H. TRACY HALL BIOGRAPHICAL SKETCH 20 April 1980

Hall, Howard Tracy

Current Address:

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Born: Ogden, Utah, 20 October 1919; parents: Howard & Florence Tracy Hall.

<u>Married</u>: To Ida-Rose Langford, 24 September 1941 in Salt Lake City, Utah.

<u>Children</u>: Sherlene (Bartholomew), Howard Tracy, Jr., David Richard, Elizabeth (Neil), Virginia (Wood), Charlotte (Weight), Nancy (Mecham).

Education: A.S. Weber College, Ogden, Utah, 1939; B.S., M.S., Ph.D., University of Utah, 1942, 1943, 1948 respectively with major in physical chemistry and mirior in physics. Special wartime training in electronics at Bowden College, M.I.T., Harvard, and Honolulu Naval Base for a total of 11 months while an ensign in the U.S. Navy, World War II.

Employment:

- 1955-1980, Brigham Young University, Provo, Utah: Distinguished Professor of Chemistry and Chemical Engineering Emeritus 1980-; Distinguished Professor of Chemistry 1967-1980; Distinguished Professor of Chemistry and Chemical Engineering 197 -1980; Director of Research and Creative Endeavor for the entire University and Professor of Chemistry 1955-1967.
- 1948-1955, General Electric Research Laboratory, Schenectady, New York, Research Associate.
- 1942-1944 & 1946, United States Bureau of Mines Research Laboratory, Salt Lake City, Utah, chemist.
- 1940-1942, Sperry Flour Mills, Ogden, Utah, Chemical Analyst (part time)
- 1939-1940, Checketts Photo, Ogden, Utah, Photographer.

Military Service:

1944-1946, United States Navy, the first six weeks a Seaman First Class and the balance of the two years as Ensign.

Consulting:

1957- for various governmental, industrial, and educational organizations (over 50 in number).

Business:

1966- co-founder of Megadiamond Industries (current name) with Bill J. Pope and M. Duane Horton. President and Board Chairman 1966-1972; Vice President and Board Chairman 1972-1975; Vice President for Advanced Research 1980-.

1957- H. Tracy Hall, Inc., President

Other Responsibilities: Director of the Brigham Young University High Pressure Laboratory, 1955-1980. Director of the National High Pressure Data Center run by Brigham Young University for the U.S. National Bureau of Standards, 1965-1980.

Professional Society Membership:

American Chemical Society, American Association for the Advancement of Science, American Physical Society, Sigma Xi, Phi Kappa Phi, Utah Academy of Science Arts and Letters, Timpanogos Club of Utah

Major Scientific Achievments:

 The invention of the "Belt" high pressure/high temp erature apparatus, U.S. patent No. 2,941,242 issued
June 1960. Conception and reduction to practice occured in 1953 but issuance of the patent was delayed by a U.S.
Government secrecy order based on the invention's importance.

This device can simultaneously maintain a pressure of one million pounds per square inch and a temperature of 2000 ,degrees centigrade. It is used throughout the world in the commercial manufacture of industrial diamond. An estimated one billion carats (about 200 tons) of diamond has been made since the Belt was invented. The Belt has been the subject of world-wide litigation, the most notable case being General Electric vs. the South African Chamber of Mines. This invention is the most highly cited invention in the field of high pressure and was so recognized in "This Week's Citation Classic", <u>Current Contents</u>, ISI Press 41, 14, 1980.

 The first synthesis of diamonds, a feat that had eluded scientists for at least 150 years. The Belt made this possible.
U.S.patents Nos. 2,947,608 & 2,947,610, 20 August 1960.

3. The invention of the tetrahedral press, U.S. patent No. 2,918,699, 29 December 1959. This invention circumvented the proprietary interest of the General Electric Company which prevented H. Tracy Hall from using the Belt for scientific research after leaving that company in 1955.

It has the same pressure-temperature capabilities as the Belt. Note that the patent on this was granted before that on the Belt. This device also became subject to a U.S. Government secrecy order.

4. Sintered diamond, a synthetic carbonado first introduced to the world by Megadiamond Industries 24 September 1970. This type of material is rapidly revolutionizing the industrial diamond industry. U.S.patents 3,816,085; 3,829,544; 3,913,280, see-Sciencey,-169,_868-69 (1970).

- 5. The determination of the first melting curve under high pressure, high temperature conditions. The material studied was germanium. See J. Phys. Chem. 59, 1144-1146 (1955)
- The first high pressure, high temperature X-ray diffraction apparatus (with J. Dean Barnett) U.S. patent No. 3,249,7533. Also, see Rev. Sci. Instrum. 35, 175-182 (1964).
- 7. Discovery of the first pressure induced phase change from a close-packed to a non-close-packed structure (FCC to BCC in Ytterbium at 40kbar) with J. Dean Barnett and Leo Merrill. Such a change was thought to be impossible before this discovery. The article disclosing this transformation appeared in Science 139, 111-112 (1963) and was featured on the front cover of that issue.
- The determination of the nature of the "resistance cusp" in cesium. This intriguing problem had remained unsolved since the discovery of the cusp by P. W. Bridgman in 1951 (with Leo Merrill and J. Dean Barnett) See Scienc, 146, 1297-1299 (1964)
- 9. The synthesis of over 100 rare earth compounds and poly morphs, impossible to synthesize by conventional means. They were prepared by the application of high pressure, high temperature techniques and "are described in a series of 20 papers beginning with an article by Norman L. Eatough and H. Tracy Hall entitled "High Pressure Synthesis of Rare Earth Diantimonides", Inorg. Chem. <u>8</u>, 1439 (1969)

HONORS, AWARDS, AND DISTINCTIONS OF H. TRACY HALL

1980

- 1. HONORARY DOCTORATE OF HUMANITIES, Weber State University, June 12, 1987.
- 2. Admitted to practice patent law before the U.S. Patent and Trademark Office.
- 3. Listed in "Who's Who In The World".
- 4. U.S. Patent No. 22,941,242, H. Tracy Hall's Belt Apparatus recognized as the most highly cited publication in the field of High Pressure and featured as THIS WEEKS CITATION CLASSIC" in <u>Current Contents. ISI Press</u> 41, 14, 1980. Over 98% of the worlds billions of carats of manufactured diamonds utilize his Belt, his Cubic Press (U.S. Patent No. 3,440,687) and his Anvil Guide (U.S. Patent No. 3,182,353).
- 5. " MAN OF THE YEAR AWARD", Abrasive Engineering Society, Milwaukee, Wisconsin.
- 6. Retired from Brigham Young University as "DISTINGUISHED PROFESSOR EMERITUS".

- 1980 Admitted to practice patent law before the U.S. Patent and Trademark office as an agent, registration number 29,800, June 23.
- 1980 "Man of the Year Award," Abrasive Engineering Society, Milwaukee, Wisconsin
- 1978 "Karl G. Maser Research Award," Brigham Young University, Provo, Utah August 31
- 1977 "International Prize for New Materials," The American Physical Society, San Diego, California, March 22
- 1975 "Distinguished Alumni Award," Weber State University, Ogden, Utah, October 16
- 1974 "IR-100 Award," Industrial Research Magazine for Indexible Sintered Diamond Tools, Chicago, October 8
- 1973 "Engineering Materials Achievement Award," The American Society for Metals, Chicago
- 1973 American Chemical Scoeity Tour Speaker (Texas, Louisiana)
- 1973 The American Society for Metals "Engineering Materials Achievement Award," Chicago, October 2, Conrad Hilton Grand Ballroom
- 1972 American Chemical Society Tour Speaker (Oregon, Washington)
- 1972 Fellow, The American Institute of Chemists
- 1972 The Intermountain Society of Inventors and Designers "Certificate for Distinguished Service and Leadership in the Field of Invention and Designing," Salt Lake City, Utah, May 20
- 1972 Fellow, The Utah Academy of Science, Arts, and Letters
- 1972 The American Chemical Society "Award for Creative Invention," Boston, Massachusetts, April 10 (Gold Medal)for being the first person to synthesize diamond.
- 1971 Honorary Doctor of Science Degree, Brigham Young University, Commencement Exercises, Provo, Utah, May 28
- 1971 "Outstanding Manhood Award," presented by Associated Men Students, Brigham Young University, Provo, Utah April 13
- 1970-1973 Member of National Academy of Science-National Research Council Evaluation Panel for the National Bureau of Standards Heat Division
- 1970 Cortez Honors Lecture, Weber State University, December 10, Ogden, Utah
- 1970 American Institute of Chemises "Chemical Pioneer Award," Pittsburgh, Pennsylvania, May 16
- 1968- Member of Joint Army-Navy-Air Force Thermochemical Tables Advisory Group
- 1967- Distinguished Professor of Chemistry and Chemical Engineering, Brigham Young University

1967 Robert A. Welch Foundation, "Lecturer in Chemistry," Texas Universities

1966-1969 Member of Editorial Board, "The Review of Scientific Instruments"

- 1965 The American Chemical Society, Salt Lake Section's "Utah Award," University of Utah, Salt Lake City, December 9
- 1965 The National Association of Manufacturer's "Modern Pioneers in Creative Industry Award," The Waldorf Astoria, New York City, December 2
- 1965 The Brigham Young university's "James E. Talmage Scientific Achievement Award," Baccaluareate Exercises, Provo, Utah, May 27
- 1964 Third Annual "Olin Mathesen Lecture," Yale University, New Haven, Connecticut, April 22
- 1964 First "Annual Faculty Lecture," Brigham Young University, Provo, Utah, April 8
- 1962 The American Society of Tool and Manufacturing Engineers "Research Medal," New York City
- 1961-1964 Member of Editorial Board, "Inorganic Chemistry"
- 1960-1961 President, Utah Academy of Sciences, Arts and Letters
- 1960 Fellow, American Association for the Advancement of Science
- 1959-1963 Alfred P. Sloan Foundation Research Fellow
- 1959 Chairman, Salt Lake Section, American Chemical Society
- 1954 First to synthesize diamond, December 16, G.E. Research Lab, Schenectady, New York

PUBLICATIONS OF H. TRACY HALL

- 1. Graham W. Marks and H. Tracy Hall, "A Method for the Spectrochemical Determination of Germanium, Tin and Lead in Ore Samples," U. S. Bureau of Mines Report of Investigations No. R.I. 3965, Nov. 1946, 38 pp.
- 2. H. Tracy Hall and Henry Eyring, "The Constitution of Chromic Salts in Aqueous Solution," J. Am. Chem. Soc., 72, 782-790(1950).
- 3. Graham W. Marks and H. Tracy Hall, "Transmission Characteristics in the Visible Spectral Region of the Quinalizarin and Beryllium - Quinalizarin Complex in N/4 Sodium Hydroxide Solution," U. S. Bureau of Mines Report of Investigations No. 4741, Oct. 1950, 5 pp.
- 4. H. Tracy Hall and Raymond M. Fuoss, "Empirical Analysis of Viscosity Data," J. Am. Chem. Soc, 73, 265-269 (1951).
- 5. H. Tracy Hall, "Molecular Weight of Polytrifluorochloroethylene by Light Scattering," J. Polymer. Sci., 7, 443-447 (1951).
- 6. H. Tracy Hall, Edward L. Brady and Paul D. Zemany, "Viscosity of Polytrifluorochloroethylene in O-Chlorobenzotrifluoride," J. Am. Chem. Soc, 73, 5460 (1951).
- 7. H. Tracy Hall, "The Solubility of Polytrifluorochloroethylene, "J. Am. Chem. Soc., 74, 68-71 (1952).
- 8. H. Tracy Hall, "A New Method of Mounting Diamonds," Rev. Sci. Instrum., <u>25</u>, 1035-1036 (1954).
- 9. F. P. Bundy, H. T. Hall, H. M. Strong and R. H. Wentorf, "Man-Made Diamonds," Nature, <u>176</u>, 51-54 (1955).
- 10-. H. Tracy Hall, "The Melting Point of Germanium as a Function of Pressure to 180,000 Atmospheres," J. Phys. Chem., 59, 1144-1146 (1955).
- 11. H. Tracy Hall, "Chemistry at High Temperature and High Pressure," Research and Engineering, 11, 27-28 (1956).
- 12. H. Tracy Hall, "Chemistry at High Temperature and High Pressure," High Temperature--A Tool for the Future, Stanford Research Institute, Menlo Park, California, 161-166(1956).
- 13. H. Tracy Hall, "What the Sunday School has Done for Me," The Instructor, <u>91</u>, 341 (1956).
- 14. H. Tracy Hall, "Chemistry at High Pressures and High Temperatures," J. Wash. Acad. Sci., 47, 300-304(1957).
- 15. H. Tracy Hail, Billings Brown, Bruce Nelson and Lane A. Compton, "I. An Apparatus for Use with Condensed Phases at 10,000 Deg. II. Some Thermodynamic Considerations at Very High Temperatures," J. Phys. Chem., <u>62</u>, 346-351 (1958).
- 16. H. Tracy Hall, "Some High Pressure, High Temperature Apparatus Design Considerations: Equipment for Use at 100,000 Atmospheres and 3000 Deg. C, "Rev. Sci. Instrum., <u>29</u>, 267-275 (1958).

- 17. H. Tracy Hall, "Ultrahigh Pressure Research," Science, <u>128</u>, 445-449 (1958).
- 18. H. Tracy Hall and S. S. Kistler, "High Pressure Developments," <u>Annual</u> <u>Review of Physical Chemistry</u>, Annual Reviews, Inc., Palo Alto, California, 395-416 (1958).
- 19. H. Tracy Hall, "Diamonds," <u>Proceedings of the Third Conference on Carbon</u> (held at University of Buffalo, Buffalo, N. Y., June 1957), Pergamon Press, London, pp. 75-84.
- 20. H. Tracy Hall, "Ultrahigh Pressures," Sci. American, <u>201</u>, 61-67(1959).
- H. Tracy Hall, "High Pressure Methods," <u>Proceedings of an International Symposium on High Temperature Technology</u>, Asilomar Conference Grounds, California, Oct. 6-9, 1959; Arranged by Stanford Research Institute, Menlo Park, California, McGraw-Hill, New York, pp. 145-156 and 355-336 (1960).
- 22. H. Tracy Hall, "Ultrahigh Pressure, High Temperature Apparatus: The Belt, "Rev. Sci. Instrum., 31, 125-131 (1960).
- 23. H. P. Bovenkerk, F. P. Bundy, H. T. Hall, H. M. Strong, and R. H. Wentorf, Jr., "The Preparation of Diamond," Nature, <u>184</u>, 1094-1098 (1959).
- 24. J. Duane Dudley and H. Tracy Hall, "Experimental Fusion Curves of Indium and Tin to 105, 000 Atmospheres," Phys. Rev., <u>118</u>, 1211-1216 (I960).
- H. T. Hall, "Some High Pressure, High Temperature Apparatus Design Considerations: Equipment for use at 100,000 Atmospheres and 3000°C, "Series of Selected Papers in Physics, Solid State Physics in High Pressure, The Physical Society of Japan, Dept. of Physics, University of Tokyo, Japan (1960), pp. 6-14, Reprinted from Rev. Sci. Instrum., <u>29</u>, 267-275 (1958).
- 26. H. Tracy Hall, "High Pressure Apparatus," <u>Progress in Very High Pressure Research</u>, Proceedings of an International Conference Held at Bolton Landing, New York, June 13-14, 1960, edited by Bundy, Hibbard, and Strong (John Wiley and Sons, Inc., Publishers), pp. 1-9 (1961).
- 27. H. Tracy Hall, "Possible Future Roles of the Utah Academy," Presidential Address, <u>Proceedings, Utah Academy of Sciences, Arts and Letters, 38, 8-10 (1961).</u>
- 28. H. Tracy Hall, "The Synthesis of Diamond," J. Chem. Educ., 38, 484-489 (1961).
- 29. H. Tracy Hall, "Anvil Guide Device for Multiple-Anvil High Pressure Apparatus," Rev. Sci. Instrum., 33., 1278-1280(1962).
- 30. H. Tracy Hall, J. Dean Barnett and Leo Merrill, "Ytterbium: Transition at High Pressure from Face-Centered Cubic to Body-Centered Cubic Structure," Science, <u>139</u>, 111-112 (1963).
- 31. H. Tracy Hall and Leo Merrill, "Some High Pressure Studies on Ytterbium," Inorg. Chem., 2, 618-624(1963).

- 32. H. Tracy Hall, "High Pressure, High Temperature," pp. 730-738, Perspectives in Materials Research, edited by L. Hinnel, J. J. Harwood, and W.J. Harris, Jr., Office of Naval Research, Dept. of the Navy, Washington, D. C., Surveys of Naval Science, No. 10, February 1963. (Note: This material was written four years before the book was published).
- 33. J. Dean Barnett, Roy B. Bennion, H. Tracy Hall, "High Pressure X-ray Diffraction Studies on Barium," Science, <u>141</u>, 534-535 (1963).
- 34. H. T. Hall, "High Pressure Apparatus," in <u>The Physics of High Pressures</u>, edited by K. Swenson, (in Russian) published in Moscow (1963). This paper translated from my paper presented at the International High Pressure Conference held at Bolton Landing, New York, June 13-14, 1960.
- 35. J. Dean Barnett, Roy B. Bennion, H. Tracy Hall, "X-ray Diffraction Studies on Tin at High Pressure and High Temperature, "Science, <u>141</u>, 1041-2 (1963).
- 36. Donald R. Hall and H. Tracy Hall, "Missionary Diary of Helon Henry Tracy in the United States and Great Britain 1881-1882," private publication, 158 pp., 300 copies printed (1963).
- 37. H. Tracy Hall, "High Pressure /Temperature Apparatus," chapter 4 (pp. 133-179), Metallurgy at High Pressures and High Temperatures, edited by K. A. Gschneidner, Jr., M. T. Hepworth, and N. A. D. Parlee; Gordon and Breach Science Publishers, New York, 1964.
- 38. J. Dean Barnett and H. Tracy Hall, "High Pressure-High Temperature X-ray Diffraction Apparatus," Rev. Sci. Instrum., <u>35</u>, 175-182(1964).
- 39. H. T. Hall, "Polymorphism and High Pressure," B. Y. U. Studies, <u>5</u>, 139-153 (1964).
- 40. H. T. Hall, Guest Editorial, "High Pressure," Experimental Mechanics, 4, 3-A (1964).
- 41. H. T. Hall, L., Merrill and J. D. Barnett, "High Pressure Polymorphism in Cesium," Science, <u>146</u>, 1297-1299 (1964).
- 42. H. T. Hall, "High Pressure Chemistry," in <u>Progress in Inorganic Chemistry</u>, Vol. 7, edited by F. A. Cotton, Interscience Publishers, pp. 1-38 (1966).
- 43. H. T. Hall, "Periodic Compounds: Syntheses at High Pressures and Temperatures," Science, <u>148</u>, 1331-1333 (1965).
- 44. H. T. Hall and L, A. Compton, "Group IV Analogs and High Pressure, Temperature Synthesis of B₂0," Inorg. Chem., 4, 1213-1216 (1965).
- 45. W. E. Evenson and H. T. Hall, "Volume Measurements on Chromium to 30 kilobars," Science, <u>150</u>, 1164-1165 (1965).
- 46. J. D. Barnett, V. E. Bean, and H. T. Hall, "X-ray Diffraction Studies on Tin to 100 Kilobars," Journal of Applied Physics, <u>37</u>, 875-877 (1966).
- 47. R. N. Jeffery, J. D. Barnett, H. Vanfleet, H. T. Hall, "A Pressure Scale to 100 Kilobar Based on Compression of Sodium Chloride, "J. Appl. Phys., 37, 3172-3180 (1966).

- 48. H. T. Hall, "Hydraulic Ram Design for Modern High Pressure Devices," Rev. Sci. Instrum., <u>37</u>, 568-571 (1966).
- 49. H. T. Hall, "A Tetrahedron Problem," Math. Mag., <u>38</u>, 241 (1965). prob. No. 598.
- 50. H. T. Hall, "A Scientist Looks at the Miracles of Jesus, " The Instructor, <u>101</u>, 86-87 (1966).
- 51. H. T. Hail, "Transformations in Solids at High Pressure," <u>Proceedings of Fourth Meeting</u>, Cape Kennedy, Florida, March 16-18, 1966, Thermochemistry Working Group, Interagency Chemical Rocket Propulsion Group, Chemical Propulsion Information Agency, Publication No. 108, June 1966, Volume 1, pp. 67-74.
- 52. H. T. Hall, "High Temperatures, High Pressures, and Periodic Compounds," <u>High-Temperature Chemistry</u>, National Academy of Sciences, National Research Council Publication 1470, Washington, D. C., 1967, pp. 65-66.
- 53. H. T. Hall (Book), "High Pressures (to 100,000 Atmospheres at 1500 Deg. C)," American Institute of Chemical Engineers, Today Series, 345 West 47th St., New York, N. Y. 10017, (1967), 179 pp.
- 54. H. T. Hail, "High Pressure Apparatus: Ram-In-Tie-Bar Multianvil Presses," Rev. Phys. Chem. Japan, <u>37</u>, 63-71 (1967).
- 55. R. B. Bennion, H. G. Miller, W. R. Myers, H. T. Hall, "100 Kbar Press for Timeof-Flight Neutron Diffraction," Acta Cryst., <u>25A</u>, S71 (1969).
- 56. N. L. Eatough and H. T. Hall, "High Pressure Synthesis of Rare Earth Diantimonides," Inorg. Chem., <u>8</u>, 1439 (1969).
- 57. N. L. Eatough, Alan W. Webb, and H. T. Hail, "High Pressure Th₃P₄-Type Polymorphs of Rare Earth Sesquichalcogenides," Inorg. Chem., <u>8</u>, 2069-2071 (1969).
- 58. J. D. Barnett, J. Pack, and H. T. Hall, "Structure Determination of a Ferroelectric Phase of Sodium Nitrate Above 45 Kilobar," <u>Proceedings of the Symposium on Crystal</u> <u>Structure at High Pressure at Pacific Science Center, Seattle, Washington, March 24, 1969,</u> Transactions of the American Crystallographic Association, <u>5</u>, 113-131 (1969), Available from Polycrystal Book Service, P. O. Box 11567, Pittsburg, Pennsylvania 15238.
- 59. N. L,. Eatough and H. T. Hall, "High Pressure Synthesis of Lutetium Diantimonide," Inorg. Chem., <u>9</u>, 416-417 (1970).
- 60. N. L. Eatough and H. T. Hall, "High Pressure Th₃P₄-Type Polymorphs of Rare Earth Sesquiselenides," Inorg. Chem., 9, 417-418 (1970).
- 61. A. W. Webb and H. T. Hall, "High Pressure Synthesis of Rare Earth Polyselenides," Inorg. Chem., <u>9</u>, 843-847 (1970).
- 62. A. W. Webb and H. T. Hall, "High Pressure Synthesis of Rare Earth Polysulfides," Inorg. Chem., <u>9</u>, 1084(1970).
- 63. H. T. Hail, "High Pressure Synthesis Involving Rare Earths," Rev. Phys. Chem. Japan, 39, 110-116 (1969).

- 64. J. F. Cannon and H. T. Hall, "High Pressure Synthesis of Selected Lanthanide-Tellurium Compounds," Inorg. Chem., <u>9</u>, 1639-1643 (1970).
- 65. H. T. Hall, "Personal Experiences in High Pressure," The Chemist, <u>47</u>, 276-279 (1970), (Chemical Pioneer Address, American Institute of Chemists, Pittsburg, Pennsylvania, May 19, 1970).
- 66. H. T. Hall, "Sintered Diamond: A Synthetic Carbonado," Science, <u>169</u>, 868-869 (1970).
- 67. H. T. Hall, "The Synthesis of Diamond," in <u>Advances in Chemical Physics</u>, Vol. XXI, entitled, "Chemical Dynamics," (papers in honor of Henry Eyring), Eds. Hirschfelder & Henderson, John Wiley, New York (1971), pp. 721-735.
- 68. H. Tracy Hall, "High Pressure Scale by X-ray Diffraction Techniques up to Approximately 100 kbar," in <u>Accurate Characterization of the High-Pressure</u> <u>Environment</u>, ed. E. C. Lloyd, U. S. Dept. of Commerce, National Bureau of Standards Special Publication 326 issued March 1971, pp. 303-306. Paper presented at the National Bureau of Standards Symposium, Gaithersburg, Md., October 14-18, 1968. Available from the U. S. Government Printing Office, Washington, D. C. 20402 (Order by Catalog No. C 13. 10:326)
- H. Tracy Hall, "Fixed Points Near Room Temperature," in <u>Accurate Characterization of the High-Pressure Environment</u>, ed. E. C. Lloyd, U. S. Dept. of Commerce, National Bureau of Standards Special Publication 326 issued March 1971, pp. 313-314. From National Bureau of Standards Symposium, Gaithersburg, Md., October 14-18, 1968. Available from the U. S. Govt. Printing Office, Washington, D. C. 20402 (Order by Catalog No. C 13. 10:326).
- 70. F. William Linsley, Jr., Jerald S. Bradshaw and H. Tracy Hall, "High Pressure Affects on Conjugated Aromatic Compounds," Rev. Phys. Chem. Japan, <u>40</u>, 69-72 (1970).
- 71. D. L. Decker, W. A. Bassett, L. Merrill, H. T. Hall and J. D. Barnett, "High Pressure Calibration: A Critical Review," J. Phys. Chem. Ref. Data, <u>1</u>, 773-836 (1972).
- 72. Karl A. Miller and H. Tracy Hall, "High Temperature Synthesis of Rare Earth Tri-Tin Compounds," Inorg. Chem., JU, 1188-1191 (1972).
- 73. J. F. Cannon, D. L. Robertson and H. T. Hall, "Synthesis of Lanthanide-Iron Laves Phases at High Pressures and Temperatures," Mater. Res. Bull., <u>2</u>, 5-12 (1972).
- 74. N. L. Eatough, H. T. Hall, "High Pressure Synthesis of REMn₂ Compounds with the MgZn₂ (Laves) Structure," Inorg. Chem., <u>11</u>, 2608-2609 (1972).
- 75. J. F. Cannon, D. L. Robertson, H. T. Hall, "The Effect of High Pressure on the Formation of LRu₂ and LOs₂ (L=Lanthanide) Compounds, "J. Less-Common Metals, <u>29</u>, 141-146 (1972).
- 76. D. L. Robertson, J. F. Cannon and H. T. Hall, "High Pressure and High Temperature Synthesis of LaCo₂," Mat. Res. Bull., <u>7</u>, 977- (1972).
- 77. J.M. Leger and H. Tracy Hall, "Pressure and Temperature Formation of A₃B Compounds. I. Nb₃Si and V3A1, J. Less-Common Metals, <u>32</u>, 181-187 (1973).

78. J.M. Leger & H. Tracy Hall, Pressure & Temperature Formation of A_3B Compounds.

II. Nb_3Ge , Nb_3Sn , Nb_3Pb , "J. Less_Common Metals, 34, 17-24 (1974)

- 79. Karl A. Miller and H. Tracy Hall, "High Pressure Synthesis of Lutetium Trilead," J. Less-Common Metals, 32, 275-78 (1973).
- 80. J., F. Cannon, D. L. Robertson, H. T. Hall and A. C. Lawson, "The Effect of High Pressure on the Crystal Structure of LaOs₂ and CeOs₂," J. Less-Common Metals, 31, 174 (1973).
- 81. A. C. Lawson, J. F. Cannon, D. L. Robertson and H. T. Hall, "Superconduc tivity of LaOs," J. Less-Common Metals, 32, 173-74 (1973).
- J. F. Cannon, D. L. Robertson, H. T. Hall and A. C. Lawson, "High Pressure Synthesis of Beta-W-Type Nb₃Te," J. Phys. Chem. Solids, <u>35</u>, 1181-82 (1974).
- 83. M. D. Horton, B. J. Pope and H. T. Hall, "Sintered Diamond," International Industrial Diamond Association Symposium, Washington, D. C. (1974).
- 84. B. J. Pope, M. D. Horton, H. T. Hall, L. S. Bowman and H. Adaniya, "Sintered Diamond: Its Possible Use as a High Thermal Conductivity Semiconduction' Device Substrate," Proc. 4th International Conference on High Pressure (AIRAPT), Kyoto, Japan (1974).
- 85. B. J. Pope, M. D. Horton, H. T. Hall and S. DiVita, "Selection and Treatment of Diamond Particulates in Preparation for High Thermal Conductivity Ceramics by Sintering at High Temperature and Ultra-high Pressure, "Proc. Ninth Annual University Conference on Ceramic Science of the American Ceramic Society, Orlando, Florida (1975).
- 86. J. F. Cannon and H. T. Hall, "Effect of High Pressure on the Crystal Structures of Lanthanide Trialuminidies," J. Less-Common Metals, 40, 313-28 (1975).
- 87. H. Tracy Hall, "Retraction System for Multi-anvil Presses," Rev. Sci. Instrum., 46, 436-38 (1975).
- H. Tracy Hall, "Sintered Diamond," Brigham Young University Studies, 16, 43-47 (1975) [Special Centennial Issue].
- 89. J. F. Cannon, D. M. Cannon, and H. T. Hall, "High Pressure Synthesis of SmB₂ and GdB₁₂," J. Less Common Metals 56, 83-90, (1977).
- 90. J. F. Cannon, H. T. Hall, "High Pressure Synthesis of Lanthanide/Boron and Actinide/Boron Compounds," in Rare Earths in Modern Science and Technology, edited by G. J. McCarthy and J. J. Rhyne, Plenum Press, New York & London (1978) pp. 219-224.
- 91. Chapter I, "Introduction" to Chemical Experimentation Under Extreme Conditions, Techniques of Chemistry, Vol IX, Eds. A. Weissberger and B. Rossiter, John Wiley & Sons (1980) pp. 1-8.
- 92. Chapter II, "High Pressure Techniques", Ibid. pp. 9-72.
- 93. H.T. Hall, This Week's Citation Classic, Ultra-high pressure, Hightemperature Apparatue: The "Belt," <u>Current Contents</u>, ISI Press, <u>41</u>, 14 (1980).

PATENTS OF H. TRACY HALL

1.	U. S. 2,728,651, December 27, 1955. Assignee: General Electric Company.	"Diamond Abrasive Whe	el. ''	
2.	U. S. 2,918,699, December 29, 1959. "High Pressure Press" (Tetrahedral Anvil Apparatus) Assignee: Research Corporation			
	Foreign Patents and Numbers:	Great Britain	847,067	
		Canada	622,429	
3.	U. S. 2,941,242, June 21, 1960.			
	"High Temperature - High Pressure Apparatus." Assignee: General Electric Company.			
	Foreign Patents and Numbers:	Canada	624,376	
		England	830,209	
4.	U. S. 2,941,248, June 1, 1960.			
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Carnegie Foundation	1956	\$ 10,000
National Science Foundation	1956	25, 500
Office of Ordnance Research (now Army Research Office)	1956	20,000
Linde	1957	300
du Pont	1957	5,000
Office of Ordnance Research (now Army	1957	15,000
Research Office)		
National Science Foundation	1958	85,000
Office of Ordnance Research (now Army	1958	14, 590
Research Office)		,
du Pont	1958	7,500
du Pont	1959	5,000
National Science Foundation (with Dr. Robert	1959	10,800
Egbert)		;
Alfred P. Sloan Foundation	1959	12,000
du Pont	1960	5,000
Army Research Office (with Dr. J. Dean	I960	103, 100
Barnett)	2,00	,
Diamant Boart	1961	1, 500
Alfred P. Sloan Foundation	1961	6, 250
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Army Research Office (with Dr. J. Dean	1962	24, 372
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Atomic Energy Commission (with Dr. J. B.	1967	\$ 27,072
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Dalley and J. D. Barnett)		
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Support)		
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Support)		
National Science Foundation	1972	30,000
National Bureau of Standards (Data Center	1972	30,600
Support)		
National Bureau of Standards	1973	30,600
National Science Foundation	1974	31,100
	TOTAL	\$ 1,304,945

Sintered Diamond

M. D. HORTON BILL J. POPE H. TRACY HALL Megadiamond Corp. and Brigham Young University

Introduction

During the past few years, researchers have learned to reconstitute diamond powder into useful products. These different products are proving to be useful with some applications being competitive with single crystal diamonds. For this reason, if for no other, anyone dealing with diamonds would be interested in the new materials.

Large quantities of fine diamond powders are the by-product of sizing and shaping operations carried out on both natural and synthetic diamond crystals. Because fine diamond powders have relatively limited applications, there is a resultant large excess of supply over demand. This leads to the powders having a comparatively low value.

Extensive research has been devoted to developing a process that can take cheap diamond powder and reconstitute it. Several processes have been developed and, judging from the resultant materials, each process is different. Were information available, this article would deal with such materials in general. However, proprietary secrecy has severely restricted the available technical material. For that reason, the balance of the article will deal with a particular sintered diamond product called Megadiamond.

Described in the next section will be the bonding process whereby diamond powder can be fused together to produce large, coherent pieces of a desired shape. Subsequent sections will then describe the properties of the sintered diamond material.

Process

The sintering of the diamond powders is accomplished by placing the suitably prepared powders in a mold. Then, the mold is placed in an ultra-high pressure press and subjected to a very high pressure. While in this high pressure field, the diamond powder is heated to such a temperature that it fuses together. Of course, each of these steps is more complex than described but this is an overview of the process.

From the information presented thus far, the reader would conclude that only fine diamond powders can be used together. This is not so. Particles as large as 150 and as small as 0.1 microns have been tested and successful fusion was achieved. However, in all cases, it was necessary that the diamond be properly prepared.

Also, there are almost no restrictions on configuration of the mold into which the diamond is placed. Figure 1 shows the mold used to make a cylindrical sintered diamond and Figure 2 shows several of the shapes that have been made. In many cases where a complicated shape is to be made, the greatest difficulty lies not in sintering the diamond but rather in preparing the mold.

Figure 3 is a picture of a cubic press closing upon the sample of Figure 1. Once the press is closed, the hydraulic pressure behind the rams is increased until the desired pressure inside the mold is achieved. Then, an electrical current is passed through the heater tube to create the required temperature for the necessary length of time. Next, the pressure is released and the press opened. After this, the mold is retrieved then the sintered diamond product recovered.

A wide selection of pressure, temperatures and times can be used although better results are obtained if the temperature is high enough that the diamond tends to revert to graphite and the pressure is as high as possible. Reference to the equilibrium diagram for the graphite-diamond system shown in Figure $4^{(1)}$ will show that at

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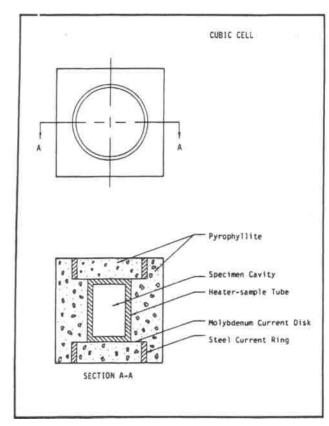


FIGURE 1. Typical mold used to make a sintered diamond.

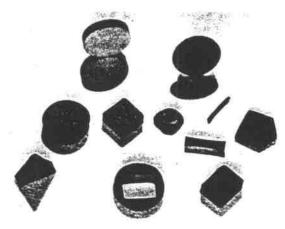


FIGURE 2. Examples of sintered diamond shapes.

any pressure a high enough temperature will cause diamond to convert to graphite. At room pressure and temperature graphite is more stable than graphite but the rate of conversion is infinitely slow. Above 1500°K however, the conversion rate becomes significant and above 2000° K very fast. Nevertheless, the sintering process works best when the temperature is slightly higher than the apparent equilibrium value.

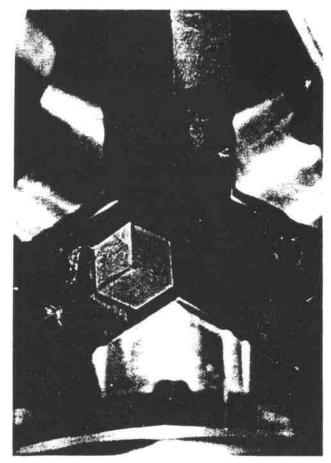


FIGURE 3. Cubic Press closing on mold shown in Figure 1.

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Hall.

n 1935 and raised in Salt Lake City, attended the University of Utah. B.S. and Ph.D. in Chemical Enree being a-warded in 1961. After in the field of solid pro-pellant ce, California. In 1963 he accepted igham Young University in the Engineering. There he began work d under the tutelage of H. Tracy Subsequently, he became

interested in diamond, its synthesis, uses, and properties. Ultimately, this interest caused him to join Tracy Hall and Bill Pope in forming a company to develop and exploit sintered diamond. Presently he occupies dual positions as Professor of Chemical Engineering and Vice President of Megadiamond Corporation.

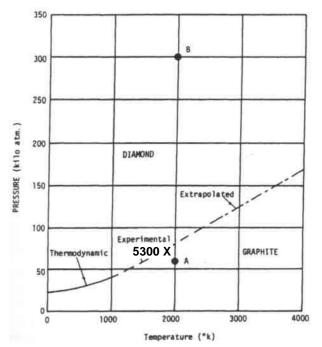
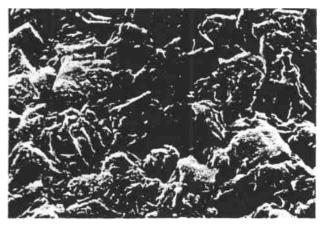


FIGURE 4. Solid carbon phase diagram.



35000 X

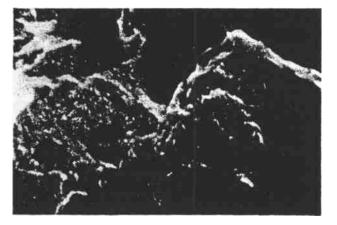


FIGURE 5. Enlarged views of sintered diamond surface.

Figure 5 shows a photomicrograph of the polished surface of a sintered diamond. It is visually apparent that the diamond particles have softened and the contact points fused together. This is astonishing since diamond has not been known to melt or even to soften. Temperatures that might be high enough to cause softening seem only to cause a rapid conversion of diamond into graphite, even at high pressures.²

Thus the bonding process contains two apparent peculiarities. First, the best bonding occurs when the temperature is such that the diamond ought to convert to graphite. Second, the diamond seems to soften, flow and fuse under conditions not known to make diamond behave in that fashion.

A more detailed consideration of the process suggests a rational explanation of these phenomena. Ordinarily, the pressure is estimated by considering the sample to be homogeneous and to transmit the pressure hydrostatically. This estimated pressure is low enough that the diamond should transist to graphite. However, the sample is not homogeneous nor is the pressure within the diamond powder transmitted hydrostatically. Instead, the externally applied force is concentrated at the diamond contact points and the resultant pressure at these points is so high that diamond is stable.

This can be understood better if a specific case is examined. For example, one where the pressure applied to the exterior of the diamond powder sample is 60 kilobars and the temperature is 2000° K (Point A on Figure 4). Figure 6 depicts an idealization of a cut-away view of the sample under these conditions. The forces on any segment of the sample must be balanced so that the "hydrostatic" pressure times the end area of the sample must be equal to the sum of the forces exerted on the diamond contact points. During the compression and packing process, these contact areas become as large or perhaps twenty percent of the cross sectional area. Therefore

(Applied pressure) x (End area) = (Contact pressure) x (Contact area) or (60 kbars) x (End area) = (Contact pressure) x (0.2 end area)

and the pressure at the contact points would be 300 kbars. Thus, point B on Figure 4 is actually representative of the conditions at the contact

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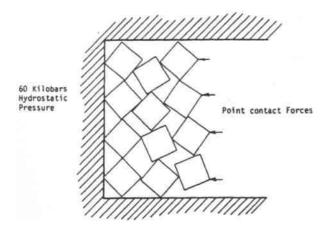


FIGURE 6. Idealized depiction of sintered diamond cross section.

points. It can be seen that this is well within the diamond stable regime so at the contact points where the fusion takes place, diamond is the stable species and no graphitization occurs to hinder the fusion process.

Also, we suggest that diamond does achieve a degree of plasticity at 2000°K but it is not ordinarily observed because the formation of graphite obstructs observation. Only when graphite formation is prevented by the special and unexplored conditions described above can the softening be observed or inferred. More particularly, only under these conditions can the slightly softened diamond be forced together for a period long enough that distortion and fusion can take place. By this explanation, the peculiarities of the bonding process disappear and are replaced by ordinary sintering behavior that is often observed with other materials.

It has been found that (See Figure 5) if pure diamond powder is used to make a sintered piece, there are many minute holes left in the matrix with the resulting apparent density ranging from 3.1 to 3.48 g/cm³. A stronger sintered product can be made if these holes contain a filler material. This is done by mixing diamond powder with another powder such as boron carbide or silicon carbide prior to placing the mixture in the mold. When the mixture is sintered, the diamond-diamond contact points fuse as described above. However, additional bonds between the filler and the diamond contribute further strength to the composite material.

Electrical Properties

From a scientific point of view, the unusual electrical properties of some sintered diamonds

are perhaps the most interesting. These properties are the result of a thin graphite film that forms on the diamond powder surfaces adjacent to the microscopic holes. Such a film forms in the manufacturing process because at these surfaces, there is not a pressure intensification effect. Therefore a high temperature causes some graphitization of these diamond surfaces.

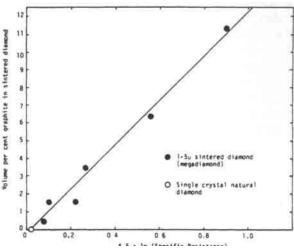


FIGURE 7. Specific resistance (in ohm-cm) versus graphite to diamond ratio in sintered diamond.

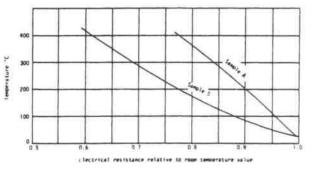


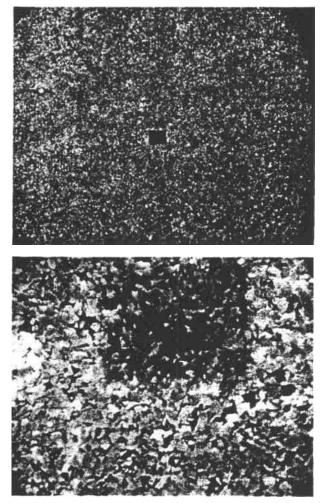
FIGURE 8. Sintered diamond electrical resistance as a function of temperature.

The most obvious electrical property of the reconstituted diamond is that it is conductive, and the conductivity correlates with the graphite content as is shown in Figure 7^3 . The amount of graphite and hence the electrical conductance can be controlled by proper selection of manufacturing parameters. Use of either a lower pressure or a longer heating time increases the thickness and hence the total amount of graphite film covering the particles. Interpolation of Figure 8 indicates that the electrical resistance (the inverse of conductance) is linear over the extraordinarily large temperature range

of 400° C. Thus the material has the properties of an excellent thermistor.

Curiously enough, the sintered diamond can be either a P or N type semiconductor with the amount of graphite present determining the type. Regardless of the amount of graphite present, the material luminesces with a blue light when subjected to a high voltage.

An even more peculiar property is illustrated in Figure 9 which is a photomicrograph of a polished. Megadiamond surface upon which an electron scanning microscope beam had focused. The larger dark image represents a discoloration produced at a lesser magnification while the darker, inner image resulted from the beam of a greater magnification. The surface retained the "photograph" of the beam but the cause of the retention is not known.



2000 X

FIGURE 9. Photomicrograph of Electron Beam Image Effect.

Other Properties

Not only the electrical but also the physical properties of sintered diamond are interesting. In the preferred form, micron-sized diamond particles are fused together. Consequently, on a macroscopic scale Megadiamond is homogeneous and isotropic. In this aspect the material is similar to carbonado. By contrast, single-crystal diamonds have different properties along different crystal axis. Being composed of randomly oriented particles, sintered diamond has a hardness and wear resistance representative of the "average" properties along the various axis of a diamond crystal. This causes it to be, like carbonado, extremely wear resistant if the wear forces are not too great. Another similarity is that, like carbonado, sintered diamond is much tougher than single crystal diamonds and does not fracture readily. When it does fracture, a rough, irregular surface is formed. Also, like carbonado, the material usually has a dark, almost black, color. If the manufacturing parameters are carefully controlled a light gray color results but this result is achieved with difficulty.

Probably due to the graphite present in sintered diamonds, they are wetted by some solders*. This property contrasts with that of other diamond which is not wetted by conventional solders.

In one way, sintered diamond is similar to single diamond crystals. Diamond has the highest known thermal conductivity (9 watts/cm°C for type I and 25 for Type IIa at room temperature) and that of sintered diamond can be nearly as large (8 watts/cm°C).⁶

The chemical properties of sintered diamond are essentially the same as those of diamond crystals. The only two known differences are slight. First, the graphite film can be leached from the sintered piece by the use of a powerful oxidizing solution. This leaves a mechanically strong, white, electrically resistive sample. Second, the sintered diamond oxidizes more readily than natural diamond; probably because of strains resulting from the sintering process.

Wear Resistance

In still another way this material differs from most single-crystal diamond. A large diamond

*An example of such a solder is Handy and Harmon 1175-1225.

can be lapped so that it has a sharp edge and can then be used as a cutting tool. After a certain amount of use the cutting edge of a single-crystal diamond becomes worn and rounded. Then the cutting forces become high and a poor finish is left on the workpiece so the tool must be refinished. In contrast to this, the cutting edge of a worn and rounded sintered diamond will continue to cut with relatively smaller cutting forces while the workpiece finish is still relatively good. (This property is more apparent with the filled type of sintered diamond.) Apparently, this is because each microscopic particle acts as an individual cutting point. As one particle becomes worn away another is exposed and the edge continues to cut well. Furthermore, the wear rate is not dependent upon the tool orientation as it is with a single-crystal. This is because as was mentioned earlier, sintered diamond ic macroscopically homogeneous.

The above result is somewhat dependent on the material being machined as sometimes the sintered-diamond tool wears just as fast as conventioned diamond tools. This was the case when sintered diamond was used to cut the hardest grades of carbon and also some fiberglass-filled phenolics. Here also, sintered diamond that does not contain a filler usually performs more poorly than the filled material.

The comparative wear rates of sintered diamond and tungsten carbide have also been measured in several tests. A sintered diamond cutting tool insert examined under comparable conditions lasted fifty times longer in cutting melamine plastic⁴. When the material cut was on 88/12 Al/Si allov, the sintered diamond had a negligible wear after cutting an amount of allow that wore out a tungsten carbide insert. Still other results of this nature are shown in Figure 10, where data for machining 390 aluminum alloy are shown.⁵ This very abrasive alloy would wear out a tungsten carbide cutting tool much more rapidly. For example, at a cutting speed of 1,000 surface feed per minute, the sintered diamond tool life was 140 times as great as that of C-2 carbide.

Conclusion

From the discussion presented above, it can be seen that sintered diamond is a new and uni-

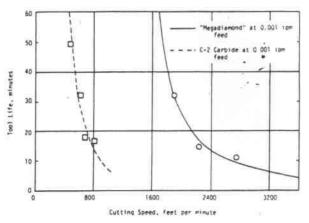


FIGURE 10. Cutting speed/tool life comparison for sintered diamond and carbide tool cutting AA 390 aluminum alloy.

que material. Of all types of diamond, it is most closely related to carbonado. However, it differs in many ways. One is that a desired shape is achieved more easily because the diamond powders can be sintered into almost any desired shape. Also, it has electrical properties that are dependent on manufacturing parameters and differ from those of other diamonds. Furthermore, its mechanical and chemical properties can be improved by the use of select fillers incorporated into the powder. Thus, this new technology adds a new dimension to the use of an already versatile substance—diamond.

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