ABSTRACT

This work is concerned with the behavior of viscous fluids (such as lubricants) as they become solids. It is motivated by the need for such information in engineering applications.

Recent studies regarding the lubrication of bearings and gears, specifically the area of elastohydrodynamic phenomena, have failed to clarify why lubricants do not provide adequate operating life at very high pressures and speeds and there are many questions as to the lubricant properties under these conditions.

The thermodynamic equilibrium or phase diagrams will be obtained for synthetic lubricants which are chemically identifiable and uniform and will be determined over a temperature range from 0° C to 70° C and a pressure range from 1 to 30,000 atmospheres. Additionally, the latent heat of the liquid-solid phase change will be obtained by using the Clapeyron equation and entropy data can then readily be generated. The method proposed is one originally used by P. W. Bridgeman at Harvard University to obtain polymorphisms of solids. ^(1, 2, 3) A device based on Bridgeman's apparatus was designed at the University of Dayton and is available for the test program suggested in this project.

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SECTION 1 INTRODUCTION

Lubricants used in rolling/sliding contact surfaces, as found in gears and bearings have overwhelmingly been characterized as viscous fluids. There are, of course, some solid lubricants and also some impregnated lubricants but the viscous fluids are the most widely used lubricants. As the loads and relative speeds of the contact surfaces have increased, the demands placed on lubricants have increased. Analytically, mechanics and applied mathematics have been employed to predict the lubricant behavior and this, in turn, has allowed for suitable design selections of the mechanical gears and bearings as well as criteria for lubricant material properties. In particular, elastohydrodynamics (EHD) theory has provided a good understanding of the dynamics of lubrication for rolling/sliding contacts over the major regime of loads and speeds. (4, 5)

Unfortunately, the solutions needed to provide specific information for design engineers and scientists are predicated upon an accurate model or description of the lubricant. Viscosity describes quite well a viscous fluid so that a mathematical model based only on viscosity has been heretofore quite adequate in solving problems. Yet, today certain high load and speed applications have shown that theory does not describe the physical happenings accurately. Thus under high loads it is apparent that the viscous fluid model does not describe accurately the lubricant in its dynamic states. Suggestions that the lubricant solidifies or acts as a visco-elastic or plastic material at high pressures have been made, ⁽⁶⁾ yet experimental verifications have not been completed. What is lacking at this time are accurate and more complete p-V-T or thermodynamic phase equilibrium diagrams of the lubricants.

For example, Trachman and Cheng⁽⁷⁾ state that EHD theory is far from adequate in predicting the friction forces between two heavily loaded

contact surfaces. They cite a complete lack of data at high pressures as one of the major reasons for this problem.

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Thus, a void exists in the information regarding behavior of lubricants at extremely high pressures. Thermodynamic equilibrium diagrams are based upon static conditions and represent a logical starting point in observing the behavior of lubricants at high pressures. The role of the thermodynamic equilibrium diagrams is analogous to that of the equilibrium phase diagrams of metals in metallurgy.

SECTION 2

OBJECTIVES

The University of Dayton is proposing a program with the following objectives:

- Obtain thermostatic (p-V-T) data on selected lubricants which can be chemically identified as to structure and degree of uniformity. This data would provide the important information of the thermodynamic phases in a static condition.
- Determine heats of transition (such as heat of solidification) which may thermally contribute to the materials behavior under high pressures.

This research will be conducted using the advanced state-of-the-art instrumentation and equipment and will seek to determine the dependence of the thermostatic data with time. That is, this proposal recognizes that lubricants can exhibit metastable states at very low shear rates and suggests that a controlled equilibrium condition can be provided in the test procedure so that a quantitative description can be obtained of that state. It is expected that the test samples may exhibit various structural phases or polymorphisms which would be identified under static conditions.

SECTION 3

TECHNICAL APPROACH AND DISCUSSION

3.1 GENERAL APPROACH

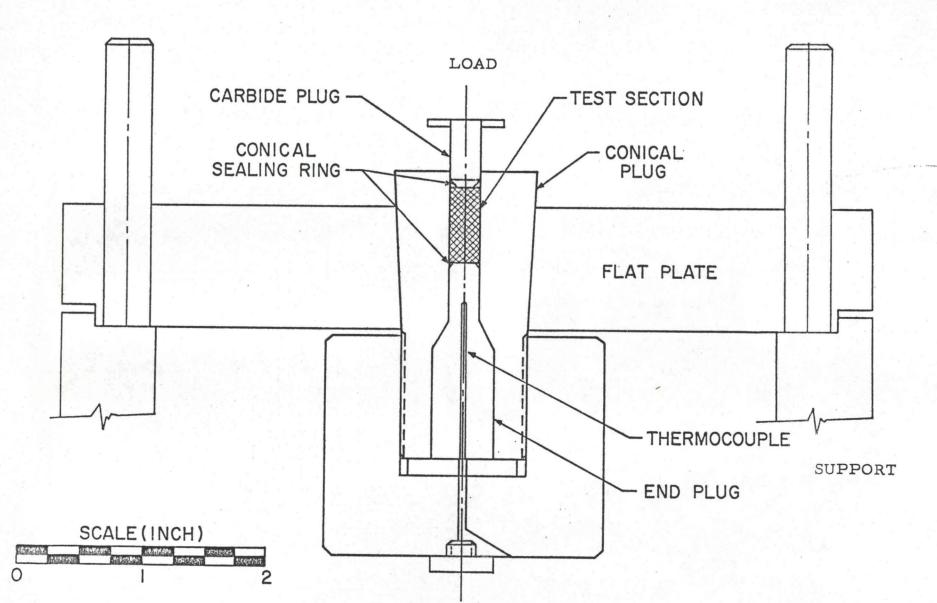
The equilibrium phase diagrams will be obtained for lubricants having diester, polyester, and polyphenolether bases. The particular region of interest will be at pressures in excess of 100,000 psi and temperatures from 32°F to 158°F. The method used to obtain the data is one commonly used to obtain polymorphisms of solids and phase diagrams of various liquids. Essentially, it involves the pressurizing of a small chamber (Figure 1), and monitoring temperature and volume as equilibrium is slowly achieved with the surroundings at constant pressure. The volume will be sensed by means of a lever system and linear voltage displacement transducer (LVDT) (Figure 2), and the temperature by means of a thermocouple placed near to the test chamber (Figure 1).

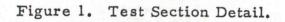
The test apparatus will be placed in an MTS Electro-Hydraulic Closed-Loop Test Machine and the load monitored accordingly. Pressure can be obtained by a calculation from the equation

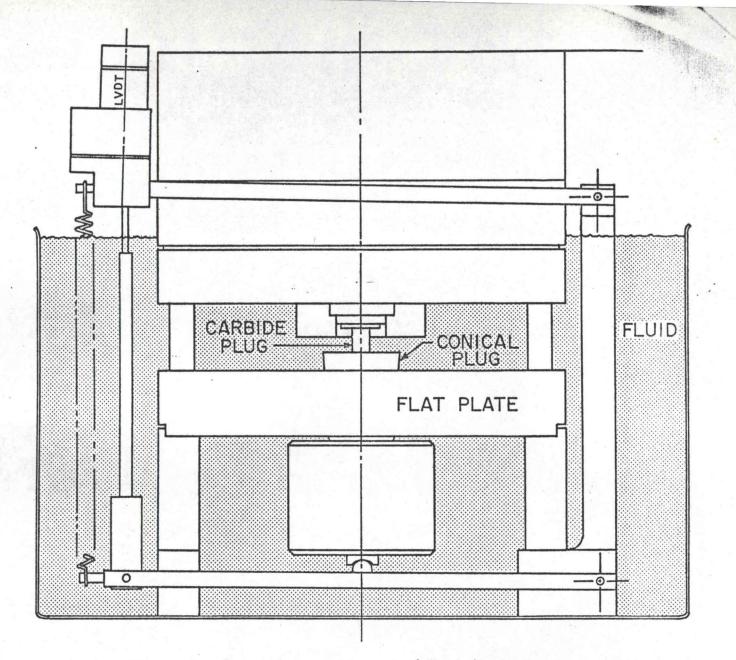
Pressure = Force/Area

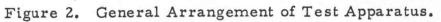
Area would represent a cross-section area of the test chamber and would be measured before testing. Of course, since yielding of material would occur during high pressure situations, the area would change. The effective area will then be predicted by means of standard stress analyses. After a series of these isobaric excursions, a phase diagram can generally be constructed.

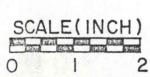
A major portion of the difficulties in this experimental program are expected to arise in preventing leakage of the test material, a lubricant. Thus, it appears that the worst of all possible worlds exists in the sealing of test material specifically designed to flow readily through confinements. To assure the containment of the material a vial of lead or woods metal will be used to encapsulate the sample of lubricant prior to testing. A soft











metal such as lead is proposed to ensure a more hydrostatic application of the pressure on the lubricant during testing. The methodology for encapsulating the fluid without entrapping air or foreign particles will need to be defined and developed.

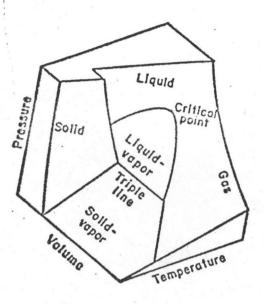
Dr. H. Tracy Hall, an international expert on high-pressure research and at present the Director of Brigham Young University High Pressure Laboratory, has agreed to act as a consultant on the research effort. He will be reviewing all critical aspects of this program. His expertise in all aspects of high-pressure research, including the first synthesis of diamond, will compliment and accelerate the test program.

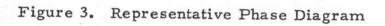
At this time, it is anticipated that the results of the measurements with various lubricants can be represented as a phase diagram as shown in Figure 3. The phase diagram is a plot of pressure, volume, and temperature on mutually perpendicular coordinates, and all possible equilibrium states are represented by a point on the surface. The data may also be presented as pressure-temperature and pressure-volume plots as shown in Figure 4. Note the transformations in the pressure-temperature plot of water at high pressures as shown in Figure 5, i.e., water can be solid at 150° F.

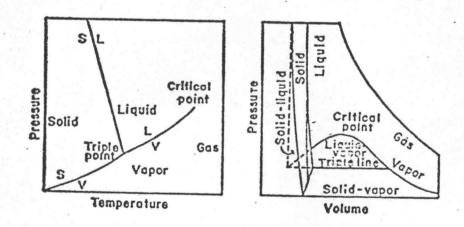
The phase boundaries are clearly indicated by discontinuities in the diagram and may be detected by an analysis of the pressure, volume, and temperature data (which also includes latent heat data). Recall that a pure substance, in the absence of motion, gravity, surface effects, electricity, and magnetism has only two independent intensive properties. Note that in multiphase regions pressure and temperature are not independent properties. For instance, for water at 32°F and atmospheric pressure of 14.7 psi, ice and liquid are in equilibrium. Thus, a true description of the equilibrium mixture requires a third property (such as enthalpy, entropy, or volume). In fact, Gibbs phase rule will give the required number of properties needed for a substance having "n" phases in equilibrium through the relationship

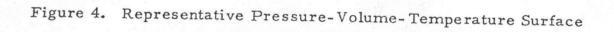
F + n = C + 2

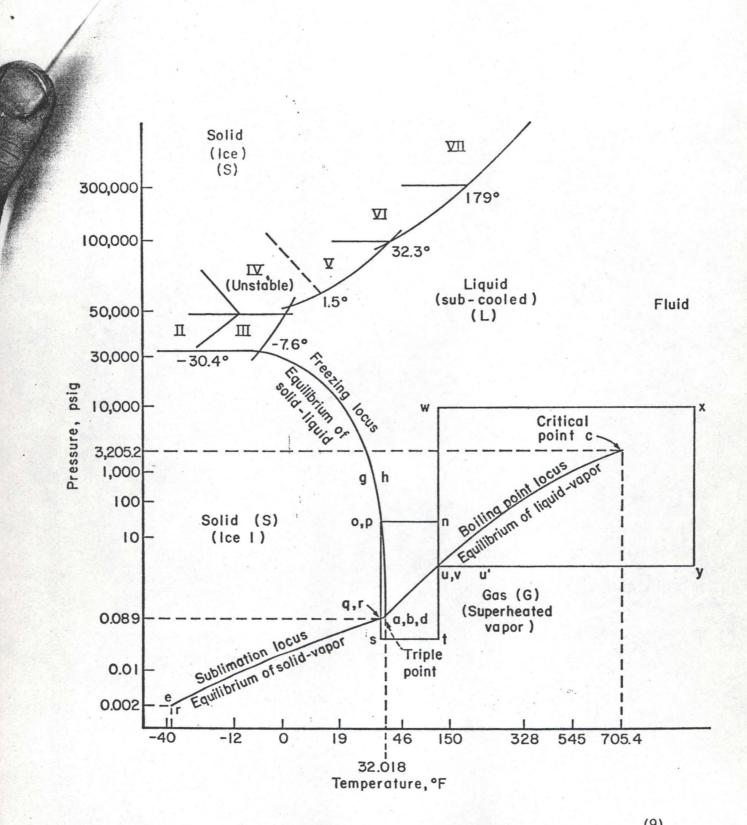
where F is the number of independent variables (properties required to determine the state) and C is the number of pure substances.

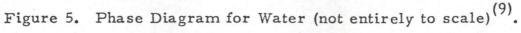












If, on the other hand, the lubricant becomes an amorphous solid or super-cooled liquid at extremely high pressures, the phase change will be more difficult to observe. It would be expected that the change in volume would be more gradual with this type of transition in contrast to crystallization. X-ray diffraction analyses of the samples will be used in the University's X-ray Diffraction Laboratory, if necessary, to ascertain the structure of the lubricant.

The significance of this work can only be as high as the degree of success in identifying the test material. The test material will be lubricants of synthetic origin and will have diester, polyester, and polyphenolether bases. It is recognized that organic compounds such as these have highly diverse chemical structures and that even the same elemental molecules of the lubricant can have many isomers.

This program will include the identification of each of the test samples as to chemical structure. The latest techniques using high-resolution liquid chromatography and/or mass spectroscopy will provide adequate information as to the "backbone" organic compound of the samples. It is anticipated that the chemical structures of the lubricants may be altered during the high pressure tests. The analyses before and after the tests will then provide the necessary information to determine the change in the chemical structure accordingly. Thus, the program seeks to use pure substances as the test material but will determine the degree of purity as well as the effect of pressure and temperature on the chemical structure of the test materials.

During the phase changes of materials, a significant amount of energy is transferred. This energy, often called latent heat, can cause a temperature drop or rise of the surrounding material. During solidification heat or energy is lost by the material. If this heat is used as "sensible" or conduction heating it may manifest itself as a temperature rise in the surroundings. Opposite to this phenomena, energy is needed to convert a solid to a liquid and thus a temperature drop may ensue in the surroundings. As illustrated in Figure 4, the interface between phases is characterized by lines on the P-T plane. Using the Clapeyron equation

$$\frac{\partial P}{\partial T} \approx \frac{\Delta h}{T(\Delta V)}$$

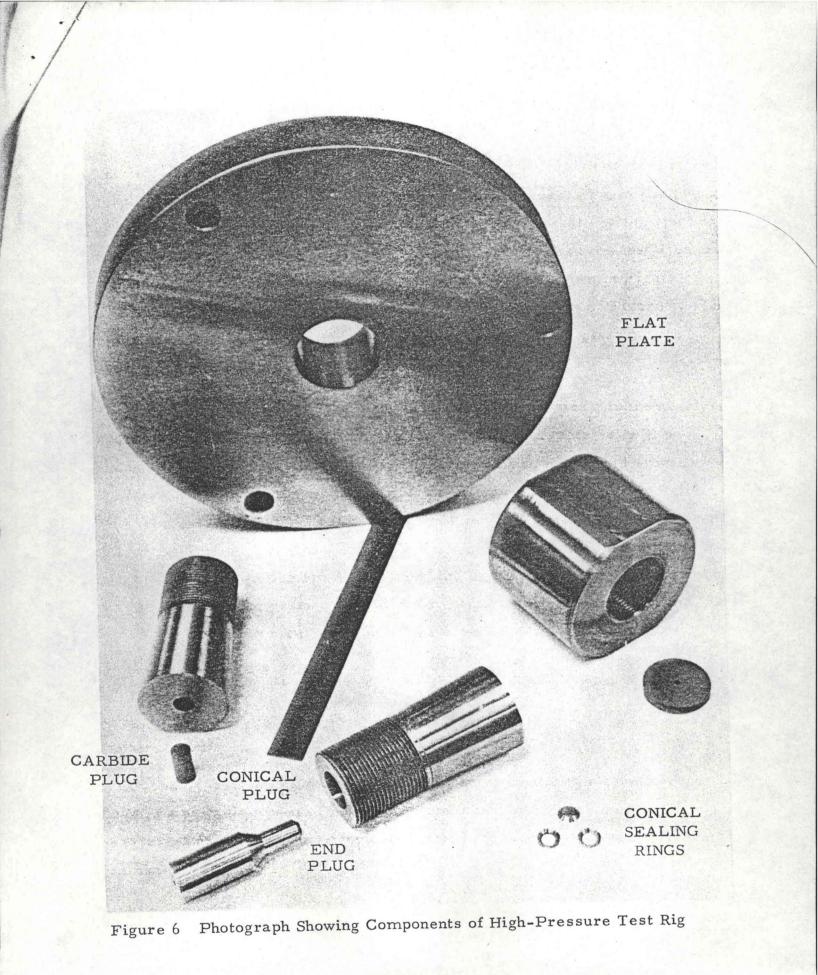
we can obtain $\triangle h$, the latent heat of solidification, by noting that the left side is the slope of the phase change curve and $\triangle V$ is the volume change during the phase change.

While this program does not consider temperature phenomena beyond 70°C, an extension in this investigation might very well be made into high temperature and pressure regions. Increasing the temperature of the test specimens much beyond 70°C will materially weaken the proposed test apparatus. This would require different test apparatii such as the Bridgeman "Anvil" technique⁽⁸⁾ but would represent a most convenient direction should the present program be developed to fruitation.

3.2 TEST APPARATUS

The University of Dayton has designed and constructed a test device necessary to perform hydro-static tests on liquids and solids to 54,000 atmospheres (800,000 psi) and 70°C (158°F). The essential festures of this mechanical apparatus are shown in Figure 1 and the assembly in Figure 2.

The design is based on P. W. Bridgeman's^(1, 3) device which utilizes an unsupported area. A conical plug is forced into a corresponding conical hole in a flat plate by the application of a force to the test specimen. A cemented carbide plug is used to apply the necessary force to the pressure chamber from above and a cap at the bottom receives the major portion of this force. The result of this action is a downward displacement of the conical plug and pressure chamber or test section (see Figure 1). The conical surface produces an increasing radial pressure on the walls of the small pressure chamber which in turn avoids rupture in the test section walls.



This technique enabled Bridgeman to conduct tests up to 50,000 kg/cm² (684,000 psi) and, with some minor modifications in the Bridgeman design, the present design is anticipated to extend the range of test pressures to 800,000 psi.

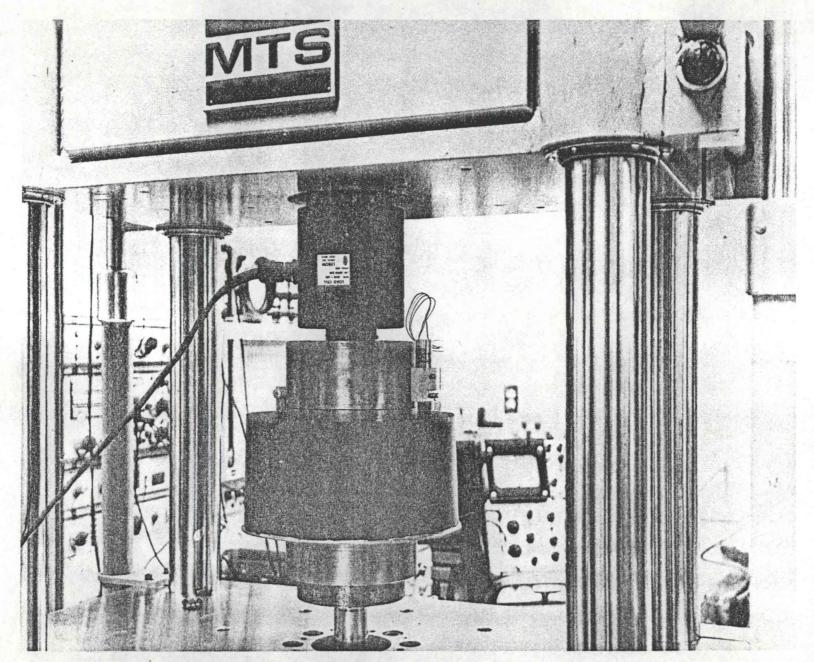
Sealing of the chamber to prevent leakage of the test material prior to solidification will be achieved in a manner which increases the sealing action with increasing pressure. This involves the use of conical sealing rings in the chamber ends, shown in Figure 1 as chamfered ends of the test section. The actual parts of the mechanical apparatus are also shown in Figure 6. The conical sealing rings are displayed at the bottom right of Figure 6. The rings, which are made of high-strength steel, will be in a plastic state during much of the high pressure tests.

To avoid extrusion of the test material and to provide a more nearly hydrostatic pressure application, the lubricant or test material will be encapsulated in a lead vile. Copper sheathing will be positioned around the lead vile to prevent possible diffusion of lead into the chamber walls. This particular diffusion (lead into steel) is reported to appreciably weaken the mechanical integrity of steel. Since the program is concerned with determining the equilibrium diagrams of materials, pressure is a property of importance. The pressure is normally defined as the hydrostatic pressure and is related to the stress tensor by the equations

$$\delta_{ij}^{p} = T_{ij}$$

where δ_{ij} is the Kronecker delta, $\delta_{ij} = 1$ for i = j, $\delta_{ij} = 0$ for $i \neq j$. T_{ij} is the stress tensor.

Thus, pressure is the normal stress component of the stress tensor. Lead is a suitable material having minimal shear strength when subjected to external forces and thus provides nearly hydrostatic pressure to the test chamber.



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Figure 7 Photograph Showing High-Pressure Test Rig Mounted in MTS Electro-Hydraulic Closed-Loop Test Machine

Data will be obtained on the chamber volume, chamber temperature, and applied force. The tests will be run on an MTS Electro-Hydraulic Closed-Loop Test Machine (precision material test machine) which is available at the University of Dayton. The High-Pressure Test Rig is shown in Figure 7 positioned in the MTS machine prior to a simulated test run. The volume will be determined by using a Linear Voltage Displacement Transducer (LVDT) to measure the chamber length during compression and upon relaxation of pressure. The length can thereby be measured continuously within ±.0001 inches. Pressure will be calculated from the relation

$$p = F/A_n$$

where F will be the force monitored by the material test machine. Friction and inertial effects will be accounted for so that F will represent the total force acting on the test chamber cross section area, A_n . It is recognized that the chamber will "barrel out" or deform slightly during the test and will not fully recover to a cylindrical shape upon release of pressure. Analytical methods are available from the theory of plasticity to accurately predict the cross-section area.

Temperature will be sensed inside the chamber by use of a miniature thermocouple embedded in the end plug as shown in Figure 1. This temperature will indicate the local instantaneous temperature of the chamber during a test. Since, however, all tests are expected to be conducted isothermally it is anticipated that this reading will represent a control for the rate of compression. The apparatus will be immersed in controlled temperature fluid so that for equilibrium conditions the temperature will remain constant.

During a test the pressure will be gradually increased in increments so that the chamber temperature remains or returns to the bath or isothermal test temperature. It is expected that with increasing pressure the test material will experience a sharp rise in temperature, followed by a gradual return to ambient. This fluctuation, in addition to being an operation control,

will also be of future interest in the program. The test temperature will be altered by varying the bath temperature accordingly.

3.3 EXPERIMENTAL APPROACH

In order to implement the research objectives the effort will be completed in two phases. Phase I is basically a calibration of the complete system and an evaluation of a lubricant with a diester base. Lubricants with polyester and polyphenolether bases will be investigated in Phase II.

In Phase I the methodology for encapsulating the fluid without entrapping air or foreign particles will be developed. The apparatus will then be calibrated by determining the thermodynamic equilibrium diagrams and heats of transitions for a well known substance, i.e. water within the range of equipment capability. Any necessary modifications of the system design will be completed at that time. Then the thermodynamic equilibrium diagram, heats of transition and changes in chemical structure will be determined for a lubricant with a diester base.

In Phase II the thermodynamic equilibrium diagrams, heats of transitions and changes in chemical structure will be determined for lubricants with polyester and polyphenolether bases.

3.4 TECHNICAL SUMMARY

Applications involving extremely high pressures such as gears, cams, and rolling bearings are enormous. At present, the properties of lubricants at very high pressures are not adequately defined with regard to elastohydrodynamic theory. Through a better understanding of the thermodynamic phase and chemical structure of lubricants at operating pressures it will be possible to design to increase fatigue life and to reduce friction and wear. This will in turn yield tremendous savings in energy.

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The present analytical treatment using elastohydrodynamic theory does not adequately predict the actual behavior of lubricants in high pressure applications. This is primarily because there is a lack of scientific information regarding the elemental behavior of lubricants under high pressures. Thus the lubricant phases (fluid, viscoelastic solid or solid) need to be identified qualitatively and quantitatively in order to establish adequate rheological models.

This program seeks to provide the thermodynamic equilibrium phase diagram of synthetic lubricants such as diester, polyester, and polyphenolether lubricants. It will also determine the heat of transition along with the effect of high pressure on the chemical structure of these lubricants. This information, obtained under static equilibrium conditions, could then be used to help predict lubricant behavior under dynamic conditions particularly regarding their phase and structure.

SECTION 4 ORGANIZATION AND PERSONNEL

4.1 ORGANIZATION

The University of Dayton has been engaged in research and development work on contracts for various agencies of the Federal Government, chiefly the Air Force, since 1949. During the period from 1949 to 1975, annual research volume has increased from \$4,000 to \$6.3 million. Since its beginning, total research volume at the University has been approximately \$68 million, and programs have been conducted for numerous military and civilian branches of the Government as well as for numerous industrial organizations. This program will be conducted in the School of Engineering of the University.

Recently, the University has taken action to expand its research capabilities in the area of Aircraft Vehicle Technology and Lubrication. Graduate engineering courses in Advanced Propulsion, Combustion, and Lubrication have been initiated and related laboratory test capabilities expanded. This expansion was brought about after much discussion with the various research organizations at Wright-Patterson Air Force Base and the conclusion that the University could satisfy a research/academic need in these areas. The University has expanded its academic engineering graduate program and initiated a Doctoral Program in Engineering. The highly educated and experienced personnel whose qualifications are presented in relationship to this program are members of the group assigned to assist the University in achieving this much needed research/academic expansion.

4.2 PERSONNEL

The Principal Investigator will be Dr. John N. Crisp, who will be directly responsible for all of the technical effort. Mr. Kurt C. Rolle will assist Dr. Crisp on the proposed effort. Dr. Crisp is Mr. Rolle's advisor for his Doctoral Program.

JOHN N. CRISP, Associate Professor and Research Engineer in Mechanical Engineering

Education

Bachelor of Mechanical Engineering, 1958, Georgia Institute of Technology, Atlanta, Georgia

M.S., Engineering, 1964, University of Akron, Akron, Ohio

Ph.D., Mechanical Engineering, 1968, Carnegie-Mellon University, Pittsburgh, Pennsylvania

Experience

Dr. Crisp has considerable experience in engineering education, design, teaching, and research. He has taught graduate and undergraduate courses including plasticity, theory of controls, heat transfer, thermodynamics, kinematic synthesis, and mechanical design. He initiated a graduate course on "Bearings and Bearing Lubrication" and conducted a short course on "The Theory and Practice of Lubrication" in August 1974 at the University of Dayton in cooperation with Wright-Patterson Air Force Base and the Dayton Section of the American Society of Mechanical Engineers. He also serves as an industrial consultant in the area of bearings and lubrication.

He is currently Principal Investigator on the University's high-temperature damping program which consists of formulating enamel materials based upon glass compounds and in determining the vibration damping properties of these materials as a function of temperature and frequency. Other current research areas include a study of the heat transfer characteristics of an absorption system and an evaluation of lubricants and bearing materials for journal bearings. The research on lubricants and bearing materials utilizes the University's Sleeve-Bearing and Lubricant Test Rig.

Dr. Crisp also has 10 years of industrial and research experience in the area of lubrication. He has conducted research on the low-temperature lubrication of bearings and on the influence of oil viscosity on roller bearing life.

He also completed a considerable amount of research on roller bearings including contact stress analysis, load zone measurement and analysis, and transfer function analysis. He has also designed and developed rolling contact test machines and bearing fatigue test rigs.

While at Carnegie-Mellon University, he completed research on the design and development of a piezoelectric force transducer which was subsequently used to measure force pulses of durations on the order of microseconds. Also, he completed an analytical and experimental study of transient thermal effects with moving heat sources over finite length intervals.

Professional Affiliations

American Society of Lubrication Engineers American Society of Mechanical Engineers American Institute of Aeronautics and Astronautics American Society for Engineering Education Scientific Research Society of North America Pi Tau Sigma P.E. Registered in Ohio

Membership in University Committees

University Scholarship Committee Physics Liaison, School of Engineering Committee

List of Publications

Crisp, J.N. and Ellis, E., "Low Temperature Performance of Greases in Railway Roller Bearings," Lubrication Engineering, July 1963.

Crisp, J.N. and Stokey, W., "Measurement of Forces During Grinding with a Single Abrasive Crain," <u>The International Journal of Production</u> <u>Research</u>, Vol. 7, No. 2, 1968.

Crisp, J.N., "Transient Thermal Effects in Surface Grinding," Ph.D. Thesis, Carnegie-Mellon University, 1968.

Crisp, J.N. and Pinson, J.D., "Engineering Analysis of a Dynamic Environmental Simulator," Technical Report UDRI-TR-73-62, Aerospace Medical Research Laboratory, WPAFB, Ohio, December 1973.

Pinson, J.D. and Crisp, J.N., "The Effective Thermal Conductivity of Fluomine in a Packed Bed; Part One - Static Thermal Conductivity," Technical Report UDSE-TR-74-01, Air Force Flight Dynamics Laboratory, WPAFB, Ohio, October 1974.

Pinson, J.D. and Crisp, J.N., "The Effective Thermal Conductivity of Fluomine in a Packed Bed; Part Two - Dynamic Thermal Conductivity," Technical Report UDSE-TR-75-05, Air Force Flight Dynamics Laboratory, WPAFB, Ohio June 1975. Kaliszewski, R.L. and J.N. Crisp, "Experimental and Computer-Aided Lubrication Analysis of Journal Bearings," Technical Report UDSE-TR-75-08, Dayton, Ohio, 1975.

Publications in industry were in-house engineering reports, contents were proprietary.

KURT C. ROLLE, Research Associate/Doctoral Student Education

B.S., Mechanical Engineering, 1960, Purdue University, West Lafayette, Indiana

M.S., Engineering Sciences, 1968, University of Dayton, Dayton, Ohio Ph.D. candidate, University of Dayton, Dayton, Ohio

Experience

Mr. Rolle has been employed by the Perfect Circle Corporation in Hagerstown, Indiana, where he was a designer of special equipment to manufacture, test, and inspect piston rings, valve seals, and other automotive parts. While here, he engaged in work relating to the lubrication of internal combustion engines through the mechanics of piston rings and valve seals. He also was with the United States Air Force at Wright-Patterson Air Force Base as a mechanical engineer. His responsibilities included the design of aircraft fuel systems and refueling components, establishment of specifications for fuel systems, and monitoring of contractor efforts in fuel system design and installation.

Since joining the University of Dayton, Mr. Rolle has taught thermodynamics, machine design, and mechanics courses in addition to authoring a new textbook in thermodynamics. Concurrent with his teaching activity, he has completed research in various areas of design and analysis. Most recently, he designed the high-pressure test rig described in this proposal. He also designed and developed a full set of equipment to test the mechanical behavior of frogs' sartorius muscles and developed suitable techniques for the methodology of testing muscles.

Also, he did analysis of clear air turbulance data to determine the validity of the data collection techniques and mechanical equipment used to gather this data.

In addition, he has developed and conducted a program to impact test various materials using a standard impact test machine. He has done consulting work in the design of various mechanical systems including central coolant systems for large machine tool facilities, retro-fitting aircraft to bleed air from engine compressor sections and other test devices. He designed a device to determine the moment of inertia in six axes and the C. G. of a pilot/seat configuration based on United States Air Force requirements and further, he assisted in the design of devices to test skin tissue in vivo.

List of Publications

Rolle, K., "Tensile Impact Test of Materials in Accordance with MIL-P-8045B," Engineering Test Report, January 1973.

Rolle, K., Introduction to Thermodynamics, Charles E. Merrill Publishing Company, Columbus, Ohio, June 1972.

Ryan, J. P., Berens, A. P., Robertson, A. C., Dominic, R. J., and Rolle, K., "Medium Altitude Critical Atmospheric Turbulence Data Processing and Analysis," Technical Report AFFDL-TR-71-82, Air Force Flight Dynamics Laboratory, Wright-Patterson AFB, Ohio, July 1971.

Rolle, K., "Experimental Determination of Mechanical Parameters of Muscle," M.S. Thesis, University of Dayton, 1968.

Rolle, K., "Metallurgical Evaluation of Cast Iron Alloy (Perfect Circle Corporation 110-63 Material)," Report 106025, 1960. HOWARD T. HALL, Director of Brigham Young University High Pressure Laboratory

Education

B.S., 1942, University of Utah

M.S., 1943, University of Utah

Ph. D., 1948, Physical Chemistry, University of Utah

Special wartime training, 11 months in electronics at Bowden College, M.I.T., Harvard, and Honolulu Naval Base while an Ensign in the United States Navy, 1944-1946.

Employment

1967	Distinguished Professor, Brigham Young University, Provo, Utah.
1955-1967	Director of Research for the entire University and Professor of Chemistry, Brigham Young University, Provo, Utah.
1948-1955	Research Associate, General Electric Research Laboratory, Schenectady, New York.
1942-1944 & 1946	Chemist, U.S. Bureau of Mines, Salt Lake City, Utah.
1940-1942	Chemical Analyst, Sperry Flour Mills, Ogden, Utah (Part-time).
1939-1940	Photographer, Checketts Photo, Ogden, Utah.

Consulting

1957 ---- For various industrial and governmental organizations (over 50 in number).

Professional Society Memberships

American Chemical Society American Association for the Advancement of Science The American Physical Society The Mathematical Association of America Sigma Xi Phi Kappa Phi Professional Society Memberships (Continued)

Timpanogos Club of Utah Utah Academy of Science Arts and Letters Newcomen Society in North America

Major Scientific Achievements

The first synthesis of diamond (1954). This feat had eluded scientists for over 150 years.

The first high pressure, high temperature apparatus, "The Belt." (100,000 + atmospheres, simultaneously with 2,000 + Degrees C.) (1953).

Determination of the first melting curve under high pressure, high temperature conditions (for Germanium) (1954).

The second high pressure, high temperature apparatus, "The Tetrahedral Press," which circumvented the proprietary interest that prevented use of the Belt for research after leaving General Electric's employ (1956).

The first high pressure, high temperature X-ray diffraction apparatus (with J. Dean Barnett) (1962).

Discovery of the first pressure-induced phase change from a closepacked to non-close-packed structure (FCC to BCC in Ytterbium at 40 kb), (with J.D. Barnett and Leo Merrill) (1963).

The determination of the nature of the "resistance cusp" in cesium. This intriguing problem had remained unsolved since discovery of the cusp by P.W. Bridgman in 1951 (with Leo Merrill and J. Dean Barnett) (1964).

The concept of "Periodic Compounds" (1965).

Sintered Diamond (similar to natural carbonado type diamond) (1970). The bonding together of fine diamond particles to produce a dense, strong, polycrystalline body of desired shape having properties equal to or exceeding those of natural carbonado heralds a new era in diamond technology. This new material is already finding use in wheel dressers, wire drawing dies, drilling stones, cutting tools, and in electronics. Thin disks, cylinders with axial holes, nozzles, rings, spheres, cubes, and other molded shapes up to 20 carat size are currently possible and will certainly revolutionize the diamond industry in the next decade. In addition, this discovery just about takes care of the problem of strategic diamond materials. In industrial diamonds, the United States will soon approach self sufficiency.

Other Responsibilities

Director of Brigham Young University High Pressure Laboratory, 1955 ----.

Director of the National High Pressure Data Center which is run by Brigham Young University for the U.S. National Bureau of Standards, 1965 ----.

Honorary Positions

President, Utah Academy of Sciences, Arts and Letters, 1960-1961. Chairman, Salt Lake Section, American Chemical Society, 1959. Fellow, American Association for the Advancement of Science, 1960----. Member of Editorial Board, "Inorganic Chemistry," 1961-1964. Member of Editorial Board, "The Review of Scientific Instruments,"

1966-1969.

NAS-NRS Evaluation panel for the NBS Heat Division, 1970----. JANAF Thermochemical Tables Advisory Group, 1968 ----.

Fellow, The Utah Academy of Sciences, Arts and Letters, 1972 ----. Fellow, The American Institute of Chemists, 1972 ----.

Distinguished Professor of Chemistry and Chemical Engineering, Brigham Young University, 1967 ----.

American Chemical Society Tour Speaker, 1972. American Chemical Society Tour Speaker, 1973.

Awards

1959-1963, Alfred P. Sloan Foundation Research Fellow.

1962. The American Society of Tool and Manufacturing Engineers "Research Medal." New York City.

1964, First "Annual Faculty Lecture," Brigham Young University, Provo, Utah. April 8.

1964, Third Annual "Olin Mathesen Lecture," Yale University, New Haven, Connecticut. April 22.

1965, The Brigham Young University's James E. Talmage Scientific Achievement Award, " Baccalaureate Exercises, Provo, Utah. May 27.

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.

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

1970, American Institute of Chemist's "Chemical Pioneer Award," Pittsburgh, Pennsylvania. May 16.

1971, "Outstanding Manhood Award," presented by Associated Men Students, Brigham Young University, Provo, Utah. April 13.

1971. Honorary Doctor of Science Degree, Brigham Young University, Commencement Exercises, Provo, Utah. May 28.

Awards, Continued

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1972, The American Chemical Society "Award for Creative Invention," Boston, Massachusetts. April 10.

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.

1973, The American Society for Metals, "Engineering Materials Achievement Award," Chicago, Illinois. October 2.

1974, IR-100 Award by Industrial Research Magazine to Megadiamond for Indexible Sintered Diamond Tools, Field Museum of Science and Industry, Chicago, Illinois. October 9.

Selected Publications

Hall, H.T. and H. Eyring, "The Constitution of Chromic Salts in Aqueous Solution," J. Am. Chem. Soc., 72:782-790 (1950).

Hall, H.T., E.L. Brady, and P.D. Zemany, "Viscosity of Polytrifluorochloroethylene in O-Chlorobenzotrifluoride," J. Am. Chem. Soc., 73:5460 (1951).

Bundy, F.P., H.T. Hall, H.M. Strong, and R.H. Wentorf, "Man-Made Diamonds," Nature, 176:51-54 (1955).

Hall, H.T., "The Melting Point of Germanium as a Function of Pressure to 180,000 Atmospheres," J. Phys. Chem., 59:1144-1146 (1955).

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Hall, H.T., "Chemistry at High Pressures and High Temperatures," J. Wash. Acad. Sci., 47:300-304 (1957).

Hall, H.T., B. Brown, B. Nelson, and L.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., 62:346-351 (1958).

Hall, H.T., "Some High Pressure, High Temperature Apparatus Design Considerations: Equipment for Use at 100,000 Atmospheres and 3,000 Deg. C," Rev. Sci. Instrum., 29:267-275 (1958). Hall, H.T., "Ultrahigh Pressure Research," Science, 128:445-449 (1958).

Hall, H.T. and S.S. Kistler, "High Pressure Developments," <u>Annual</u> <u>Review of Physical Chemistry</u>, Annual Reviews, Inc., Palo Alto, California pp. 395-416 (1958).

Hall, H.T., "Diamonds," <u>Proceedings of the Third Conference on</u> <u>Carbon</u> (held at University of Buffalo, Buffalo, New York, June 1957), Pergamon Press, London, pp. 75-84.

Hall, H.T., "High Pressure Methods," <u>Proceedings of an International</u> Symposium on High Temperature Technology, Asilomar Conference Grounds, California, 6-9 October 1959; Arranged by Stanford Research Institute, Menlo Park, California, McGraw-Hill, New York, pp. 145-156 and 355-336 (1960).

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Dudley, J.D. and H.T. Hall, "Experimental Fusion Curves of Indium and Tin to 105,000 Atmospheres," Phys. Rev., 118:1211-1216 (1960).

Hall, H.T., "Some High Pressure, High Temperature Apparatus Design Considerations: Equipment for use at 100,000 Atmospheres and 3,000°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., 29:267-275 (1958).

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SECTION 5

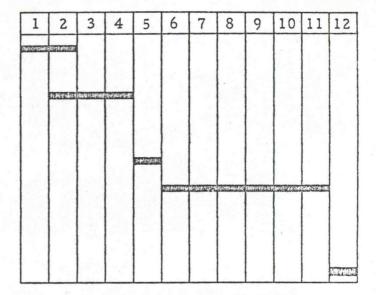
PROGRAM PHASES AND REPORTS

The research plan which describes the phases for the two year research effort is shown in Figure 8. $^{\circ}$

Phase I - Calibration of System and Evaluation of Lubricants with Diester Bases

- a. Develop methodology for encapsulating fluid
- b. Calibrate apparatus by determining thermodynamic equilibrium diagrams for water
- c. Modify system design as needed
- d. Determine thermodynamic equilibrium diagrams, heats of transitions and charges in chemical structure for: lubricant with diester base

e. Prepare Report and Recommendations

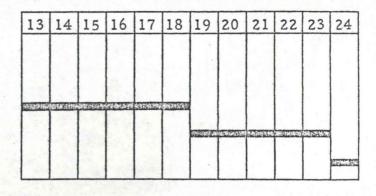


Phase II - Evaluation of Lubricants with Polyester and Polyphenolether Bases

Determine thermodynamic equilibrium diagram, heats of transitions and changes in chemical structure for:

Lubricant with polyester base

Lubricant with polyphenolether base Prepare Final Report and Recommendations



SECTION 6

FACILITIES AND SUPPORT CAPABILITIES

6.1 INTRODUCTION

The School of Engineering is located in the research and engineering facilities of the Eugene W. Kettering Engineering and Research Laboratories building. The excellent modern laboratories and the equipment contained in this building give the University a broad capability and provide the needed atmosphere for the development of engineering educational and research programs. The following paragraphs describe some of these excellent facilities.

6.2 LUBRICATION FACILITIES

The University has designed and developed the necessary equipment to collect the data required for the research. The basic device consists of a solid plate supported rigidly and containing a tapered cylinder. The test specimen or lubricant would be enclosed inside the cylinder with carbide plugs at each end. This device is indicated in Figures 1 and 2 of the proposal. A load cell and thermal transducer monitors the pressure and temperature while a Linear Voltage Displacement Transducer monitors the volume of the specimen. Oscillographs, cathode ray oscilloscopes, and other standard recording instruments are readily available to assist in data collection. The tests will be run on an MTS Electro-Hydraulic Closed-Loop Test Machine (precision material test machine) which is available at the University of Dayton. The High-Pressure Test Rig is shown in Figure 7 of the proposal.

Over the past few years, there has been much effort expended at the University of Dayton on research related to studying bearings, gears, and lubricants. The University of Dayton presently has six Pope spindle grease testers used to obtain data on the fatigue and life of greases. The

University also has an eight-spindle sleeve-bearing and lubricant test rig which is used to evaluate boundary lubricants and bearing materials. Additionally, some special equipment used to observe oxidation characteristics of lubricants is available.

The University has an extensive capability in the material properties area and among the equipment available is a different thermal analyzer (DTA). This device allows for observing the equilibrium phase diagrams for mixture of rare-earth metals and while its applicability is not direct, this capability exists to refine or review the technology as it would apply to this effort.

6.3 CHEMICAL INSTRUMENTATION LABORATORY

Gas chromatography is utilized extensively in this laboratory for the analysis of gaseous, liquid, and solid samples. This laboratory is equipped with three different gas chromatographs which employ a variety of sensitive detectors. A highly versatile Varian Aerograph 1800 Series Gas Chromatograph is capable of performing high resolution separations. Also, a 2440 Series Varian instrument is available for analyzing trace components by electron capture detection. Non-volatile materials such as polymers and other high molecular weight organics are investigated by pyrolysis gas chromatography. This technique subjects a small quantity of material to a controlled temperature increase of up to 1400°C, while in a defined atmosphere.

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Thermogravimetric and differential thermal analysis examination can be conducted in this laboratory. A Fisher 100 A Series TGA/DTA System is used for examining a wide range of materials at temperatures up to 1000°C. Two other TGA instruments are available within the Research Institute. A Chevenard Model 249 and an Aminco Model 101-1517 are routinely employed for specimen temperature extending up to 900°C.

This particular laboratory is also equipped for other conventional instrumental analyses. As examples, a Perkin-Elmer Model 257 Infrared

Spectrophotometer is available for nondestructive infrared analysis. Also, a Perkin-Elmer Model 240 elemental analyzer can be used for determining the carbon, hydrogen, nitrogen, and oxygen content of samples.

6.4 DIRECT ENERGY CONVERSION

The Direct Energy Conversion Laboratories provide the opportunity to study new concepts to convert available energy directly to useful work.

The Thermionic Conversion Laboratory's equipment permits the adjustment of many parameters to provide numerous combinations of operating conditions for changing thermal energy into electrical energy by the emission of electrons from a heated plate. Much of the effort in the Solar Cell Laboratory is directed toward the goal of developing more efficient solar cells for the future. In the Bio-Energy Conversion Laboratory unique muscle test equipment is used to study how a muscle directly converts chemical energy to mechanical work.

Devices in the Thermoelectrics Laboratory permit the study of the various parameters of thermoelectricity, including the opportunity to study magneto effects on thermoelectric materials.

6.5 MATERIALS ANALYSIS LABORATORIES

The Materials Analysis Laboratories contain advanced analytical instruments for measuring both physical and chemical properties of solidstate materials--metals and alloys, ceramics, plastics and polymers, glasses, composites, and biological materials.

The Thermal Properties Laboratory is equipped to detect and measure changes in phase and state that occur in materials on thermal cycling, and to correlate these with simultaneous changes that occur in the physical, chemical, and mechanical properties of the materials. This laboratory contains a differential thermal analyzer, which is a precisely controlled high vacuum or inert atmosphere furnace equipped with very sensitive temperature sensors and recorders.

The X-Ray Diffraction Laboratory is equipped to conduct a wide range of investigations on crystalline materials. These include phase identification, crystal structure analysis, precision lattice constant determination, grain size, and orientation texture analysis, stress analysis, thermal expansion measurements, solubility of one solid in another, and phase transformation studies.

The Electron Optical Systems facility contains equipment to analyze, by means of electron probe, surface and subsurface detail in metallic and nonmetallic subjects.

6.6 MATERIALS ENGINEERING

6.6.1 High Temperature Materials Laboratory

This facility is equipped to provide static and dynamic testing and measurement of optical properties of a large spectrum of metallic and ceramic materials under a wide range of temperatures and a variety of loading conditions. Tests performed in this laboratory include tensile and compressive strength, creep, stress rupture, modulus of rupture, stress corrosion, and oxidation resistance studies. The laboratory is also equipped to determine the dynamic properties of metallic, ceramic, refractory, and composite materials at temperatures that range from -100°F to 3,000°F and to measure optical properties of a wide range of materials at elevated temperatures.

6.6.2 Ceramic and Refractory Materials Laboratory

The Ceramic and Refractory Materials Laboratories provide for the fabrication and evaluation of ceramic materials for biomedical and aerospace applications. The Physical Properties Section has the facilities for measuring the mechanical, electrical, and thermal properties of ceramic, metal, and composite materials of interest. The hot pressing laboratory area is devoted to the fabrication of ceramic test samples. The primary method of fabrication is pressure sintering, which is accomplished in any one of three hot presses. This equipment is also used to vacuum

melt rare-earth metal alloys and to pressure bond metal matrix-ceramic fiber-reinforced composite materials.

The Plasma Spray Laboratory is designed for the development and evaluation of flame-sprayed plastic, metal, or ceramic coatings. In addition, this laboratory has a vacuum coating unit for applying thin film coatings to a wide variety of substrates.

The Petrography Laboratory is a "clean room" for the purpose of microscopy, X-ray film evaluation, and accurate weighing of materials used in the Ceramic Laboratory. All powder materials prepared for study are processed in the Powders Preparation Laboratory.

6.6.3 Composite and Polymeric Materials Laboratory

This group of laboratories provides an extensive capability for fabricating, testing, and evaluating both metallic and nonmetallic composite materials, adhesives, and elastomers. The laminating laboratory is equipped for the fabrication of laminated structural plastic composites, adhesive bonds, pressed powder castings, and ablative composites. If necessary, special enclosures are available for employing an inert gas or vacuum environment.

The Curing and Aging Laboratory houses a variety of equipment for use in exposing experimental plastic, elastomeric, lubricant, and adhesive materials to deleterious environments.

The Composite Preparations Laboratory contains filament winding machines, fume hoods, and a laminating table, used to prepare fiber-reinforced plastic. Metal, graphite, and glass fibers are the usual reinforcements, and epoxies are the primary resins utilized.

The Elastomers Laboratory contains equipment for fabricating and testing newly developed experimental elastomers.

6.7 STRUCTURAL MECHANICS LABORATORIES

The Structures and Vibrations Laboratories are equipped to study and analyze the strength and fatigue characteristics of structures and materials used in aerospace vehicle designs.

The equipment in the Structural Analysis Laboratory provides the capability for applying and controlling loads, deflections, and strains in the performance of tests upon structural members in tension, compression, flexure, creep, and fatigue. The system has four channels of closed-loop control with the capability of using each channel independently or using all four to load a single structure. The hydraulic actuators can be used in load frames for coupon testing, or mounted directly on the floor or wall for testing of a structure. The system includes a control console, a cyclic function generator, servo controllers, cycle counters, load frames, and a hydraulic power supply.

Equipment in the Vibrations Laboratory includes a 100-lb electromagnetic shaker with associated wave generator and amplifier, accelerometers, signal conditions, oscilloscopes, and recorders. This vibration facility is designed to conduct research programs in the areas of dynamic response, fatigue, and shock of structural materials and material systems.

6.8 NOISE AND VIBRATION LABORATORY

The Noise and Vibration Laboratory contains acoustical and audio frequency equipment and instrumentation essential for efficient evaluation and testing of noise and vibration related problems. The laboratory has the capability of simulating a noise environment approaching a free sound field within a recently constructed anechoic room. A full spectrum of research as related to acoustical phenomena can be tested and analyzed in this Laboratory.

6.9 SUPPORT FACILITIES

6.9.1 Electronics Laboratory

The Electronics Laboratory specializes in design, prototyping, fabrication, and testing of precision instrumentation and complex electronic control devices. These systems are in the areas of analog-to-digital conversion and logic interfacing, closed-loop servo systems, automatic data logic acquisition systems, special electronic measurement devices, transducer conditioning/readout devices, operational amplifier applications for differentiators, integrators, comparators, current and voltage modelts, etc. All of the above provide a unique service that is not available from an outside supplier. In addition, the laboratory includes an array of primary and secondary standards and comparison equipment essential to calibration, maintenance, and repair of test instrumentation.

6.9.2 Magnetics Materials and Measurements Laboratory

The Magnetics Materials and Measurements Laboratory is equipped for the preparation and evaluation of magnetic intermetallic compounds and permanent magnet materials of extremely high coercivities and energy products. Magnetic measurements are performed with the aid of a unique electronic laboratory hysteresigraph.

6.9.3 Special Instrument and Holography Laboratories

In this laboratory, facilities and equipment are available for development, assembly, and performance evaluation of electromechanical research apparatus. The laboratory contains two complete holographic camera systems. Both systems are presently being used to study nondestructive testing techniques for the Composite and Polymeric Materials Laboratories. In addition to holography, the laboratory supports the design and development of special optical and electro-optical apparatus.

6.9.4 Fabrication Shop

The University has a fully-equipped machine shop where specimens may be fabricated from complex and difficult-to-machine materials. Specialized testing apparatus, jigs, and fixtures are also fabricated in this facility. Its available equipment includes lathes, milling machines with numerous accessories, a shaper, a disk grinder, a band saw, surface grinders, drills, oxyacetylene and electrical welding rigs, and a large assortment of small tools, as well as extensive layout and inspection equipment. A special preparation area is separately located in this facility for the machining of exceptionally dirty or hazardous materials.

6.9.5 Programming Facilities

Full time professional programmers and student assistants comprise the programming staff of the Aerospace Mechanics and Applied Systems Analysis Division of the University of Dayton Research Institute (UDRI). In addition, many of the faculty and other engineers on the staff are competent FORTRAN programmers. Also available for consultation and assistance are the staff members of the Computer Science Department and the Office for Computing Activities.

6.9.6 Glass Blowing

A complete laboratory glass blowing facility is capable of handling all of the needs of the researcher. The laboratory personnel have experience with the fabrication of distillation apparatus, gas handling and vacuum systems, glassware for high and low temperature research, and analytical ware and vacuum forming of precision parts as well as general glass blowing. In addition to glass blowing facilities, the laboratory is equipped to slice and grind glass and ceramics, to silver and evacuate jacketed vessels, and to perform all of the other services common to a glass shop.

6.9.7 Information and Publication Services

UDRI operates its own technical library which serves the professional personnel within the Research Institute and the School of Engineering. The library function provides document acquisition by request, performs literature searching, provides current awareness services, and maintains an extensive collection of Defense Documentation Center, National Aeronautics and Space Administration, and Jet Propulsion Laboratory abstract journals. Grouped with the library activities are a technical editing service which assists in the writing and production of reports and publications, and the security function which processes and maintains security records for documents and personnel.

Included in the Graphic Arts Section are a complete drafting room, a photographic laboratory, and an offset reproduction facility. In addition to offset processes, reproduction can also be accomplished by means of Xerox, Ozalid, Mimeograph, and Ditto. All phases of report preparation including color and halftone offset work, collating, headlining, and binding can be accomplished by this Section.

SECTION 8

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