffusion studies as well. We will analyze our experimental data and report our findings to the earth-science community.

Chromium-doped forsterite single crystals are to be grown for a joint MSC-sponsored research effort involving James Burlitch (Chemistry), Rüdiger Dieckmann and Christopher K. Ober (Materials Science and Engineering), and Clifford R. Pollock (Electrical Engineering). Chromium-doped forsterite is a promising candidate for fiber-optic telecommunication applications because it shows laser activity in the infrared region between 1.1 and 1.3 micrometers. The chemistry group will prepare precursor materials with various dopant concentrations; we will grow single crystals under various conditions; the electrical engineering group will analyze the specimens for their laser-relevant properties (see Pollock's article in this issue).

Although the floating-zone method with focused radiation heating generally works well with dark, strongly absorbing materials such as CoO, optically transparent materials such as Al_2O_3, which absorb the focused radiation very poorly, cannot be melted and grown as single crystals without additional heating. We are developing an accessory that will alter the temperature distribution along the lower rod and heat, in particular, the rod's surface area. This will expand the range of materials that can be grown as single crystals.

We are also developing a procedure for growing materials for which the floating-zone method cannot be used: materials that have low surface tension and are therefore unable to carry the weight of a molten zone 8 to 12 millimeters in diameter. We want to apply the Verneuil method, in which powder grains are fed directly into the molten top of a vertically growing crystal; because this requires a much smaller melt volume than the floating-zone method, larger crystal diameters can be achieved. In order to use the Verneuil method, we are designing an adjustable, low-volume powder feeder for our image furnace.

Our image furnace is a valuable instrument for the Cornell materials science community and other researchers because it provides the ability to grow a variety of single crystals. The image furnace and other related equipment we expect to acquire will constitute an important facility for basic and applied research on materials. ■

Roland Geray is a postdoctoral researcher with Professor Rüdiger Dieckmann in materials science and engineering at Cornell. A native of Germany, Geray earned a diploma degree in mineralogy at the University of Freiburg in 1984 and a Ph.D. at the University of Marburg in 1990. His thesis research was on crystal growth and magnetic properties of cobalt and nickel silicates.

Rüdiger Dieckmann, a specialist in the point-defect structure of ceramics, related transport of matter and charge, and the kinetics of solid-state reactions, has been a Cornell professor of materials science and engineering since 1987. He holds a diploma in chemistry (1972) and a doctorate in engineering (1975) from Technical University of Clausthal, Germany. Subsequently, he joined the Institute for Physical Chemistry and Electrochemistry at the University of Hannover, where he received, in 1983, the "habilitation" for professorship in physical chemistry. Before coming to Cornell, he spent three years as a Heisenberg fellow at Hannover and at the Nuclear Research Center at Jülich. In 1984 he was awarded the Nernst Prize. At Cornell he is currently serving as chairman of the Master of Engineering program.

ADVENTURES WITH FORSTERITE

by Clifford Pollock

"I tell callers that currently there is no broadly tunable laser suitable for probing fiber-optic devices at 1.3 μm, but that soon there may be."

I often get phone calls from people looking for lasers that operate over certain wavelength regions. Over the past five years, the most common request has been for a tunable source in the near-infrared region around 1.3 micrometers (μm). This is the wavelength at which distortion is lowest for many optical fibers used in data transmission. Researchers want a smoothly tunable, continuous-wave source in order to characterize the performance of fiber-optic devices or systems that operate in this vicinity. Lasers are particularly well suited for probing fiber-optic devices because they have a well defined beam that can be efficiently focused to small dimensions, and the wavelength of the light is essentially monochromatic.

I tell callers that currently there is no broadly tunable laser suitable for probing fiber-optic devices at 1.3 μm, but that soon there may be. My research group in electrical engineering and several groups in other fields at Cornell have combined efforts to develop suitable materials for lasers in the 1.3-μm wavelength region, and we are well on the way to achieving exciting results. Indeed, we have come to regard the course of our research as an adventure.

Tunable Lasers and How They Work

The effect that leads to the generation of monochromatic light by a laser is *stimulated emission*. When certain atoms or molecules are excited by photons of a particular frequency, they emit photons that are identical in frequency and phase to the incident photons. If this process can be repeated many times, as by trapping the photons between two mirrors, then the intensity of the radiation can build up and monochromatic light can be generated.

The lasers we build are fairly simple. The schematic in Figure 1 shows the essential parts: the solid-state lasing material, called the *gain medium*; a pump beam, supplying the energy to the gain medium; a pair of lenses to focus the radiation; a pair of mirrors to reflect light back and forth through the gain medium; and a prism to disperse the emitted light, allowing the laser to be tuned to a particular wavelength. The gain medium is the heart of the laser, as it generates and amplifies laser light of the desired wavelength. It also determines key characteristics of the laser, such as its power and tuning range. Developing good gain media is the focus of my group's research.

Finding a Material That Lases in the Needed Range

The spectral range that is appropriate for work in optical instrumentation (1.3 μm) has not been easily accessible for tunable lasers. For wavelengths greater than 1.45 μm, there are color-center lasers, which span wavelengths all the way from 1.45 to 3.9 μm, with continuous-wave output power and smooth tuning. For wavelengths shorter than 1.1 μm, there are a number of systems, including the dye laser and a more recently developed solid-state laser based on titanium-doped sapphire, which can produce useful power over a range that is tunable from 700 to 1,100 nanometers (nm). But in the spectral range between 1.1 and 1.4 μm, there are presently no powerful continuous-wave sources. Developing a tunable laser for this spectral window has challenged my research group for several years. We are expert with color-center lasers that use alkali-halide crystals (see "Tunable Infrared Lasers Based on Color Centers," *Engineering: Cornell Quarterly*, 19:12–17, Summer 1984), so we explored many color-center systems that might lead to a tunable source around 1.3 μm. But for one reason or another, these systems have not worked out. At one point, one of my graduate students thought he might

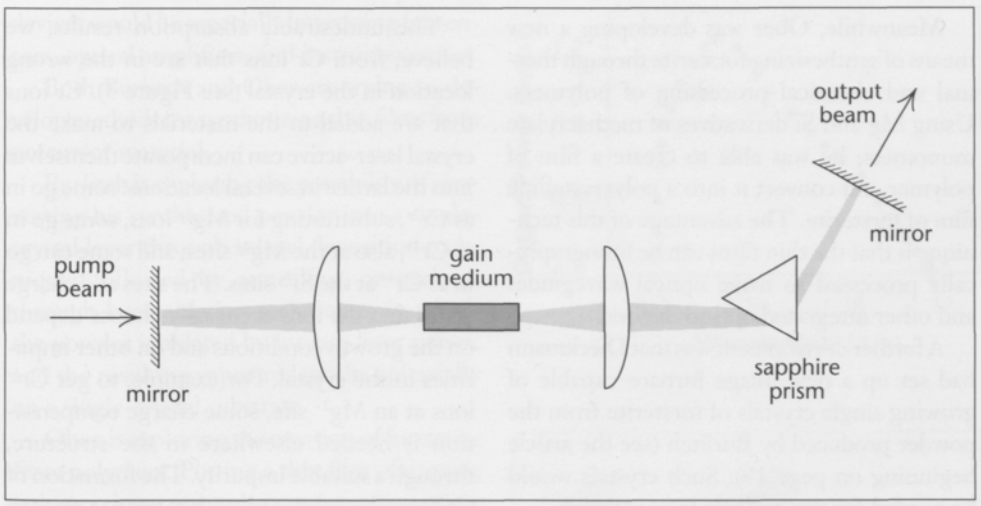


Figure 1. A schematic representation of a tunable solid-state laser. The laser cavity is formed by two mirrors that reflect light back and forth through the lasing material (called the gain medium), which amplifies the passing light. Tuning is accomplished by adding a dispersive element, such as the prism shown, into the cavity. Adjusting the angle of the end mirror adjusts the operating wavelength.

In the research described here, the gain medium is forsterite doped with chromium ions.

have to write a thesis entitled, "Things That Don't Lase."

Then, about two years ago, we became aware of a material called forsterite that was being investigated at Columbia University and at the Army Night Vision Laboratory. Forsterite is a naturally occurring mineral that is principally composed of Mg_2SiO_4, with small amounts of metallic impurities such as chromium and iron. Early results showed that Cr-doped forsterite could be made to lase in the 1.25-μm region. We were cautious: laser media are reported frequently and many fall short of their initial promise. A good tunable laser should have a gain medium that is reasonably inexpensive, easy to pump (energize), and easy to handle—and it should be smoothly tunable and usefully powerful. The initial reports on forsterite looked promising, so we decided to explore its potential.

Having spent most of my career working on alkali halide crystals, I didn't know much about forsterite. In truth, I didn't have the slightest idea what the stuff was. But soon after I first heard about it, I happened to mention forsterite during an informal conversation with Christopher Ober of the Department of Materials Science and Engineering. To my amazement, I discovered that not only Ober, but James Burlitch in the Department of Chemistry and David Kohlstedt, then in MS&E, had been working intensively with forsterite for about a year within the MSC Ceramics Study Group. Thus began my adventure with forsterite.

The Story of Work at Cornell with Forsterite

Forsterite, it turns out, is part of the olivine family of minerals that form a major part of the earth's mantle (see *Engineering: Cornell Quarterly* 23:29-33, Winter 1989). Understanding the mechanical properties of mantle rocks allows geologists to better understand the dynamics of the earth's crust. Measuring the mechanical properties of such materials requires very pure, fine-grained samples.

Burlitch tackled the problem of synthesizing forsterite while spending a sabbatical leave in Kohlstedt's laboratory. Drawing on several years' work with transition-metal carbonyls, he synthesized olivine, $(Mg,Fe)_2SiO_4$, by combining magnesium with an iron carbonyl compound and a silicon alkoxide in methanol. He then treated the mixture with hydrogen peroxide to form a sol (a colloidal suspension), and when the solvent was removed by heating, the residue was a finely divided olivine powder. By following the same steps without iron, he was able to synthesize a precursor for forsterite. (Hydrogen peroxide is essential to keep the magnesium from precipitating out of solution as magnesium peroxide while the silicon reacts with water.) A patent, recently issued to Burlitch, describes how the resulting sol can be prepared and used to make thin films of various magnesium silicates, including forsterite. The process results in samples of exceptional purity, which have played a central part in Kohlstedt's research on mechanical properties, and have also aided Rüdiger Dieckmann (of MS&E) in his research on the defect properties of olivine.

"So just when I began to get interested in forsterite, I found myself surrounded with experts who could synthesize it in an extremely pure form, process it in thin films, and perhaps grow it in single crystals. All I had to do was get busy and make some lasers."

Meanwhile, Ober was developing a new means of synthesizing forsterite through thermal and chemical processing of polymers. Using Mg and Si derivatives of methacrylate monomers, he was able to create a film of polymer and convert it into a polycrystalline film of forsterite. The advantage of this technique is that the thin films can be lithographically processed to make optical waveguides and other integrated optical devices.

A further development was that Dieckmann had set up a new image furnace capable of growing single crystals of forsterite from the powder produced by Burlitch (see the article beginning on page 19). Such crystals would be needed for any realistic laser research.

So just when I began to get interested in forsterite, I found myself surrounded with experts who could synthesize it in an extremely pure form, process it in thin films, and perhaps grow it in single crystals. All I had to do was get busy and make some lasers.

Early Results with Forsterite As a Gain Medium

This brings us up to the present. My students have installed a forsterite crystal in one of our lasers, and found that it is tunable from 1.2 to 1.32 μm—very close to the desired range—with almost two watts of output power in a continuous wave. A plot of output power as a function of wavelength (Figure 2) shows that forsterite is already a powerful laser that should satisfy many of the users who call me with requests.

We think, however, that we can make this laser even better. The crystal we are now using displays a lot of unwanted absorption, especially at the longer wavelengths. This absorption increases the overall loss of laser light in the optical cavity (the zone between the two mirrors), reducing power, efficiency, and tuning range. We suspect that the tuning range for an optimized crystal could extend up to 1.4 μm, and the overall power output could nearly double. While few people need four watts of power, improved efficiency would mean that less pump power would be needed to achieve current levels of performance.

The undesirable absorption results, we believe, from Cr ions that are in the wrong location in the crystal (see Figure 3). Cr ions that are added to the materials to make the crystal laser-active can incorporate themselves into the lattice in several locations: some go in as Cr^{2+}, substituting for Mg^{2+} ions; some go in as Cr^{3+}, also at the Mg^{2+} sites; and some can go in as Cr^{4+} at the Si^{4+} sites. The sites and charge states that the ions eventually choose depend on the growth conditions and on other impurities in the crystal. For example, to get Cr^{3+} ions at an Mg^{2+} site, some charge compensation is needed elsewhere in the structure, through a suitable impurity. The formation of Cr^{2+} can be inhibited with a proper oxygen atmosphere during the growth of the crystal.

Our effort to create crystals of the best optical quality is a major challenge. It requires close collaboration involving optical characterization by my group, crystal-growth optimization by Dieckmann's group, and adjustment of precursor composition by Burlitch's group. The identity of the laser-active Cr ion is not yet well established, although the consensus is that a Cr^{4+} ion located at a tetrahedral site is responsible for the laser action. We hope to confirm this through optical spectroscopy in my laboratory, electrical conductivity measurements in Dieckmann's laboratory, and high-frequency electron spin resonance (ESR) studies by Jack Freed in the Department of Chemistry. If Cr^{4+} is the desired dopant, then Cr ions at other sites and in other charge states represent, at best, unused Cr, and at worst, unnecessary absorption losses. Much of the loss in our laser, we believe, is due to excess Cr^{2+}. If this can be eliminated through careful synthesis and growth, laser performance should improve dramatically.

Recently, Burlitch and his graduate student Dong Gon Park prepared pure Cr-doped forsterite from a magnesium silicate sol and a chromium salt, and from the resulting microcrystalline powder, Dieckmann is growing a single crystal in a new arc-image furnace (see page 19). This ability to synthesize and grow our own material makes possible a wide range of experiments. For example, if thin films of single-crystal forsterite can be grown on a substrate, integrated optical circuits based on tunable sources could be constructed. Such

devices would be especially interesting for sensors, optical amplifiers, and instrumentation.

Both Burlitch and Ober are trying to develop methods for synthesizing thin films from precursor material.

Burlitch is exploring the possibility of converting his synthesized powder into a single-crystal layer through sol-gel deposition techniques followed by annealing, or perhaps through laser ablation techniques, in which the powder is ablated by intense laser pulses, and the resulting vapor redeposits epitaxially on a single-crystal substrate.

Ober is exploring the creation of forsterite from polymers. Putting a thin film of organic polymer down on a substrate is fairly routine; the difficult part is to process that thin film so as to transform it into the desired material. In view of Ober's success in converting thin-film organometallic polymers to forsterite through controlled thermal and chemical processing, it appears that it may be possible to process a thin film of polymer lithographically, creating the outline of an optical structure, and then convert the remaining film into active forsterite. By processing large areas, laser arrays and signal-processing optics could be made.

A little farther in the future are possibilities that might be called "materials engineering." For example, one could replace the chromium impurity with vanadium. Vanadium ions are laser-active in certain crystals, and these or other metallic impurities might produce gain media that would operate at different wavelengths. Or the crystal structure might be modified slightly by changing the atomic constituents. For example, instead of growing Mg_2SiO_4, one might be able to find a way to substitute germanium for silicon, forming Mg_2GeO_4. Electronically, the crystal would be identical to forsterite, but the mass of certain ions would change and this would have a slight effect on the crystal field splitting that determines the laser emission lines of the chromium dopant—we might be able to shift the operating wavelength of the laser to new regions. Such an ability only arises when the synthesis, growth, and evaluation processes are closely linked.

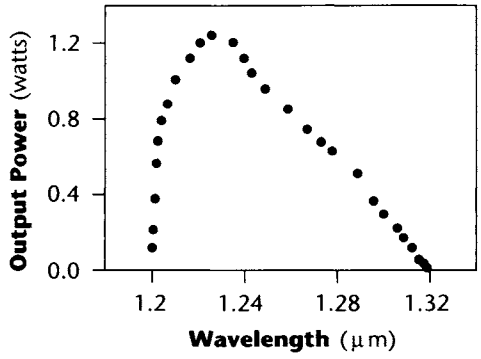


Figure 2. A tuning curve for the forsterite laser. Presently the laser being developed by Pollock and his group spans from 1.2 to 1.32 μm, but with better optics and crystals of better quality, they hope to extend this range in both directions.

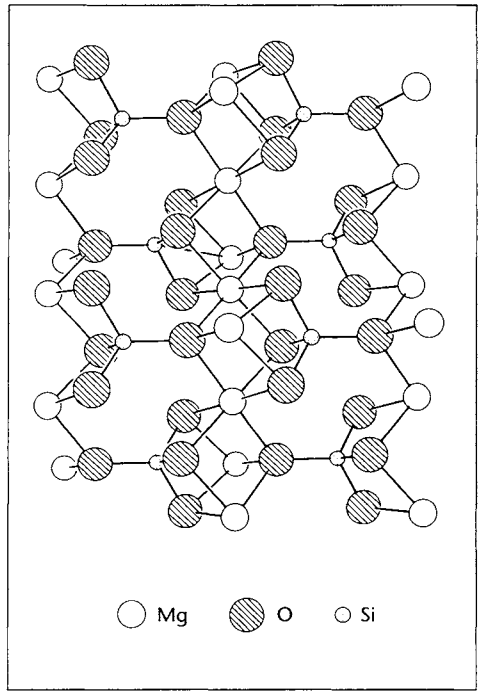


Figure 3. "Ball and stick" diagram of the forsterite lattice, showing the location of Si, Mg, and O atoms. Added Cr ions can substitute into the lattice at either a Mg or a Si site. According to Pollock, which site is best is a mystery still to be solved, and once that is figured out, the Cr has to be convinced to go there.

Clifford R. Pollock, shown in his laboratory, focuses much of his research effort on the development of lasers. His interests extend to fiber optics and quantum electronics.

Future Adventures with Forsterite

Our adventures with forsterite have just begun. Initially, it looks as if forsterite will make a good laser, once we master the problems of crystal growth and doping. But there is much basic research yet to do on this puzzle before we can say that we really understand the system. There is the problem of identifying the critical location of the Cr ion. And then we must determine the optimum synthesis and growth conditions needed to put that ion in that spot. Once these steps are mastered, we can begin to look at new materials and new systems.

Finding four research groups, all in one place, with complementary expertise demonstrates the wonderful advantages of university research. Our project is making good progress because interdisciplinary collaboration is not only possible, but fostered through Cornell's Materials Science Center. As a laser specialist, I would find it difficult—if not impossible—to grow and characterize new lasing materials such as forsterite, as well as to build the lasers. Similarly, my colleagues probably would not have made laser performance an important consideration in their work on the synthesis and characterization of materials. But together we can add new dimensions to each others' research, broaden the horizons of our students, and maybe even make a worthwhile contribution to society. ■

Clifford R. Pollock, an associate professor in the School of Electrical Engineering, is associated with the Cornell Optoelectronics Consortium as well as the Materials Science Center.

Before joining the Cornell faculty in 1983, Pollock earned B.S., M.S., and Ph.D. degrees at Rice University and spent two years as a National Research Council postdoctoral fellow at the National Bureau of Standards.

In 1984 he was one of the first recipients of the Presidential Young Investigator Award, sponsored by the National Science Foundation. This award provides research support for five years.

POLYMERS AND POLYMER COMPOSITES:

A Study Group of the Materials Science Center

by Edward J. Kramer

The development of polymers and polymer-based materials over the last fifty years has affected practically every branch of technology. Once thought of as "low-tech" or "cheap" substitutes for traditional materials such as metallic alloys or soda-lime glasses, polymers and polymer composites have been developed to the point where they can be tailored for a variety of demanding applications. They are used extensively in medicine; for example, devices such as heart-assist pumps that come into contact with blood are made of polymer because parts are easy to fabricate and surfaces can be made nonthrombogenic. Polymers can be made into precise thin films for use as photoresists or interlayer dielectrics in the manufacture of electronic components. High-modulus polymer fibers are used for applications as diverse as airframes and bullet-proof vests (some fibers have an elastic stiffness per unit weight that is eight times that of steel).

At universities, though, progress in polymer science may be impeded by departmental organization. Often people who ought to be working together are separated. For example, the skills in synthetic chemistry that are needed to make polymer molecules for a particular application may be found in one department, while the facilities to characterize the important properties of these polymers may be housed in another department, and faculty members interested in the theoretical underpinnings of the field may be in a third. The study of polymers is thus a natural area for interdisciplinary research, and here at Cornell the Materials Science Center (MSC) plays a vital role in encouraging such research.

Polymer Research in the Materials Science Center

The MSC fosters the formation of interdisciplinary groups that can work together to take advantage of new techniques, new faculty expertise, and new theoretical insights. When the Polymers and Polymer Composites Study Group was formed in the fall of 1989, an important motive was to forge stronger links between faculty groups that were working on the characterization of polymers, and those that were synthesizing them. This formal study group has succeeded in strengthening collaborative research efforts that were already underway, and it will enhance Cornell's ability to capitalize on the burgeoning capability for the synthesis of novel organic and inorganic polymers.

The group consists of faculty members from six schools and departments; five members are principally involved in synthesis, nine in characterization, and four in theory (see Figure 1). Research being carried out within the study group comprises projects in three principal areas—polymer interfaces and films, nanoscale polymer composites, and polymer dynamics.

Funding for the research comes from three sources in addition to the Materials Science Center itself. One source is grants and contracts that are applied for independently by members of the study group. (Not surprisingly, many of these involve collaborations between two or more members.) Another funding source is the MSC-sponsored Polymer Outreach Program, under which corporations fund graduate-student traineeships and send their own scientists to collaborate in the research. Also, there is a grant from the Department of Education for graduate fellowships in the area of polymers—a grant that was awarded to members of the study group under the National Need Fellowships Program.

"... interdisciplinary groups... can work together to take advantage of new techniques, new faculty expertise, and new theoretical insights."

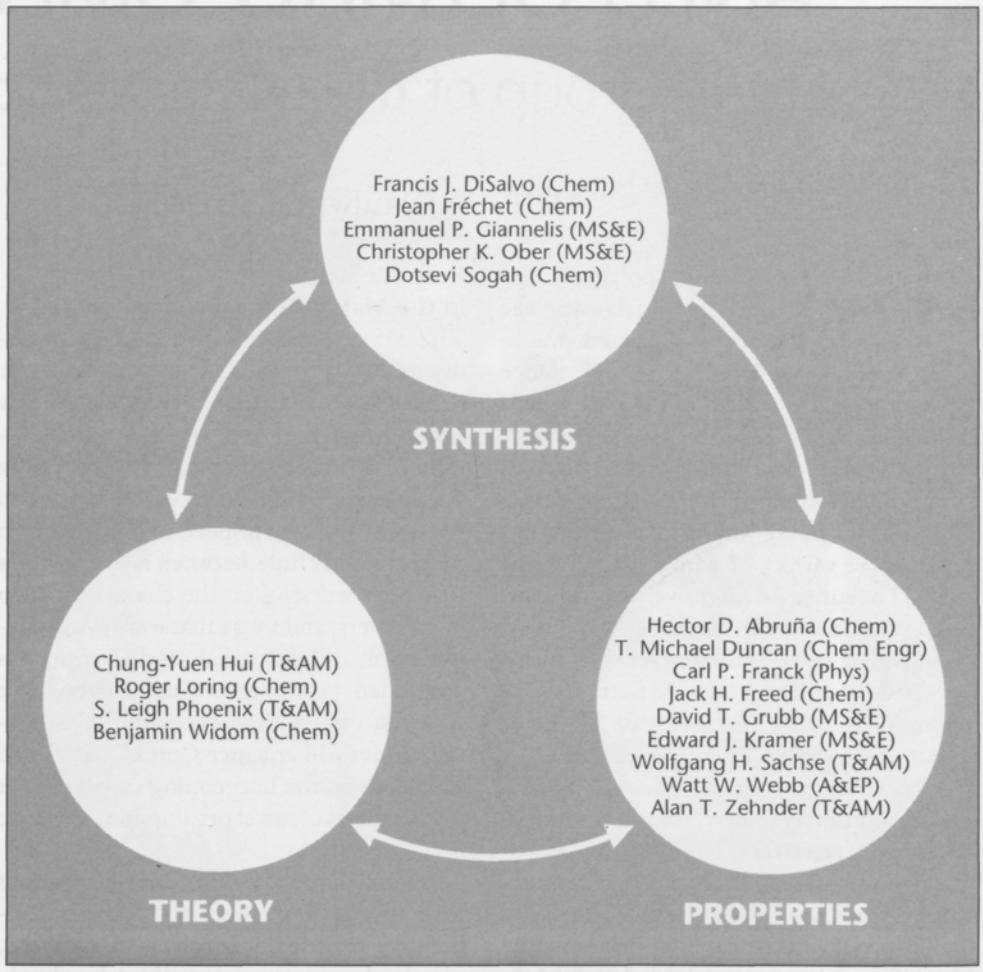


Figure 1. The Polymers and Polymer Composites Study Group. The members are professors who head research programs in six schools or departments: Applied and Engineering Physics, Chemical Engineering, Chemistry, Materials Science and Engineering, Physics, and Theoretical and Applied Mechanics.

In the area of synthesis, three members of the group (Fréchet, Ober, and Sogah) are interested in organic polymers, and two (DiSalvo and Giannelis) are doing work on inorganic polymers.

The theorists develop models for polymer molecules (Loring and Widom) or for polymer composites (Hui and Phoenix).

A number of group members use novel characterization methods to study the properties of polymers and polymer composites. They are Abruña (electrochemical methods), Duncan (nuclear magnetic resonance spectroscopy), Franck (optical reflectometry and ellipsometry), Freed (electron spin resonance spectrometry), Grubb (micro-Raman spectroscopy), Kramer (forward recoil spectrometry), Sachse (acoustic emission and ultrasonic characterization), Webb (two-photon fluorescence microscopy), and Zehnder (speckle measurements of strain).

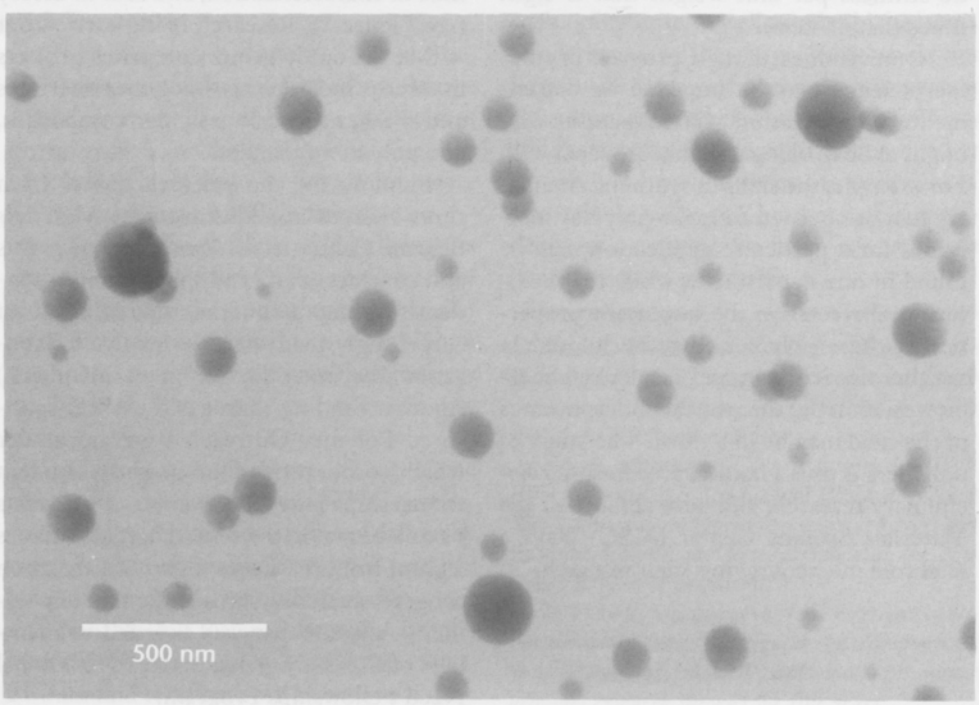


Figure 2. A transmission electron micrograph of a two-phase polymer blend, showing spheres of polyvinylpyridine (PVP) in a matrix of polystyrene (PS). The sample was cast from solution as a thin film and the PVP phase has been stained by exposure to iodine vapor.

The micrograph was taken by Costantino Creton, a graduate student in materials science.

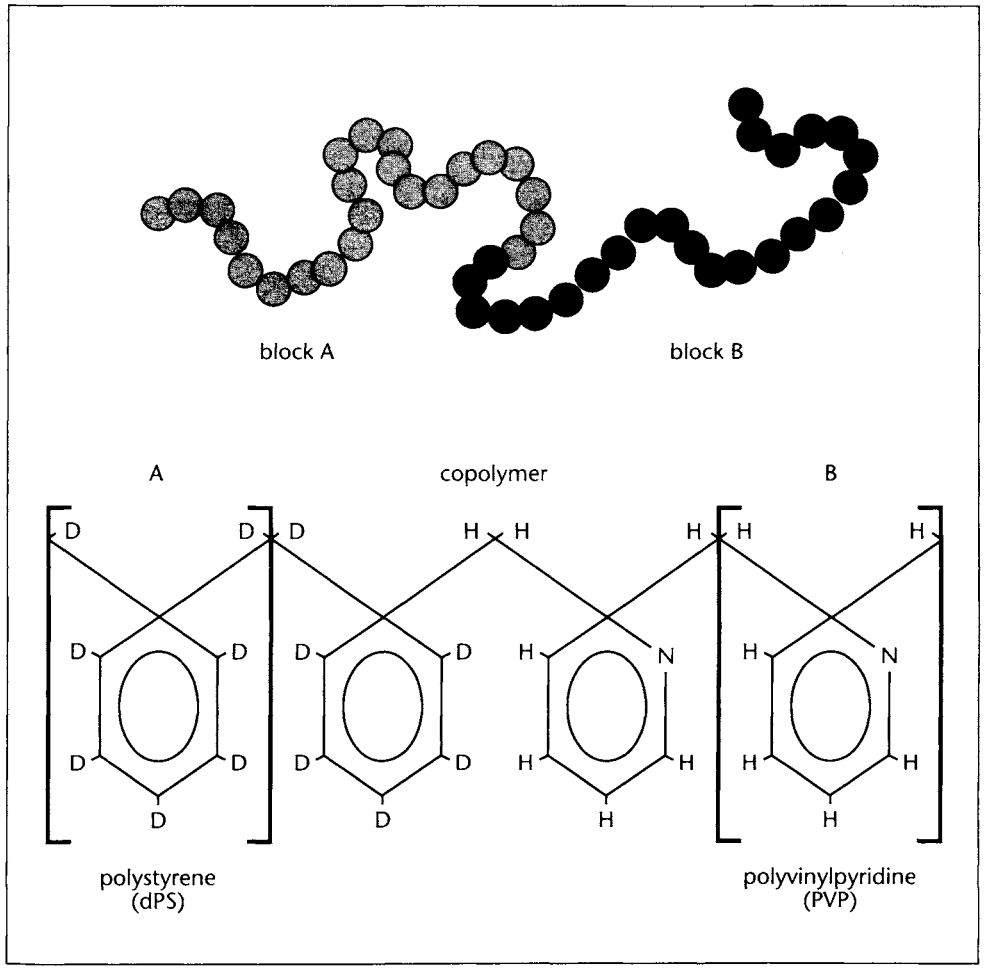


Figure 3. A diblock copolymer, consisting of a block of polymer A joined covalently to a block of polymer B. This structure, which resembles a string of beads, is shown schematically for the dPS-PVP block copolymers discussed in the text. Deuterium atoms (D) in the dPS block act as labels, making it possible to measure the amount of block copolymer at an interface before fracture, as well as how the block copolymer behaves during fracture.

One Group Project: Interfaces in Polymer Blends

One project that exemplifies the collaborative research conducted by members of the Polymers and Polymer Composites Study Group is concerned with interfaces in two-phase polymer mixtures or blends. This work involves several faculty members and their graduate students.

Polymer mixtures can possess some very attractive properties. For example, adding poly(styrene-acrylonitrile) and butadiene rubber to polycarbonate, an expensive polymer, reduces the cost and improves the ease of processing. Effective recycling of plastics also demands that we develop strategies for remelting and reusing polymers—strategies that naturally depend on mixing different polymers together.

Chemically different polymers are almost always immiscible, however, even when melted. They can only be combined so that one polymer forms droplets in the other by mechanical mixing, in much the same way that oil and water mix when they are rapidly stirred. The interface between the polymer phases has a large interfacial tension, which leads to the formation of large droplets of the component polymers after melt processing. (Figure 2 shows a typical two-phase microstructure.) The interfaces are weak, making the entire structure subject to brittle fracture.

The addition of a small amount of a block copolymer, which consists of a block of polymer A joined covalently to a block of polymer B (see Figure 3), is effective in solving this problem. It works in two ways: The block copolymer segregates to the interface between the polymer phases and orients itself with the A block on the A side of the interface and the B block on the B side, acting like a surfactant to lower the interfacial tension. In addition, if

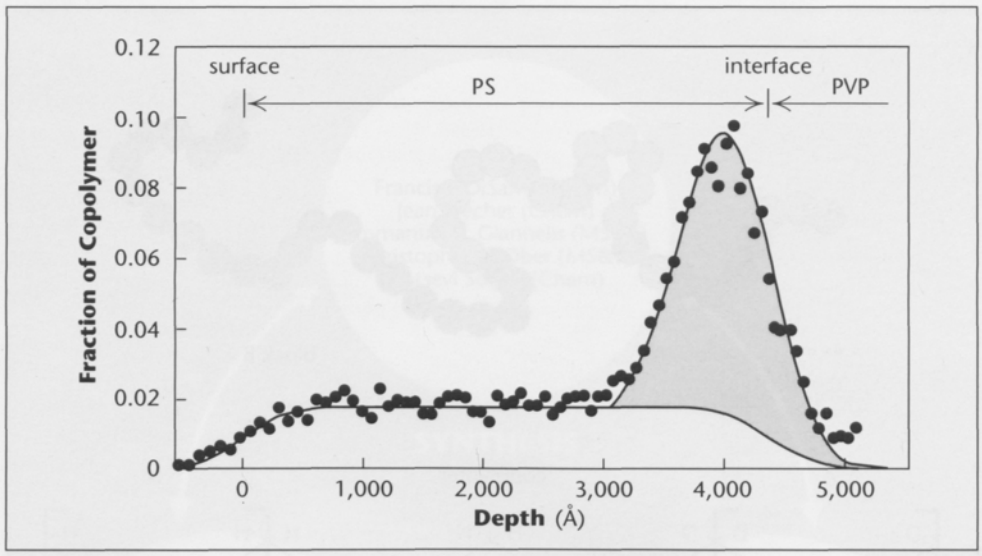


Figure 4. Segregation of the dPS-PVP block copolymer to the interface between PS and PVP.

The profile shows how concentration varies with depth. Each block copolymer chain at the interface has its PVP head in the PVP phase and its dPS tail in the PS phase. The depth profile is determined by using an ion-beam technique, forward recoil spectrometry (FRES), to analyze the sample's deuterium content as a function of depth.

The shaded area under the curve can be used to calculate ν, the number of block copolymer chains per unit area at the interface.

the block copolymer is long enough and is present at the interface in a high enough concentration, it reinforces the interface by entangling with its corresponding polymer. In effect, the block copolymers act as molecular "stitches" that strengthen the interface.

Studying Processes at Interfaces

To achieve practical results, it is necessary to develop a more detailed understanding, at the molecular level, of the processes that are involved in the action of block copolymers. Many skills are required.

We need to know, for example, under what conditions block copolymers are effective in decreasing interfacial tension and increasing interfacial strength. What concentration is high enough, and how long does the block have to be? To answer these questions, block copolymers with controlled chain lengths (such as the one illustrated in Figure 3) must be synthesized.

To study a wide range of chemical structures (a block copolymer with an A block containing a functional group that will hydrogen bond to a C polymer, for example) it will be necessary to use sophisticated techniques such as anionic polymerization of "protected" monomers or group-transfer polymerization.

To be able to measure copolymer concentrations at subsurface interfaces, it is necessary to label the block copolymers with something that does not markedly perturb the way they interact chemically with their surroundings, but that can be detected with depth-profiling techniques. The most convenient way to do this labeling and detection is to replace the hydrogen with deuterium, and then locate the deuterium with one of three techniques that have different and complementary depth resolutions: forward recoil spectrometry (FRES), dynamic secondary ion mass spectrometry (SIMS), and neutron reflectrometry (NR).

Other properties of the interface that must be determined are interfacial tension and fracture toughness. Because interfacial tension is difficult to measure directly in viscous polymer melts, it is necessary to estimate it on the basis of measurement of the concentration of block copolymer at the interface. Fracture toughness of the interface must be determined as a function of the concentration of block copolymer at the interface and the lengths of the two blocks. Finally, models of the micromechanics of crack growth along the interface must be developed so that the measurements can be understood and extrapolated to other systems.

Properties of Model Polymer Interfaces

Planar interfaces between the polymers polystyrene (PS) and polyvinylpyridine (PVP) make excellent models for studying the molecular aspects of reinforcement. Block copolymers (dPS-PVP) can be syn-

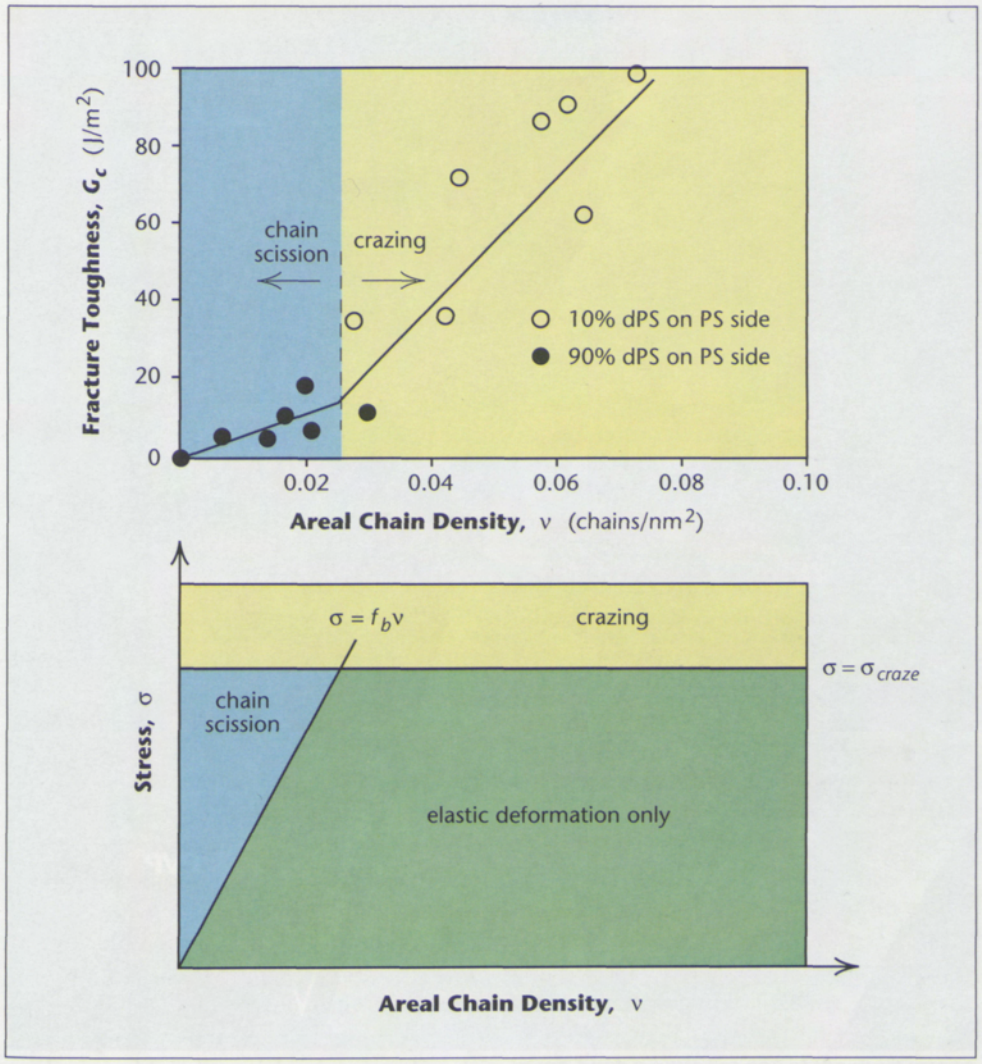


Figure 5. Results of a series of experiments on a two-phase polymer blend of polystyrene (PS) and poly-2-vinylpyridine (PVP). Various amounts of deuterium-labeled block copolymer (dPS-PVP) have been added in order to study its effect on the strength of the interface between PS and PVP. The chains of copolymer are quite long—800 segments of PS and 870 segments of PVP—and so they are well entangled on their respective sides of the interface.

In the top part of the figure, the fracture toughness, G_c, of the PS-PVP interface is plotted versus the areal density, ν, of the copolymer chains at the interface. The transition point in the plot locates a change in how and where the sample fractures under tensile stress.

The figure below, showing tensile stress, σ, in relation to ν, illustrates the transition from *chain scission* at low ν to *crazing* at high ν. At low ν, failure occurs when $\sigma = f_b \nu$, where f_b is the force necessary to break a single block copolymer chain. At higher ν, on the other hand, the interface stress is limited by the crazing stress, σ_{craze}; a craze will form and widen before chains break, thus dissipating much more energy and leading to a higher G_c. From measurements of σ_{craze} ($\approx$ 30 MPa in PS) and the value of ν, at the transition, the force needed to break a single block copolymer chain can be calculated: $\approx 1.4 \times 10^{-9}$ Newtons.

thesized, with one of the blocks labeled with deuterium; we designate the copolymer of PVP and deuterium-labeled PS as dPS-PVP. Then the density of block copolymer chains per unit area at the interface (ν) can be measured by FRES (Figure 4).

Not surprisingly, the fracture toughness (G_c) of the interface increases as ν increases. If both the dPS and PVP blocks of the block copolymer are long enough to become well entangled in their respective sides of the interface, fracture toughness increases significantly. This improved adhesion appears as a transition in the G_c vs. ν curve, as shown in Figure 5. Whereas at low ν, G_c increases hardly at all, at high ν, G_c increases rapidly in proportion to the increase in copolymer molecules at the interface.

This transition also marks a change in where the break occurs when the interface fractures under tensile stress, σ. At low ν, FRES measurement of the two halves of the sample after the fracture shows that almost all of the dPS block has remained on the PS side of the break, indicating that the block copolymer chain has come apart near where the dPS and PVP blocks were originally joined. This form of separation is called *chain scission*. At higher ν, the dPS block is found on the PVP side of the break due to the formation and subsequent fracture of a *craze* in the PS. A craze is a plastically cavitated region of highly oriented polymer in the form of fibrils about ten nanometers in diameter (an example is shown in Figure 6). These fibrils are drawn

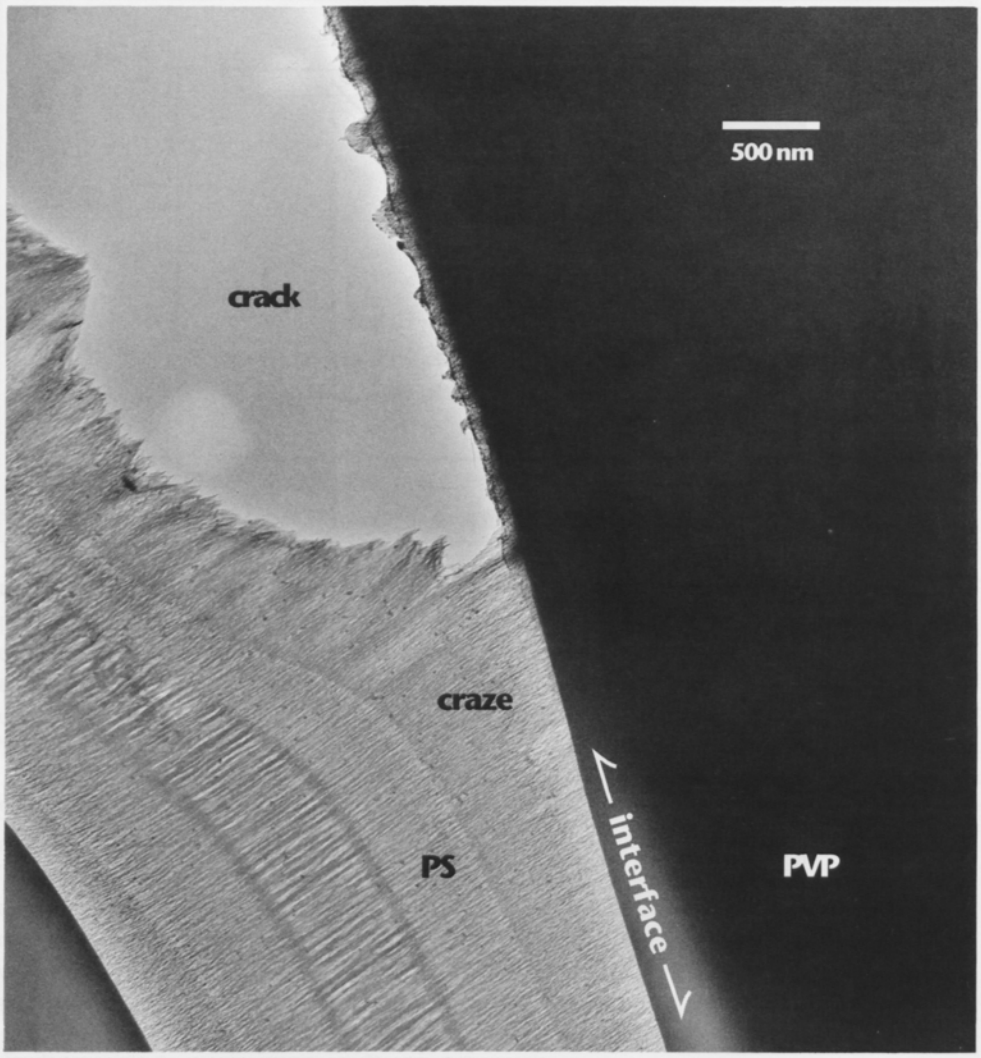


Figure 6. A transmission electron micrograph showing crazing and fracture at an interface in a two-phase polymer blend. A craze has grown on the PS side of the planar interface, and a crack has opened at the interface.

This specimen was produced by applying tension to a thin slice cut from a bulk sample which was then stained with iodine and viewed through a transmission electron microscope at the MSC Electron Microscopy Facility. The interface was formed by spin-coating a PVP slab with a large amount of a dPS-PVP block copolymer that has long dPS and PVP blocks, and then joining this slab to a similar PS slab under heat and pressure. The high areal density of the block copolymer makes the bond relatively strong; weaker interfaces fracture with either a much thinner craze at the crack tip or with no craze at all.

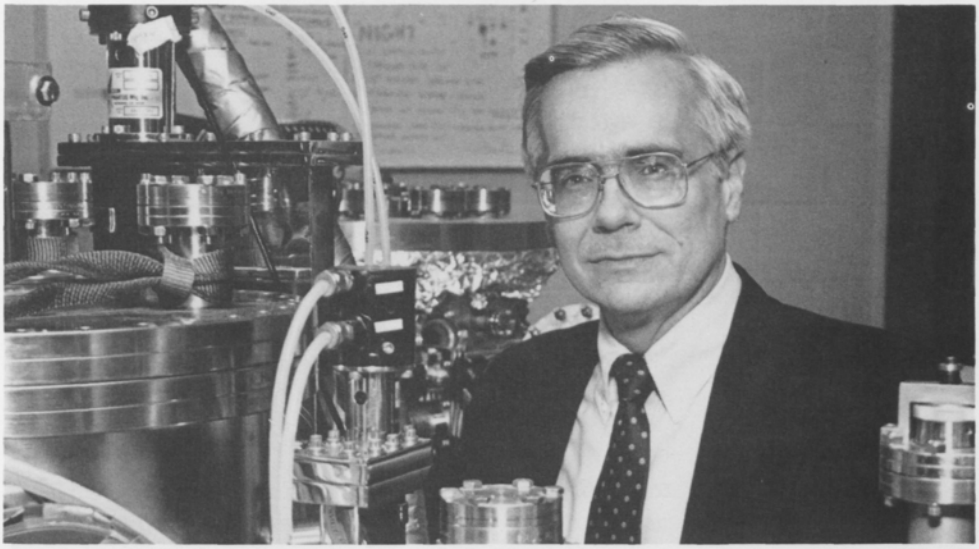
The sample and the micrograph were prepared by Junichiro Washiyama, a visitor from Japan.

from a thin zone of active plastic deformation at the boundary between the craze region and the solid polymer at or above the crazing stress, σ_{craze}.

The hypothesis that craze formation, followed by fracture, occurs on the PS side of the interface at high v is supported by the results of experiments on interfaces in thin films. A slice was microtomed from a bulk sample perpendicular to a reinforced interface, strained in tension, and then examined by transmission electron microscopy. The micrograph (Figure 6) shows that a craze does indeed form and then break on the PS side of the interface.

The situation is different if one of the copolymer blocks is relatively short (shorter than about 200 segments). Then the fracture toughness increases only slowly with areal density and the interface remains very weak regardless of how much block copolymer is added to it. Forward recoil and Rutherford backscattering spectrometry analysis of the fracture surfaces show that the short block "pulls out" of its side of the interface so that all the block copolymer ends up on the opposite fracture surface. This change in fracture behavior is attributed to the inability of the shorter chains to entangle strongly with the polymer on their side of the interface.

Now that we have an understanding of these molecular principles of interface strengthening, we aim to develop new block copolymers that can be securely anchored even when the chains are short.



The Need for Interdisciplinary and Cooperative Research

The project to study the effects of reinforcing two-phase polymer blends with block copolymers provides an example of the advantages of interdisciplinary, collaborative research. This project would be nearly impossible to carry out under the usual "single-investigator" funding of the National Science Foundation, but is typical of the type of problem that can be tackled through the Materials Science Center. Jean Fréchet and his students in chemistry are involved in the synthesis of block copolymers with functional hydrogen-bonding groups and other novel architectures. My students and I, in materials science and engineering, are measuring the concentrations of deuterium-labeled block copolymers at interfaces, using forward recoil spectrometry and neutron reflectometry. Chung-Yuen Hui and his students in theoretical and applied mechanics collaborate with us in measuring the fracture properties of interfaces "stitched" with known concentrations of block copolymer, and in constructing micromechanical models that allow the fracture process of these molecularly reinforced interfaces to be understood. And in its early stages, the project was aided significantly by Hugh Brown of the IBM-Almaden Research Laboratories, who helped develop both the fracture methodology and the micromechanics modeling.

Of course, the strengthening of interfaces is only one of many challenging scientific and technological puzzles in this area. Our study group encourages the formation of interdisciplinary teams, including scientists and engineers from both Cornell and industry, that can focus on specific problems. In such an enterprise, the university researchers benefit from the insights, "real world" knowledge, and points of view that their counterparts in industry can provide, and companies benefit not only from direct participation in the research itself, but also from access to the primary Cornell product: graduates. Those who have worked with the Polymers and Polymer Composites Study Group are well-trained polymer scientists and engineers, experienced in working in an interdisciplinary environment. Such people are one of the most important elements in the effort to improve the competitiveness of American industry. ■

Edward J. Kramer is the Samuel B. Eckert Professor of Materials Science and Engineering. He joined the Cornell faculty in 1967 after earning the B.Ch.E. degree at Cornell in 1962 and the Ph.D. from Carnegie Mellon University in 1966.

Kramer was elected to the National Academy of Engineering in 1989. In 1987 he received a von Humboldt Award and spent the academic year as Gast Professor at the Johannes-Gutenberg Universität in Mainz, Germany. He has also been a visiting professor at other universities in Germany and Switzerland, and a visiting scientist at the Argonne National Laboratory. A fellow of the American Physical Society, he was a corecipient of its 1985 High Polymer Physics Prize for his work on crazing in polymers.

MODULAR PROGRAMS FOR PHYSICAL RESEARCH

by James P. Sethna

"Available computers can generate amazing movies; the bottleneck is in writing appropriate software."

Ready-to-use computer software has revolutionized bookkeeping and publishing, but has had much less impact on scientific research.

The available options for graphics—packages to visually display the results of computation—simply do not satisfy the needs of people working at the leading edge of research in the physical sciences. One can find packages to make two-dimensional *x-y* plots, packages for image analysis that will make a Fourier transform from a picture of an orangutan, and ray-tracing packages that are good for drawing teapots. Such software can be used in the presentation of final results, but it has little value as an aid to research. Physicists need visual feedback that is more sophisticated than the bar charts and graphs used in business. They do not need realistic images; cartoons of their results are quite adequate. But they do want to animate their results—adding the extra dimension of time.

A key deficiency of currently available software packages is their lack of flexibility. Research is, by nature, exploratory. When scientists enter unknown territory, unforeseen numerical and graphical problems arise constantly, and researchers must have broad control of their working environment. Also, performance standards are continually advancing, and it is often necessary to tinker with algorithms and graphics hardware.

In addition to being flexible, software should be efficient to work with. Researchers in the physical sciences have spent years individually writing routines in PostScript®, capturing interrupts and mouse input, and controlling the screen on a pixel level. The edit-compile-run-edit cycle that is needed to explore complex simulations and physical phenomena without interactive control is arduous and time-consuming. Software modules with interactive control, which is the hallmark of commercial packages, could greatly facilitate this work. Available computers can generate amazing movies; the bottleneck is in writing appropriate software.

To meet this need, my students and I have been attempting to write a series of modular programs for graphics and numerical analysis that can be used to expedite research in the physical sciences. Our programs are run directly from the Unix shell, and are intended as graphical extensions of Unix. They are written using X-windows, a user interface analogous to Microsoft Windows®, that has become, in the last few years, the official standard of the workstation market. This software development is Cornell's part of an exchange agreement with IBM. Cornell has received, as IBM's contribution to the work, thirty-four RS-6000 workstations with a retail value of over three million dollars, as well as software and staff support.

During the first year of development, our software modules have had a remarkable impact on the research carried out by the theory group in the Laboratory of Atomic and Solid-State Physics. Some have been ported to the Cornell National Supercomputer Facility. Many are now available by anonymous FTP (a File Transfer Program used by Unix) to users elsewhere in the country (see the box on the opposite page).

The examples presented in this article suggest the capabilities of this new software, although the printed page can only suggest the dynamic potential of animated sequences.

PlotAtoms: A Useful Software Package Developed at Cornell

One of the most useful modules is *PlotAtoms*, which was written by graduate student Bruce Roberts. *PlotAtoms* has been of considerable help to researchers studying everything from the structure of quasicrystals to the molecular dynamics of buckminsterfullerene.

The "unit cell" in a quasicrystal is infinite in extent: all attempts to deduce the atomic configurations directly have failed so far. The

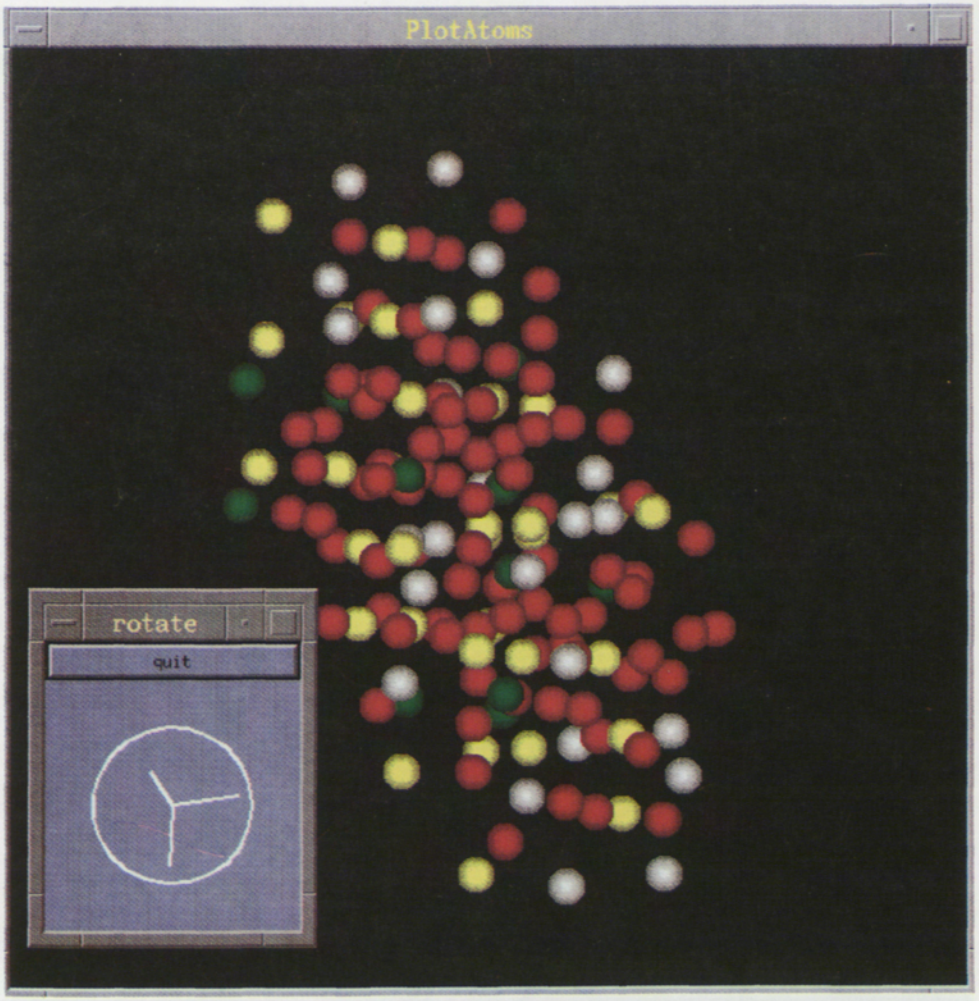


Figure 1. A unit cell from the crystalline phase of T_3(AlMnZn), whose structure is believed to approximate that of decagonal quasicrystals such as $Al_{65}Cu_{20}Co_{15}$ and AlCuNi.

Quasicrystals have long-range translational order, but not of the periodically repeating kind found in ordinary crystals. Since the "unit cell" in a quasicrystal is theoretically infinite, direct determination of its atomic configuration is not possible. Instead, researchers extrapolate from analogous crystalline phases with large unit cells, which are rational approximants to the quasicrystal.

Here, the red atoms are aluminum, the green atoms are zinc, the white atoms are randomly 80 percent aluminum and 20 percent zinc, and the yellow atoms are randomly 80 percent manganese and 20 percent zinc.

most successful method for deducing the structure was pioneered by Cornell Professors Christopher Henley and Veit Elser, who worked from analogous crystalline phases with large unit cells. Figure 1 shows a unit cell from one of these crystalline phases, T_3(AlMnZn), which is thought to approximate the structure of certain quasicrystals. The three-dimensional atomic coordinates were generated by a general-purpose program written by Sergei Burkov, a visiting professor from the Landau Institute for Theoretical Physics, near Moscow. They were then passed through a series of simple filters, the most interesting of which is *rotate*, an input tool written by graduate student Maynard Handley. Appearing in the lower left side of the screen, *rotate* allows one to select a rotation matrix dynamically, by "rolling" the sphere on which the three unit axes are inscribed. The rotated coordinates are then sorted and passed to

Readers who have a machine that runs Unix and X-Windows may obtain a copy of the software featured in this article via "anonymous FTP" by using Internet.

Type:

```
ftp ept. msc.cornell.edu
Name: anonymous; Password: (your e-mail address)
cd pub
get LASSPTools.src.tar.Z
get LASSPTools.demos.tar.Z
```

If you have an RS-6000, you can copy the executables:

```
get LASSPTools.rs6000.tar.Z
```

Or if you have a sparc station from Sun you can type:

```
get LASSPTools.sparc.tar.Z
```

Then type:

```
quit
```

Next, type:

```
uncompress *
tar –xvf LASSPTools.src.tar. . .
```

The compressed files are between one and two megabytes each. Uncompressed, the code currently takes 11 megabytes of storage; about half of this consists of examples. The manual pages are in src/manpages.

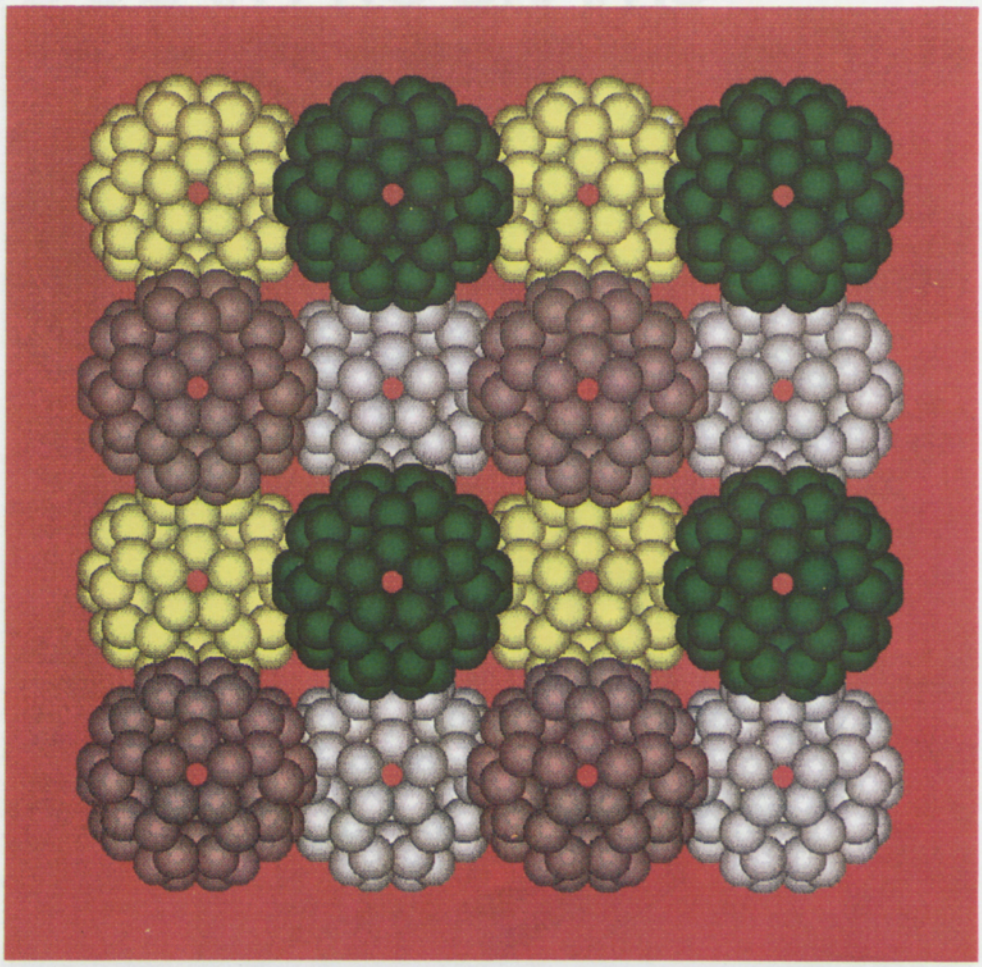


Figure 2. The structure of crystalline buckminsterfullerene. This recently discovered allotropic form of carbon has icosahedral molecules composed of sixty atoms. The substance is named in honor of Buckminster Fuller, inventor of the geodesic dome, which has a similar structure.

Since the molecules are nearly spherical, they continue rotating at temperatures below the point at which the fcc crystal is formed. But at sufficiently low temperature, the slight corrugation of the molecules leads to an ordered state in which molecules in different sublattices assume different orientations. Understanding these phenomena is facilitated by seeing, in real time, how the simulated molecules respond to changes in temperature and pressure at various intermolecular potentials.

This simulation was generated by graduate student Zheng Chen and Assistant Professor Veit Elser.

Developers of modular software for physical research. Around the circle, starting from the left, are graduate student Albert Putnam; undergraduate physics major Jet Ho; graduate students Bruce Roberts and Christopher Myers; James Sethna; graduate student Tom MacFarland; undergraduate physics major David Chin; graduate students J. Ken Burton, Jr. and George Stecher; and undergraduate physics major Eric Lochstet. Visible on the computer screen is *XYInput*, which was developed by Ho.

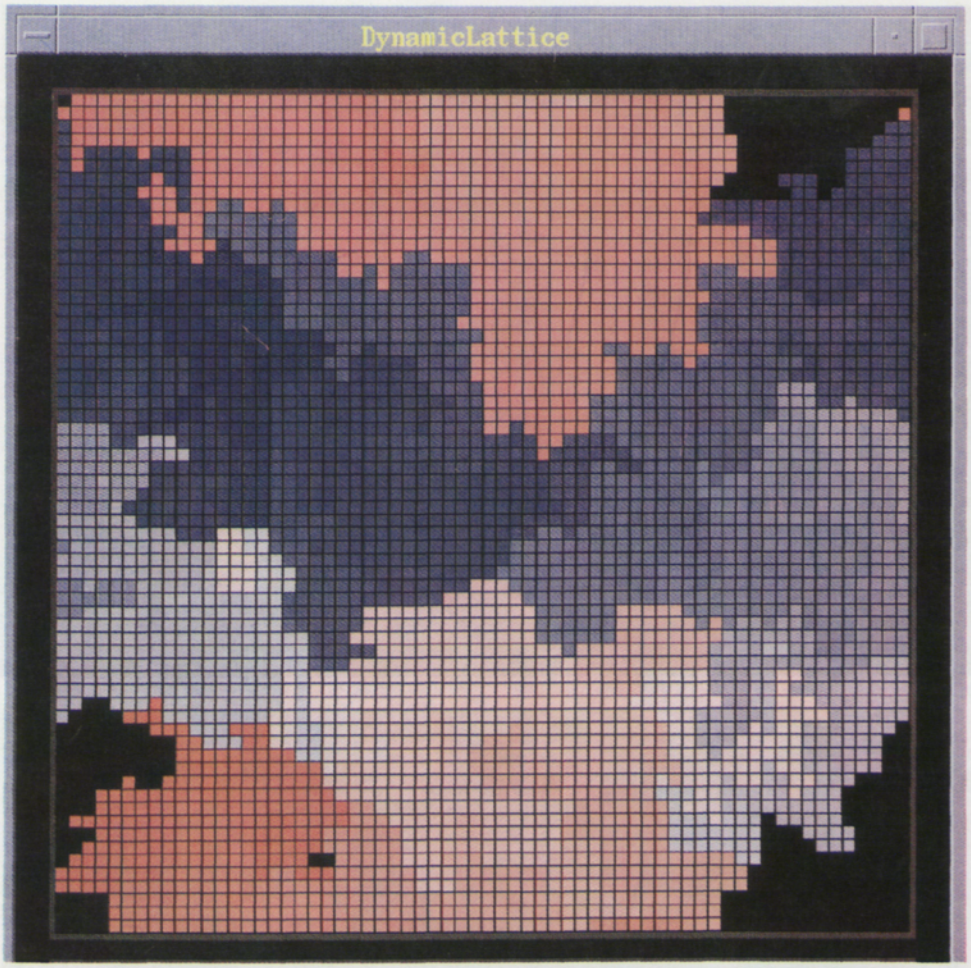


Figure 3. A snapshot of the evolution of a model charge-density wave as it slides through a period. Many metallic materials, especially those with highly anisotropic electronic structures (quasi-one-dimensional), undergo a phase transition in which electron density and lattice positions modulate spontaneously along one axis. This modulation reduces the net conductivity of the material, sometimes making it into an insulator.

At one time the idea was advanced that the modulation, once started, would not stop, and it was proposed as a mechanism for superconductivity. Actually, the opposite is true: the charge-density wave is pinned down by impurities in the material, and does not conduct. However, if a large enough field is applied, the wave can "depin." As the depinning threshold is approached from above, the periodic motion of the wave becomes increasingly jerky—a consequence of large avalanches triggered by the slippage of individual sites.

In this simulation, color indicates time of firing: blue sites have already fired, pink sites are currently firing, and red sites have yet to fire.

This simulation was generated by graduate student Christopher Myers.

PlotAtoms, which displays them using a series of preformed bitmap images. Turning the unit cell around in all directions makes it possible to glean real information from an otherwise inscrutable mass of atoms.

PlotAtoms has also been indispensable in the study of the molecular dynamics of buckminsterfullerene (Figure 2). The software was not just used to make pretty pictures of results, but provided an essential interactive tool right from the debugging stage of the research. *PlotAtoms* has also been used by Professors Albert Sievers and Michael Teter to study molecular dynamics, and by Associate Professor Barbara Cooper's group to study the way ions scatter off surfaces. With the assistance of the Cornell Theory Center, we have used *PlotAtoms* to generate a video tape on ion scattering that graduate student Bruce Roberts showed at the March Meeting of the American Physical Society.

Software for Studies from Atomic Structure to Earthquake Dynamics

Another very useful module, *DynamicLattice*, was written by graduate student Christopher Myers while doing the research that led to his doctoral dissertation. Later he adapted it for general use. Myers's study of the evolution of charge-density waves (see Figure 3) is one example of what it can do. In addition, its arrow mode makes it useful in studying magnetic spin systems, and its bond mode has been employed in modeling dynamical percolation. Jean Carlson and Jim Langer, faculty members of the University of California at Santa Barbara, have been using it to animate their simulations of earthquake dynamics.

Grid2Contour, written by adjunct professor Michael Teter and graduate student Maynard Handley, was used to generate an electron-density contour surface for rutile (Figure 4). The electronic structure was determined us-

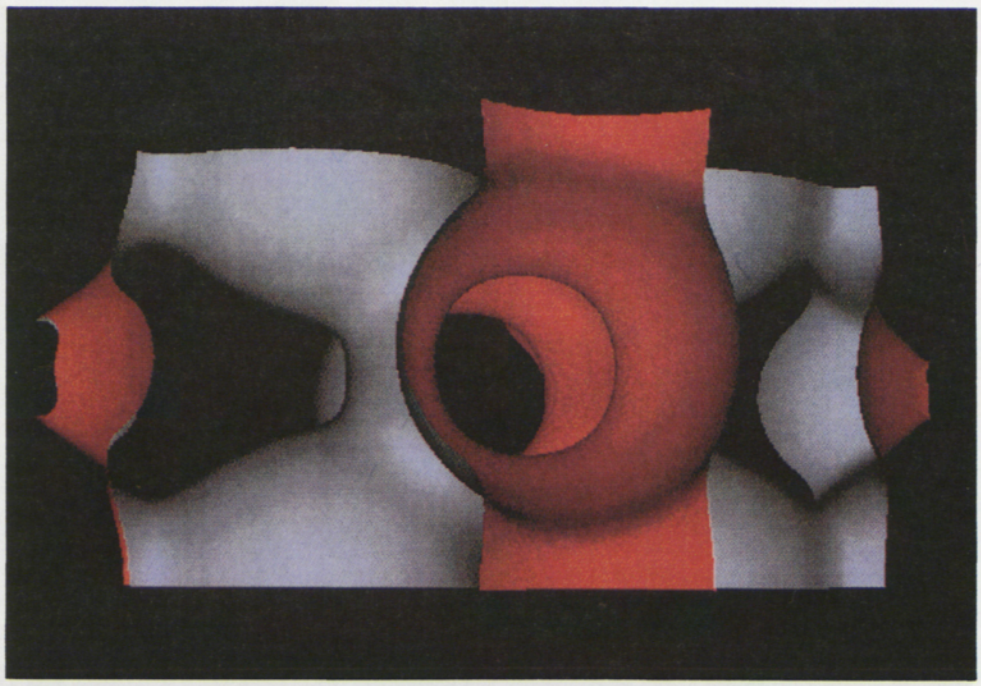


Figure 4. An electron-density contour surface for titanium oxide (TiO_2) in the rutile structure. It was constructed with use of *Grid2Contour* from a 3-D grid of electron densities by using an interpolation algorithm and a simple ray-tracing method.

Here the surface chosen for simulation is at the Van der Waals radius of the oxygen atom. The interior of this surface is colored red, and the exterior, blue. The atom shown in the middle is titanium, and the blue tubes represent strongly polarized oxygen atoms.

This simulation was generated by Michael Teter (an adjunct professor) and Douglas Allen, both of Corning Incorporated. Teter likes to compare this calculation with that of stichiovite, a high-pressure form of SiO_2 found in meteor craters. Stichiovite shares the rutile structure, but is not as stable. Partly this is because the titanium atom is larger, but mostly it is because the oxygen atoms are not nearly as polarized in stichiovite: the d-orbitals of titanium are important for polarizing the oxygen atoms (the blue tubes) and stabilizing the structure.

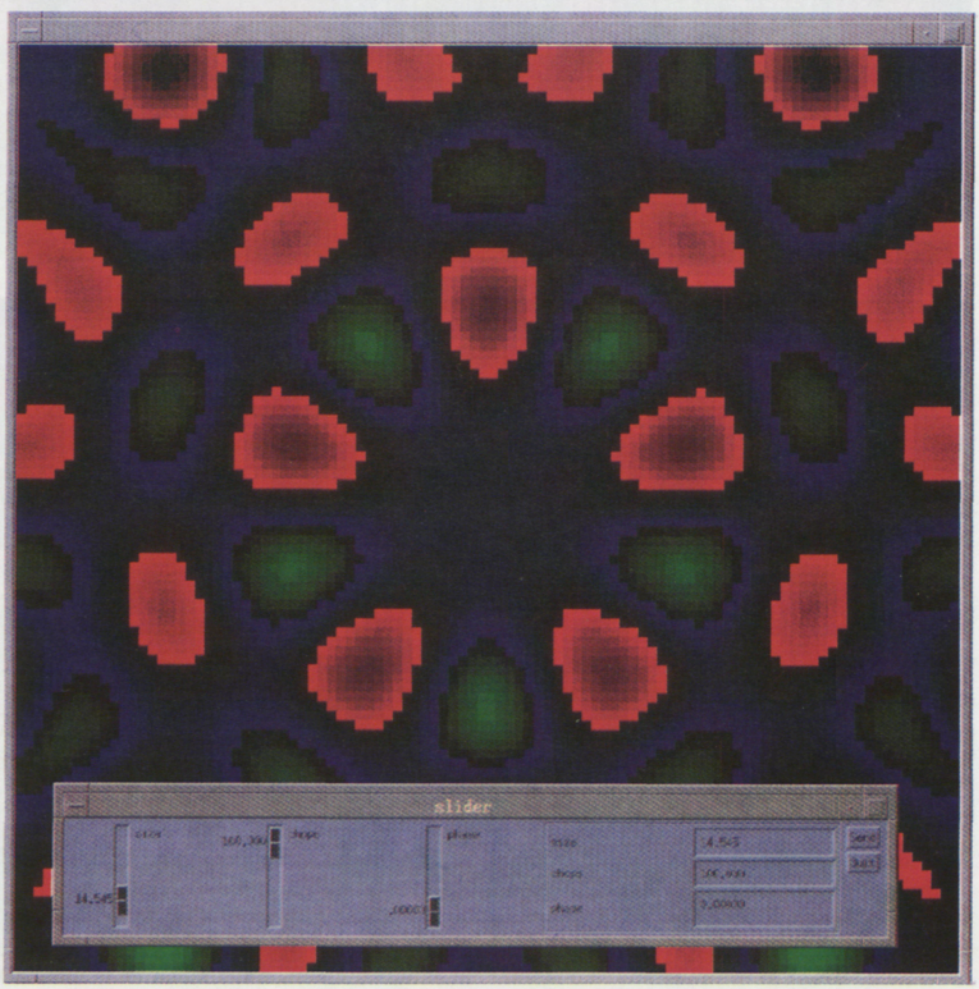


Figure 5. The sum of five cosine waves traveling in the directions defined by the sides of a regular pentagon. The red regions, which may be presumed to represent atoms, are logically and regularly set out, with sensible separations, despite the lack of a periodically repeating pattern.

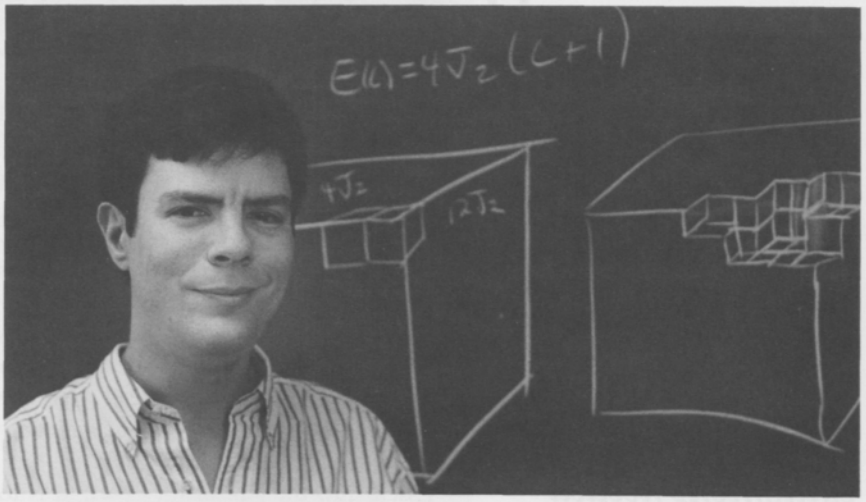
A similar construction was proposed earlier by Professor David Mermin and graduate student Sandra Troian for their theories of quasicrystals.

ing an all-plane-wave local-density code. The contour surface was constructed from a three-dimensional grid of electron densities by using an interpolation method and a simple ray-tracing method that generates a smooth surface without the traditional ambiguities of polygonal surface reconstruction. *Grid2Contour* is about two orders of magnitude faster than traditional ray-tracing methods, but it still takes several seconds to construct a contour surface from a given viewpoint. Frames can be stored up, however, and shown in sequence to form a movie.

Figure 5 shows the sum of five cosine waves in an animated demonstration that can be used in class discussions of quasicrystals. Two important tools are demonstrated: *Slider* and *Matrix2X*. *Slider* was written by undergraduate physics major Jeffrey Osterman and is our most heavily used analog input device. It allows parameters to be adjusted either by sliding on-screen controls with a mouse or by editing numerical values. Three sliders were used to prepare this graphical presentation: the first controlled the size of the quasicrystalline region depicted, the second controlled the resolution (100 x 100), and the third controlled the sum of the phases of the plane waves. The output uses *Matrix2X*, which takes the values of a sequence of two-dimensional grids and displays them as a color animation.

Graduate student Sivan Kartha and I have been using *Matrix2X* to study the "tweed" deformations found in certain metallic alloys. Before undergoing a spontaneous stretching "martensitic" transition, these materials show a cross-hatched pattern in electron micrographs. I showed movies of our model for tweed made with *Matrix2X* in meetings held in the Los Alamos and Brookhaven National Laboratories.

Other important tools developed by our project are *PolyDraw* (written by graduate student Stephen Townsend) and *Poly2ps* (written by graduate student Joel Shore). These packages allow animations of general polygonal constructions to be drawn on the screen or to be output through a laser printer. *PolyDraw* will eventually be used to study polygonalized three-dimensional surfaces, but it already has been invaluable in studying martensites and slow, glassy coarsening in frustrated Ising models. With the aid of the Cornell Theory



Center, Joel Shore developed a videotape showing this coarsening, and I presented it recently during talks delivered at the University of California. Much physical research has been done with the aid of transparencies, but animations are the transparencies of the future.

Numerical analysis tools currently under development are filters for interpolation and smoothing, filters for nonlinear fits to data (using *Slider* to interactively find approximate fits, and nonlinear least-squares to fine-tune), and filters for matrix manipulations, Fourier transforms, and eigensystems.

In a grand view, we hope to do for numerical analysis and graphics what most operating systems do for file manipulation and text processing. We hope to provide the obvious components necessary for use in a wide variety of scientific and engineering applications. ■

"... we hope to do for numerical analysis and graphics what most operating systems do for file manipulation and text processing."

James P. Sethna, an associate professor of physics, has been at Cornell since 1984. A member of the Laboratory of Atomic and Solid State Physics as well as the Materials Science Center, he conducts research in the areas of glasses, defects in liquid crystals, quantum tunneling, and dynamical systems and has published some fifty papers.

Sethna received the B.A. degree in physics from Harvard University in 1977 and the Ph.D. from Princeton University in 1981. Before joining the Cornell faculty, he did postdoctoral work here and at the Institute for Theoretical Physics at Santa Barbara.

In 1985 he received a Sloan Resesarch Fellowship and a Presidential Young Investigator Award, which provided research support for five years through the National Science Foundation.

THE MSC FACILITIES: A USER'S POINT OF VIEW

by Jean Lee

". . . they provide the sophisticated equipment needed for the various steps in the sample-making process, and expert help in its use."

As most electron microscopists know, the ability to consistently produce good ceramic TEM samples overqualifies one for an alternate career in neurosurgery. This is, at any rate, one of the conclusions I have reached in carrying out my thesis project.

Essentially, the aim of my research is to check experimentally the theoretical prediction of an interesting property of a particular ceramic-ceramic interface. It is part of an investigation of defects in materials that is headed by Stephen L. Sass, professor of materials science and engineering. The most difficult job encountered in my project is to prepare good samples for examination in a high-resolution transmission electron microscope (TEM).

This is where the facilities of the Materials Science Center (MSC) contribute: they provide the sophisticated equipment needed for the various steps in the sample-making process, and expert help in its use. The work is accomplished conveniently and economically, if not exactly easily.

The Project: NiO Structure at a Grain Boundary

The focus of Professor Sass's overall program is the structure of two-dimensional defects—in particular, grain boundaries and interfaces. This is an important study because defects are intimately involved in determining the physical behavior of materials. For instance, the mechanical, electrical, and transport properties of a material may be affected by the presence of zero-, one-, two-, or three-dimensional defects.

My project deals with the structure of ceramic-ceramic interfaces; specifically, I am using a high-resolution TEM to probe the structure of NiO/NiO coincidence twist grain boundaries. (A coincidence twist grain boundary is the interface between two adjacent single-crystal grains that are oriented the same way except that one grain is rotated with respect to the other by a misorientation angle θ_t about an axis normal to the interface.) NiO is a good material for this study because its physical characteristics are well documented and it has a simple structure in its perfect, single-crystal form. In addition, it has that interesting theoretically-predicted property: computer simluations show that the $\Sigma 5$, [001] twist boundary in NiO is unstable unless a Schottky defect is introduced in each of the grain-boundary unit cells.[1] (In this designation, Σ indicates the ratio of the density of perfect lattice points to the density of grain boundary lattice points, and [001] indicates the rotation axis about which one grain is misoriented with respect to the other.)

I am seeking to check this prediction by seeing how the atomic structure may vary at the grain boundary in an appropriately aligned NiO–NiO sample. Using the high-resolution TEM, I hope to see contrast changes at the boundary that would indicate a change in the mean inner potential at the boundary[2] and hence possibly a change in atomic density and at the boundary.

Preparing Specimens: the Initial Task

The first step in the sample-preparation process is to grow single crystals of NiO onto substrates of commerically available, polished [001] MgO. The NiO is grown to a thickness of approximately 20 to 30 micrometers by chemical vapor deposition in a dedicated furnace in our lab. In order to produce a coincidence twist boundary, one of the single crystals of NiO is rotated to the appropriate misorientation angle θ_t with respect to another single crystal, and then the two are hot pressed in this configuration (Figure 1).

It turns out, however, that because of the

[1]Tasker, P. W., and D. M. Duffy, *Phil. Mag. A* 47:6 (1983).

[2]Ruhle, M., and S. L. Sass, *Phil. Mag. A* 49:6 (1984).

Left: Jean Lee works with the high-resolution transmission electron microscope in one of the eight facilities of the Materials Science Center.

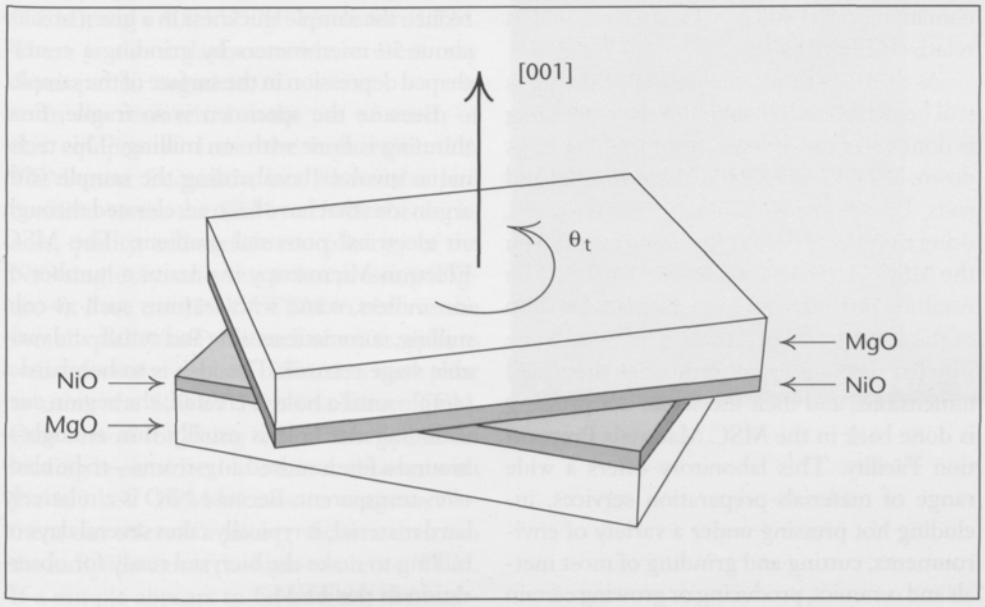


Figure 1. The configuration of a [001] twist grain boundary with misorientation angle θ_t. The NiO single crystal is about 20 to 30 micrometers thick, and the MgO substrate is approximately 1 millimeter thick.

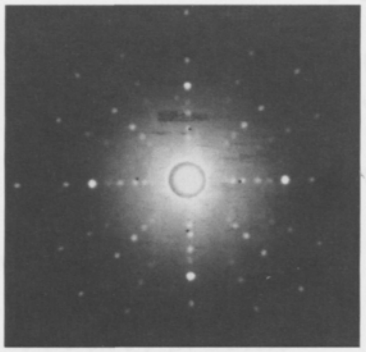


Figure 2. Laue back-reflection photograph of a bare (001) single-crystal MgO substrate. The photograph shows that the substrate is well oriented; the round diffraction spots indicate very little distortion of the crystal.

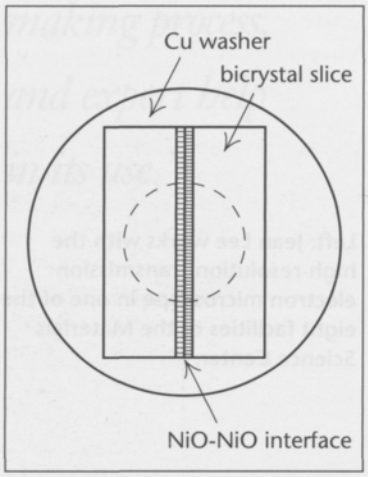


Figure 3. Top view of a bicrystal slice mounted for cross-sectional TEM observation. The copper washer provides support during the thinning process.

growth morphology of [001] NiO, better bonding between the two crystals is achieved when they are polished prior to the hot pressing. The MSC Metallography Facility, which has grinding and polishing equipment, comes in handy here. Since NiO is a relatively hard material, I polish these crystals using diamond paste (containing diamond particles on the order of a few micrometers in size) to smooth out the pyramidal structures that may occur on the NiO surface. Other substances for grinding and polishing, such as SiC and alumina, are also available.

At this point it is sometimes useful to check the quality of the NiO film or the MgO substrate. This can be done with diffraction techniques using the machines in the MSC X-Ray Facility. The substrate may be examined using the Laue back-reflection or the real-time Laue equipment (Figure 2), and diffractometers designed for thin-film analysis are available at this facility.

After cleaning, the NiO crystals are ready to be placed in a specially designed jig for orientation and hot pressing. Being a refractory material, NiO requires a relatively high hot-pressing temperature—about 1300°C. Since most metals melt at this high a temperature, and the few that don't are expensive and difficult to machine, jigs are usually made of a ceramic material (although ceramics are also generally difficult to machine into complicated shapes). For my project I am using an alumina jig, which apparently does not contaminate the NiO-NiO interface and is relatively inexpensive.

As of this writing, the design of the jig is still being refined, so much of the machining is done in-house as a means of keeping costs down. When I receive the alumina tubes and rods, I make the preliminary cuts on them, using a universal cutting/grinding machine in the MSC Materials Preparation Facility. The resulting pieces are taken to the machine shop of the Laboratory of Atomic and Solid State Physics, where they are ground to their final dimensions, and then the actual hot pressing is done back in the MSC Materials Preparation Facility. This laboratory offers a wide range of materials-preparation services, including hot pressing under a variety of environments, cutting and grinding of most metals and ceramics, producing or growing certain materials (such as the rod of NiPt that a fellow group member requires for his work), and mechanical testing (also of interest to other members of my group).

If the bicrystal that results from the hot-pressing step does not break upon handling or exhibit excessive cracking, it is ready to be cut into pieces measuring about 3 millimeters by 1 millimeter for cross-sectional examination. The cutting is done in our lab with a wire saw fitted with a diamond-impregated wire blade.

The pieces that survive the sawing undergo a thinning process in order to make them electron-transparent. This is accomplished by first mounting a bicrystal slice on a specimen holder and then, back at the MSC Metallography Facility, mechanically grinding and polishing it using successively finer diamond pastes. This thins the sample to approximately half its original thickness. At this point, a small copper TEM washer is glued to the surface of the specimen to provide support as the specimen becomes thinner (Figure 3). After the washer has bonded to the sample, the thinning process is continued by removing, inverting, and remounting the sample/washer assembly onto the specimen holder and repeating the mechanical polishing procedure on the other side of the bicrystal slice. The sample is thinned to approximately 100 micrometers in this way.

Next the bicrystal—provided it is still unbroken—is further thinned with the use of a dimple grinder in our lab. This apparatus can reduce the sample thickness in a given area to about 50 micrometers by grinding a crater-shaped depression in the surface of the sample.

Because the specimen is so fragile, final thinning is done with ion milling. This technique involves bombarding the sample with argon ions that have been accelerated through an electrical potential gradient. The MSC Electron Microscopy maintains a number of ion millers, some with features such as cold milling, automatic sensing and cutoff, and variable stage control. The idea is to bombard a sample until a hole is created; the region surrounding the hole is usually thin enough—around a few hundred angstroms—to be electron-transparent. Because NiO is a relatively hard material, it typically takes several days of milling to make the bicrystal ready for observation in the TEM.

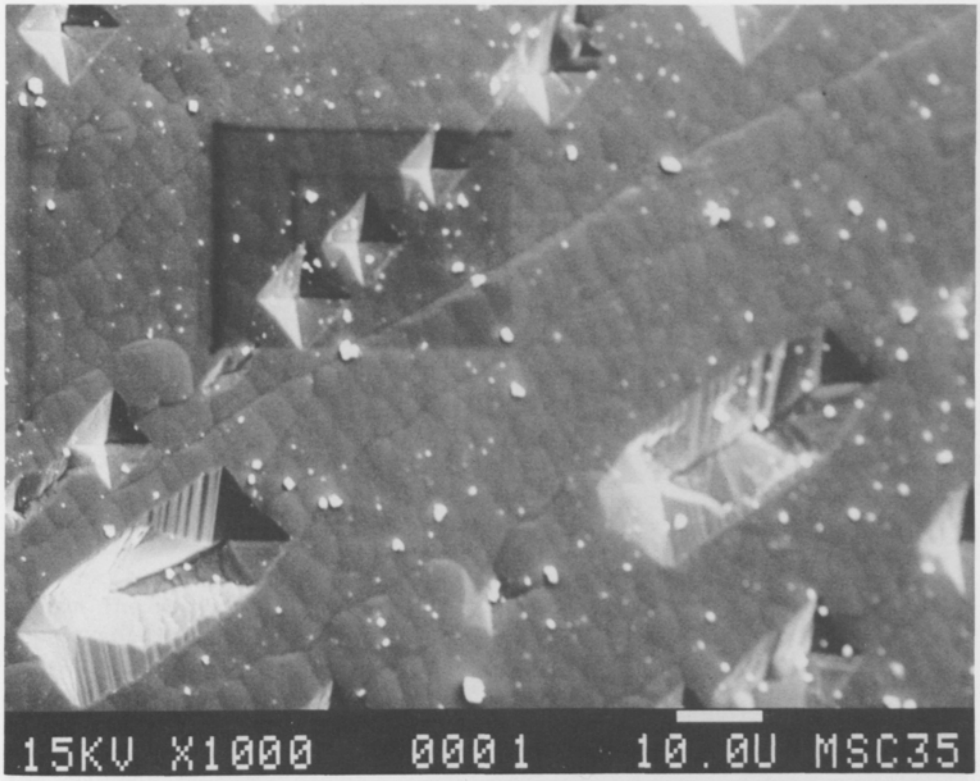
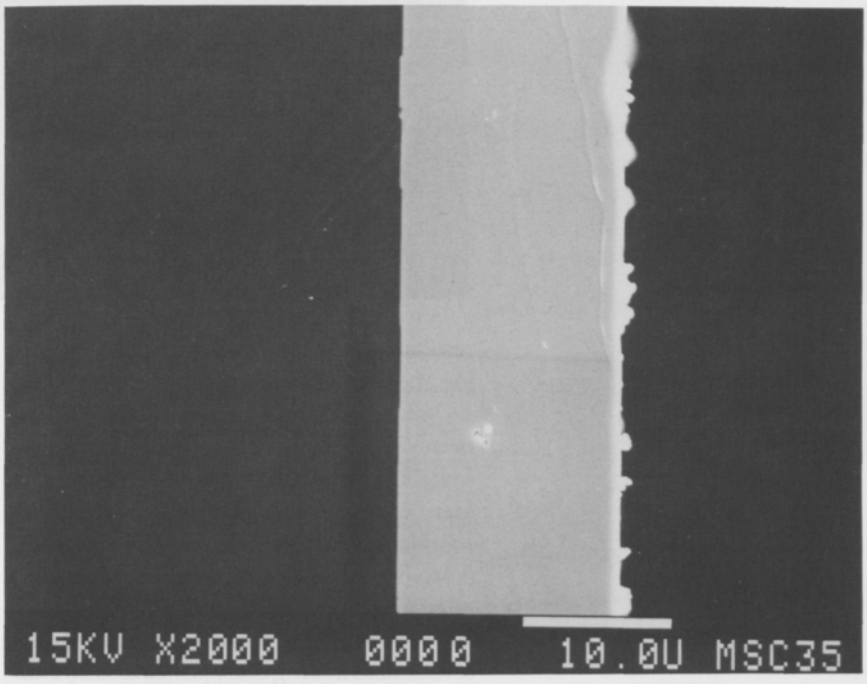


Figure 4a (left). A SEM micrograph showing the surface morphology of a NiO single crystal. The {111} planes of NiO form the faces of the square pyramid structures. Polishing the NiO single crystals prior to hot pressing flattens the surface and helps to provide more contact between the two NiO crystals.

Figure 4b (below). The SEM may also be used for direct observation of the thickness of a NiO single-crystal film.



Examining the Samples by Electron Microscopy

Among the microscopes available in the Electron Microscopy and Microanalysis Facility are a scanning electron microscope (SEM) with energy-dispersive x-ray spectroscopy (EDS) capability; a microprobe; a scanning transmission electron microscope (STEM); a 120 kV transmission electron microscope (TEM), and a 400 kV high-resolution TEM. There is also a darkroom where users may develop their negatives.

In my research, I make use of much of this equipment. I use the high-resolution TEM most frequently, but I have also had occasion to use the SEM to check the thickness, quality, and surface morphology of the NiO single crystals prior to hot pressing (Figure 4). I use the 120 kV TEM to observe the overall quality and structure of my sample, as well as for some electron diffraction work prior to using the 400 kV high-resolution TEM. One reason for doing this is that specimen loading is considerably easier and faster with the 120 keV TEM because its tungsten filament can tolerate a poorer vacuum than can the LaB_6 filament of the high-resolution TEM. If a sample appears to be suitable, the high-

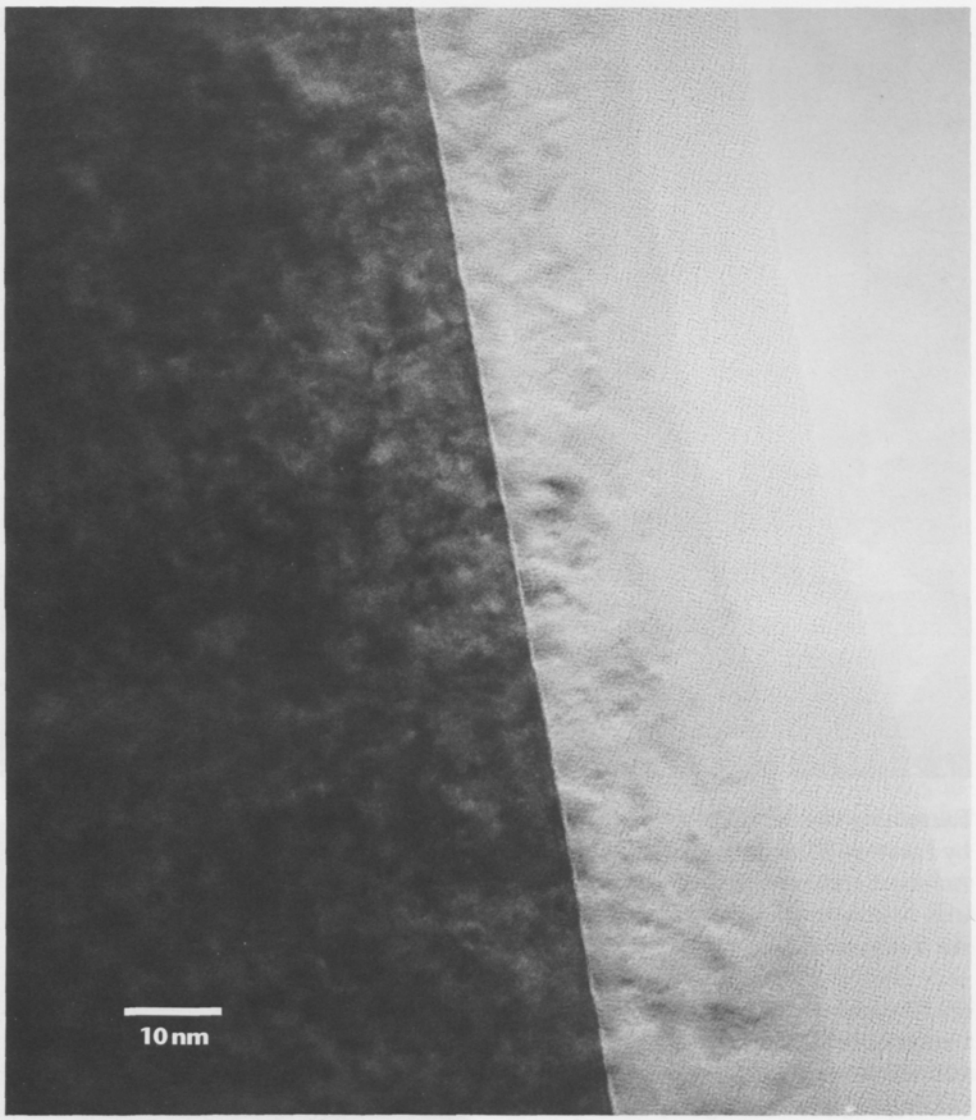


Figure 5. A high-resolution TEM image of a [001] NiO twist grain boundary. The NiO single crystal to the left of the interface is at the [100] orientation. The misorientation angle between the two NiO single crystals is approximately 38°. The periodic change in contrast along the interface is most likely due to grain boundary misfit dislocations.

This photograph was taking using the JEOL 4000EX high-resolution TEM.

resolution TEM is then used for lattice imaging and microdiffraction (Figure 5).

When good electron micrographs have been obtained, they may be brought to the Metallography Facility to be printed. Users may be trained to use the contact printer and an enlarger, allowing them a fair amount of flexibility in choosing the features they wish to emphasize in their prints. Other scientific photography services, such as the production of slides and thesis-quality prints, are also available at this facility, although this work tends to be done by professionals on the staff.

Image Simulation As Part of an Investigation

Once I have pictures of my sample, I can finally sit down and analyze them. However, it might be dangerous for me to draw conclusions about the structure of the NiO grain boundaries solely on the basis of micrographs. Some way of checking these experimental observations is needed, for it is possible to obtain imaging artifacts—introduced during specimen preparation, perhaps, or caused by microscope instabilities.

This is where I turn to the MSC Multiuser Computing Facility in Thurston Hall (one of two maintained by the MSC). Among the TEM image-simulation programs available here is TEMPAS, which can produce the hy-

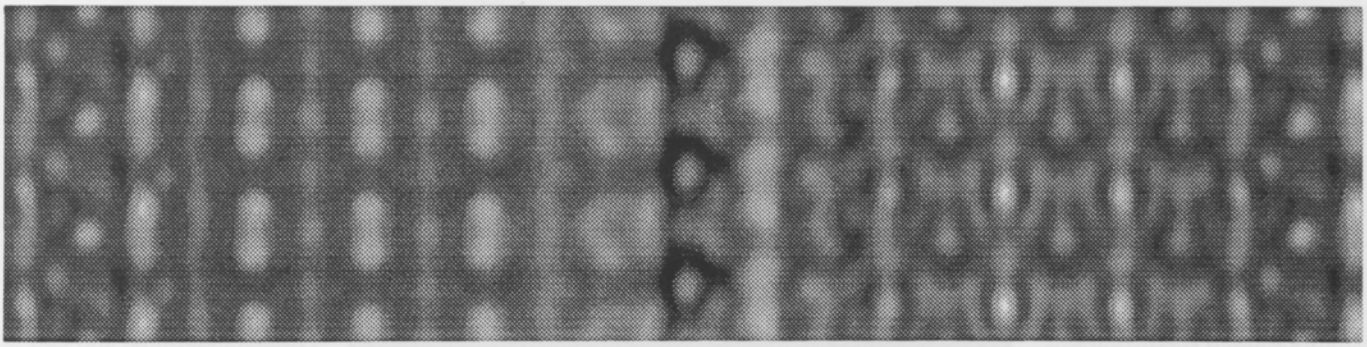
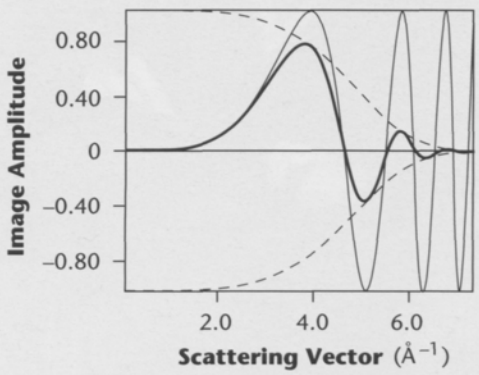


Figure 6. Image simulation with the program TEMPAS for the JEOL 4000EX TEM.

Above: An attempt at simulating the NiO Σ5 [001] twist grain boundary. The foil thickness is 50 Å and the defocus is 0 Å.

Right below: A contrast transfer function (CTF), which gives information about how the microscope can produce imaging artifacts. In particular, it shows the range of scattering vectors over which image contrast may be reversed (for example, black atoms to white atoms).

The CTF shown here corresponds to a 150Å-thick NiO specimen viewed at 0 Å defocus. The heavy curve is the result of imposing damping on the undamped sine curve. The lighter oscillating curve is the undamped sine part of the complex phase change in the electron wave function due to spherical aberration of the objective lens. The "envelope" represents damping due to effects of the spatial and temporal coherence of the beam.



pothetical image one would obtain on a given microscope under given conditions of defocus, specimen thickness, and specimen orientation. For my project, I use a piece of software called *rotncut* (also accessible through the Computer Facility) to construct the grain boundary, and then feed the resulting structure to TEMPAS as an input file. TEMPAS can also produce diffraction patterns, contrast transfer functions, and other information that can help electron microscopists verify their experimental data (Figure 6). Another available image-simulation program can digitize and "massage" a given image—a capability that may be of interest to researchers in other fields as well as to those in electron microscopy.

The Use of MSC Facilities throughout a Research Project

One of the most useful aspects of the various MSC facilities is their accessibility. Staff members are happy to instruct researchers in the use of particular pieces of equipment, and to give advice based on their extensive experience and knowledge. Some people see this as a waste of time—after all, if an apparatus is not a major component of your research setup, why bother spending the time to learn how to use it when there are technicians available to operate it for you? But I believe that hands-on experience is an integral and indispensable part of a researcher's training. ■

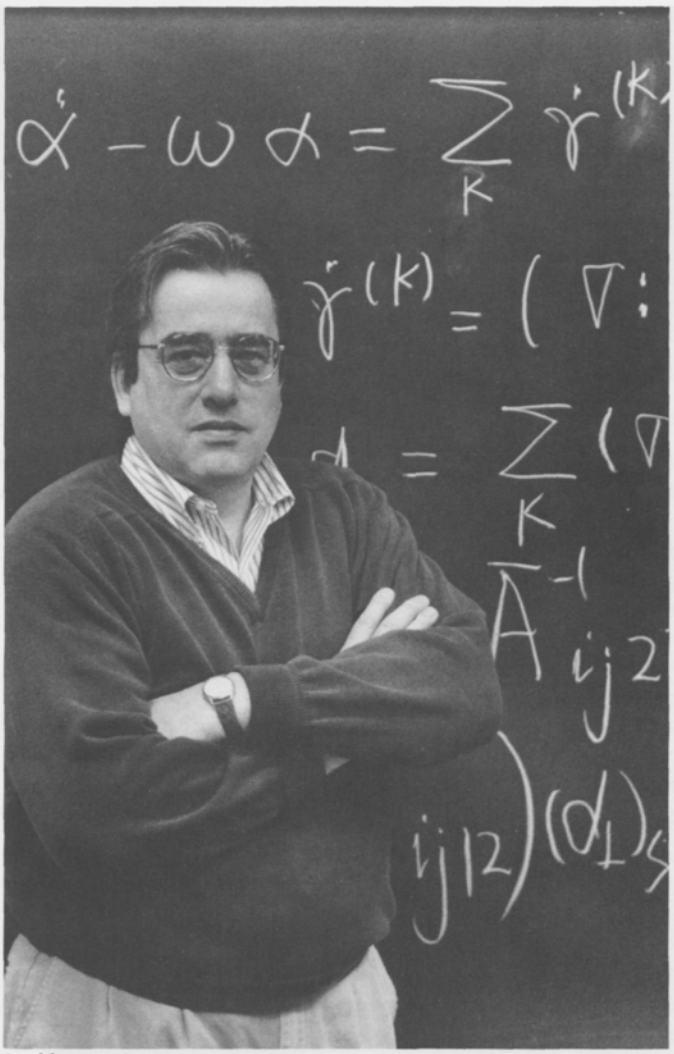
Jean Lee is a graduate student in materials science and engineering. She earned bachelor's degrees in both materials science and mathematics at the Massachusetts Institute of Technology and has worked as a summer intern at AT&T Bell Laboratories.

Lee received a fellowship from the U.S. Department of Education under the program on Grants in Areas of National Need. The grant is administered by Dr. A. Cissell of the Department of Education and by Professor Rishi Raj of the Department of Materials Science and Engineering at Cornell. Lee has also received support from the U.S. Department of Energy.

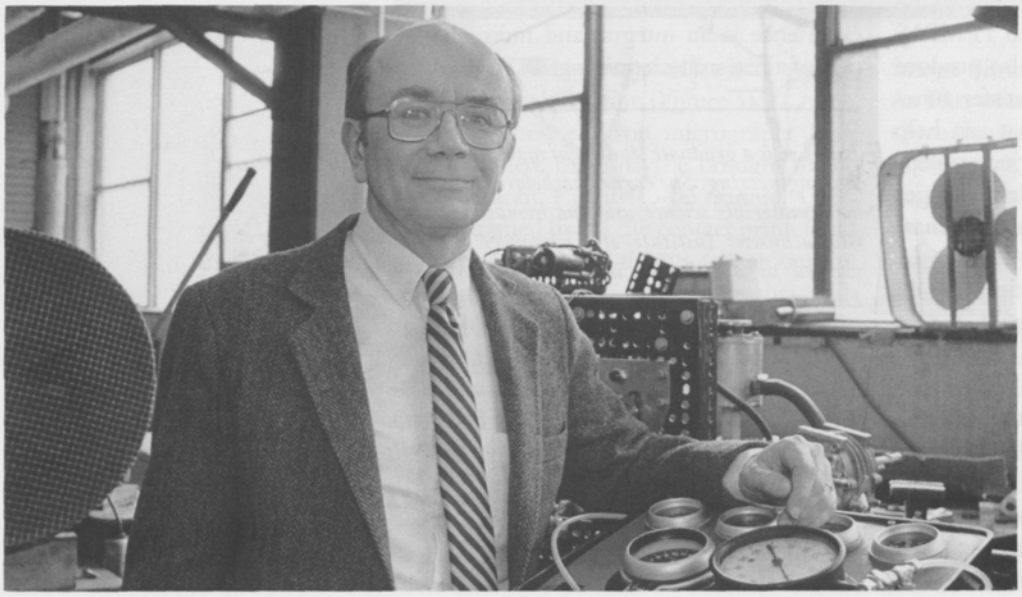
■ ***James T. Jenkins*** has been appointed to succeed Joseph A. Burns as chair of the Department of Theoretical and Applied Mechanics.

Jenkins received his Ph.D. in 1969 from The Johns Hopkins University and spent the following year as a research associate at the Solid State Physics Facility of the University of Paris at Orsay, France, developing continuum theories for structured fluids. He continued this research as a visiting lecturer in the Department of Mathematics at Strathclyde University in Glasgow before joining the Cornell faculty in 1971.

He has held visiting positions at Sandia National Laboratory, as well as at several universities: Johns Hopkins, Tokyo, Tulane, and Minnesota. He has been the recipient of distinguished fellowships from the University of Pisa, in Italy; McGill University, in Montreal; and the University of Canterbury, Christchurch, New Zealand.



Jenkins



George

■ ***Albert R. George***, professor of mechanical and aerospace engineering, was named director of the Cornell Manufacturing Engineering and Productivity Program (COMEPP). He succeeds Herbert B. Voelcker.

COMEPP facilitates and promotes cross-disciplinary research, teaching, and outreach programs in manufacturing systems, science, and engineering. It involves Cornell faculty members in many of the engineering fields and in the Johnson Graduate School of Management, as well as engineers and scientists from industry.

George, who received his Ph.D. in 1964 from Princeton University, joined the Cornell faculty in 1965, after teaching for a year at the University of Washington. During leaves, he has been a visiting senior fellow at the University of Southampton, England (1971–72), a section head at BMW (1987–88), and a senior research associate at the Aeromechanics Laboratory of the National Aeronautics and Space Administration's Ames Research Center (1988). At Cornell he has served as director of the Sibley School of Mechanical and Aerospace Engineering and was one of the founders of the Cornell Manufacturing Engineering and Productivity Program (COMEPP).

George is an associate fellow of the American Institute of Aeronautics and Astronautics and a member of several professional societies. Currently, he is serving as chairman of the Wind Noise Committee of the Society of Automotive Engineers.

■ The Department of Computer Science has three new faculty members: ***Paul Pedersen, Carlo Tomasi, and Lloyd N. Trefethen.***

Pedersen, who joined the faculty as an assistant professor, earned M.S. and Ph.D. degrees from New York University. He held a four-year full-tuition fellowship from AT&T and was awarded the NYU Dean's Dissertation Award in recognition of excellence in a dissertation. Before he began graduate study, Pedersen was a programmer for Merrill-Lynch and for Kidder-Peabody in New York.

Tomasi, a specialist in computer vision with emphasis on the interpretation of visual motion, earned the "Laurea" degree in electrical engineering at the University of Padova, Italy; an M.S. from the University of Massachusetts at Amherst; and a Ph.D. from Carnegie Mellon University. He received a Rotary Foundation Scholarship for one year of study at the University of Massachusetts at Amherst, and following his graduate work there, he received a Bottani Fellowship, awarded by the Italian Association of Electrical Engineers, for a year of research with Italtel, a major Italian telecommunications company in Milan.

Trefethen, formerly at the Massachusetts Institute of Technology, joined the faculty this fall as associate professor. His degrees are the A.B. from Harvard University, and the M.S. and Ph.D. from Stanford University. His recent honors include the NSF Presidential Young Investigator Award, 1986–91; IBM Faculty Development Award, 1986–88; and first prize in the Fox Competition in Numerical Analysis, 1985. Trefethen's research interests are numerical analysis and scientific computing, in particular the finite difference and spectral methods for partial differential equations; numerical linear algebra; numerical conformal mapping and applications; and approximation theory and applications. The author of over fifty articles and editor of *Numerical Conformal Mapping* (Elsevier, 1986), Trefethen recently completed work on a graduate textbook, *Finite Difference and Spectral Methods.*

■ ***Yu-Hwa Lo*** joined the School of Electrical Engineering as an assistant professor. He received a B.S. degree from National Taiwan University, and the M.S. and Ph.D. degrees from the University of California at Berkeley. After completing his doctoral studies, Lo worked for Bellcore as a member of the technical staff. His research interests include optoelectronic materials and devices, integrated optoelec- tronic circuits; microfabrication technology; high-speed electronic circuits; and optical interconnect and communication systems. Lo has published more than fifty journal articles and conference papers.

■ Two assistant professors were appointed to the Sibley School of Mechanical and Aerospace Engineering this fall: ***Elizabeth M. Fisher*** and ***Nicholas J. Zabaras.***

Fisher has been awarded a five-year Clare Boothe Luce Professorship (these professorships, administered by the Clare Boothe Luce Foundation, are awarded to encourage the participation of women in science and engineering). Fisher holds a B.S. degree from Yale University and M.S. and a Ph.D. degrees from the University of California at Berkeley. Before beginning graduate studies, she was an associate engineer at the Solar Energy Research Institute. After completing her graduate studies, she came to Cornell in the spring 1991 term as a postdoctoral associate. Her research interests are in hazardous-waste incineration and combustion chemistry.

Zabaras holds a diploma from the National Technical University of Athens, a M.S. degree from the University of Rochester, and a Ph.D. from Cornell University. After completing his doctorate, he taught for four years at the University of Minnesota. He received a Presidential Young Investigator Award from the National Science Foundation in 1991. His research focuses on the development of accurate and innovative mathematical techniques for realistic engineering simulation and design of manufacturing processes.

■ ***Barbara L. Cain*** was appointed an editor in the college's Office of Publications this summer. Her responsibilities include serving as an associate editor of the *Cornell Engineering Quarterly* and as a staff member of *Cornell Engineering News.*

Cain received a B.S. degree in journalism from the University of Southern Mississippi in 1974. For the next eight years she worked as a public information specialist with the Army Corps of Engineers, first at the Waterways Experiment Station in Vicksburg, Mississippi, and then in the Albuquerque, New Mexico District office. Since 1982, when she came to Cornell, she has been an editorial assistant for the *Administrative Science Quarterly* and, most recently, managing editor of the *Sociological Forum.*

■ The most recent national center to be established at Cornell is the Science and Technology Center for Computer Graphics and Scientific Visualization. It is located in the new Engineering and Theory Center Building, where the Cornell National Supercomputer Facility is also housed.

Cornell and four other universities were awarded a five-year, $14.68 million grant by the National Science Foundation to develop the center. The other universities are Brown, the California Institute of Technology, North Carolina at Chapel Hill, and Utah.

The director of the facility is Donald P. Greenberg, the Jacob Gould Schurman Professor of Computer Graphics and director of Cornell's Program of Computer Graphics.

Current research activities at the Cornell University College of Engineering are represented by the following publications and conference papers that appeared or were presented during the four-month period February through May 1991. (Earlier entries omitted from previous Quarterly *listings are included here with the year of publication in parentheses.) The names of Cornell personnel are in italics.*

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Chaimov, T. A., M. Barazangi, L. Alexander, W. Bohrer, S. Losh, L. Cathles, R. Culotta, J. Mondary, J. Boll, and *T. S. Steenhuis.* 1991. Uses of the LandMark workstation at Cornell University. Paper read at 4th Annual LandMark Graphics–University Partners Research Conference, 25–26 April 1991, in Houston, TX.

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Liu, Y., J. S. Selker, T. S. Steenhuis, and *J.-Y. Parlange.* 1991. Role of hysteresis in fingered flow in homogeneous sandy soils. In *American Geophysical Union Spring Meeting 1991 abstract volume,* p. 120. Washington, DC: AGU.

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Selker, J. S., and *T. S. Steenhuis.* 1991. Large-scale laboratory studies of transport through unsaturated soils with sloping interfaces. In *American Geophysical Union Spring Meeting 1991 abstract volume,* p. 130. Washington, DC: AGU.

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Bilderback, D. 1991. X-ray optics for protein crystallography. Paper read at Center for Macromolecular Crystallography workshop, 17–20 May 1991, in Gulf Shores State Park, AL.

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A NOTE TO OUR READERS

This is the first issue of the new, redesigned *Quarterly*. Having celebrated our first twenty-five years of publication with a retrospective silver-anniversary issue, we are beginning our second quarter century with a new look. The old 9"x9" design won many awards in competitions sponsored by the Council for Advancement and Support of Education, and a few other publications flattered us by imitation. But design is sensitive to fashion, and what was new and refreshing in 1966 has begun, in recent years, to look a bit dated.

The new design was created by Deena Wickstrom of Cornell's Office of Publications Services. We asked her to develop something contemporary, appropriate to its subject, easy to work with, and inexpensive to produce. We are pleased with the result, and hope that readers will be, too.

We are also making a slight change in the name of the magazine. *Engineering: Cornell Quarterly* has always looked good on the cover, but it sounds backward when read out loud. In changing the name to *Cornell Engineering Quarterly*, we risk increased confusion with another publication, *The Cornell Engineer*. But there is no dishonor in occasionally being confused with the nation's oldest student-run engineering magazine, and we are willing to tolerate this inconvenience for the sake of having the magazine's name become what most people think it already is.

We have prepared an index to the subject matter in the first twenty-five volumes. This index makes accessible, through listings by both author and subject, a twenty-five-year record of Cornell's College of Engineering—its research, its educational programs, and the professional lives of its faculty members. We are sending a copy to all the libraries on our mailing list, and other subscribers may have copies upon request.—DP

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