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SOLID FILM LUBRICANT EVALUATION TECHNIQUES

R. J. Benzing

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R. J. BENZING

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FOREWORD

This report was prepared by the Fluid and Lubricant Materials Branch, Nonmetallic Materials Division, Air Force Materials Laboratory, Research and Technology Division with Mr. R. J. Benzing acting as project engineer. The preparation of the report was initiated under Project 7343, "Aerospace Lubricants", Task 734302, "Dry and Solid Film Lubricant Materials."

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R. L. ADAMCZAK, Chief
Fluid & Lubricant Materials Branch
Nonmetallic Materials Division
Air Force Materials Laboratory

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ABSTRACT

The use of mechanical, physical, and chemical evaluation techniques for solid lubricants is discussed. Those mechanical evaluation tests reviewed include simulative friction and wear tests and component test employing specimens such as gears and bearings. Physical and chemical tests include those for studying adhesion, thermal resistance, fluid compatability, oxidation behavior, film thickness, and thermal shock tests. In addition to the description of the various tests, the use of each in the various phases of film development is reviewed.

INTRODUCTION

There are a large number of standardized test methods and techniques available to evaluate conventional lubricants such as oils and greases. Early development effort on these materials employ chemical and physical bench tests quite extensively prior to testing in full scale components. Some of these methods are shown in Table I. For the most part, the lubrication engineer is able to take results from these tests and, with a minimum of wear testing, predict, rather successfully, a lubricant's performance in an actual application. It is possible to do this because of the long history and sound understanding of these tests and their correlation with actual applications.

Solid film lubricant developers, on the other hand, rely heavily on friction and wear testing in an effort to evaluate their lubricants. There are, however, a number of chemical and physical tests that are employed. Since the major effort in evaluating solid film lubricants is expended in testing their lubrication behavior, we may look at most programs in this area as a mechanical evaluation approach to material development. It is a quite different approach from that used with the oils and grease where the majority of initial evaluation is of a chemical nature. There are several reasons why this approach prevails with solid lubricants:

1. There are few chemical tests for solid lubricant components which can be related to the performance of the film in its final formulated condition. Oxidation behavior would be one possible exception.
2. Most of the tests now employed have no direct correlation with service even when run on fully formulated films
3. In many programs the research on a new film is concerned with the development of new or improved methods of bonding and/or application

and as a result the investigator may already know that the lubricating component is satisfactory for the intended use.

4. The solids of interest are less adversely affected by chemical reactivity than the fluid considered for oils and therefore chemical properties are not as critical as with oils.
5. Because of interactions of the various constituents in a solid film lubricant it is often simpler to run one type of friction or wear test to get an overall rating rather than a series of physical and chemical tests.

Of course, as in most things, there are exceptions to the rule. We have already seen this in other papers and will note it in future portions of this paper. It should also be noted that chemical analysis of solid film lubricants is being used more and more in the research laboratory and in particular in those laboratories involved mainly in fundamental studies on the films and/or their constituents.

The purpose of this paper will be to review methods for evaluation of solid film lubricants. Particular emphasis will be placed on the area of lubrication performance evluation with a somewhat briefer section on chemical and physical testing. In order to do this, and also orient the subject of solid film evaluation techniques as they are used, we will first review the evaluation involved in the selection and development of a typical bonded solid film. Having done this, we will then describe the tests and factors relative to their use in more detail. Since previous discussions have covered both the fundamentals of lubrication with solid lubricants and the evaluation of other types of solid lubricants, we will not repeat these areas in this paper although in most cases they lay the foundation for initial material selection.

EVALUATIONS CONDUCTED IN THE DEVELOPMENT OF A BONDED SOLID FILM

Figure 1 depicts briefly the development and evaluation cycle as it might occur for a bonded solid film lubricant. Since this is a bonded film it would consist of a pigment, binder, and any additives required to improve film deficiencies. It would likely be bonded to a metal surface which has been pretreated to improve adhesion of the film to the metal. Each block has been numbered for ease in discussion in this paper but the numbering does not indicate the order in which that step might be carried out.

In the selection of a binder you rely on the operating temperature to define the basic type. For example, at temperatures in the region of 1000°F you would proably select a ceramic binder while around 200-300°F you would pick a resin binder. The evaluation that would be carried out to further define the binder would be the chemical or physical types. You would be primarily concerned with thermal and oxidative stability as well as resistance to degradation by any fluids with which the binder could come in contact. These can be evaluated by coating panels with the binder and subjecting the panels to the test conditions of interest to observe any detrimental effects. A series of tests used for resin bonded films are described in Table II. Similar ones can be employed for ceramic bonded films with modification made where dictated by the eventual use of the films. In the case of ceramic materials you might also employ a phase diagram of the system should one be available. Friction and wear tests could be run if it were expected that the binder had some self-lubricating properties of its own. If the binder met the criteria set in this step it would then be checked for bonding with the metal surface of interest. The major criteria in this step (block #3) would be adhesion and surface continuity or uniformity. In adhesion it is self evident that a non adhering film would not be expected to last for very long. One test procedure is given in Table II. Another that is commonly used is to rub a tip or scribe over the film to see if you obtain breaking up of the film. The importance of adherence of the film would apply to all types of bonding materials. In the case of ceramic films you could encounter a problem with differences in thermal expansion of the substrate metals resulting in cracking and poor adherence. While this can be measured it is often much simpler to observe the results in a microscope. Figure 2 shows the effect of cracking of one film when used with conventional steels versus a uniform appearance obtained when coating the same film on Ni based alloys. The curing cycle and temperature should be evaluated here and in block 4 where you are checking the compatibility of the binder with the pigment. Effects on the metal substrate such as changes in hardness due to the curing cycle can be detrimental.

In selecting the pigment there are several properties which should be evaluated. Some of these are listed in Table III. The first four are generally available from a chemical or physical handbook. Their importance has also been reviewed and will not be repeated here. Thermal stability can be checked in various test procedures such as DTA and TGA. Oxidative stability can be evaluated by heating the material at the desired temperature in air and then analyzing weight and composition changes. Friction and wear behavior of the pigment are of the utmost importance since the pigment is expected to provide the lubrication to the system. In general, this will be carried out in some sort of simulative test rig. In this paper we will use the term simulative test rig to define those friction and wear rigs which employ a test specimen other than an actual machine element. (A flat bearing on a rotating ring is one possible example). The test rigs which employ actual machine elements, such as bearings and gears, will be referred to as component test rigs. The most common simulative test rig used in evaluating a pigment would be of the pellet type. In such a rig the pigment can be compressed and formed into a test specimen or pellet which is then run against a metal surface.

After one or more pigments are selected they must be matched with the binder to see if you have a set of compatible materials. Information of interest would mainly involve changes in adhesion and chemical changes of the pigment due to the curing process. These could be evaluated in much the same manner as described above.

The next step in the cycle would couple blocks 5 through 7 where a formulated film is prepared and coated on the metal substrate. Various bonding techniques, including curing cycles, would be investigated and might include spray, dip or brush coating. If a problem existed in corrosion it would be evaluated here by a method as outlined in Table II and possibly solved by means of an additive. Pretreatment of the metal surface to improve performance of the film might be carried out by phosphating or sandblasting for example. Film thickness would be studied. The results of all these steps are then evaluated in block number 8.

Many of the physical and chemical tests described previously are also used in this phase of evaluation of the film. There is, however, a considerably larger effort generally devoted here to simulative wear testing of the material. In some cases component evaluation may also be carried out.

Most of the various physical and chemical test methods previously used are of a go/no-go type. The simulative wear tests, on the other hand, are used to define performance limits of the film when subjected to varying environmental test conditions as shown in Table IV. This then defines whether the film will meet the actual application requirements if it is being developed for one application. If it is not being developed for one give application the tests define the overall limits of the film. These simulative tests do not give direct correlation with service but point the way as to relative ratings of various films.

The final step in the development cycle would be testing for an actual application. This generally employs either an actual piece of equipment or a component test rig. The objective of such evaluation would be to obtain life ratings on the film under the conditions to be encountered in actual service.

As shown in block 9 there can be reformulation work required as a result of the evaluation in blocks 8 and 10. This reformulation can be simple or extensive based on the results of testing. It could actually involve any of the effort carried on in blocks 1 - 7 or simply a change in additives. It should be noted that, based on experience, a person might start at any phase of the development cycle and go immediately to testing as indicated in blocks 8 or 10. This is actually quite a common approach where a film is formulated and applied with no previous testing and then evaluated in a simulative test rig. It often is done in areas previously studied or in areas where known materials are used. It is a trial and error type approach and if successful can save considerable time and effort. In the least it often guides one to a reformulation type effort.

The final type of evaluation is shown in block 11 and this meant to imply testing for acceptance or quality control of a developed material. Table V shows condensed evaluation requirements of a government specification (1) for resin bonded materials. It is when one reaches this phase of evaluation that you most nearly approach standardized testing as normally employed with greases and oils.

Having now shown various phases of testing we should delve in more detail on the actual methods or tests employed. We have seen where the various ones are used, let us now turn to their actual merits and what they can tell us.

DISCUSSION OF EVALUATION TEST METHODS

I. Mechanical Testing

Both simulative tests and component tests will be reviewed under the general classification of mechanical testing. Many aspects are applicable to both types of tests. When conducting the various mechanical evaluation tests one is generally interested in obtaining performance data on the solid films subjected to a series of environmental test conditions. Very often the materials themselves are also varied giving further factors to consider in assessing the results.

The actual performance of a solid film lubricant can be rated in many ways. The most common yardsticks are those of friction, wear, and operational life. Friction behavior in some cases might be of interest during the full cycle of testing while in other cases the investigator may only be interested in time to a given level. Wear measurements may be studied in a similar fashion where wear rate or total wear is the criteria. Operational life is often expressed in load cycles, time, or number of oscillations before some failure point. The failure point is generally defined in terms of some friction and/or wear level as above but can include complete destruction of the test specimen. It is impossible to define or classify the type or quality of data versus the type of testing taking place. Quite often the performance test in an actual piece of equipment may be every bit as sophisticated as the test run by the most meticulous basic research man.

In turn, sound basic data can be taken with a simple rig. At the 1965 ASME-ASLE joint Conference, one researcher brought his test device to the meeting in a suitcase so that he might actually demonstrate results on PTFE for the audience while presenting his paper.

In obtaining performance data on solid film lubricants the investigator if often interested in a wide variety of conditions. Table IV lists some of the factors that are commonly studied in evaluating the solid film lubricants. In addition you can study such properties as surface finish, substrate materials, and material hardness. As the introduction to the couse indicated, the advent of environmental extremes has stimulated research on solid film lubricants and this has resulted in increased emphasis in the areas of testing at high and low temperatures and extremely low pressures. It is possible, however, to point to many major programs for investigating all of the other factors. To completely define the effect of these factors here, however, is another matter. They vary from material to material and from time to time one finds materials where an increase in severity of a test condition may be beneficial rather than detrimental.

Later in this section we will briefly summarize the results of changes in these factors on lubricant performance from a general aspect with some illustrations. However, before discussing this I would like to turn to the type of testers employed as this also enters into the reults obtained. All these various considerations are interrelated. Knowing the type of data required, the environmental test conditions desired, and the type of program to be conducted one can best define the test device to use.

A. Simulative Devices

As previously stated, the simulative test device employs test specimens which can be any of a series of different geometries but which do not employ actual machine elements, such as bearings or gears, as the test specimen. There is a broad spectrum of simulative type test devices. A recent survey report by the ASLE (2) included over 100 individual test rigs reported in the literature from 1945 to the present. Although many of these rigs were of similar contact geometry each was an individually unique tester. Even though some of these rigs are only employed with more conventional lubricants there were by far a large majority which were also used on solid film lubricants. Table VI lists some of the more common solid film lubricant test rig contact geometries.

As can be seen from Table VI, there are many different test specimen configurations employed in simulative type test rigs. The resulting <u>initial</u> contact geometry, however, is either one of point, line, or area contact. The initial contact is stressed here for as the specimens wear there can be a change in the geometry with a tendency to go in the direction of area contact. Several contact combinations are also possible with one type of tests specimen. If one were to take two cylinders you could get area, line or point contact by arranging them as shown in Figure 3. In addition to the obvious effect on load, the contact configuration can affect other test conditions such as motion and temperature. Temperature can be affected by the type of contact and heat conduction paths for example. Two different combinations of the configuration of a rotating annulus rubbing on a disk moving at a different rotational speed can give widely different types of motion when oriented as shown in Figure 4. Without going into a complete analysis it becomes obvious that in sketch A the two rubbing surfaces are moving at a constant differential speed for the complete revolution while in sketch B the motion is cyclical and quite complex. It is quite essential in designing the test to be employed that full consideration be given to test geometry as well as environmental conditions. While this is particularly true with simulative type rigs it also holds for many of the component type rigs too. The relative sliding and rolling speeds in a 20 mm ball bearing and a 100 mm ball bearing are quite different.

Various factors enter the picture when selecting the specimen configuration. Cost is one factor. Rod materials are generally readily available for most metals and therefore cylindrical type specimens are less expensive than most others. Forming an hemispherical tip to the cylinder can add considerably to the cost. With some materials it is possible to mold the test specimen from the solid lubricant being tested - a pellet device uses this technique. The method of application of the solid lubricant influences the type of specimens and which surface should be coated. In some cases it may be desirable to coat both surfaces. Many of these factors will be covered when we discuss how data is obtained and used.

I would now like to briefly describe five of the more commonly used simulative devices. The test capabilities of the first three, which are commercially available units, are shown in Table VII.

<u>Falex Lubricant Test Machine - See Figures 5 and 6</u>

A 1/4 inch diameter rotating pin is clamped between two "V" shaped blocks 1/2 inch in diameter by 3/8 inch long. Initially four line contacts result but with wear you obtain area contact. Two type tests are generally run, one for wear with time and one which is a step wise load capacity test.

<u>Dual Rub Shoe Tester - See Figure 7</u>

There are many devices of this type but the Hohman A-6 shown in Figure 6 is probably the most common. It consists of two rub shoes 1/4 inch wide bearing against a 1.375 inch diameter rotating ring. Wear and friction of a solid film coating on the disk is measured. Line contact develops into area contact with wear of the rub shoes.

Single Rub Shoe Tester - See Figure 8 and 9

Again there are many versions of this device. The most commonly used is the Alpha Molykote Model LFW-I as shown. It consists of a .250 inch wide rub shoe bearing against a rotating disk 1.375 inches in diameter. As with the dual rub shoe machine line contact goes to area contact. The film may be coated on the disk.

Pin on Disk

There are many types of devices that fall in this category. In most cases they consist of a hemispherical rider loaded against a rotating flat disk. In some cases the hemispherical rider is replaced with the flat end of a small cylinder or by a collet holding a ball, in others the rider is pressed against the curved portion of the disk. Since there are a wide number of versions no one is specifically shown. The solid lubricant being evaluated can be applied to either surface. Although there is generally only one pin there are cases where multiple pins are employed.

Pellet Device

Again there are a large number of these type devices. The most common consist of compressed pellets of solid lubricants in the form of cylinders rubbing against a flat disk of varying types. The pellet can be wearing on either its curved or flat surface. Multiple specimens are more common than single test specimen units. They are mainly used in evaluating the solid lubricant pigment but can be employed with fully formulated and compacted materials.

B. Component Test Devices

The component test devices are those that employ actual machine elements as the test specimen. In some cases the component might even be a fully assembled piece of equipment but this type will not be covered in this paper. Most of the devices are built to simulate given applications or provide life data in a given element rather than provide data, such as friction coefficients, which can be compared to another rig as you might do with a simulative device. The test element performance thus becomes the prime criteria or yardstick. Because of this we will not cover individual test units in this paper but rather discuss the topic in terms of overall testing concepts.

A wide variety of machine elements have been employed as test sepcimens for components test devices. These include ball bearings, roller bearings, plain spherical bearings, journal bearings, screw actuators, ball screw actuators, and gears. This list is not complete but does show the broad spectrum of test specimens employed. They range from predominantly rolling, through combined rolling and sliding, to pure sliding contacts. Not only are there more types of bearings used as test specimens than other types of components but there are in most cases a larger number of bearing rigs within each type than there are rigs using other element types.

In general, there is very little, if any, standarization on component test devices. This is a result of the fact that most component test rigs are unique to one organization and not duplicated by any other facility. Unlike simulative rigs, such as the Falex Machine, you do not see wide spread use of these units. One possible exception to this would be the bearing test rig shown in Figure 10. This is a unit made by the Pope Machinery Company for grease testing which has been employed by at least three different organizations in the evaluation of solid lubricants. Because of the lack of standardization there is a minimum of component testing by people engaged in solid film development. Unlike greases, where the Coordinating Research Council's L-45 technique is widely used, there is no standard bearing test for solid films. As a result, most component testing is done by design or application engineers with one or two specific end uses in mind when setting the test conditions. This results in a considerable amount of duplicate or repetitive testing and a plea by most design engineers for data on solid films in components. A recent survy made by a member of our organization pointed this out as a critical problem to the use of solid films.

As has already been indicated, bearing rigs are the most common type of component test device. For this reason, we will discuss component test concepts in light of bearing experience. While other test element types may give somewhat different experience most of the factors should be the same. Bearing rig experience, itself, can vary quite a bit from rig to rig.

In addition to the obvious problem of lack of correlation from rig to rig because of non standardization there is also the problem of being faced with using a bearing designed for operation with greases and/or oils. Therefore, if you wish to evaluate a solid film lubricant for ball bearing use you must consider redesign of the bearing test specimen itself. One approach used by the Boeing Company (3) is shown in Figure 11. Here a lubricant compact was made into the cage of the bearing and provides lubrication by means of a transfer film formed on the contacting surfaces. Other approaches to bearing modification for solid film lubrication have also proved successful. Prior to such attempts, however, initial evaluation testing was unsuccessful and tended to leave the general feeling that solid film lubricants were not satisfactory with rolling element bearings. Another problem that we have encountered in our own laboratory involves the selection of the proper wearing surface to lubricate even in bearings designed for solid film lubricants. Figure 12 shows a bearing which we employ in one test and lubricate on the spherical surface. With one school of thought this practice is acceptable. With another, however, this is considered as the self-aligning portion of the bearing and in use the wearing surface should be the inner bore of the bearing where it attaches to the shaft. Obviously those with this school of thought would not lend much weight to our results. Finally, one other major point remains in specimen selection and that is the factor of bearing material. Figure 2 has already demonstrated the effect of substrate materials on lubricant adhesion. This can be expected to also affect lubricant performance. It will suffice to state that you cannot always get bearings made of the optimum material for the solid film. This is particularly true if you are interested in high temperature performance or if you do not have unlimited funds for their purchase.

Criteria of evaluation of a solid film in a bearing are also a factor which must be given serious thought. Various failure points have been used. One common approach is film and/or bearing final failure. Another is to use torque input into the rig as the means of setting a failure point. Another approach is temperature rise due to frictional heating. And yet another is the coast down time of the bearing when power is removed. These serve to illustrate the problem of correlating data from one test to another test employing a different set of failure detection criteria. Some films have high friction to start with an might fail rapidly in coast down or torque input measurementsbut run for a long period of time under the condition of film rupture and bearing failure. In selecting component rig test failure points you must keep the application very closely in mind or the results will not have much meaning.

The degree of sophistication of the test rig is another factor that can vary. Figure 13 is a simple schematic of a vacuum bearing test rig in which failure is measured by coast down time. This probably is one extreme simplification of a rig. On the other end of the spectrum are full scale bearing rigs (4) which can take full loads for plain bearings expected to operate in aircraft and are instrumented to measure many significant parameters. Such rigs probably cost in the region of $100.000. Many other types of bearing rigs exist as shown in Table VIII. Obviously cost, data required, and time available for testing dictate the degree of sophistication and type rig used in bearing studies.

C. Acquisition and Utilization of Test Data

We have briefly touched on some of the factors that affect solid film lubricant test data as we discussed the operation of the equipment. We will now turn to a more detailed discussion including factors such as environmental effects, significance of test data, correlation of simulative devices and component devices with each other, repeatibility, reproducibility, and instrumentation.

There are many environmental factors which can affect solid film lubricants. In this paper we will include mechanical factors as well as the state of the surrounding atmosphere under the term of environmental factors. Some of these factors have an affect on all types of solid film lubricants while others only basically affect one or two types. Even though most of these effects have been discussed in other papers we will review them here from the point of considerations involved in properly assessing the effects. Table IX lists many of the most common factors and those that will be discussed in this paper.

Temperature is probably the most widely varied parameter in evaluation of solid film lubricants by mechanical test devices. Tests have been run from the cryogenic region to in excess of 2000°F. Based on the ASLE (2) survey it appears that the most widely used rigs have an upper limit of temperature in the region of 400° to 1200°F. In the case of cryogenic testing, you encounter specialized rigs designed for this one test condition but in many cases also having other temperature capabilities. In most cases, temperature extremes are detrimental to film performance. That is, cryogenic temperatures or elevated temperatures generally reduce wear life. This is not always the case and I would like to just mention one film to illustrate this point. The film, a $PbS-B_2O_3$ composition, has given optimum results at 1000°F over its performance at lower temperatures. This was due to softening of the film in the 1000°F region. Thus one can see that care must be taken in setting temperature levels for testing and it is best to select more than one level. The design of the test rig can influence the actual temperature also. Generation of heat due to friction can influence the temperature at the contacting surface. Although this is an obvious problem at cryogenic levels it can also affect results in tests run at 1000°F. Temperature can influence other factors such as metal substrate hardness and, as we will see, chemical reactivity.

Gaseous composition is a factor affecting film performance life. Oxidation factors are probably the first that come to mind. We have seen how oxidation of MoS_2 adversely affects its performance. Temperature obviously is involved in such an affect since the rate of oxidation would be increased with an increase in temperature. Humidity has been shown to affectresin bonded solid films and must be taken into account when testing this and possibly other types of films. In the case of insitu formed films the composition of the reacting gas is the major factor in obtaining a workable film.

Gaseous pressure effects have been mainly evaluated in the lower than atmospheric region, i.e. under vacuum conditions. Much of this work either directly or indirectly has been stimulated by the space program. In the first case there is a great interst in materials for operation in vacuum. In the indirect sense the development of better vacuum pumping systems has led many researchers to employ vacuum environments for controlling the atmosphere and surface composition. Like temperature, vacuum generally has an adverse effect on film life but at least in one case, that of MoS_2 films, this does not hold. Pressure levels of 10^{-9} torr are a quite common level for thos engage in vacuum studies with 10^{-6} torr almost a routine everyday occurrence. Until about 10 years ago this was not the case and a rig with a 10^{-6} torr capability was a rarity.

Load and speed, for the most part, adversely affect solid film wear performance. Some materials such as the plastics have well documented PV limits. This PV limit is a product of the load, in pounds per square inch, and velocity, in feet per minute, and should not be exceeded in operation. Polytetrafluoroethylene has a limit of about 1000 for most applications. Load and speed effects then are important parameters if a film is to be evaluated for more than one set of operational test conditions.

The type of motion and contact have considrable effect on wear life of a solid film. Motion can either be unidirectional or oscillatory. The contact can be rolling, sliding, point, line, and area with combinations of the first two and last three. All of these factors are found in simulative and/or component test rigs. The major problem of interpreting test results is probably due to disregarding these factors in translating from one rig to another or to an actual application. Less consideration is generally given here than in any other factor. It is also worth noting that contact geometry and even motion to some extent can affect loads and temperatures. Some of this has already been discussed.

Radiation has less of an effect on solid films than on conventional oils and greases. Resin bonded materials and plastics are stable in the region of 10^9 to 10^{12} ergs/gm C while oils are limited to 10^6 to 10^8 ergs/gm C. Ceramic bonded materials would be expected to be even more resistant. In evaluating for radiation effects two procedures have been used. One is to first statically irradiate the test specimen and then run the test on the fully irradiated film. The other is to conduct the test during irradiation giving it a combined environment of radiation and whatever other test conditions are of interest. There has not been sufficient work to say which is the most severe condition. One problem with radiating during testing is that if the sample does not run long enough you do not accumulate a very high dosage. Of course, if you statically irradiate and then test you may not get the thermal stresses coupled with the irradiation.

The final point that must be considered in any environmental testing is interaction of various test conditions. Some of these have already been pointed out particularly where one condition aggrevates another. There are also cases where interactions can reverse trends. Figure 14 shows such an effect. Here we see the case for one ceramic bonded solid film. The small letters of s and t denote low levels of sliding speed and temperature while the capital letters denote higher levels of these test conditions. Thus, there is a reversal of wear life trend due to temperature for the two speed levels. Little work has been doen in statistical evaluation of such interactions but one such approach has been reported by Lavik (15) on resin bonded films.

The significance of data obtained from mechanical tests is of utmost concern to the person doing the testing. In general one wants to know "what is the data telling me". In many cases this question is asked in terms of the use of a film in an actual application while in others it is in regard to comparison of one film to another. There are no concrete guidelines or numbers that can be placed on significance of data. Because of this, different people have widely differing opinions of the interpretation of test results. These can range from the opinion that a given unit produces data of no significance to the opinion that it provides data which can be fully relied on to guide development efforts.

There are many factors that enter into the significance of the data from a mechanical unit. The significance of data from any mechanical test device can be improved if one studies the unit and test conditions so that you know what you are actually testing. In too many cases tests are run without the person actually knowing what is being evaluated. That is - what is the critical test condition. This is more true when employing simulative devices. There are cases where people have attempted to develop lubricants for ball bearings with nothing more than a simple block on rub shoe type tester. In addition to the factor of geometry, you must also know whether a device is properly evaluating such factors as environmental effects before you can place significance on the test results. For example, if an actual end use or film is particularly sensitive to a given speed and motion and you evaluate it in a device similar to the one shown in sketch B or Figure 4 it is questionable that you would obtain significant results. Also if you want to know what temperature a film will fail at and you use test specimens which soften below the films limit you do not have a valid test.

Of course, the closer a test device simulates the actual intended use the more significance can be placed on its data for the purpose of correlation and/or guiding development. If one is to develop a lubricant for a gear the most significant data probably would be that from an actual gear. Lacking this, then if one knew that the gear were critical from combined stresses of rolling and sliding then data on a lubricant coated on two contacting cylinders rotating in the same direction but at opposite speeds might be of significant value. The closer the rolling and sliding speeds approached the gear conditions the better it should be.

Significance is closely related to correlation of different types of equipment. If two different devices are evaluating the same parameter then one can expect that there is a good possibility of the results from the two rigs providing similar results. However, if different parameters are affecting the results then no correlation will result.

In this paper we will review the general topic of correlation of test devices to each other. Most of what is said in this regard also applies to actual correlation with applications. Good correlation is basically dependent upon obtaining data under as well documented and controlled conditions as possible with the same critical parameter being evaluated in the units you wish to correlate. The correlation of data even under identical conditions, however, often is quite a major task.

Correlation between two rigs would appear to be quite simple. Without care taken in running tests, however, you can not always be certain. Table X shows results from our laboratory and one other laboratory on one film composition in two identical dual rub shoe machines. In this series of tests we were attempting to rate the optimum curing cycle. Although in some cases the AFML friction cut off was higher than the other unit they in all cases gave lower results. Even accepting the different levels one encounters the fact that the order of the films is also different in the two rigs. This then shows that two identical rigs do not always correlate but it should be noted that no particular effort was taken to standardize and calibrate between rigs for this series of runs. We will see data in a following section on repeatability and reproducibility which shows what can be done with care being taken.

Another example I would like to present involves the results of testing on three different films developed using three different types of test rigs. The three films were developed by Midwest Research Institute, NASA, and the Navy Aeronautical Materials Laboratory. The first was developed using a dual rub shoe machine, the second with a pin and disk machine, and the final with various devices including ball bearing tests and a Falex test. These three films were compared in a dual rub shoe machine on a program concerned with radiation effects. (16) They were coated on Inconel-X substrate disks and Rex AAA rub shoes were employed. Table XI gives the conditions of test. Film A was basically developed for use at 1000°F, Film B for use at 1200°F and higher, and Film C for use in the region of room temperature to 750°F. As can be seen, even though the films were developed on different types of devices they do appear to check out at similar temperature ranges in the dual rub shoe tester. Thus it can be seen that films developed using one type rig can operate on another rig in a satisfactory manner. In this case the three films had all been subjected to sliding test in their development cycle.

While we have cited two cases of rig correlation or lack thereof they have both been somewhat the opposite of normal expectations. This has been done purposely to point out that you do not always get what you expect in rig correlations. The analysis of what is being evaluated, however, will improve your predictions.

When correlating simulative rigs with component rigs you can have a more complex problem than between two simulative rigs. In a ball bearing you have rolling and sliding contacts and therefore to get correlation you should have simulative rig data of each type to obtain a good chance of getting correlation. Of course this would apply also to actual applications only to a much more extent as there you could encounter bearings, gears and other wearing components.

There has been only a minimum effort in studying repeatability and reproducibility of mechanical tester evaluation data. What has been done is mainly in the area of repeatability studies.

One organization (17) quotes repeatability of ± 1% on runs from a dual rub shoe machine for one particular experimental film at 200 rpm. The sample was from one spray batch and gave results in the order of 60,000 load cycles. This would give a spread of ± 600 cycles. They state however, and this author agrees, that such repeatability is almost unbelievable. The same organization also quotes ± 15% repeatability for the Falex Machine, variations as high as 10-1 on pellet and gear testers with 3-1 being more typical.

The standard deviations shown in Table XI are in the order of 25 to 50% of the average with the exception of the 1500°F film which may be affected by rub shoe softening rather than film variations.

The CRC (18) has conducted an extensive survey on the Falex and various single rub shoe devices (two basic types). Eighteen laboratories participated with fourteen running Falex tests and eleven running single rub shoe tests. Unfortunately a full statistical analysis of the results is not available although the work tends to point out that the Falex is more repeatable than the single rub shoe devices.

In the ASLE Survey (2) various comments were given on repeatability and reproducibility. One user quotes ± 10% reproducibility for the Alpha LFW-1 (a single rub shoe tester). A user of the Hohman A-3 quotes 10-15%

repeatability while another on the Hohman A-6 states reproducibility is a function of test materials. Both are dual rub shoe machines. In general, however, little attention is given to this area.

Control of test conditions, calibration of instrumentation, and standard film application are all factors that must be well checked to maintain good repeatability and reproducibility. In order to obtain sound data multiple testing is required for each point unless a statistical safeguard is built in by a series of tests over varying conditions which can be used as checks on each other.

It is impossible to cover the full scope of instrumentation within this paper. Instrumentation, however, plays an important role in obtaining test data. The proper control of test parameters is often the function of proper instrumentation. One point that was obvious in the ASLE survey (2) was that instrumentation and equipment descriptions were often inadequate in papers on lubricant evaluation so that it becomes difficult for one to make his own assessment of the data by independent analysis. Exact location, sensitivity, degree of calibration,etc. are not always reported for the instrumentation.

II. Physical and Chemical Property Testing

Finally I would like to cover the area of physical and chemical bench tests of solid film lubricants. As mentioned previously these methods are generally of a go/no-go type. Most of these methods have not been standardized throughout the industry but there is activity in this area by the ASTM, ASLE, CRC and various governmental agencies. As we have seen several of the methods can be employed throughout the full development cycle. The following is a description of some of these test methods:

A. Adhesion Properties: The adhesion of the film to the substrate or wearing surface is of utmost importance in the performance of a film.

Various techniques are employed to study this property. One, as described in Table II, involves the use of a so-called tape technique. In this method adhesive tape is pressed on a film and then removed. The amount of film removed or surface change is noted. This type test is currently under consideration by ASTM for standardization and has been standardized by the Government. It finds its main use with resin bonded films. Corelation with adhesion under wearing conditions probably would not be very good and thus the test has limited use. Another method employed is to move a scribe over the surface of the film and observe the behavior of the film under such working. Usually a hemispherical tipped rider is employed in such a test. Again one can only measure gross effects. Simple observation of a film under a microscope can often indicate lack of adhesion of a film by cracking or other surface defects. This can be seen from the photographs of Figure 2. There are no adhesion tests which will directly predict if a film will adhere to a surface when subjected to sliding except actual wear where one is also investigating other phenomena. Adhesion tests are frequently used to assess the results of some of the other tests we will discuss below.

B. Thermal Properties: The behavior of the constituents of a solid film when subjected to extremes of temperature is of considerable interest in predicting film performance. The tape type test described in Table II combines a study of thermal shock and possible oxidation effects. As such the classification as a thermal resistance test is partially a misnomer if one used the concept of only thermal as opposed to combined thermal and oxidative behavior. The factors involved in this test include assessment in terms of loss of adhesion. Modifications of such a test to other conditions would be simple to perform. Within the laboratory other methods are employed including DTA and TGA. In such techniques pure thermal behavior can be measured if oxygen is excluded and an inert or vacuum environment maintained. Because a film generally is expected to operate over a wide temperature range it is often wise to evaluate its behavior at low temperatures as well as at high temperatures. One area where this would be critical is in thermal expansion of the films.. If the film does not match the base metal fairly well, loss of adhesion will result. Simple observation of the surface often is used to detect this. In the research laboratory the thermal properties of individual constituents have as much value in film formulation as most other properties.

C. Oxidation Properties: The result of chemical degradation or change of the film constituents due to rapid oxidation at elevated temperatures is, in most cases, detrimental to solid film performance. A simple method for evaluation of this is to heat the film or its individual constituents in air and observe the change in weight, appearance, or chemical composition. In many cases this type information can be found in chemical handbooks.

D. Fluid Compatibility Effects: The resistance of a film to degradation when subjected to a test as indicated in Table II is again a go/no-go type test. Although most solid films are not recommended for use in conjunction with other lubricants they often are contaminated in actual service by contact with such materials. It is necessary therefore to know what the effect of such a contact will be. Another type of fluid interaction of interest is that of solubility. In spraying films it is necessary to know what potential solvents can be employed, in other cases it is necessary to know if the final film might be soluble in various fluids. In the case of a ceramic film, for example, it is important that the final cured film not be soluble in water if it is to be used in environments with high humidity, especially where it is subjected to rain.

E. Other Properties: Surface wetting is one property that has been studied in development of ceramic bonded films. In one program (19) the investigator studied wetting of both the substrate and pigment with the binder in a combustion tube furnace where he was able to photograph contact angles as the temperature was raised. Both air, inert gases, and vacuum were employed for surrounding gases. Melting points of the constituent materials, as measured by conventional means, must be employed if the material is marginal at the use temperature. Phase diagrams and associated studies (20) have been employed to explain film performance of ceramic films and can also be used in selection of ceramics for binders. One reference of aid in evaluating this would be the compilation of phase diagrams by Levin, McMurdie and Hall (21). Film thickness is often measured to ensure proper coating thickness since this has an affect on performance of some films. One common technque employed here is the use of a Magna Gauge where reduction in magnetic attraction is correlated to film thickness. Of course in such a technique one must have a substrate with magnetic properties and a film which is non magnetic in nature. Another technique is in using a simple micrometer but here one must be careful not to damage the film. The type designed for measuring thickness of paper would be useful. Crystal structure of the lubricating pigment is often of interest in assessing the potential of new pigments. Standard X-ray diffraction techniques are used here and often the material may have known crystal structures as reported in physical or chemical handbooks. Corrosion properties are often of interest and the method described in Table II is the only standardized one. It was basically developed for resin bonded films.

Knowing the above factors enables you to better explain the behavior of fully formulated films when run in mechanical test devices. In most cases this also allows one to rapidly adjust film composition to improve deficiencies. In other cases it can quickly explain results. The oxidation of MoS_2 in the region of 750°F explains poor behavior of this type film in air at this temperature level while it is still usuable in vacuum at much higher temperatures, for example. The chemical test data can also be used in service trouble shooting to explain failure modes. There will undoubtedly be more chemical and phsyical test methods developed as the field of solid films expands but at this time there is a long way to go before such testing will reach the level as employed in conventional lubricant development.

TABLE I

Some Physical and Chemical Test Methods for Oils and Greases

Viscosity

Flash Point

Fire Point

Oxidation Corrosion Test

Neutralization Number

S I T

Evaporation Test

Dropping Point

Deposition Rating

TABLE II

Some Solid Film Lubricant Test Method Descriptions

Thermal Resistance -

A steel panel having the solid film lubricant deposited on one surface is subjected to 500°F followed by immediate exposure to -65°F. The solid film is then examined for cracking, flaking, blistering, or other evidence of thermal instability.

Fluid Resistance -

Aluminum panels with the solid film coating is immersed half-way into various fluids for a period of 24 hours at 73.5°F. The panels are removed, cleaned and examined visually for evidence of softening, lifting, blistering, cracking or peeling.

Adhesion -

The dry solid film lubricant is applied to anodized aluminum panels and immersed in water for 24 hours and then wiped dry. A strip of masking tape is pressed onto the panel and removed abruptly. Film removal exposing the surface of the metal panel is the criteria for failure.

Corrosion Resistance -

An aluminum panel having a solid film deposited on one surface is contacted under load with the surface of an unlubricated panel. The specimen is subjected to 95 ± 3 percent relative humidity at 120°F for a period of 500 hours. After this period, the surface of the unlubricated panel is examined for evidence of corrosion.

TABLE III

Properties of Interest in Evaluating Pigments

Melting Point
Hardness
Solubility in H_2O and Organic Solvents
Crystal Structure
Oxidation Resistance
Thermal Stability
Friction and Wear

TABLE IV

Test Conditions of Interest

Atmospheric Composition

Atmospheric Pressure

Temperature

Load

Speed

Type of Motion

Radiation

TABLE V

Condensed Specification Requirements for a Resin Bonded Solid Film

Requirement	Type of Test Method
1. Film Appearance and Thickness	Visual in microscope and Magna Gauge.
2. Film Adhesion	Panel test as in Table II.
3. Thermal Stability	Panel test as in Table II.
4. Fluid Resistance	Panel test as in Table II.
5. Endurance Life	Falex test.
6. Load-Carrying Capacity	Falex test.
7. Corrosion Resistance	Panel test as in Table II.
8. Storage Stability	Store for six months, apply film and test by methods 2, 3, 4 and 5 above.

TABLE VI

Some Simulative Test Rigs

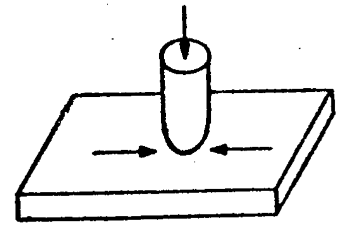
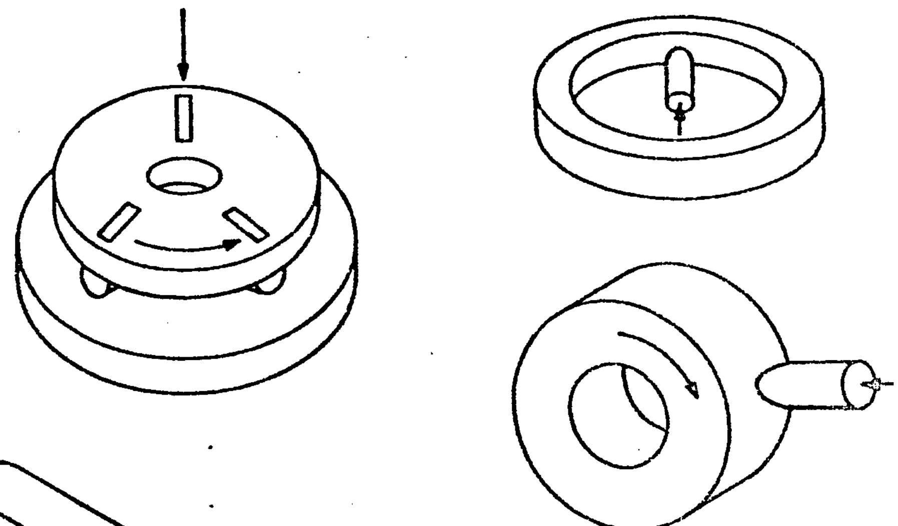
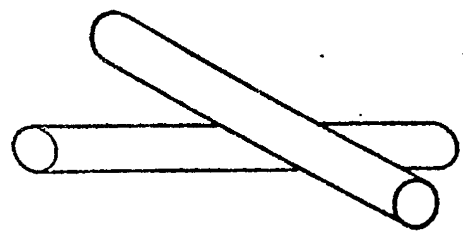
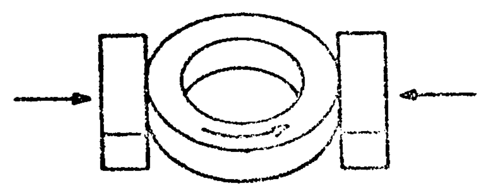
Classification	Typical Rig	Contact Geometry
Hemisphere on Flat	Bowden Leben various NASA Rigs	
Pin on Ring and Pellet Devices	Many research devices	
Crossed Cylinders	Lathe type	
Dual Rub Shoe	Hohman A-6	

TABLE VI - (Cont'd)

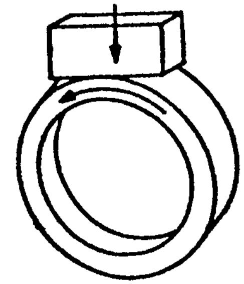
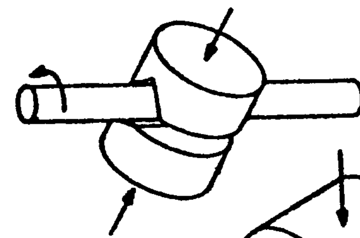
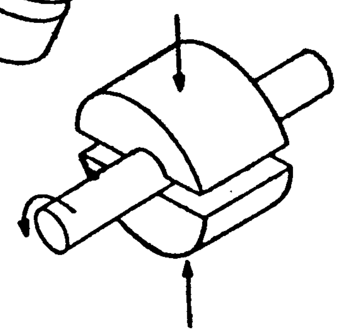
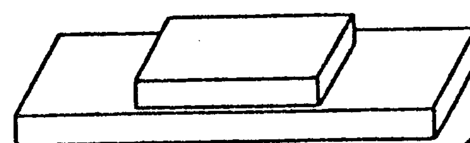
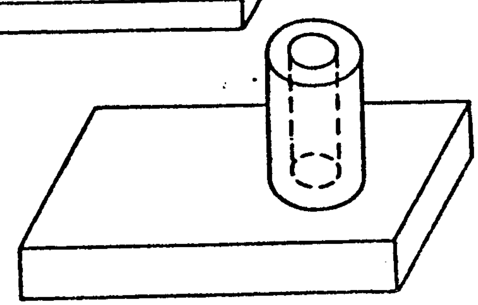
Classification	Typical Rig	Contact Geometry
Single Rub Shoe	Alpha LFW-1	
Nutcracker Loading on Rotating Cylinder	Falex	
	Almen	
Flat on Flat	Various research rigs	
Annulus on Flat	Various research rigs	

TABLE VII

Test Capabilities of Various Commercially Available Solid Film Lubricant Test Devices

	Temperature (°F)	Speed (RPM)	Load (Pounds)	Atmosphere
Falex	Ambient	290	To 4,500	Air
Hohman A-6	60-1500	60-3,000	0-800	Air and Inert
Alpha LFW-1	Ambient	0-200	30-630	Air

TABLE VIII

Some Bearing Component Tests Used for Evaluation of Solid Films

Type Bearing	Type Solid Lubricant	Temperature, °F	Ref.
204 ball	Ceramic-bonded	To 750	5
204 ball	Gas-entrained powders	To 1200	6
206 ball	Vapor-deposited	To 900	7
209 ball	Vapor-deposited	To 900	7
Instrument-sized ball	Soft metal	To 600	8
204 ball	Gas-entrained powders	To 1000	9
75-mm bore roller	Gas-entrained powders	To 1000	9
Plain spherical	Organic powders	To 1200	10
Plain spherical	Ceramic-bonded	To 750	11
Spherical	P.T.F.E.-fabric	Ambient	12
Plain	T.F.E.	550	13
Cylindrical	P.T.F.E.	Ambient	14

TABLE IX

Environmental Conditions Affecting Solid Films Lubricants

Temperature

Gaseous Composition

Gaseous Pressure

Load

Speed

Type Motion and Contact

Radiation

TABLE X

Wear Life in Air of a Solid Film as Obtained in two Dual Rub Shoe Machines

Film Cure Temp (°F)	Wear Life (revolutions) / μ, Steady State / μ, Cut Off — AFML Rig[1]	MRI Rig[2]
925	9,400/.045/.160	13,200/-/.15
960	12,000/.085/.155	15,600/-/.15
1010	8,400/.055/.190	13,200/-/.15
1105	*9,400/.07-.145/.185 (↖1/2 way point)	19,600/-/.15
1250	*9,200/.065→.14/.20 (↑slowly)	16,000/-/.15
1400	11,000/.05/.20	13,600/-/.15

Test Conditions

Apparatus - - - - - Disk-Rub Shoe
Load - - - - - - - 100 lbs.
Speed - - - - - - - 400 RPM
Temperature - - - - 400°F
Disk Material - - - M-10
Rub Shoe Material - Inconel-X

Films: $MoS_2:B_2O_3$, 12:1 by wt.
Approx. 1 mill thick.

*These films would probably have shut off the machine earlier if the cut off point had been 0.15. The wear lives of the other films would probably not have been affected.

1/ - Automatic cut off at 0.20 or by hand when loud noise accompanied sharp increase in μ.
2/ - Automatic cut off at 0.15

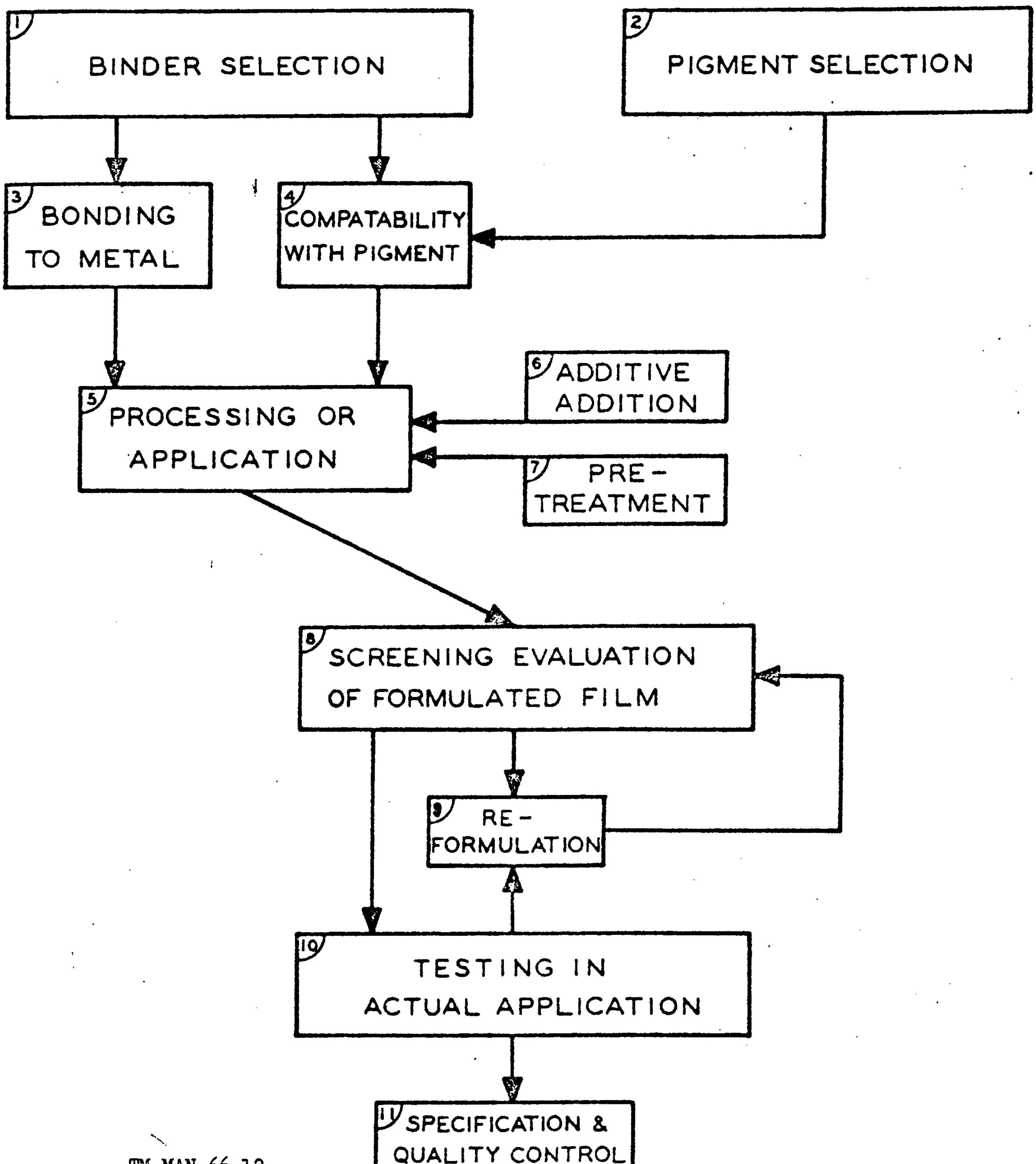
TABLE XI

Comparison of Three Films

Lubricant	Composition	Wear Life/Standard Deviation(±)*					
		80°F	600°F	900°F	1000°F	1200°F	1500°F
A(MRI)	$PbS+MoS_2+B_2O_3$	58,228 / 26,996	19,323 / 5,836		14,224 / 6,468		
B(NASA)	CaF_2+Oxide Frit			3,603 / 1,158		5,016 / 2,136	26,798 / 28,225
C(NAVY)	MoS_2+Graphite and Sodium Silicate	54,964 / 16,532	12,255 / 4,293			10,979 / 4,134	

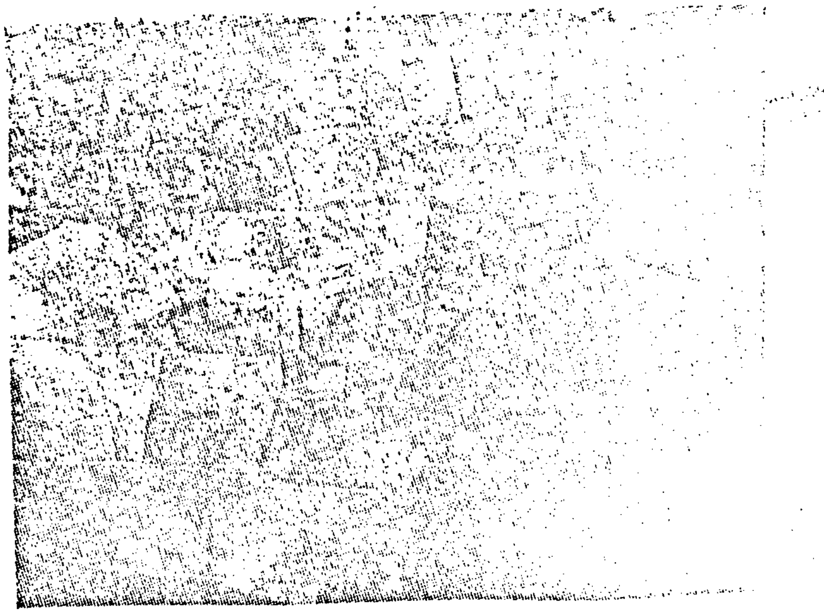
*Average of six runs.

DEVELOPMENT AND EVALUATION CYCLE FOR A BONDED SOLID FILM

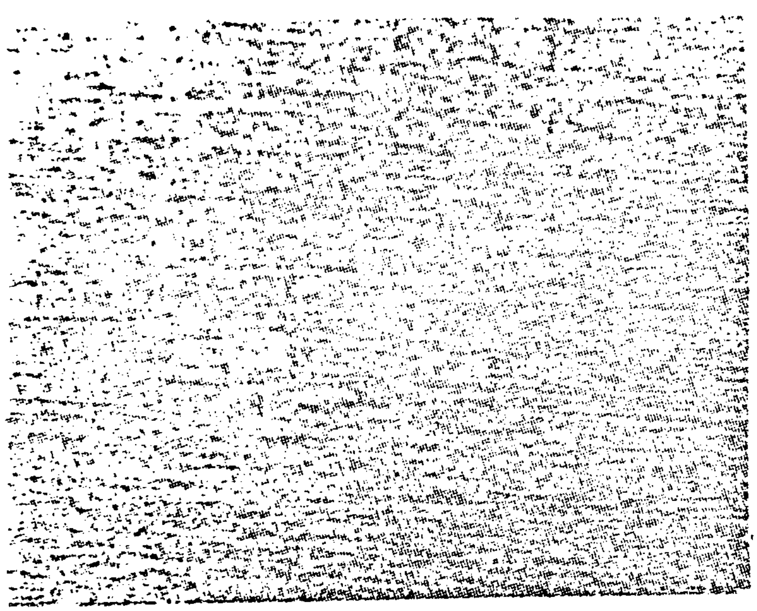


APPEARANCE OF A CERAMIC FILM*
AS BONDED TO VARIOUS SUBSTRATES

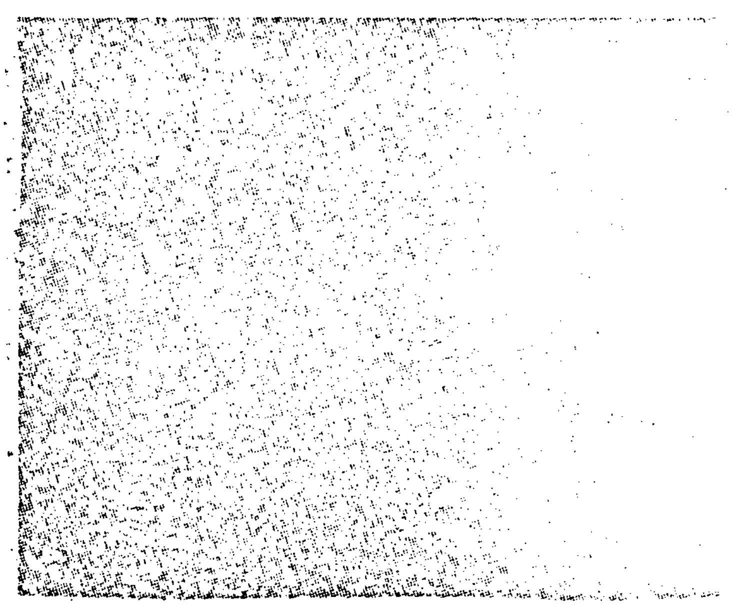
BONDED ON REX AAA



BONDED ON 440C



BONDED ON RENĒ 41



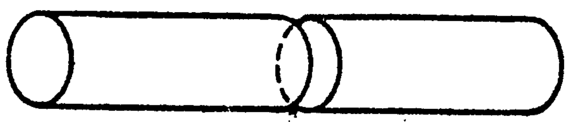
BONDED ON INCONEL X

TM-MAN-66-12

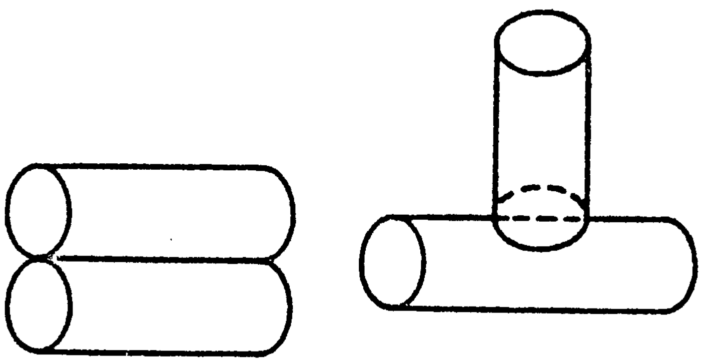
* NBS 332

FIG. 3

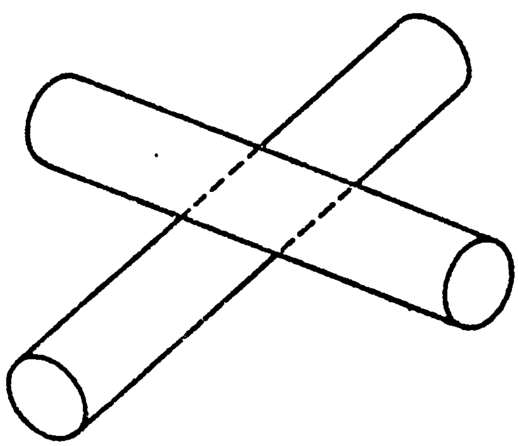
CONTACT GEOMETRY FOR TWO CYLINDERS



AREA CONTACT



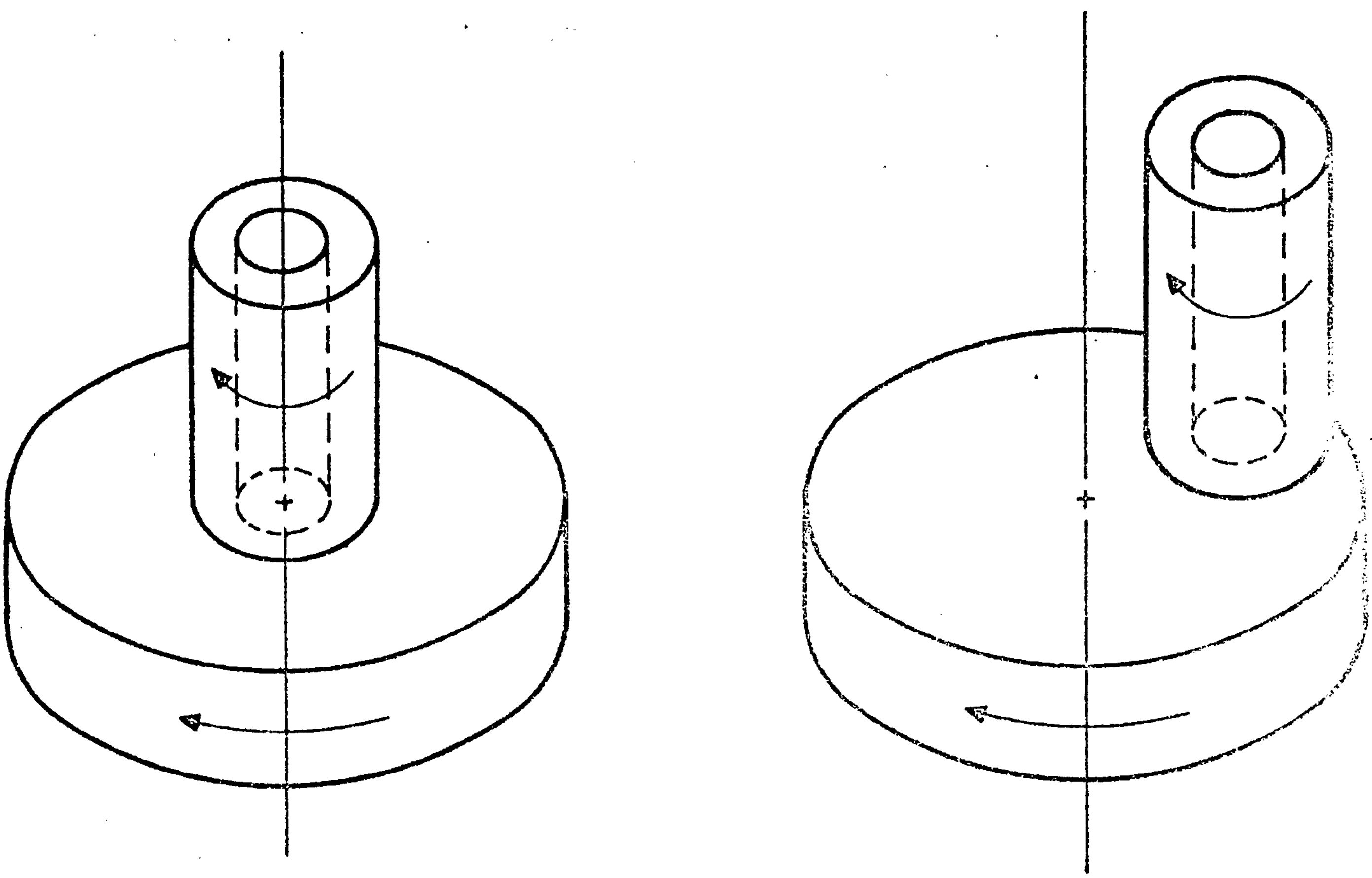
LINE CONTACT



POINT CONTACT

FIG. 4

TWO POSSIBLE ORIENTATIONS FOR AN ANNULUS RUBBING ON A FLAT DISK



SKETCH A SKETCH B

FIGURE 5

Falex Lubricant Test Machine

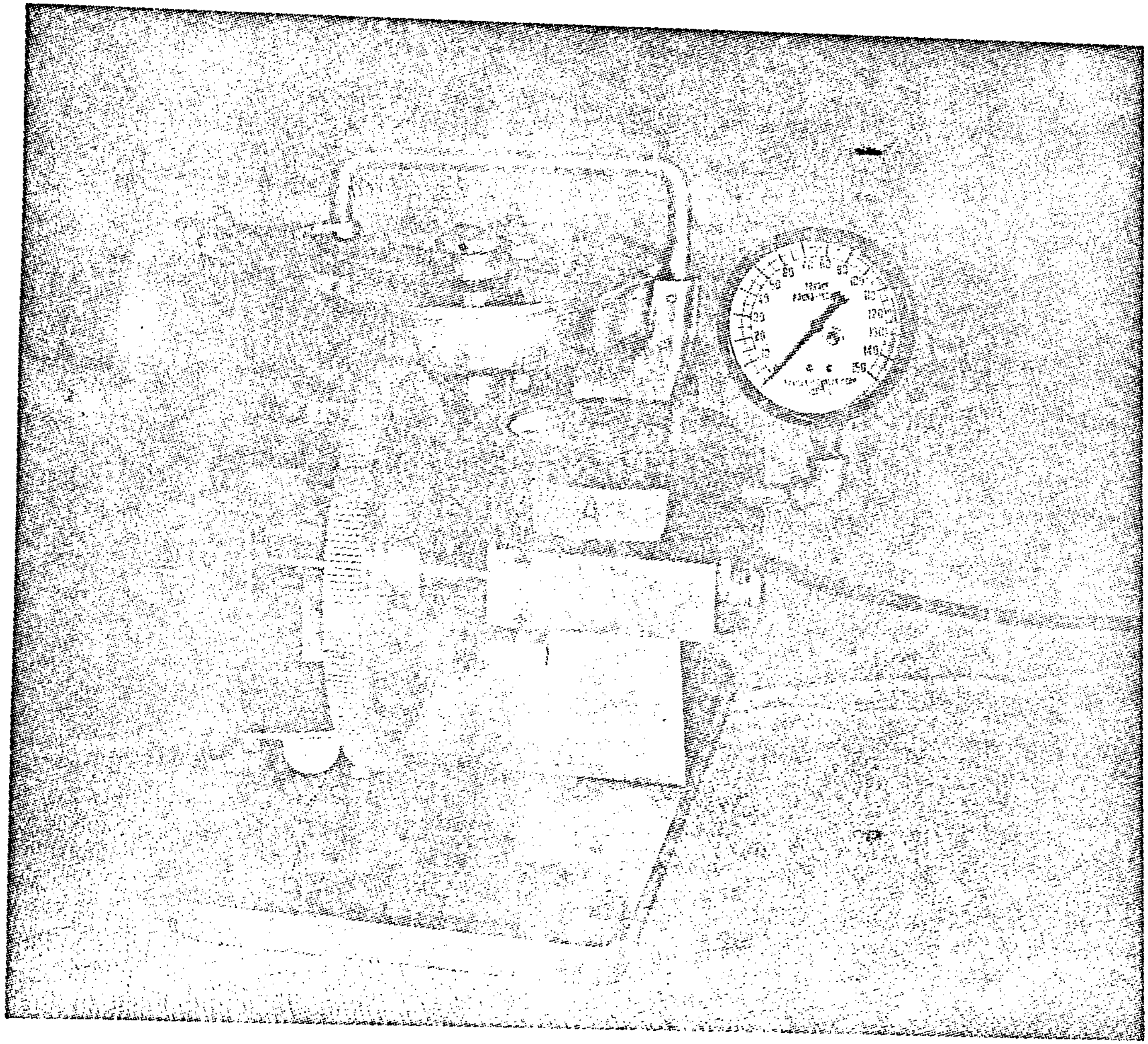
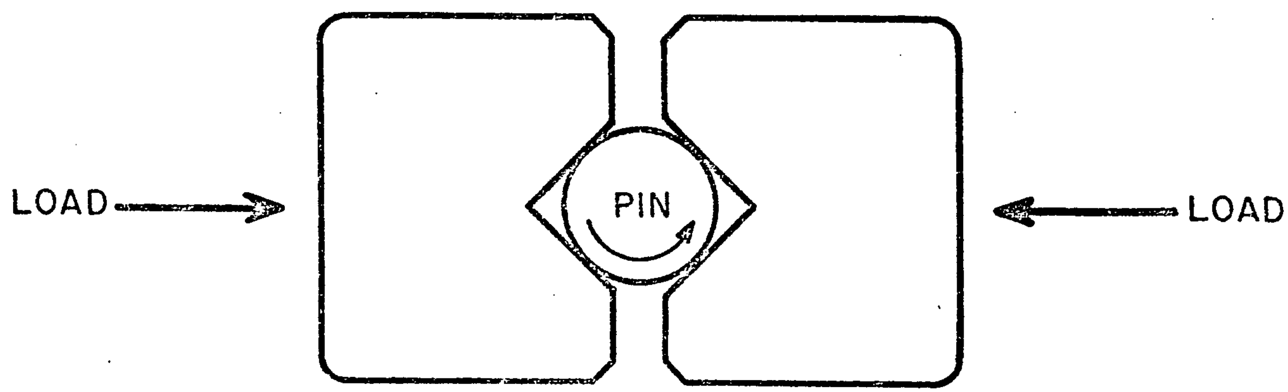
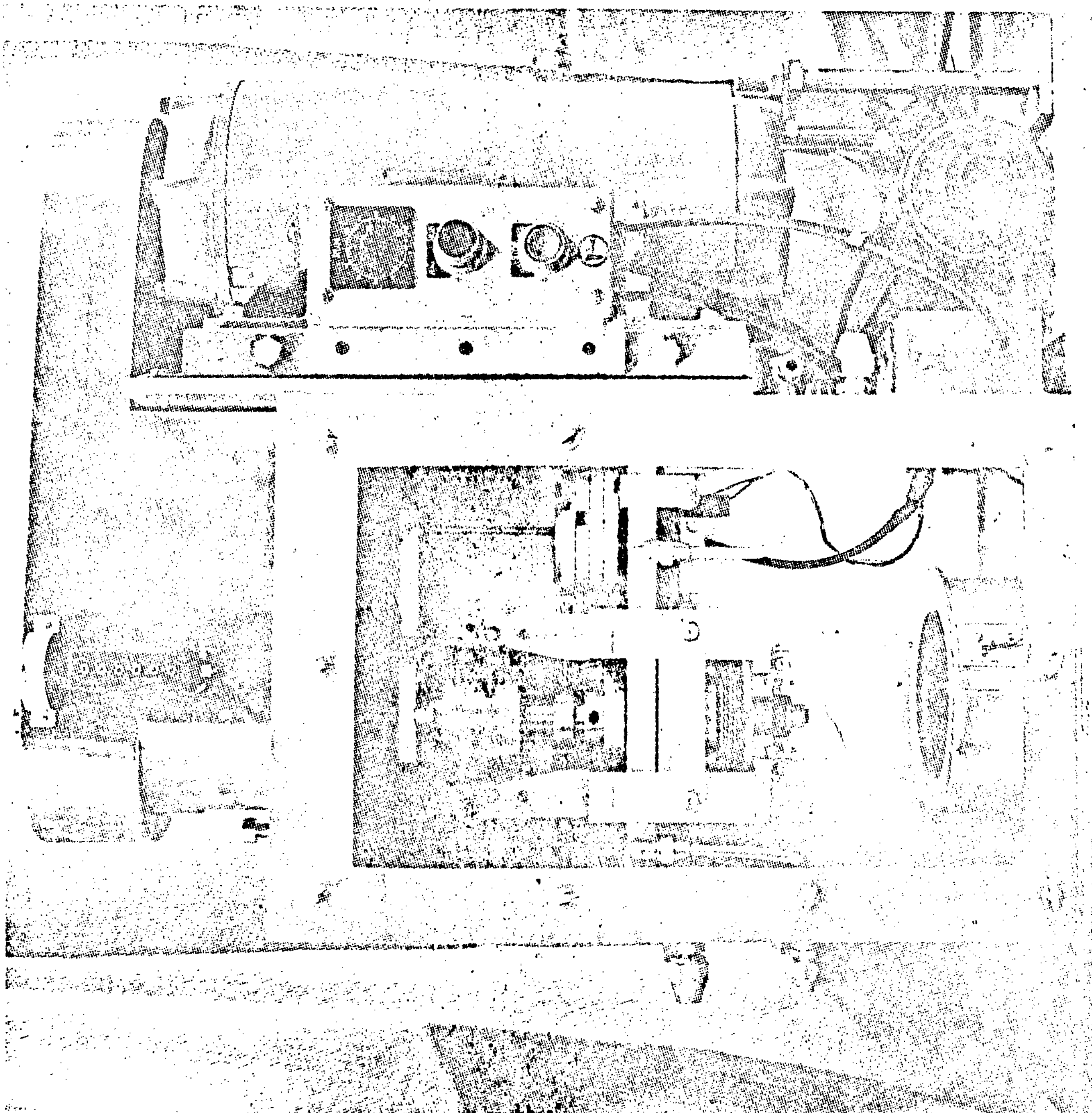


FIGURE 6

Principle of Loading and Operation Falex Lubricant Tester





Hohman A-6 Machine

FIGURE 8

Alpha LFW-1 Test Machine

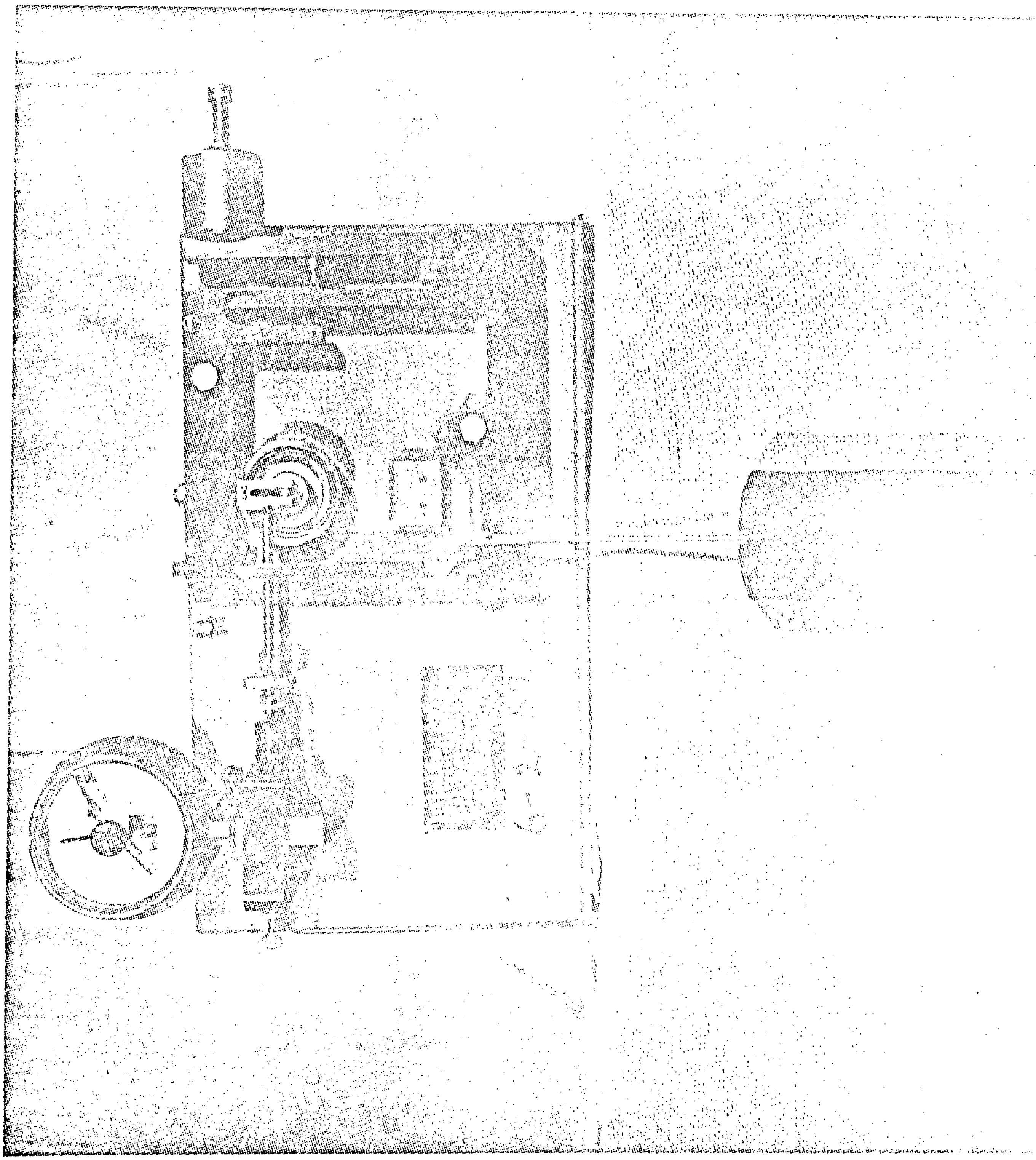


FIGURE 9

Principle of Loading and Operation of Alpha LFW-1

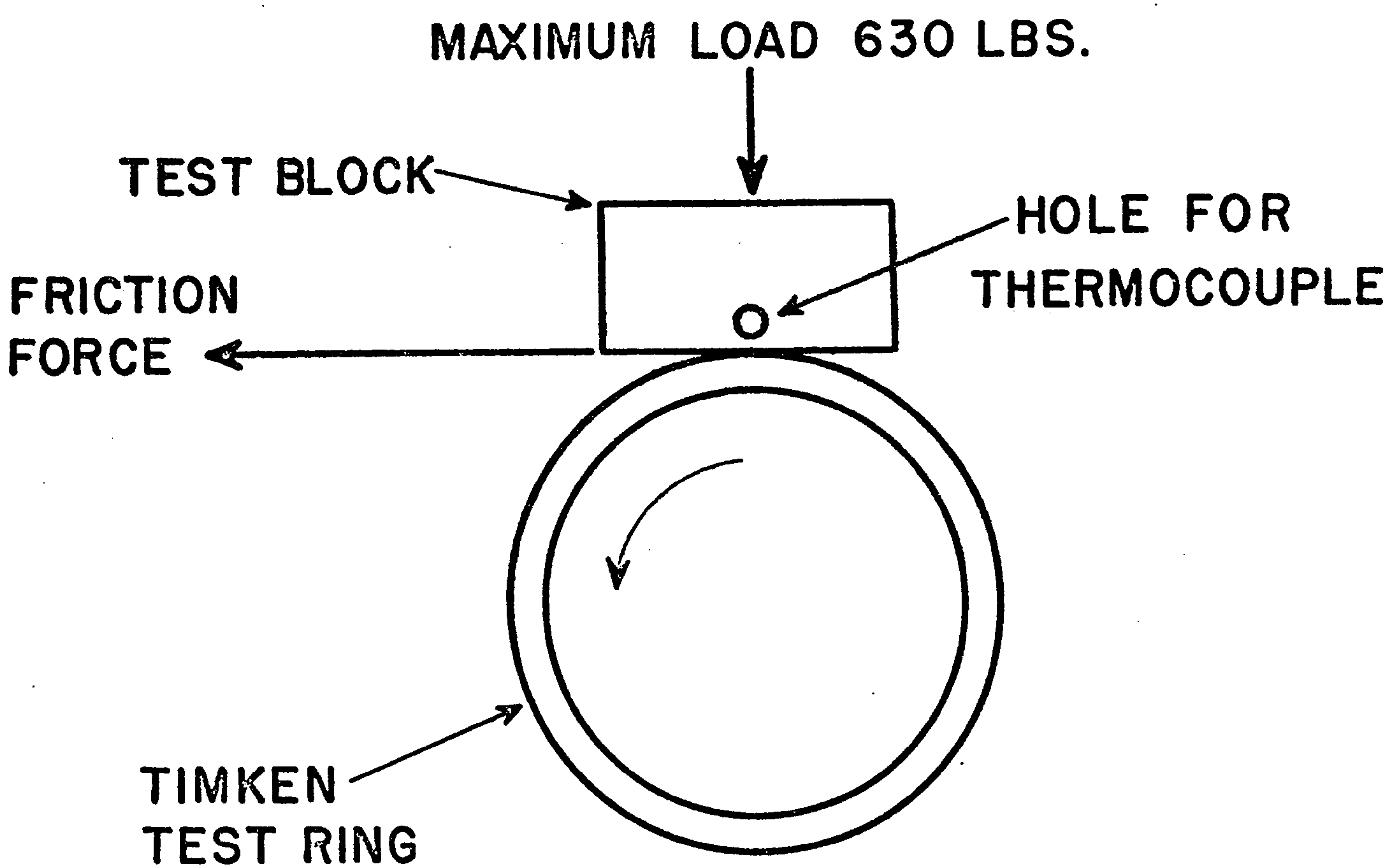


FIGURE 10

Bearing Test Rig

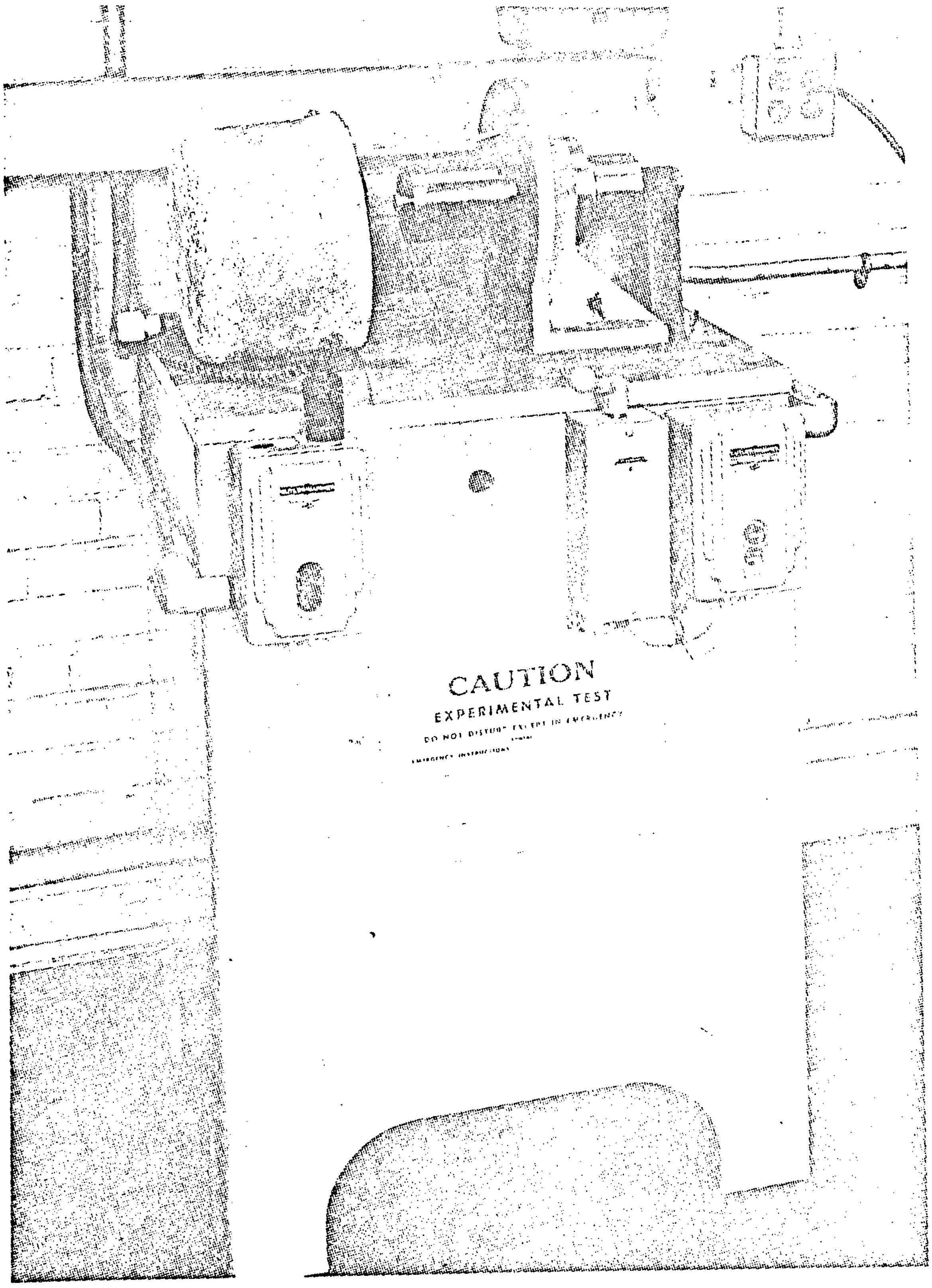


FIGURE 11

Ball Bearing Employing Lubricant Compact

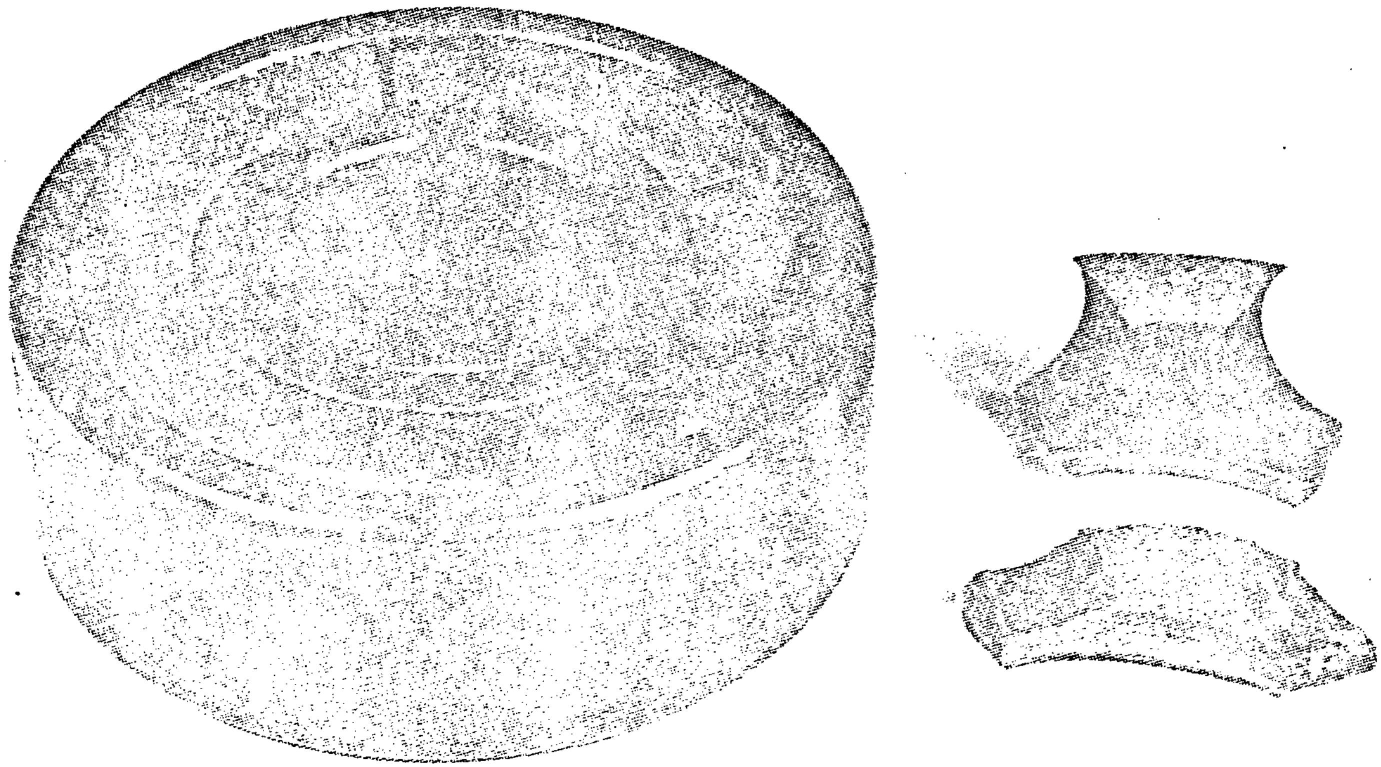


FIGURE 12

Plain Spherical Bearing

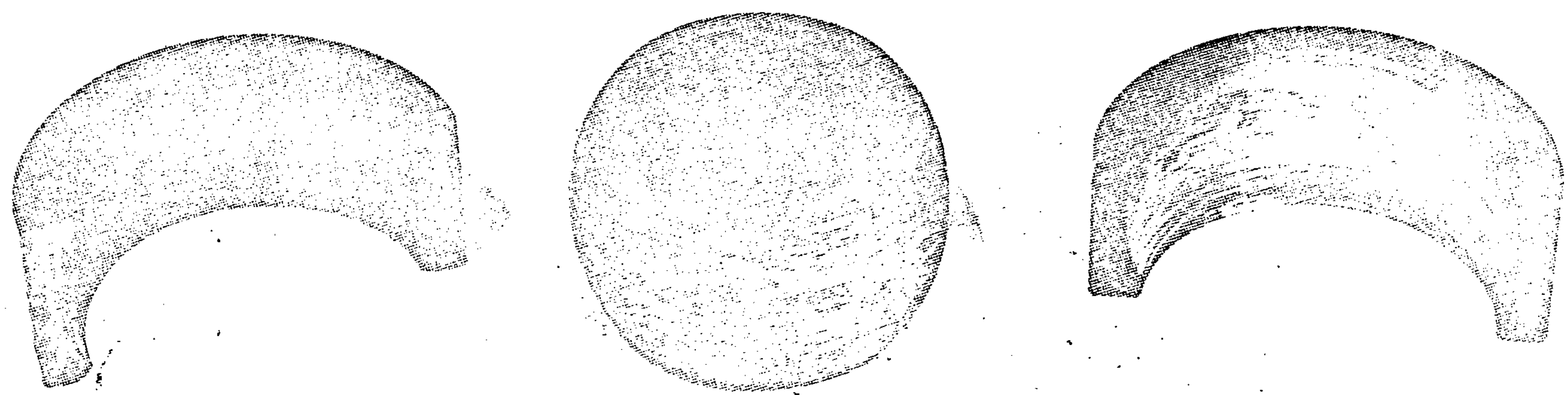


FIG. 13

ROLLING FRICTION EXPERIMENTAL APPARATUS

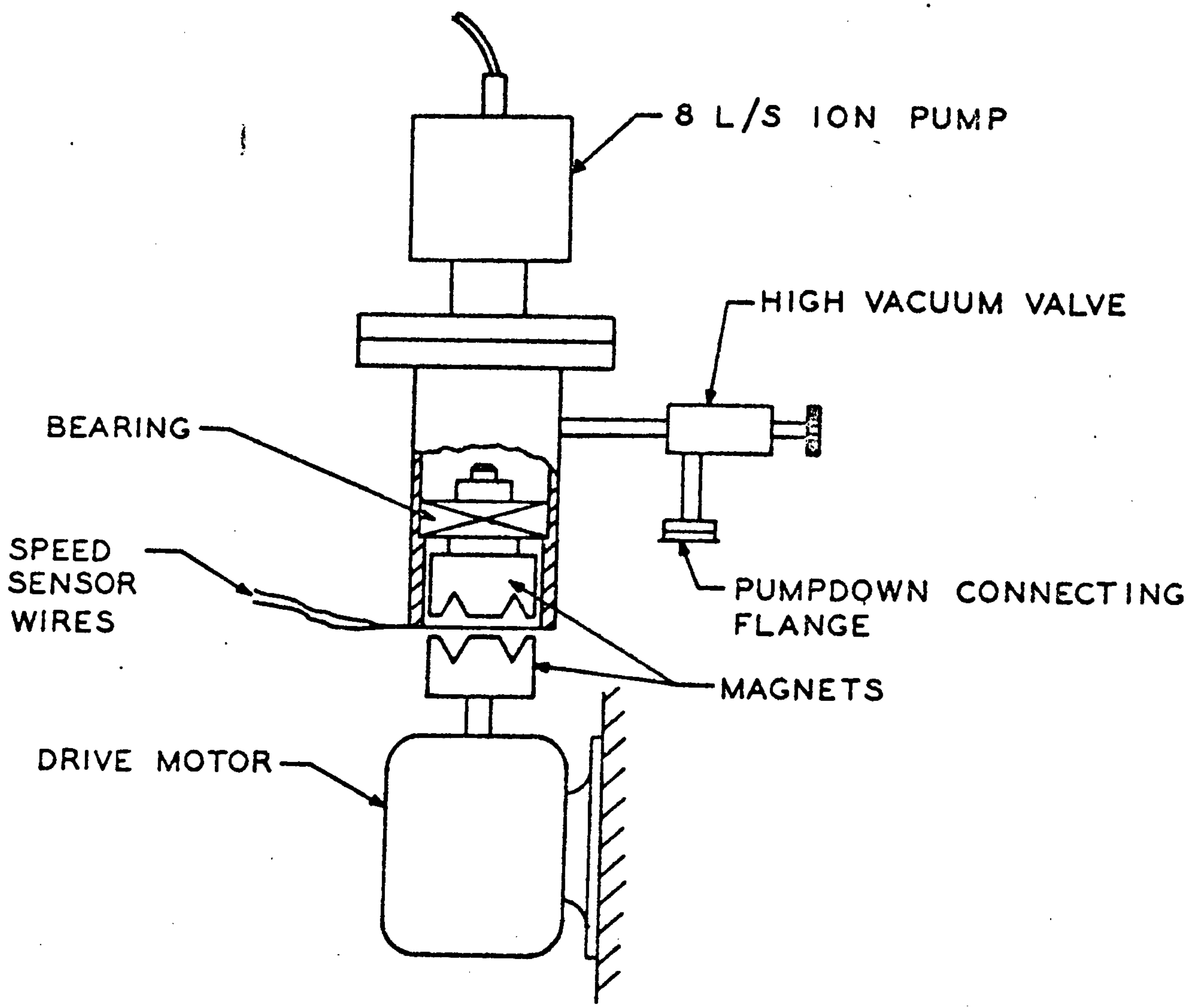
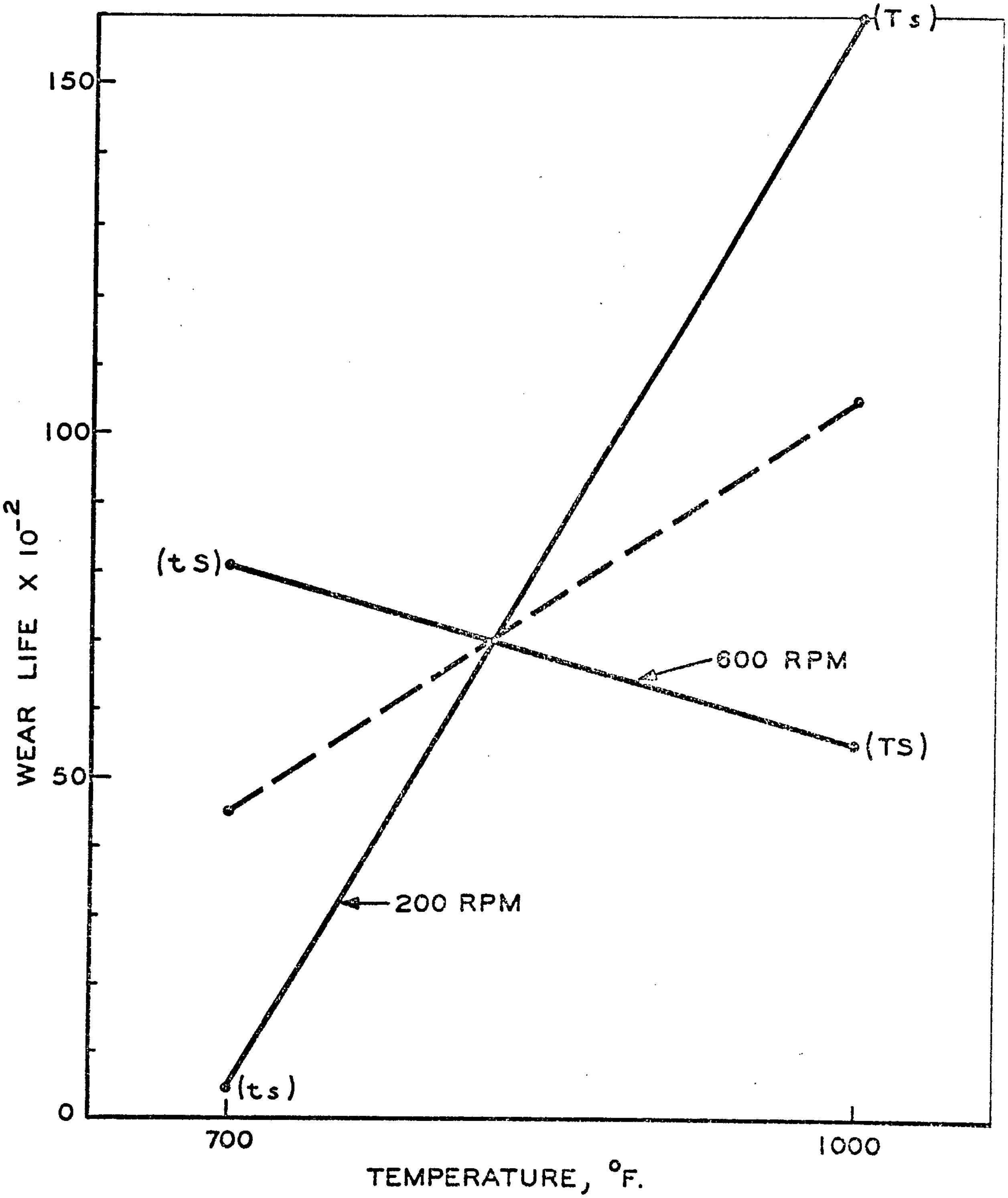


FIGURE 14

Main Effects and Interactions for Ceramic Bonded Films



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