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Program urges undergrads to try research. Cornell Chronicle 026_06 p1&4 September 29, 1994

Symposium to honor Professor Arthur Ruoff’s 50 years at Cornell. Cornell Chronicle 038_06 p6 September 14, 2006
Metallic Hydrogen: Engineers Racing Russians

High pressure engineers at Cornell University could be engaged in an unpublicized race with Russian scientists to create metallic hydrogen — a substance that could revolutionize rocketry and make possible a perfect conductor of electricity at room temperature.

Using a 1000-ton press in a laboratory in Thurston Hall, the engineers already have created pressures up to 700,000 atmospheres — approximately — one fifth the pressure at the center of the earth and 700 times the pressure at the deepest point in the ocean. One atmosphere is 14.7 pounds per square inch.

Despite these tremendous pressures, the Cornell team, headed by Arthur L. Ruoff, professor of materials science and engineering, is pushing to reach pressures of at least 1,000,000 atmospheres.

Ruoff doesn’t like to think of his group as being in a race with the Russians. However, in 1967 a Russian acquaintance sent Ruoff a news item from Pravda, the Russian newspaper, stating that a huge multi-story press was being constructed in the Soviet Union with the express purpose of reaching pressures up to 2,000,000 atmospheres. The Russian scientist suggested that one application of the new press might be the production of metallic hydrogen.

“It’s clear the Russians are very much interested in metallic hydrogen and that they have some pretty good hardware to make it with,” Ruoff said. “It would be nice if we could get there first.”

Cornell physicist Neil W. Ashcroft, professor of atomic and solid state physics, in 1968, at the same time as the Russian physicist Abrikosov, first suggested that metallic hydrogen might be a room temperature superconductor.

Although the engineering problems and costs involved in its production are enormous, the rewards that could come with the creation of metallic hydrogen are almost incalculable.

Since metallic hydrogen would be a superconductor of electricity at room temperatures, it might be used for power lines. Present day electric power lines are inefficient conductors. Superconducting power lines can be made now, but they might be too costly to be practical since they would have to be kept at extremely low temperatures — lower than 20 degrees above absolute zero.

Magnets for industry or research use are now generally made of copper and generate tremendous heat, which means that energy is lost. Although magnets can now be made of superconducting materials, they must be kept at very low temperatures. Metallic hydrogen might make possible magnets that would have almost no energy losses and that could be operated without the need for creating low temperatures.

Tremendous advances in rocketry also could follow the creation of metallic hydrogen. Because rockets now use liquid hydrogen as fuel, they must be made like giant thermos bottles to assure low temperatures. If metallic hy-
Hydrogen were used, rockets could be made smaller because low temperatures would not be necessary and because hydrogen in this form is one tenth as dense as liquid hydrogen.

Since it is necessary to start their research using solid hydrogen, the researchers must be concerned with the low temperatures involved in solid hydrogen as well as the high pressure needed to obtain metallic hydrogen.

Using a sample of solid hydrogen weighing only one ten thousandths of an ounce, tremendous pressure is applied to tapered pistons in a pressure vessel into which the solid hydrogen is placed. The pistons are tapered to take advantage of what is called Pascal’s Law. The law states that modest pressures applied to a large area are intensified in a smaller area.

Theoretical physicists are not sure exactly what pressure is needed to cause the transformation. Current estimates are 800,000 atmospheres to 2,600,000 atmospheres. One of the problems facing the Cornell engineers is to obtain data which will pin down this transformation pressure more precisely. The other problem is to generate the higher pressure needed to create metallic hydrogen.

When the engineers finally create metallic hydrogen, they know it will be a material stiff as steel but only one eighth as dense.

Working with Ruoff on the project are Ashcroft; Geoffrey V. Chester, director of the Laboratory of Atomic and Solid State Physics, and James A. Krumhansl. professor of physics. Funds for their research are provided by the Advanced Research Projects Agency and the Atomic Energy Commission.

*from Cornell Chronicle 001_21 p1&4 March 12, 1970*
Pushbutton Learning: Device Aids Students

A casual visitor to the Physics 234 class in Rockefeller Hall might think he’d stumbled onto a television quiz show complete with pushbuttons and electronic gadgetry.

What the visitor really has come upon is an experimental class that features a series of rapid fire electronic quizzes enabling the professor to keep a finger on the learning pulse of a class at all times.

The teaching method, called a Student Response System, was designed and installed by Raphael M. Littauer, who teaches the class in quantum mechanics.

The concept is not a new one, in fact commercial systems of this type are available. A pilot system of a similar nature was constructed last year by Arthur L. Ruoff, professor of materials science. Ruoff, who is active in seeking better teaching methods at Cornell, built his system in Bard Hall where it was used by himself and other professors. But until this semester, when Littauer has his system ready for his 200-member class, no permanently installed, large-scale system had been in use at Cornell.

Basically, the system includes a group of five pushbuttons placed at each student’s seat and a display unit that tells the professor at the front of the class at a glance, how many students have pushed any given button. During the lecture, Littauer asks the students to respond to multiple choice questions he flashes on a large screen. Each possible answer has a number and the student chooses by pressing the appropriately numbered button. If a vast majority of the students have gotten the point, Littauer pushes ahead with his classwork. If, on the other hand, the responses show that many students are not clear on a given topic, Littauer can go back and review that particular aspect.

Littauer sees two principal benefits for the system. The professor finds out quickly and painlessly whether or not he is communicating with the class. The system also helps keep the students alert since they are required to participate in the quizzes, which may number as many as a dozen in the hour-long class.

One possible disadvantage Littauer sees is that he can’t tell which students have given the correct answer and which have missed the point. The system has been designed so that this limitation can be remedied and enable the professor to know exactly how each student has answered. The wiring is there to do this, but Littauer is reluctant to hook it up because he wants to avoid a “big brother is watching you” atmosphere in the classroom. Anonymity, Littauer feels, will encourage a student to give an answer, even if he’s not sure of a topic.

So far, Littauer is cautiously optimistic about the future of the teaching system.

“It may prove to be a dud,” he said, “but at the moment it looks very promising. The kids enjoy it now. It’s like a new toy. But I may yet have to make some revisions at the end of the year.” Littauer said he got the idea for installing the system during final examinations in the spring of 1970.

“It just struck me,” he said, “that students retain no more than a fraction of the material they’re presented, and that’s pretty inefficient. If you know this, it’s silly to keep using the same method of teaching. Some change, it appeared to me, was needed.”
Littauer learned that the system could be purchased — but the price tag was $40,000 and the money was not available. So he designed and made his own after he applied for and got a $2,000 grant from Cornell’s Center for Research in Education. He also got a little financial help from the Department of Mathematics and from the College of Arts and Sciences. Labor was donated by the Department of Physics for actual installation of the various pushbutton units and other electronics parts necessary to make the system operate.

So far, there have been no hitches either in installation of Littauer’s home-made system or in the teaching results. But Littauer is reserving comment on whether the system is a success or failure. Littauer plans to write a report on the design of the system and the educational results obtained for the Center for Research in Education at the end of the Semester.

_from Cornell Chronicle 002_21 p4  February 18, 1971_
A Cornell engineer has tailored a course which not only permits students to set their own learning pace but which also removes a perennial headache of many students — that of scheduling a class that conflicts with others.

The course, developed last year by Arthur L. Ruoff, professor of materials science and engineering, has been refined this semester to allow tests to be taken any time instead of on a fixed day.

Besides its built-in learning flexibility, the course is adapted to permit students to take a general approach to materials science or to concentrate on either the mechanical properties or the electrical properties of materials. In effect, three courses are taught simultaneously.

The 35 students taking the course, titled “Introduction to Materials Science,” do their work in a Learning Center in Room 303 Thurston Hall. The center includes a room where a tutor has a desk, a movie room where films may be viewed privately and heard through earphones so as to cause no disruption, and 11 carrels.

The carrels themselves are sophisticated learning mini-centers with earphones and slide projectors with rear view projection. They also have electric power outlets so that microscopes can be used and simple experiments conducted.

Ruoff has one general meeting with students at the beginning of the term at a time when no conflict is possible with other classes. He tells students how the course will be conducted in the Learning Center, gives them assignment sheets and asks that they keep a notebook to list two types of questions that may arise.

The two types of questions encouraged are the “I don’t understand” questions and the “why” questions. Answers to the former can be gleaned from tapes which are part of the course or from the tutor; the latter, which require deep individual thought, can be discussed with Ruoff or the tutor. Ruoff places great stress on the “why” type question and frequently prods students to come up with queries.

“The primary and secondary school kids have had the desire to ask questions knocked out of them,” Ruoff said. “I want to get it back for them.”

Since the course has done away with ordinary lectures, Ruoff tells the students they’ll see him again when he’s on duty in the Learning Center. Elimination of the scheduled-time lecture in favor of taped lectures enables students to do their learning anytime between 1:30 and 5:30 p.m. from Monday through Friday or between 7:30 and 10:30 p.m. Monday through Thursday. It’s this flexibility in study times that enables students to schedule other classes that they might not normally be able to schedule.

The course is organized so that a chapter in the text used is related to a taped lecture and the accompanying color slides. Students may read the text first and then listen to the taped lecture or reverse the process.
Records are kept on how much time a student spends in the Learning Center. This, however, is used to evaluate the course rather than the student and has nothing to do with grading. “We just want to see if there is a correlation between the time a student spends in the center and his grade,” Ruoff said.

The course includes a mid-term and final examination. But before a student may take either, or before he may take a quiz, he has to show the tutor or Ruoff a list of his “why” questions. It’s a case of no question, no quiz. Moreover, performance at a mastery level of 80 percent is required on each quiz.

With the choice of study hours, the students may take as little or as much time during the semester to finish their work. Some expect to finish this semester’s work by the recess break on March 18.

Ruoff not only designed the course and its content, he had a hand in making the specialized carrels used and the instrumentation in them.

To those who suggest that use of tapes and films has depersonalized the course, Ruoff points out that the contrary is true.

“All the lectures are prepared in advance so that gives me a lot of time to spend in the Learning Center with students,” Ruoff said. “All they have to do is come to the desk and see me. Then we really have one-to-one or personal contact.”

The Learning Center was designed with expansion in mind. Along with the existing 11 carrels, there is space for nine more if needed. Other professors in other courses have begun to use the Learning Center facilities to supplement their courses.

*from Cornell Chronicle 003_24 p16-6 March 9, 1972*
Scientists Seek to Produce Hydrogen in Metallic State

A materials science experimentalist and a theoretical physicist are working together at Cornell to turn hydrogen, one of the elements in drinking water, into a metal which might revolutionize chemical rocketry, provide convenient fuel for controlled nuclear fusion, and make possible a perfect conductor of electricity at room temperature.

With a $60,000 contract from the National Aeronautics and Space Administration (NASA), Arthur L. Ruoff, professor of materials science and engineering, and Neil W. Ashcroft, associate professor of physics, are collaborating in a joint experimental and theoretical study to produce and understand metallic hydrogen.

Normally, hydrogen is a gas, but at very low temperatures (about -400 degrees Fahrenheit) it becomes a liquid and, at even lower temperatures, an insulating solid. Theorists predict that squeezing this solid with tremendous pressures will convert it into a metal.

“Lots of things which aren’t metals under ordinary circumstances become metals under great pressures,” Ruoff explained. He cited iodine as one such element which changes from a reddish-brown solid to a shiny metal.

Hydrogen is the lightest of all elements, the simplest in structure and the most abundant material in the solar system. Astronomers have calculated that some 40 percent of the hydrogen in the planets exists in the metallic state, most of it in the giant planets, Jupiter and Saturn.

Metallic hydrogen is 10 times as dense as molecular hydrogen. Pressures inside the giant planets must be high enough to drive the molecule into the metallic form of the element. Just what these pressures are has not been exactly determined. Ashcroft and Ruoff estimate that the pressures needed will be in excess of 15 million pounds per square inch, or more than a thousand times the pressure at the bottom of the deepest ocean.

“If we can keep it long enough at room temperature to prove that it exists even temporarily under these conditions,” Ruoff said, “that’ll be long enough for me. If pressed, we could make several scientific tests in a matter of milliseconds. Given half an hour, we can conduct numerous other studies on the product.”

Ruoff will first try to conduct electricity through the product. Molecular hydrogen is an insulator (does not conduct electricity). Metallic hydrogen would be a conductor, as all metals are, and may even prove to be a superconductor. This would mean that wires of metallic hydrogen would transport electric power with no waste.

Other tests will measure the density of the product, observe its behavior in a magnetic field and try to determine what, if any, crystal structure it has.

*from Cornell Chronicle 004_24 p8 April 12, 1973*
$7.5 Million Ceramics Program Started Here

Ceramics today are most commonly found in bricks, bathroom fixtures, and fine dinnerware. The ceramics of the future, though, will be widely used in engines, electronic components, and maybe nuclear reactors.

Before ceramics can replace metals in a wide range of products, however, researchers must discover how to reduce their brittleness in order to utilize their hardness and resistance to extreme heat.

Toward that end, Cornell has created a $7.5 million high-technology ceramics research program in cooperation with industry and possibly the federal and state governments.

Three corporations, including a leading computer company and a major ceramics firm, have been asked to commit at least $1.5 million each over five years. They have already indicated their intent to provide substantial funding. Additional corporations may participate: $600,000 per year is being sought from the National Science Foundation, and discussions have been held with the New York State Science and Technology Foundation, says Arthur L. Ruoff, director of the program and Director of the Department of Materials Science and Engineering at Cornell.

“The ceramics industry is going through a revolution,” Ruoff says. “But, there is a severe shortage of scientists and engineers trained in high-technology ceramics. Without an immediate and substantial effort, the United States could lose much of the future ceramics market to Japan.”

Cornell’s program will begin this fall and will eventually train five postdoctoral researchers and award seven Ph.D.’s in ceramics each year, placing Cornell among the top half-dozen American universities conducting ceramics research, Ruoff says.

Unlike traditional ceramics, which are made from clay, sand, and other materials, modern ceramics are produced from materials such as silicon, carbon, aluminum, and nitrogen. Those raw materials are much more abundant in nature than many of the metals currently used in manufacturing.

In addition, high-tech ceramics are harder than metals, have much higher melting points, and, depending on the particular ceramic, can serve as electrical insulators or conductors. They also may be magnetic.

Ceramics may be used to make pumps, bearings, turbine blades, or other parts that could allow automotive and jet engines to operate at a much higher temperature than is now possible, increasing engine efficiency by as much as 50 percent.

In the electronics industry, improved ceramic packaging of computer chips “could be as significant as the development of the chip itself,” Ruoff says. Ceramic packaging serves as both an electrical insulator for the numerous leads needed to distribute electrical signals, and as a conductor of heat, helping to cool increasingly powerful computer components.

But, as anyone who has dropped a piece of china realizes, the strength of ceramics is no match for their brittleness when they are hit with a sudden force.

Researchers are hoping to solve the brittleness problem by studying the molecular bonding in ceramic materials, and by improving the process used to manufacture ceramics, Ruoff explains.

Discovering a more cost-effective way to mold ceramics into complex shapes is also needed to give ceramics wider industrial uses, Ruoff adds.

Cornell’s ceramics program is a collaborative venture of the Materials Science Center at Cornell, which is sponsored by the National Science Foundation, and the Department of Materials Science and Engineering. At maturity, the program may involve 10 or more Cornell scientists and their research groups, two or three participating scientists from each collaborating industry, and a support staff.
The Materials Science and Engineering department at Cornell was the first to integrate the study and teaching of ceramics, metals, polymers, and semi-conductor materials, Ruoff says. Funding for materials research at Cornell is approximately $20 million per year.

The new program will draw on the previous research of three ceramic scientists at Cornell, including Rishi Raj, associate director of the new ceramics program and a professor of materials science and engineering. A visiting professorship will be established to bring one researcher from elsewhere to Cornell each year to participate in the program.

Scientists in the program will conduct basic research, rather than research aimed at a specific commercial problem. All patents will be held by Cornell.

*from Cornell Chronicle 016_39 p1 June 20, 1985*
New Major Program Here Now Combines Electrical Engineering, Materials Science

A new course of study combining the high-technology fields of electrical engineering and materials science has been established here.

The first of its kind in the nation, the program will expose undergraduate students to the latest research discoveries related to the fabrication and packaging of electronic devices, according to Arthur L. Ruoff, director of the materials science and engineering department.

“The joining of these two fields is essential if the United States hopes to regain its leading position in the semiconductor field,” Ruoff said. “I expect that some other leading research and engineering universities will follow our lead into this new undergraduate major.”

The new major, called “electronic materials,” is based in Cornell’s College of Engineering. Five students who completed the course requirements for the new major before the program was formally adopted graduated with double-major bachelor of science degrees in electrical engineering and in materials science in June 1985. About one dozen students are expected to graduate with the double-major degree next spring.

Cornell has a reputation as a national leader in electrical engineering and materials science teaching and research. Cornell’s academic departments in the two disciplines are both ranked in the top five nationally, and the university operates the Materials Science Center and the National Research and Resource Facility for Submicron Structures.

Research executives at firms such as General Electric, AT&T, IBM, and Xerox, in letters to Ruoff, have praised the new program for its comprehensive approach to the electronics and packaging obstacles confronting scientists and engineers striving to improve semiconductor performance.

“I cannot over emphasize how important it is to have students well-grounded in both materials science and devices,” wrote Venkatesh Narayanamurti, director of the Solid State Electronics Research Laboratory at AT&T Bell Laboratories. “The idea of a dual major is an excellent one.”

“I am happy to see a program which teaches students in depth both about electronic materials and electronic devices,” wrote John A. Armstrong, research division vice president of logic and memory at IBM. “These topics are of great interest to the computer industry and having both in one curriculum is doubly interesting.”

Integrated circuits are small, delicate semiconductor chips containing a half-million or more transistors that communicate with each other through electrical interconnections. These interconnections consist of thin metal lines with a width 100 times smaller than the diameter of a human hair. The interconnections rest on a layer of oxides that, in the most advanced chips, are one thousand times thinner than a human hair.

Typically, electrical engineers have been concerned with improvements in circuit design, processing methods, and applications of electromagnetic theory relative to semiconductors. Materials science engineers, on the other hand, have focused on the properties of materials, the growth of crystals, and the analysis of ceramics, polymers, and other materials with potential integrated-circuit applications.

With the growing demand for more powerful, efficient, and smaller chips, a new type of scientific specialist who has a knowledge of high-tech materials and electronics is needed. Improvements in electrical interconnection techniques and the use of new materials will both be needed for the integrated circuit of the future, Ruoff said.

Yet, “An electrical engineer whose curriculum stresses circuit and transistor design theory receives surprisingly little instruction on the processes and materials used to form an integrated circuit,” he said. “The curriculum in the new program covers basic electrical design and solid state theory.”
“Just as we need increased university, industry, and government cooperation in scientific and engineering research to improve the quality of our manufactured products and the efficiency of their production, we also need new and imaginative educational programs to meet the growing electronic and computing needs of the information society,” Ruoff added.

No new courses were created for the program, but students participating in the new dual-major must study a common core curriculum that emphasizes mathematics, chemistry, physics, computing, engineering distribution, materials science, electrical sciences, and probability and statistics. The course can be completed within the typical eight semesters of full-time study.

“It has always been possible to major in one field and take several electives from the other,” Ruoff said. “This new program, though, allows students to graduate as fully qualified electrical engineers and as materials science engineers.”

One of the program’s first graduates, Elaine Lui, conducted research in electron beam lithography at AT&T Bell Laboratories. She is conducting graduate study at Princeton University in electronic materials and devices.

*from Cornell Chronicle 017_01 p4 September 5, 1985*
Cornell’s Diamond Anvil Puts Big Squeeze on Tiny Materials

The spike heel of a 90-pound woman’s shoe exerts more pressure — per square inch — than the sneaker of a 300-pound wrestler.

Cornell scientists are using that principle, together with a special diamond “anvil,” to squeeze semiconductor and insulating materials into crystalline forms never seen before.

In search of new arrangements of crystals, a team led by Yogesh K. Vohra and Arthur L. Ruoff has compressed the semiconductor material germanium to less than half its original volume with an atom-crushing pressure of 1.25 megabars (or 1.25 million atmospheres). That is the highest pressure at which crystalline structure change in any material has been directly observed by X-ray diffraction.

While there is not much market for crushed germanium, the Cornell researchers say their experiments are useful in predicting high-pressure behavior for all kinds of materials.

The record-breaking pressure and the crystal structure determination of germanium will be reported in the May issue of the journal Physical Review Letters. In the March 7 issue of the journal Science, the Cornell researchers reported crystal phase changes when cesium iodide and rubidium iodide were subjected to pressures of 950,000 atmospheres and 890,000 atmospheres respectively.

Such pressures exist naturally only deep within the Earth and on other planets. For comparison, the pressure in a municipal water system is three to five atmospheres and in the deepest parts of the oceans is about 1,000 atmospheres. Pressure at the center of the earth is estimated at 3.5 million atmospheres.

Extremely high pressures such as those achieved at Cornell are possible by concentrating the force in a small area, between the flat faces of two diamonds. The device known as a diamond anvil operates like a miniature workbench vice and resembles a piston and cylinder of an automobile engine. Pressure is exerted as the two opposing sides are screwed together.

The tips of gem-quality 1/3-carat diamonds are ground to flat surfaces 300 microns across. (A micron equals one millionth of a meter, or about 1/70 the diameter of a human hair.) Between the faces of the diamonds the researchers insert a thin strip of spring steel, drilled to form a gasket with a hole 25 to 50 microns across. Minute amounts of samples to be squeezed are placed in the gasket.

As the diamond anvil is tightened, an intense, focused X-ray beam shines through the diamond (which is relatively transparent to high-energy X-rays) and into the crystalline sample. The X-rays are diffracted (or scattered) by the electrons in the materials, producing distinctive patterns that reveal crystal structure. Materials scientists use X-rays because, unlike visible light or other forms of radiation, their wavelengths are approximately equal to the distance between atoms.

The X-rays come from CESR (the Cornell Electron Storage Ring), where electrons and positrons from a synchrotron are accelerated nearly to the speed of light around a half-mile long, underground beam pipe. Synchrotron radiation in the form of X-rays is a “waste product” from elementary particle physics experiments at CESR. The
extremely bright X-rays are diverged from the storage ring, then beamed into the laboratories of CHESS (the Cornell High Energy Synchrotron Source), and through materials placed in their path.

A technique called energy-dispersive X-ray diffraction lets the materials scientists follow both the changes in internal structure — known as crystallographic phase transition — and the volume of substances as they are squeezed tighter and tighter.

Compressing cesium iodide with 950,000 atmospheres of force reduced its volume to 46 percent; as pressure increased, the cesium iodide atoms were moved from a cubic arrangement, to a tetragonal arrangement (resembling a shoe box with square ends) to an orthorhombic phase (like a standard shoe box). The Cornell materials scientists found that germanium changed from tetragonal crystals to hexagonal (resembling a six-sided soda can) to a form called double hexagonal close-packed structure as pressure approached 1.25 megabars and the volume decreased to 45 percent.

The “shrinking” of seemingly solid crystalline materials is possible because the outer shells of electrons in the atoms are crowded closer to the nucleus under tremendous pressure. Some materials, such as cesium iodide, recover their original crystalline structure and volume when released from the diamond anvil. Others, including silicon, never fully recover, or take different forms.

Under pressure, some materials take on different properties. Germanium, for instance, is a semiconductor at room pressure; at 110,000 atmospheres, germanium transforms into a metal that also is a superconductor at extremely low temperatures.

Before the Cornell high-pressure experiments with germanium in early 1986, crystallographic phase changes had never been documented at megabar pressures — those greater than a million atmospheres. The only indication materials scientists had of megabar phase changes were subtle shifts in color. The double hexagonal close-packed structure had been seen in rare earth elements only.

The diamond anvil technique shows little immediate likelihood of becoming an important industrial process, according to Ruoff. “It may be possible — one day — that giant diamond anvils will exist for industrial production of specialty materials,” he says.

In the meantime, tests at Cornell and about a half dozen other ultrahigh-pressure laboratories around the world are helping materials scientists learn to predict the behavior — under stress — of all kinds of substances.

from Cornell Chronicle 017_32 p3 May 1, 1986
Packaging microchips challenges engineers

Cornell’s materials scientists and electrical and mechanical engineers are collaborating in an industry-funded attempt to overcome a serious bottleneck facing microelectronics: the packaging of integrated circuits.

Maintaining a constant operating temperature for microelectronic equipment and ensuring the reliability of microchips in climates ranging from the equator to the polar caps is one goal of Cornell’s Electronics Packaging Sciences Research Program.

“Electronic system performance improvement will result more from packaging technology development than from integrated circuit technology during the next 10 years,” says D. Howard Phillips of the Semiconductor Research Corp.

SRC, the consortium of U.S. firms in the electronics, computer, and communications businesses initiated the packaging sciences program with a first-year contract for $250,000 and plans to continue this research as a multi-year program.

Support for the packaging sciences program also is coming from individual industries, including IBM and General Electric Co., both SRC members.

Electronic packaging is the means of protecting the wafer-thin integrated circuit chips and connecting them to the rest of a system. Included in packaging are the substrates — usually ceramic or plastic materials on which chips are mounted — as well as the dozens or hundreds of fine wires or solder joints carrying signals to and from the chips, and tiny metal couplings connecting the package to larger circuit boards.

While great progress has been made in increasing the speed and capacity of integrated circuits, packaging technology has lagged.

“We are now at a turning point in electronics technology; the chip has out-paced the package,” says Che-Yu Li, professor of materials science and engineering, and one of the two principal investigators in the new program.

“Improvements in electronic packaging should enhance U.S. competitiveness,” says Arthur L. Ruoff, the other principal investigator and a professor and director of Cornell’s Department of Materials Science and Engineering.

The Japanese already dominate the market for ceramic packages for low-end uses. They have long emphasized manufacturing technology and miniaturization,” Ruoff says.

“Establishment of this program represents a continued effort at Cornell to contribute to areas of technology that are vital to U.S. industries,” says Joseph M. Ballantyne, Cornell’s vice president for research and advanced studies and a professor of electrical engineering. “The SRC-supported research is a direct response to the needs of the semiconductor and information industry.”

The packaging program will draw on the resources of several national centers at Cornell: the National Research and Resource Facility for Submicron Structures, the national supercomputer center, and the X-ray diffraction laboratory at the Cornell High Energy Synchrotron Source.

Related activities include the recent establishment of a ceramics initiative program and the expansion of the polymers program in the Department of Materials Science and Engineering. Both programs have the support of industry for new faculty members and research, and are expected to have a direct impact on electronic packaging research.

Five universities, out of nine that originally sought the designation, were invited by the Semiconductor Research Corp. to prepare formal proposals for the packaging program. SRC chose Cornell and Lehigh University.

from Cornell Chronicle 018_01 p8 August 28, 1986
Grant supports ceramics studies

The belief that engine turbines someday will be made from ceramics instead of metal has prompted an Ithaca-based company to provide the university $50,000 for ceramics research.

“The Japanese are running way ahead of this country in ceramics development,” said Robert R. Sprole Sr., chairman of Therm Inc. “We have to develop ceramic materials and make them stronger to remain competitive.”

Therm Inc. machines turbine blades for all types of turbomachinery, including applications for use in ships and airplanes. Therm’s clients include General Electric Co., Rolls Royce, Pratt and Whitney, and Dresser Industries, Sprole said. The company has created its own ceramic division to fabricate silicon nitride ceramic components.

“We think ceramics are the material of the future. We can’t afford to allow the United States to be left behind,” Sprole said.

Today’s turbine blades are made from metal alloys. Ceramic blades can make engines more efficient by allowing them to operate at temperatures that would melt current metal components, according to Arthur L. Ruoff, director of materials science and engineering.

Cornell researchers already are studying how ceramics can be used in engines, as packaging for integrated circuits and in other ways, Ruoff said. Cornell’s ceramic research program is supported by the National Science Foundation and grants from Corning Glass Works and International Business Machines Corp.

“The grant from Therm will support ongoing studies, and will fund renovations to provide space for a new professor,” Ruoff said. “There is a severe shortage of scientists and engineers trained in high technology ceramics. This award will help us attract another expert in the field.”

High-technology ceramics are made from materials such as silicon, carbon, aluminum and nitrogen, which are more abundant and harder than natural metals and have a higher melting point. They must be machined by using diamonds and ultrasound techniques. Metal is not strong enough to cut modern ceramics, Ruoff said.

The key disadvantage of ceramics is their brittleness. Cornell researchers are studying the molecular bonding of ceramics to find ways to make them more durable, Ruoff said.

Eventually, it may be possible to manufacture a complete ceramic jet turbine rotor in one mold pressing, Ruoff said. That would save time, energy and money compared to the current machine manufacturing and assembly of metal turbines, he said.

Sprole’s grandsons, Cornell graduates William and Robert West, are helping guide Therm’s ceramic work, Sprole said. Robert is manager of Therm’s ceramic fabrication division and William is working as a research engineer while completing graduate study in materials science. Sprole is a member of the Cornell Class of 1935.

— Mark Eyerly

from Cornell Chronicle 018_15 p8 December 11, 1986
Oxygen may become metallic at high pressure: researchers

Using diamonds, rubies, gold dust and the world’s most powerful X-ray source, Cornell researchers have made significant advances in exploring the effects of super-high pressures on matter.

In a series of papers to be delivered March 19 and 20 at a meeting of the American Physical Society in New York, they will announce:

- The first evidence that oxygen may become a solid metal at high pressures.
- The discovery of a new form of silicon that appears at pressures over 780,000 times atmospheric pressure.
- The discovery of three new forms of germanium, two of which appear above a million atmospheres, the first time new forms of any material have been discovered at such pressures.
- The highest pressures ever achieved with a vicelike “diamond anvil” that uses synthetic diamonds rather than natural ones, an achievement that could open the way for cheaper, stronger apparatuses for achieving high pressures.

The advances were reported by a research team led by Professor Arthur L. Ruoff, director of the Department of Materials Science and Engineering. Members included Serge Desgreniers, Yogesh K. Vohra, Keith E. Brister, Steven J. Duclos and Samuel T. Weir.

Ultrapressure research has enabled scientists to produce and detect numerous new forms of solids, said Ruoff, and these findings have contributed to better theories of chemical bonds and the behavior of electrons in solids. He also said that the study of the earth’s depths and of the structure of other planets had benefited from such high-pressure studies.

The diamond anvil that scientists use to achieve high pressures typically consists of a pair of brilliant-cut diamonds, each with a tiny flat area polished off its tip. Mounted on this flat area — which is about the diameter of a human hair (100 micrometers, or millionths of a meter) — is a tiny steel gasket with a hole about one-quarter the diameter of a human hair drilled in the middle.

The substance to be tested is added to the hole, and the diamonds are mounted on a powerful vice, tip-to-tip with the gasket between them. When the diamonds are clamped together using a system of screws, scientists can achieve pressures well over one megabar, the equivalent of 980,000 atmospheres.

To measure the immense pressures in diamond anvil cells, scientists usually include in the sample chamber a substance that changes in a known way as pressure increases. For example, they may add an infinitesimal chip of ruby, which fluoresces at a wavelength that changes with the pressure. Or, they may use a small sample of gold powder, which reduces in volume at a calibrated rate as it is squeezed.

In one paper, Desgreniers, Vohra and Ruoff report the first studies showing that oxygen may become a metal at one megabar.

In their experiments, the researchers subjected oxygen samples to pressures up to 1.3 megabars using their diamond anvil apparatus. As had previous researchers, they saw the sample change from yellow to red to opaque as the pressure increased. Then, at 1 megabar, the oxygen sample became reflective to infrared light, taking on the shine characteristic of metal.

“We still have to test the electrical conductivity of such a sample, to make sure that it is a metal,” said Vohra. “But this discovery is nevertheless striking, because it shows that oxygen is actually a very exotic material at high pressures.”

1 Corrected title: 018_26 April 2, 1987, p2
The discovery of the new form of silicon by Duclos, Vohra and Ruoff represents the latest in a series of changes silicon is known to undergo at high pressures. These changes in structure occur because the atoms in the silicon crystal shift and rearrange themselves in different ways as they are squeezed closer and closer together. Researchers already had discovered that silicon changes to such forms as “diamond cubic,” “beta-tin,” “primitive hexagonal” and “hexagonal closest packed” with successive pressure increases. The Cornell scientists found that the transformation to the new structure, called “face centered cubic,” occurs at about .78 megabars.

They detected the new structure by subjecting the diamond anvil to the intense beam of X-rays generated as a byproduct of Cornell’s high-energy physics particle accelerator, the Cornell Electron Storage Ring, operating at more than 5 billion electron volts. This “synchotron radiation” — the most powerful beam of X-rays available — is created by the bending of the particle beam as it speeds around the storage ring. The X-ray beam is diverted into the laboratories of the Cornell High Energy Synchotron Source (CHESS), where scientists use it to deduce the structure of a substance by studying how it diffracts the X-rays.

“Significantly, this is the lowest-atomic-number, or lightest, element that has ever had a structural determination made above 1 megabar,” Ruoff pointed out.

Ruoff also reported new forms of germanium that occurred at .75 megabars, 1.02 megabars, and 1.25 megabars. The first two represent transitions to “primitive hexagonal” and “double hexagonal closest packed,” said Ruoff, but the scientists have still not determined what structure germanium transforms to at 1.25 megabars. The transition at 1.02 megabars represents the first time any material has been found to change structure above 1 megabar. So far, said Ruoff, the scientists have studied germanium up to 1.6 megabars.

The Cornell researchers also reported the highest pressures yet achieved, up to 1.25 megabars, using synthetic diamonds. Scientists doing high-pressure studies now must sift through thousands of natural diamonds to find two perfect enough for use in diamond anvils. And even these stones may have tiny flaws, or dislocations, that may cause them to crack or shatter under pressure.

— Dennis Meredith

from Cornell Chronicle 018_26 p1&8 March 19, 1987
Rutherford ‘atomic machine gun’ installed in Bard Hall

Although its official name is the Rutherford backscattering system, perhaps “atomic machine gun” might be the best way to describe the large, tan T-shaped arrangement of cylinders now installed in Bard Hall. An accelerator capable of spewing multimegavolt streams of charged ions at a small target, the latest addition to the materials sciences department, has literally extended the vision of Cornell material scientists.

Purchased under a $980,000 contract with IBM to Professors Jim Mayer and Ed Kramer and dedicated in February, the new accelerator is allowing researchers, both from Cornell and from industry, to probe more deeply into solids to understand their structure. It is also allowing them to implant ions more deeply into solids, and to “stitch” dissimilar layers of metals or ceramics together to create totally new materials.

“The addition of this new machine is another example of why Cornell’s materials science and engineering department is considered one of the best in the country,” said Arthur Ruoff, director of the department. “Our faculty and students create and study all types of materials — ceramics, metals, polymers, and semiconductors — and this device offers a powerful tool for working with all these materials.

“Enormous credit should go to Jim Mayer and Ed Kramer for creating the superior research environment in which such a collaboration with IBM is possible,” said Ruoff.

The Rutherford system is used to analyze the composition of materials at and just below their surface through the reflection of beams of particles accelerated to high energies. By loading the accelerator with different “ammunition” and shooting it at the target in different ways, researchers can use their atomic machine gun for a variety of purposes.

“Rutherford Backscattering” is the technique for analyzing materials made of heavy atoms such as silicon or metals by measuring how the target reflects a beam of helium ions. Researchers can analyze samples by means of “proton-induced X-ray emission,” studying the X-rays produced when a beam of hydrogen ions, or protons, is shot at the target. The Rutherford system can also analyze the structure of light molecules such as polymers with “forward recoil spectroscopy” by studying how atoms of hydrogen are blasted off the material itself when a helium ion beam impacts at a low angle. By varying the energy of the beam, researchers can reach deeper and deeper beneath the surface of the material to learn of its structure.

Such accelerators can also be used to create new materials by embedding elements such as nitrogen or carbon into solid surfaces. Such bombardment can toughen metals used in such applications as artificial joints and machine tools. It can also create new semiconductors for microelectronics, by implanting various metal ions in silicon.

Cornell’s ion beam facility already includes an accelerator capable of generating beams ranging from 1.5 to 3 million volts for Rutherford Backscattering analysis. The IBM-provided machine, however, can launch helium ions with energies of 2 to 5 million electron volts. It can also generate 1 to 3 million-volt proton beams and accelerate nitrogen or carbon to energies necessary for implantation.

This means researchers will be able to reach twice as deeply into materials to study their composition. In the case of silicon targets, this would mean extending their reach from about one millionth of a meter to about two millionths of a meter.

It also means they will be able to do new kinds of analyses. They will be able to launch protons at targets with such high energies that atoms such as fluorine, oxygen and nitrogen can be made radioactive. The decay of such “activated” atoms will yield gamma rays at characteristic energies that the researchers can use to identify and measure their concentration.

The new system can also be used in a process called ion-beam mixing, in which the scientists first deposit a thin film of one material on another, and then zap the combination with a high-energy ion beam to mix the two layers.
This creates alloys that could not otherwise be made. For example, auto manufacturers are interested in producing metal with tightly bonded ceramic coatings, to make engines that would need no lubricants.

Finally, because of its high power, the beam from the new accelerator can be brought outside the machine, to be directed at targets in air. The previous machine could only probe samples within a special ultra-high-vacuum chamber. This means that Rutherford backscattering can be used to analyze such delicate materials as rare books, archaeological artifacts, and biological samples.

Professor Ed Kramer’s research offers a good example of how the new system will be applied.

Kramer and his colleagues use forward recoil spectroscopy to study how substances such as solvents diffuse into polymers and how polymers diffuse into one another. His basic research is important in a huge range of applications, just about anywhere advanced plastics are used, from microelectronics to plastic moldings.

Since the ion beam will bounce off heavier molecules with greater energies than off lighter ones, the scientists can distinguish, say, a solvent containing chlorine or oxygen from the polymer that has neither of these molecules. Or, if the scientists are studying two polymers, they can “tag” one with heavy hydrogen, or deuterium, and study how the two intermix.

The new system will enable Kramer to reach much farther into polymer layers to see how the long molecules “slither” together as they intermix.

— Dennis Meredith

from Cornell Chronicle 018_29 p9 April 16, 1987
Pressures greater than Earth’s core reached here

Within a hair-thin chamber squeezed between two flawless diamonds, static pressures greater than those at the center of the Earth have been achieved for the first time by Cornell materials scientists.

The development will allow scientists to produce and study new forms of solids that do not exist at normal pressures. Such solids could yield better basic theories of chemical bonds and the behavior of electrons in solids.

Graduate student Hui Xia displays the diamonds that were used to attain the highest static pressures yet achieved — greater than the pressure at the Earth’s center. The bottom diamond is mounted in part of the apparatus used to hold the diamonds, tip-to-tip, as they are squeezed to produce the high pressures in a tiny chamber between the tips.

The ultrapressures will also facilitate insights into the structure of the Earth and other planets, according to the scientists.

In a scientific paper in the December issue of the Review of Scientific Instruments, the researchers describe using an apparatus known as a diamond anvil cell to reach pressures more than 4,000 times greater than the pressure at the deepest spot in the ocean.

The pressure they reached was 4.16 megabars, while the pressure at the center the Earth is about 3.6 megabars. One megabar is about 980,000 times sea-level atmospheric pressure.

Reporting the experiments were Professor of Materials Science and Engineering Arthur Ruoff, graduate students Hui Xia and Huan Luo, and Assistant Professor of Materials Science and Engineering Yogesh Vohra.

Other research groups have achieved instantaneous pressures far higher — up to 100 megabars — in shock experiments using special gas cannons or nuclear explosions. However, the extremely high temperatures and fleeting nature of those experiments do not allow study of the structure of the compressed substances using lasers, X-rays and other probes.

The diamond anvil cell is widely used to achieve high pressures for long periods of time and with samples of a size that can be analyzed. The device typically consists of a pair of brilliant-cut diamonds, chosen for their crystal per-
fection, each with a minuscule flat area polished off its tip. The diamonds are mounted tip-to-tip, and a tiny steel gasket with a hole drilled in the middle is placed between them. The substance to be tested is added to the hole and the diamonds are mounted within a powerful vice and clamped together using a series of screws.

The Cornell scientists achieved the highest static pressures ever by using diamonds with especially small tips of about 20 micrometers, or about one-fifth the diameter of a human hair. (A micrometer is one-millionth of a meter.) The scientists obtained the specially cut one-third-carat diamonds from the diamond merchant D. Drukker & ZN of the Netherlands.

The sample hole in the steel gasket was even smaller — 10 micrometers, or about one-tenth the diameter of a hair and a few micrometers thick. The researchers produced the tiny sample chamber by drilling through the steel with a 10-micrometer-diameter carbide drill.

The Cornell scientists used tungsten and molybdenum as test substances in the sample chamber, because their physical properties at high pressures are experimentally and theoretically well known. Thus, these metals could act as a means of measuring the pressure. The sample volumes were about 40 trillionths of a cubic centimeter, approximately the volume of a droplet of fine aerosol mist.

After the diamond anvil cell was brought to the maximum pressure, the scientists studied the structure of the sample by subjecting it to the intense X-ray beam from the Cornell High Energy Synchrotron Source (CHESS). The CHESS beam is produced as a byproduct of Cornell’s high-energy particle accelerator, the Cornell Electron Storage Ring, operating at more than 5 billion electron volts. The resulting synchrotron beam has roughly the intensity of 1 million medical chest X-rays.

To perform X-ray studies of the tiny chamber, Ruoff and his colleagues constructed a special collimator to narrow the CHESS beam down to four micrometers in width.

By analyzing the diffraction of the X-rays by the sample of known material under pressure, the scientists could calculate the pressure being put on the sample. Besides X-ray studies, the scientists also use high-powered lasers to determine the optical properties of materials under pressure.

X-ray diffraction techniques are the most dependable for measuring the highest pressures reached in diamond anvil cells, emphasized Ruoff.

For example, two other research groups claimed in 1986 to have achieved pressures above the current 4.16-megabar Cornell mark, based on a pressure measurement technique that depended on the fluorescence under pressure of samples of ruby in the diamond anvil chamber.

However, said Ruoff, these reports are not considered valid, because of the unreliability of ruby fluorescence as a pressure measurement technique. At pressures above about 2.0 megabars, ruby fluorescence is rendered inappropriate for pressure measurements by such possible complicating factors as fluorescence of the diamonds, structural changes in the ruby or changes in the fluorescence spectrum of the ruby. To be valid, ruby fluorescence must be calibrated over the entire pressure range for which it is to be used, said Ruoff.

The groups reporting the high pressures based on ruby fluorescence measurements were Willie Moss and his colleagues at Lawrence Livermore Laboratory, who reported achieving 4.6-megabar pressures, and David Mao and his colleagues at the Carnegie Institution of Washington, who reported achieving 5.5 megabars. In both cases, said Ruoff, the groups used ruby beyond pressures where it had been calibrated, which was only to 1.8 megabars.

Extremely small diamond tips are required for diamond anvils to achieve ultrahigh pressures, said Ruoff, and the relatively large size of the diamond tips used in the 1986 experiments makes their claims extremely unlikely.

The highest static pressure previously reported, based on X-ray diffraction measurements, was 3 megabars in 1989 by Mao and Russell Hemley.
The Cornell researchers plan to use the improved diamond anvil cell for numerous basic studies of crystal structure. For example, they plan to attempt to create for the first time a particular “phase transformation” theorized to occur in molybdenum at multimegabar pressures, in which the metal changes crystal structure, from “body-centered cubic” to “hexagonal closest packed.”

The scientists also plan to explore whether diamond will become a metal at ultrapressures. They have already gathered data indicating that diamond is altering its structure as pressures rise above about 2.5 megabars.

Using the improved diamond anvil, the Cornell scientists will also attempt to squeeze hydrogen and nitrogen enough to cause them to become metals and to confirm that metallization by measuring the samples’ electrical conductivity. These studies would extend earlier research on oxygen by Cornell graduate student Serge Desgreniers and Ruoff, in which they found that the reflectivity of oxygen at high pressures resembles that of silver and indicates metallization.

The researchers plan studies of numerous materials important in the electronics industry, including germanium, silicon, gallium arsenide and gallium antimonide.

The Cornell work is supported by the U.S. Department of Energy, as well as the National Science Foundation through the Cornell Materials Science Center. CHESS is also supported by the National Science Foundation.

— Dennis Meredith

_from Cornell Chronicle 022_15 p1e68 December 13, 1990_
Metallic-hydrogen claims disputed by CU researchers

Scientists who presented evidence last year that they had transformed hydrogen into a metal by squeezing the gas at ultrahigh pressures were probably observing contaminating aluminum created by a chemical reaction in the sample chamber, according to Cornell researchers.

In the Feb. 11 issue of Physical Review Letters, materials scientists Arthur Ruoff and Craig Vanderborgh presented evidence questioning the conclusions of a 1990 article in that journal by David Mao and colleagues at the Carnegie Institution of Washington. In that article, the Carnegie researchers reported results which they interpret as evidence of metallization in hydrogen samples squeezed above pressures of 1.49 megabars. A megabar is about 1 million times sea-level atmospheric pressure.

The Carnegie scientists reported observing a reflectivity of 5 percent in the infrared, which they interpret as characteristic of metal in the pressured hydrogen sample. For comparison, a clean silver surface would give essentially a 100 percent reflectivity in the infrared.

Also inside the sample chamber in the Carnegie experiments was ruby powder. The fluorescence of ruby powder — or aluminum oxide with about 0.5 percent chromium — is commonly used as a secondary measure of ultrahigh pressures, “but in the Carnegie experiments, the scientists had purposefully added a lot more ruby powder to eliminate ‘fringes’ in their optical output pattern,” said Ruoff.

According to Ruoff and Vanderborgh, aluminum oxide and hydrogen would react at those pressures to produce aluminum metal, which would coat the ruby particles.

“When I saw their results, I realized that since there was a lot of contaminating aluminum oxide in that chamber and since hydrogen is a strong reducing agent, that there was a chance of a reaction at high pressure,” said Ruoff.

After exploring possible chemical reactions, Ruoff and Vanderborgh concluded that the reaction to form metal aluminum and aluminum oxide hydroxide had a very strong tendency to proceed under the influence of the extremely high pressures. Thermodynamic calculations showed that pressures above about 1.36 megabars would push the reaction between aluminum oxide and hydrogen toward forming aluminum metal, said Ruoff.

Such a ruby-hydrogen sample would have also misleadingly shown the characteristic electrical conductivity expected of hydrogen metal, because the aluminum-coated ruby particles would give a conductive aggregate, said the Cornell scientists. However, the Carnegie scientists studied only the reflectivity of the samples in the experiments reported last year.

The Carnegie scientists use a device known as a diamond anvil cell to achieve the high pressures. The device consists of a pair of brilliant-cut diamonds chosen for their crystal perfection, each with a minuscule flat area polished off its tip. The diamonds are mounted tip-to-tip, and a tiny steel gasket with a hole drilled in the middle is placed between them. The substance to be tested is added to the hole and the diamonds are mounted within a powerful vice and clamped together with screws.

“This example of one ultrapressure chemical reaction really opens the door to the whole subject of ultrapressure chemistry,” said Ruoff. “It brings up such questions as whether hydrogen at ultrapressures goes into diamond as tiny hydrogen atoms and greatly changes the properties of the diamond.” Such properties might include the diamond’s optical absorption or mechanical properties, said Ruoff.

“Also, does hydrogen go into the ruby and react with the chromium ion to change the pressure scale?” he asked.

Ruoff noted that when he produced metallic oxygen at high pressures in previous experiments, the oxygen reacted continuously at room temperatures with the stainless steel apparatus, while it did not do so at atmospheric pressure. — Dennis Meredith

from Cornell Chronicle 022_24 p7 March 7, 1991
‘Soccer-ball’ molecule is far stiffer than diamond

Theoretical calculations by scientists at IBM Corp. and Cornell have shown that the soccer-ball-shaped carbon compound known as buckminsterfullerene is far stiffer than diamond, currently the hardest known substance.

Nicknamed “buckyballs,” the 60-carbon-atom compound has generated enormous interest among researchers. Besides the intrinsic interest in the properties of the new substance, they foresee the possibility of a new class of materials that could have applications in electronics or as the basis for structural materials or lubricants. Buckminsterfullerene is named for the late architect Buckminster Fuller, who invented the geodesic dome, which has an identical structure of connected hexagons and pentagons.

The IBM-Cornell calculations suggest that the material’s remarkable resistance to compression could play a role in those applications.

In an article in the April 25 issue of Nature, the father-son team of Arthur and Rodney Ruoff describe their calculations showing that the individual buckyball molecule could prove more than twice as resistant to pressure as the corresponding single diamond structure. A crystal of many buckyball molecules, they calculated, could prove 50 percent more resistant to compression than diamond.

Rodney Ruoff is a postdoctoral fellow at IBM’s Thomas Watson Research Center in Yorktown Heights, N.Y., and his father, Arthur, is a professor of materials science and engineering and director of the Cornell Ceramics Program.

The two scientists based their calculation, called a “continuum elasticity approach,” on the known resistance of another carbon compound, graphite, to tensile stretching. Graphite, whose structure consists of stacks of flat layers, is the other form of pure carbon compound besides diamond, carbon black and buckminsterfullerene.

Once the scientists calculated the resistance to strain of a single layer of graphite, they calculated the resistance to stretching of that same layer if it were wrapped into a buckyball-like sphere. This resistance to stretching is functionally equivalent to the same molecule’s resistance to compression.

The resulting incompressibility was about twice that of the diamond structure, they found. However, said the Ruoffs, a crystal of many molecules of buckminsterfullerene would be only about 50 percent harder than diamond. The crystal, like a pile of soccer balls, would have empty spaces in the crannies between the molecules, which would reduce the stiffness of the crystalline material.

In fact, because the individual molecules in the crystal would not touch if the crystal were not under pressure, the crystal would be easily compressible until the molecules were squeezed together by moderate pressure. Once this “mushiness” was squeezed out, however, and the individual molecules were touching, the crystal would resist further compression far more than would diamond.

The Ruoffs are now exploring still another way to calculate how incompressible buckyballs might be. The second technique involves calculating the stiffness of the individual carbon-carbon chemical bonds in the molecule and extrapolating to the entire molecule.

While the Ruoffs are reluctant to speculate on specific applications of buckminsterfullerene, Arthur Ruoff said that the properties of the buckyballs could hint at new realms of materials.

“The fact that we’ve found one material harder than diamond means that we will probably find others,” he said.

Rodney Ruoff also pointed out that buckyball-type molecules could be made even stiffer by “reinforcing” them with an atom such as xenon added to the interior of their spherical structure.

“Preliminary calculations show that if a xenon atom was incorporated in the C-60 [60-carbon] molecule, the C-60 would be many, many times stiffer at moderate pressures, than a C-60 without the xenon atom inside,” he said.
The scientists said that experimental studies on the compressibility of buckyballs are being carried out by Steve Duclos of AT&T's Bell Laboratories and Keith Brister at the Cornell High Energy Synchrotron Source. Buckyballs, first made in quantity by Donald Huffman of the University of Arizona, were the subject of a major symposium at the American Physical Society meeting in Cincinnati in March. The Ruoffs were first motivated to do their calculations by that symposium.

Rodney Ruoff’s research is supported by IBM; Arthur Ruoff’s research is supported by the Department of Energy.

— Dennis Meredith

from Cornell Chronicle 022_31 p7 May 2, 1991
Scientists find pressures that transform diamond into metal

By squeezing tiny diamonds to pressures 4 million times greater than atmospheric pressure, Cornell scientists have gathered data revealing for the first time the ultimate pressures to which diamond may be squeezed before transforming into a metal.

That ultimate pressure, about 9 million atmospheres, will mark the upper limit of static ultrapressure research, because diamonds are the only known substance hard enough to form the chambers for squeezing substances to ultrapressures.

As the diamond transforms into a metal, it will become opaque to light used to probe the structures of pressurized samples. More importantly, however, the metallized diamond will lose its inherent strength.

Researchers can achieve instantaneous pressures far higher — up to 100 million atmospheres — in shock experiments using special gas cannons or nuclear explosions. However, the fleeting nature of those experiments does not allow study of the structures of the compressed substances.

Professor of Materials Science and Engineering Arthur Ruoff and Assistant Professor of Materials Science and Engineering Yogesh Vohra reported their findings in the May 1 Journal of Applied Physics.

The researchers used a “diamond anvil cell” which typically consists of a pair of brilliant-cut diamonds chosen for their crystal perfection, each with a minuscule flat area polished off its tip. The tip used in this study was 20 microns in diameter, about one-fourth the diameter of a human hair.

The diamonds are mounted tip-to-tip, and a tiny steel gasket with a hole drilled in the middle is placed between them. The substance to be studied is added to the hole, and the diamonds are mounted within a powerful vice and clamped together.

“Metallization will represent the end of the game as far as diamond anvils go,” said Ruoff. “When diamond becomes a metal, the bonding electrons within the crystal are ‘delocalized,’ that is, they can move throughout the crystal; delocalization means that the crystal becomes far weaker.

“Nobody has ideas at this point for making useful substances substantially harder than diamond at atmospheric pressures,” he added.

“However, it is conceivable that an inner stage of a diamond anvil cell could be an ‘intensifier’ of a crystal of buckminsterfullerene,” he speculated. (See article above.)

The substance to be subjected to ultrapressures would be encased in the buckminsterfullerene, he explained.

The limit on ultrapressure studies is particularly unfortunate, said Ruoff, because at higher pressures, scientists could study the behavior of the core electrons of atoms under pressure. Such studies would yield new insights into the nature of materials.

In their latest studies, Ruoff and Vohra subjected two kinds of diamonds to ultrapressures. Natural diamonds with nitrogen impurities were stressed to 4.05 megabars (4.05 million times atmospheric pressure). Also, relatively pure natural diamonds were subjected to 1.67 megabars.

The researchers could study the optical characteristics of the squeezed diamonds by placing tiny samples of metal in the sample chamber and using the metals as mirrors to reflect light shone into the diamonds.

As diamond under pressure begins to alter its structure toward a metal, it would be expected to transmit less and less light. By extrapolating the observed light absorption to that expected at higher pressures, the scientists could determine the point at which diamond would transform into a metal. They calculated that diamond would become a metal at pressures of 9.1 megabars.
Ultrapressure studies allow researchers to study forms of solids that do not exist at normal pressures. Such studies yield better theories of chemical bonds and the behavior of electrons in solids. Ultrapressure studies also offer scientists insights into the structure of the Earth and other planets.

— Dennis Meredith

from Cornell Chronicle 022_31 p7 May 2, 1991
Putting the squeeze on diamonds achieves top high-pressure award

By Larry Bernard

Arthur L. Ruoff, professor of materials science and engineering, assembles a pressure vessel in which twin diamond anvils squeezed together create the highest static pressures ever achieved.

Arthur L. Ruoff takes some of the world’s most beautiful diamonds, polishes them to a finish rarely seen in a Fifth Avenue jewelry store, and squeezes them with a thin metal gasket between their tips until one of them virtually explodes.

Rather than savor these brilliant-cut gems for a bright gold setting to adorn some lucky fiancée’s hand, Ruoff appreciates the beauty of these precious jewels for their role in science.

Using two 1/3-carat diamonds of exceptional quality, Ruoff, professor of materials science and engineering, has created pressures far greater than that at the center of the Earth to create new materials that do not exist at normal pressures, study crystal structure, the behavior of electrons in solids and chemical bonding of solids of various elements in a chamber one-tenth the diameter of a human hair.

“I know women who just cringe that I blow apart some of these diamonds,” said Ruoff, fingering a 16-sided flawless gem that provides one half of the diamond anvil pair that helps him attain such high pressures.

Such work has earned Ruoff, at Cornell since 1955, the highest award in high-pressure research — the Bridgman Award of the International Association for the Advancement of High Pressure Science and Technology. The award is given every other year to a premier researcher for outstanding contributions to high-pressure science. Percy W. Bridgman was the outstanding high pressure scientist of his time, who won the Nobel Prize in 1946.
Ruoff and his research group have achieved 560 GPa static pressure, or 5.6 million atmospheres, in pressure vessels using diamond tips squeezed together. As a comparison, the pressure at the center of the Earth is 3.6 million atmospheres; the deepest part of the ocean, 1,000 atmospheres (36,000 feet deep); car tires, 2 atmospheres. Ruoff was the first to reach static pressure greater than that at the center of the Earth in 1990 with 4.16 million atmospheres and reported in *Review of Scientific Instruments* (October 1992) the maximum pressure of 5.6 million atmospheres.

Such high pressures allow researchers to create novel materials that are not found under normal pressure. Using the diamond anvil technique, Ruoff has found that oxygen becomes an extremely reflective metal at 95 GPa, so reflective that it appears shinier than the metal pressure vessel itself. You can see it start to change color. It’s blue, then orange red, deep red and black, and that suggests it’s now a semiconductor, and further squeezing makes it a metal,” Ruoff said.

“Sulfur does the same thing,” Ruoff said. “At high pressures, it becomes a metal. First it becomes amorphous, then after further pressure increases it transforms to a new crystal structure and then at higher pressure still to a different crystal structure, which is metallic. As a result, it’s possible that both oxygen and sulfur could be excellent superconductors.”

Ruoff helped pioneer the use of X-ray diffraction to measure such ultrapressures. Using the Cornell High Energy Synchrotron Source, an X-ray source for scientific studies, he can “illuminate” the structure of solids used as the samples.

Ultrahigh pressure dramatically decreases the interatomic spacing, and so high-pressure studies provide a harsh test of the theories of bonding, Ruoff said. “Our results,” he said, “make it possible to improve these approximate theories, so that we will with confidence be able to calculate the properties of materials that have not yet been made and then, if they look interesting, proceed to try to synthesize them.

Recently, it was calculated from theory that aluminum nitride would transform to a new crystal structure at high pressure. “We have made this and found that it remained in this new crystal structure when the pressure was removed,” he said.

Ruoff, who teaches courses in thermodynamics, kinetics and a senior laboratory course, regularly has undergraduate students work in his laboratory. He hopes to explore the outer realm of ultrahigh pressures, eventually achieving 1,000 GPa or 10 million times atmospheric pressure.

For such studies, he will have to shave the diamond tips down to about 10 microns — to about eight times smaller than the diameter of a human hair. Currently, the outer limit is double that, about 20 microns, Ruoff said, the size of the diamond tip he used to exceed the pressure at the center of the Earth.

*from Cornell Chronicle 025_06 p1e&8 September 30, 1993*
Program urges undergrads to try research

By Rachel F. Preiser

One Thursday last June in Clark Hall, Bob Richardson, professor of physics, enjoyed impersonating the mad scientist as he poured smoking liquid nitrogen into a Styrofoam cup. The demonstration was part of his talk about “Low-Temperature Science” that he gave to student researchers participating in Cornell’s 10-week Research Experience for Undergraduates program.

“The whole idea of this is to give you a sense of the width and breadth of modern research in materials science,” explained Richardson at the start of his talk. The liquid nitrogen transforms from a gas into a solid as pressure above the flask is decreased.

“I love this demonstration,” Richardson said. “It’s amazing that while the nitrogen in the air in the lecture room is a gas, we can be watching the nitrogen in the flask become solid.”

The Research Experience for Undergraduates, sponsored by the Cornell Materials Science Center, provides undergraduates from Cornell and other institutions across the country with the opportunity to pursue their own research and to interact with scientists in other research fields in a 10-week summer program. It was started in 1987 to foster undergraduate interest in pursuing graduate work in engineering and the physical sciences, explained Wolfgang Sachse, professor of engineering, who is the program’s current director. Funding is allocated by the Materials Science Center to faculty members who agree to sponsor a student researcher interested in working in their field of interest.

Students apply to participating faculty members, and, if successful, are granted a $3,000-or-more stipend and the opportunity to work alongside graduate students and professors, preparing and analyzing specimens, calibrating and fabricating equipment, and pursuing their own research projects. After 10 weeks of intensive research, the 26 REU students presented the results of their research to their peers and shared what they had learned.

Half the researchers were from Cornell and half were from other universities, including Harvard, City College of New York and the University of Puerto Rico. Most found out about the Cornell program when they applied to their faculty advisers for summer research opportunities. In addition to working 8 a.m. to 5 p.m. in the lab, REU participants attended Thursday lectures and demonstrations given by faculty members about aspects of their field of research. Sachse emphasized that these lectures not only are occasions for students to become acquainted with the academic work of preeminent scientists in different fields of materials science, but also to meet one another and exchange ideas.

Among the Thursday lecturers: Roald Hoffmann, professor of chemistry and Nobel laureate; Joel Brock, professor of applied and engineering physics; Mike Thompson and Art Ruoff, professors of materials science and engineering, and Clif Pollock, professor of electrical engineering.

Sean Smith, an REU researcher who is now a senior at Albright College, did his summer research in Olin Lab with Hector Abruna, professor of chemistry.

“The lectures give us a chance to hear top researchers talk about their work,” he said. “There is so much going on at Cornell, and the talks allow us to find out about it.” Smith’s research involves synthesizing transition metal complexes that catalyze the reduction of oxygen to water and carbon dioxide to carbon monoxide, a process which is particularly useful in fuel cells. He hopes to do his graduate work at Cornell and speaks highly of the REU program as “a great opportunity to get your feet wet.”

Stephen Gomez-Diaz came from the University of Puerto Rico, where she is a senior chemistry major, to work with Paul Houston, professor of chemistry, in Cornell’s Summer program. She is examining laser photo dissociation of small molecules.
Graduate students Wei Kong and Laura Dobeck worked with undergraduates in the laser facility at Bake Lab as part of the Research Experience for Undergraduates program last summer.

Although most of her work has been with solutions, she took up gases at Cornell and appreciated the opportunity to learn a new field: “It’s a great chance to meet people in other fields of science. The REU program encourages us to do interdisciplinary research and to exchange ideas with students who don’t work in our specialty. The most modern research techniques have applications across many different fields,” she said. Gomez-Diaz studied the dynamics of photo dissociation of trans-glyoxal.

The program also sponsors Cornell students, like Kai Wu, a junior in physics at Cornell. He worked with Wilson Ho, professor of physics, studying chemical vapor deposition of thin diamond films under conditions of low temperature and pressure.

‘Before I started the research, I was reading about the methods and experiments. In just a few weeks I’m actually doing what I’ve been reading about. It never ceases to amaze me.’ —Kai Wu

The research has “an enormous range of applications,” Wu explained, “because of diamond’s unique properties.” Wu gives as an example the production of ultrahigh-performance speakers with a diamond core, which will give a better-quality sound because diamond’s lightweight strength allows it to transmit sound without being deforming. Wu sees the REU program as immensely important to his professional trajectory:

“It’s a must for people who are even considering graduate work in science. A program like this is the only way to get a sense of what you will be committing yourself to for the next five years.”

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Symposium to honor Professor Arthur Ruoff’s 50 years at Cornell

By Bill Steele

Being married to a university for 50 years is an occasion to celebrate, so the Department of Materials Science and Engineering (MS&E) and students of Arthur Ruoff, the Class of 1912 Professor of Engineering, have organized a symposium to honor his golden anniversary, slated for Monday, Sept. 18, 9 a.m. to 5 p.m., in the Statler Hotel Carrier Grand Ballroom.

Speakers at the symposium, which is devoted to several aspects of high-pressure physics, will include many of Ruoff’s former students, now professors and industrial scientists, Cornell Professors Roald Hoffmann and Neil Ashcroft, and Ruoff’s son Rodney Ruoff, the John Evans Professor of Nanoengineering at Northwestern University.

Arthur Ruoff has dedicated his career to the study of the effect of very high pressure on materials, including the making of metallic oxygen, xenon and sulfur. In 1990, by squeezing small samples between two diamond anvils, he reached a static pressure of 416 GigaPascals (GPa), becoming the first scientist to create a static pressure greater than at the center of the Earth, 361 GPa. Scientists had theorized that at such a pressure, hydrogen would become a metal and a superconductor, but in 1998 Ruoff disproved the theory, cracking several diamond anvils in the process. He later obtained a pressure of 560 GPa, the highest static pressure obtained to date.

After earning his Ph.D. at the University of Utah in 1955, Ruoff joined the Cornell faculty as an assistant professor of mechanics and materials. In 1965 he was a founding member of the new Department of MS&E and later served as its director (1978-88). On July 1 of this year he became professor emeritus, but he intends to continue his research. Although he says he will miss some aspects of teaching, “It will be great to have the time to travel to more meetings and get new ideas.”

Among other awards, Ruoff received the Bridgman Medal for outstanding high pressure research from the Association Internationale pour l’Avancement de la Recherche et de la Technologie aux Hautes Pressions and the Westinghouse Award for Outstanding Teaching. He received a National Science Foundation Science Teacher Fellowship in 1962. He is the author of two books on materials science and developed an audio-tutorial course on introductory materials science, which has been used at 60 universities.

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