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MS&E NEWS

Cornell University
College of Engineering

Department of Materials Science & Engineering

May 2006

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 renown[ed] 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 sons:

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

CORNELLCHRONICLE

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.

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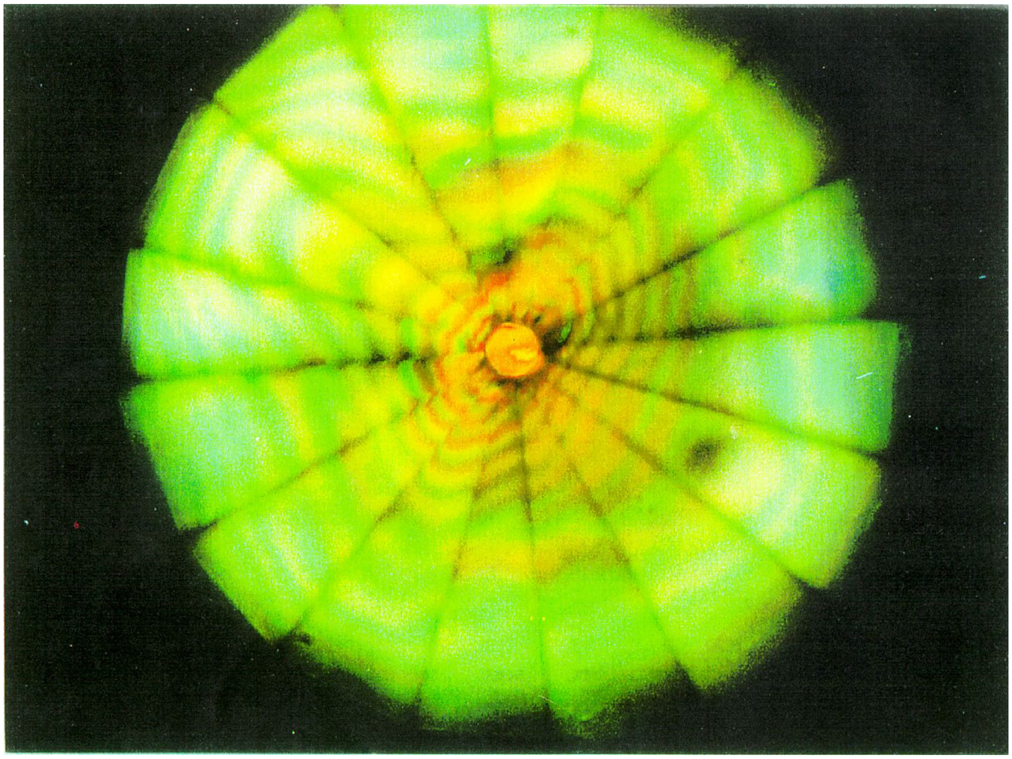
Golden Anniversary for Founder of High-pressure Program at CHESS

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 every 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.

References:

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2. Duclos, S.J., Y.K. Vohra, and A.L. Ruoff, Hcp-to-Fcc Transition in Silicon at 78-Gpa and Studies to 100-Gpa. *Physical Review Letters*, 1987. 58(8): p. 775-777.

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7. Desgreniers, S., Y.K. Vohra, and A.L. Ruoff, Optical-Response of Very High-Density Solid Oxygen to 132-Gpa. *Journal of Physical Chemistry*, 1990. 94(3): p. 1117-1122.

8. Luo, H., et al., High-Pressure Optical Studies on Sulfur to 121 GPa - Optical Evidence for Metallization. *Physical Review Letters*, 1991. 67(21): p. 2998-3001.

9. Luo, H., R.G. Greene, and A.L. Ruoff, Beta-Po Phase of Sulfur at 162 GPa - X-Ray-Diffraction Study to 212 GPa. *Physical Review Letters*, 1993. 71(18): p. 2943-2946.

10. Narayana, C., et al., Solid hydrogen at 342 GPa: no evidence for an alkali metal. *Nature*, 1998. 393(6680): p. 46- 49.

11. Ruoff, A.L., 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. Instr.*, 1992. 63

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

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Department of Materials Science and Engineering
Bard Hall, Cornell University
Ithaca, NY 14853-1501
(607) 255-4161

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 indentor 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.

<u>Other Professional Activities</u>
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: <u>Advances in Materials Research</u>
Regional Editor: <u>Phase Transitions</u>

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 Poly-acrylonitrile 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).

7. A.L. Ruoff, D.L. Prince, J. Calvin Giddings and George H. Stewart, "The Diffusion Analogy for Solvent Flow in Paper", Kolloid-Zeitschrift 166, 144 (1959).

8. A.L. Ruoff, and J. Calvin Giddings, "Paper Geometry and Flow Velocity in Paper Chromatography", J. Chromatog 3, 438 (1960).

9. J. Calvin Giddings, George H. Stewart and A.L. Ruoff, "Zone Migration in Paper Chromatography", J. Chromatog 3, 239 (1960).

10. A.L. Ruoff, George H. Stewart, Hyung Kyu Shin and J. Calvin Giddings, "Diffusion of Liquids in Unsaturated Paper", Kolloid Zeitschrift 173, 14 (1960).

11. Che-Yu Li, A.L. Ruoff and C.W. Spencer, "Effect of Pressure on the Energy Gap of Bi_2Te_3", J. Appl. Phys. 32, 1733 (1961).

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16. 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).

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

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).

21. A.L. Ruoff, "Creep by Grain Boundary Diffusion", Cornell Materials Science Center Report #298 (1965).

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).

24. B.M. Butcher, H. Hutto and A.L. Ruoff, "Activation Volume and Energy for Self-Diffusion in Aluminum", J. Appl. Phys. Letters 7, 34 (1965).

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27. J.E. Hill and A.L. Ruoff, "Temperature Dependence of the Velocity of Sound in Some Liquid Metals and Eutectic Alloys", J. Chem. Phys. 43, 2150 (1965).

28. P.J. Reddy and A.L. Ruoff, "Pressure Derivatives of Elastic Constants in Some Alkali Halides",in Physics of Solids at High Pressures, Ed. by C.T. Tomizuka and R.M. Emrick, Academic Press, New York (1965) p. 510.

28. P.B. Ghate and A.L. Ruoff, "Energy of Formation of a Vacancy in Sodium", Atomic Energy Commission Report NYO-2504-13 (1966).

29. C.R. Kohler and A.L. Ruoff, "Two Ranges of Creep Behavior in Potassium as a Function of Temperature and Pressure", J. Materials 2, 20 (1967).

30. A.L. Ruoff, "A Comparison of Virial Coefficients Obtained from Shock and Ultrasonic Data for Al and Cu", J. Phys. Chem. Solids 28, 453 (1966).

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303. C. Narayana, A. Ho, M.-F. Pai and A.L. Ruoff, "Seven-Fold Coordinated MgSe at 202 GPa", Rev. High Pressure Sci. Technol. 7, 260 (1998)

304. A.L. Ruoff, J. Orloff and C. Narayana, "The Behavior of Solid Hydrogen at 342 GPa", Rev. High Pressure Sci. Technol. 7, 772 (1998)

305. A.C. Ho, M.K. Granger and A.L. Ruoff, "X-ray Structural Study of Li_3N at High Pressure", Mat. Res. Soc. Sym. Proc., 499, 133-138 (1998), eds. R.M. Wentzcovitch, R.J. Hemley, W.J. Nellis, and P. Yu

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307. A.L. Ruoff, T. Li, C. Narayana, H. Luo and R.G. Greene, "Novel Structure of MgSe in the Multimegabar Regime: Positional Parameter Determination", Mat. Res. Soc. Sym. Proc., 499,. 429-434 (1998), eds. R.M. Wentzcovitch, R.J. Hemley, W.J. Nellis, and P. Yu

308. A.L. Ruoff, C.O. Rodriguez, and N.E. Christensen, "The Elastic Moduli of Tungsten to 15 Mbar, Phase Transition at 6.5 Mbar and Rheology to 6 Mbar", Phys. Rev. B, 58, 2998 (1998)

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311. Y. Mori, C.-S. Zha, and A.L. Ruoff, "Solid Molecular Lithium at 100 GPa", Proceeding of the AIRAPT Conference held in Honolulu, Hawaii, August, 1999 (in press)

312. J. Orloff, C. Narayana and A.L. Ruoff, "Use of focused ion beams for making tiny sample holes in gaskets for diamond anvil cells", Rev. Sci. Instr., 71 (2000)

313. C.H.B. Zhang, Y. Mori, C. Narayana, and A.L. Ruoff, "X-ray Studies of BeO to 126 GPa", Proceeding of the AIRAPT-17 Conference held in Hyderabad, India, 2000

314. A.L. Ruoff, C.O. Rodriguez and N.W. Christensen, "Pressure Strengthening and its Application to the Analysis of Hydrogen Sample-Radius Behavior in a Tungsten Gasket as a Function of the Initial Sample Radius to Tip Radius Ratio", Proceeding of the XVIII International Conference of AIRAPT, High Pressure Science and Technology (2002), J.Phys. Condensed Matter, 14, 10907 (2002)

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)

317. A.L. Ruoff, L. Sun, C-S. Zha, and G. Stupian, "Optical Properties of Silane to 218 GPa: Black Silane", Proceeding of Joint XX AIRAPT International Conference and XLIII EHPRG meeting of High Pressure Sci. and Tech., T12-KN, p. 188, Karlsruhe (2005)

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)

320. L. Sun, A.L. Ruoff, C-S. Zha, and G. Stupian, "High Pressure Studies on Silane to 210 GPa at 300 K: Optical Evidence of an Insulator-Semiconductor Transition", J. Phys.: Condensed Matter 18, 8573-8580 (2006)

321. L. Sun, A.L. Ruoff, C-S. Zha, and G. Stupian, "Optical Properties of Methane to 288 GPa at 300 K", J. of Phys. And Chem. Of Solids 67, 2603-2608 (2006)

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 0.01C in a room controlled to 0.1C.

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: $7565.4 \pm 3.7, 7569.2 \pm 1.2$ 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 synchrontron 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''. 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 Gernanium 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 $a = 2.491 \pm 0.003A$ and $c = 4.020 \pm 0.005A$.

> 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 Hugoniots) 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 $3.7eV$ at 1 atm to $3eV$ at 300 GPa and $2.5eV$ 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 $21\mu m$ with an $8\frac{1}{2}$ degree bevel and the collimated X-ray beam diameter to $4\mu m$, 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 qradient: 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, M_0, 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).

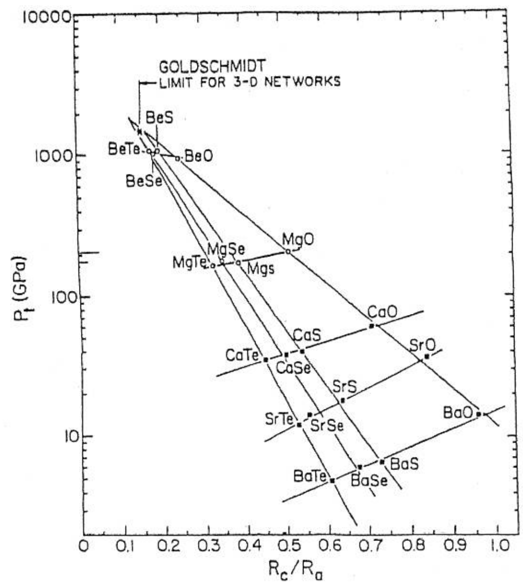
A metal is characterized in part by a low electrical resistivity. Cu is a very good metal with $\rho = 1.6 \times 10^{-8}\Omega$ at 300K while a less robust metal is Zr with $\rho = 43 \times 10^{-8}\Omega - m$. Somewhat smaller values are permitted. The next necessary condition is that ρ decreases as 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 $NiAa$ (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, Hyderbad, 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),* $n^2 E_g(Ryd) = \pi/2$, *where* n *is the refractive index and* Eg *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 metallization 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 metallization by Struzkin et al., Nature, **360**, 382 (1997), when they showed that at 95 GPa and 10K, sulfur became a superconductor, and that Tc increased to 15K as P increased to 160 GPa, and then jumped to 17K and then Tc 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 $1.9eV$ and the refractive index was 3.2 with $k = 0$; hence n^2Eg =$19.2eV$, compared to the expected $n^2Eg(Ryd) = \pi/2$ or $n^2Eg(eV) = 21.4$, 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(GPa)	V_{H_2}(cm^3/mole)	$\Delta\mu(eV)$	B(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:

$$H_2 + \frac{1}{2}O_2 = H_2O \quad \Delta\mu^0 = -2.505eV$$

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:

$$2A\ell + \frac{3}{2}O_2 = A\ell_2O_3 \quad \Delta\mu^0 = -15.8eV$$

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

However, the famous thermite reaction

$$2A\ell Fe_2O_3 = 2Fe + A\ell_2O_3\Delta\mu = \text{neg}$$

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 includes 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 at 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.