



On the Legacy of **Arthur Louis Ruoff** Collected Resources



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Cover: Stills of Art Ruoff and Roald Hoffmann from the video:
A Conversation with Arthur Ruoff

This compilation consists of Background Resources and previously published articles from *Engineering: Cornell Quarterly* and the *Cornell Chronicle*.

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<http://hdl.handle.net/1813/39289>

proofreader: Dianne Ferriss
produced by J. Robert Cooke

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Published by The Internet-First University Press
Founded by J. Robert Cooke and Kenneth M. King



Photo: Cornell University Photography (Harrington)

Arthur L. Ruoff: Background Resources

Professor Arthur L. Ruoff retires from MS&E

Symposium to Honor Professor Arthur Ruoff's 50 Years at Cornell

Golden Anniversary for Founder of High-Pressure Program at CHESS

Personal Data: Arthur Louis Ruoff

Research Contributions and Scientific Achievements of Arthur L. Ruoff



Professor Arthur L. Ruoff retires from MS&E

At the end of the Spring 2006 Semester, Arthur L. Ruoff, the Class of 1912 Professor of Engineering in the Department of Materials Science and Engineering at Cornell University will retire. Professor Ruoff helped form MS&E into a department that produces innovative and cutting-edge research while remaining dedicated to excellence in education. His distinguished career in teaching and research has been an inspiration to faculty and students for fifty years.

Professor Ruoff received his BS, with honors, in chemistry from Purdue University in 1952 and his PhD, under the direction of Henry Eyring, in Physical Chemistry and Physics from the University of Utah in 1955. He arrived at Cornell in the fall of 1955 as an Assistant Professor in the Department of Mechanics and Materials. He then moved to the Department of Engineering Physics and Materials Science (EP &MS). In 1965, the Department of Materials Science and Engineering was created and Ruoff, along with thirteen other faculty members, became the first faculty of the newly formed department. This is also the year in which Ruoff became a full Professor with tenure. In 1978 he was named a chaired professor.



Professor Ruoff has dedicated his career to the study of the effect of very high pressure on materials. His research has been marked by many impressive achievements. In 1990 he reached a static pressure of 416 GPa, becoming the first scientist to create a static pressure greater than at the center of the earth, 361 GPa. He has carried out optical studies on diamonds and has obtained x-ray diffraction patterns of tungsten at 560 GPa, the highest static pressure obtained to date. Professor Ruoff has published over 300 scientific publications and has been invited to give talks in 18 countries. He wrote two books on Materials Science published by Prentice Hall in 1972 and 1973, and developed an audio-tutorial course on Introductory Materials Science which has been used in 60 universities.

Throughout his career, Professor Ruoff has won many prestigious awards for his achievements in research. In 1993, Professor Ruoff received the Bridgman Medal for outstanding high pressure research from the Association Internationale Pour L'Avancement De La Recherche Et De La Technologic Aux Hautes Pressions (AIRAPT), the International Association for Research at High Pressure and Temperature. In 2004 he was named a Distinguished Alumnus of the University of Utah and in 2005 a Distinguished Alumnus of the Chemistry Department of Purdue University. In addition to awards for excellence in research, Professor Ruoff has received many awards for excellence in education. Even while managing an internationally renowned research group Professor Ruoff remained dedicated to mentoring and educating his students. In 1956 he won the Westinghouse Award for Outstanding Teaching. He has also won the National Science Foundation Science Teacher Fellowship (1960-1962). During his fifty years of teaching he has mentored thirty-nine students who received their PhD's and four who received their MS degrees. In addition, he has had nineteen post-doctoral research associates work with him at Cornell.

Professor Ruoff led a distinguished career as both a researcher and educator: he has been a leader in his field and a mentor to students for fifty years. His service to Cornell includes being a member of the committees which

wrote the research proposals for the Materials Science Center, The National Facility for Submicron Research and the Cornell High Energy Synchrotron Source. He was one of the founding faculty members of MS&E and served the Department as Director from 1978 to 1988. MS&E has been privileged to have had Arthur Ruoff as a faculty member for forty years. His legacy of excellence in research and teaching will continue to be an example to which faculty and students aspire for many years to come.

Professor Ruoff and his wife Enid have five sons¹:

William graduated from Cornell in 1978 and has a Ph.D. in toxicology from Illinois. He is the Senior Project Risk Assessor at URS Greiner-Woodward Clyde Consultants in Denver.

Stephen graduated from the Cornell Materials Science and Engineering Department in 1979. He is CEO of IMR Test Labs in Ithaca, NY, Charleston, SC, and Louisville, KY. He is on the Board of the Cornell Center for Materials Research.

Rodney graduated from the University of Texas in 1981. He has a Ph.D. in chemical physics from Illinois. He was a Fulbright Scholar in Germany and is The John Evans Professor of Nanoengineering at Northwestern University.

Jeffrey graduated from Cornell in 1985, and received an M.F.A at Temple and a Ph.D. at Iowa in film studies. He was a Fulbright Scholar in France and is a professor of Film and Television Studies at Dartmouth College.

Kenneth graduated from Harvard in 1989. He has a Ph.D. in Japanese Studies from Columbia. He was a Fulbright Scholar in Japan. He is the Director of the Institute for Japanese Studies at Portland State University and the winner of the 2004 Osaragi Prize for Commentary for his book, *The People's Emperor*.

http://www.mse.cornell.edu/news/upload/MSE_News06.pdf

¹ See page 15 for a version that was updated on April 12, 2015.

Sept. 13, 2006

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

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.

Source URL:

<http://www.news.cornell.edu/stories/2006/09/symposium-honor-arthur-ruoff-50-years-cornell#comment-0>

Golden Anniversary for Founder of High-pressure Program at CHESS



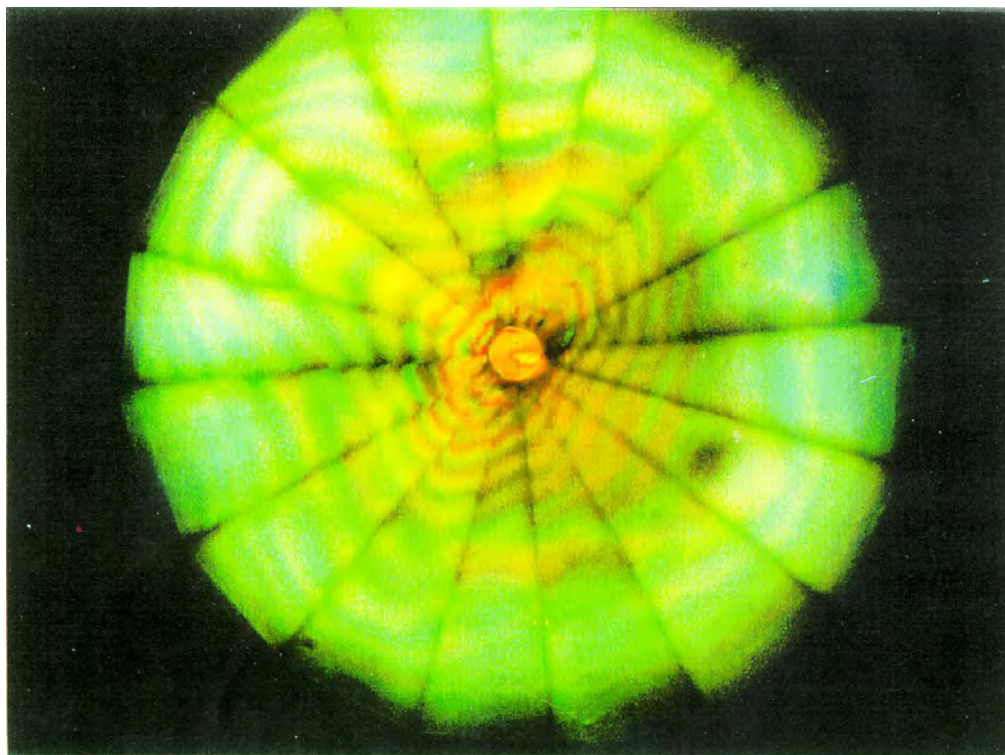
Professor Arthur L. Ruoff

The life and career of Arthur L. Ruoff was celebrated on September 18, 2006 on the occasion of his 50th anniversary as a Cornell Professor. Ruoff, the Class of 1912 Professor of Engineering, was honored by fifteen speakers who recalled fondly his career as both an innovative scientist in the field of high-pressure research and a dedicated educator and advisor.

Organized by former students and his home department of Materials Science and Engineering (MS&E), the speakers were mostly related to Ruoff as one of his 43 former graduate students or 19 post-doctoral research associates, fellow faculty or collaborators – from the US and abroad. Among the list were former students, many now professors and industrial scientists: Prof. M. Baublitz (1982) from Boston University, Dr. S.J. Duclos (1990) from General Electric Company and Prof. Y.K. Vohra (1992) from the University of Alabama. Baublitz was the first scientist user of the high pressure x-ray facility started by Ruoff at the Cornell High Energy Synchrotron Source, CHESS. Also paying tribute were Cornell Professors Roald Hoffman and Neil Ashcroft, and Ruoff's son Rodney Ruoff who is the John Evans Professor of Nanoengineering at Northwestern University.

Ruoff was a founding member of the MS&E department in 1965 and focused his career on studying materials under high pressure. He is a master of designing and building diamond anvil cells and squeezing small specimens to very high pressures. In the 1990s he reached a static pressure of 416 GigaPascals (GPa), becoming the first scientist to ever reach a static pressure greater than the center of the earth (360). Among many topics, his group studied virtually all the alkaline chalcogenides [1], was the first to obtain x-ray diffraction patterns above 200 and 400 GPa for a variety of elements [2-5], and created and studied the stable phases of metallic oxygen, metallic xenon, and metallic sulfur [6-9]. Ruoff commented that “the chalcogenides showed an interesting correlation with ionic radii which would have pleased Linus Pauling”. By 1998 he had disproved theoretical expectations that hydrogen

would form a metal at expected pressures [10] and, along the way, reached upwards of 560 GPa [11], the highest man-made static pressure to date.



Polarized light photograph of a solid hydrogen specimen inside a faceted diamond-anvil cell. The solid piece of hydrogen is 10 microns in size, glowing yellow inside the 20 micron cell volume at center. If the hydrogen were metallic it would not transmit light and appear black. [From reference 10]

Surveying a wide variety of materials like this was bound to turn up surprises. For instance, about xenon, an inert gas at standard atmospheric conditions, Ruoff recalls “we studied the FCC to HCP phase transition up to 170 GPa and found xenon turning metallic at 140 GPa, with no noticeable volume change in the HCP phase.” The acronyms refer to arrangements of the atoms in crystal lattices that are either face-centered-cubic or hexagonal-close-packed. “It was a beautiful study. When metallized, xenon looks sky blue because an indirect bandgap transition (in the electronic structure) allows it to absorb red light. Theory calculates it nicely.” As a second example, it was a long held opinion that all elements, at very high pressures where they become metallic, would assume close-packed arrangements with twelve nearest neighbor atoms. Ruoff’s group showed that they instead formed body-centered structures with only eight nearest neighbors.

His outstanding achievements garnered praise in science and education circles. In 1993 Ruoff was awarded the prestigious Bridgman Medal for outstanding high-pressure research, citing his work in high-pressure phenomena, particularly insulator-to-metal transitions and x-ray diffraction studies at pressures in the multimegabar regime. In 1956 he won the Westinghouse Award for Outstanding Teaching and in 1962 a National Science Foundation Science Teacher Fellowship. He has written two books on materials science and developed audio-tutorial course materials in materials science that have been used at many universities. In addition to being chairman of the MS&E department for 10 years he initiated the Cornell Industrial Affiliates Program, the MS&E News, and the Cornell Ceramics Program. His undergraduate school, Purdue (1952), presented Ruoff a Distinguished Alumnus of Chemistry Award in 2005, and his graduate school, Utah (1955), gave him a Distinguished Alumnus Award in 2004. Ruoff was a principal investigator on an NSF award in 1987 to develop a national high-pressure user facility at the Cornell High Energy Synchrotron Source. Co-investigators included B.W. Batterman, CHESS founder and first director, D.H. Bilderback, CHESS Associate Director, W.A. Bassett (Cornell Geology) and Y.K. Vohra. This

facility grew to encompass two experimental stations. Today scientists from over the world visit to use high-energy x-ray beams as structural probes of materials held inside diamond-anvil cells. Sometimes together and sometimes separately, Ruoff and Bassett have both pioneered new types of high-pressure apparatus, cells, and techniques that shaped the research capabilities of both the x-ray and materials science fields. The facility has trained students and post-doctoral associates who have gone on to help develop similar x-ray-based, high-pressure materials programs at other synchrotron light sources, universities and industries around the world.

“Ruoff is amazingly focused and hardworking, and is a marvelous example of what persistent, creative energy can do for a project”, says Bilderback. “He was and is a visionary in high-pressure science and has shown how important studying materials under extreme conditions can be for materials sciences in general”. “The highlight of the symposium evening dinner was Ruoff personally praising the technical help and collaborators for their necessary and critical contributions to his career – he’s a real gentleman in that way”.

Outside his career of applying pressure to diamond-anvil cells, Ruoff has withstood pressure applied to himself as a coach for little league soccer, youth hockey, and two terms as President of the Ithaca Youth Hockey Association! Retired from teaching this past July 1st, Ruoff says he will miss teaching but will continue his active research program and travel more.

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Extracted from: <http://news.chess.cornell.edu/articles/2006/RuoffAnnv.html>

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from:

http://people.ccmr.cornell.edu/~ruoff/alr_cv_2_14_12.pdf

PERSONAL DATA

ARTHUR LOUIS RUOFF

The Class of 1912 Professor of
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and Engineering
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Home Address:

216 Texas Lane
Ithaca, NY 14850
(607) 257-0695

Born: September 17, 1930, USA

Married: Enid Frances Seaton, January 24, 1954

Children: Five sons

Education Information: B.S. Chemistry, 1952, Purdue University
Ph.D. Physical Chemistry and Physics, 1955,
University of Utah

Honors:

Standard Oil of California Fellowship, 1952-1955
National Science Foundation Science Teacher Fellowship, 1960-1962
Western Electric Fund Award for Excellence in Instruction of
Engineering Students, 1966-1967, presented by ASEE
Engineer of Distinction - Engineers Joint Council, 1970
American Men and Women of Science, 1970
Fellow of the American Physical Society, 1971
Who's Who in the East, 1971
The Class of 1912 Professor of Engineering, 1979
Who's Who in America, 1980
Who's Who in Technology Today, 1980
New York Academy of Science, 1984
Fellow, the Böhmische Physical Society, 1988
Bridgman Award (Presented by AIRAPT (The International High
Pressure Association) for Outstanding High Pressure Research),
1993. This is the prize to win in high pressure.
Honored by the University of Utah as a Distinguished Alumnus, 2003
Honored by Purdue University as a Distinguished Alumnus, 2005

Employment Record: (Major Positions only)

Assistant Professor, Cornell University, 1955-1958
Associate Professor, Cornell University, 1958-1965

Visiting Associate Professor, University of Illinois, 1961-1962
Professor, Cornell University, 1965-
Class of 1912 Professor of Engineering, 1978-
Director, Materials Science and Engineering Dept., 1978-88
Chairman, Program Committee of the National Research & Resource Facility
for Submicron Structures, 1979-1986
Currently Emeritus Professor in graduate fields of Materials Science and
Engineering, Applied and Engineering Physics, and Earth and
Atmospheric Sciences, 2005

Research:

Director of Thurston High Pressure Laboratory. Research in various areas of Materials Science: High Pressure including liquid and gas systems; Ultra Pressure including diamond indenter system and opposed diamond anvil system (which included reaching the highest static pressure 560GPa, (earth's core is at 360GPa)); energy dispersive X-ray diffraction in the megabar regime using synchrotron radiation; Study of insulation to conductor transitions at megabar pressures (which included making metallic oxygen and metallic sulfur); optical absorption, reflectivity and Raman spectroscopy at megabar pressures; study of strength of diamond; study of various mechanical properties; microfabrication including study of inorganic resists, development of X-ray sources, submicron fabrication of interdigitated electrodes, reactive ion beam etching.

Teaching:

Lecturing in graduate course work and undergraduate Materials Science courses. Development of audio-tutorial undergraduate courses, learning center, etc.

Committee and Administrative work at Cornell:

Of which the most important assignments have been:

- a. Member of Committee to write proposals and solicit ARPA BLOCK FUNDING SUPPORT for Materials Science. We won Member of the Executive Committee of the Cornell Materials Science Center for first five years.
- b. Organized the Graduate Field of Materials Science and Engineering, and was the first Field Representative.
- c. Work on Lloyd Smith Committee to study future organization of the Engineering College at Cornell (this reorganization was carried out).
- d. Member of Faculty Council (Advisory Committee to President).

- e. Member of University Senate
- f. Originate and run Materials Science and Engineering Research Colloquium.
- g. Organize and run College-wide Colloquium in Instructional Technology.
- h. Major contributions to Preparation and Presentation of Proposals for the National Research and Resource Facility for Submicron Structures; was the Chairman of its Program Committee for six years.
- i. Contributor to proposal for the Cornell High Energy Synchrotron Source, Member of its Executive Committee.
- j. Member of committee which wrote and made presentation for the Resource Facility for Submicron Structures.
- k. Director, Materials Science and Engineering (1978-1988).
- l. Originate and edit MS&E News (1979).
- m. Originate and run MS&E Industrial Affiliates Program (1982).
- n. Originate new Electronic Materials undergraduate degree program (1983).
- o. Originate and first Director of the Cornell Ceramics Program 1985-1995.

Other Professional Activities

Member, American Ceramics Society

Member, Materials Research Society

Member (and Fellow) American Physical Society

Co-organizer, Western New York Section, Materials Research Society

Policy Committee, University Materials Council

International Advisory Committee of AIRAPT

Committees of the Materials Advisory Board

Editorial Board: Advances in Materials Research

Regional Editor: Phase Transitions

Consultant:

To numerous industries and laboratories on high pressure phenomena, including diamond production, to several universities on multimedia instruction.

Other Activities:

Manager and coach for youth hockey teams and youth soccer teams

President of Ithaca Youth Hockey Association, 1972-1973

(youth hockey program for over 500 Tompkins County boys)

Director, Ithaca Youth Hockey Association

Studies of the History of Knowledge and Economics

Travel, classical music, opera and championship finals in sports

Intensive all consuming study and analysis of energy for the past ten years

TALKS AT CONFERENCES

Has presented invited lectures at international conferences in Canada, Mexico, Brazil, Japan, India, Greece, Russia, Iran, England, Scotland, Sweden, Germany, East Germany, France, Poland, Portugal, Italy, and Holland as well as the United States.

PUBLICATIONS

Articles Published

Has published over 322 articles in scientific journals and in books.

The publication list is available.

A FEW HIGHLIGHTS

- First to reach static pressure above that at earth's core (360 GPa).
- Eventually reached 560 GPa.
- First to make metallic oxygen
- First to make metallic sulfur. At pressure of 150GPa it is a superconductor, $T_c=17$
- Succeeded in showing that hydrogen is not yet a metal at 420GPa.

BOOKS AND OTHER WORKS

1. With H.D. Block, Differential Equations for Engineers and Scientists, Pennysaver Press, Interlaken, New York (1961).
2. Introduction to Materials Science, 697 pp., Prentice-Hall, Englewood Cliffs, New Jersey (1972). Now published by Krieger Publishing Co., Melbourne, Florida.
3. Solution Manual for Introduction to Materials Science, 158 pp., Prentice-Hall, Englewood Cliffs, New Jersey (1972).
4. Materials Science, 926 pp., Prentice-Hall, Englewood Cliffs, New Jersey (1973).
5. Introductory Materials Science, (Audio-tutorial course) Packaged Courses, Ithaca, New York (1973).
6. Concepts of Packaged Courses (Slide-tape presentation), Packaged Courses, Ithaca, New York (1973).
7. The Declaration of Energy Independence, 148 pp., Linus Publications Inc. (2011).

ENERGY

Statement by the author.

I have spent the last ten years thoroughly studying energy consumption and energy production, including 3D seismic surveying, drilling, chemical analysis and production, pipelines, coal production and transportation and use, uranium, mining, refining, nuclear reactor design and construction and operation and nuclear waste disposal. I also studied the world peak production rate. As I have a long term interest in economics, I also studied how the United States government spends its money. First comes the military, then entitlements, leaving nearly nothing for infrastructure, especially non-fossil fuel plants and seriously damaging manufacturing and hence destroying jobs.

I made visits to energy facilities from 3D seismic shots in Wyoming, refineries in Long Beach and Houston, oil (tar) sand production in Athabasca, Canada, geothermal production in California and New Zealand and hydroelectric facilities in the U.S., Brazil, New Zealand and China (under construction) to G.E. nuclear

reactors in the U.S. and Areva reactors in France, to G.E. windmills in Lethbridge, Canada, in California, Costa Rica and New York, and solar sites in Georgia, California, Texas, and Germany.

FAMILY

Wife: **Enid Frances Seaton Ruoff**

Elected to the Ithaca City School Board – 2 terms.

Trustee of the Tompkins County Library Board, 10 years.

Distinguished Alumna of the College of Eastern Utah, 2008.

Sons:

William graduated from Cornell in 1978 and has a Ph.D. in toxicology from the University of Illinois. He is the Senior Project Risk Assessor at URS Greiner-Woodward Clyde Consultants in Conshohocken, PA. He has made assessments which required improvement in Sitka, Alaska, the Grand Canyon, and Salt Lake City.

Stephen graduated from the Cornell Materials Science and Engineering Department in 1979. He established and was CEO of Ithaca Materials Research Test Labs in Ithaca, NY, Charleston, SC, Louisville, KY, and Portland, OR. He recently sold the company. He is on the Board of the Cornell Center for Materials Research.

Rodney graduated from the University of Texas in 1981. He has a Ph.D. in chemical physics from the University of Illinois. He was a Fulbright Scholar in Germany and the John Evans Professor of Nanoengineering at Northwestern University, and now is Director of the Ulsan National Institute of Science and Technology (UNIST), in Ulsan, South Korea. He recently received the David Turnbull Award from the Materials Research Society.

Jeffrey graduated from Cornell in 1985 and received an MFA at Temple and a Ph.D. at Iowa in film studies. He was a Fulbright Scholar in France and is a Professor of Film and Communication Studies at Dartmouth College. His documentary “Pilobulus” received praise from Ken Burns.

Kenneth graduated from Harvard in 1989. He has a Ph.D. in Japanese Studies from Columbia. He was a Fulbright Scholar in Japan. He is the Director of the Institute for Japanese Studies at Portland State University, and the winner of the 2004 Osaragi Prize for Commentary for his book, “The People’s Emperor”. Since the publication of his study of the monarchy in postwar Japan, he has become recognized as the leading western authority on the imperial house in Japan. He recently received the Japanese Consul General’s Commendation.

All five sons were most valuable players in their high school sport, were starters and lettered in sports in college.

A.L. Ruoff's Publications

1. A.L. Ruoff and H. Eyring, "The Mechanical Properties due to the $\alpha \rightarrow \beta$ Transformation in Natural Keratin Fibres", Proc. Intl. Wood Conf., Australia, 1955, p. D-9.
2. C.E. Reese, A.L. Ruoff and H. Eyring, "The Mechanical Behavior of Polyacrylonitrile Fibres", *ibid.*, p. D-27.
3. F. Frank and A.L. Ruoff, "A Method of Measuring Poisson's Ratio of Fibers", *Textile Research Journal* 28, 213 (1958).
4. Arthur L. Ruoff, "An Alternate Solution of Stefan's Problem", *Quarterly of Applied Mathematics* 15, 197 (1958).
5. D.A. Stuart and A.L. Ruoff, "Impact Loading of Structures Using Double Acting Bombs", *Society Experimental Stress Analysis* 16, 7 (1959).
6. A.L. Ruoff, S.W. Liu and F. Frank, "Aerodynamic Heating of Parachutes", WADC T.R. 57-157 ASTIA Document No. AD 142261, (December 1957).
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15. R.W. Balluffi and A.L. Ruoff, "On Strain-Enhanced Diffusion in Metals. I. Point Defect Models", J. Appl. Phys. 34, 1634 (1963).
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18. A.L. Ruoff, "Diffusion During Deformation by Surface Intensity Methods", J. Appl. Physics 36, 2207 (1965).
19. R.H. Cornish, B.M. Butcher and A.L. Ruoff, "Techniques for Measuring Creep Strains at High Hydrostatic Pressures", ASME Publication #64-WA/PT 30.
20. J.E. Hill and A.L. Ruoff, "Velocity of Sound Measurements in Liquid Metals", Rev. Sci. Instr. 36, 1465 (1965).
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22. A.L. Ruoff, "Mass Transfer Problems in Ionic Crystals with Charge Neutrality", J. Appl. Phys. 36, 2903 (1965).
23. A.L. Ruoff, "Space Charge Effects Near Surfaces of Stoichiometric Ionic Crystals", Materials Science Center Report #309 (1965).
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315. A.L. Ruoff, L. Sun, S. Natarajan, C-S. Zha, and G. Stupian, "Technique for X-ray Markers at High Pressure in the Diamond Anvil Cell", *Rev. of Scientific Instruments* 76, 036102 (2005)
316. L. Sun, A.L. Ruoff, and G. Stupian, "Convenient Optical Pressure Gauge for Multimegabar Pressures Calibrated to 300 GPa", *Applied Physics Letters* 86, 014103 (2005)
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318. A.L. Ruoff, L. Sun, C-S. Zha, and G. Stupian, "Optical Studies on Methane to 300 GPa: Black Methane at 300 GPa", Proceeding of Joint XX AIRAPT International Conference and XLIII EHPRG meeting on High Pressure Sci. and Tech., T10, p. 359, Karlsruhe (2005)
319. A.L. Ruoff, L. Sun and G. Stupian, "The Diamond Anvil Tip Pressure Scale Calibrated to 300 GPa", Proceeding of Joint 20th AIRAPT – 43rd EHPRG International Conference on High Pressure Sci. and Tech., (2005)

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- 322. C. Narayana, R.G. Greene and A.L. Ruoff, "Studies on Silane to 70 GPa", Joint 21st AIRAPT and 45th EHPRG International Conference on High Pressure Science and Technology, Journal of Physics: Conference Series 121, 042019 (2008)
- 323. A.L. Ruoff, "World Poverty, Another Look", Environment, Development and Sustainability

RESEARCH CONTRIBUTIONS AND SCIENTIFIC ACHIEVEMENTS OF ARTHUR L. RUOFF

While Ruoff was working on his thesis research with Henry Eyring, Prof. Peter Gibbs (who did high pressure science) introduced him to Percy Bridgman's book which included studies of equation of state, phase transitions, mechanical properties, etc., to pressures of up to 78 kb (7.8 GPa) when corrected by Harry Drickamer, George Kennedy and others.

I. High Temperature Creep of Metals

HTC of metals is controlled by bulk diffusion. Measurements of the pressure dependence of HTC yield the activation volume for self-diffusion, ΔV_{SD} . Ruoff's group was the first to do this and did it in an ingenious way by pressure cycling, so they obtained as many as twenty independent values from one sample. Their study on *Al* was especially significant as ΔV_{SD} was found to be greater than the atomic volume: $\Delta V_{SD} = 13.6 \pm 0.6 \text{ cm}^3/\text{mole}$ compared to the molar volume of $9.97 \text{ cm}^3/\text{mole}$. For several other metals studied by the Ruoff group, ΔV_{SD} is about 2/3 of the molar volume.

B.M. Butcher, H. Hutto and A.L. Ruoff. Activation Volume and Energy for Self-Diffusion in Aluminum. *Applied Phys. Lett.*, **7**, 34 (1965).

- *As a result of theoretical studies by Ashcroft, Ruoff realized that the pseudopotential for Al was different than for other metals and studied Al because of this, which is why we have the interesting result that the activation volume for self-diffusion in Al is LARGER than the atomic volume (by the factor 1.36).*

Later pseudopotential calculations confirmed the result.

II. Pressure Dependence of Yield Stress

Percy Bridgman, who won the Nobel Prize for his extensive research on high pressure (up to an actual 7.8 GPa) had attempted to measure the pressure dependence of the yield stress of steel but was unable to observe an effect. Ruoff who had taught the first dislocation course at Cornell knew that in an elastically isotropic solid (which could be an equiaxed polycrystalline aggregate of randomly oriented crystals having any symmetry) the yield stress σ_0 at zero pressure, $\sigma_0(0)$, is proportional to G (the shear modulus) if the process is controlled by screw dislocations, or to $G/(1 - \nu)$ where ν is the Poisson ratio if controlled by edge dislocations. In the first case

$$\sigma_0(P) = \sigma_0(0)[G(P)/G_0],$$

where G_0 is the shear modulus at zero pressure. With

$$G(P) = G_0 + G'_0(P), \text{ so that } \sigma_0(P) = \sigma_0 G_0(1 + G'_0 P/G_0).$$

Because G'_0 was in the neighborhood of 1.5 for metals and to make it easier to find an effect, Ruoff studied a metal with a much smaller G_0 . In fact, the experiment showed that the scaling factor $[G/(1 - \nu)]/[G_0(1 - \nu_0)]$ fitted the data best, showing that the motion of the edge dislocations is the controlling factor.

J.O. Chua and A.L. Ruoff. Pressure dependence of the yield stress of potassium at low homologous temperature. *J. Appl. Phys.*, **46**, 4659 (1975).

Hence

$$\sigma_0(P) = \sigma_0 F(P)$$

where $F(P)$ was called the *pressure strengthening factor* by Ruoff.

In later years, Ruoff, Christensen and Rodriguez collaborated to obtain $F(P)$ theoretically to above the 1 TPa range for *Mo* and *W*.

N.E. Christensen, A.L. Ruoff and C.O. Rodriguez. Pressure Strengthening: A way to multi-megabar pressure. *Phys. Rev. B*, **52**, 9121 (1995).

A.L. Ruoff, C.O. Rodriguez and N.E. Christensen. The elastic moduli of tungsten to 15 Mbars, phase transition at 6.5 Mbar and rheology to 6.5 Mbar. *Phys. Rev. B*, **58**, 2998 (1998).

At 550 GPa, $F(P) = 4.5$ for W . Without the pressure strengthening factor metal gaskets could not contain the huge multimegabar static pressures attained.

- *The pressure strengthening of the gasket material first suggested by Ruoff and then proven experimentally by his group has enabled multimegabar pressures to be reached and is expected to make it possible to reach pressures over 1 TPa.*

III. Pressure Dependence of the Elastic Behavior of Liquids and Solids

Ruoff has contributed substantially to the understanding of the elastic behavior of materials at high pressure. This includes elastic studies in which his group made highly accurate studies of the pressure dependence of the bulk modulus of two liquid metals and 25 solids. His group was the first to measure d^2c_{ij}/dP^2 values (on sodium) and hence to provide accurate values of the coefficients of its bulk modulus, B_0 , B'_0 and B''_0 .

P.S. Ho and A.L. Ruoff. Analysis of Ultrasonic Data and Experimental Equation of State of Sodium. *J. Phys. Chem. Solids*, **29**, 219 (1968).

These coefficients along with the Birch (winner of the Bridgman Award in 1983 for high pressure studies) equation of state provide an accurate $P(V)$ to high pressures.

- *The measured pressure derivatives of the elastic constants of many materials by Ruoff's group provided a trove of data for theorists developing algorithms to make quantum mechanical calculations accurate with finite computational capacity.*

IV. Direct Length Measurements and the Invention of a New Absolute Pressure Gauge Concept with Implementation

Direct volume measurements or length measurement also can be used to measure

$$B_0^T = -V(dP/dV)_T = -(\ell/3)(dP/d\ell)_T$$

(for a solid with an isotropic linear compressibility). Ultrasonic measurements give B_0^S (constant entropy) at zero pressure and a small measurable correction gives $B_0^T = B_0^S/(1 + \Delta)$. Ruoff realized that an absolute pressure gauge could be based on simultaneous length measurements on say a silicon crystal and the appropriate ultrasonic transit times in silicon crystals. A system was designed for this purpose. A meter long single crystal in a pressure vessel was used for length measurements accurate to $0.03\mu m$. A connected parallel pressure vessel had three crystals of silicon for transit time measurements. These pressure vessels were in a temperature bath controlled to $0.01^\circ C$ in a room controlled to $0.1^\circ C$.

R.C. Lincoln and A.L. Ruoff. Absolute Length Measurements at High Pressure. *Rev. Sci. Instrum.*, **44**, 1239 (1973).

The system was used to determine the freezing pressure of mercury of 7571.2 ± 1.6 kb. Values obtained by other groups based on the use of the “free-piston” gauge are: 7565.4 ± 3.7 , 7569.2 ± 1.2 and 7571.0 ± 1.2 .

A.L. Ruoff, R.C. Lincoln and Y.C. Chen. High pressure calibration with a new absolute-pressure gauge. *Appl. Phys. Lett.*, **22**, 310 (1973).

C-S. Zha, a former operator at CHESS and a friend of Ruoff, later used this concept 27 years later to make absolute pressure measurements to 50 GPa using Brillouin scattering and X-ray based lattice-parameter measurements at CHESS.

- *Ruoff’s invention of a totally new pressure-gauge concept (with synchrotron radiation sources as they now exist or are on the horizon) could become the penultimate way of calibrating static pressure scales in the TPa range.*

V. Development of a Purely Thermodynamic Method of Converting Hugoniot Equations of State to Isotherms

In dynamic shock experiments using explosives or gas-gun loading two quantities are measured: the shock-front velocity, u_s , and the velocity of the particle after the front has passed, u_p . These give the well-known three Hugoniot relations. It is found experimentally that u_s vs. u_p is nearly a linear relationship with a small quadratic term: $u_s = c + su_p + s'u_p^2$. Ruoff showed that the values of c , s and s' and the Hugoniot relations could be used to obtain the adiabatic coefficients: $B^S(P) = B_0^S + B_0^{S'}P + B_0^{S''}P^2/2$.

A.L. Ruoff. Linear shock-velocity particle-velocity relationship. *J. Appl. Phys.*, **38**, 4976 (1967).

Using the thermodynamic relationships of Cooke, Ruoff obtained B_0^T , $B_0^{T'}$ and $B_0^{T''}$. Morris Keeler tells the story that when he introduced Ruoff to Andre Sakarov, perhaps the greatest shock experimentalist and the inventor of the USSR atomic bomb and hydrogen bomb.

*Sakarov said to Ruoff, "so **you** discovered the thermodynamics approach to get the isotherm from the $u_s(u_p)$ relation. We are using that to get isotherms from Hugoniots."*

VI. Creation of the High Pressure Beam Line at CHESS (Cornell High Energy Synchrotron Source)

Ruoff designed and with the help of his able machinist, Volker Arnold (trained in Germany) built the first pressure stage at CHESS (available for inside and outside users). His group made major contributions to the study of EOS of solids from lattice parameter measurements as well as finding new crystal structures caused by pressure. The high quality of their diffraction studies is illustrated in the 1986 studies on *CsI* which transforms from the cubic *CsCl* or *B2* structures to tetragonal and then orthorhombic.

Y.K. Vohra, K.E. Brister, S.T. Weir, S.J. Duclos and A.L. Ruoff. *Science*. **231**, 1136 (1986). This was a generation ago.

● *Numerous X-ray diffraction studies of EOS and phase transition pressures were very useful to theorists who were developing techniques and algorithms which would make, in time, quantum mechanics a quantitative predictor of the solid state as shown by a joint paper with Marvin Cohen's group which resulted after it was learned that they had independently completed work on this problem.*

Y.K. Vohra, K.E. Brister, S. Desgreniers, A.L. Ruoff, K.J. Chang and M.L. Cohen. Phase-transition studies of Germanium to 1.25 MBars. *Phys. Rev. Lett.*, **56**, 1944 (1986).

VII. Creation of the Field of Multimegabar Research (at Static Pressures)

In 1987, the Ruoff group obtained X-ray data at 216 GPa on rhenium ($V/V_0 = 0.734$) with 12 peaks with $a = 2.491 \pm 0.003 \text{ \AA}$ and $c = 4.020 \pm 0.005 \text{ \AA}$.

Y.K. Vohra, S.J. Duclos and A.L. Ruoff. High-pressure X-ray diffraction studies on rhenium to 216 GPa (2.16 Mbars). *Phys. Rev. B*, **36**, 9790 (1987). This work was complemented by their 1988 study of rhenium to 255 GPa.

Y.K. Vohra, S.J. Duclos and A.L. Ruoff. Static Pressures of 255 GPa (2.55 Mbar) by X-ray diffraction: Comparison with extrapolation of the ruby pressure scale. *Phys. Rev. Lett.*, **61**, 574 (1988).

- In both of the above experiments and several others detailed pressure profiles were measured by Ruoff's group.

This was followed in 1990 by the comparison (from lattice parameter measurements) of the simultaneously determined P vs. V for Pb , Mo and Pt (all of which had isotherms derived from Hugoniot) to 278 GPa.

Y.K. Vohra and A.L. Ruoff. Static Compression of Metals Mo , Pb and Pt to 272 GPa: Comparison with Shock Data. *Phys. Rev. B*, **42**, 8651 (1990).

Then came the study of Si to 248 GPa in which it was shown that Si transforms to the fcc structures at 79 ± 2 GPa which persists to 248 GPa ($V/V_0 = 0.361 \pm 0.006$). Earlier predictions of the fcc phase provided a test of three different theoretical calculations (GPT, LMTO and AP) of the transition pressure and the volume.

S.J. Duclos, Y.K. Vohra and A.L. Ruoff. Experimental study of the crystal stability and equation of state of Si to 248 GPa. *Phys. Rev. B*, **41**, 12021 (1990).

Also in 1990 it was shown that the secondary absorption edge of diamond dropped from 3.7 eV at 1 atm to 3 eV at 300 GPa and 2.5 eV at 364 GPa.

Y.K. Vohra, H. Xia, H. Luo and A.L. Ruoff. Optical properties of diamond at pressures of the center of the earth. *Appl. Phys. Lett.*, **57**, 1007 (1990).

In 1990 by *miniaturization* of the flat tip diameter to $21 \mu\text{m}$ with an $8\frac{1}{2}$ degree bevel and the collimated X-ray beam diameter to $4 \mu\text{m}$, Ruoff et al., reached 378 GPa in tungsten and

416 GPa in molybdenum, easily surpassing the pressure at the center of the earth of 361 GPa. A pressure profile to 364 GPa was measured.

- Ruoff *et al.*, had surpassed Bridgman's highest pressure, 7.8 GPa (corrected) by a factor of 50.

A.L. Ruoff, H. Xia, H. Luo, and Y.K. Vohra. Miniaturization techniques for obtaining static pressures comparable to the pressure at the center of the earth: X-ray diffraction at 416 GPa. *Rev. Sci. Instrum.*, **61**, 3830 (1990).

In 1991, a pair of Type-IIa diamonds were used to generate a pressure of 338 GPa, showing that the strength of diamond depends primarily on crystal perfection.

A.L. Ruoff, H. Luo, C.A. Vanderborg and Y.K. Vohra. Generating near-earth-core pressures with type-IIa diamonds. *Appl. Phys. Lett.*, **59**, 2681 (1991).

Finally, in 1992 a static pressure of 560 GPa was obtained.

A.L. Ruoff, H. Xia and Q. Xia. The effect of a tapered aperture on X-ray diffraction from a sample with a pressure gradient: Studies on three samples with a maximum pressure of 560 GPa. *Rev. Sci. Instrum.*, **63**, 4342 (1992).

Einstein's following quote is one of Ruoff's favorites. When asked what his research philosophy was, Einstein said, "I don't saw sawdust."

In all the Ruoff group experiments discussed so far the pressure vessel used was a large, stiff pressure vessel in which the long piston and cylinder weighed 52oz. (BIG BERTHA) which was machined at the high precision machining research laboratory at NASA. When others used a tiny compliant vessel (piston and cylinder) weighing only 13 oz. (BABY BERTHA) they have only reached a pressure somewhat over 300 GPa. Ruoff notes the following analogy. In Australia they have huge semi-trailer-tractors (powered by liquid natural gas (LNG)) which pull a "train" of three trailers each with a load of 55 tons down a highway at 60⁺ MPH. Could a U.S. semi-tractor which pulls a single trailer with a 40-ton load pull the Australian train at 60⁺ miles per hour?

VIII. Pioneering Studies of the Basis for Reaching Higher Pressures

On the road to pioneering multimegabar research and with the purpose of reaching TPa pressures Ruoff has studied five areas:

1. The design of the pressure vessel.
 - *Ruoff knew that a large diameter piston and a larger thick-walled cylinder allowed machining that leaves a small gap and hence less tilt and that a longer piston cylinder contact also decreases tilt.*
2. The diamonds (how to determine diamonds of the highest perfection). What really matters (absolute perfection), Types IIa. **The presence of a dislocation in the high stress region near the tip is lethal if one is dealing with a chemical (such as hydrogen) which diffuses rapidly down dislocation pipes.**
3. Pressure strengthening of the gasket and the diamonds.
4. Optimum shape design of the diamonds.
5. Microminiaturization.

Optimum Design

The first paper related to diamond design was simple but very helpful. It lead to a triangular pressure profile, $P = P_M(1 - r/a)$ if the yield stress was constant.

K.S. Chan, T.L. Huang, T.A. Grzybowski, T.J. Whetten and A.L. Ruoff. Pressure concentrations due to plastic deformation of thin films or gaskets between anvils. *J. Appl. Phys.*, **53**, 6607 (1982).

Not bad, but not quantitative. See the measured pressure profiles in Fig. 2 of Vohra, Duchos, Brister and Ruoff, PRL **61**, 1988. Many of the papers listed in Section VII also show measured pressure profiles.

- *Ruoff initiated systematic theoretical studies of the effect of the diamond tip profile and the gasket [including finite non-linear elasticity of the diamonds and plasticity of the gasket, with the “pressure strengthening factor” noted earlier and extensive related experimental results on pressure profiles were obtained].*

H. Poon, A.L. Ruoff and S. Mukerjee. "Optimal Design of Diamond Anvils Using Finite Element Analysis and Simplified Equilibrium Equations. *Inverse Problems in Engineering*, **2**, 20 (1996).

A.L. Ruoff, H. Poon and S. Mukerjee, *Optimal Design in High Pressure Science and Technology*, [Proceedings of AIRAPT XV and EHRPG33 Conference] ed. by W. Trzeciakowski, World Scientific, Singapore (1996) p. 25.

These studies, a "Tesla approach" for attaining ultra pressure, extended what had often been an "Edisonian approach." The application of the plasticity theory in the paper with Poon and the strengthening factor from *Mo* with Christensen in Section II enabled the calculation of the final gasket thickness at $r = 0$ of $2.2\mu m$ in *Mo* at 416 GPa. Ruoff has plans to measure thickness in the metals in the future by X-ray absorption and is carrying out a full plasticity analysis with faster computers now that his book, *The Declaration of Energy Independence*, is in press.

Microminiaturization

One of Ruoff's most important discoveries related to getting ultrapressures in the diamond anvil cell is found in Fig. 3 of H. Xia, H. Luo and A.L. Ruoff. Miniaturization techniques for obtaining static pressures comparable to the pressure at the center of the earth: X-ray diffraction at 416 Gpa. *R.S.I.*, **61**, 3830 (1990).

In this case luck had fallen into Cornell's and Ruoff's laps. His group had placed indigitated metal electrodes on a diamond tip, using lithographic techniques, for two-lead resistance measurements.

D.A. Nelson, Jr. and A.L. Ruoff. *PPL* **42**, 383 (1979).

This was one of several non-electrical engineering applications which helped Cornell get the National Submersion Facility (now Cornell Nanoscale Facility). After Edward Wolf became the Director of the former, Ruoff became the Program Committee Chairman and became aware of many of the capabilities in this area. Subsequently, his group in cooperation with other groups, made some extremely important pioneering advances in this area. Originally, sample holes in gaskets were made with carbide drills and a special drill press down to about $50\mu m$ in gasket materials, *W*, *Mo*, etc., Ruoff initiated drilling them with ion beams with the help of Jon Orloff.

J. Orloff, C. Narayana and A.L. Ruoff. Use of focused ion beams for making tiny sample holes in gaskets for DAC. *Rev. Sci. Instrum.*, **II**, 216 (2000).

The initial holes were $17\mu m$ in diameter. In cooperation with former postdoctoral associate Gary Stupian, $4\mu m$ diameter sample holes have been made. It should be noted that it is expected to use these at Grenoble where the beam diameter is $1.8\mu m$.

Another impressive micro-miniaturization feat is shown in the following:

A.L. Ruoff, L. Sun, S. Natarajan, C-Z. Zha and G. Stupian. Techniques for X-ray markers at high pressure in the DAC. R.S.I., **76**, 036102-1 (2005).

Here a tiny narrow strip of $Pt \frac{1}{2}\mu m$ thick, was sputtered on the cylindrical wall of an ion-beam produced hole. Four or even six different strips could have been easily made. This can be achieved with smaller gasket holes.

● *While Janieson and others introduced miniaturizing with the diamond anvil cell, (relative to piston-cylinder devices), Ruoff took it another giant step further with the introduction of microminiaturization, which, he has calculated will lead to TPa static pressure.*

IX. Insulator to Metal Transitions in BaX, etc.

The first insulator converted to a metal in Ruoff's group in a DAC was *BaTe*.

T.A. Gryzbowski and A.L. Ruoff. Band-Overlap Metallization of *BaTe*. *Phys. Rev. Lett.*, **53**, 489 (1984).

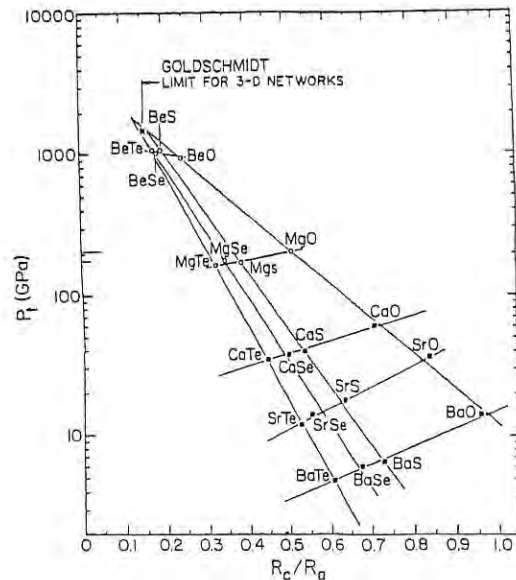
A metal is characterized in part by a low electrical resistivity. *Cu* is a very good metal with $\rho = 1.6 \times 10^{-8} \Omega$ at 300K while a less robust metal is *Zr* with $\rho = 43 \times 10^{-8} \Omega - m$. Somewhat smaller values are permitted. The next necessary condition is that ρ decreases as the temperature decreases approximately according to $\frac{1}{T}$ where T is the absolute temperature. Metals can also be characterized by their refractive index, $n + ik$, and by their absorption coefficient. The quantity k is called the extinction coefficient.

Gryzbowski measured ρ in the DAC by the 4-lead van der Pauw technique which was first used at high pressure by the Ruoff group. Resistivity studies showed band overlap at $V/V_0 = 0.65$ and $P = 20$ GPa and that $\rho(100K) = \frac{1}{3}\rho(300K)$. Unfortunately, the diamonds broke on further cooling. Optical studies showed metalization at $V/V_0 \sim 0.65$. From X-ray studies they found a phase transition at $V/V_0 = 0.907$ and $P = 4.8$ GPa from the *NaCl* (B_1) structure to the *CsCl* (B_2) structure. *BaSe* and *BaS* show the same transition at 6.0 and 6.4 GPa while *BaO* transforms first to the *NiAs* (B_8) structure (also 6-fold coordinated) and then to a near 8-fold tetragonal structure at 15 GPa, first observed by Lin-gun Lui.

S.T. Weir, Y.K. Vohra and A.L. Ruoff. High-Pressure Phase Transitions and the Equation of State of *BaS* and *BaO*. *Phys. Rev. B*, **33**, 4221 (1986).

- Ruoff's group made careful and complete metallization studies of *BaTe*, *BaSe* and *BaS* in the diamond anvil cell with the introduction four-lead van der Pauw technique into high pressure studies with complementary optical techniques.

The figure shows the pressure at which phase transitions to 8-fold coordinated structures (closed circles) and the expected pressures for transitions to 8-fold coordinated structures (open circles).



C.H.B. Zhang, Y. Mori, C. Narayana and A.L. Ruoff. *X-ray studies of BeO to 126 GPa. Science and Technology at High Pressure*, ed. by M.H. Manghnani, W. J. Nellis, and M.F. Nicol, Universities Press, Hyderabad, India (2000) p. 518.

In the *Mg* group, it was shown for the first time that the stable phase at $P = 0$ was the *NiAs* phase (also 6-fold coordinated).

T. Li, H. Lui, R.G. Greene, A.L. Ruoff, S. Trail and F.J. Di Salvo, Jr. High Pressure Phase of *MgTe*: Stable Phase at *STP*? *Phys. Rev. Lett.*, **74**, 5232 (1995).

MgSe has a *B1* to *B8* transition at low pressure (1-3 GPa) and persists in the *NiAs* phase to 99 GPa where it begins a prolonged continuous transformation by internal atom motions ending at about 202 GPa to the 7-fold coordinated *B28* structure (also called the iron silicide structure). There was no noticeable volume change from 99 to 202 GPa, a characteristic of second-order phase transitions; theoretical calculations showed a tiny change.

A.L. Ruoff, T. Li, A.C. Ho, M-F. Pai, H. Luo, R. Greene, C., Narayana, J.C. Molstad, S. Trail and F.J. Di Salvo, Jr. *Phys. Rev. Lett.*, **81**, 2723 (1998).

- *This was the first determination of internal atom positions above 100 GPa.* It was shown that the isoelectronic set of *BeTe*, *CsI* and *Xe* follow the Goldhammer and Herzfeld dielectric catastrophe model as do the chalcogenides *BaTe*, *BaSe* and *BaTe*.
- *Ruoff discovered that, for the alkaline chalcogenides, the alkali metals and the rare gas solids (all closed shell systems), $n^2 E_g(\text{Ryd}) = \pi/2$, where n is the refractive index and E_g is the energy band gap.*

A.L. Ruoff. Empirical Relation Between the Energy Band Gap and the Refractive Index in Closed Shell Systems, *Mat. Res. Soc. Symposium Proc.*, **22**, Part 1, 279 (1984).

Ruoff's group found the alkaline chalcogenides to be a happy hunting grounds, inspired by Pauling, J.C. Phillips, Goldschmidt and Sheraga.

X. Metalization of Molecular Solids

Following their metalization of *BaTe*, *BaSe* and *BeS*, Ruoff's group began the hunt that led to the metalization of oxygen (95 GPa) and sulfur (95 GPa) at 300K.

S. Desgreniers, Y.K. Vohra and A.L. Ruoff. Optical Response of Very High Density Solid Oxygen to 132 GPa. *J. Phys. Chem.*, **94**, 1117 (1990).

A.L. Ruoff and S. Desgreniers. *Very High Density Solid Oxygen: Indications of a Metallic State, In Molecular Systems Under High Pressures*, ed. by R. Pucci and G. Piccitto, Elsevier Science. North Holland, **123**, (1991).

H. Luo, S. Desgreniers, Y.K. Vohra and A.L. Ruoff. High Pressure Optical Studies on Sulfur to 121 GPa: Optical Evidence for Metallization. *Phys. Rev. Lett.*, **67**, 2998 (1991).

H. Luo, S. Desgreniers, Y.K. Vohra and A.L. Ruoff. Optical Absorption and Reflection Studies on Sulfur: Evidence for Metallization. *Recent Trends in High Pressure Research*, ed. by A.K. Singh (1991), Oxford and IBH Pub. Co., New Delhi (1992), p. 374.

It should be noted that in order to load these gases into the pressure cell, the piston-cylinder was placed inside a large steel cylinder in which the gas pressure was raised to 0.2 GPa. For the sake of convenience, the small cylinder-piston noted earlier (BABY BERTHA) used was satisfactory for these lower pressure studies but would not suffice to get, say, 600 GPa with current techniques.

• *These elegant experimental results proving the metallization of oxygen and sulfur rigorously passed the test of time.*

Eight years later, Shimizu et al., *Nature*, **390**, 3767 (1998) confirmed the metalization of oxygen and showed that at 93 GPa, oxygen became a superconductor with $T_c = 0.6K$.

After the metalization of sulfur by Ruoff's group Prof. Marvin Cohen's group did theoretical calculations and predicted that sulfur would be a superconductor.

The result of both groups was subsequently confirmed six years after the initial metalization by Struzkin et al., *Nature*, **360**, 382 (1997), when they showed that at 95 GPa and 10K, sulfur became a superconductor, and that T_c increased to 15K as P increased to 160 GPa, and then jumped to 17K and then T_c decreased as P increased. It should be noted that previous X-ray studies on *S* to 212 GPa had found a transition to the $\beta - Po$ structure at 162 GPa.

H. Luo, R.G. Greene and A.L. Ruoff. *B – Po* Phase of Sulfur at 162 GPa: X-ray diffraction studies on *S* to 212 GPa. *Phys. Rev. Lett.*, **71**, 2943 (1993).

Huan Luo won the Jamieson Award given by AIRAPT in 1993 for his studies of sulfur.

Ruoff's group also showed in a cooperative effort that xenon became a transparent conductor at 150 GPa.

R. Reichlin, K.E. Brister, A.K. McMahan, M. Rose, S. Martin, Y.K. Vohra and A.L. Ruoff. Evidence for the Insulator-Metal Transition in Xenon from Optical, X-ray and Band Structure Studies to 170 GPa. *Phys. Rev. Lett.*, **62**, 669 (1989).

This paper was accompanied by a similar success by Silvera's group on xenon published in the same issue (instant confirmation).

XI. Attempted Metallization of CsH

As a result of a discussion between Ruoff and Baranowski about CsH . Ruoff looked further into CsH . He noted that Hockheimer et al., (1985) had shown that CsH has a bulk modulus at 300K of 7.6 GPa while Mao et al., had determined that the room temperature freezing point of hydrogen was 5.4 GPa, and that its bulk modulus there was 15.6 GPa. [For comparison, lithium at 300K has $B_o = 12$ GPa].

Studies on CsH led by Ruoff's graduate student, Kouros Ghandehari resulted in three publications involving Ruoff's group and DiSalvo's group.

K. Ghandehari, H. Luo, A.L. Ruoff, S. Trail and F.J. DiSalvo, Jr. New High Pressure Crystal Structure and Equation of State of Cesium Hydride to 253 GPa. *Phys. Rev. Lett.*, **74**, 2264 (1995).

A.L. Ruoff, K. Ghandehari, H. Luo, S. Trail and F.J. DiSalvo, Jr. Phonon Reflectivity Behaviour in CsH and CsD at Megabar Pressures: Phonons in the Near IR ($\lambda = 1 - 2\mu m$). *Solid State Comm.*, **100**, 777 (1996).

K. Ghandehari, H. Luo, A.L. Ruoff, S. Trail and F.J. DiSalvo, Jr. Band Gap and Index of Refraction of CsH to 251 GPa. *Solid State Comm.*, **95**, 385 (1995).

While highly compliant at $P = 0$ ($B_0 \sim 8$ GPa), CsH , when squeezed to 253 GPa, was found to have $B(251 \text{ GPa}) = 881$ GPa, over twice the value of diamond at $P = 0$. The structure at $P = 0$ is the $NaCl(B1)$ type which transforms to the $CsCl$ type at 37 GPa and then at 17.5 GPa ($V/V_0 = 0.53$) to the orthorhombic CrB structure (space group $Cmcm$). The $Cmcm$ space group is predicted to be a product space group of the parent $CsCl$ structure involving a displacive phase transitions with X_5^+ or X^- mode softening. Nineteen peaks were indexed with excellent positional fits to the CrB structure and very good intensity fits. At 253 GPa and 300K $V/V_0 = 0.26$ and as noted it is extremely stiff elastically. Theoretical studies of CsH confirmed the crystal structure.

R. Ahiyu, O. Erickson, J.M. Wills and B. Johansson. Theoretical high-pressure studies of caesium hydride. *Journal of Physics: Condensed Matter*, **10**, L153 doc:10.1088/0953-8994.

The energy band gap at 251 GPa was 1.9 eV and the refractive index was 3.2 with $k = 0$; hence $n^2 Eg = 19.2 \text{ eV}$, compared to the expected $n^2 Eg(Ryd) = \pi/2$ or $n^2 Eg(eV) = 21.4$, by Ruoff's relation noted earlier. This suggests that at 250 GPa CsH is an ionic solid.

- *CsI showed interesting similarities to H_2 : (1) The energy band gap was 1.9eV at 250 GPa for CsH which is the same as for hydrogen (shown later) at 342 GPa. (2) The calculated volume of solid hydrogen at the freezing point at 300K shows the same factor of four reduction as does CsH at 300K and $P = 0$ when the pressure is increased to 250 GPa.*
- *The rising reflectivity in the $i - r$ (second paper) was shown by isotopic-studies to be owing to phonons and not to a rising band edge.*

XII. Why a Sequence of High Pressure Studies on a Solid Sample is often not a Static High Pressure Experiment

Researchers say “static high pressure measurements” because that may be a good approximation in some cases after a sufficient time is allowed after the pressure is changed.

However, during the pressure change in the DAC, plastic deformation (of both the GASKET and the SOLID SAMPLE) is occurring, most dislocations are moving, and there is a net increase of dislocations and vigorous production of point defects. This leads to greatly enhanced diffusion rates as clearly shown by Balluffi and Ruoff.

R.W. Balluffi and A.L. Ruoff. Enhanced Diffusion in Metals during Plastic Deformation. *Appl. Phys. Lett.*, **1**, 59 (1962).

R.W. Balluffi and A.L. Ruoff. On Strain-Enhanced Diffusion in Metals. I. Point Defect Models. *J. Appl. Phys.*, **34**, 1634 (1963).

A.L. Ruoff and R.W. Balluffi. Strain-Enhanced Diffusion in Metals. II. Dislocation and Grain-Boundary Short-Circuiting Models. *J. Appl. Phys.*, **34**, 1848 (1963).

A.L. Ruoff and R.W. Balluffi. On Strain-Enhanced Diffusion in Metals. III. Interpretation of Recent Experiments. *J. Appl. Phys.*, **34**, 2862 (1963).

A.L. Ruoff. Diffusion During Deformation Measured by Surface Intensity Methods. *J. Appl. Phys.*, **36**, 2207 (1965).

Ruoff had spent his first Sabbatical leave at University of Illinois. It was the year after Ruoff had taught the first dislocation course at Cornell (which inspired Prof. H.D. Block to write the poem “A Crystal Lament.” See

A.L. Ruoff. *Materials Science*, Prentice Hall, Englewood Cliffs, NJ (1973) p. 686.

Diffusion occurs very much faster in grain boundaries and down dislocations (pipe diffusion) than in the bulk. Even if the dislocations are static, they can readily fill with an impurity and with the very high density present in heavily plastically strained materials become colored and blackened by the solute in the dislocations. Now imagine a dislocation filled with impurities. The impurity atoms in the dislocation will slowly diffuse outward into the bulk. However, if stress moves the dislocation during a pressure change, the debris (solute)

remains behind and the dislocation then readily accumulates more solute. The repetition of this process leads to enhanced “bulk” diffusion.

- *Ruoff notes: the process of diffusion enhancement by plastic deformation during pressure changes can wreak havoc in the study of hydrogen, if metal electrodes or gaskets which are nascent hydrogen producers are present.*

Ruoff published a warning about this in his 342 GPa paper on hydrogen in 1998 (to be discussed later).

XIII. Hydrogen at Ultrapressures is a Voracious Scavenger

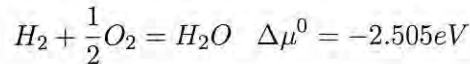
In 1988 Mao et al. published room temperature $P(V)$ data for H_2 from the freezing pressure (5.4 GPa, where $V = 8.291\text{cm}^3/\text{mole}$) to 26.5 GPa where $V = 4.791\text{cm}^3/\text{mole}$. Ruoff computed the chemical potential of H_2 vs. pressure. At $STP_0 \Delta\mu^0 = 0$ there is good $P - V$ data available at 300K to the freezing point at 5.4 GPa where $V_{H_2} = 8.29\text{cm}^3/\text{mole}$. Loubeyre et al. Nature (1996) obtained the EOS to 109 GPa to 300K. Their Vinet equation was used to calculate the quantities in Table 1.

Table 1: Hydrogen Variables vs. Pressure

$P(\text{GPa})$	$V_{H_2}(\text{cm}^3/\text{mole})$	$\Delta\mu(\text{eV})$	$B(\text{GPa})$
5.4	8.29	0.85	18.5
10	6.70	1.20	32
20	5.28	2.16	60
60	3.50	2.93	140
100	2.92	5.18	250
200	~ 2.08	~ 7.9	~ 480
250	~ 1.88	~ 9.1	~ 590
300	~ 1.74	~ 10.2	~ 720
350	~ 1.60	~ 11.1	~ 760
600	~ 1.23	~ 15.4	~ 1273

Note: the estimated ratio $V(250)/V(5.4)$ is 3.8 for H_2 and the measured ratio is 3.9 for CsH discussed in Section XI.

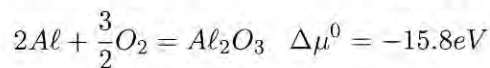
Consider:



These two reactants can be comingled without reaction in a metastable state. A tiny spark causes an explosion.

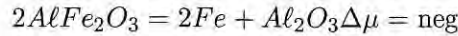
- H_2 , at a high pressure is (in a thermodynamic sense) a voracious scavenger.

Another metastable state is aluminum on the wings of airplanes:



A thin layer of coherent oxide on the surface prevents further oxide formation.

However, the famous thermite reaction



used to bring down old, high buildings (straight down) works because the enormous heat given off by the burning of the aluminum melts the steel beams to which the thermite is attached. H_2 as a reactant at high pressure also often causes a similar negative $\Delta\mu$ for a reaction product.

If, e.g., there is a metal which, perhaps by raising the pressure in the presence of H_2 , has $\Delta\mu$ become negative, (the reaction $M + \frac{1}{2}H_2$ has $\Delta\mu$ negative) it may remain in a metal stable state because the activation energy, $\Delta\mu^*$, is too large and the rate of reaction, being proportional to $e^{-\Delta\mu^*/kT}$ and is too slow. The kinetics need a spark.

This can be accomplished with a catalyst which absorbs H_2 and releases H atoms. Such nascent hydrogen formers are widely used in the petroleum industry and the chemical industry. They include Re, Ag, Cu, Au , etc. For the reaction shown, the activation energy, $\Delta\mu^*$, is reduced by $2.2eV$ and the reaction readily proceeds at $300K$.

Inasmuch as “like dissolves like” metal atoms which form hydrides are likely to be soluble in hydrogen. Moreover, because of the presence of dislocations and pipe diffusion and the enhanced diffusion owing to plastic deformation during pressure change, a solution of metal in hydrogen can readily form.

XIV. Studying Pure Hydrogen is Difficult Because It Has to be Kept Pure

If nascent hydrogen formers are present in the cell, it is extremely unlikely that the hydrogen itself will remain pure. It is likely that metal hydrides will form, and it is likely that metals will dissolve in the hydrogen.

It is likely that “black hydrogen” will be “observed” but what is being observed is a solution in a hydrogen solvent or suspended MH particles in a solution, not hydrogen.

Ruoff was intrigued by studies on metal hydrides and carefully followed the work of the Ponyatovsky group and the studies of Baranowski and Thacz.

He had highlighted in his notebook in 1984 the following comment:

“To date hydrides of all transition metals have been synthesized except those of the remaining four platinum metals and **TUNGSTEN**.”

V.E. Antonov, I.T. Belash, V. Val Malyshev and E.G. Ponyatovsky. The solubility of Hydrogen in Platinum Metals under Pressure. *Platinum Metals Rev.*, **28**, 158–163 (1984).

This is why Ruoff used costly ultrapure gaskets of *W* which were checked by surface analysis for nascent hydrogen formers by three different analytical laboratories who found none.

Ruoff notes that along with the bulk *W* and the bulk H_2 there is another phase, the interfacial phase and it is entirely possible that an impurity such as *Re* in low concentration in the bulk *W* gasket, may be at a high concentration in the interface, and may even be a monolayer.

Ruoff points to the example in which his son Rodney produced graphene, by use of carbon dissolved in copper, with partitioning on the surface.

● *All data obtained on H_2 at high pressure with nascent hydrogen formers present are likely to be data on solutions and/or MH suspended in solution.*

A.L. Ruoff. “The difficulty of studying pure hydrogen at ultrapressure, in preparation.”

XV. Faulty Claims of Metallic Hydrogen

Two early claims not using diamond cells faded away because:

1. The necessary pressures could not possibly have been reached. It is doubtful that 100 GPa was reached.
2. The electrical resistance drops observed were most assuredly shorts.

The third claim that H_2 became metallic in the hydrogen cell at 150 GPa based only on reflectivity, ignoring absorption data, was published with great news and excitement and was retracted in a few lines in another paper several years later.

The fourth claim was made in 2011 by Eremets and Troyan who earlier had led the group who claimed to have made superconducting SiH_4 at 50 GPa. The “superconducting SiH_4 ” turned out to be platinum hydride as shown by

O. Degtyareva et al. *Solid State Commun.*, **149**, 1583 (2009).

This fourth claim of making metallic H_2 at 275 GPa at 300K in 2011 did not give a single measured property of a material. There were severe impurity problems and electrical shorts (see Figure 6 of the reference to Poon et al. in Section VIII). Their resistance dependence as a function of temperature is **TOTALLY CONTRARY** to that required of a metal. Their “black hydrogen,” i.e., polluted hydrogen occurred at a substantially lower pressure than observed by other finders of “black hydrogen.” That is a necessary and sufficient condition which disproves metallization. Einstein wrote, “A thousand experiments which agree with my theory do not prove it is right, while one experiment can prove me wrong.” Repeating a technique, proved wrong in one case, is likely to be courting trouble.

XVI. Two Results Which Showed No Signs of Metallization: one at 290 GPa and one at 342 GPa

Before 2012 there were two published papers on H_2 at 300K which showed no “black hydrogen,” one at 290 GPa and one at 342 GPa. Both used ultrapure W as gaskets as noted earlier.

In the first Raman measurements were made at 34 pressures to 273 GPa and X-ray pressure measurements were made to 290 GPa and a micrograph was taken which showed only transmitted yellow light through the well-centered round sample but then a fracture occurred. A metal sample would not transmit light.

A.L. Ruoff. *Hydrogen at Multimegabar Pressures in High pressure Science and Technology*. Ed. by W. Trzeciakowski, World Scientific. Singapore (1996), p. 25

The second experiment showed no black sample but showed yellow (nearer to orange) light transmitted and a Raman peak at 342 GPa. The band gap is near 1.9eV.

C. Narayana, H. Luo, J. Orloff and A.L. Ruoff. Solid Hydrogen at 342 GPa: No Evidence for a Alkali Metal. *Letters to Nature*, 393, 46 (1998).

Ruoff notes that tungsten hydride has since been produced by others, occurring in the presence of free H atoms.

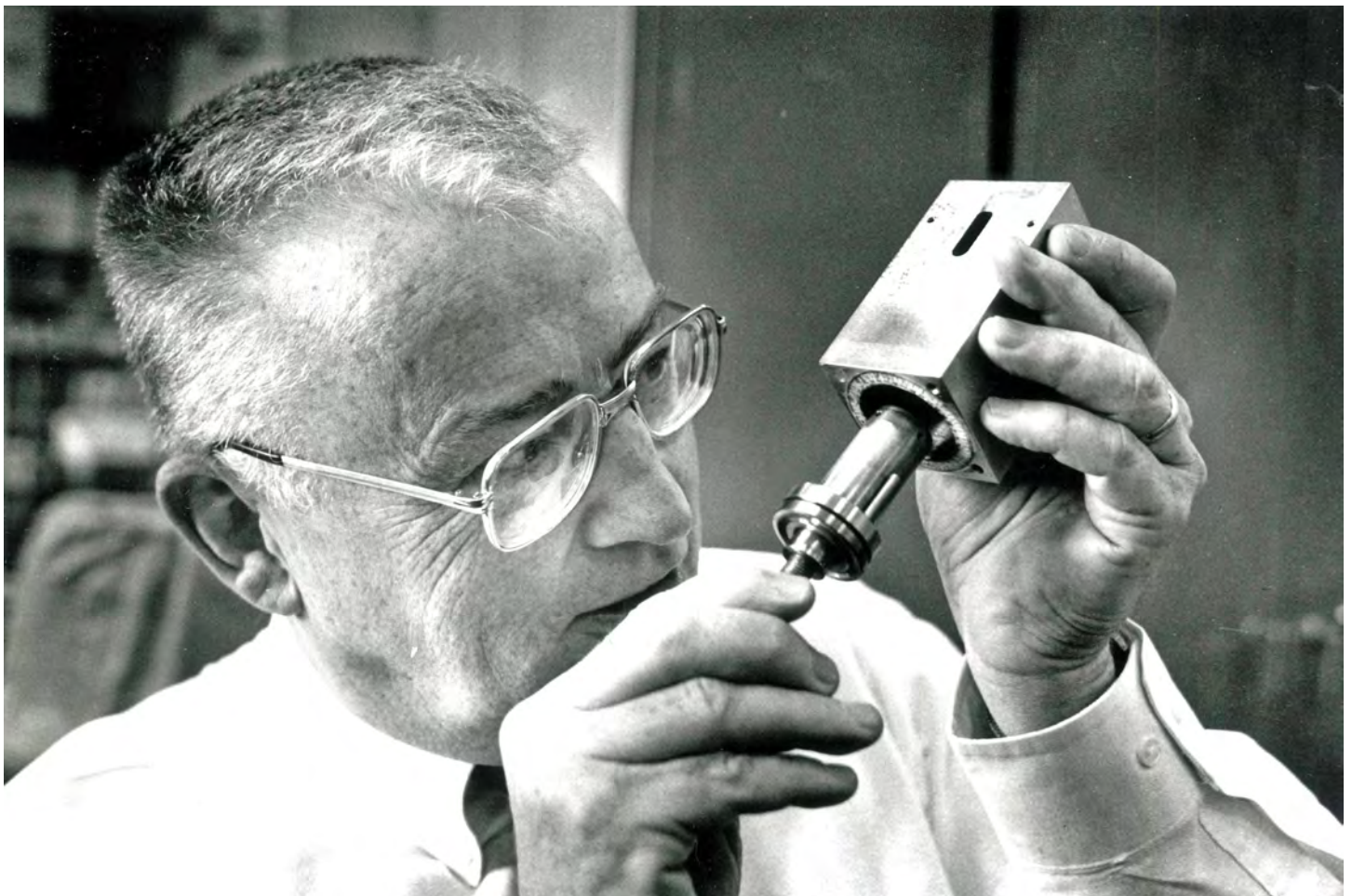
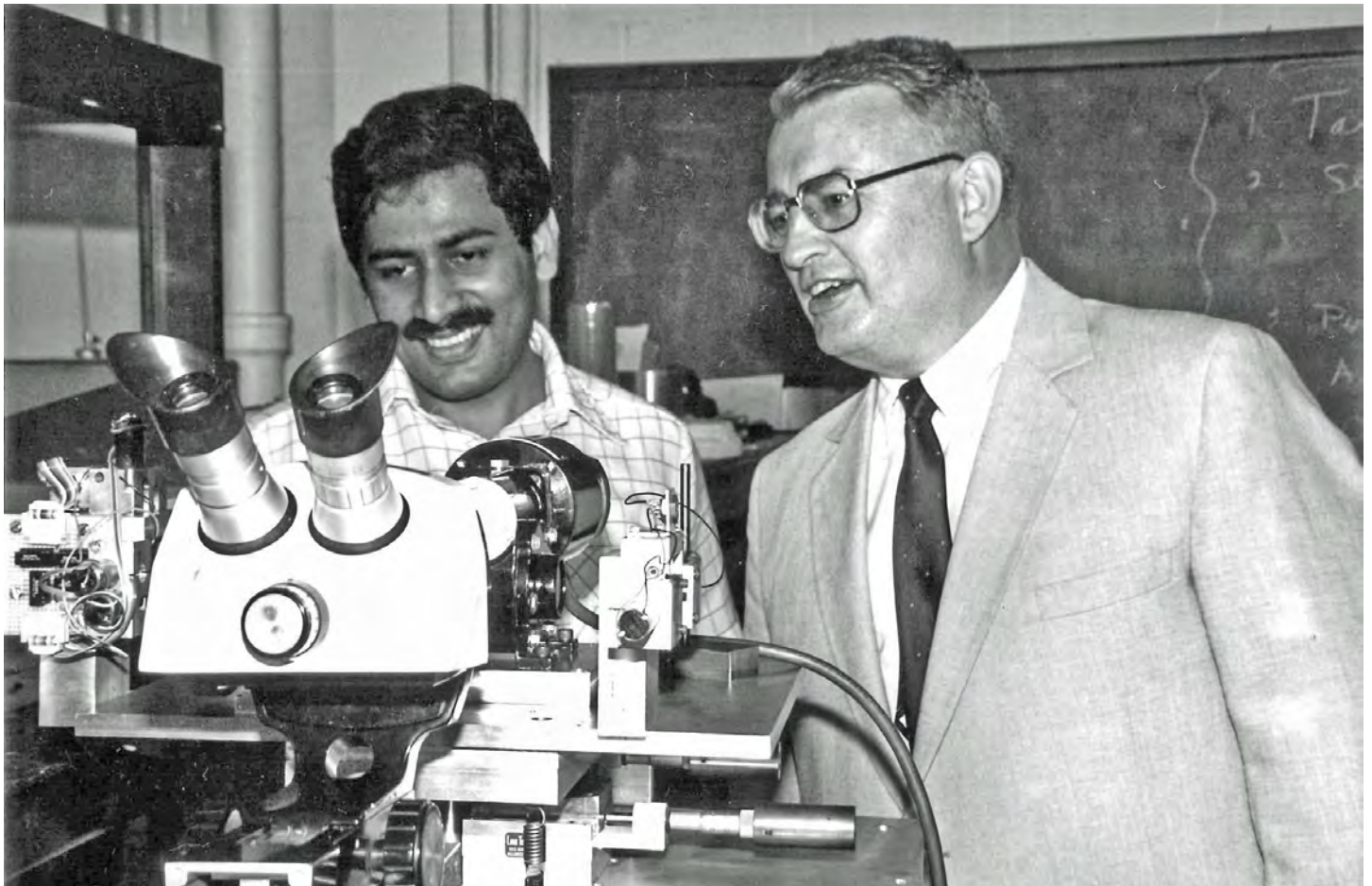
It was first produced by Indian scientists using a **rhenium** gasket with powdered W in the sample hole with H_2 . It was then produced by Akahama et al., in the same way.

It was next produced by Strobel et al. using a W gasket from a mixture of SiH_4 and/or H_2 plus $(SiH_4)(H_2)_2$ probably with some decomposition at modest pressures producing SiH_{4-m} and mH .

Recently M. Hanfland, J.E. Proctor, C.I. Guillaume, O. Degtareva and E. Gregoryanz wrote: “High Pressure studies on silane (SiH_4) revealed that it does not metallize at 50 GPa, but instead goes through a pressure induced amorphization above 60 GPa recrystallizing into a polymeric phase at around 90 GPa. Silane remains insulating up to at least 130 GPa.”

- *The conclusion of Ruoff’s group: H_2 is still a large band gap semiconductor with a gap of about 1.9eV at 342 GPa.*

Addendum: Another study (14 years later) by Zha et al., PRL, 2012 confirms this conclusion to 360 GPa.







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Arthur Ruoff in Engineering: Cornell Quarterly

MEGABAR PRESSURES IN SUBMICRON VOLUMES

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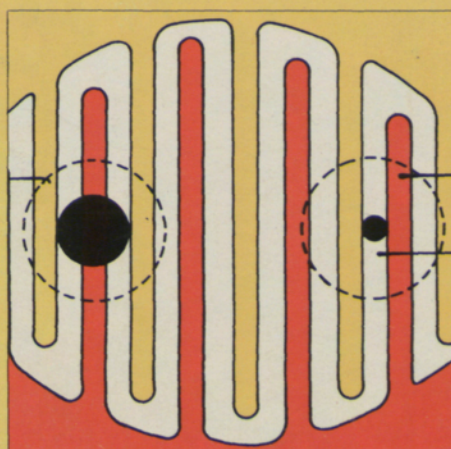
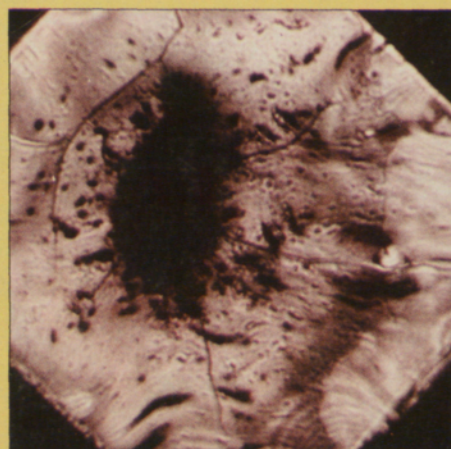
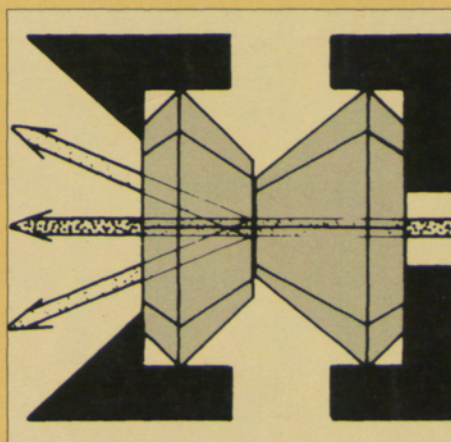
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Engineering Quarterly 1991_v25_n4 p37

ENGINEERING

CORNELL QUARTERLY



VOLUME 14
NUMBER 1
SUMMER 1979

**MATERIALS
AT ULTRAHIGH
PRESSURES**

MEGABAR PRESSURES IN SUBMICRON VOLUMES

by Arthur L. Ruoff

The pressure in an ordinary automobile tire is about 3 bars, approximately three times atmospheric pressure. City water pressure may be 4 to 5 bars. The pressure at the deepest spot in the ocean is about 1,000 bars or 1 kilobar (kbar). Many commercial processes are carried on at pressures of 1 to 5 kbars; diamonds are synthesized at about 50 kbars. The pressure at the center of the Earth is about 3.7 million bars or megabars (Mbars). It is at pressures in the megabar range that the work of my research group is focused.

Under these extreme pressures, materials have properties very different from those they exhibit under ordinary conditions. If the material is a liquid, there can be an enormous increase in viscosity and a drastic change in the freezing point. A solid sometimes assumes a new, more condensed structure. New chemical compounds may form, or existing compounds may break up into their elements. Nonconductors become metals, and often these new metals are superconductors at low temperatures.

The industrial potential of this modern-day alchemy is enormous. For

example, if pressure-induced superconductors prove to be metastable—if they can be kept in metallic form after the pressure is released—they could be used in many electrical and electronic applications. But current research is directed primarily to basic scientific studies and to the development of necessary techniques. Significant progress has been made in attaining ultrahigh pressures and in producing and examining new forms of materials. Our group at Cornell has had some exciting successes: we have made metallic xenon, for example, and we have converted sulfur, one of the best electrical insulators, to a metal. A new approach is to make use of unique new submicron fabrication and x-ray facilities at Cornell to create and examine materials in new forms.

HIGH-PRESSURE EFFECTS ON PHASE AND STRUCTURE

What happens to materials as a result of pressure? Essentially, the atoms are pushed closer together, drastically altering the macroscopic properties as well as the molecular structure.

The magnitude of viscosity change, for example, is difficult to conceive. A mixture of three parts methanol and one part ethanol increases in viscosity by a factor of 10 trillion (10^{13}) when a pressure of 100 kbars is applied. Under pressure, materials freeze at higher temperatures than they do under atmospheric conditions. Liquid hydrogen, for instance, freezes at 16°K at atmospheric pressure, but at 56 kbars, the freezing point is 300°K, about room temperature. A solid may change to a new structural form with a higher coordination number (the number of nearest-neighbor atoms or ions). In sodium chloride, for example, each sodium ion has six chlorine ions surrounding it at atmospheric pressure, but above a few hundred kbars each sodium is surrounded by eight chlorines. Materials scientists say that the compound has undergone a phase transformation from the sodium-chloride-type structure to the cesium-chloride-type structure.

The transformation of graphite to diamond is another example of a pressure-induced increase in coordina-

“The industrial potential of this modern-day alchemy is enormous.”

tion number. In graphite, which is the equilibrium structure of carbon at atmospheric pressure, each atom has three nearest neighbors. Above about 14 kbars at room temperature, diamond is the equilibrium structure, and each carbon has four nearest neighbors. This may seem puzzling, for we know that both graphite and diamond exist at atmospheric pressure. The explanation is that diamond is metastable under ordinary conditions. A thermodynamicist would say that graphite has a lower free energy than diamond at room temperature and pressure; the carbon atoms in diamond would “like” to arrange themselves into the graphite structure and thereby lower their free energy. The rate of this rearrangement is slow, however: one need not worry about a diamond gem reverting to graphite. But if the temperature were raised substantially, say to 1,700°C, this reversion would occur rapidly.

The transformation of graphite to diamond at high pressure is the basis of a large industry: synthetic diamond production. In fact, most industrial

ally. They are used for such diverse operations as grinding hard materials such as the turbine blades in a jet engine, cutting grooves in highways, scratching designs on Steuben glass, slicing silicon crystals into wafers, and drilling oil wells. In the United States, the General Electric Company has a major synthetic diamond plant in Columbus, Ohio. There are other synthetic diamond plants in France, Ireland, Japan, Russia, South Africa, and Sweden.

Silicon, both as an element and in compounds, also undergoes an increase in coordination number under high pressure. Normally silicon exists in silicate structures, as part of the $(\text{SiO}_4)^{4-}$ radical and has four nearest-neighbor oxygen atoms, but at a few hundred kbars, six-fold coordination occurs; this process is of enormous importance in geology. Elemental silicon shows an increase in coordination number from four to approximately eight at about 120 kbars; in the process, it changes from a semiconductor to a metal. Diamond, which has the same crystal structure as silicon, will probably transform to a metal at about 2 Mbars.

COMPOUND FORMATION AND DECOMPOSITION

Not only changes in crystalline structure, but also the formation of crystalline compounds can be accomplished under high pressure. Cubic boron nitride, for example, is a metastable material commercially made by a process similar to that used for diamond: ordinary BN is dissolved in liquid metal, pressure-treated, and then precipitated as the cubic form. But research now underway is showing how the cubic form can be produced at much lower pressures and in larger quantities by growing it from a solution containing boron and nitrogen compounds.

The opposite process—the decomposition of a compound into its elements—can also occur under high pressure. This happens with C_3S_2 , a compound we have worked with in our laboratory. Our hope was that the carbon produced at high pressure and room temperature would be in the form of diamond. It wasn't; but had this process worked, we might have synthesized diamond at 15 kbars instead of the 50 kbars used in commercial practice.

A variation of decomposition under pressure is a reaction in which the original compound forms a different compound plus an element. Recently there has been much excitement about results suggesting that cuprous chloride exhibits unusual magnetic behavior (characteristic of superconductors) when it is subjected to high pressures. There is some evidence that the reaction taking place is $2\text{CuCl} \rightarrow \text{CuCl}_2 + \text{Cu}$.

TRANSFORMING INSULATORS INTO CONDUCTING METALS

At sufficiently high pressures, all semiconductors and insulators are expected to be metals (pure semiconductors at low enough temperatures are also insulators). For example, zinc sulfide, an excellent insulator, becomes metallic at about 150 kbars. The semiconducting III-V compound gallium phosphide becomes metallic at about 200 kbars. Mike Chan, a graduate student in my group, is looking at aluminum nitride; there is a theoretical prediction that it will transform to a metallic phase at 0.9 Mbar. An indication of the relative pressures needed for metallic transformation is given by the position of the elements in the periodic chart (see Table I).

In our laboratory at Cornell, we have conducted high-pressure experiments with several of these nonmetallic elements. We have cooled iodine metal (at pressure) to 1°K with no sign of superconductivity. Currently Millard Baublitz, a student in my group, is attempting to study it at temperatures down to 0.3°K, but perhaps even that will not be low enough to produce superconductivity. In other work, we showed that sulfur, which is ordinarily an extremely good insulator, becomes

Table I METALLIZATION OF NONMETALLIC ELEMENTS					
Groups					
IIIb	IVb	Vb	VIb	VIIb	VIII
				H	He
B	C	N	O	F	Ne
	Si	P	S	Cl	Ar
	Ge	As	Se	Br	Kr
			Te	I	Xe
					Rn

There are two general rules about the pressure needed to transform the non-metallic elements to metallic conductors: (1) in a given row of the periodic table, the required pressure increases from left to right; and (2) in a given column the transition pressure increases from bottom to top. Thus helium will require the most pressure. None of the first- or second-row elements have been made metallic; boron should transform at a modest pressure of a few hundred kbars, although the kinetics of the transition may make high temperatures necessary. The elements Si, Ge, P, As, Se, Te, and I all become metallic below 200 kbars, and all of these metals except iodine have been found to be superconductors at high pressure.

metallic at room temperature at 300 kbars. And we found that solid xenon became metallic when pressurized to 320 kbars at 32°K. (Of the inert gas solids, radon should transform at the lowest pressure, but because it is radioactive, we chose not to work with it.)

We think that oxygen and hydrogen are exciting possibilities for future research. A metal transition in oxygen should require a pressure of about 1 Mbar. For hydrogen, a pressure variously estimated at 1.6 to 7 Mbars might be needed.

ATTAINING HIGH PRESSURES IN THE LABORATORY

There are two general rules for obtaining high pressures: (1) use the hardest substance known for the apparatus; and (2) create the pressure in the smallest volume possible. These criteria determine our laboratory technique.

Diamond is the stiffest and hardest material on Earth, with the highest yield strength. It has the same crystal-line structure as silicon and germanium,

but is about ten times stiffer and stronger. All three of these materials are brittle—tiny scratches on their surfaces will greatly decrease their fracture strength—and all three plastically deform if fracture is prevented. The bulk compressive strength of diamond is about 350 kbars, which is very high; the next strongest common material, sintered tungsten carbide, has a yield strength of 52 kbars (as determined by Prakash Panda, a graduate student, and me), and ultra-strength steels have yield strengths in the region of only 20 kbars. Anvils made of diamond would begin to yield at 500 kbars and could sustain a maximum pressure, when heavily deformed, of about 1 Mbar.

If truly perfect diamonds were available, much higher pressures could be attained. It has been predicted that the yield strength of perfect diamond would be 4.1 Mbars. But single crystals of diamond, regardless of their sparkle and clarity, are neither pure carbon nor perfect crystals; they contain about 1 percent of impurities, either in solution or as tiny precipitates, and they exhibit crystalline irregularities amounting to about 10^5 centimeters of dislocation line per cubic centimeter of material. (Dislocations are significant because plastic deformation occurs by motion of these line defects.) Large dislocation-free crystals of silicon can be grown, and it is not inconceivable that this could be accomplished with diamond. But at the present time, the best alternative is to use a diamond of very small size; if small enough, it will be free of dislocations. Such a region must be about 10 microns (μm) in each dimension ($1 \mu\text{m} = 10^{-6}\text{m} = 10^{-4}\text{cm}$).

Accordingly, the technique we use to obtain very high pressures is to press

Figure 1

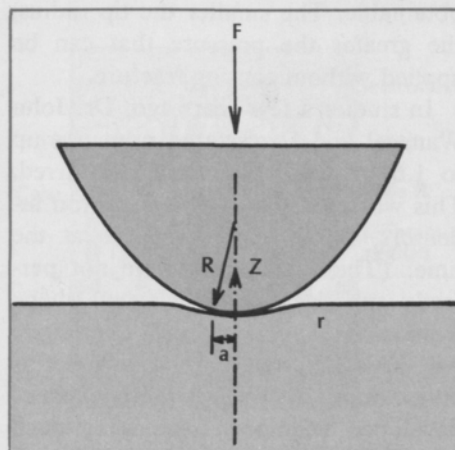


Figure 1. A schematic of the spherical indenter-flat anvil pressure-generating system. A force F is applied on the indenter, which has a spherical tip of radius R . Pressure is exerted over a contact area of radius a , with a maximum value at the center. The smaller the tip, the larger the pressure attained: with tip radii in the micron range, megabar pressures can be achieved at the center.

Figure 2. The pressure distribution of the system shown in Figure 1. The maximum pressure, P_0 , is produced at the center of the area of contact, where $r=0$ and $z=0$. The area of contact has a radius a .

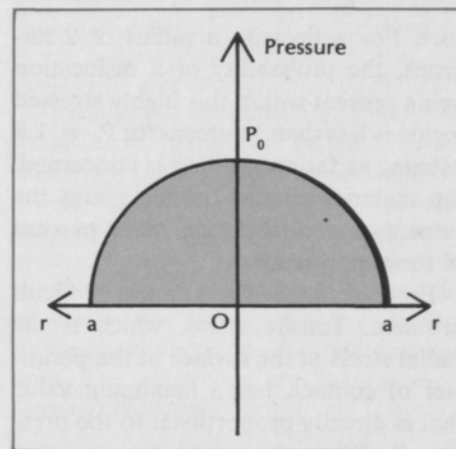


Figure 2

a tiny diamond with a spherical tip—the indenter—against a flat diamond, as shown in Figure 1. This results in a contact pressure distribution, as shown in Figure 2, with a maximum, P_0 , that can be calculated from elasticity theory. The equation we use was formulated by Hertz:

$$P_0 = (3/2)^{1/3} \pi^{-1} (E/1-\nu)^{2/3} R^{-2/3} F^{1/3}$$

where E is Young's modulus, ν is Poisson's ratio, R is the initial tip radius, and F is the applied force. This shows that the maximum pressure, P_0 , is pro-

portional to the applied force and inversely proportional to the radius of the indenter tip.

To understand the eventual failure of the diamond, we must consider the shear stress, which causes yielding by dislocation motion, and also the tensile stress, which causes fracture.

The shear stress is a maximum along the axis of revolution at a distance of about $a/2$ from the contact point (see Figure 2), and falls to about a quarter of this value on a spherical surface of radius a centered at the point of con-

tact. For a tip with a radius of 2 microns, the probability of a dislocation being present within this highly stressed region is less than 1 percent for $P_0 = 1.8$ Mbars: as far as yielding is concerned, the material should behave about the same as a perfect crystal in 99 percent of the experiments.

Fracture presents a more difficult problem. Tensile stress, which is the radial stress at the surface at the perimeter of contact, has a maximum value that is directly proportional to the pressure P_0 . When the tensile stress reaches a certain critical value—the fracture strength—a ring crack forms. (The actual shape of the ring crack is determined by the way in which the cleavage planes intersect the surface.) This limits the maximum value of P_0 . Happily, there is an important size effect that helps out, however. It was discovered by Auerbach in 1890 that when a steel bearing is pressed against glass, the fracture strength of the glass increases as the tip radius decreases; we have found that this law applies to diamond over a wide range of tip radii, from 2,000 microns down to the smallest

obtainable. The smaller the tip radius, the greater the pressure that can be applied without causing fracture.

In studies a few years ago, Dr. John Wanagel and I registered pressures up to 1.6 Mbars before failure occurred. This work was done with 2-micron indentors, the smallest available at the time. (These indentors were not perfectly spherical; if their actual shape were taken into account, the pressure we achieved would be calculated as larger than 1.6 Mbars.) We have since developed techniques for making much better tips, with radii as small as a tenth of a micron. Dr. Dan Golopentia and I hope to report soon on the results of tests on these tips, which provide a means of checking theoretical predictions.

WORKING WITH SAMPLES IN THE DIAMOND CELL

A sample of material we wish to study under high pressure must be introduced as a very thin film between the flat diamond anvil and the indenter. If the film is sufficiently thin, the pressure distribution will still be given by our basic equation relating maximum pressure, P_0 , to tip radius and applied force. The film may be introduced by vapor deposition, by sputtering, or by evaporation from solution. Very thin samples can also be prepared by thinning a bulk sample by grinding, polishing, and etching.

The pressure attained in an experiment is calculated directly from the Hertz equation. Since this expression includes a constant elastic modulus (E), it depends on the assumption of linear elastic behavior of diamond, an assumption that has been justified experimentally. The reliability of the pressure calculation has been verified up to 0.5

Mbar by a direct measurement using Newton's rings (an interference technique), and is considered applicable to about 2 Mbars. Other proved methods of determining high pressure also depend upon the assumption of linear behavior; for example, a measurement based on a shift in the ruby fluorescence peak (see the article in this issue by William Bassett) assumes a linear relation to pressure, and the method is known to be accurate at least to pressures of 100 Kbars.

Since one of the effects of high pressure in which we are most interested is the transition from nonconductor to metal, we must be able to measure changes in electrical resistance. We have developed two techniques to achieve this. By either method, the measurements are taken in a tiny area at the center of the sample, where pressure is near maximum. We call one technique the coated metal film method, and the other the interdigitated electrode method.

In the coated film method, thin films of metal are sputtered onto the highly polished diamond anvil and the diamond indenter, and leads are attached to each. The sample is placed on top of the metal film on the anvil, and the indenter is pressed against the sample. If the sample undergoes a transition to metal, this occurs in the region of highest pressure, along the axis of the indenter, so that resistance measurements pertain primarily to the first material to transform. This method has been used successfully in our laboratory, but it has one shortcoming: if the indenter were to punch through the specimen, a short would occur, and this would be difficult, as a rule, to distinguish from a transition. To avoid

this problem, we invented the second method.

The interdigitated electrode system is diagrammed in Figure 3. This technique, which was developed by Kam-Shui Chan and me, not only prevents electrical shorting, but also eliminates the difficult problem of positioning the indenter precisely so that the tip makes contact between the electrodes. (This is necessary to ensure that the electrodes measure the point of highest pressure, where the transition to metal occurs first.) With the interdigitated system, the indenter tip can be positioned anywhere in the electrode area, since the electrodes are fabricated as grids of seventy-five or more electrode fingers.

Of course, the use of very small tips—needed, as explained, to permit the attainment of very high pressures—requires comparably small electrodes. The grids we use are produced by photolithography in Cornell's new National Research and Resource Facility for Submicron Structures (see the April 1978 issue of this magazine). The width of each finger (d in Figure 3) was 6 microns in the first experiments we carried out. In the work on xenon, d was 3 microns, and we are working to reduce the spacing to 0.6 micron using projection lithography. We are also beginning to work with electron beam lithography, which is capable of producing an even finer pattern, and we hope later this year to be able routinely to make electrodes with a width of 0.1 micron.

The electrodes that we have used so far are nickel and are 350 Angstroms thick; in the future, other materials will be used for electrodes for different applications.

Figure 3

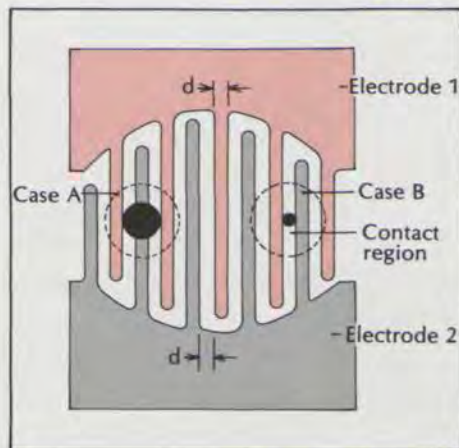


Figure 3. The nonshorting interdigitated electrode system for resistance measurements in very small areas. This assembly is placed on the diamond anvil and covered with a film of nonconducting sample, and then the indenter tip is pressed against it from above. If a transition to metal occurs, the maximum size of the conducting area is determined by the spacing between the electrode fingers and their width (d); such areas are indicated in the sketch by black circles corresponding to two possible indenter positions. Finger diameters can be made as small as a fraction of a micron. This system, developed in Professor Ruoff's laboratory, offers the advantage that the indenter can be positioned anywhere in the interdigitated area. The electrodes are fabricated in Cornell's National Research and Resource Facility for Submicron Structures.

Once the electrodes are in place, electrical leads are attached to the anvil base. Then the sample is deposited on the anvil. Finally, the indenter is pressed against the sample-electrode-anvil assembly, and contact is established over the region shown by the dashed circle in Figure 3. When the insulator-to-metal transition pressure of the sample is reached, the newly formed metal closes the circuit. The metallic region is represented in the figure by two solid circles, representing two extremes of possible indenter location. The center pressure will be different for these two cases; but if the finger widths and spacings are very small, the pressure at which the transition is observed will approach P_0 .

EXPERIMENTAL RESULTS WITH OUR APPARATUS

Our diamond indenter-diamond anvil system has enabled us to make some interesting studies of what happens to various materials under extreme pressure.

One of the substances we investigated is aluminum oxide, of interest because

early work at the Moscow High Pressure Institute had indicated a metallic transition at 0.4 Mbar. We did not observe a transition to a conducting phase, however, at pressures up to 1.2 Mbars. It should be noted that an electrical short would give the appearance of an insulator-to-metal transformation. Had we observed a sharp resistance drop, we would have used our nonshorting interdigitated-electrode method to confirm that a transition had actually taken place. The results we obtained demonstrate the necessity of ensuring against

Figure 4. Representative experimental results for the determination of the pressure at which zinc sulfide transforms to a metal. The transition is detected in the diamond-anvil cell as a drop in resistance. These experiments established that the transition occurred at an applied pressure of 14.0 ± 0.5 GPa (1 GPa = 10 kbars); a value of 15.0 ± 0.5 GPa has been obtained on the ruby scale. A control experiment, in which no sample was present, showed no resistance drop.

Figure 5. Experimental results showing the creation of metallic xenon. Transitions from insulator to conductor were observed as drops in resistance (as measured on a Keithley 160B multimeter). The temperature was 32°K and the interdigitated electrode system was used in the measurements. The readings show a drop in resistance by a factor of 10^4 to 10^5 , occurring at about 33 GPa (or 330 kbars). This work was done by David A. Nelson, Jr.

electrical shorting in high-pressure resistance measurements.

Using our interdigitated electrode technique, we detected a metallic transition with zinc sulfide (see Figure 4). This was the first observation of the transition in which pressures were measured by a primary method.

Our work with sulfur, in which we produced a transition to metal, revealed another interesting property: resistance was found to drop continuously as the pressure increased, rather than sharply as in most experiments. It appears that

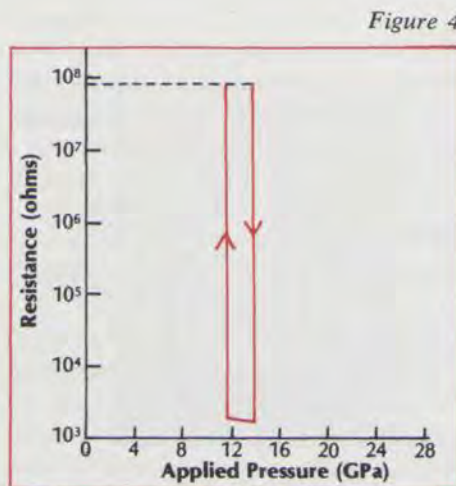


Figure 4

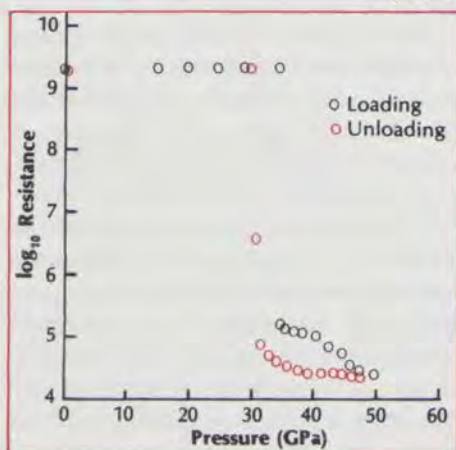


Figure 5

in sulfur the band gaps, and therefore the metallic behavior, vary linearly with pressure. This summer we plan to look for superconductivity in sulfur. Recent reports in the literature suggest that peculiar effects are involved and we are anticipating experiments of great interest.

For our research with xenon, we developed special techniques for handling gaseous samples. We introduced the gas into an evacuated chamber containing the anvil, and cooled it by means of liquid helium flowing through a sup-

porting plate. The thickness of the xenon film condensed on the anvil was measured by a quartz thickness monitor, which uses quartz crystals on the cold plate—one crystal adjacent to the diamond anvil and the other in a permanently evacuated chamber. Results for a representative experiment, showing the transition from insulator to conductor, are given in Figure 5.

We have also studied the electrical behavior of ice at 78°K in the same apparatus with the same electrodes and indentors. We found that ice remains an insulator to 0.72 Mbar, the highest pressure used.

EXPERIMENTS UNDERWAY AND PLANNED

Current and future work will further improve the experimental techniques and equipment, and explore the high-pressure properties of additional materials.

We are continuing to develop tinier tips in order to achieve higher pressures; the ultimate static pressure attainable by use of diamonds will be known soon. Tinier tips will require smaller electrodes, and fabrication techniques for electrodes will also be refined. Three students will be working on these problems.

A special cell that can be used at temperatures as low as 2°K and with loads as small as 10 grams has been completed and is now being tested. Provided that the diamonds do not fail, this cell should produce a pressure of 2.4 Mbars with a load of 22 grams and a 1-micron indenter tip.

We also have an interesting piece of work in progress which uses a kind of diamond cell developed at the National Bureau of Standards. Here we are using



A new diamond indenter-anvil system in Professor Ruoff's laboratory will be used at temperatures as low as 2°K. Researchers involved in the experiments include (left to right) Robert Terry, high-pressure technician; Dan Golopentia, senior post-doctoral associate; and Volker Arnold, high-pressure technician. The diamonds and the load cell for measuring the force are in the cylinder just above the bench top.

We are also beginning to examine the effects of high pressure on the conductivity of a polymer, polyacetylene. This material, which has fibrils containing one-dimensional chains, conducts electricity much better along the chains than perpendicular to them (it has a band gap of about 1.4 eV and can be doped to be a p-type or an n-type semiconductor), and high-pressure studies may elucidate the conductivity mechanism. Perhaps we will convert this semiconducting polymer into a metal; conceivably, however, high pressure will decompose it.

LOOKING AHEAD TO HIGH-PRESSURE POSSIBILITIES

Since high pressure drastically alters the characteristics of materials, it raises hopes that radical new technologies may become available. For example, superconductivity effects that occur at high pressures look attractive as transmission costs increase. An idea that is particularly enticing to many people is the formation of metallic hydrogen, which could be used for rocket fuel or as pellets for energy production in fu-

an x-ray technique called Energy Dispersive X-ray Diffraction to study the crystal structure of the new phases produced. This technique has been used in earlier work at Cornell to look at the high-pressure structure of silicon, and is currently being used to study the high-pressure phase of gallium arsenide. With the high-energy x-ray source now available, experiments take a week or more to perform, but we hope soon to be able to use x rays of much higher energy from the Cornell High Energy Synchrotron Source (CHESS) when

construction of this facility has been completed. The shorter time required to gather data—perhaps minutes instead of weeks—will enable us to study the kinetics of transformations occurring at high pressure.

Of the materials we hope to study, oxygen and hydrogen have high priority. We will also continue our studies of metallic iodine, sulfur, and xenon, to see whether we can detect superconductivity. Other materials of considerable interest are methane, ammonia, nitrogen, and, of course, diamond itself.

sion reactors. NASA (which is supporting much of the high-pressure research we are conducting at Cornell) is interested in this possibility.

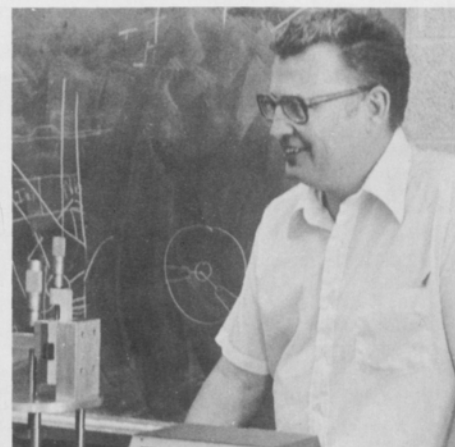
I am often asked whether I believe that metallic hydrogen will be metastable—that is, whether it will persist at ordinary pressures if kept at low temperatures. My answer is and has been: No. My reason is that the metallic state involves long-range forces, and the only examples of metastability involving large energy differences (0.1 eV per atom, say) involve short-range covalent bonds. Moreover, the degree of “unhappiness” of metallic hydrogen—expressed as a free energy of 4 eV per atom at zero pressure—constitutes a tremendous driving force to return to the molecular state. I could be wrong, but I’ll be very surprised if I am. I am also asked whether I think hydrogen will be a superconductor at relatively high temperatures (above 100°K and possibly at room temperature), as some theorists have predicted. Again, I have to say no. Here my reason is that the theorists have calculated the electron behavior of hydrogen metal on the as-

sumption of a static lattice; but in hydrogen the ions (protons) are very light and have appreciable kinetic energy. I expect that accounting for this properly will drastically alter the theoretical predictions.

If I were asked to suggest what will be the highest transition temperature of an element to a metal, I would say that it may be as high as 30°K at pressures of a couple of megabars, and that the metal will be carbon, nitrogen, or oxygen.

Much is still unexplored and unknown in the realm of ultrahigh pressure. There is a long way to go from millions of atmospheres of pressure exerted over dimensions of less than a millionth of a meter to ultrahigh pressures of practical use. But this is partly what makes high-pressure studies an exciting field of research: the possibilities for innovative work are great, and the potential for discovery and application is enormous. Remarkable results are anticipated, hoped for—or perhaps still unimagined.

Professor Arthur L. Ruoff is the director of the Department of Materials Science and Engineering and is the Class of 1912 Professor of Engineering. He is known especially for his research in high-pressure phenomena: in his ongoing effort to produce metallic hydrogen, he has developed techniques of applying pressures as high as 1.4 million atmospheres. Ruoff's recent success in producing metallic xenon was announced by the National Aeronautics and Space Administration as an important



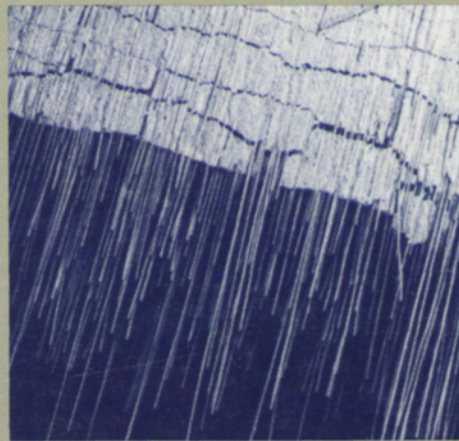
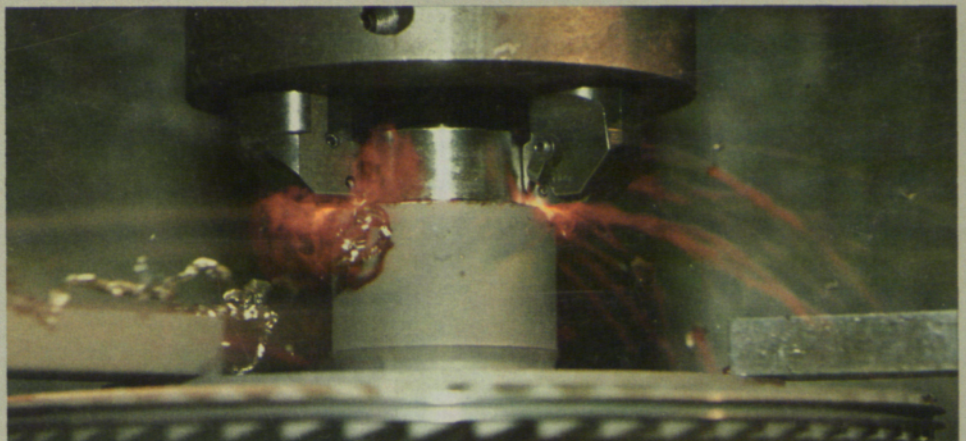
step toward the goal of producing metallic hydrogen.

Ruoff joined the faculty here in 1955 after earning the B.S. degree at Purdue University and the Ph.D. in physical chemistry at the University of Utah. At Cornell he has served as graduate faculty representative for materials science and engineering, and he is a member of the graduate Fields of Applied Physics and Geological Sciences. He has twice served on the executive committee of the University's Materials Science Center, and currently is active in the National Research and Resource Facility for Submicron Structures, both as a researcher and as a member of the program committee. Outside the University he serves as a consultant to numerous industries and laboratories on high-pressure phenomena, and to several universities on techniques of multi-media instruction, a teaching method he has developed in classes here.

His honors include a National Science Foundation Senior Science Faculty Fellowship, and the 1967 Western Electric Fund Award, presented by the American Society for Engineering Education for excellence as an educator. He is a fellow of the American Physical Society and has been named Engineer of Distinction by the Engineers Joint Council.

ENGINEERING

CORNELL QUARTERLY



VOLUME 23
NUMBER 2
WINTER 1989

A NEW ERA
OF CERAMIC
MATERIALS

THE CORNELL CERAMICS PROGRAM

by Arthur L. Ruoff

The time preceding the seventh millenium is known as the preceramics age, a designation that acknowledges the vital role of ceramics in the development of human society. Pottery, which made possible the storage of agricultural products, was introduced along with agriculture itself; clay tablets were the first durable historical and commercial documents.

Archaeological discoveries, including pottery and tablets, provide clues to history and reveal links to our past. For instance, we know that the cities of Mycenae and Troy in Homer's epic poems truly existed. The island to which Odysseus returned must also have been a real place, probably visited by Homer; perhaps it is the Greek island of Ithaki, after which our upstate New York city of Ithaca is named.

Today we no longer rely on clay tablets, but we do use a great many ceramic materials. We take for granted such common modern artifacts as brick buildings, concrete highways, window glass, ceramic flower pots, and bathroom fixtures. If reminded, we recall that ceramics are a vital component of the sparkplugs in our automobiles and the insulators that hold high-voltage transmission lines. Perhaps

we recall that ceramics line the furnaces in which iron is melted to produce cast iron and steel. Indeed, ceramics made possible first railroads, and then the automobile and trucking industries.

CERAMICS—FROM CLAY POTS TO SUPERCONDUCTORS

The first ceramic objects were molded from clay and simply dried in the sun. Then it was learned that heating in a fire could make them rock-hard. Over the millenia, both materials and techniques have expanded to the point that definitions are difficult.

What are ceramics? Sometimes it seems easier to say what they are not: they are those solid materials that are not metals and are not based on organic compounds. If forced to be more specific, one could begin by saying that ceramics include oxides (such as aluminum oxide), nitrides (such as silicon nitride), and carbides (such as boron carbide). Sometimes a definition includes nonmetallic inorganic substances in general, and even diamond might be considered a ceramic. Often the term is reserved for materials that can withstand very high temperature.

Ceramics include crystalline materials (such as quartz, one of several crystalline forms of silicon dioxide) and amorphous or glassy substances (such as the material obtained when molten silicon dioxide is cooled rapidly). More often than not, ceramics are impure materials—solid solutions and mixtures of several components. Unlike archaeologists, materials scientists would include in their definition a number of geological materials, mostly oxides. To the materials scientist, marble and granite are ceramics, and so are gemstones such as ruby (which is aluminum oxide, Al_2O_3 , contaminated with chromium) and sapphire (which is Al_2O_3 contaminated with iron).

In 1987 still another kind of ceramic—complex oxides prepared in the laboratory—made a stir in the scientific world and the news media. The furor began in earnest with the announcement by Paul Chu, a professor of physics at the University of Houston, that he and his colleagues had made a ceramic compound, copper barium yttrium oxide, that was a superconductor at 96°K , approximately four times the temperature at which metals become superconducting.

“Today the uses and potential uses of ceramics boggle the imagination.”

THE USEFUL PROPERTIES OF SYNTHETIC CERAMICS

Over the centuries, man-made ceramics have been useful because of their physical properties (animals and insects could not penetrate clay storage vessels, for example), their chemical resistance (the clay pots of Jarmo in Kurdistan have persisted for some nine thousand years), their high melting point (crucial for the lining of steel furnaces), their transparency (valuable for glass objects), and, surely, their low cost.

Today the uses and potential uses of ceramics boggle the imagination. They include:

- *Applications in communications.* The optical fiber, which is revolutionizing the communication industry, is an example of a high-tech ceramic material whose development is an almost unbelievable achievement of materials science and engineering.

Also important are ceramic magnetic materials that are electrical insulators. These are ideal for transformers, such as those in television sets, that are used at high frequencies. (The reason these ceramic materials are good for this application is that they do not exhibit eddy-current heat-

ing, a problem with conducting materials such as magnetic iron.)

- *Applications based on piezoelectric effects.* When placed in vibration by the application of an alternating electric field, piezoelectric ceramic materials can create sound waves (sonar). Conversely, a sound wave can be detected by the electrical pulse it creates when it hits and hence slightly deforms a material such as barium titanate.

- *The fabrication of heat engines.* Unlike metals, ceramics have very high melting points and so retain their strength at the high temperatures at which heat engines are more efficient. Actually, ceramics are already used as part of the electronic controls of automobile engines, as sensors of oxygen in the hot gases.

The great drawback of ceramics as structural materials is their brittleness; unlike most metals, ceramics generally have no ductility or almost none. If this drawback can be overcome by design ingenuity or by making ceramics with greater toughness, a large-volume industry will open for the use of these materials for building engines—more than fifteen million new motor vehicles were sold in the United States in 1988.

PROCESSES USED FOR MAKING CERAMIC PRODUCTS

The usual procedures for making metals—melting and casting—won’t do for ceramics. Iron can be melted in ceramic-lined containers, but there are no containers for materials such as magnesium oxide, which has a melting temperature of 3175° K.

One process that is used for making ceramics (and also refractory metals such as tungsten) is quite akin to the method used for making snowballs. We start with a powder of, say, Al_2O_3 particles and compact them in a die. At this point the component has the approximate consistency of chalk. Then the compacted powder is heated in a furnace to a temperature that is high but substantially below the melting point. In the process, called *sintering*, the powder grows together by the motion of atoms until—ideally—a nonporous solid is formed. Sometimes this sintering is carried out with the powder enclosed in a metal jacket which is surrounded by argon at a pressure of 30,000 psi; this is called *hot isostatic compaction*. Sometimes the powder is first held together at low temperatures with an organic binder (glue) which is driven off at intermediate temperatures; a

Table I

GOALS OF THE CORNELL CERAMICS PROGRAM**Input:**

- Increase in faculty size
- New equipment
- Visiting industrial scientists
- Visiting professors

Output:

- Increased understanding of ceramics science
- More and faster exchange of information
- More ceramicists with B.S. degrees
- Twice as many Ph.D.s by 1992–93
- Further increase in Ph.D. output during 1990s
- Education of Ph.D.s who will have a fuller understanding of how to maximize university-industrial cooperation

further rise in temperature completes the sintering. With this method, sheets of ceramic as thin as paper can be produced.

Other ways of producing ceramics are by precipitation from solution and by deposition from the vapor phase. For example, aluminum, which has a low melting point, can be vaporized in a low-pressure oxygen atmosphere; when the vapor is passed over a cold substrate, a thin film of Al_2O_3 is formed. Alternatively, such a film can be produced by passing a vapor of an organo-metallic compound, aluminum ethoxide, over a hot substrate; the ethoxide decomposes, leaving an Al_2O_3 film on the substrate, and organic fragments in the vapor flow downstream.

An incredible number of different processes are being used to make ceramics. The number of possible ceramics appear endless, and the properties they exhibit are extremely diverse.

THE ORGANIZATION OF CORNELL'S CERAMICS PROGRAM

In 1984, while serving as director of the Department of Materials Science and Engineering, I felt that the time had come to expand our research in the field of high-technology ceramics. It was apparent that new materials with properties not yet achievable would be urgently needed, and that ceramics are leading candidates. In

fact, the federal government was beginning to plan a new initiative in ceramics.

The faculty of our department decided that the proposed ceramics program should continue to emphasize fundamental research—studies that would lead to an understanding of how the properties of ceramic materials can be enhanced. Specifically, we wanted to expand our efforts in areas in which industrial need is likely to be acute:

- ceramic thin films and coatings;
- ceramics for electrical-magnetic and optical applications;
- tough ceramics.

With the help of Professor David Kohlstedt, I prepared a prospectus of a Cornell Ceramics Program that would be jointly supported by industry and government. Subsequently, Provost Robert Barker and William B. Streett, who was then acting dean of the College of Engineering, reached an agreement with our faculty. We were given permission to add three professors if we brought in three industrial sponsors that would each contribute \$1.5 million over a five-year period.

With the help of all the members of our department, but especially those who were already working full-time on ceramic materials, we succeeded in enlisting two excellent companies—Corning Glass Works and IBM. This support, along with funding

from the National Science Foundation, has resulted in an expanded, active program.

As summarized in Table I, we have used the funds obtained from the university, industrial, and government sources to purchase new equipment, add faculty members, and bring in visiting professors and industrial scientists.

THE CORE OF THE PROGRAM: THE RESEARCH FACULTY

At the time the program was organized, the department had three faculty members—C. Barry Carter, David Kohlstedt, and Rishi Raj—who were working full-time in the field of ceramics, and others who were working part-time or sometimes on ceramic materials. The program has resulted in an expanded effort by these faculty members, and the addition of two people who already had active programs in ceramics research underway.

Kohlstedt and Raj were carrying out research on the production of ceramic materials (chiefly from powder). Carter was an expert in the use of transmission and scanning electron microscopy for studying the microstructure of materials and the mechanisms of solid-state reactions. We decided, therefore, to seek faculty members who were active in two other areas.

One of the new people, we decided, should be an expert in the study of point defects in solids. This is an important area because the motion of defects is the mechanism essentially responsible for sintering and for reactions in the solid state. The person we found is Rüdiger Dieckmann, who joined us from the University of Hannover in Germany. He brings to us an expertise in handling materials at extremely high temperatures under carefully controlled conditions.

We also sought an expert in the chemi-

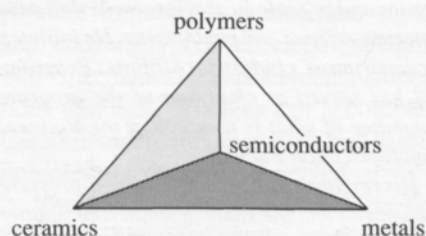
cal synthesis of inorganic materials by processes related directly to the production of ceramic materials. We were fortunate to find a young man, Emmanuel Giannelis, whose experience was in this field; he joined us as an assistant professor.

Cornell faculty members with a primary interest in ceramics include, in addition to these five in our department, James Burlitch (Chemistry), who is working on the synthesis of ceramic materials. A number of other Cornell professors are working part-time in the field; they include Robert Pohl and Albert Sievers (Physics), Robert Buhrman (Applied and Engineering Physics), Frank DiSalvo (Chemistry), and Jack Blakely, Herbert Johnson, Stephen Sass, and myself (Materials Science and Engineering).

CERAMICS AS PART OF AN OVERALL PROGRAM

An important aspect of the ceramics research is its integration into the overall program of the department. Ceramics are one of the four kinds of material we study, and research on ceramics is directly connected to work with the other types.

The overall field of materials science and engineering can be thought of in terms of the connecting edges and faces of a tetrahedron:

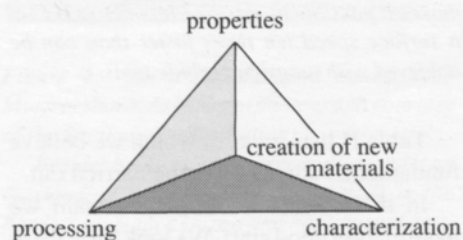


This issue of the *Quarterly* focuses on ceramics (all the professors who are authors are members of the Cornell Ceramics

Program), but we recognize the connections with polymers, semiconductors, and metals. For instance, the superconducting ceramics I have mentioned fall on the ceramics-metals line. Glass-fiber composites—ceramic fibers embedded in a polymer matrix—are “located” along the ceramics-polymers edge. A computer chip comprises silicon (a semiconductor), an electrically insulating barrier of silicon oxide (a ceramic), and electrical leads of aluminum or tungsten (metals) to transmit power and signals; thus the chip involves a semiconductor, a ceramic, and a metal, represented by one side of our tetrahedron.

The value of interplay among people working in different ways with these various materials was recognized in 1961 when the graduate Field of Materials Science and Engineering was formed at Cornell. The field faculty includes professors from our department and from a half-dozen other departments. This facilitates the interaction of polymer scientists with ceramicists, for example, or of semiconductor experts with metallurgists. It is a synergism that leads to more innovation and better teaching and research results for all.

This interplay of disciplines is illustrated by a second tetrahedron representing the scientific approach of materials scientists:



Suppose we make a new material—for example, a high-temperature superconducting ceramic. We will investigate dif-

ferent ways of processing it. We will also experiment to find out how processing affects the microstructure (the positions of the atoms in the crystal structure and the presence of features such as point defects, dislocations, and grain boundaries), and how the microstructure in turn is related to the properties of interest (such as the current-carrying capacity, the superconducting transition temperature, and the mechanical strength).

Eventually, an overall picture begins to emerge and it is seen that certain compositions and defect structures give better properties. Accordingly, changes in processing procedures are made to obtain the best microstructure. What is learned can also be the base for creating other new materials.

TOWARD THE HIGH-TECH CERAMICS OF CENTURY 21

Many of the ceramics of the future will be composites.

An example of the possibilities is the recent development of a composite made up of tiny silicon carbide whiskers in a matrix of aluminum oxide—a material hard enough to make a remarkably good cutting tool for machining superalloys such as Inconel. An important new technique was used to make this composite: the whiskers of silicon carbide were grown by submicron fabrication methods. The processing involved hot-pressing a mixture of the whiskers (about 30 percent) with aluminum oxide powder.

This example suggests the wide range of studies that contribute to the development of these new materials. In such a composite, the bonding at the interface of the ceramic whisker and the matrix material is very important; and indeed, the study of surfaces and interfaces is an important area of research in ceramics.



Table II
**AREAS FOR FUNDAMENTAL
 STUDIES IN CERAMICS**

- Surface chemistry and physics
- Interfaces: ceramic-ceramic, ceramic-metal, ceramic-polymer, ceramic-semiconductor
- Microstructure, defects, grain boundaries, creep, cavitation
- Multi-component kinetics, dopants in sintering processes
- Coating technologies, sol-gel processes
- Vapor deposition processes: sputtering, CVD (chemical-vapor deposition), MBE (molecular-beam epitaxy)
- Laser and plasma processes
- Colloids, fine powders, whiskers, fibers
- Composites: combinations of ceramics, polymers, metals, and semiconductors
- Ceramic machining, hole-making
- Thermal structures
- Graded interfaces

Above: A cutter made of a revolutionary new ceramic material is used at Therm, Inc. of Ithaca to machine the alloy Inconel. Left to right are Robert West, director of Therm's Ceramics Division; Arthur L. Ruoff, director of the Cornell Ceramics Program (who served as consultant); and Robert Sprole II, president of Therm.

The composite cutter (of silicon carbide whiskers in an aluminum oxide matrix) was produced by hot pressing. This new material makes it possible to machine Inconel 713LC at a surface speed ten times faster than can be achieved with tungsten carbide tools.

Table II lists areas in which we believe fundamental studies must be carried out.

In the Cornell Ceramics Program we have made a good start. We look forward to contributing significantly to the realization of new high-tech ceramics for the early twenty-first century.

Arthur L. Ruoff, the Class of 1912 Professor of Engineering, organized the Cornell Ceramics Program during his recent ten-year term as director of the Department of Materials Science and Engineering, and is currently serving as director of the program.

Ruoff joined the faculty in 1955 after earning a B.S. degree at Purdue University and a Ph.D. at the University of Utah. At Cornell he is a member of the graduate fields in applied physics and in geological sciences, as well as in materials science and engineering. He initiated his department's Industrial Affiliates Program, and has served as chairman of the program committee of what is now called the National Nanofabrication Facility.

His research is in the areas of high-pressure phenomena and the study of sputtering, sputter etching, and reactive-ion-beam etching. He is currently heading a project to create a national high-pressure x-ray facility at the Cornell High Energy Synchrotron Source (CHESS). Ruoff is a fellow of the American Physical Society.

ENGINEERING

CORNELL QUARTERLY

25

ENGINEERING
CORNELL QUARTERLY

ANNIVERSARY ISSUE

VOLUME 25 NUMBER 4 SUMMER 1991

Arthur L. Ruoff

Materials Science and Engineering

It was in 1955—eleven years before the advent of *Engineering: Cornell Quarterly*—that I arrived at the Cornell College of Engineering. What was it like at the college then? The faculty was dedicated to teaching undergraduates; there was little research underway. Usually the parents of the undergraduates paid for most of the students' education. The students understood the source when you quoted from *Hamlet*, cited Seneca or Marcus Aurelius, or referred to classical Greek art or the Renaissance. The administration was capable of providing free parking. At Cornell freedom of speech was practiced and strongly defended and we were free of social engineering.

What is the college like in 1991? We now have a major research effort; there are currently 801 graduate students in M.S./Ph.D. programs in engineering, and 247 in the professional Master of Engineering program. We have new departments, including Materials Science and Engineering, which was established in 1965; in MS&E we now have an undergraduate program (rated first in the nation in the Gourman report) with twenty-five or thirty seniors graduating each year. Our students are bright, but I find they are less knowledgeable in mathematics and liberal arts than students used to be. Parking is inadequate and expensive. The "politically correct" syndrome is prospering at Cornell and is the most serious threat to free speech since the time I arrived on campus.

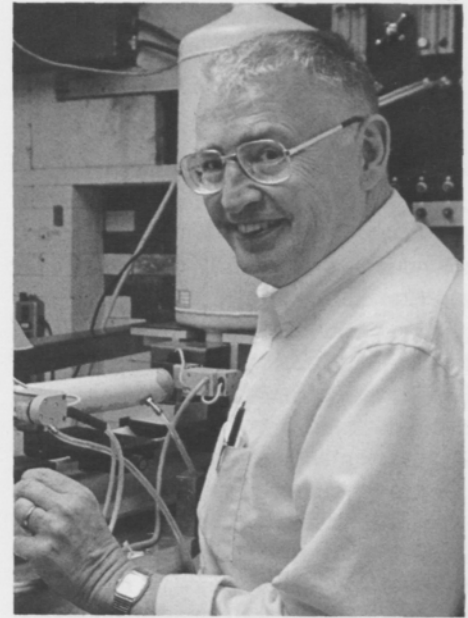
There are now major facilities that make Cornell a mecca for research in materials science. These include the Materials Science Center, the Cornell High Energy Synchrotron Source (a national resource), the National Nanofabrication Facility, and the

"There are now major facilities that make Cornell a mecca for research in materials science."

Cornell Theory Center (which includes a national supercomputer facility). These, and the research performed, are primarily supported by the federal government, but close to 20 percent of our research support now comes from industry, much of it via the Cornell Ceramics Program, the SRC Program on Microscience and Technology (Cornell is a SRC "center of excellence"), and the Industry-Cornell Alliance for Electronic Packaging.

In our department we have expertise in ceramics, metals, polymers, and semiconductors, and on materials for structures large and small: composites for airplanes, for example, or carefully made materials for computer chips. We have achieved static pressures greater than those at the center of the earth, carried out x-ray diffraction on samples as small as 10^{-11} cubic centimeter, and studied polymer interdiffusion with use of Rutherford backscattering. Our research is internationally respected.

Of our more than one hundred Ph.D. students, about 25 to 30 percent are foreign—the best and brightest from their native lands. Since many of these foreign students remain in the United States, this constitutes a giant brain drain—a lend-lease in reverse. If better United States applicants were available, we'd take them. Our criterion is: excellence. A great department, a great college, a great university, and a great country owe their eminence to a relentless drive for excellence.



Arthur L. Ruoff came to Cornell in 1955 as a member of the Department of Mechanics and Materials. He is now the Class of 1912 Professor of Engineering in the Department of Materials Science and Engineering.

At Cornell he has served as director of his department and as chairman of the program committee of the National Nanofabrication Facility. He initiated the Industrial Affiliates Program in Materials Science and Engineering, and also the Cornell Ceramics Program, which he directs.

Ruoff received the B.S. degree from Purdue University in 1952 and the Ph.D. from the University of Utah in 1955. A specialist in ultra-pressure phenomena, he has published two books as well as several hundred papers. His research includes studies on x-ray diffraction at pressures greater than those at the earth's center.

Ruoff has been a visiting professor at the University of Illinois as a National Science Foundation fellow. In 1966–67 the American Society for Engineering Education presented him the Western Electric Fund Award for Excellence in Instruction of Engineering Students. He is a fellow of the American Physical Society.

Cornell CHRONICLE

Ruoff in the *Cornell Chronicle*

The entires below are linked to the articles

Metallic Hydrogen: Engineers Racing Russians. *Cornell Chronicle* 001_21 p1&4 March 12, 1970

Pushbutton Learning Device Aids Students. *Cornell Chronicle* 002_21 p4 February 18, 1971

Education Is Flexible in 'Learning Center'. *Cornell Chronicle* 003_24 p1&6 March 9, 1972

Scientists Seek to Produce Hydrogen in Metallic State. *Cornell Chronicle* 004_24 p8 April 12, 1973

\$7.5 Million Ceramics Program Started Here. *Cornell Chronicle* 016_39 p1 June 20, 1985

New Major Program Here Now Combines Electrical Engineering, Materials Science. *Cornell Chronicle* 017_01 p4
September 5, 1985

Cornell's Diamond Anvil Puts Big Squeeze on Tiny Materials. *Cornell Chronicle* 017_32 p3 May 1, 1986

Packaging microchips challenges engineers. *Cornell Chronicle* 018_01 p8 August 28, 1986

Grant supports ceramics studies. *Cornell Chronicle* 018_15 p8 December 11, 1986

Oxygen may become metallic at high pressure: researchers *Cornell Chronicle* 018_26 p1&8 March 19, 1987

Rutherford 'atomic machine gun' installed in Bard Hall. *Cornell Chronicle* 018_29 p9 April 16, 1987

Pressures greater than Earth's core reached here. *Cornell Chronicle* 022_15 p1&8 December 13, 1990

Metallic-hydrogen claims disputed by CU researchers. *Cornell Chronicle* 022_24 p7 March 7, 1991

'Soccer-ball' molecule is far stiffer than diamond. *Cornell Chronicle* 022_31 p7 May 2, 1991

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

Putting the squeeze on diamonds achieves top high-pressure award. *Cornell Chronicle* 025_06 p1&8 September
30, 1993

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-



HIGH PRESSURE MEN — Arthur L. Ruoff (right), professor of materials science and engineering, glances at some notes compiled by his research associate C. C. Chao, while Volke Arnold, high pressure technician, makes an adjustment to a chamber which contains an ultrahigh pressure vessel. Pressure in the vessel is generated by a 1,000-ton press. The insert shows a gasket which illustrates the sample size.

drogen 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.

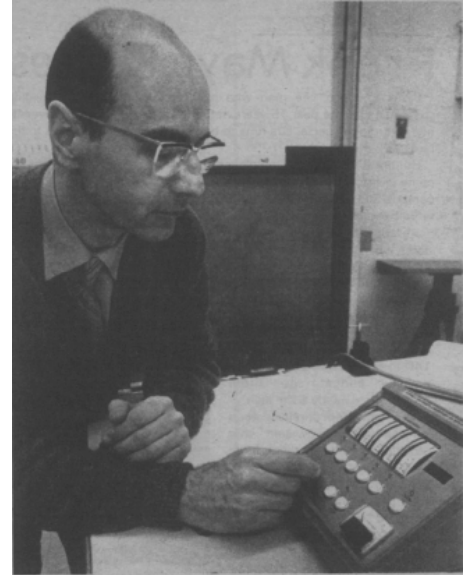
Littauer sees two principal benefits for the system. The professor finds out quickly and painlessly whether or not he is communicating with the class. The system also helps keep the students alert since they are required to participate in the quizzes, which may number as many as a dozen in the hour-long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



Prof. Raphael M. Littauer looks at a display unit which tells him how students have responded to a multiple choice question he has asked.



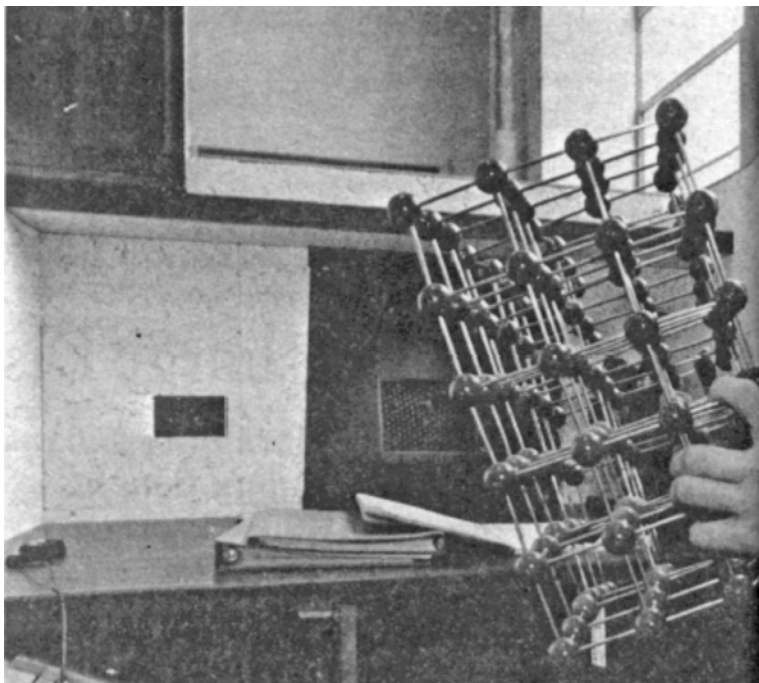
Pushbuttons at each student's seat enable him to reply to the professor's questions, or indicate whether he understood a particular point of the lecture.

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

Education Is Flexible in 'Learning Center'



David Cox, a freshman from Honolulu, looks over a molecular model. The model is one of several visual aids in the Center in Thurston Hall.

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 p1&6 March 9, 1972



Two students in specialized carrels designed by Ruoff use slide projector in their learning experience. The Learning Center has 11 carrels now.

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



Half a diamond anvil (with a coin to show size), capable of simulating pressures deep within the Earth.

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

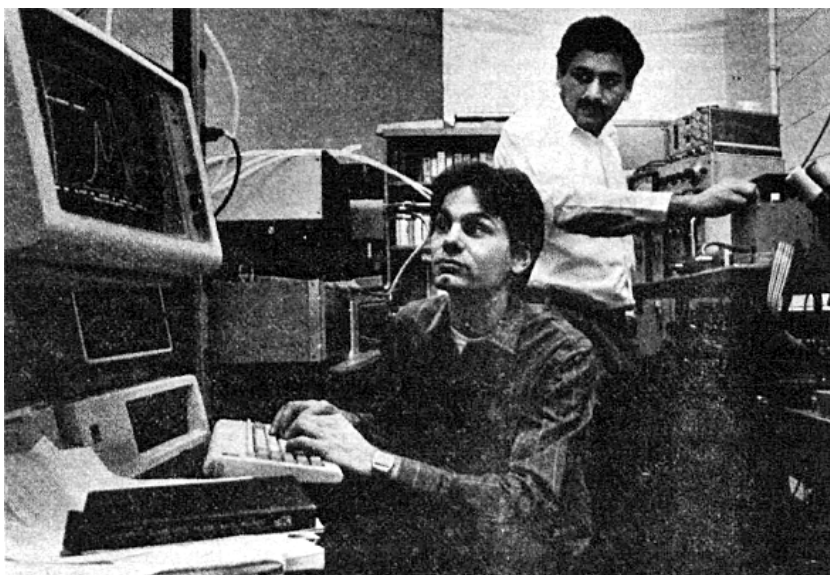
extremely bright X-rays are diverged from the storage ring, then beamed into the laboratories of CHESS (the Cornell High Energy Synchrotron Source), and through materials placed in their path.

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.



A laser beam excites ruby fluorescence as graduate student Keith Brister, left, and research associate Yogesh Vohra measure pressure inside the diamond anvil.

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.

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: researchers¹

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.

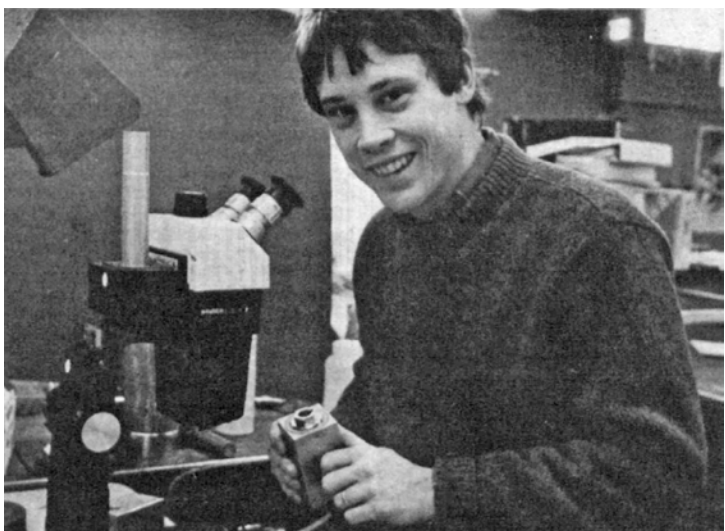
“We still have to test the electrical conductivity of such a sample, to make sure that it is a metal,” said Vohra. “But this discovery is nevertheless striking, because it shows that oxygen is actually a very exotic material at high pressures.”

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 “synchrotron 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 Synchrotron 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.



Steven J. Duclos, a graduate student in the Department of Materials Science and Engineering, prepares to demonstrate a “diamond anvil” used to test the effects of very great pressure on materials.

photo: Claude Levet

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.

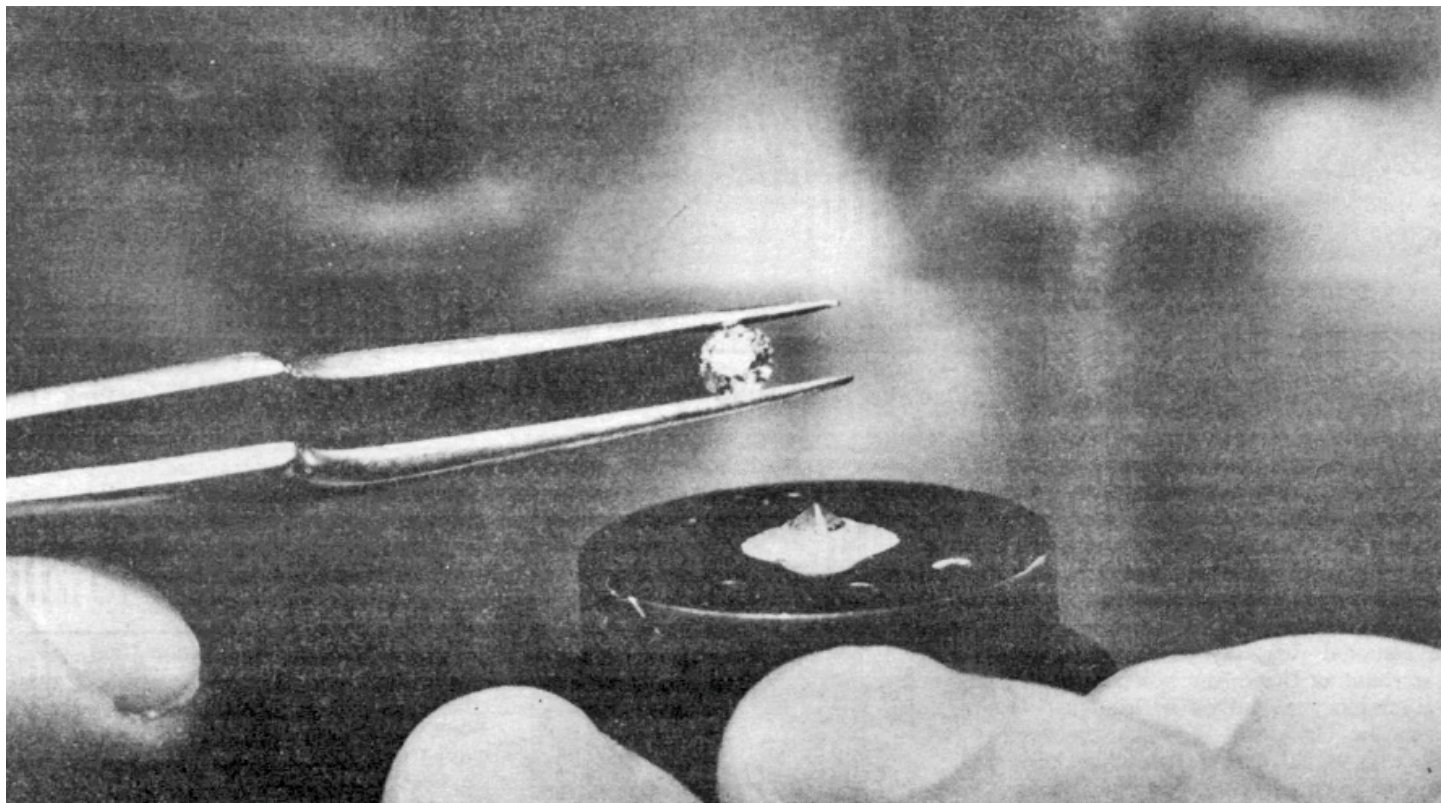
— *Dennis Meredith*

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 p1&8 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 ultrahigh pressure research, because diamonds are the only known substance hard enough to form the chambers for squeezing substances to ultrahigh pressures.

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 ultrahigh pressures would be encased in the buckminsterfullerene, he explained.

The limit on ultrahigh pressure 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 ultrahigh pressures. 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.

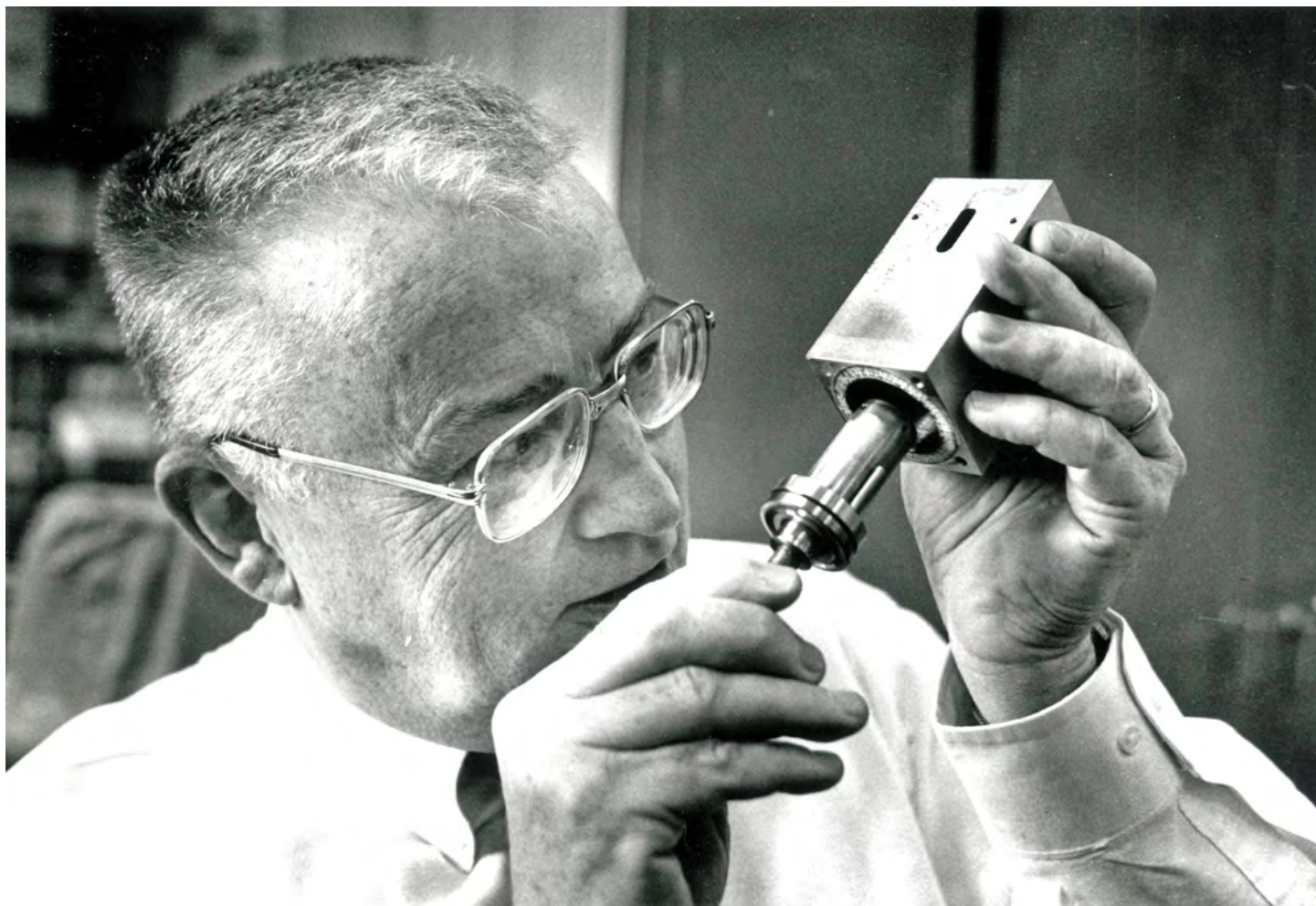
Ultrapressure studies allow researchers to study forms of solids that do not exist at normal pressures. Such studies yield better theories of chemical bonds and the behavior of electrons in solids. Ultrapressure studies also offer scientists insights into the structure of the Earth and other planets.

— *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 ultrahigh pressures. 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 p1&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.”

from Cornell Chronicle 026_06 p1&4 September 29, 1994

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."

'It will be great to have the time to travel to more meetings and get new ideas.'

– Arthur Ruoff

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.

from Cornell Chronicle 038_06 p6 September 14, 2006

