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RESEARCH ON OPTICAL CONTACT BONDING

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241 Crescent Street
Waltham, Massachusetts 02154

Contract No. AF 19(628) - 3237

Project No. 4600,5635,0000

Task No. 460003,563503,000029

FINAL REPORT

Period Covered: 15 May 1963 thru 28 February 1966

Date of Report 30 May 1966

This research was partially supported by the
Air Force In-House Laboratory
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Prepared
for

AIR FORCE CAMBRIDGE RESEARCH LABORATORIES
OFFICE OF AEROSPACE RESEARCH
UNITED STATES AIR FORCE
BEDFORD, MASSACHUSETTS

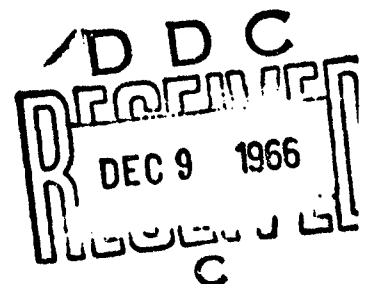
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ABSTRACT

The adhesion of optically polished surfaces - "optical contact" - was investigated both under room conditions and ultrahigh vacuum with the twofold objective of determining the adhesion mechanism and its characteristics and extending the technological applications. It is shown that optical contact adhesion occurs readily under ultrahigh vacuum, thus demonstrating that the widely held liquid layer theory is not complete. Adhesion mechanisms are discussed, and it is concluded that London dispersion forces are responsible for the adhesion under ultrahigh vacuum. For optical contacts made under room conditions, surface tension of a liquid layer probably also contributes. It is shown that optical contact techniques can be used to obtain extremely efficient transducer - sample bonds for gigacycle ultrasonic work. Reflections as low as 1% of the incident acoustic power were obtained at 3 GC and 9 GC. Techniques for making such bonds are discussed. It is pointed out that the incorporation of evaporated metal films should enhance the properties of optical contact bonds and eliminate some practical difficulties. The design and development of a high vacuum evaporator are described.

ACKNOWLEDGEMENTS

Sections I to IV of this report cover some basic studies of optical contact and related phenomena. H.I. Smith and M.S. Gussenhoven carried out most of this work. Section V covers the development of the high vacuum evaporator; work which was performed by R.B. Holt. In all of this work, the technical assistance of John McCloskey and Wallace Coffin was an invaluable asset.

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1. INTRODUCTION

If two clean optically polished surfaces are brought into intimate contact, they will often adhere to one another. This phenomenon is known as "optical contact." The objectives of the present effort can be divided into two main categories: (1) basic studies of the phenomenon of optical contact, including experiments at ultrahigh vacuum, (2) the practical application of optical contacting techniques, especially in the bonding of transducer-sample assemblies for gigacycle ultrasonic studies.

Section 11 discusses, in brief and general terms, commonly observed surface adhesion phenomena, in order to set the stage for a specific discussion of optical contact, and the findings of the present study.

11. ADHESION BETWEEN SOLID SURFACES

1. Metals

It is well known that metal surfaces can seize or cold-weld if surface contamination and oxide layers are either broken through mechanically or removed under vacuum¹⁻⁴.

A metallic bond is formed between the pure metals thus brought into contact which is presumably the same as the bond present in the bulk material. In the case of metals, the area over which bonding occurs can become quite large due to plastic flow of microscopic surface asperities. The application of small tangential stresses further enhances junction growth^{1,2,4}. The phenomenon of friction is closely related to adhesion. According to the theory proposed by Bowden and Tabor, metallic friction is due to the shearing of microwelds plus a contribution from mechanical plowing action^{1,2}.

2. Brittle Solids*

In the case of brittle solids, adhesion phenomena are not so readily observed. The area of intimate contact between such materials is almost always a minute fraction of the geometric area. If this area of intimate contact is increased by the application of a normal load, any microwelds which might be formed would generally be broken upon load removal, due to the release of elastic stresses in the bulk of the sample. Under extreme compressive forces, sufficient to induce plastic flow, some brittle substances such as rock salt can be made to adhere (if proper crystallographic alignment is provided) with strengths comparable to bulk tensile strength⁵. An exceptional case is that of mica which when cleaved is molecularly smooth over rather large areas. Adhesion between rejoined cleaved surfaces is readily observed and has received considerable attention^{2,6,7}. Both ionic and van der Waals forces contribute to the adhesion, but the key factor is obviously the extreme flatness and consequent large area of intimate contact upon rejoining.

*The term "brittle solids" is intended to include non-metallic inorganic single crystals and glasses. Specifically excluded from the discussion are polycrystalline materials, metals, plastics, elastomers, and other organic materials.

3. Effect of Bulk Bonding Mechanisms on Adhesion

The above remarks indicate that lack of sufficient surface area in intimate contact is the main reason for the paucity of adhesion phenomena observed with brittle solids. Another reason has to do with the type of bonding which characterizes the solid. A pure covalent bond is highly directional and requires precise near neighbor positioning. Precise near neighbor positioning is also required in ionic solids because of the alternating array of positive and negative ions. Thus, if two perfectly planar and identical crystal faces of an ionic or covalent solid are brought into contact, the possibility of ionic or covalent bonding across the interface would appear to be extremely remote since perfect crystallographic coincidence would be almost impossible to obtain. The metallic bond is considerably less sensitive to bond angles and deviation of the near neighbors from equilibrium positions, and this is an important factor in the adhesion of metals³.

III. OPTICAL CONTACT*

1. Characteristics

A careful observation of the optical contacting process reveals several interesting features. As the clean polished flats are brought close together first order interference fringes are observed, indicating the presence of an air wedge. These first order colors go over to a uniform gray appearance at a separation of about $1000 \text{ \AA}$.

*For a description of optical contacting techniques see section IV, 1. and 2.

If the separation is decreased further, a sudden seizure of the surfaces occurs and the appearance is a uniform black, indicating a reduction in the optical reflectivity of the interface of several orders of magnitude. According to Lord Rayleigh's measurements, the optical reflectivity of an optical-contact bond is between 2×10^{-4} and 2×10^{-5} , if the incident light intensity is taken as unity⁸. On this basis, he calculated that the average separation of surfaces in optical contact is between 10 and 30 Å. This figure is clearly too small, since the mean height of surface irregularities is at least 100 Å. The actual separation probably ranges from 0 to 200 Å.

The tensile strength of an optical contact bond is surprisingly large. Lord Rayleigh measured strengths up to 46 Kg/cm^2 (Ref. 8). Several tests were made during the course of the present studies using the apparatus shown in Figure 1. Special care must be taken to eliminate vibration and to insure that the pull is normal to the plane of the interface. Bond strength was approximately 14 Kg/cm^2 for Z-cut quartz samples. It was observed that breakage of an optical contact bond always occurs as a slow progressive stripping initiated at some imperfection at the border of the contact area. The area of non-contact would slowly grow larger over the course of several minutes (no more weight being added) until only about 2/3 of the original area was still in contact. At this point, sudden breakage would occur. Thus, the actual tensile strength is probably many times greater than what is measured.

One possible means of measuring more accurately the adhesive strength of optical contact is to use a well-polished convex lens of large radius and an optical flat. The central black spot of the Newton rings formed when the lens is placed on top

of the flat is an area of optical contact. If the lens is set into a rocking motion, one could probably relate the damping of this oscillation to the adhesive strength. This type of experiment would eliminate the problem of a perfectly normal pull, and would not be as sensitive to the presence of dust as is a straight tensile test. This experiment could be done under a variety of environmental conditions, including high vacuum.

When an optical contact bond is made under normal room conditions, there are generally a few dust particles trapped between the surfaces. These dust particles are easily detected if the interface is viewed at a glancing angle. They appear as white specs against the black background. What one is actually seeing is the area around the dust particle which is not in contact and hence has a much higher optical reflectivity. Occasionally, a trapped dust particle is large enough for some of the first order interference colors to appear, but this is usually not the case, at least with the flats used in these studies (up to 1 inch diameter). Such large dust particles usually prevented contact altogether. Appendix 1 describes some work on the local stress induced in pyrex flats by trapped dust particles.

2. Liquid Layer Theory

Most authors attribute the adhesive force of optical contact entirely to surface tension of an extremely thin film of liquid (usually condensed water) at the interface. Water from the atmosphere collects around hygroscopic impurities on

surfaces. It has been reported that the equivalent of about 20 molecular layers is present at a relative humidity of 80% (Ref. 9). The attraction of two flat parallel plates with a thin film of liquid between them is given by the equation:

$$F = \frac{2TA}{d} \quad (1)$$

where T is the surface tension of the liquid, d is the separation of the plates, and A is the area in contact¹⁰. For a water film 1000 Å thick, the adhesive force per unit area is 15 Kg/cm². McFarlane and Tabor studied the adhesion of glass beads to flat glass plates for various liquid layers, and found a direct variation of adhesion with surface tension¹¹. They reported no adhesion in air at a relative humidity below about 50%.

One of the consequences of the surface tension theory is that the force of adhesion between plates should disappear if the junction is surrounded by the same liquid as produces the adhesion. This effect has been observed in this laboratory in several experiments using water. However, this is not considered conclusive proof that surface tension of a liquid film is the sole mechanism of adhesion.

One of the first and most important objectives of the present work was to determine if covalent, ionic or van der Waals forces contribute to optical contact adhesion. To do this, optical contacting experiments were carried out at ultrahigh vacuum where water films could be readily eliminated or at least reduced to single monolayer coverage, in which case the liquid layer theory would clearly not apply. These experiments are described in the next section.

3. Optical Contact at Ultrahigh Vacuum

a. Vacuum System

The vacuum system is shown schematically in Figure 2. The 8 liter per second Varian ion pump (Model No. 911-5000) is connected on the low vacuum side through a stainless steel, teflon seated valve to a Vac-Sorb pump (Varian Model No. 941-5610). On the high vacuum side, a bakeable valve (Varian Model No. 951-5027) permits isolation of the pump. The test chamber and 1 liter per second ion pump (Varian Model No. 913-0008) are connected via a glass seal-off tube to a larger Pyrex chamber. The system was mounted on a specially constructed bench with transite top. The bakeable valve and test chamber were located above bench level, the large ion pump and Vac-Sorb pump were located below. Bakeout of the system was accomplished by a 450°C oven which could be lowered onto the bench top. Temperature was controlled to within a few degrees by a Fenwall thermoswitch. Several different test chamber designs were experimented with. The final configuration is shown in Figure 3, and photographs are given in Figure 4. In all of the ultrahigh vacuum experiments performed, 1/4 inch diameter Z-cut quartz rods and 1/2 inch diameter Z-cut quartz flats were used as samples.

b. Experimental Procedure

Before placing the sample rod and flat in the test chamber, the two were optically contacted together. If microscopic inspection revealed the presence

of trapped dust particles, the contact was simply broken and remade in trial and error fashion until a good quality, dust-free contact was obtained. This procedure insured that no dust collected on the optically polished surfaces during test chamber assembly and attachment to the main vacuum system. After system assembly and pump-down to the 10^{-6} torr range, the optical contact was broken by means of the manipulator rod, and the two surfaces were held apart during a 12 hour 450°C bakeout. After the system returned to room temperature, the one liter per second ion pump was started and the tubulation from the test chamber to the main vacuum system sealed off. The pressure in the test chamber rapidly fell below 8×10^{-9} torr*.

The manipulator rod was then used to bring the end face of the rod into contact with the flat. The interference colors could be viewed through the rear end of the Pyrex test chamber, and thus the progress to optical contact could be checked in much the same manner as when one makes contacts under normal room conditions.

*Pressure was determined by monitoring the ion pump current, and using a conversion chart supplied by the manufacturer. 8×10^{-9} torr is approximately the lower limit using this method. The actual pressure was probably considerably lower.

In the first experiment, optical contact could be established over only about half of the 1/4 inch diameter area owing to a dust particle that had become wedged between the surfaces. Nevertheless, adhesion was evident. Dust was also responsible for three more failures⁹. As a result, the dust shield was added to the test chamber (see Figure 3). The copper pieces of the shield were polished and mated to provide an intimate fit. In a subsequent experiment, optical contact was made over the complete area. The effect was quite striking, a sudden seizure occurring as soon as a certain critical separation was reached. Contact was broken and remade several times with no difficulty. It was not possible to accurately measure the adhesive strength under vacuum, since a precise normal pull is required, but it was at least a few kg/cm^2 . When removed from the vacuum system, a force of 14 kg/cm^2 was required to break the bond, which is the same as for contacts made in air. Since optical contact bonds are good vacuum seals¹³, it is unlikely that water could have penetrated the interface and formed an adhesive film.

⁹We were rather surprised to find that dust collected on the surfaces even after high vacuum was established. We performed some experiments at ultrahigh vacuum to study the mobility of dust particles using the rod and flat, so to speak, as a "dust detector." It was found that a gentle tapping of the test chamber wall caused dust particles to change their positions and brought additional dust onto the surfaces. These simple experiments are of some significance since they show that dust particles are quite mobile and can be dislodged by minor shock and vibration. This should have a bearing on thin film evaporation work, where dust particles cause pinholes, and in areas as far afield as the question of dust mobility on the moon.

c. Analysis of Results and Critique of Liquid Layer Theory

In another experiment, the same procedure as described above was followed except that a longer time was allowed for pumpdown before the baking cycle was initiated. The pressure had fallen to a steady value of 6×10^{-8} torr. When the optical contact between the rod and flat was broken, it was noted that a small burst of gas was released causing the pressure to rise sharply from 6×10^{-8} torr to 15×10^{-8} torr and then return to 6×10^{-8} torr. It can be assumed that any water layer present would immediately evaporate when the surfaces were exposed to the vacuum¹⁴. On this assumption, a simple calculation shows that the gas burst was equivalent to only 1/100 of a monolayer coverage. The gas release was probably due largely to the flexing of the bellows which occur when the manipulator rod is pulled back to break the contact.

This evidence, together with the experiments where optical contact was made under ultrahigh vacuum, have demonstrated that the liquid layer theory is not complete. There exist forces between highly polished surfaces which of themselves are sufficient to produce adhesive strength of a few kg/cm^2 . It appears most likely that for optical contact bonds made under room conditions, two mechanisms are present—surface tension of a water film enhancing an independent attractive mechanism. In support of this, some experiments were performed with thin disks contacted on one edge to large flats. It was observed that if the surrounding atmosphere was saturated with water vapor, the contact area would increase, thus indicating that surface tension of condensed water tends to pull the disk into closer contact with the flat.

It is unlikely that the observed adhesion under ultrahigh vacuum resulted from a non-uniform distribution of electrostatic mosaic charges. Kitchener and Prosser¹⁵ have shown that such charges can be eliminated by exposing the surfaces to a glow discharge for several seconds. The ion pump conveniently provided this condition both at the start of pumping and during the early phase of bakeout. Also, there would be a certain amount of ionic backstreaming from the pump even at low pressure.

In accordance with the discussion of section II, it would not appear reasonable to attribute the adhesion to ionic or covalent bonding. The ductility of quartz is negligible, so that the area of intimate contact was probably only a minute fraction of the total interface. This, coupled with the necessity of precise near neighbor separation and orientation, would tend to rule out such forces. However, one cannot make an absolute statement on this matter. As mentioned above, under high compressive loads properly oriented rocksalt crystals will adhere (compressive loads had no apparent effect on adhesion in the present experiments). Studies of the frictional properties of diamonds have shown that the coefficient of friction increases from 0.05 at 760 torr to 0.9 at 10^{-10} torr with the frictional resistance attributed to the shearing of microwelds.¹⁶

The adhesion was most likely due to van der Waals forces or, more specifically, London dispersion forces. These forces, which produce a universal attraction between atoms and molecules, produce, as well, an attractive force between solid bodies. This effect has been directly observed by several investigators at separations ranging from 1000 to 10,000 Å using optically polished surfaces.^{15,17,18}

Their results agreed, within experimental error, both with the microscopic theory of Casimir and Polder¹⁹ which assumes additivity of the London forces between individual molecules, and the more recent macroscopic theory of Lifshitz²⁰. According to the proponents of the latter theory, there is no justification for extending the additivity of London forces to the case of solid bodies. The Lifshitz approach makes no assumptions about the nature of the interaction between individual atoms, but instead attributes the interaction between macroscopic bodies to the fluctuating electromagnetic field which is always present in the interior of an absorbing medium and extends beyond its boundaries. In any event, both theories predict that for separations less than about $200 \text{ \AA}$, the attractive force per unit area between flat parallel surfaces should be proportional to the inverse third power of distance. This has been confirmed by some recent work of Bailey²¹ on the attraction between molecularly smooth mica sheets, although there is some doubt that ionic attraction was entirely eliminated. Attractive forces of 1100 kg/cm^2 at $10\text{-}\text{\AA}$ separation and 1.5 kg/cm^2 at $100 \text{ \AA}$ were reported. Unfortunately, it is not possible to make a sensible calculation of the attraction to be expected between surfaces in optical contact because the distance between them varies over such a wide range due to surface roughness (approximately $150\text{\AA}$). Nevertheless, since the separation distance over a large percentage of the interface was probably less than $100 \text{ \AA}$, van der Waals forces could easily account for an adhesive strength of 14 kg/cm^2 . If smoother surfaces can be produced, a considerably enhanced adhesion should result.

IV. APPLICATION OF OPTICAL CONTACTING TECHNIQUES

1. Preparation of Surfaces

In order to make good optical contact bonds, the surfaces must be free of grease and other contaminants, and large dust particles must be excluded. It is not necessary to use a dustfree room, but this would certainly be helpful and reduce the number of tries that are normally required to obtain a reasonably good bond. For cleaning the flats, we have generally found it sufficient to wipe them first with acetone-soaked lens tissue and then with lens tissue soaked in purified petroleum ether. A chromic acid solution can be used to remove grease. We have occasionally used a cellulose acetate tape available from Ernest F. Fullam, Inc., Schenectady, New York for removing dust just prior to contacting. This tape is normally used in electron microscopy surface replicating work. Surfaces are cleaned first in the manner described above, then tape, softened in acetone, is evenly applied. The tape is removed when dry and the two surfaces can often be contacted without any apparent dust. However, pieces of the tape are sometimes left sticking to the surface so that this method was not used when very high quality contacts were required.

2. Contacting Techniques²²

Once surfaces have been cleaned, they must immediately be brought into contact, otherwise dust will recollect. The seizure of the surfaces, which signals the onset of optical contact, is achieved by making the two surfaces parallel and pressing with a normal force of a few pounds per square inch. We have found that this process must be carried out manually while observing the interference fringe pattern to assure parallelism. Since work must be done by hand and eye, results improve

significantly with experience, as in many other areas of the optical art.

If rod specimens are to be bonded, it is best to first contact them to auxiliary flats. This permits one to inspect the bond carefully under a microscope for freedom from dust and other irregularities. If a white light source is aligned so that the microscope views the reflection from the interface, the area in optical contact will appear black because of the almost negligible reflectivity. Areas of noncontact caused by dust particles will show up clearly as white specks against this background. Optical contact is made between the rod-end faces by simply snapping them off the auxiliary flats and pressing them quickly together. If a disk is to be contacted to a rod, only the latter uses an auxiliary flat. In both cases, it is important to carefully recheck the contact area of the assembly under a microscope.

We have succeeded in making optical contact bonds between various combinations of the following materials: Pyrex glass, fused quartz, single crystal quartz, MgO, KCl, Al_2O_3 , Ge, and YIG - the latter 5 in the form of single crystals. And we have made optical contacts between quartz and evaporated layers of gold and permalloy. It would appear that any two surfaces can be optically contacted if they can be made sufficiently flat and smooth. This observation supports our contention that the bonding is due to van der Waals forces (in the absence of the ultrahigh vacuum experiment, it could also support the liquid layer theory).

3. Temperature Cycling Experiments

Experiments were conducted to determine if optical contact bonds retained their strength at low temperature. These experiments have a significance as a critique of the liquid

layer theory, but their main purpose was to determine which optically contacted transducer-sample combinations could be used in low temperature gigacycle ultrasonics research.

We have found that optical contact bonds between rods of dissimilar material break when cooled to liquid-nitrogen temperatures, and this limits, to some extent, the practical applications of the optical-contact technique. We attribute this breakage to differential thermal expansion since breakage does not occur if the two rods are of the same material and crystal orientation. A bonded pair of X-cut quartz rods and a bonded pair of AC-cut quartz rods were cooled to 1.5°K where ultrasonic experiments were performed on them (see section IV, 4. below) and then cycled back to room temperature. There was no apparent change in the bond as a result. Also, we have found that thin disks bonded to rods or large flats can be cycled to cryogenic temperatures even if the larger piece is of a different material. Apparently, the thin disks can deform and still retain their adhesion.

4. Gigacycle Ultrasonic Experiments

Several optically contacted transducer-sample combinations were supplied to Dr. Paul Carr of Air Force Cambridge Research Labs who performed experiments on them to determine the reflectivity of the bonds for acoustic waves of 3 and 9 gigacycles ($\lambda = 19,500^{\circ}\text{A}$ and $6,500^{\circ}\text{A}$ respectively). The apparatus and technique employed are described elsewhere²³. The combinations supplied included:

- (1) X quartz rod bonded to X quartz rod
- (2) X quartz disk bonded to Z quartz rod
- (3) X quartz disk bonded to ruby rod
- (4) AC quartz rod bonded to AC quartz rod

The two rods of the first combination were of different lengths so that echoes from the bond and from the far end face could be easily distinguished. The optic or Z axes of the rods were aligned to within a few degrees. For longitudinal waves of 3 GC and 9 GC, it was found that less than 1% of the incident acoustic power was reflected by the optical contact bond. This is a significant improvement over standard adhesive bonds which normally transmit only about 1% of the incident power. It is worth noting that a small dust particle was trapped between the rods producing an area of non-contact over about 1% of the cross sectional area. The observed reflectivity could have been due almost entirely to this.

The second combination also demonstrated excellent acoustic transmission properties, although it was not possible to measure directly the bond reflectivity. On resonance, these disks gave conversion efficiencies of the same order of magnitude as with surface excitation of an X-cut quartz rod in the same cavity²³. If it is assumed that the bond thickness is negligible compared to the acoustic wavelength, the reflectivity is given by:

$$R = \frac{Z_1 - Z_2}{Z_1 + Z_2} \quad (2)$$

where Z , the characteristic acoustic impedance, is the product of the mass density and the relevant acoustic velocity. For combination (2), the reflectivity would be 0.093 for longitudinal waves. About a dozen echoes were obtained with combination (3) and considerable ringing was observed. From equation (2), the bond reflectivity should be 0.49, which would not account entirely for the poor results. The quality of the contact was poor; about one third of the interface area was not in contact and it is believed that there was a rather thick water layer present.

With combination (4) using transverse waves of 3GC and 9GC, the reflection from the optical contact bond was about an order of magnitude larger than for longitudinal waves in combination (1), or about 10%. The AC rods were aligned by matching up scribe lines which were cut in the sides of the rods by the manufacturer. However, by using a polarizing microscope, it was found that the rods were actually misaligned by approximately 17° . Letting E represent the amplitude of the elastic displacement vector, the amplitude of the wave transmitted through the bond, E_t , is related to the amplitude of the incident wave by:

$$E_t = E_i \cos 17^\circ = (0.96) E_i .$$

Acoustic power, P , is proportional to E^2 .

Thus,

$$P_t = (0.96)^2 P_i = (0.92) P_i .$$

Assuming loss is negligible,

$$P_r = 0.08 P_i , \text{ a reflectivity of 8\%}.$$

Thus, the large reflection could have been due primarily to this misalignment rather than the bond itself.

Another pair of optically contacted X-cut rods, similar to combination (1) were used in a reentrant cavity with rounded posts so that both longitudinal and transverse waves could be generated. It was found that the reflectivity for transverse waves was higher than for longitudinal waves by about a factor of 10, but it is not certain that there was perfect crystallographic alignment of the two X-cut rods. The polarizing microscope was used to align the Z axes to within 1° . However, with this technique, it is not possible to distinguish between the +Z and -Z directions. The transverse waves are polarized at $+30^\circ$ and -60° with respect to the Z axis. Hence, if the +Z axis of one rod was lined up with the -Z axis of the other, a high reflection would be expected at the interface. Thus, the question of whether an optical contact bond actually reflects transverse waves to a greater extent than longitudinal waves is still open.

5. Extension of the Technical Applications of Optical Contact

With respect to low temperature gigacycle ultrasonic work, there are two major problems which must be overcome before optical contact can be considered a general solution to the bond attenuation problem. The first is the breakage of the bond when large specimens of dissimilar materials, joined by optical contact, are cooled to low temperatures. The second is the reflection due to acoustic impedance mismatch at a bond between dissimilar materials (see eq. 2). Fortunately, it may be possible to solve both problems together by evaporating metal layers onto the surfaces, and contacting them while still under ultrahigh vacuum. This approach may, in addition, result in stronger adhesion since metallic bonding should contribute.

It should be possible to choose a metal or alloy whose ductile-brittle transition point lies a few degrees above helium temperature. A film of this metal between rods of dissimilar materials should solve the thermal breakage problem. If this film is also chosen so as to be of approximately intermediate acoustic impedance, and a quarter wavelength thick, the reflection from the bond can be eliminated.

Section V, below, describes the development of an ultrahigh vacuum evaporator designed to accomplish the above. It can also be used for other general evaporation work, and for depositing double layers of suitably chosen quarter wave films to improve the coupling between electromagnetic sources and acoustic waves in accordance with the recent paper by Haydl et al.²⁴

V. DEVELOPMENT OF ULTRAHIGH VACUUM EVAPORATOR

1. Design Considerations

Considerable effort was expended toward finding the optimum design for a laboratory installation which would be suitable for performing optical contact bonding under ultrahigh vacuum after the deposition of suitable thin films. First, a survey of existing evaporators was made to determine whether commercially available units would have sufficient flexibility for the work intended. It was concluded that none of the units examined fitted the requirements exactly. Consequently, construction of such a system, using commercially available and tested parts, was undertaken.

Early design centered around the use of Vac-sorb pumps coupled with a large ion pump. This basic approach was followed in the final model.

Its principal features include interchangeable (glass and stainless steel) bell jars, multiple access ports to the working area, adequate room inside the system for installation of E-guns or similar special sources, and provision for introduction through the sides of the chamber of manipulators to aid in the making of contacts under ultra-high vacuum conditions.

As the work progressed, it became evident that it would be necessary to consider the use of materials having relatively high vapor pressures. Calculations showed that the use of an ion pump as the principal method of achieving the vacuum desired would be impractical, because of the necessity for disposing of large amounts of foreign vapors produced in the process of evaporation. Furthermore, it was concluded that a somewhat larger system than had been originally contemplated would be required to allow adequate spacing between evaporation source and substrate. This consideration led to the final adoption of a system employing a large diffusion pump with a refrigerated baffle, backed by a second diffusion pump inserted ahead of the fore pump. A diagram of the structure of the main chamber of the system is shown in Figure 5. Attached to the main chamber are a standard six-inch Heraeus oil diffusion pump backed by a two-inch oil diffusion pump and a standard roughing pump. The main chamber is equipped with an ionization gauge and is also provided with two viewing ports, several side tubes for the introduction of manipulators, an E-gun port, and other appurtenances necessary for satisfactory operation. The electrical control of the unit is centered in a control chassis (see Figure 6).

The system is intended to be well suited to the three generally used methods of thin film deposition (evaporation, sputtering, and vapor-phase reaction) and is intended to operate from conditions of moderately high vacuum with relatively high vapor pressure materials which require very rapid pumping to maintain proper conditions in the chamber, up to and including the ultrahigh vacuum range (below 10^{-10} torr) if proper precautions be taken.

High current feed-throughs are provided in case it is desired to use resistance heating of the materials to be placed upon the substrate, which may be desirable with low melting point materials. These same high current feed-throughs can be used for the introduction of relatively low-frequency rf power in case induction heating is desired. Direct provision has been made for inclusion of a Varian E-gun type of apparatus.

In the use of any of the above methods, relatively good vacuum conditions are desired, but in most cases the ultrahigh vacuum range need not be reached. In the high vacuum range, the chamber should be operated with a neoprene gasket (without refrigeration) and the baffle need not be employed for the average application. To attain ultrahigh vacuum, the gasket should be refrigerated, and liquid nitrogen placed in the baffle.

In case sputtering is the desired method of deposition, a positive ion source can be placed in one of the feed-throughs to the chamber (this has not been implemented as yet) and the conventional technique applied. Here, high pumping speed is vital, and the baffle will probably be useful to increase the effective pumping speed.

In case glow-discharge type sputtering is used, a high voltage feed-through is provided to allow the power supply to be connected. Since the optimum pressure range is approximately 10^{-2} torr, no elaborate precautions concerning leaks need to be taken and the vapor pump need not even be used. Similar considerations apply to vapor-phase reaction deposition.

2. Mechanical Design

The main vacuum chamber was constructed from stainless steel, due to its desirable outgassing performance and relatively low cost. The chamber is 18" in diameter, and weighs approximately 1000 lbs. It is provided with high current electrical feed-through (20 amp rating), multiple connection general purpose feed-throughs, and ion-constantan feed-throughs for the introduction of thermocouples, two viewing ports (2" diameter), and a standard ionization gauge.

The pumping system includes a thermocouple gauge for monitoring the fore pressure of the system, which is interlocked with the oil diffusion pump to prevent their being turned on under adverse conditions. A pneumatically operated bellows type valve is included for isolating the system from the fore pump. Water cooling is provided for the oil diffusion pump, and a quench coil is also included to allow rapid shut down of the main pump. The main bell jar, which weighs several hundred pounds, is provided with air-operated lifts to allow easy access to the chamber itself. A photograph of the mechanical arrangement of the completed system is shown as Figure 7.

3. Electrical Design

Basic control of the unit is designed to prevent contamination of the oil in the pumps and other undesirable effects in case of a power failure or other catastrophic failures involving loss of vacuum. The control system requires (through the use of interlocking relays) a proper sequence of turn-on and turn-off for the system. For example, the power to the main diffusion pump is interlocked both by the supply of an adequate flow of water and by the presence of a suitable fore vacuum. Likewise, the quench coil in the main diffusion pump is connected in such a way that in case of power failure, water is automatically introduced into the coil to effect rapid cooling and thereby minimize damage. Also, a bi-metallic element thermostat is provided to assure proper cooling water temperature and to automatically turn off the diffusion pump power in case of inadequate cooling.

Pressure is measured by the thermocouple gauge and by the ionization gauge, using a standard commercial ionization gauge control unit. The thermocouple gauge is connected so as to monitor the fore pressure and to prevent operation of the diffusion pump without an adequate fore vacuum; the circuit employed for this purpose is shown in Figure 8.

The power supply for the E-gun source employed is a standard Varian unit, and is completely described in the instruction book for this unit. The only special provision made in the control circuitry is the provision of an interlock to prevent the E-gun from being activated unless the fore pressure is adequate and the diffusion pumps are on.

4. Performance

The system is designed to operate satisfactorily from the moderate vacuum range to the range somewhat beyond 10^{-8} torr without refrigeration equipment being provided for the O-ring seal, and to pressures of 10^{-10} torr in case such refrigeration is provided. Although a refrigerating coil has been incorporated in the system, it has not been felt desirable to extend the range to the ultrahigh vacuum region until experimental requirements demand it. A standard commercially available manipulator has been included in the system, but this will have to be replaced later by an improved design if ultrahigh vacuum performance is required. The system is designed to permit bake-out (using fibreglass blankets and standard strip heaters) when ultrahigh vacuum operation is desired.

Other than routine leak-checking with a helium leak detector and the repair of leaks so revealed, no particular operating problems have been encountered and the performance of the system is good.

APPENDIX I

Study of Trapped Dust Particles

Small dust particles (on the order of $1000 \text{ \AA}$ diameter or smaller) are often trapped between surfaces in optical contact, and these produce local areas of non-contact which are readily observed because of the enhanced local optical reflectivity. These dust particles also set up large local stresses which, in the case of pyrex flats, can be readily detected with a polarizing microscope. It is well known that glass becomes birefringent when stressed. The phase difference between the ordinary and extraordinary wave being given by

$$\Delta = \frac{2\pi}{\lambda} (n_2 - n_1) d$$

where n_1 , is the index of refraction for polarization parallel to the lines of stress and n_2 for polarization perpendicular to the stress lines ($n_2 > n_1$); d is the thickness of the birefringent medium. The equation can also be written as:

$$\Delta = \frac{2\pi}{\lambda} C T d$$

where C is the relative stress optical coefficient and varies between 1×10^{-13} and $10 \times 10^{-13} \text{ cm}^2 / \text{dyne}$ for common optical glasses. T is the stress.

Because of the circular symmetry, one sees a four lobed star pattern, the nodes being at multiples of 90° with respect to the direction of the analyser. We were able to determine that the phase difference was less than π . If it is assumed that the deformation is spherically symmetric, leading to an effective d on the order of 10^{-2} cm , we obtain $10^{10} \text{ dynes/cm}^2$ for the stress. This matter was not pursued further, and the exact significance of the results is not clear. Nevertheless, it should be recognized that small dust particles can be used as a quasi microscopic probe for investigating adhesion forces between surfaces.

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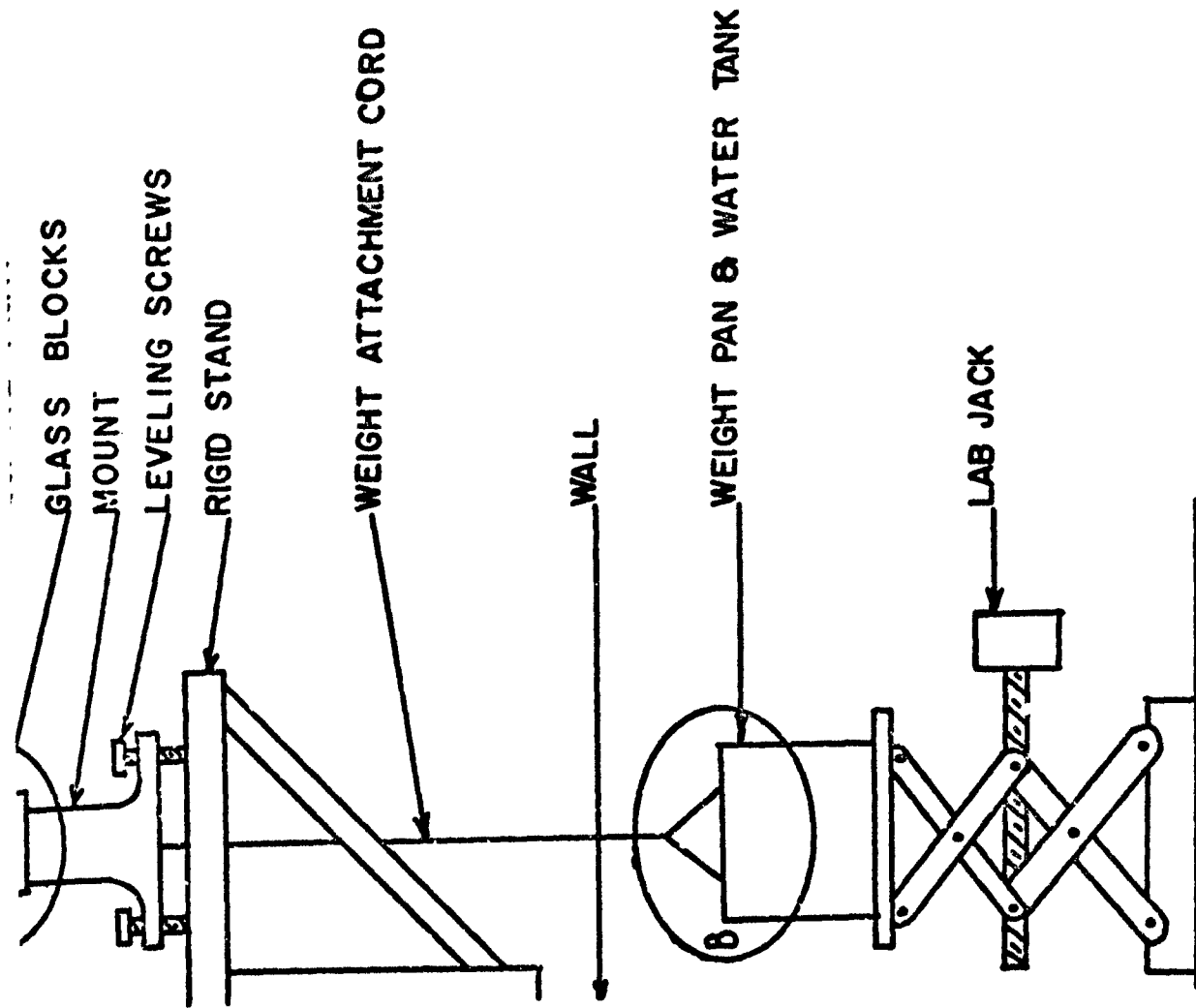
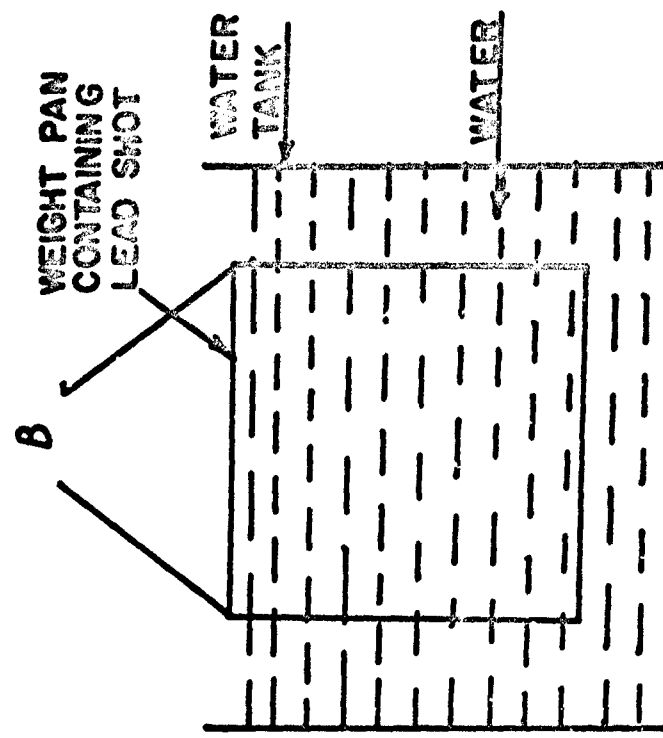
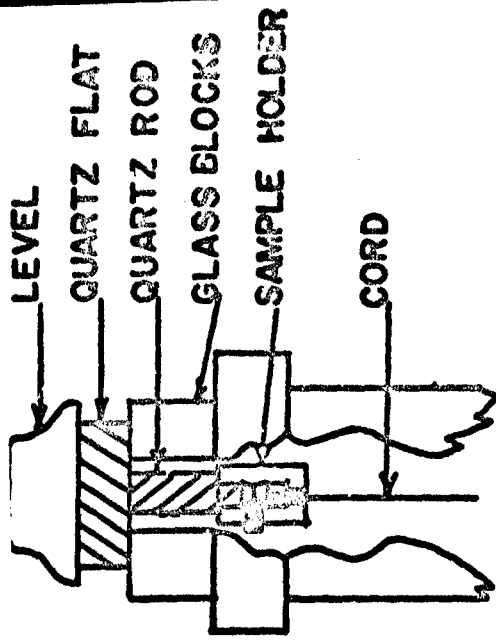


FIGURE 1
APPARATUS FOR MEASURING TENSILE STRENGTH OF OPTICAL CONTACT BONDS.

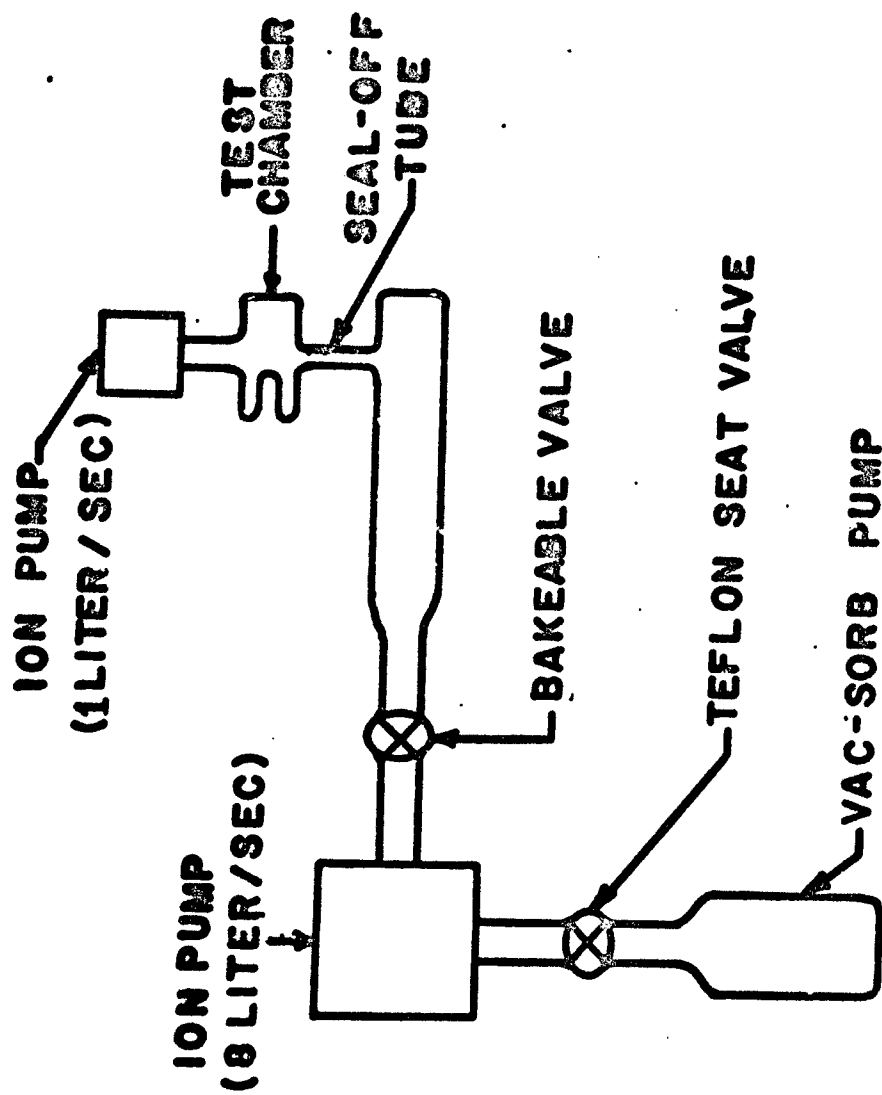


FIGURE 2
SCHEMATIC OF ULTRAHIGH VACUUM SYSTEM.

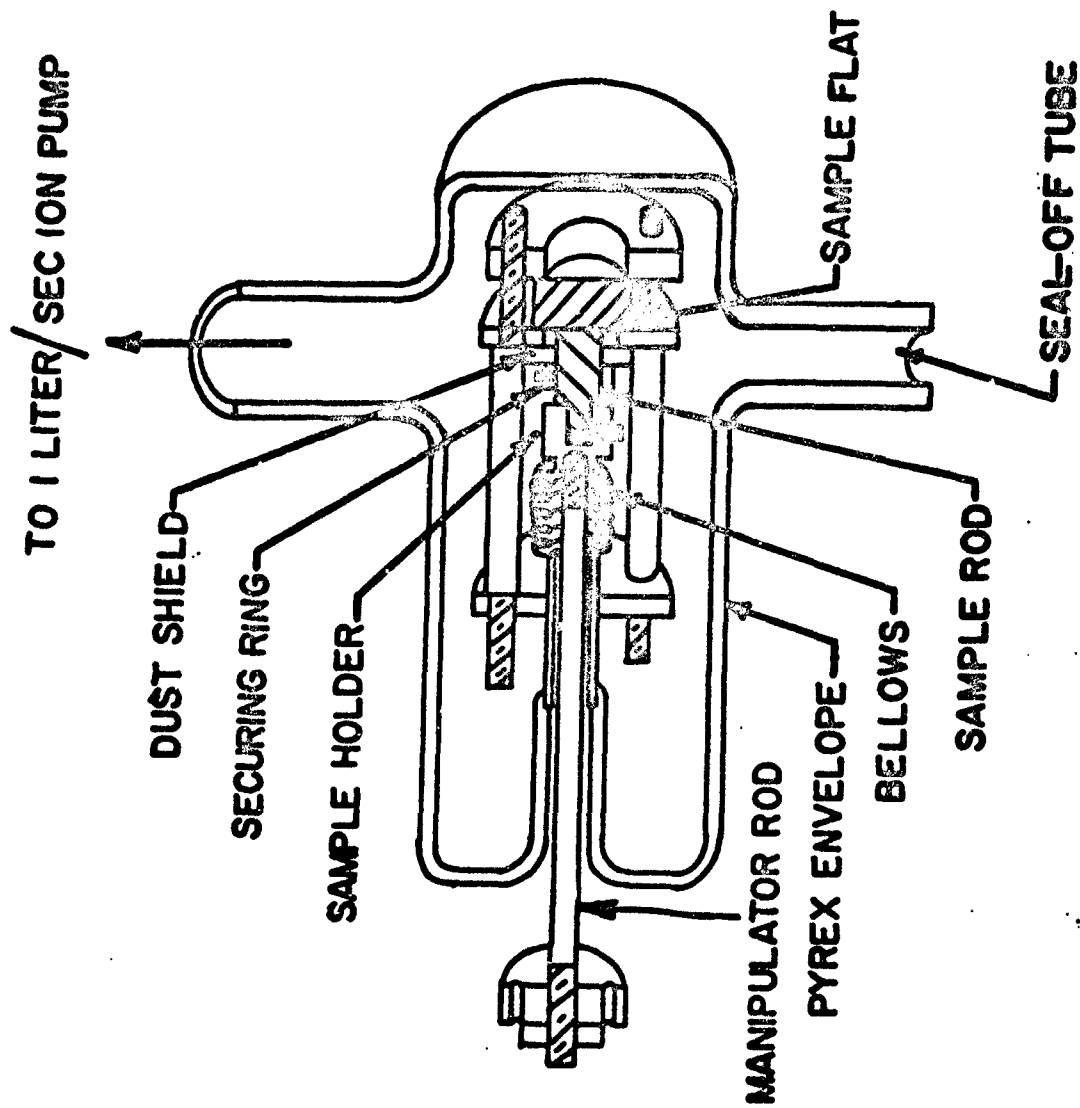


FIGURE 3

SCHEMATIC OF TEST CHAMBER. (Interior parts are made of OFHC copper except for Kovar tube, monel bellows and 304 stainless steel rods.)

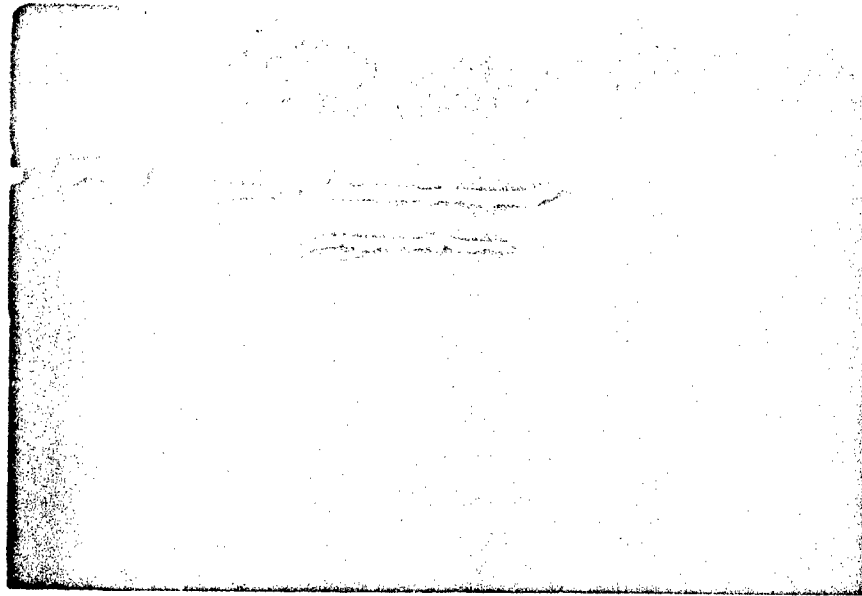
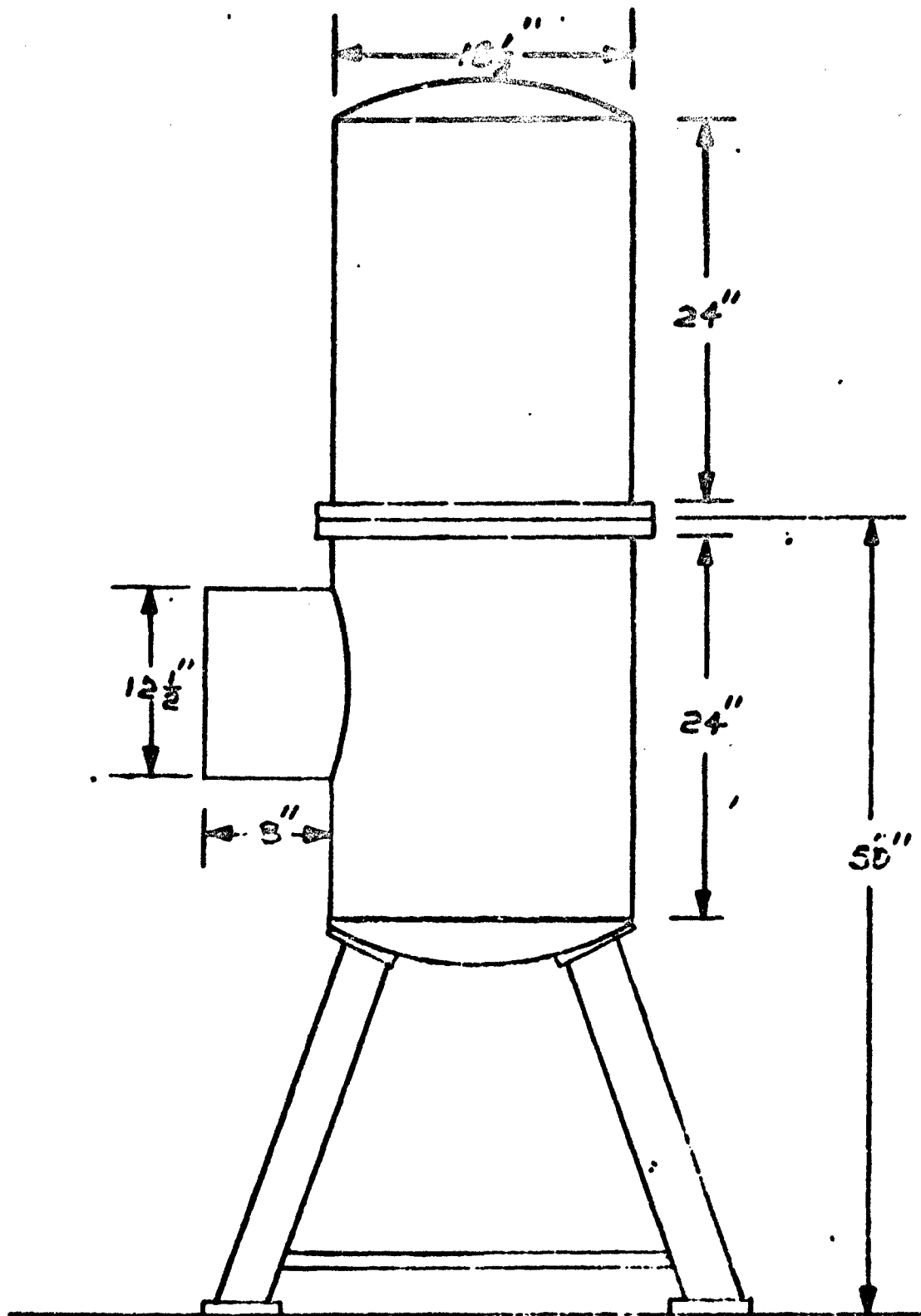


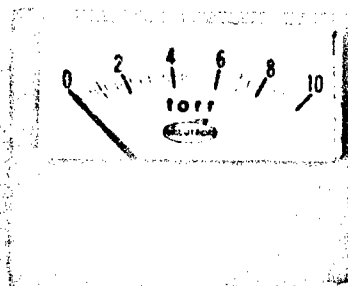
FIGURE 4
PHOTOGRAPHS OF TEST CHAMBER AND VACUUM SYSTEM.



MATERIAL: STAINLESS STEEL TYPE 304
O-RING GROOVE IN UPPER
SECTION FLANGE ONLY
INSIDE SURFACE POLISHED

FIGURE 5
MAIN CHAMBER OF VACUUM SYSTEM

ENTRANCE TO CHASSIS
OUT OF CHASSIS



VACUUM GAUGE
CONTROL



VACUTRON COMPANY

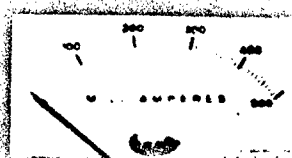


FIGURE 6
CONTROL CHASSIS

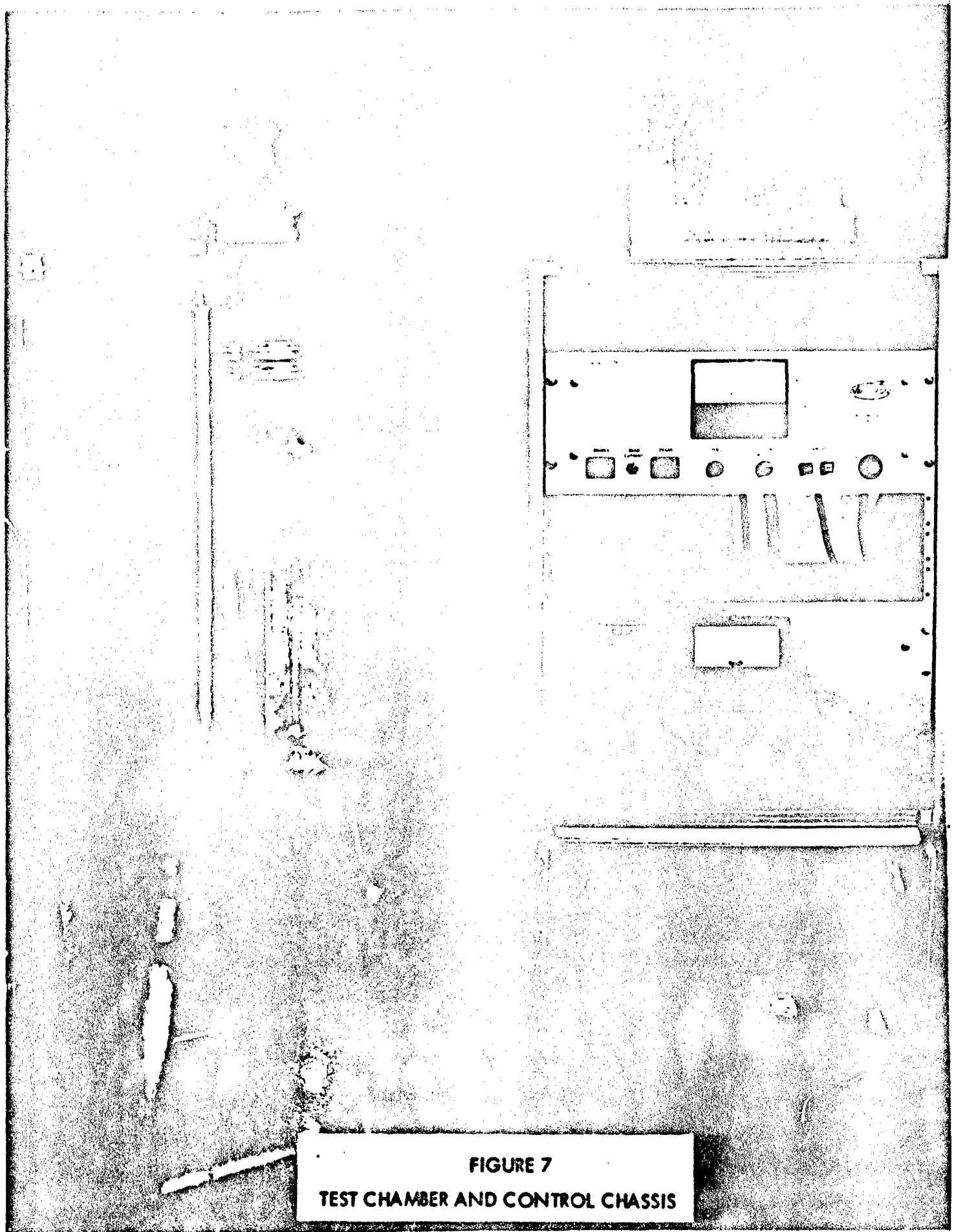
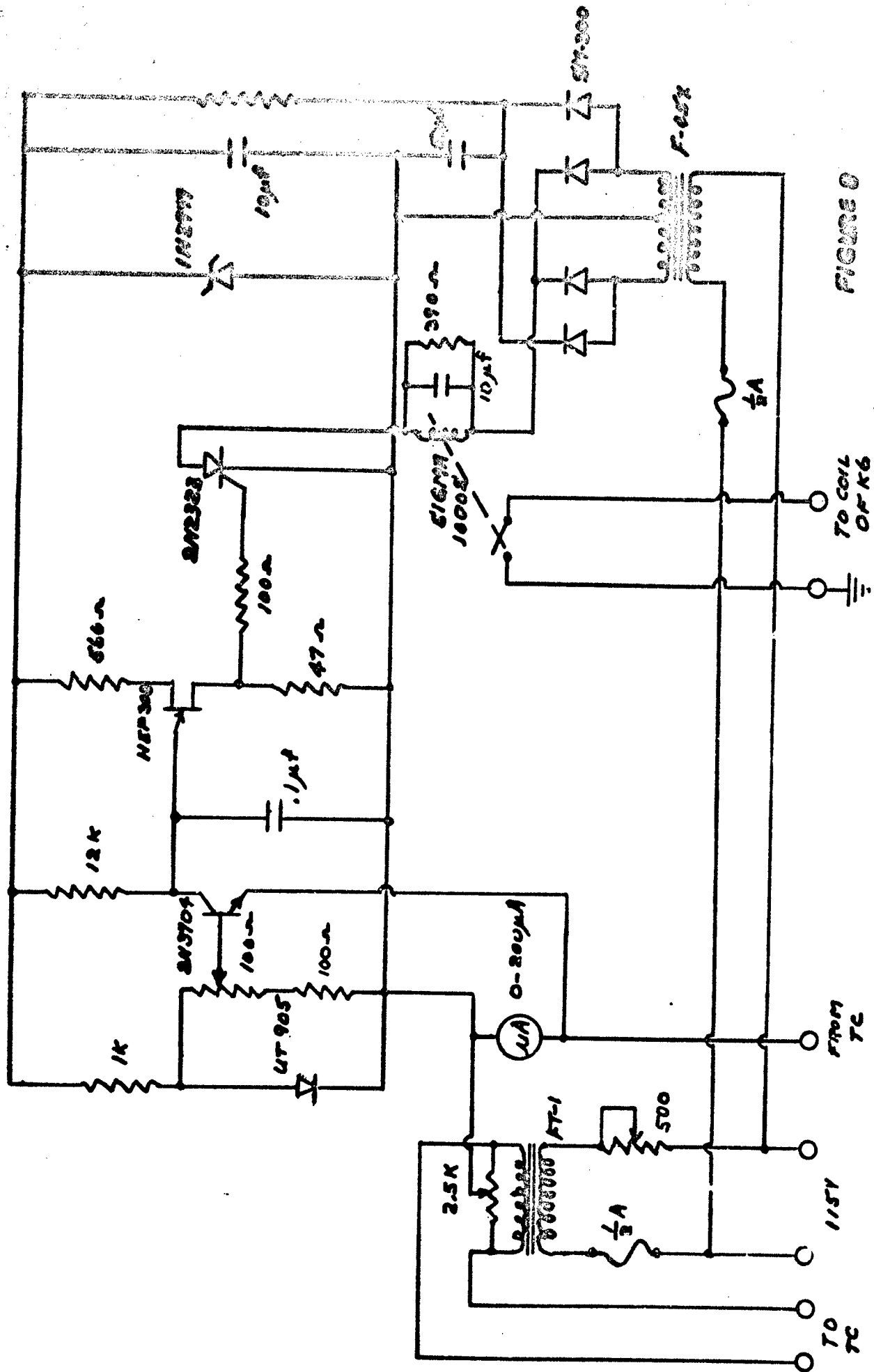


FIGURE 7
TEST CHAMBER AND CONTROL CHASSIS



LOW VACUUM TIGHT-CAPABLE

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The adhesion of optically polished surfaces ("optical contact") was investigated both under room conditions and ultrahigh vacuum with the twofold objective of determining the adhesion mechanism and its characteristics and extending the technological applications. It is shown that optical contact adhesion occurs readily under ultrahigh vacuum, thus demonstrating that the widely held liquid layer theory is not complete. Adhesion mechanisms are discussed, and it is concluded that London dispersion forces are responsible for the adhesion under ultrahigh vacuum. For optical contacts made under room conditions, surface tension of a liquid probably also contributes. It is shown that optical contact techniques can be used to obtain extremely efficient transducer-sample bonds for gigacycle ultrasonic work. Reflections as low as 1% of the incident acoustic power were obtained at 3 GC and 9 GC. Techniques for making such bonds are discussed. It is pointed out that the incorporation of evaporated metal films should enhance the properties of optical contact bonds and eliminate some practical difficulties. The design and development of a high vacuum evaporator are described.	

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