MATERIALS TESTING IN A VACUUM ENVIRONMEN

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ABSTRACT

Some general problem areas of materials testing in a vacuum environment are discussed in terms of component equipment with reference to specific requirements. These problem areas include: (1) the selection of a vacuum pumping system, as related to surface contamination, (2) pressure requirements as related to the particular materials test, (3) pressure measurement and residual gas composition analysis, (4) measurement of the investigative parameter when tests are conducted in a vacuum, (5) specimen thermal control at high and low temperatures in vacuum, (6) electrical, fluid, and motion transmission through a vacuum chamber wall. Selected case histories of vacuum apparatus, as applied to a particular materials test, are discussed. Also, when possible, the types of measurements made with each apparatus are presented. The systems discussed include zone-refining and monocrystal growing apparatus; tensile, fatigue and creep testing apparatus; cold-welding or adhesion apparatus; a combined space environment system; as well as vacuum applications to research instruments such as the electron microscope. Finally, the possible significance of the effects of vacuum environment on material properties is discussed in terms of both the scientific and engineering disciplines.

INTRODUCTION

It is natural to think of the earth's atmosphere as a benign environment. However, this environment is not passive when one considers its effects on some properties of materials - atmospheric corrosion of metals has been a problem for decades. Only recently have the more subtle effects of this atmosphere been shown to influence a material's behavior significantly. An example of this influence is the increase in fatigue life of lead by more than a factor of 60 in a vacuum environment, as compared to an air environment.[1] One of the first investigations which implied that air may affect the mechanical properties of metals was the early studies of Roscoe in 1934.[2] He noted that oxide films only 20 atoms thick on the surface of cadmium monocrystals caused a large increase in their strength. Since this beginning, the effects of various surface films,[3,4] surface conditions[5,6] and surface environments from liquids[7] to gases,[8,9] including vacuum[10,12] on the mechanical properties of metals have been investigated. From these and other studies, it has been demonstrated that many properties of materials are affected by active gases such as oxygen and water vapor in the environment, even when the concentrations relative to a pressure of 1 atmosphere are less than parts per billion to parts per trillion. This, of course, effectively eliminates, or at least severely limits, the usefulness of inert gases as a test environment - the lowest impurity levels of active gases attainable to date for inert gases are parts per million.

Until recently investigations of vacuum-sensitive material properties have been of interest primarily to the scientist or engineer who is studying a basic materials property. For example, in an investigation of the mechanics of plastic deformation within a metal, since the measured parameters

may be influenced by the interaction of the environment with the metal surface, the complete elimination of the surface environment (by testing in ultrahigh vacuum) may be required before the plastic deformation process occurring within the metal can be directly observed. With the advent of space exploration where a vacuum replaces air as the normal environment, these investigations have taken on new meaning and there has been increasing interest in the effects of vacuum on material properties.

Much progress has been made in the last few years in the state of the art of vacuum testing. These advances in the technology and economics of vacuum apparatus now permit the attainment of vacuum levels of the order of 10^{-11} torr and below, which are required in some materials tests, with relative simplicity and at a fraction of the cost that was heretofore possible. This paper will review some procedures, techniques, and equipment involved in the testing of materials in a vacuum environment, as well as results of some vacuum-sensitive property investigations. Because nearly all such materials tests to date have been performed on metals, this paper will primarily consider metals; however, the general vacuum techniques and apparatus to be discussed can be applied to the testing of all materials. The purpose of this paper is to make the test engineer more aware of the importance of and the techniques available for conducting materials tests in a vacuum environment. Problem areas as related to vacuum testing will be discussed, as well as specific test apparatus for particular test conditions with selected case histories from our laboratory.

GENERAL

The vacuum equipment and techniques employed in the testing of materials in a vacuum environment are dictated by the type of test, as well as by the

economics of the situation. However, the basic components of the vacuum system will, generally, remain the same with only the degree of sophistication varying. These basic components are (1) the pumping system made up of rough and fine pump stations with associated instrumentation, (2) the vacuum chamber with its associated equipment, and (3) the vacuum measuring apparatus. It will be seen that because of the wide variety of vacuum components available to the equipment designer, it is easy to custom-build a vacuum system for a particular materials test.

I have not the time to enter into an extended discourse on the merits and faults of available vacuum components - their merits can be obtained readily from the various vacuum component manufacturers - however, there do exist some special considerations, techniques, and components involved in the testing of materials in a vacuum environment which I will discuss in some detail.

Pumping System

The vacuum pumping system consists of two pumping stations - the rough pump station and the fine pump station. The rough pump station is used to evacuate the vacuum system initially to pressures at which the fine pump station can be started. This pressure is in the range of 1 to 20 microns, depending on the characteristics of the fine pump station. One can choose roughing pumps from a variety of mechanical and cryosorption models. When cryosorption pumps are used, several are employed at various stages of pump-down. These pumps have an advantage over mechanical pumps in that they are free of any possible oil vapor contamination of the vacuum system and can generally obtain lower pressures. However, unlike the mechanical pump, they

are not capable of continuous operation and require periodic bakeout. When oil or mercury diffusion pumps are employed in the fine pump station, the rough pumping station must serve a dual role since it also acts as a back-up or foreline pump to the fine pump station. Under these conditions, continuously operating mechanical pumps must be used.

Vacuum pumps in the fine pumping station serve to evacuate the vacuum system from roughing pressure down to the system's ultimate pressure. These pumps are generally oil or mercury diffusion pumps or sputter-ion pumps, the latter usually including a titanium sublimation pump, TSP. The sputter-ion pump, together with a TSP, has the advantage of eliminating any possible oil contamination of the vacuum system. However, when compared to a diffusion pump system, this pumping system has the disadvantages of a higher cost for a given pumping speed and a wider variation in pumping rates for various gas species - the easily ionized species are pumped much more rapidly than the more inert species.

Chamber Materials and Construction

The vacuum chamber material and the method of chamber fabrication can determine the ultimate pressure attainable in a vacuum system. In the past, all-glass systems were required if the system was to attain ultrahigh vacuum, 1×10^{-9} torr or lower. However, materials, fabrication, and sealing techniques have recently been developed which permit the use of single-wall, all-metal, or metal plus glass systems even at these low pressures.

The chamber material, as well as other components exposed to the vacuum environment, is usually 300 series stainless steel relatively free of porosity. When welding is required, gas shield methods are employed to minimize porosity

and welds are made only on the interior of the chamber to eliminate any gas pockets between the exterior and interior of the chamber wall. If gas permeation through the chamber wall is a problem, which may be the case at very low pressures or at high temperatures, a thin oxide coating on the exterior of the chamber has been found to minimize this problem. The development of the now standard all-metal seal flanges is a major contribution to the use of metal vacuum systems. With this technique, a metal gasket - usually made of OFHC copper - is compressed between two ridges on the two mating flanges.

System Contamination

In many materials tests conducted in a vacuum to investigate a particular material property which is environment sensitive, it is important that the surface of the test material be initially free of foreign contamination, or, in some tests, that this contaminant (say an oxide film) be inherent to the test material. This means the material surface must be cleaned by heating the specimen to high temperatures,[13] by ion-sputtering,[14] or by some other in situ technique, or that the nature of the contaminant be known.

The vacuum level, of course, determines the degree of residual gas contaminants in the environment. However, foreign contaminants, such as oil, cannot be tolerated in many materials tests. The source of this contaminant is usually the diffusion- or mechanical-pump. The method of pumping often depends on the degree of oil contamination that can be tolerated in a particular materials test.

The pumping system most free of oil contamination utilizes a cryosorption-type roughing pump together with a sputter-ion fine vacuum pump. However, many times the use of such a pumping system is not economically feasible.

If a mechanical roughing pump is used, the vacuum environment should always be shielded from oil contamination by a zeolite trap and/or a liquid-nitrogen trap with antimigration baffles. Likewise, when oil diffusion pumps are employed, the same types of traps must be used. Even with these traps, oil migration into the vacuum environment can be a problem; therefore, methods of determining the existence and extent of oil contamination are important.

There are several methods of detecting oil in a vacuum. A mass spectrometer may be used to examine the residual gas spectrum.[15] This technique is very sensitive but the mass spectrometer is expensive. A standard dual-beam ultraviolet absorption spectrophotometer may be used to determine the total amount of oil collected during a run.[16] This technique is very sensitive but is not an in situ technique and gives only the average collection rate over the period of the test. A radioactive tracer technique[17] has also been developed. This technique is extremely sensitive but is limited by the short half-life available and requires the use of an irradiating reactor. A quartz-crystal microbalance has been used[18] but this technique does not discriminate among oils and other condensables. And, lastly, a single-beam ultraviolet spectrophotometer technique has been developed which will continuously monitor the presence and extent of oil contamination in situ.[19] Such a system is shown schematically in Figure 1 and consists simply of radiation from a mercury vapor lamp which penetrates a transparent cooled collecting disk in the vacuum system. The transmitted beam is then analyzed by means of a monochromator and photomultiplier system. With the present state-of-the-art, this technique is not quite as sensitive as some other techniques; and, in determining the extent of contamination, it is limited to film growth rates for oil of greater than 2 Å in thickness per minute.

Pressure Requirements

The pressure requirements for any particular materials test will depend on both the material and property being investigated. Assuming that every molecule striking the surface of a test specimen will adhere, Table I lists values of the molecular incidence rate and time to form a monolayer for air at 25°C.[20] It can be seen that if the investigation involves a property which is very sensitive to any surface contaminant - for example, the nucleation of a metal vapor on a freshly cleaved substrate - pressures will be required that will allow the surface to remain essentially clean for the duration of the test.

Tests that involve more dynamic properties, such as tensile and fatigue deformation studies, can be accomplished at pressures higher than would be apparent from Table I. Examples of results of such tests are shown in Figures 2 and 3. Figure 2 is a plot of the change in shear strain at the end of the first linear stage in vacuum, as compared to that in air at 760 torr, versus pressure for magnesium monocrystals oriented for basal glide which were tested in tension[21] at various strain rates. It can be seen that the transition from properties characteristic of air to those of vacuum occurs in the pressure range from 10^{-6} to 10^{-10} torr, for the strain rates of these tests. Figure 3 is a plot of number of cycles to failure versus pressure for polycrystal magnesium. As can be seen, the transition of the fatigue properties of magnesium polycrystalline specimens occurs at 10^{-3} to 10^{-6} torr at 2 cycles per second[22] and at 10^{1} to 10^{-2} torr at 30 cycles per second.[23] The reasons for these variations in the transition pressures from property to property are that competing processes are occurring such as the rate of

TABLE I

Time to Form a Monolayer for Air at 25° C

Pressure, torr	Time to form monolayer, sec
760	2.63×10^{-9}
1	2.00×10^{-6}
10^{-3}	2.00×10^{-3}
10^{-6}	2.00×10^{0}
10^{-9}	2.00×10^{3}
10^{-12}	2.00×10^{6}
10^{-15}	2.00×10^{9}

clean surface formation by deformation in a tensile test or by crack propagation in a fatigue test and the rate of clean surface contamination by adsorption.

Many other examples could be given to demonstrate the dependence of the pressure requirement on the particular test being conducted; however, it is sufficient to say that this can be a major consideration in the design and required sophistication of the vacuum apparatus for a particular set of test conditions.

Pressure Measurement

A knowledge of the pressure of the vacuum environment is fundamental to studies of pressure-dependent phenomena. Additionally, it is often important that the composition of the vacuum environment be known. There are many types of vacuum gages, some of which are capable of measuring total pressures below 10^{-12} torr. Some of the commonly used pressure gages are listed in Table II by operational mode with their approximate useful pressure range. The principles on which these gages are based, as well as the details of the specific gages, have been discussed by others in great detail[20,28] and thus will not be presented here.

The ability to measure pressure with a reasonable degree of confidence for a given vacuum gage is, of course, related to the method of calibration, as well as to its inherent repeatability. Several gage calibration systems have been developed. Probably the most simple is the direct-comparison type in which a standard gage is used to measure the pressure at which the test gages are then calibrated. Another method of calibration utilizes the pressure-ratio technique.[29] This technique has the advantage of extending

TABLE II

The Basic Operational Modes of Pressure Gages Employed to Measure Vacuum and Their Approximate Useful Pressure Range

Operational Mode	Approximate useful range (torr)	Typical vacuum gages
Force	760 - 1	Liquid manometer
Compression	100 - 10^{-5}	McLeod gage[24]
Transport	760 - 10^{-3}	Thermocouple gage
Radioactive ionization	760 - 10^{-5}	Alphatron[25]
Discharge ionization	10 - 10^{-11}	Cold-cathode-ionization gage (Redhead gage[26])
Thermionic ionization	10^{-3} - 10^{-9}	Hot-cathode-ionization gage (Bayard-Alpert gage[27])
Gas analysis ionization	10^{-2} - 10^{-12}	Residual gas mass spectrometer

the pressure range in which the test gage can be compared to the standard gage. A pressure-ratio gage comparison system suitable for gage calibration to 1×10^{-13} torr is shown in Figure 4.[30] This figure is shown to indicate the complexity of such an apparatus. This particular system consists of the two test chambers and associated equipment suspended from a bulkhead. An outer chamber shown at the right can enclose this equipment with a vacuum environment to facilitate attaining the very low required pressures in the test chambers. With this system a test gage can be calibrated at 2×10^{-13} torr against a standard gage at 1×10^{-10} torr.

As has been mentioned, a knowledge of the residual gas composition in the vacuum can often be important because various component gases in the vacuum environment can affect the results of a materials test. An example of this effect is shown in figure 5 in which the fatigue life in the test-gas environment is compared with the fatigue life in vacuum for magnesium, copper, brass, and two aluminum alloys. It is seen that the fatigue life for a given metal varies significantly when tested in different gases, and, for a given gas, it varies significantly for the metals tested.

An additional consideration in gas composition monitoring is that the residual gas composition can vary significantly from vacuum system to vacuum system and, in fact, from pumpdown to pumpdown in the same system. This latter effect is due to small leaks which may develop, changes in the pumping speed of the vacuum pumps due to contamination, etc. Thus, for consistent and meaningful results in many materials tests, a knowledge of the residual gas composition during each test may be imperative. Several instruments exist for determining the residual gas environment on the basis of gas

ionization and separation. These instruments include quadratic-sector, quadrupole, monopole, and time-of-flight type mass spectrometers. The theory and use of these instruments have been discussed in detail elsewhere[31,32] and will not be presented here.

Sensors for Measuring Material Properties

Sensing the particular test parameters on which the materials test is based, such as stress and strain in a deformation test, can be a severe problem when the test is conducted in a vacuum. It is many times imperative that the sensing instrument be placed in contact with the specimen. In an air test, this requirement is not a particular problem and standard sensing instruments can be used; however, in vacuum this is not necessarily the case because of volatile components in the instruments.

Let us consider a deformation test as an example of the problems encountered in sensing in a vacuum environment. At pressures greater than about 10^{-7} torr, the standard load cell and strain measuring apparatus, such as a resistance strain gage or LVDT, linear-variable-differential-transformer, may be used. However, even at these relatively high pressures, care must be exercised to ensure that the instruments used do not contain highly volatile materials. At pressures in the range of 10^{-7} to 10^{-10} torr, specialized load cells and strain sensing apparatus must be used. These instruments are commercially available and contain such things as potting compounds free of volatile components, or enclosed in a hermetically sealed case, etc. At pressures less than 10^{-10} torr, most commercial stress-strain sensing apparatus is not compatible with the vacuum environment and many times complex external sensor systems must be developed. One such typical system is shown

in figure 6. This system employs an external load cell connected to the tensile test system through a welded metal bellows. Specimen elongation can be measured externally by the use of, for example, an optical strain sensing system. By sighting through a transparent window on the vacuum chamber, the optical system would measure the elongation of the specimen by comparing the relative motion of the two ends of the gage section. Depending on the optical system, this comparison would be made physically by the investigator or electronically by rather complex electronic equipment.

Specimen Heating and Cooling

When specimen temperatures other than ambient are required in a vacuum environment, temperature uniformity and control becomes a problem partly because of the lack of convective heat transfer. At temperatures below ambient, the desired specimen temperature can best be obtained by conduction. This is done through grips or blocks in intimate contact with the specimen which are cooled by some coolant, such as liquid nitrogen. Depending on specimen length and temperature uniformity required, it is often necessary that the specimen be shielded from sources of radiative heat (e.g., chamber walls) by a cold shroud.

At specimen temperatures above ambient, conductive, radiative, and induction heating can be used. If radiative heating is employed, it is many times desirable to isolate the resistance heating filament from the vacuum environment by the use of an envelope such as a sealed quartz tube. Such infrared heaters with tungsten filaments and quartz envelopes are commercially available. If the resistance element is in direct contact with the vacuum environment, care must be taken to select high purity elements that have a minimum of out-gassing when they are hot.

In high-temperature testing, consideration must also be given to minimizing heating of chamber walls and internal components. This is because of the increased outgassing rates and volatilization of surface contaminants which occur when these components are heated. Cooled shrouds and/or heat reflectors around the specimen are often required to minimize this problem.

Dynamic Feedthroughs

In most materials involving the manipulation of the specimen in a vacuum environment, it is often physically impossible and also undesirable to have the integrated test system within the vacuum jacket. Therefore, motion feedthroughs are an important part of the test design. Depending on the test requirements, many commerical systems are available, some of which are shown conceptually in figure 7. The most simple motion feedthrough uses the simple "O-ring" seal, figure 7(a), which permits the transmission of linear or rotary motion. This seal is a compressed polymeric "O-ring" on a shaft. Such a seal is generally limited to use at pressures to about 10^{-7} torr because outgassing and unacceptable leak rates. Below 10^{-7} torr, the methods of motion transmittal become more complex. For positive linear motion, the welded metallic bellows is generally employed, figure 7(b). This feedthrough has excellent vacuum integrity. It should be pointed out that consideration must be given to forces applied to the load rod by the vacuum force component on the bellows as well as by its spring constant. There are several excellent feedthrough systems for transmitting rotary motion. When positive motion is not required, a magnetic feedthrough (Figure 7(c)) is commonly used. In this system, a magnet outside the vacuum chamber is placed around an iron core within the vacuum chamber. The core and magnet are separated by a nonmagnetic

can which is an integral part of the vacuum jacket. Thus, any rotation of the external magnet will result in a coupled motion of the iron core. When positive rotary motion is required, the metallic bellows sealed wobble stick, figure 7(d), or a harmonic-drive system[33] with a flexible vacuum can, figure 7(e), may be employed.

Static Feedthroughs

Many static feedthroughs for such entities as low frequency and R.F. electrical transmission through a vacuum jacket are commercially available. Detailed information on these can easily be obtained and thus will not be discussed here. However, fluid transmission through a vacuum environment can be a significant problem, primarily because of leaks, and will be discussed briefly. Figure 8 is a diagram of a method that eliminates any mechanical connections in the transmission tubes within the vacuum chamber. Since mechanical connections in the transmission lines have the greatest propensity to leak, this transmission method has a distinct advantage over most others. As is seen from the figure, a continuous transmission tube is welded to a metallic flange which has a reverse seal; that is, it is sealed at the interior rather than the exterior of the vacuum jacket. Additionally, this system allows easy removal of the transmission tube assembly from the vacuum chamber.

CASE HISTORIES

Because numerous vacuum components with various degrees of sophistication are readily available and are interchangeable, and because each materials test requires a specific set of test conditions, vacuum apparatus is generally designed for a particular materials test. The apparatus and techniques

discussed above, as well as other ideas, have been applied by investigators in our laboratory to the design of vacuum apparatus for particular test requirements. Selected case histories will be presented for a variety of materials tests. Each test system will be discussed, first in terms of any meaningful observations stemming from the use of this or similar apparatus with an example, when possible, and, second, in terms of the system's basic capabilities to demonstrate the use of vacuum techniques as discussed above.

Vacuum Zone-Refining and Monocrystal Growing Apparatus

It is generally known that small amounts of impurities in a metal may rather drastically affect many mechanical and physical properties of that metal. An example is the effect of purity on the deformation behavior of iron single crystals at -196°C. It has been observed that in relatively impure iron crystals at -196°C, all orientations are not ductile; rather a ductile-brittle boundary exists where failure mode changes from ductile tearing to brittle cleavage.[34] Edmondson[35] observed that this boundary is about 16° from the (100) pole of the unit triangle, and crystals oriented nearer than 16° to the (100) were completely brittle. Biggs and Pratt[36] have further shown that increasing the purity of iron crystals has a tendency to move this boundary toward the (100) pole. Studies conducted by Hughes and Barton[37] on crystals grown by the floating-zone technique showed that sufficient purity could be obtained that all orientations of crystals tested at -196°C were ductile and, in fact, the greatest strain to fracture was observed for orientations near the (100) pole.

To investigate the effects of selected impurities, as well as the properties of the pure metal itself, it is essential that materials be produced

having the highest possible purity. Figure 9(a) is a photograph of a zone-refining and monocrystal growing apparatus in which such high-purity materials can be processed. This system can accommodate specimens from 4 to 12 inches long and up to 1/2 inch in diameter, depending on the specific material, and has the capability of ultrahigh vacuum purification of refractory metals and ceramics. Purification occurs by the standard zone refining technique, as well as by vacuum degassing and evaporation of volatile impurities from the molten zone. Additionally, this system can grow smooth, concentric, cylindrical or hemispherical monocrystals of random or predetermined orientations.

The vacuum portion of the apparatus has an all-metal-sealed stainless steel vacuum chamber to lessen the possibility of leaks and to permit high temperature bakeout of the chamber to facilitate the removal of volatile contaminates from the chamber walls. The system uses an ion-pump together with a titanium sublimation pump to eliminate oil contamination and is capable of operating at pressures of the order of 10^{-10} torr.

The titanium sublimation pump is surrounded by a liquid-nitrogen-cooled shroud on which the titanium is sublimed. The use of this shroud decreases the pump-down time by increasing the sticking coefficient and residence time of species that contact the titanium-covered shroud. Employing this technique the system can be pumped to 5×10^{-9} torr in 2-1/2 hours.

The zone refining module within the vacuum jacket is all-metal. One contact surface of all sliding surfaces is gold-plated to reduce friction and minimize cold-welding. Figure 9(b) is a photograph of the module assembly. This assembly supports the specimen to be purified, rotates the specimen, and contains a small goniometer which may be used for the growth of oriented

monocrystals. The selected material is purified or a crystal is grown by the movement of a narrow molten zone along the specimen. This zone, formed by an electron beam, is sufficiently small that the configuration of the specimen is retained by surface tension without the use of external constraints - the floating zone technique. This technique facilitates purification by maintaining the vacuum environment in contact with the zone during solidification, thus eliminating contamination by containers.

Uniaxial Testing

To date, the bulk of the studies conducted on the effects of a vacuum environment have been concerned with the mechanical behavior of metals. It has been observed that the significant effect of vacuum on magnesium, for example, is to increase its ductility. The magnitude of this effect is dependent on grain size, as shown in figure 10.[22] This figure is a plot of stress versus strain for polycrystal magnesium at two pressures and grain sizes. It is seen for this specimen cross section, that a vacuum environment of 5×10^{-8} torr had no observable effect on tensile properties at a grain size of 0.01 mm but did at a larger grain size of 0.20 mm. A more pronounced effect of vacuum was observed in the change in tensile properties of monocrystals as shown in figure 11.[21] This figure is a plot of shear stress versus shear strain at a constant strain rate and varying pressures for monocrystal magnesium oriented for basal glide. It is seen that a vacuum environment mainly affects the extent of the first linear stage. Similar effects have been observed in investigations of other metals.

Equipment designed to investigate the effects of vacuum on such properties as materials' strength and ductility in uniaxial tension must have the

capability of reaching pressures from 1×10^{-8} to 1×10^{-10} torr, depending on the desired parameters of the test. Figure 12 is a schematic diagram of an apparatus designed for uniaxial testing. The vacuum system for this apparatus is relatively inexpensive. For reasons already discussed, it consists of a small stainless steel chamber with all-metal seals, is rough pumped by a zeolite-trapped mechanical pump, and is fine pumped by a sputter-ion pump integrated into the test chamber, as well as by a titanium sublimation pump exposed to a liquid-nitrogen-cooled shroud. The system is designed to fit between the columns of a standard tensile testing machine to eliminate any specially designed loading apparatus. This vacuum system has a pressure capability of 1×10^{-10} torr or better. Additionally, preselected gases can be introduced into the chamber, when desired, by the use of a controlled leak valve, and the gas composition can be measured by a quadrupole-type residual gas analyzer. Motion is transmitted into the chamber by bellows-sealed load rods. Both load and specimen deflection are measured externally.

To facilitate the attainment of low pressures, bakeout heaters are used for rapidly removing volatile species from the chamber walls. While the heating elements for many bakeout systems are external to the chamber, the elements for this system are contained within the vacuum chamber. They consist of tungsten resistance elements within sealed quartz tubes; thus, they are isolated from the vacuum environment, Figure 13. This bakeout system has several advantages over the normally used external system in that it is relatively inexpensive to install, it uses a nominal amount of power (8 amp at 110 V for the system shown), it has a much faster response time, it heats primarily those surfaces and components exposed to the vacuum

environment, it does not require external insulation to obtain reasonable bakeout temperatures (up to 350°C), and it maintains the clean exterior of the vacuum chamber thus facilitating the mounting of equipment, etc.

Fatigue Testing

From the known effect of vacuum on the deformation behavior of a metal, particularly near-surface deformation, one must deduce that a vacuum environment may have a major influence on the fatigue life of a material. This has been studied extensively.[10,11] One such study is included in the paper by Sumsion in this Proceedings, and is also reported elsewhere.[23] Briefly, figure 14 is a typical example of the effect of vacuum on the fatigue properties of metals. This figure is the data for a polycrystal magnesium-thorium alloy and is a plot of maximum stress versus fatigue life. As is seen from the figure, a significant increase in fatigue life is observed for a given maximum stress in vacuum, when compared to an air environment.

Figure 15 shows a simple vacuum fatigue apparatus which employs a cantilever type specimen. As is seen from the figure, the vacuum system is designed to be mounted on a standard commercial fatigue machine to eliminate the need for a special test apparatus. The vacuum system is capable of pressures down to 1×10^{-8} torr and is pumped by the use of a sputter-ion pump together with a titanium sublimation pump. The cyclic load is transmitted to the specimen by a metal-bellows-sealed load rod. Also included in this system is a residual gas mass spectrometer due to the extreme importance of the composition of the residual environment on the observed fatigue properties, figure 5.

Creep Testing

Creep deformation has been shown to be an environment-sensitive mechanical property for several metals. An example of this effect is shown in figure 16[22] for polycrystal, high-purity magnesium. This figure is a plot of steady-state strain rate versus the reciprocal of absolute temperature at a constant applied stress. The dashed lines are curves determined from data obtained in an air-argon environment and the solid line with data points is for tests conducted at pressures less than 4×10^{-9} torr. As can be seen, the effect of vacuum is a function of the test temperature. The creep resistance of magnesium is increased at low temperatures and decreased at high temperatures in vacuum as compared to an air-argon environment.

Figure 17 is a schematic drawing of a medium-temperature, vacuum creep apparatus capable of pressures of the order of 1×10^{-9} torr and temperatures up to 600° C. It is pumped with a sputter-ion pump integral with the stainless steel vacuum chamber and a titanium sublimation pump having a liquid-nitrogen-cooled surface. Dead-weight load is applied to a hollow cylindrical specimen in the torsion mode by the use of a large magnetic rotary-motion feedthrough. Specimen deformation is monitored external to the chamber. The specimen is heated by quartz-sealed resistance heaters surrounded by stainless steel heat reflectors to eliminate excess heating of other components in the system.

Figure 18 has photographs of a high-temperature ultrahigh-vacuum creep apparatus designed to fit between the columns of a standard testing machine. This system has a single-wall stainless steel vacuum chamber, pumped with a sputter-ion pump and a titanium sublimation pump and is capable of ultimate

pressures of less than 5×10^{-12} torr as measured by a residual gas mass spectrometer. The operating temperature range of the test zone is -196° to 3000° C. The low-temperature capability is, of course, obtained by liquid-nitrogen cooling and the high temperatures are obtained by a resistance-heated tungsten mesh element. At test temperatures of 1000° C, the system is capable of pressures of less than 1×10^{-10} torr and at 3000° C, 1×10^{-8} torr. These pressures appear to be limited only by the vapor pressure of the tungsten filament. Because the system is designed to fit a standard tensile test machine, this system may also be employed for tensile testing, uniaxial fatigue testing, etc.

This test apparatus is being applied to the study of the high-temperature creep behavior of refractory metals. Such a system is required because of the high chemical reactivity of these alloys at elevated temperatures with environments containing carbon, oxygen, or nitrogen. This reaction in refractory metals containing alloy additions of such elements as hafnium, zirconium, and titanium results in the formation of stable carbides, oxides, or nitrides which can cause significant changes in the mechanical behavior of the alloy.

Adhesion Testing

Adhesion, or cold welding, and a related phenomenon, friction, are environment-sensitive properties of metals. Uncontaminated metal surfaces (clean) will form an interface bond having a mechanical strength as great as the parent metal. Investigators have shown that small amounts of oxygen, air, and other such gases will significantly decrease the adhesion of initially clean metal surfaces.[38,39] Thus, surface films, such as oxides, inhibit

the adhesion of most metals and make the use of a vacuum environment mandatory for the preparation and study of the adhesion behavior of uncontaminated metals.

Numerous methods exist for the study of the phenomenon of adhesion; however, in all studies the metal surfaces must be clean initially. The simplest approach to this requirement is to work with specimens which are fractured in situ at pressures of the order of 10^{-10} torr.[40,41] Such adhesion studies have been conducted in the vacuum apparatus shown in figure 12, which fits between the columns of a standard testing machine. The capabilities of this apparatus have been discussed earlier. The test specimen consists of a simple notched bar. The test procedure is to fracture the specimen in vacuum, expose the fracture surfaces to the desired environment for a measured interval, and rejoin the surfaces with a given compressive load. The tensile force for subsequent fracture is a measure of the adhesion. Figure 19 is an example of the data obtained in this apparatus.[41] This figure is a plot of the adhesion coefficient, α, versus the exposure to selected environments. The adhesion coefficient is defined as the load required to separate an interface joint divided by the load applied to form the joint. The exposure time is simply the partial pressure of the gas of interest multiplied by the length of time the clean interfaces are exposed to this environment. As is seen from the figure, various gases affect adhesion to different degrees. Of those gases shown, oxygen is the most severe.

Studies have been conducted by several investigators to delineate the important variables involved in controlling the friction coefficient of metals in vacuum. Some of the more extensive work is that conducted by

Buckley.[42] The system employed by Buckley is shown in figure 20.[43] This system is pumped by the use of a cryosorption rough pump, a sputter-ion pump, and a cryopump and is capable of pressures less than 10^{-10} torr. The cryopump is simply a metal surface cooled to temperatures near liquid helium and exposed to the vacuum environment. An electron beam gun is employed to help clean contacting surfaces by electron bombardment. This gun is also used to heat the contacting surfaces to the desired temperature and the cryopump is employed to cool the contact surfaces when desired. A disk in contact with a rider is rotated by a magnetic drive and the friction force is determined by a strain gage on the rider arm. The contact load between the disk and rider is applied by a dead-weight load system as shown. An example of data taken in such a system is work conducted on copper. Copper, when exposed to laboratory air, has little tendency to self-weld and has a friction coefficient of 1.2; however, in a vacuum environment, copper self-welds readily and its friction coefficient prior to complete seizure is in excess of 40.[44]

Space Environment Simulation

Vacuum is, of course, one important parameter in any system designed to simulate the space environment. Figure 21(a) is a schematic of a space environment simulator capable of operating at pressures below 10^{-10} torr[45] in combination with photon and charged-particle radiation, micrometeoroid bombardment, and temperature which is used for studies of material properties which may be influenced by exposure to the space environment.

The vacuum chamber is shown in greater detail in figure 21(b). It is 8 ft in diameter and 11 ft long with a hinged end door. The chamber is pumped

by a 32-inch oil-diffusion pump due to its large volume and a 5-ft diameter by 5-ft-long cylindrical cryopump array to facilitate the attainment of low pressures. The cryopump array is designed to use liquid nitrogen for the outer shell and for chevron panels which protect the 20° Kelvin helium cryopanels from thermal radiation. The system is designed to achieve a pressure of 10^{-10} torr in 12 hours after bakeout of the chamber.

Some Unique Devices and Concepts

Because a vacuum is, in general, the most nonreactive environment available, it has application to apparatus or devices which are used to study fundamental characteristics of materials, such as the electron microscope, low-energy-electron diffraction apparatus, etc. These apparatus differ from what has generally been discussed to this point in that they are tools for study of materials and not for making materials tests per se. Some of these devices which utilize rather unique vacuum techniques and concepts will be discussed briefly.

The contamination of targets in such instruments as ion accelerators and electron microscopes has always been a problem. This contamination originates from such components as the electron or ion source, the numerous polymeric materials generally used as both static and dynamic vacuum seals, the focusing and condensing coils, the vacuum pumping system itself, etc. As has been discussed earlier, such items in the vacuum environment generally tend to limit system capability to above 10^{-7} torr. Additionally, because of the high gas loads required in these instruments, oil-diffusion and mechanical pumps are used which can contribute oil vapor as a contaminant. A simple and relatively inexpensive solution to this problem of target

contamination is to establish and maintain a relatively high vacuum only in the immediate vicinity of the target. One successful approach to this solution is the use of the differential pumping technique. In this technique two independent pumping systems are used - one system for the target chamber and the other for the remainder of the instrument vacuum column. The main column and the target chamber are connected by a small orifice which passes the beam but which minimizes flow of contaminants into the target chamber. Under such conditions, a pressure difference of three to four orders of magnitude can be maintained with the target chamber having a reasonably low pumping speed.

When the primary beam is too wide to permit the use of a limited-conductance orifice, it is many times possible to separate the vacuum of the target chamber from that of the instrument by the use of a membrane which is permeable to the beam and impermeable to other gases. Such a technique has been developed for use on a conventional proton accelerator.[46] A thin (200 to 1000 Å) film of Al_2O_3 is used as a beam window between the normal accelerator vacuum of 2×10^{-6} torr and the clean target-area vacuum of 2×10^{-10} torr. This window is essentially transparent to the proton beam but impermeable to other gases.

Another example of the use of the differential pumping technique is an ultrahigh vacuum electron microscope in use in our laboratory. The modified specimen chamber of a standard electron microscope is isolated from the main instrument by both the limited-conductance and the film-window techniques. The electron beam is small in cross section before it impinges on the specimen; thus, the limited-conductance technique is feasible at the beam entrance to the specimen chamber. As the beam passes through the specimen, it spreads

and the emergent beam is so large that the limited-conductance technique is not feasible for the chamber exit window. In this case, a carbon-film window is employed. In situ studies of nucleation on clean substrates[47] and of other phenomena which require the presence of an ultrahigh vacuum environment in such an instrument can thus be made.

The development of other instruments, which are proving and have proved to be very useful tools in the study of materials, have also come about by the development of ultrahigh vacuum technology. Such instruments are the low-energy-electron-diffraction apparatus,[48] the field-ion microscope,[49] and the surface-ion mass spectrometer.[50] It is thus only reasonable to assume that in the future many more such instruments will be developed to help understand both the bulk and surface behavior of materials.

SIGNIFICANCE OF VACUUM TESTING

It has been shown that materials testing in a vacuum environment can involve simple or complex vacuum apparatus, depending on the vacuum requirement of the materials test as dictated by the particular property and material being investigated. Additionally, an attempt has been made to indicate the influence of a vacuum environment on some properties of materials. To date, most of these studies may be considered to be preliminary and, in general, the findings have not been used in a practical manner by design engineers.

It is the author's opinion that materials studies in a vacuum environment will, in the future, contribute significantly to both the scientific and engineering understanding of materials; as a result, improved materials will be developed for use both on earth and in space. The scientist interested

in a better understanding of the fundamental mechanisms involved in the behavior of materials has begun to realize the significance of contributions of surfaces and the surface environment to the experimentally observed property. It is reasonable to assume that, when the mechanics of the surface effects are understood, methods for improving materials for use in various environments will become apparent to the engineer. Even now progress in this direction is under way. As an example, the fatigue of metals is a materials property which is generally improved by the presence of a vacuum environment. Recently, it has been shown that fatigue life can also be improved in the earth's environment by the application of a simple coating.[23]

REFERENCES

1. Snowden, K. V. and Greenwood, J. N., *Trans. AIME*, 212, 626 (1958).
2. Roscoe, R., *Nature*, 133, 912 (1934).
3. Garstene, J., Honeycombe, R. W. K., and Greetham, G., *Acta Met.*, 5, 484 (1956).
4. Mitzger, M. and Read, T. A., *Trans. AIME*, 212, 236 (1958).
5. Sherman, R. J. and Achter, M. R., *Trans. AIME*, 224, 1072 (1962).
6. Kramer, I. R., *Trans. AIME*, 227, 1003 (1963).
7. Kramer, I. R., *Trans. AIME*, 221, 989 (1961).
8. Hancock, G. G. and Johnson, H. H., *Trans. AIME*, 236, 513 (1966).
9. Bradshaw, F. J. and Wheeler, C., *Applied Mat. Res.*, 5, 112 (1966).
10. Hordon, M. J., *Acta Met.*, 14, 1173 (1966).
11. Grosskrentz, J. C. and Bowles, C. Q., "Proc. Conf. Environment-Sensitive Mechanical Behavior of Materials," 1966, Westwood, A.R.C. and Stoloff, N. S., Eds. (Gordon and Breach, N. Y. 1967), p. 67.
12. Williams, D. P. and Nelson, H. G., *Trans. AIME*, 233, 1339 (1965).
13. McCarroll, B., *Surface Sci.*, 7, 499 (1967).
14. Farnsworth, H. E., Schlier, R. E., George, T. H., and Burger, R. M., *J. Appl. Phys.*, 29, 1150 (1958).
15. Gosselin, C. M. and Bryant, P. J., *J. Vac. Sci. Technol.*, 2, 293 (1965).
16. Langdon and Fochtman, *Trans. 10th AVS Vac. Symp.*, 1963 (The Macmillan Co., N. Y. 1963), p. 128.
17. Carter, J. G., Elder, J. A., Birkhoff, R. D. and Roecklein, A. K., *J. Vac. Sci. Technol.*, 2, 59 (1965).

18. Deville, J. P. Holland, L. and Laurenson, L., "Advances in Vacuum Science and Technology," H. Adams, Ed. (Pergamon Press, Inc., N. Y., 1967), Vol. 2, Part 1, p. 153.

19. Supple, R. W. and Gloria, H. R., *J. Vac. Sci. and Technol.*, 4, 276 (1967).

20. Santeler, D. J., Holkeboer, D. H., Jones, D. W. and Pagano, F., "Vacuum Technology and Space Simulation," NASA SP-105, 1966, p. 5.

21. Nelson, H. G. and Williams, D. P., "Proc. Conf. Environment-Sensitive Mechanical Behavior of Materials," 1966, Westwood, A.R.C. and Stoloff, N.S., Eds., (Gordon and Breach, N. Y., 1967) p. 107.

22. Nelson, H. G. and Williams, D. P., "Proc. Conf. Effects of Space Environment on Materials, SAMPE," 1967 (Western Periodicals Co., Hollywood, Calif., 1967), p. 291.

23. Sumsion, H. T., to be published, *J. Spacecraft and Rockets* (1968).

24. Lott, P., *Anal. Chem.*, 28, 276 (1956).

25. Vacca, R. H., "Trans. National Symposium of Vacuum Technology," 1957 (Pergamon Press), p. 93.

26. Redhead, P. A., *Can. J. Phys.*, 37, 1260 (1959).

27. Nottingham, W. B., "Trans. Vacuum Symposium," 1955 (Committee on Vacuum Techniques, Inc., Boston), p. 76.

28. Dushman, S., "Scientific Foundations of Vacuum Technique," 1958 (John Wiley and Sons, Inc., N. Y.).

29. Hobson, J. P., *Can. J. Phys.*, 37, 300 (1959).

30. Supple, R. W., Petrucci, L. J. and Bruce, H. W., *Abstracts 14th AVS Vac. Symp.*, 1967 (Herbick and Held Printing Co., Pittsburgh, Pa., 1967), p. 3-1.

31. Kiser, R. W., "Introduction to Mass Spectrometry and Its Applications," (Prentice-Hall, Inc., Englewood Cliffs, N. J., 1965).

32. "Fifteenth Annual Conference on Mass Spectrometry and Allied Topics," Denver, Col., 1967, ASTM.

33. Harmonic Drive Division, United Shoe Machinery Corp., Beverly, Mass., Bulletin No. 46-0432310-63.

34. Allen, N. P., Hopkins, B. E., and McLennan, J. E., *Proc. Roy. Soc.*, 234A, 221 (1959).

35. Edmondson, B., *Proc. Roy. Soc.*, 264A, 176 (1961).

36. Biggs, W. D. and Pratt, P. L., *Acta Met.*, 6, 694 (1958).

37. Hughes, E. J. and Barton, P. W., *RMIC Bulletin*, Oak Ridge National Lab., June, 1964.

38. Bowden, F. P., and Tabor, D., "Friction and Lubrication of Solids, II," Oxford University Press, London, 1964.

39. Gilbreath, W. P., "Symposium on Adhesion or Cold-Welding of Materials in Space Environments," *ASTM STP-431*, 1968, p. 128.

40. Ham, J. L., *Trans. ASLE*, 6, 20 (1963).

41. Gilbreath, W. P. and Williams, D. P., *Abstracts 13th AVS Vac. Symp.*, 1966 (Herbick and Held Printing Co., Pittsburgh, Pa., 1967), p. 181.

42. Buckley, D. H., "Symposium on Adhesion of Cold-Welding of Materials in Space Environments," *ASTM STP-431*, 1968.

43. Buckley, D. H., *NASA TN-D-4351*, 1967.

44. Buckley, D. H., *J. Appl. Phys.*, Aug. 1968.

45. Cunningham, B. E., and Eddy, R. E., *J. Spacecraft and Rockets*, 4, 280 (1967).

46. Snouse, T. W., *Applied Physics Letters*, 5, 122 (1964).

47. Poppa, H., *J. Applied Physics*, 38, 3883 (1967).

48. Lander, J. J., *Prog. Solid-State Chem.*, 2, 26 (1965).

49. Muller, E. W., "Advances in Electronics and Electron Physics," Vol. 13 (Academic Press, N. Y. 1960), p. 83.

50. Lichtman, D. and McQuistan, R. B., *Prog. Nuclear Energy*, 4, 95 (1965).

FIGURE LEGENDS

Fig. 1.- Schematic drawing of a single-beam spectrophotometer attached to a vacuum system.

Fig. 2.- The effect of pressure and strain rate on the change in the shear strain at the end of first linear hardening of the first linear stage for the tensile deformation of monocrystal magnesium.

Fig. 3.- The effect of pressure on the fatigue properties of polycrystal magnesium at 2 cps and 30 cps.

Fig. 4.- A pressure-ratio gage comparison system capable of gage calibration down to 1×10^{-10} torr.

Fig. 5.- The effect of various gas environments on the fatigue life of metals at 2 cps.

Fig. 6.- Schematic drawing of a typical load-displacement sensing apparatus for tensile testing at vacuum levels less than 10^{-10} torr.

Fig. 7.- Dynamic feedthroughs for the transmission of motion into vacuum. (a) "O"-ring compression seal for rotary and linear motion; (b) Bellows seal for linear motion; (c) Magnetic feedthrough for rotary motion; (d) Bellows sealed wobble stick for rotary motion; (e) Harmonic drive feedthrough for rotary motion (ref. 33).

Fig. 8.- Simple method of fluid transmission through a vacuum-chamber wall.

Fig. 9.- Electron-beam, ultrahigh vacuum, zone-refining, and monocrystal growing apparatus. (a) Overall view of system; (b) Zone-refining module.

Fig. 10.- The effect of vacuum on the tensile properties of magnesium.

Fig. 11.- The effect of vacuum on the tensile properties of magnesium monocrystals oriented for glide on the basal plane.

Fig. 12.- Schematic drawing of vacuum system designed to fit between the columns of a standard tensile test machine.

Fig. 13.- Internal bakeout system for vacuum apparatus using quartz-sealed tungsten resistance heaters.

Fig. 14.- The effect of vacuum on the fatigue properties of polycrystal magnesium-thorium alloy.

Fig. 15.- Perspective drawing of vacuum fatigue apparatus designed to fit on a standard fatigue testing machine.

Fig. 16.- Comparison of the creep properties of polycrystal magnesium in vacuum and in an air-argon environment at 760 torr.

Fig. 17.- Schematic drawing of a vacuum torsional-creep apparatus capable of pressures down to 10^{-9} torr and temperatures up to 600°C.

Fig. 18.- Ultrahigh vacuum, high-temeprature creep apparatus capable of pressures down to 5×10^{-12} torr and temperature up to 3000°C. (a) Overall view of system; (b) View of vacuum chamber interior showing furnace.

Fig. 19.- Effect of exposure to various gas environments on the coefficient of adhesion of aluminum.

Fig. 20.- Vacuum apparatus for friction testing.

Fig. 21.- Schematic drawing of a space environment simulator designed to simulate vacuum, photon, and charged-particle radiation, micro-meteoroid bombardment, and temperature. (a) Plan view of overall system; (b) Cross-sectional drawing of vacuum chamber capable of pressures down to 10^{-10} torr.

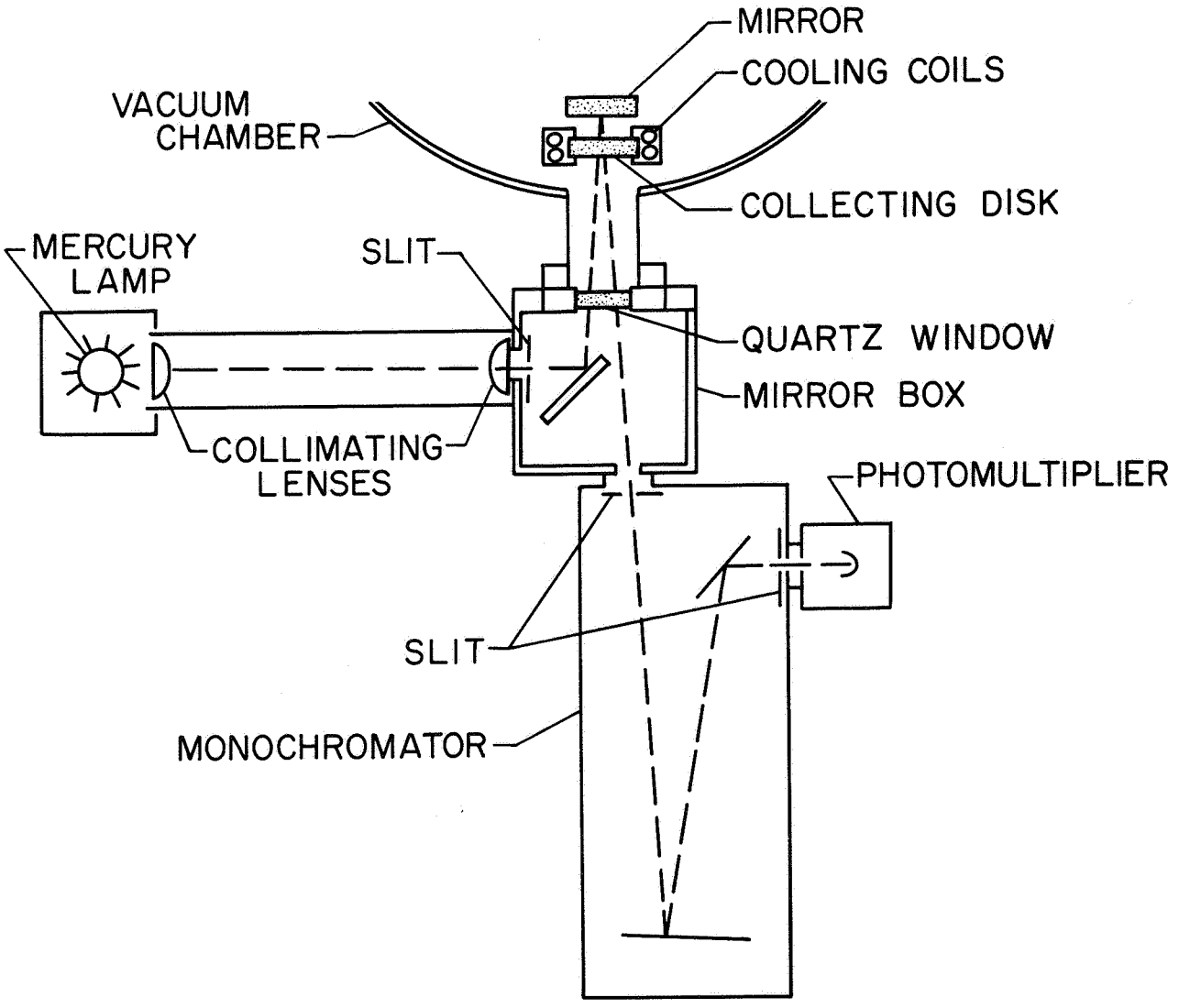

Figure 1.

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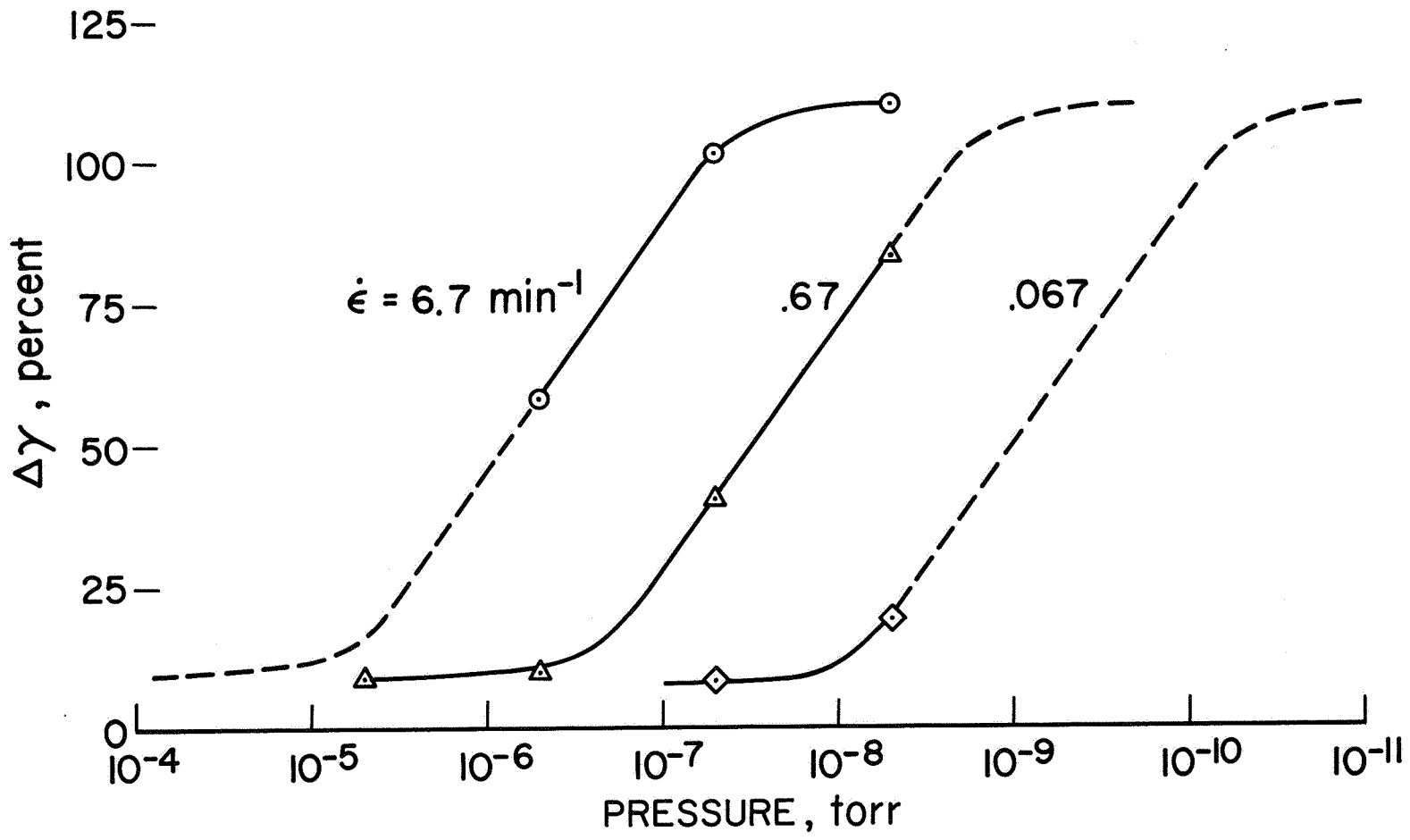

Figure 2.

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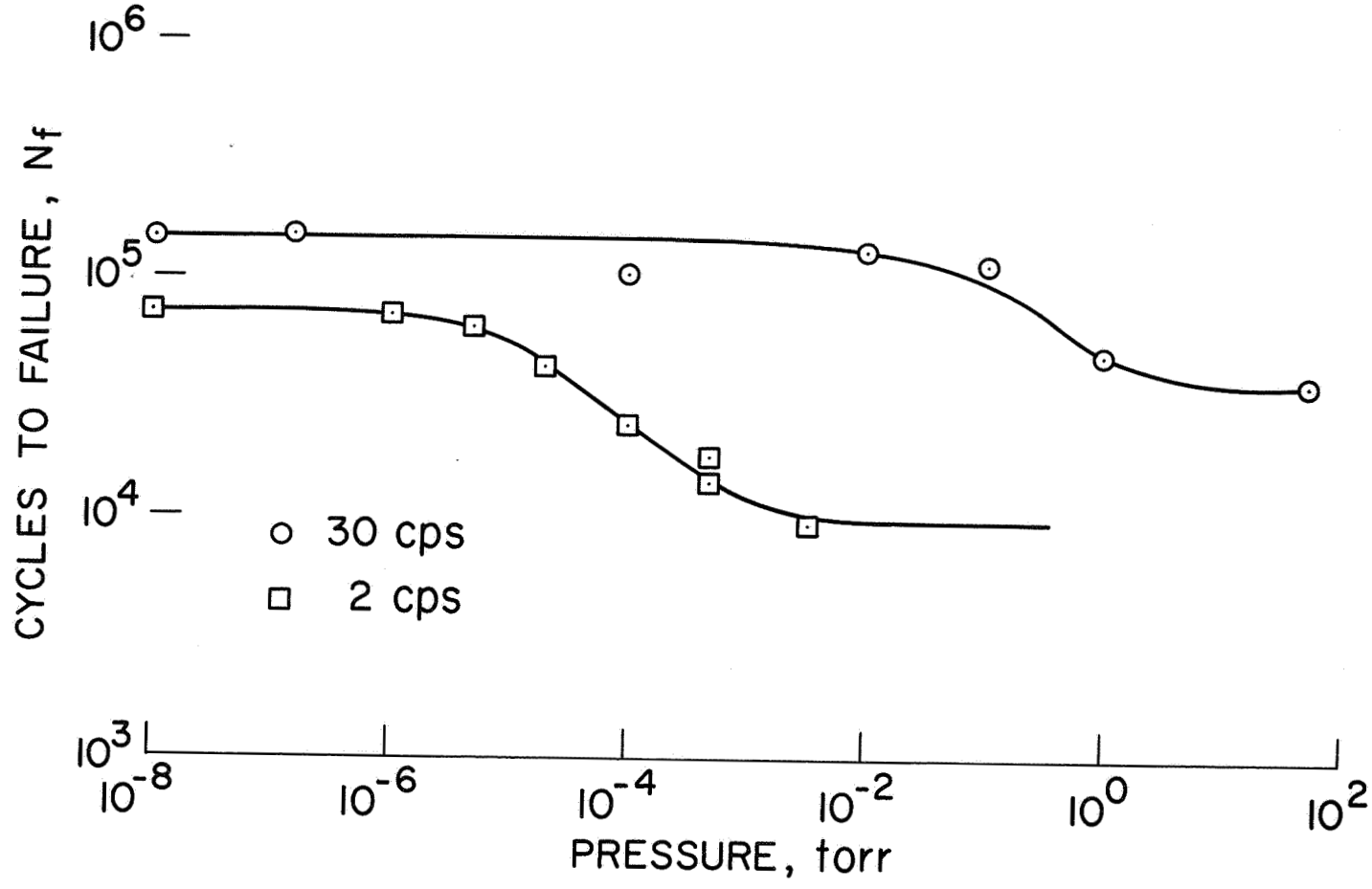

Figure 3.

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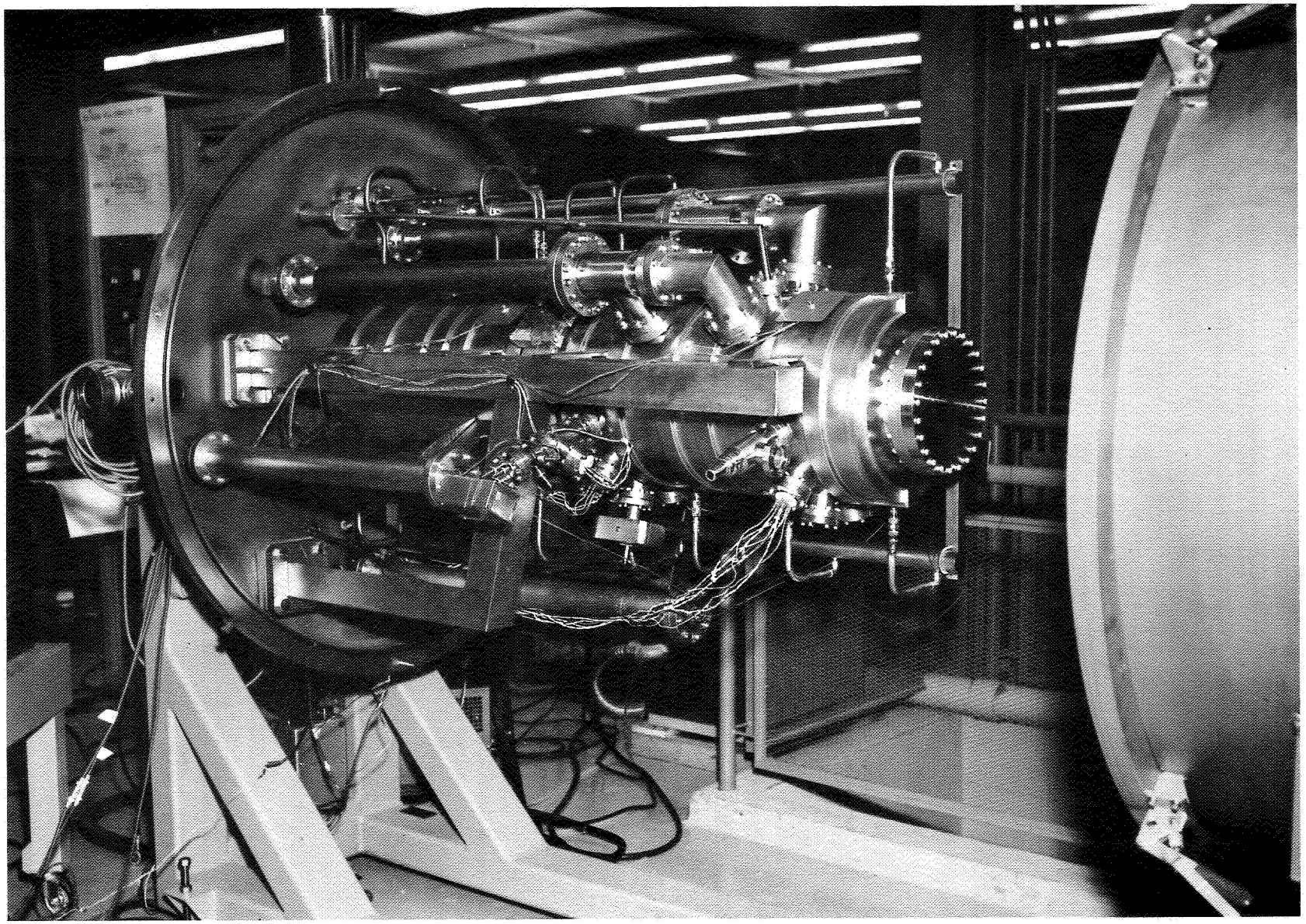

Figure 4.

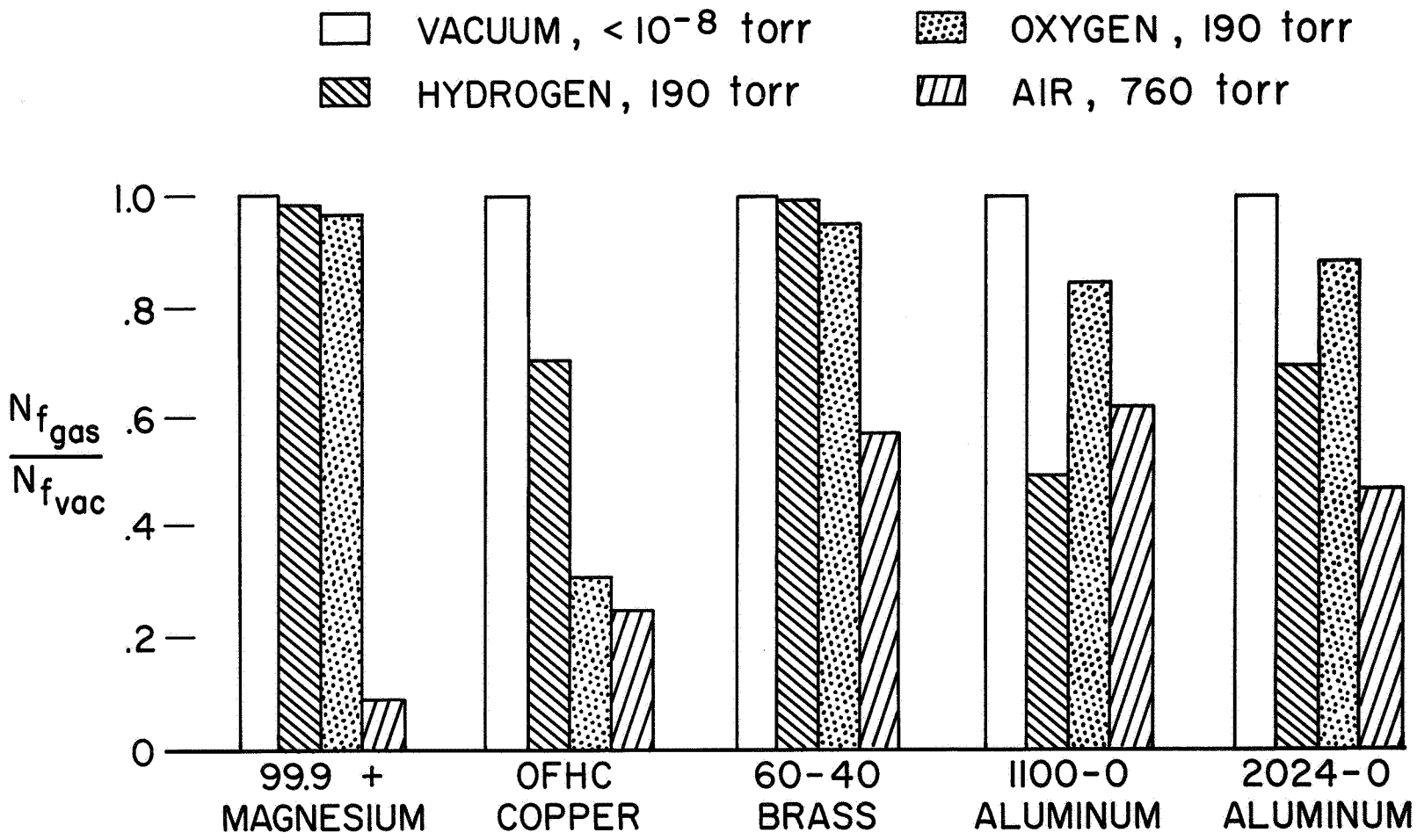

Figure 5.

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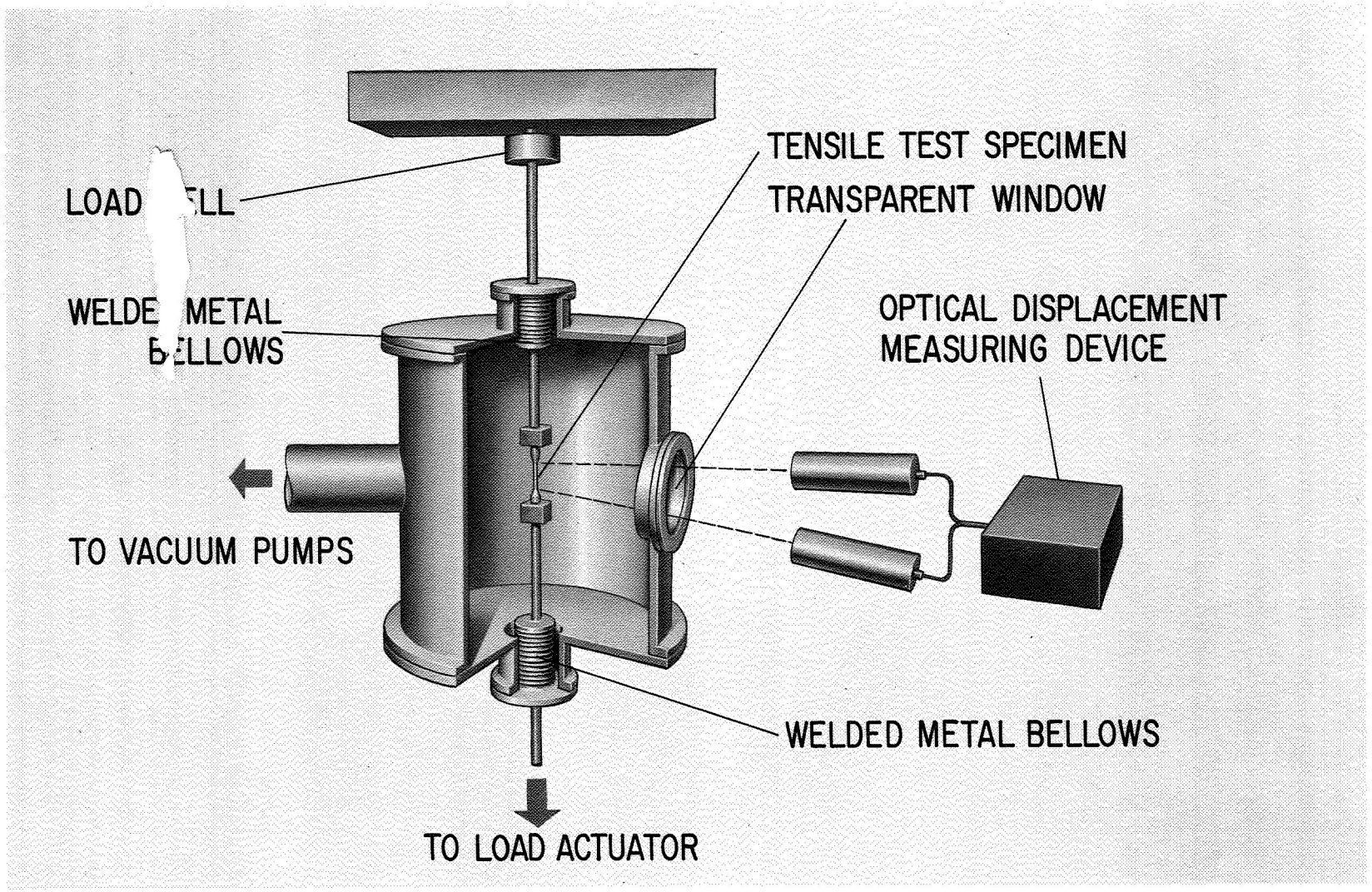

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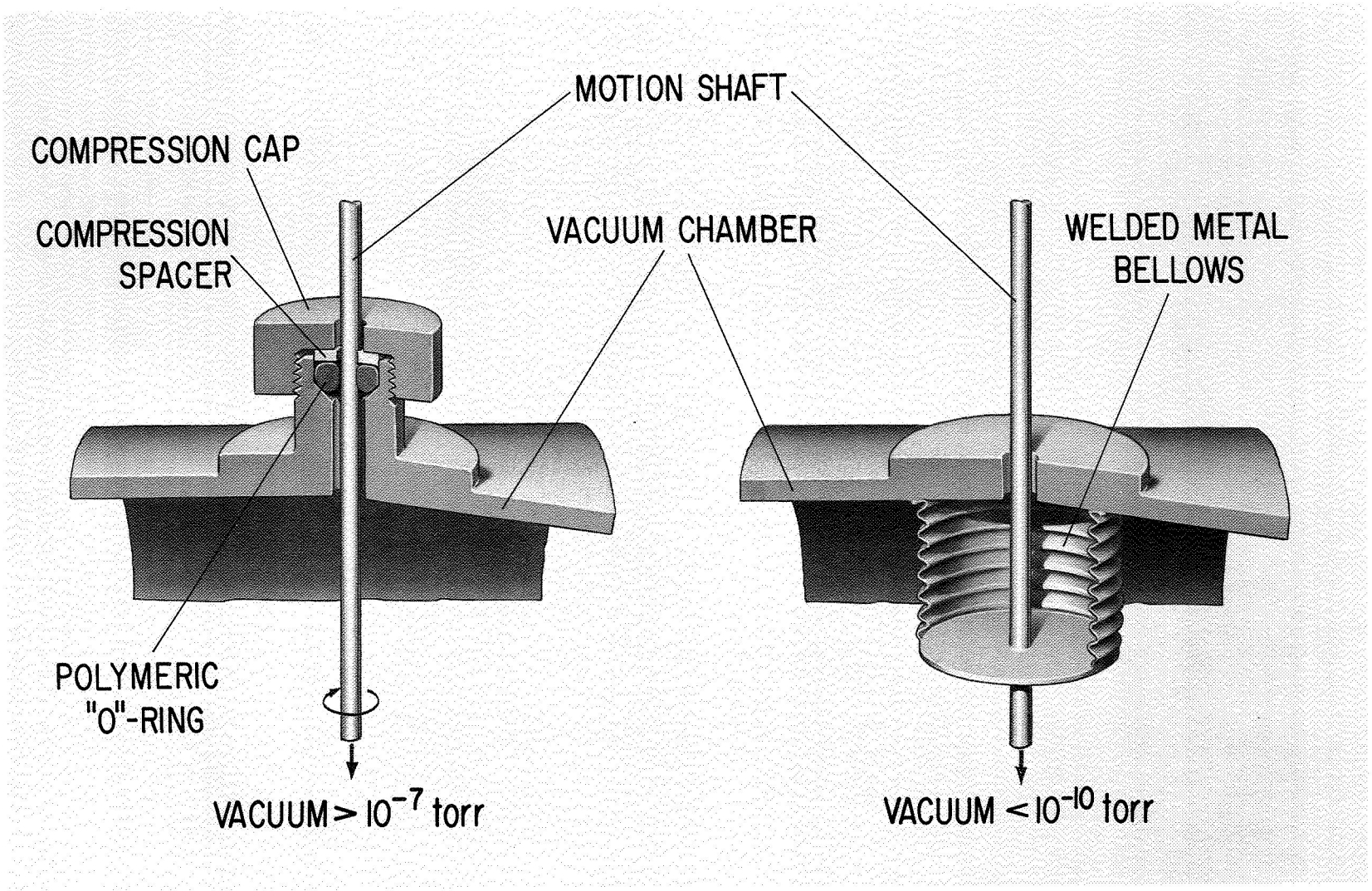

Figure 7(a) and (b)

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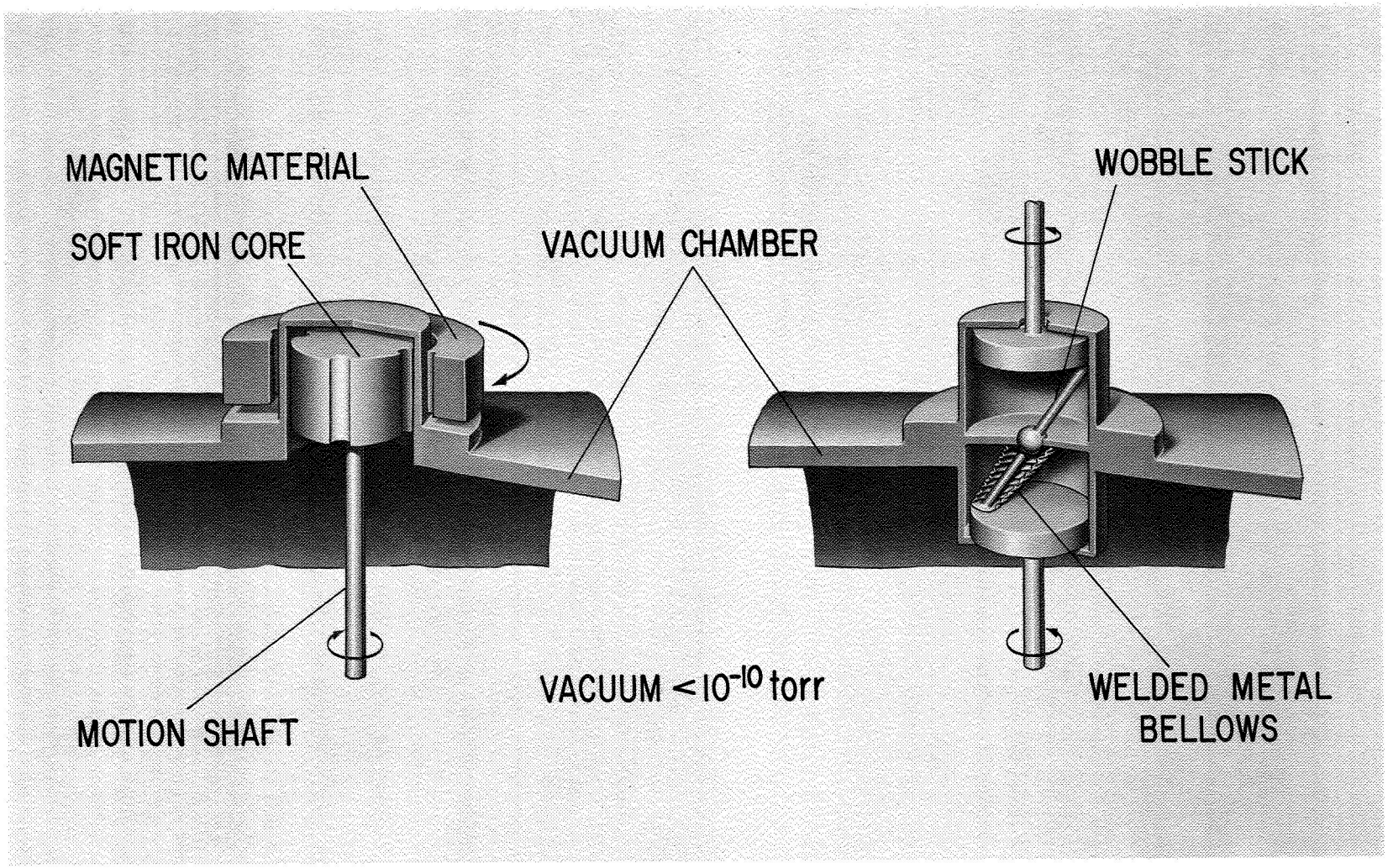

Figure 7(c) and (d)

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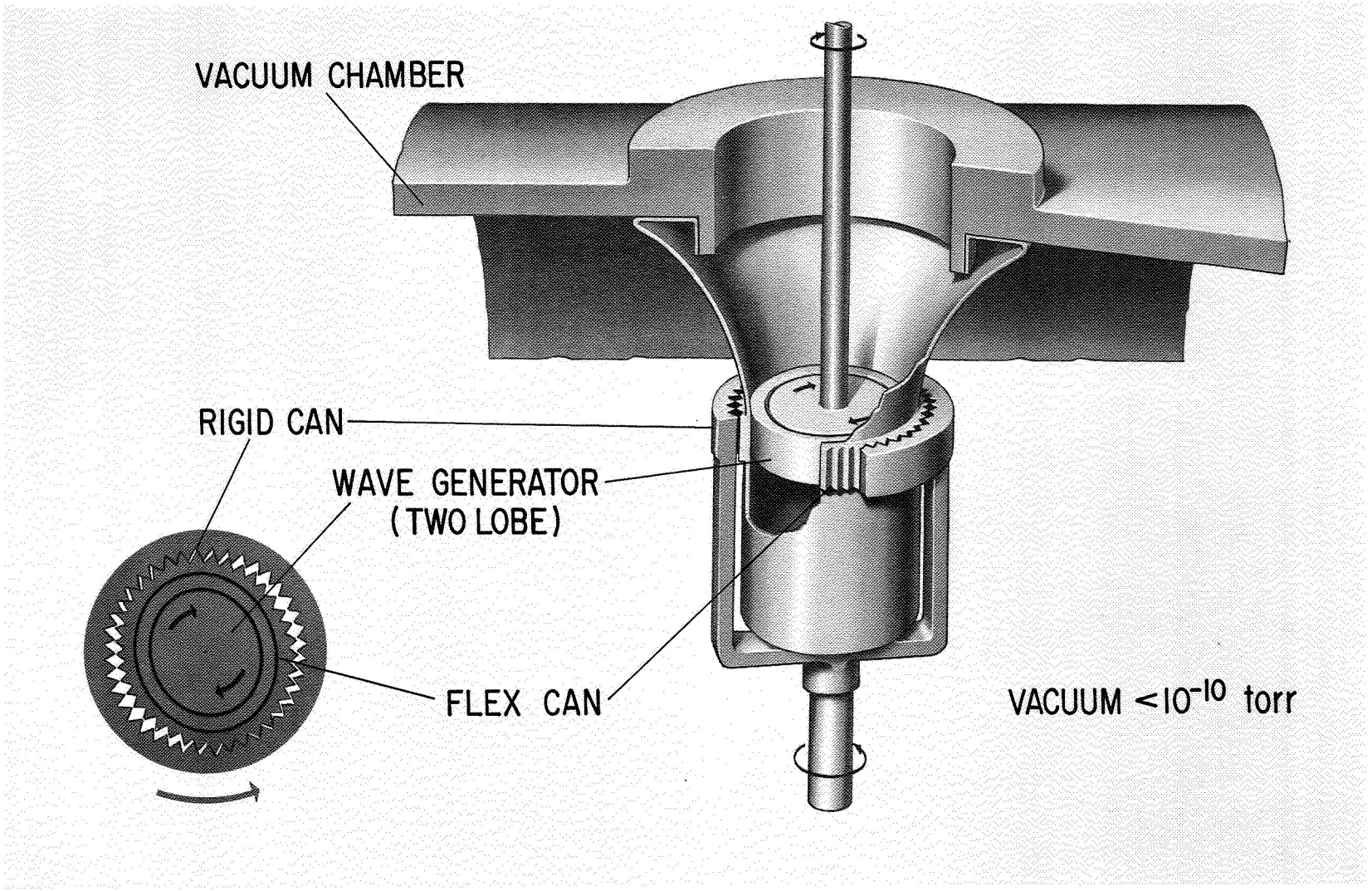

Figure 7(e)

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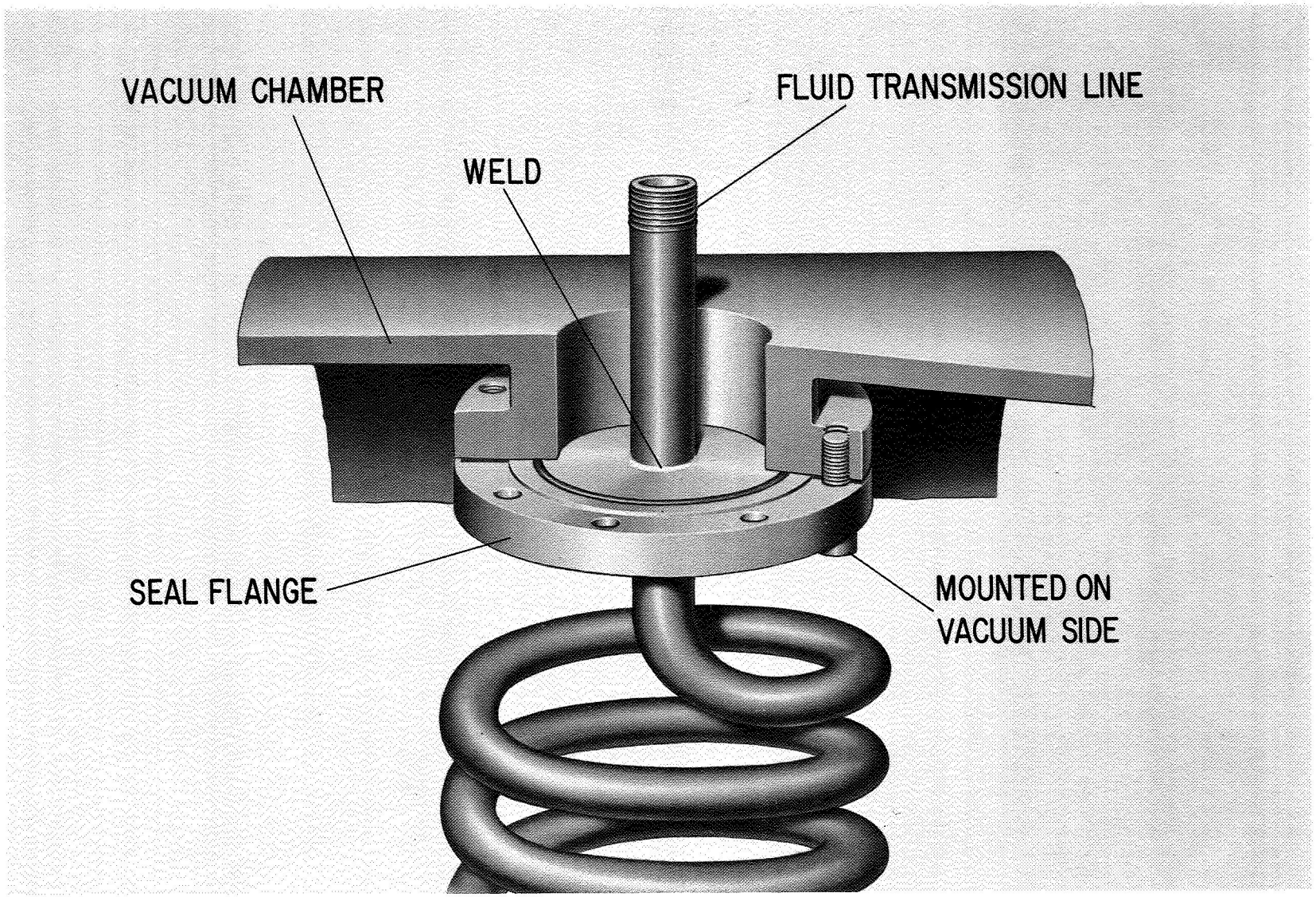

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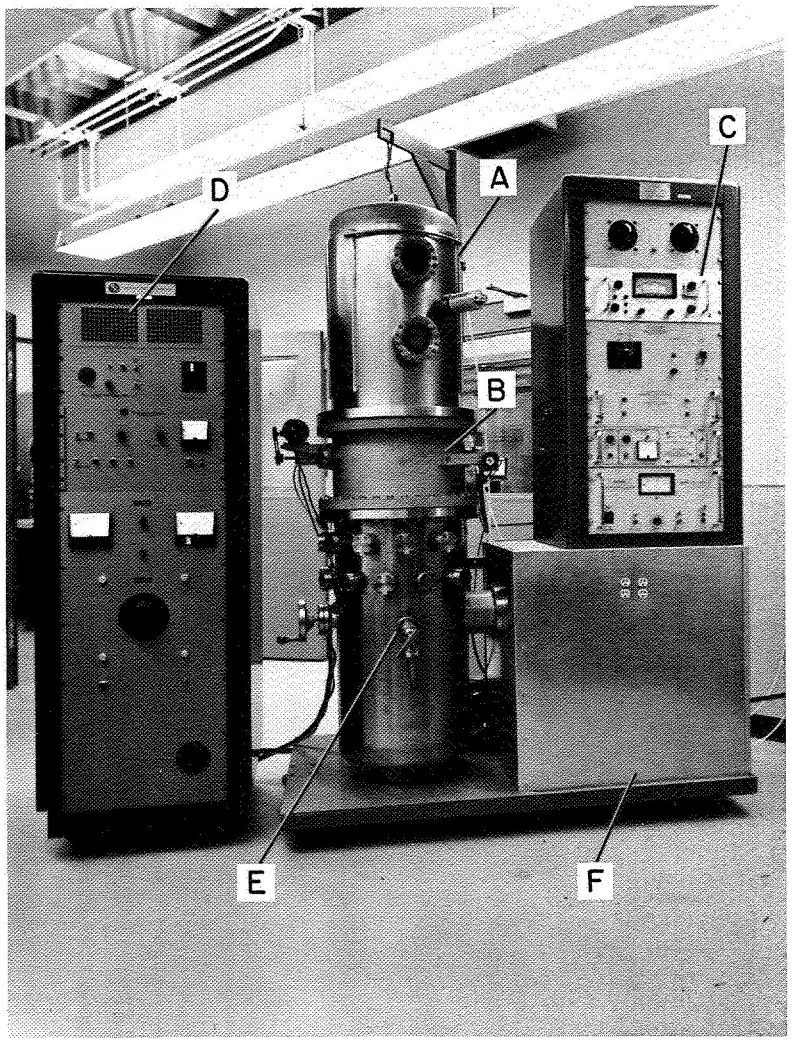

A. VACUUM BELL JAR
B. SUPPORT CHAMBER
C. CONTROLS FOR VACUUM SYSTEM
D. CONTROLS FOR ZONE-REFINING MODULE
E. VACUUM SUMP CONTAINING CRYOPANEL AND TSP
F. SPUTTER-ION PUMP

Figure 9(a).

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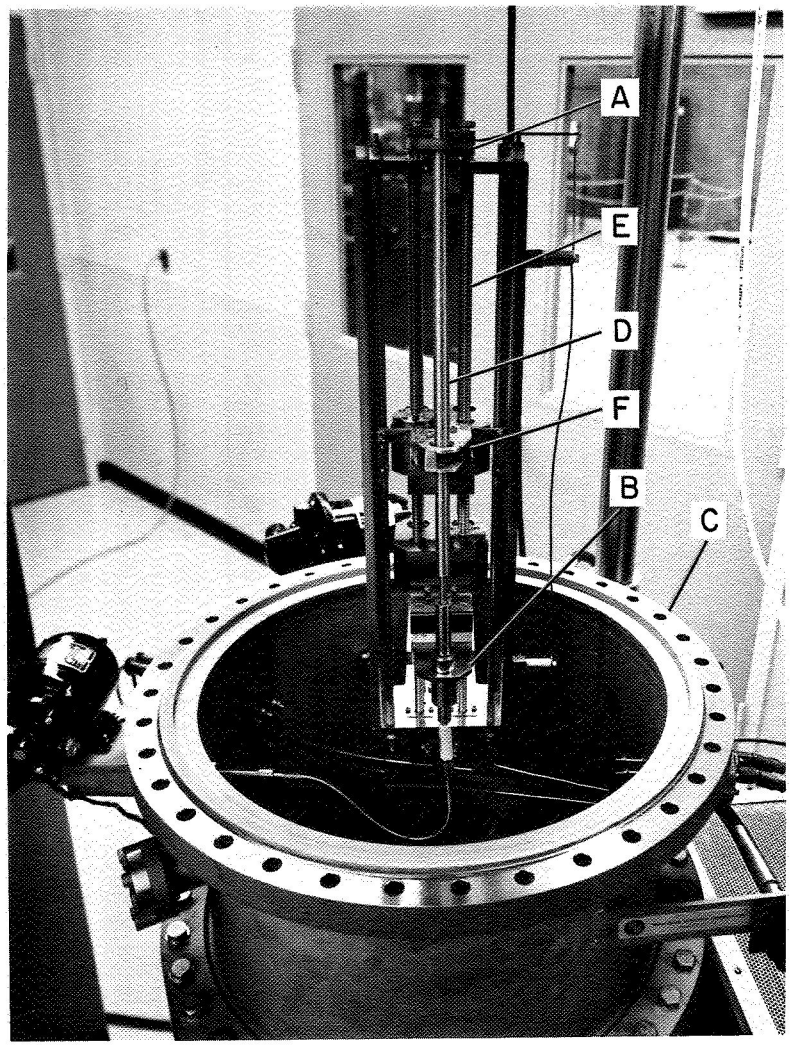

A. UPPER SPECIMEN SUPPORT
B. LOWER SPECIMEN SUPPORT
C. UPPER FLANGE OF VACUUM SUMP
D. SPECIMEN
E. BALL-SCREWS FOR SCANNER TRANSLATION
F. ELECTRONIC GUN AND SCANNER ASSEMBLY

Figure 9(b).

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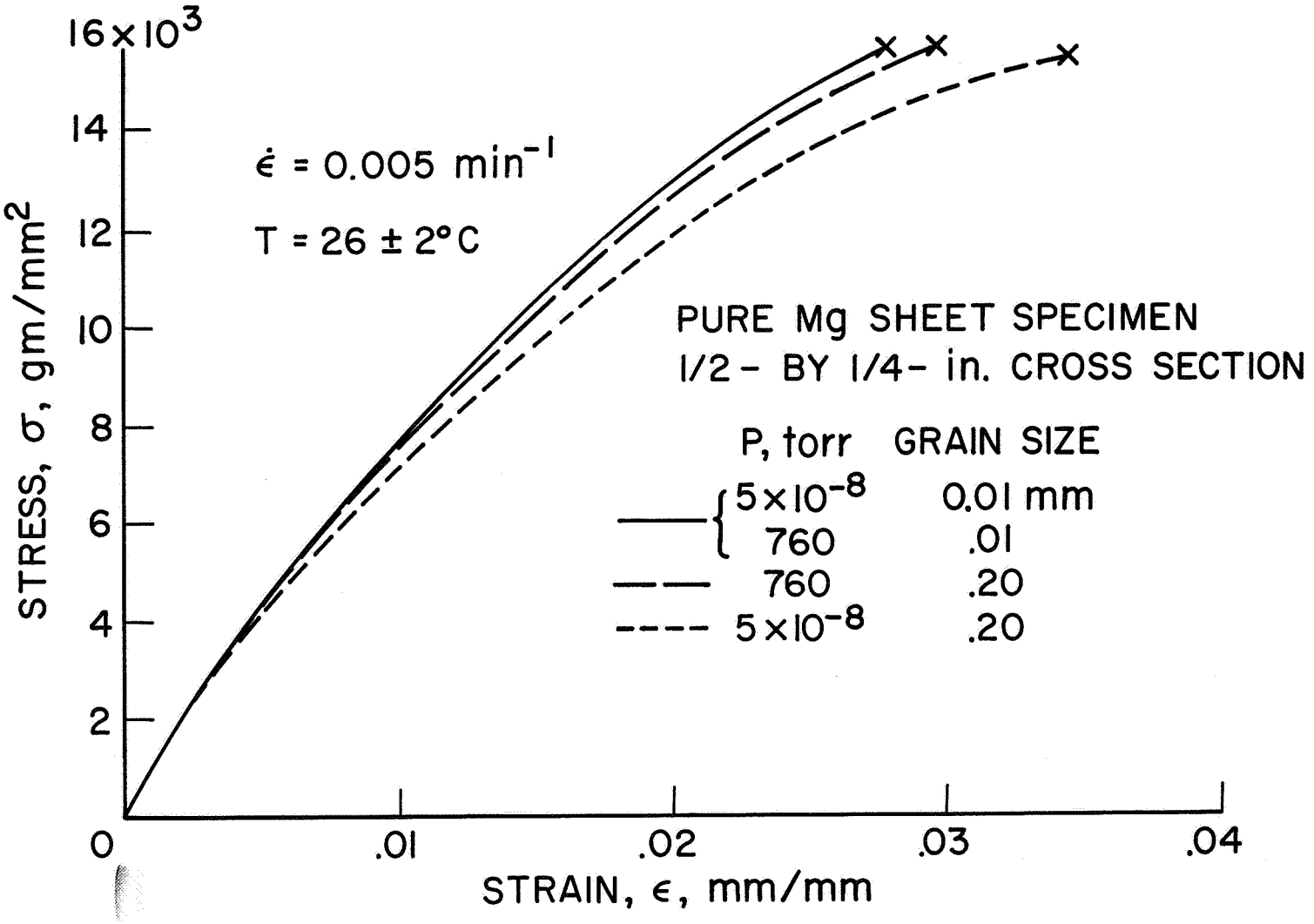

Figure 10.

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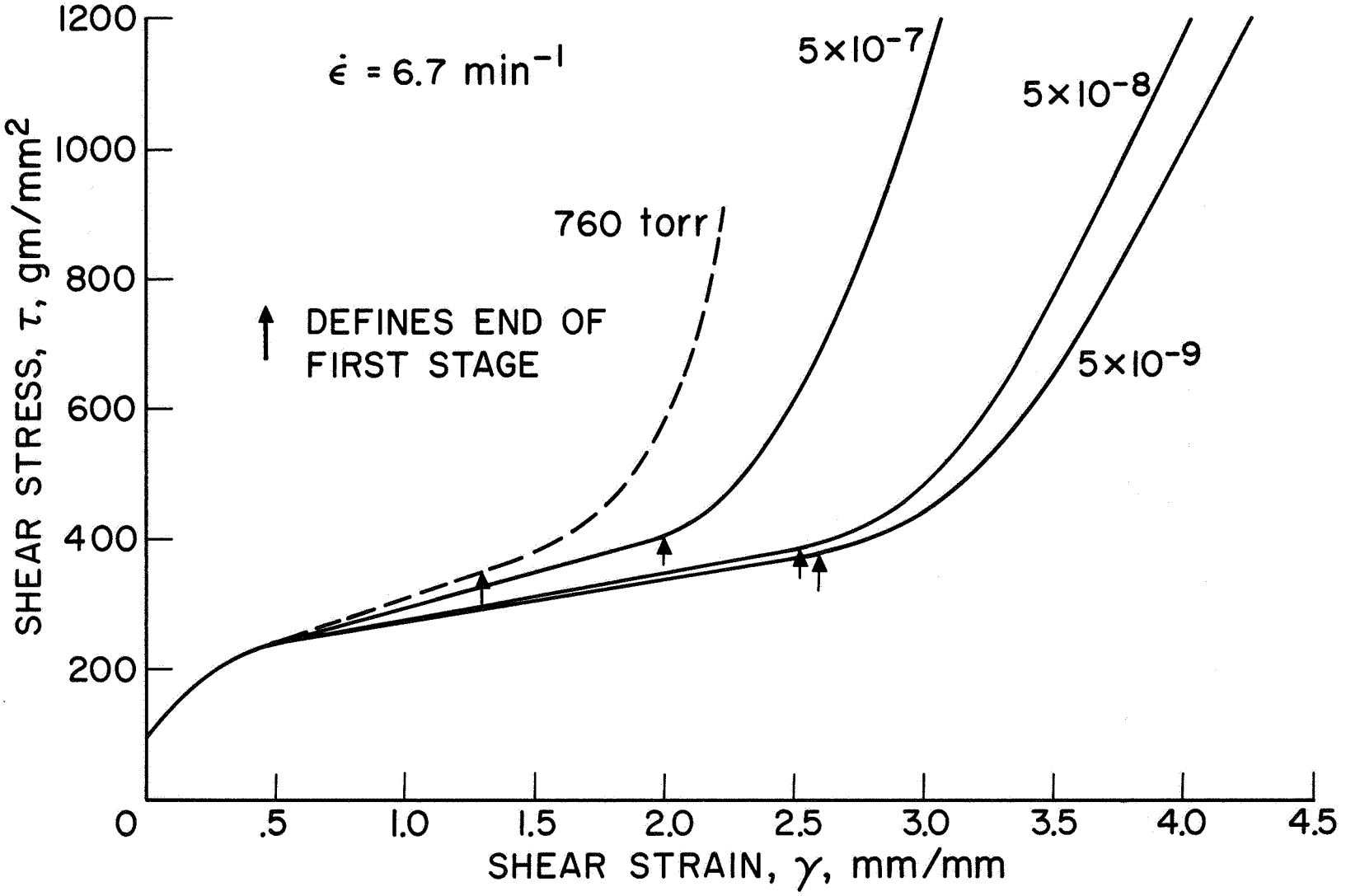

Figure 11.

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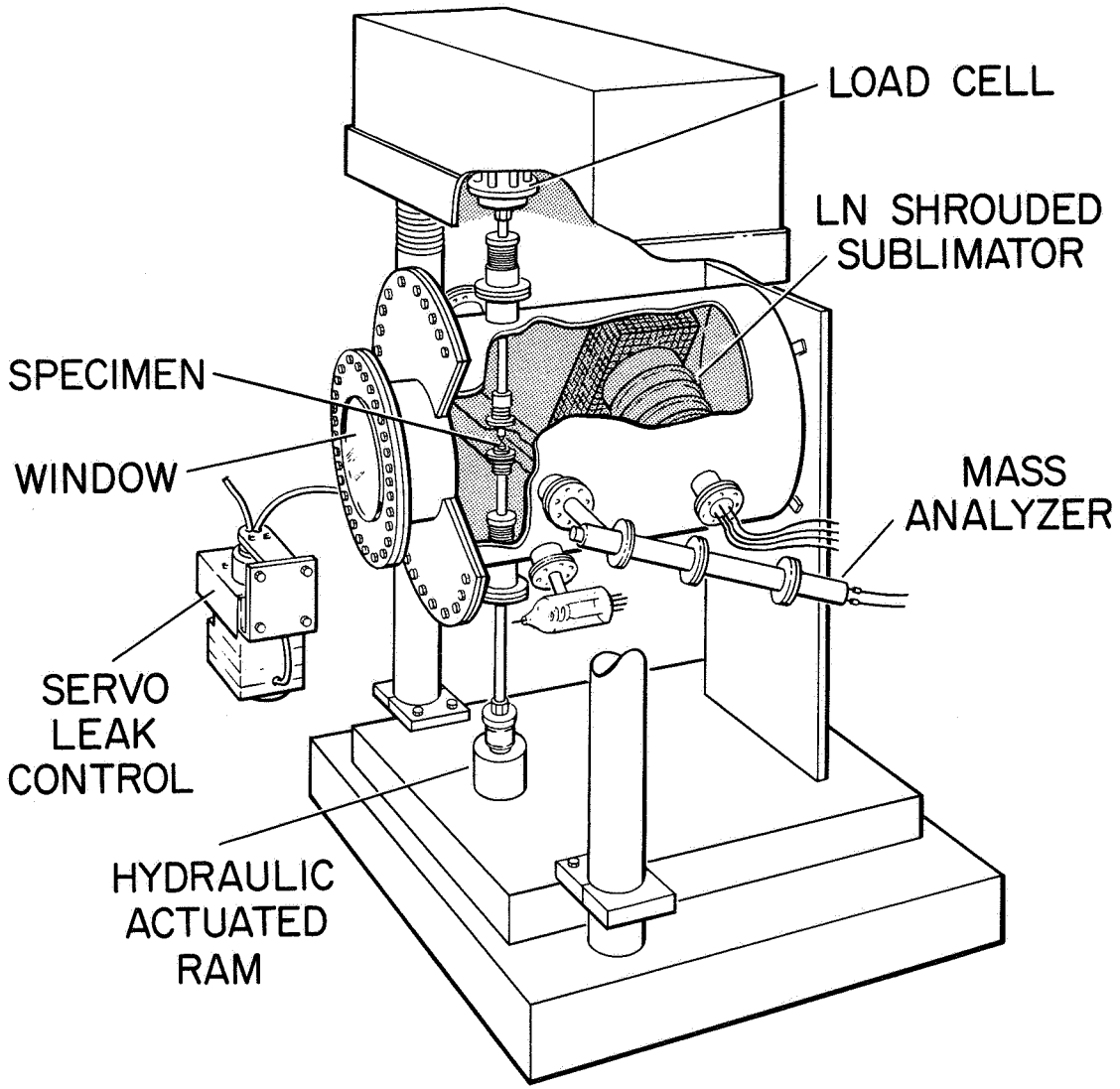

Figure 12.

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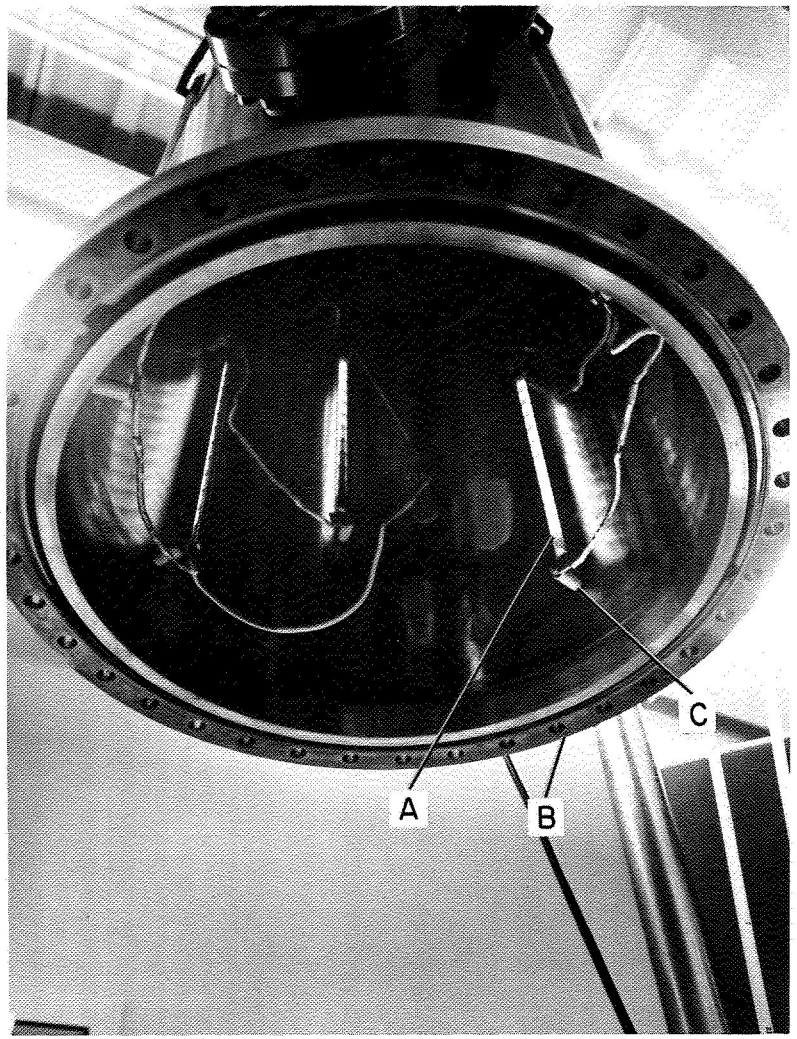

A. TUNGSTEN FILAMENT, QUARTZ LAMP HEATERS

B. ELECTRICAL INSULATORS

C. VACUUM CHAMBER AND FLANGE

Figure 13.

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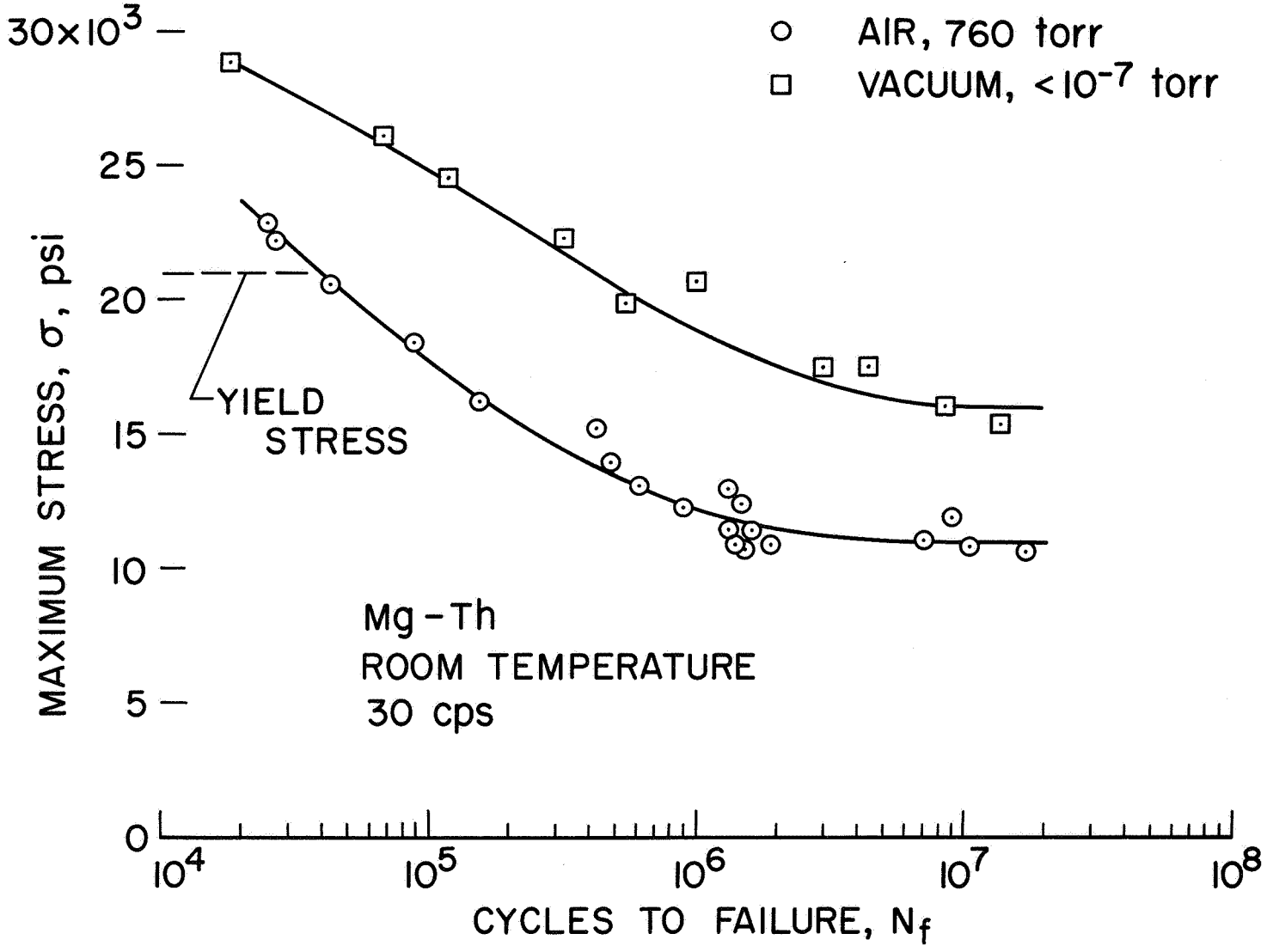

Figure 14.

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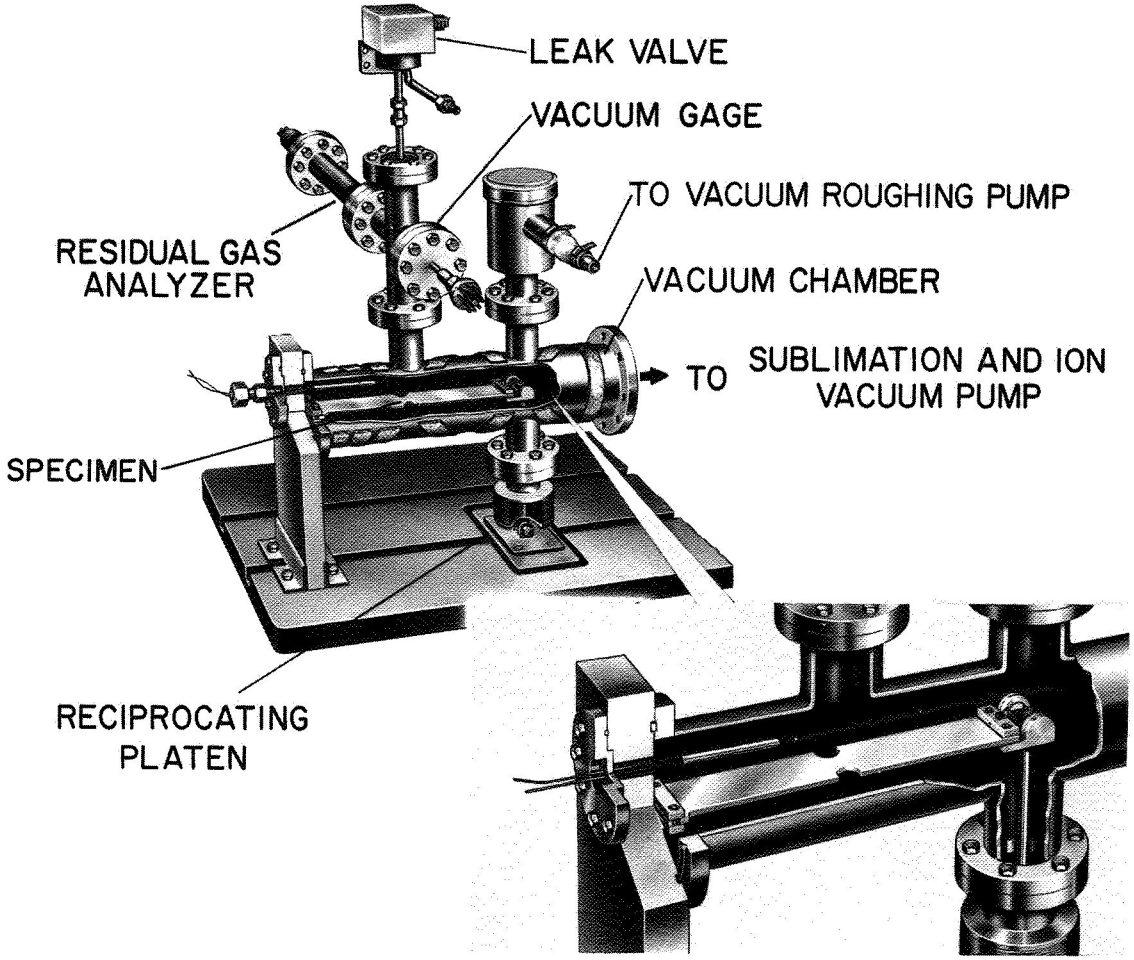
LEAK VALVE
VACUUM GAGE
TO VACUUM ROUGHING PUMP
RESIDUAL GAS ANALYZER
VACUUM CHAMBER
TO SUBLIMATION AND ION VACUUM PUMP
SPECIMEN
RECIPROCATING PLATEN

Figure 15.

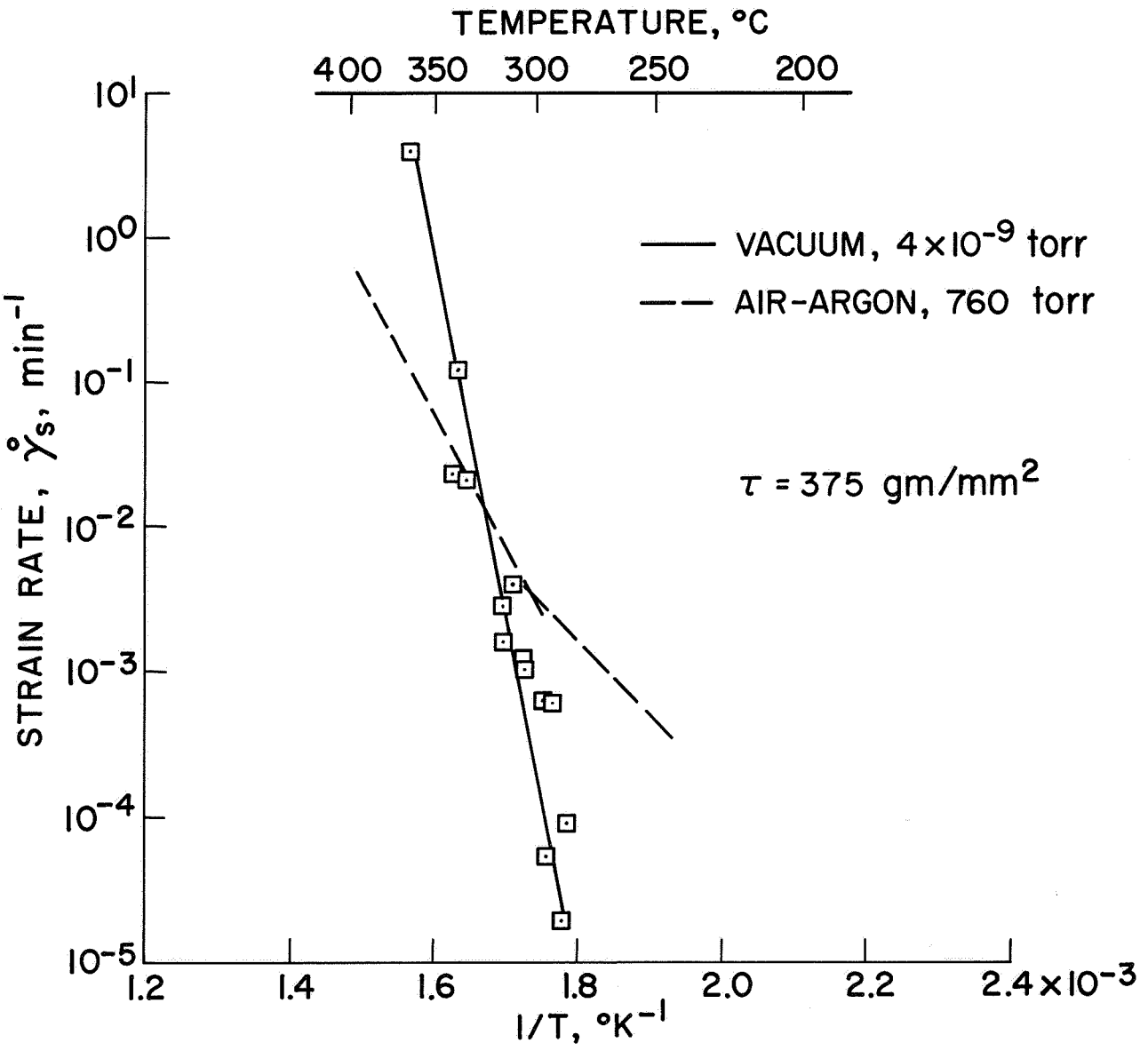

Figure 16.

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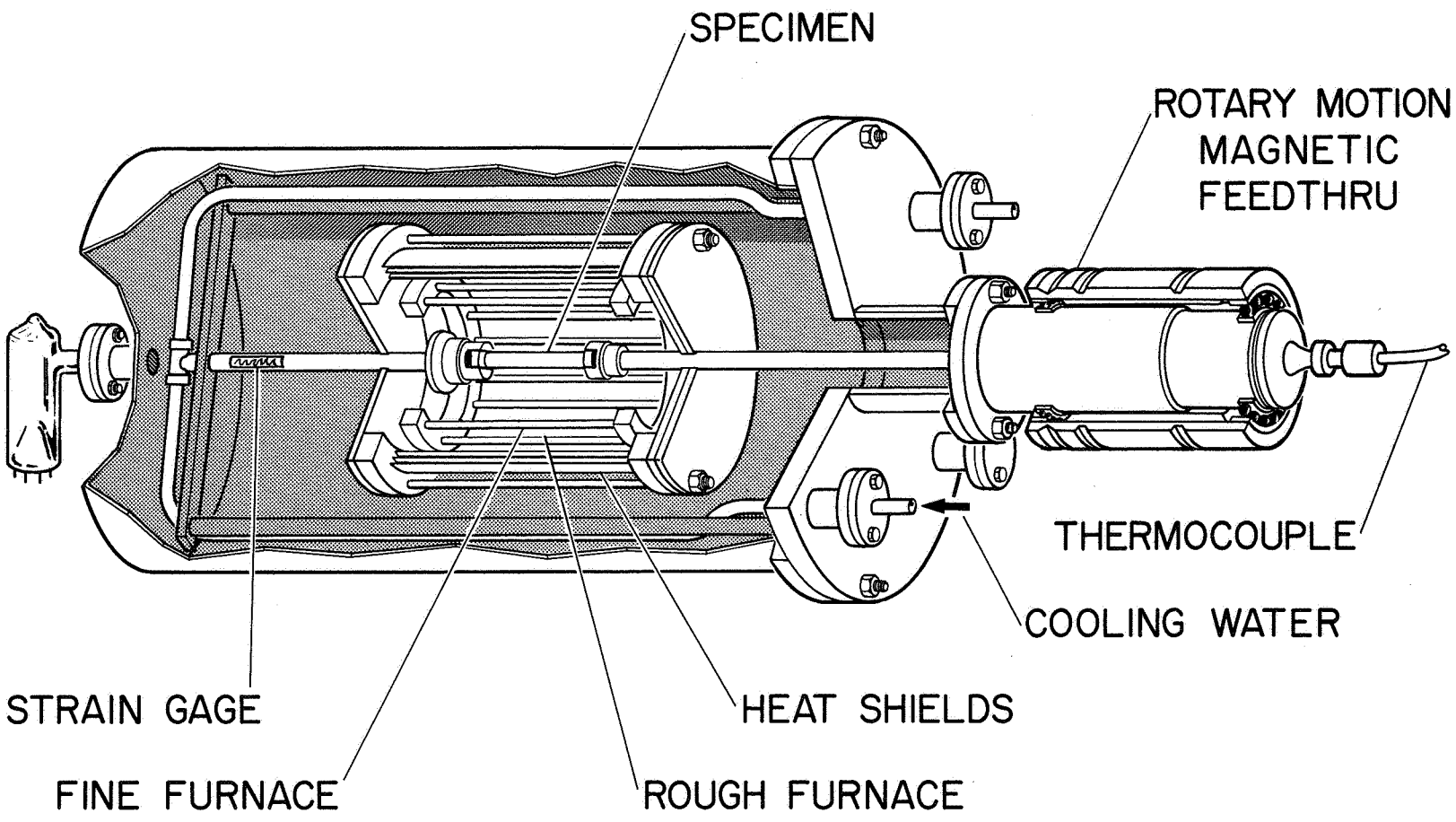

Figure 17.

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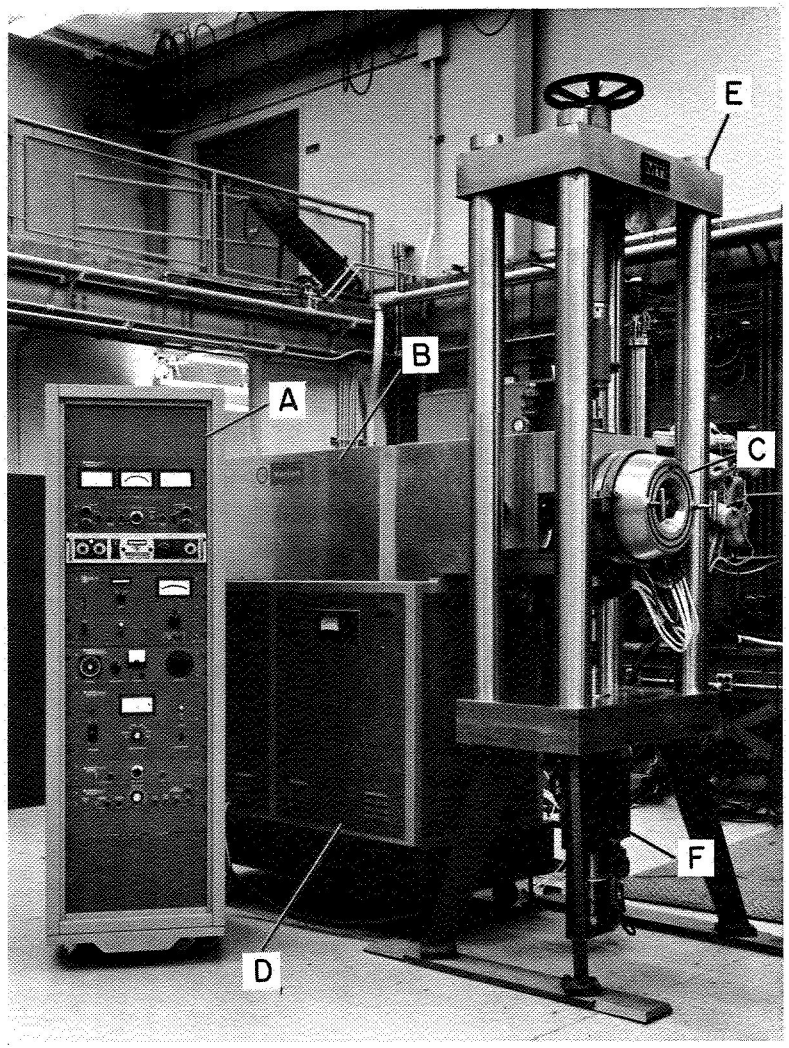

A. VACUUM AND FURNACE CONTROLS
B. SPUTTER-ION PUMP
C. FRONT SWING-OUT VACUUM CHAMBER DOOR
D. FURNACE POWER SUPPLY
E. LOAD FRAME
F. HYDRAULIC LOAD ACTUATOR

Figure 18(a).

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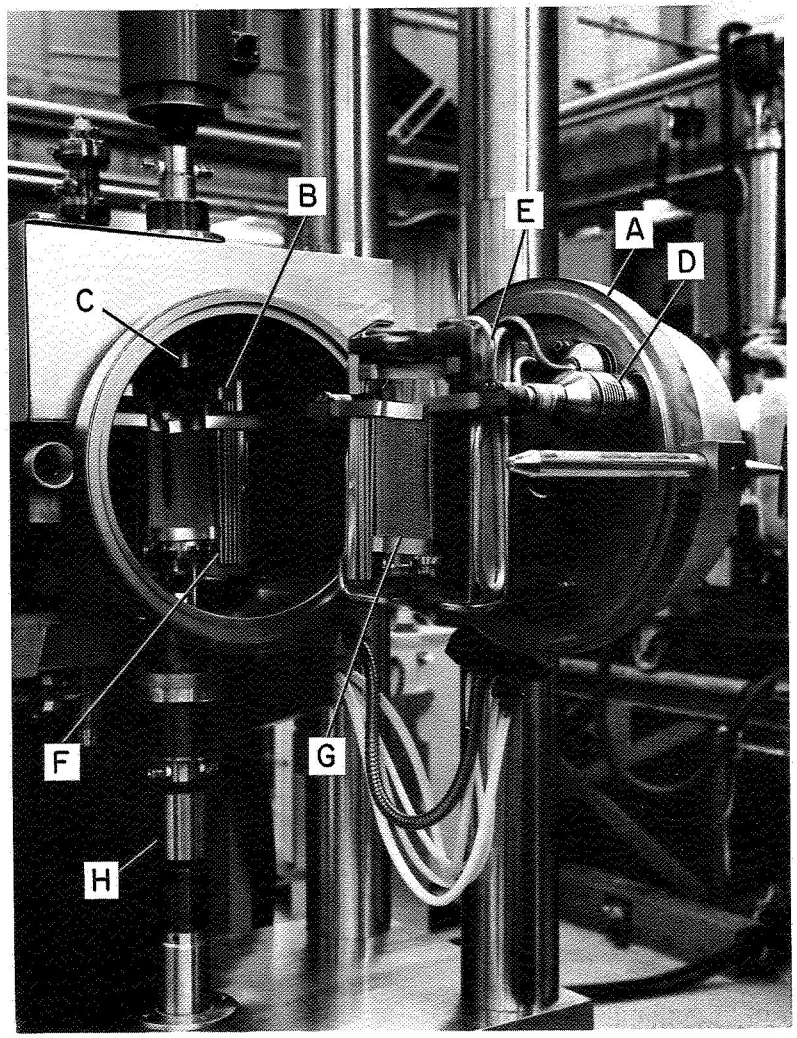

A. VACUUM CHAMBER DOOR WITH HALF OF FURNACE
B. INNER HALF OF FURNACE
C. UPPER LOAD ROD
D. FURNACE POWER LEAD
E. WATER COOLED COPPER SHROUD
F. TUNGSTEN HEAT REFLECTORS
G. TUNGSTEN WIRE MESH ELEMENT
H. LOWER LOAD ACTUATOR

Figure 18(b).

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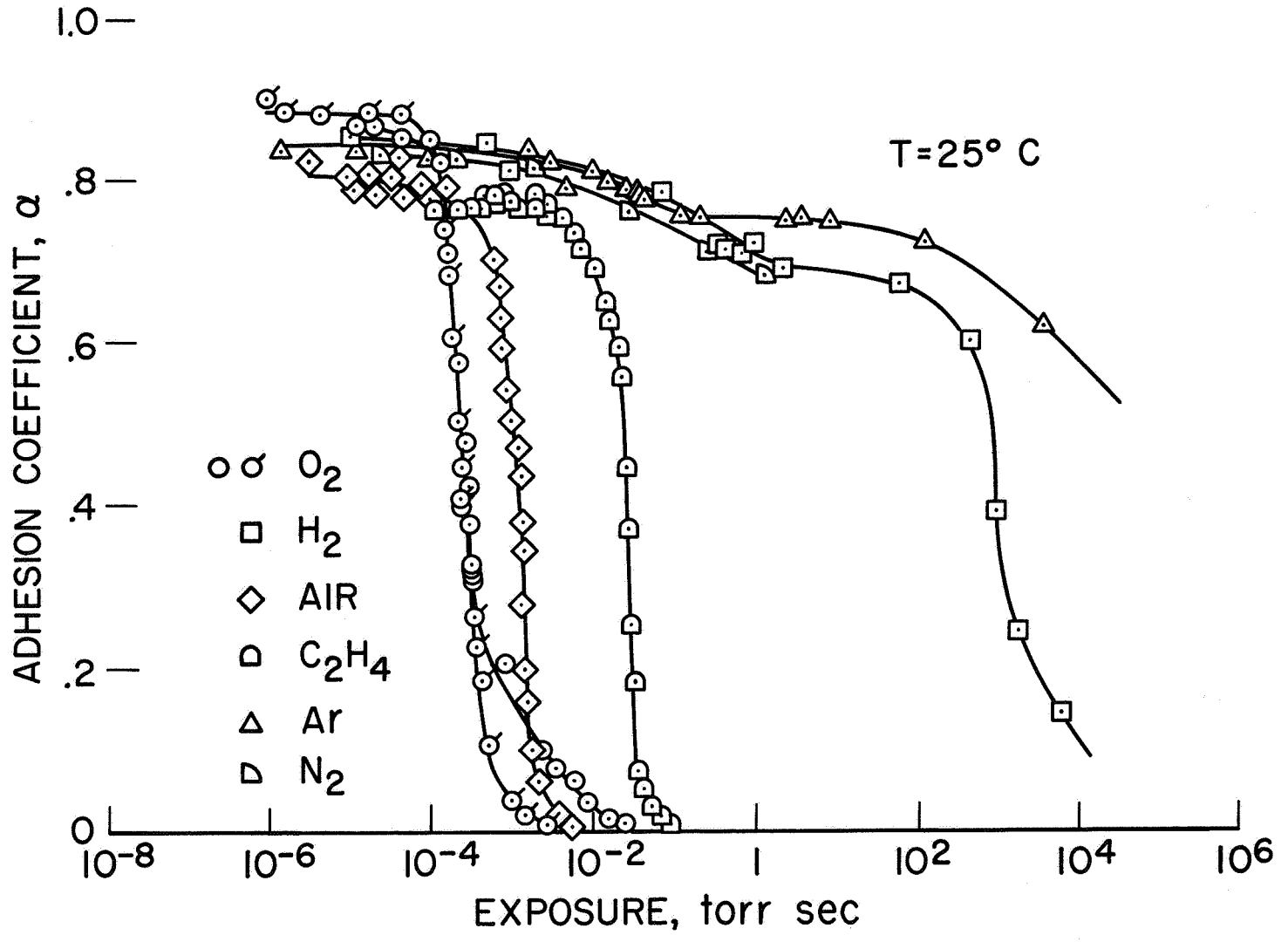

Figure 19.

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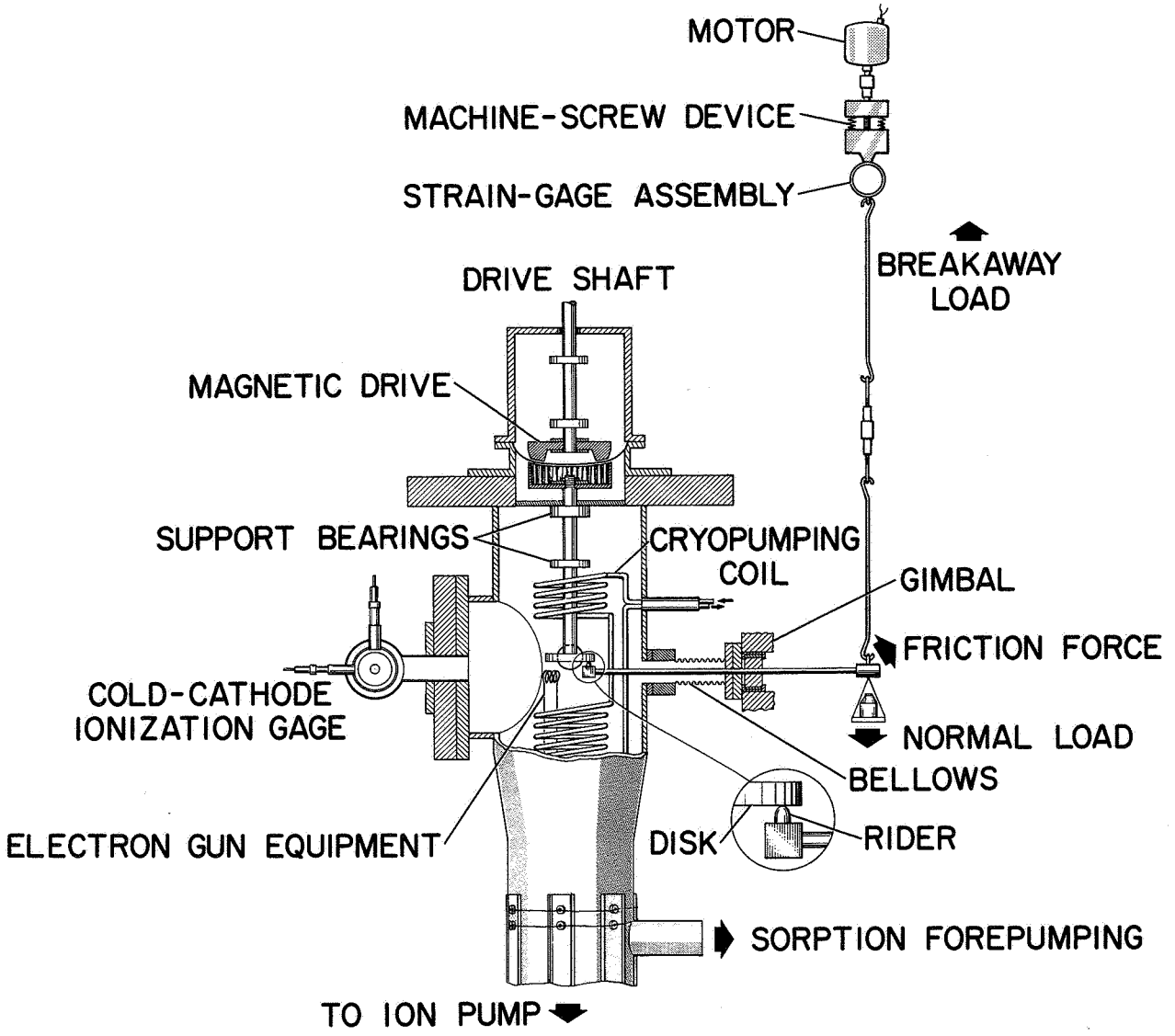

Figure 20.

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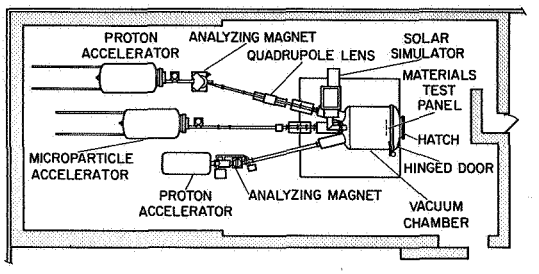

Figure 21(a).

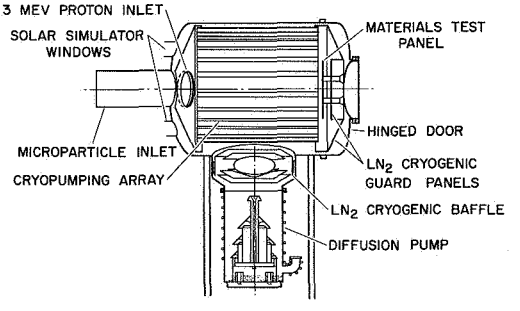

Figure 21(b).