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ELASTOMERIC SEALS AND MATERIALS AT CRYOGENIC TEMPERATURES

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Forward

This report was prepared by the National Bureau of Standards Cryogenic Engineering Laboratory under WADD order No. (33-616) 61-04. It was administered under the direction of the Nonmetallic Materials Laboratory, Materials Central, Elastomers Section, with Mr. R. E. Headrick acting as project engineer.

The present research is a continuation of work previously performed under sub-contract from Boeing Airplane Company. Previous work is reported in Boeing Airplane Company progress and summary reports issued under contract No. AF 33(616)-5722 and entitled Design Criteria for O-Rings and Similar Elastic Seals.

Introduction

This research divides logically into three distinct but related areas. These are Static Seals, Moving Seals, and Elastomer Properties.

The work on static seals includes (1) elastomer o-rings in confined and unconfined flange designs; (2) high strength plastic gaskets which seal under localized high stress; and (3) vendor seals of metal, fluorocarbon plastics, or various combinations.

The study of moving seals is concerned initially with methods of effecting a seal around a rotating shaft. Engineering terminology distinguishes between "mechanical packing" and "mechanical seals". Mechanical packing refers to any seal material compressed into the annular space between rotating shaft and stationary housing. This annular space is commonly known as the "stuffing box". Mechanical seals, on the other hand, have a rotating face sealing against a stationary face at right angles to the shaft. Mechanical seals are initially more expensive than mechanical packings, but they have a number of important advantages, including the fact that they prevent shaft wear. Both types of shaft seals are being studied for use at cryogenic temperatures.

The work on low temperature properties of elastomers is concerned initially with a study of elastomer samples which are compressed at room temperature, then cooled under constant stress to cryogenic temperatures.

1. Static Seals

A fixture for testing highly compressed elastomer o-ring seals is shown in Figure 1. This fixture and the seal test procedure have been described in previous reports[1]. It has been found that standard commercial o-rings of 1/8 inch cross section diameter can be used to make excellent seals at cryogenic temperatures.

It has also been shown by previous work[1] that thin flat gaskets of Mylar[R] or nylon make excellent cryogenic seals with relatively low flange loads. The key to success in this case is the method of applying sealing force to the gasket, and consists essentially of setting up a narrow ring of highly concentrated stress to form the seal.

[R] Du Pont registered trademark

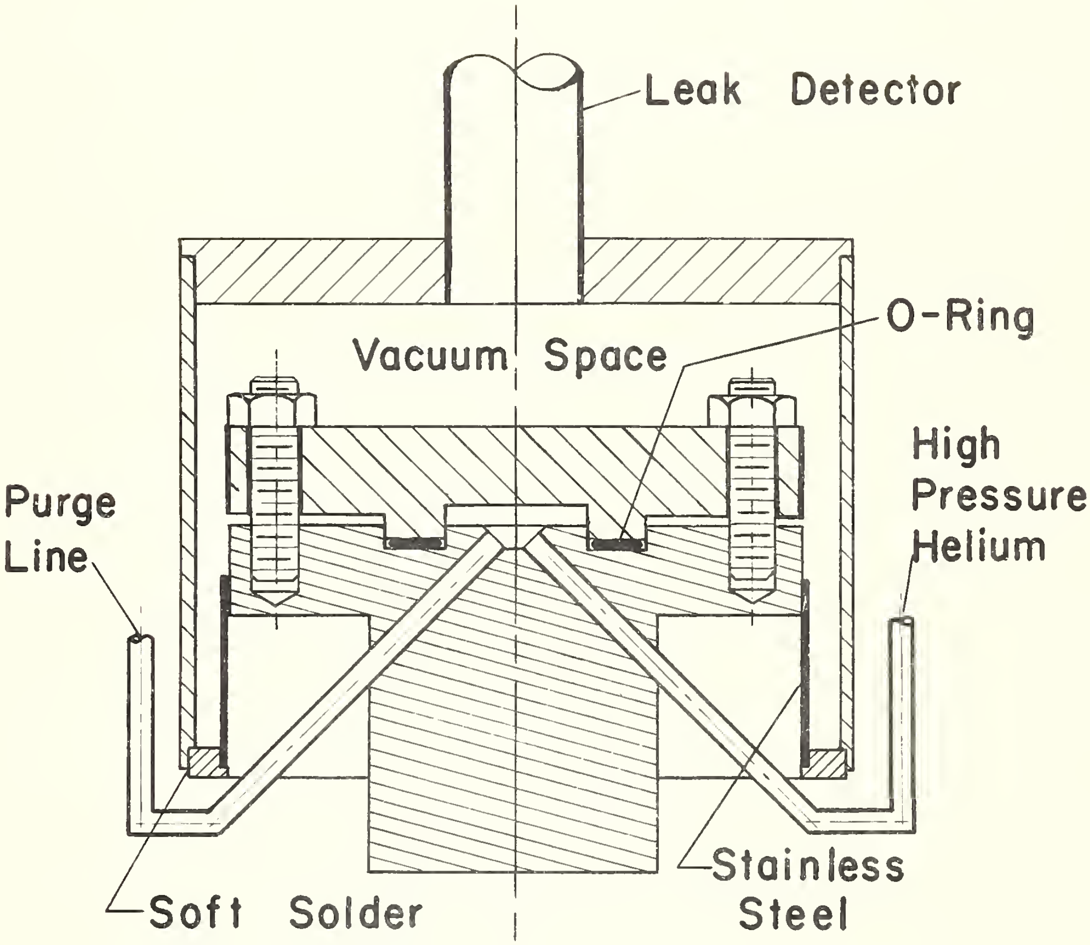


Fig. 1· O-Ring seal test jig .

Both of these basic seal designs have been extended by the present work.

1.1 O-Ring Seals

The elastomer o-ring concept has been explored in two additional ways. These are the use of o-rings of small cross section in the tongue and groove flange design and the use of o-rings between flat plates. Viton A[R] o-rings of 1/16 inch cross section diameter have made satisfactory seals in a tongue and groove flange design and required about half as much bolt loading as 1/8 inch o-rings of the same material. Thus one of the principal disadvantages of the original seals of this design can be substantially reduced. The limiting factor would appear to be width and uniformity of the compressed o-ring.

1.1.1 O-Rings Between Flat Plates

The original tongue and groove flange design was dimensioned to closely confine the o-ring at the end of the linear compression, and then subject the confined elastomer to about 5% volume compression. This required close machining tolerances in the flange construction, and high bolt loads at the end of the compression. Although the resulting seal is very reliable, and can be made with a large number of elastomer compounds, it has now been found that some of the elastomers make satisfactory seals without lateral or "side" confinement.

There are several advantages to this modification: (1) the flange loading is about half that required for a confined o-ring of the same cross section; (2) the flanges require a minimum of machining and there are no close machining tolerances; and (3) the surface finish of the flanges is not critical - in fact, a somewhat rough machine finish is advantageous since it helps prevent excessive flow of the otherwise unconfined elastomer.

It is apparent now that minimum bolt loading should be required to make the seal if an o-ring of minimum cross section is compressed between ungrooved flat flanges. With this in mind, a number of seals were made by compressing 1-inch diameter by 1/16 inch thick o-rings between rigid flat flanges. Bolts and flanges were sufficiently heavy to eliminate any significant spring loading effects due to flexing of metal parts. All of the elastomers were standard "Parco" brand o-rings, supplied at no charge by Plastic and Rubber Products Company of Los Angeles.

In Table 1, column 2, the dash number behind the compound number identifies the Shore A Hardness. Column 3 gives the maximum test pressure for seals which were tight at 76°K, or the pressure during cooldown if the seal failed before reaching 76°K. If the temperature in column 4 is 76°K, the seal leaked less than 10^{-4} standard cc of helium per hour per inch of seal at the given pressure. Other values in column 4 indicate the temperature at which the seal leaked more than 0.2 cc per hour per inch of seal. The compressions given in column 5 are maximum values tried for the seals which failed and minimum values tried for the seals which were tight. Column 6 gives the torque used to tighten the six 3/8 - 24 UNF flange bolts. The Teflon o-ring was supplied by Boeing Airplane Company and the Teflon-coated stainless steel o-ring was purchased from Advanced Products, Inc., North Haven, Connecticut.

The following comments supplement the results shown in Table 1.

1. Hypalon. A very good seal resulted at 85% compression, and this compression required only medium bolt loads.

2. Natural Rubber. Although natural rubber has been very successful for confined o-ring seals, it did not make a good seal between ungrooved flanges. This is probably due to its low compression modulus at room temperature.

3. Paracril. The first seal of this compound leaked, but the o-ring recovered quickly when the seal was disassembled and warmed. On this basis a higher squeeze was applied and the seal held 1000 psig at 76°K. When the jig was opened, however, the o-ring was found to be shredded and broken in many places. This mechanical failure under load at low temperature leads us to advise against use of Paracril.

4. Neoprene. Neoprene was tested at 94% compression, but this was obtained with only medium bolt loads. The o-ring appeared to be undamaged by the low temperature and high compression. At 85% compression the seal failed at 86°K, indicating that a compression of about 90% would probably be adequate.

5. Butyl. This material was compressed 85% and failed at the relatively high temperature of 135°K. It's softness evidently makes it unsuitable for this type of seal.

1. Polymer	2. Parco Compound No.	3. Test Pressure (psi)	4. Minimum Temperature ($^{\circ}$K)	5. Final Compression ($^{o}/o$)	6. Bolt Torque (ft-lb)	7. Recommended
Hypalon	921-65	1000	76	85	10-15	Yes
Natural Rubber	634-50	50	86	90	16-17	No
Paracril	427-50	1000	76	90	20-25	No
Neoprene	307-50	1000	76	94	10-15	Yes
Butyl	805-40	50	137	85	5-10	No
Hycar	228-50	250	76	86	10	Limited
GR - S	132-50	50	158	85	5	No
Thiokol	501-70	125	155	85	5-10	No
Teflon		1000	76	90	25	Limited
Teflon-coated Stainless Steel		125	76	50	5	Limited

Table 1. Seals made by compressing 1/16" o-rings between flat flanges

6. Hycar. Hycar has held moderate pressures at $76^{\circ}K$ when compressed 86%. This indicates at least some usefulness for cryogenic seals. Higher compressions will be tried when more stock is available.

7. GR - S. Failed at $158^{\circ}K$ when compressed 85%. Does not appear to be a good candidate for this type of seal. Very low strength material.

8. Thiokol. Failed mechanically at 85% compression and $155^{\circ}K$. Not a good candidate.

9. Teflon. A 1/10 inch thick o-ring was compressed 90%, requiring relatively high bolt loading. The seal held 1000 psig at $76^{\circ}K$. When the jig was opened, the o-ring remained completely flat, as might be expected of this plastic material. This property, together with the low coefficient of friction, casts some doubt on the reliability of Teflon for this type of seal.

10. Teflon-coated Stainless Steel. This was a hollow o-ring of 321 stainless, made of .094 inch diameter tubing with .010 inch wall. The finished o-ring was 1.25 inch O.D. When compressed 50%, which required bolt torque of only 5 ft-lb, the seal was good to 125 psig at $76^{\circ}K$.

Viton A, which will probably make a successful seal of this type was not tested because of depleted stock. It will be tested later. Silicone rubber was not tested because of its softness and low mechanical strength.

The principal advantage of low bolt loading is the light weight flange construction which it makes possible. This is especially attractive for airborne equipment where weight must be kept as low as possible. Light weight flanges automatically introduce another factor, namely flexibility, and this can be used to advantage in the overall seal design. With proper choice of materials and proper positioning of bolts and o-ring, flexing of the flange results in a very efficient spring loading which greatly reduces the rate of force decay during cooldown of the seal.

The fuel line flange currently used on the Centaur employs a pressure actuated Teflon jacketed metal seal. This seal has failed to perform properly in this application. Accordingly, an investigation to study seals for this application has been undertaken. Two test flanges (similar to the Centaur flange) were constructed as illustrated in Figure 2. A test container was also constructed to allow the assembled

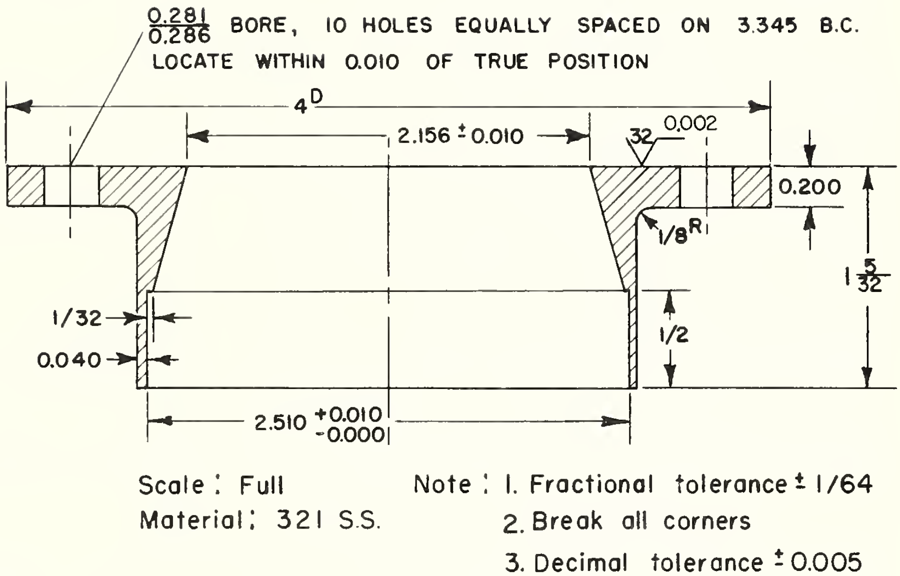


Figure 2

Centaur Fuel Line Flange

flange to be pressure tested at liquid nitrogen temperature (-320°F). The flange was assembled as shown in Figure 3 and placed inside the test container. The volume around the assembly was evacuated and connected to a mass spectrometer helium leak detector. The volume inside the flange assembly could then be cooled to liquid nitrogen temperature and pressurized with helium gas. Any seal leakage would immediately be shown by the leak detector. The assembly was proof-tested to 225 psi and leak checked at 165 psi.

Three tests have, thus far, been performed with this apparatus. The first of these employed a neoprene o-ring, I.D. = 2.614 in., width = 0.070 in., obtained from Plastic and Rubber Products, Inc. (Parco), Los Angeles, California. The neoprene was designated as compound 307-50. The seal was made by installing the o-ring, as received from the manufacturer, between the two clean (degreased with trichloroethylene) flange faces. The unit was assembled by evenly tightening ten 1/4 inch N.F. cap screws to a torque of 120 in.-lb. When pressure tested, the seal was found to be leak tight on the most sensitive scale of the mass spectrometer at both room temperature and liquid nitrogen temperature. The seal assembly was temperature cycled from ambient to liquid nitrogen temperature three times, and pressure tested after each cycle. No leaks were indicated at any time.

Identical tests were made with two additional o-rings:

1. Parco Compound 921-65 ("Hypalon"), I.D. = 2.614 in., width = 0.070 in. Bolt torque = 70 in.-lb.

2. Neoprene, O.D. = 2-3/4 in., width = 0.139 in. This standard o-ring was a rather hard compound carried in NBS-CEL stock for general high-vacuum use. Bolt torque = 120 in.-lb.

Both of these tests were as promising as the first, with no leaks indicated.

It is interesting to note that the flange edges, on tests one and two, were touching at the bolt torque indicated. It is evident, therefore, that the spring loading effect mentioned above is active in this seal.

These seals appear promising for the Centaur application. The present testing was done at liquid nitrogen temperatures only. Past experience with these materials, however, indicates that service at liquid hydrogen temperature would have no further detrimental effects, which were not already apparent at liquid nitrogen temperature.

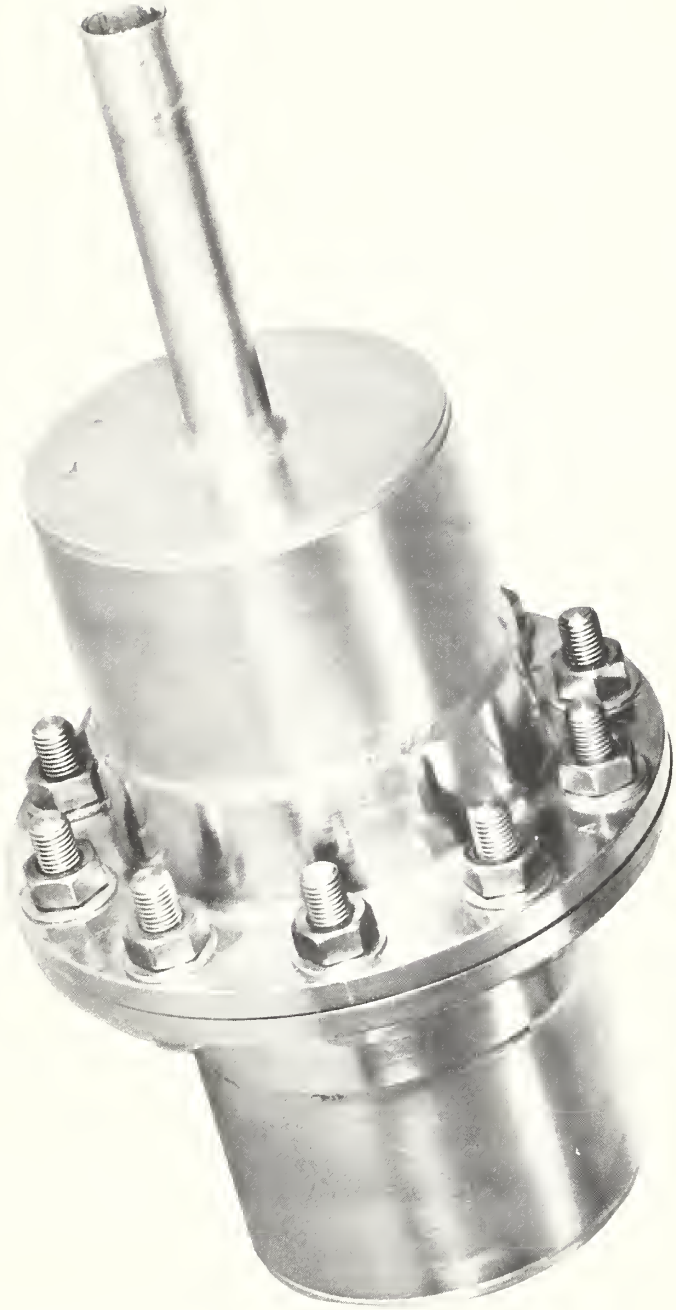


Figure 3. Flange Seal Test Assembly

These encouraging results warrant further investigation of the possible application to the fuel line flanges. The preliminary results have been given to Convair Astronautics and a joint program established. NBS will perform additional thermal and pressure tests on the above sealing method and on other more conventional commercial seals. CVA will perform vibration tests on the successful seals. Physical retainment of the seals for field installation will be considered by NBS and CVA.

The concept of a seal formed by compressing a narrow ring of thin Mylar has also been adapted to use with simple flat flanges. This was accomplished by making a sandwich of two Mylar gaskets with a solid metal o-ring (made of about .050 nichrome wire) between them. Thus, the wire takes the place of the machined compression ring described in previous reports, and forms a seal against each of the two flat flanges. This design has proven convenient and completely reliable.

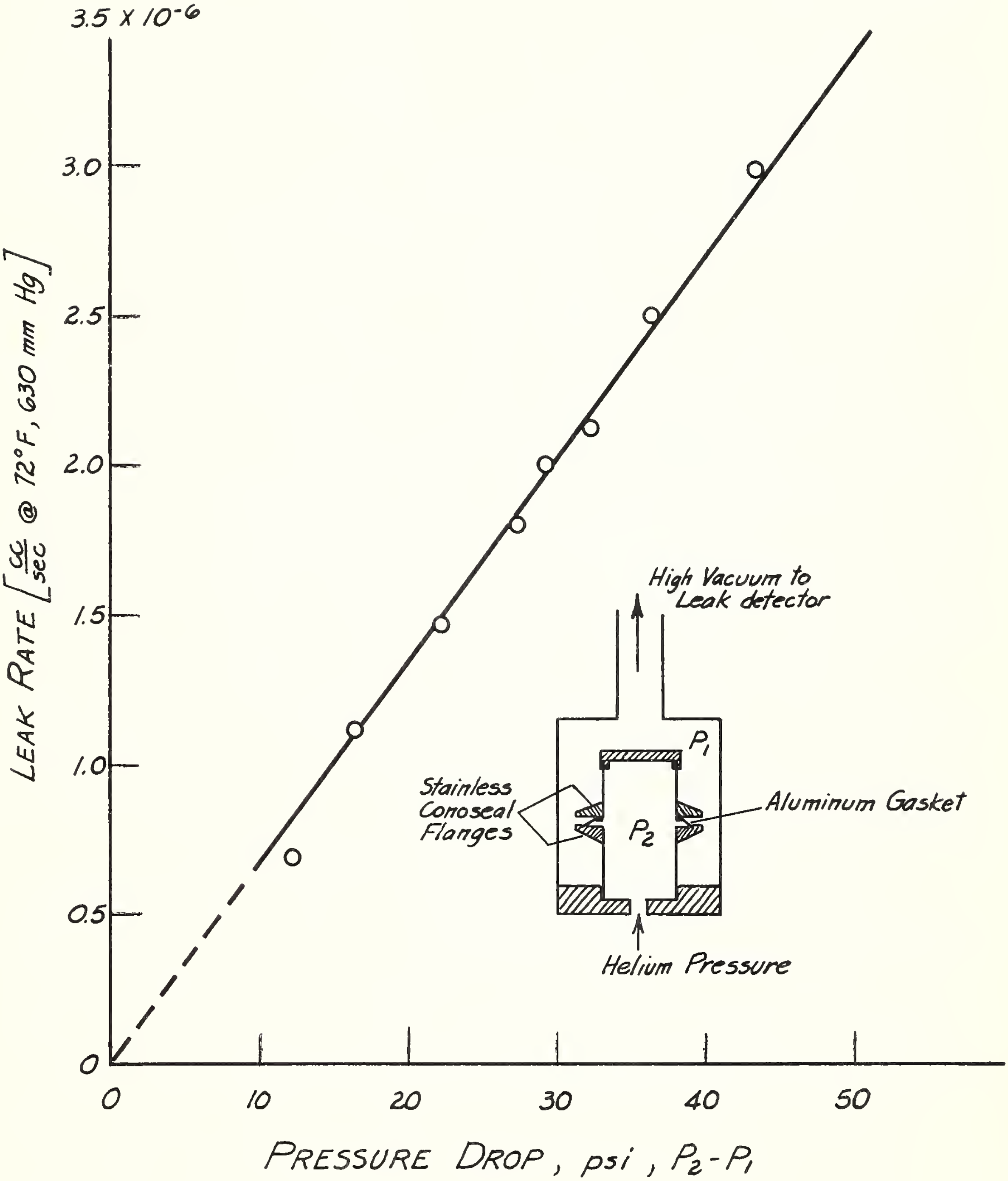
1.2 Vendor Seals

A metal-to-metal flange seal produced by Marmon Division of Aeroquip Corporation was adapted to our test jig by Mr. R. F. Ward of Marmon. In this seal a conical aluminum washer is compressed between mating stainless steel flanges, effecting line contact on the inside and outside circumferences of the washer. Attempts were made to use an aluminum flange with the aluminum gasket and a stainless steel gasket with the stainless flange, but aluminum welding difficulties and unsatisfactory performance of the "V-band" coupling used to compress the gasket made these combinations unsatisfactory.

Test results at $20^{\circ}K$ together with a rough schematic of the test jig are shown in Figure 4. The seal was leak tight from room temperature to somewhat below $76^{\circ}K$, when it began to leak. At $20^{\circ}K$ a stable leak rate was observed at various pressures as shown in Figure 4. These leak rates are of the order of 1 cc every 10^{6} seconds, which is serious for a seal between high vacuum and some moderate pressure, but of little concern in many other applications.

It is believed that the leak resulted from the greater contraction of the aluminum gasket which reduces the high forces initially present. This difficulty could be remedied by using a stainless steel gasket. In conclusion, this seal using an aluminum gasket in conjunction with at least one stainless steel flange (necessary in any stainless-to-aluminum transition joint application) performed satisfactorily at $76^{\circ}K$ but developed a pressure dependent leak at $20^{\circ}K$.

Figure 4
Leak rate, MARMAN CONOSEAL®
transition joint,
at 20°K (-424°F).



This test was of a preliminary nature and should not be interpreted as NBS endorsement of the Marmon conoseal for cryogenic applications.

2. Moving Seals

An apparatus to test seals between a 1-inch diameter rotating shaft and a 1-1/4 inch diameter housing is shown in Figures 5, 6 and 7. This apparatus incorporates a sliding seal which separates helium pressure from vacuum or from atmospheric pressure. If a sufficiently high vacuum can be maintained, the leak rate is measured by a helium leak detector; at higher leak rates the "vacuum" space is kept at atmospheric pressure and the leak rate is measured through a precision west test gas meter. The part containing the seal can be cooled by immersion in liquid nitrogen or liquid hydrogen, while the shaft is rotated by means of a motor-driven flexible shaft. A spring-loaded clutch protects the motor and slips with an audible clicking when the torque required to turn the shaft exceeds a pre-set value.

Figure 5 shows details of one assembly designed to test various materials in the form of interchangeable inserts. The inserts are statically sealed to shaft and housing by means of highly compressed elastomer o-rings, while the flat faces of the inserts themselves slide on one another to make the seal. Force to compress the static o-rings, as well as to maintain close contact between the sliding seal faces, is applied through a ball bearing and maintained constant during cooldown by means of a large cone-shaped compression washer through which the shaft passes. This compression washer has an accurately known spring constant, so that micrometer measurement of washer deflection gives the sealing force. Figure 6 shows the test fixture in two stages of assembly, and Figure 7 is a diagram showing the cryostat arrangement for testing in cryogenic environments.

This test fixture can also be used for the study of shaft packings of various types. For this purpose a second ball bearing is positioned below the top static seal, and the packing is compressed between metal inserts which replace the two parts of the sliding seal.

Tests of both types of shaft seals are in progress, but results have not yet been sufficiently reproducible to justify any detailed reporting. Although some reasonably tight seals of both types have been achieved with filled and unfilled polytetrafluoroethylene, the torques required to turn the shaft have in every case been too high for any practical application which involves continuous rotation over a period of time. Some of the packings might be suitable for valve stems

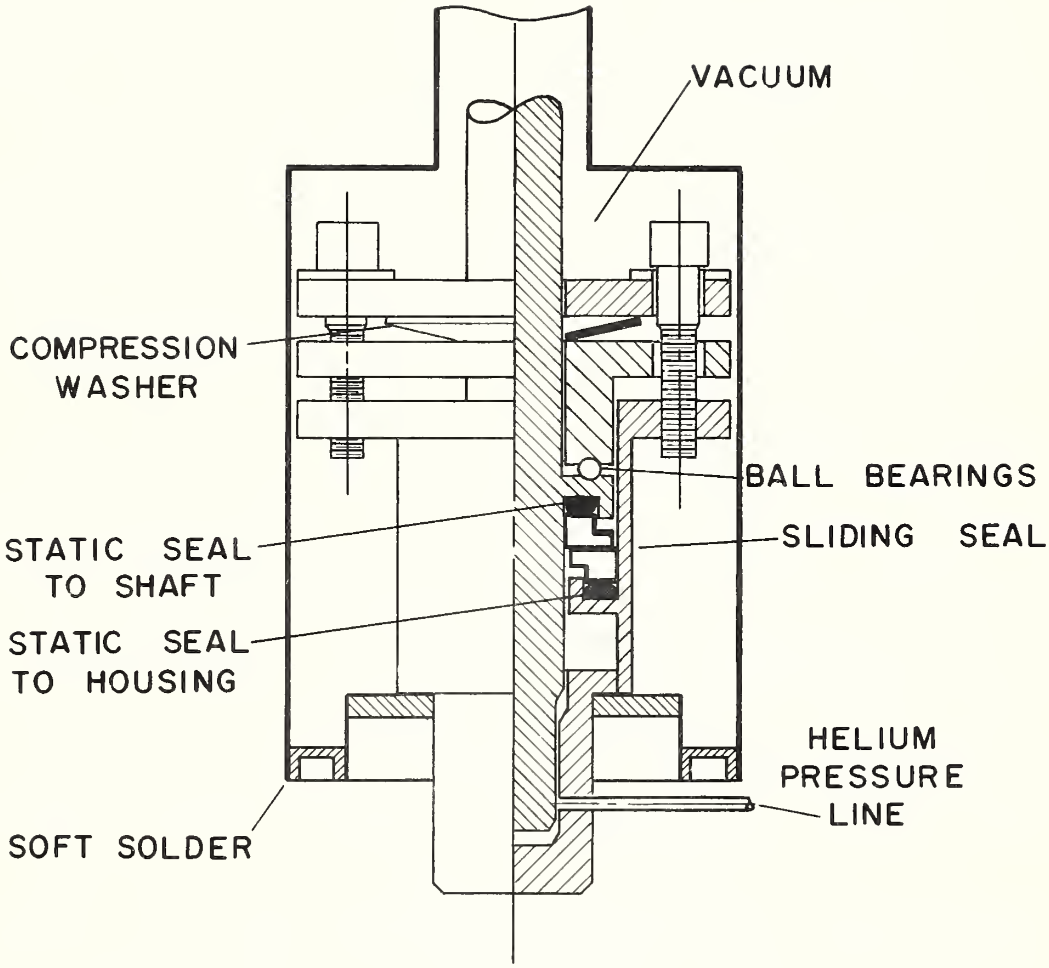


Fig. 5. Detail of shaft seal tester.

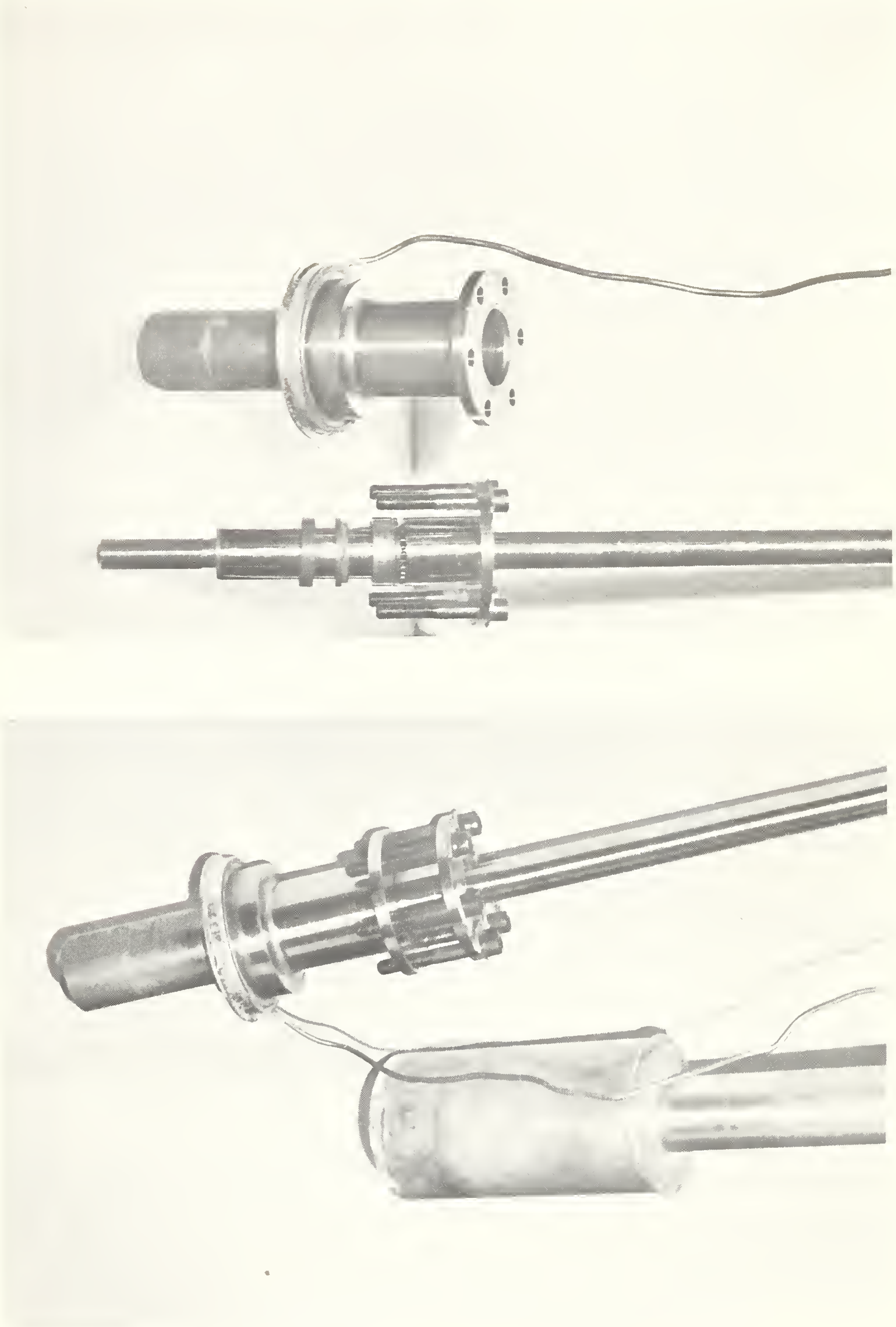


Figure 6. Rotating Seal Test Fixture

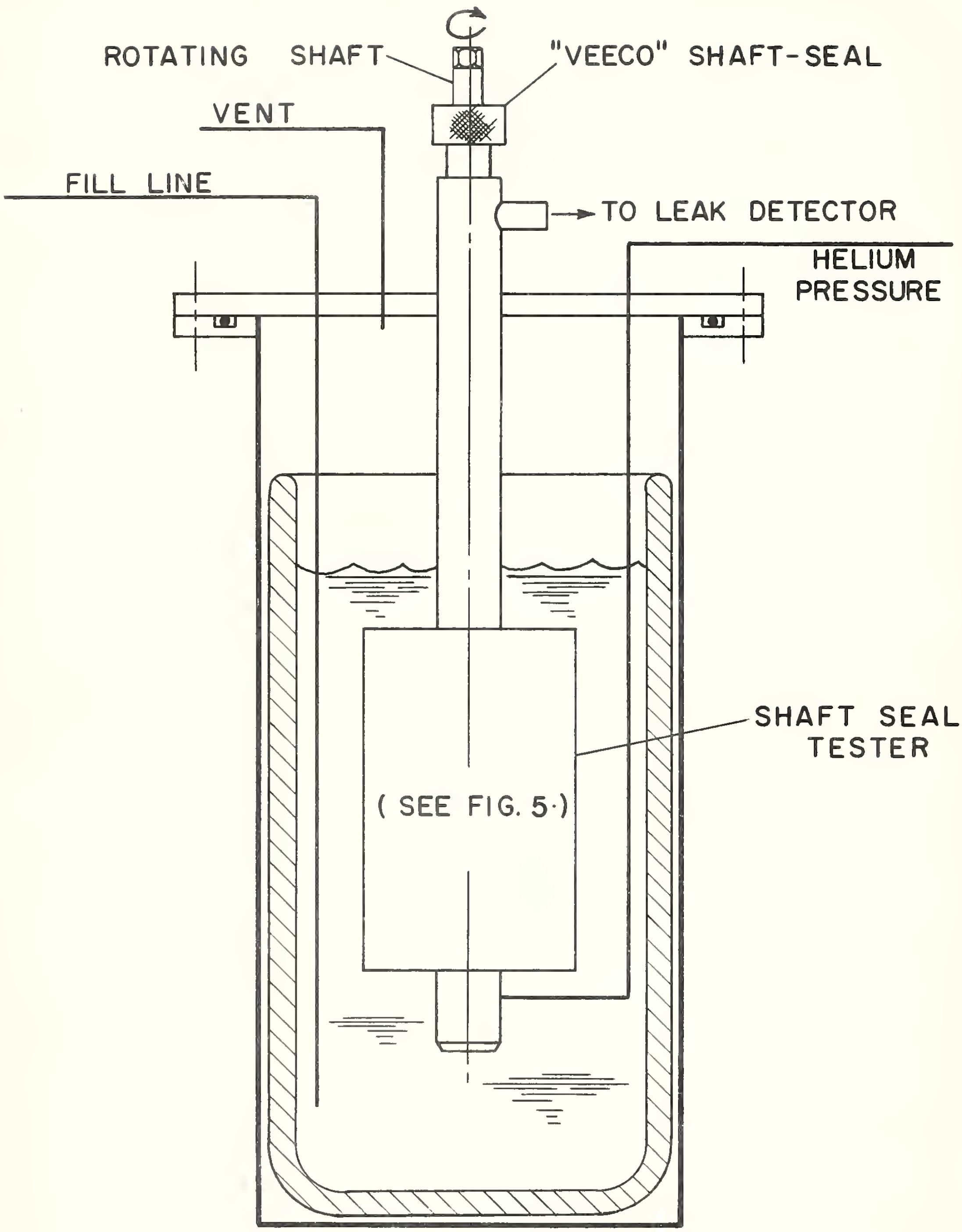


Fig. 7· Seal tester assembly .

which are subjected to intermittent turning at cryogenic temperatures. These are all more or less standard designs and will be discussed after more detailed testing has been completed.

After considerable study of mechanical seals which are currently in use throughout the cryogenic industry we have decided to rely on the best available engineering for the seal design and concentrate on the study of seal face materials. This is appropriate not only because the study of seal materials is consistent with the stated objectives of this research, but also because leading mechanical seal engineers have told us that seal face materials and combinations constitute the area of greatest uncertainty in present mechanical seal design.

A good mechanical seal, proven in cryogenic service, will accordingly be obtained from Chicago Rawhide Manufacturing Company. This seal will be tested with various face materials in a test rig similar to those currently used at NBS for testing of cryogenic bearings. More details of this program will be presented in our next report.

3. Elastomer Properties

Studies of the physical properties of elastomers have continued during the reporting period. In all applications of concern to this project a material is subjected to an initial compressive force (which may result in shear and tensile forces as well) at room temperature, then cooled to a point far below the glass transition temperature. Much attention has been directed toward the behavior of elastomers as they are treated in the above manner.

The linear thermal expansion of any material is the change in length with a change in temperature, the force acting parallel to the length in question always being held constant. To study the thermal expansion of elastomers as they are cooled after an initial compression, the compression dilatometer shown in Figures 8 and 9 was designed and built. Figure 8 pictures the dilatometer and associated equipment, including the 50L cryogen container, thermocouple recorder, water bubbler used to regulate the cooling rate of the sample, and the glass, high vacuum insulated dewar which surrounds the dilatometer proper during a test. Length measurements are achieved using a lever which translates vertical motion of the sample and the stainless steel compression platens into horizontal motion outside the cold area, as shown in Figure 9. A micrometer screw is used to measure the travel of the lever. Helium gas pressure in the bellows maintains a constant applied force on the sample.

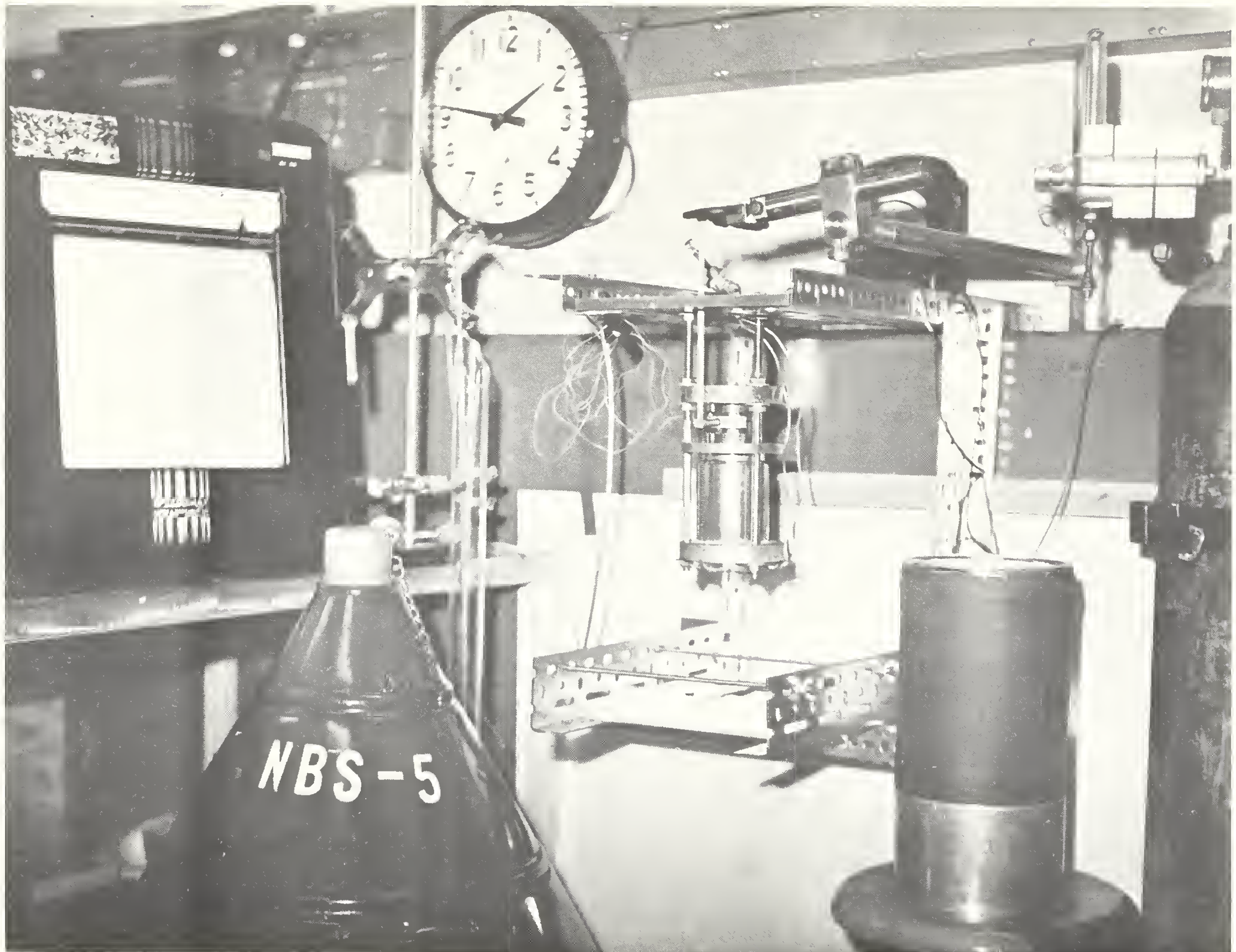


Figure 8. Compression Dilatometer and Associated Equipment

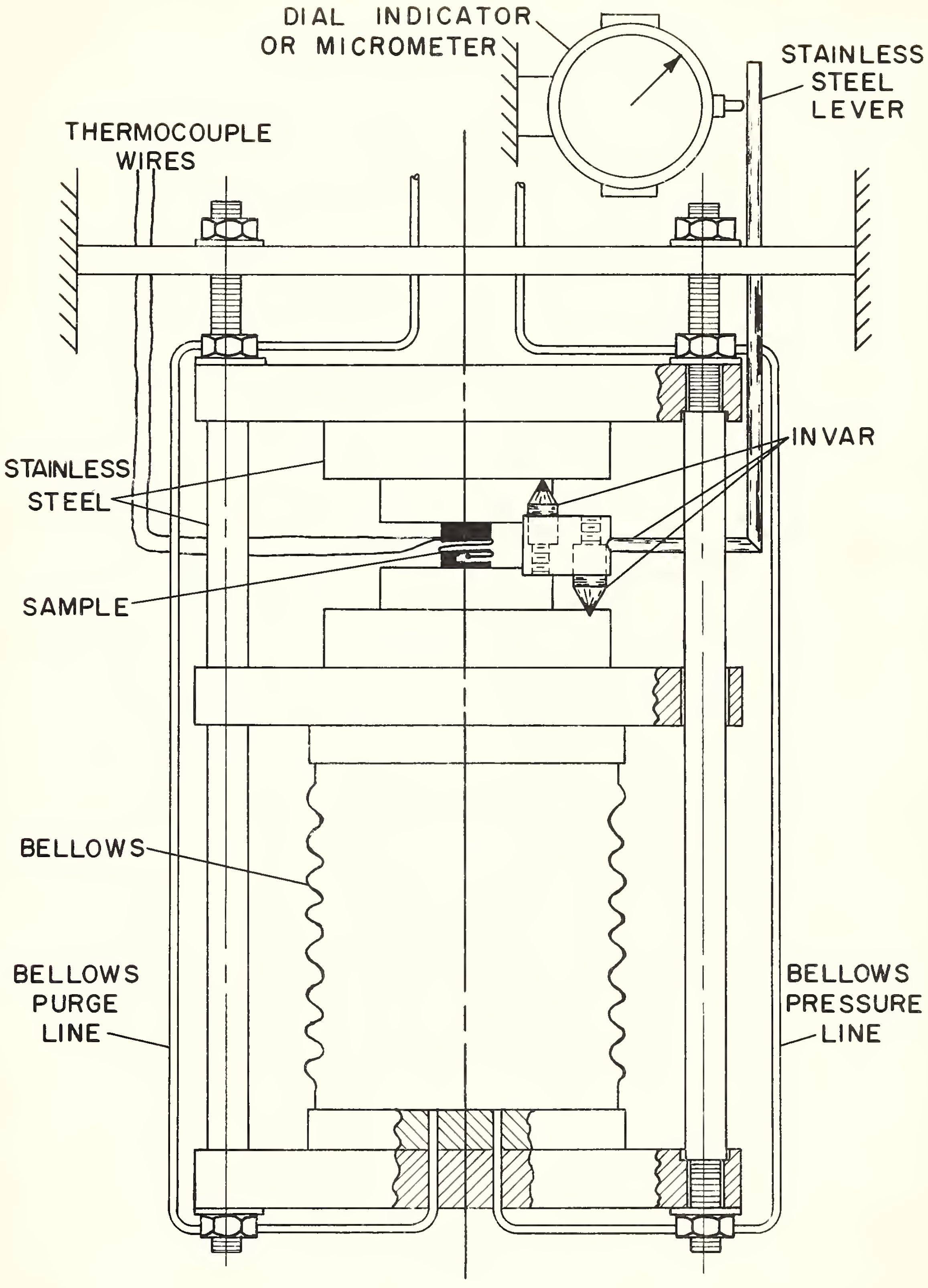


Fig. 9· Compression dilatometer .

Some preliminary test results are shown in Figure 10, in which the unit change in length after compression is plotted vs temperature. The Viton A buttons were cylindrical; unfortunately it was necessary to vary the size of the button to accommodate the apparatus. For the 31% squeeze test, a 1 inch^2 x 1/2 inch compression set button was ideal, however to apply the stress (2655 psi) needed for 79% squeeze, it was necessary to use a sample 1/2 inch in diameter and 1/4 inch thick. Accuracy of the data was therefore reduced in the 79% test, since after the initial compression, the sample length was only 0.052 inches. The configuration tested at 0% squeeze was 1/2 inch in diameter and 2 inches long, and only the overall contraction from 297°K to 76°K was measured. From considerations of the force decay tests taken at constant length and previously reported[1] and previous work reported by Logan[2] it was felt that high compressions might reduce the unit thermal contraction; however, the opposite effect is indicated by Figure 10. The rapid change in slope around the glass transition temperature has been observed by many[3, 4, 5] investigators, and was expected.

Viton A was chosen as the test material since it was successful in the functional seal tests; however, knowledge of the complete history of the samples and the material itself was not adequate to describe the above behaviors by classical elastic theory. Even with more knowledge of the material, the classical theory would not completely describe the experiments, since sample geometry and friction forces at the loading faces vary with each test. Within these limitations, there are still reasons to believe that the test results should have been expected, as described in the following paragraphs.

It is well known that the properties of high extensibility and forcible quick retraction common to all rubber-like materials are not characteristic of the chemical structure, but rather of the molecular structure, of the materials[6]. In fact, these properties suffice as the definition of an elastomer. The molecular requirements necessary to produce an elastomer are as follows: (1) the molecular chains must be long; (2) there must be freedom of rotation about most of the bonds joining adjacent chain atoms; (3) the molecules must be linked together occasionally, either by chemical cross-links or by chain entanglements; and (4) the secondary forces between molecules must be small. When a (non-crystalline) elastomer is cooled, the free volume allowing freedom of rotation around bonds is reduced, and the secondary (van der Waal) forces have more effect.

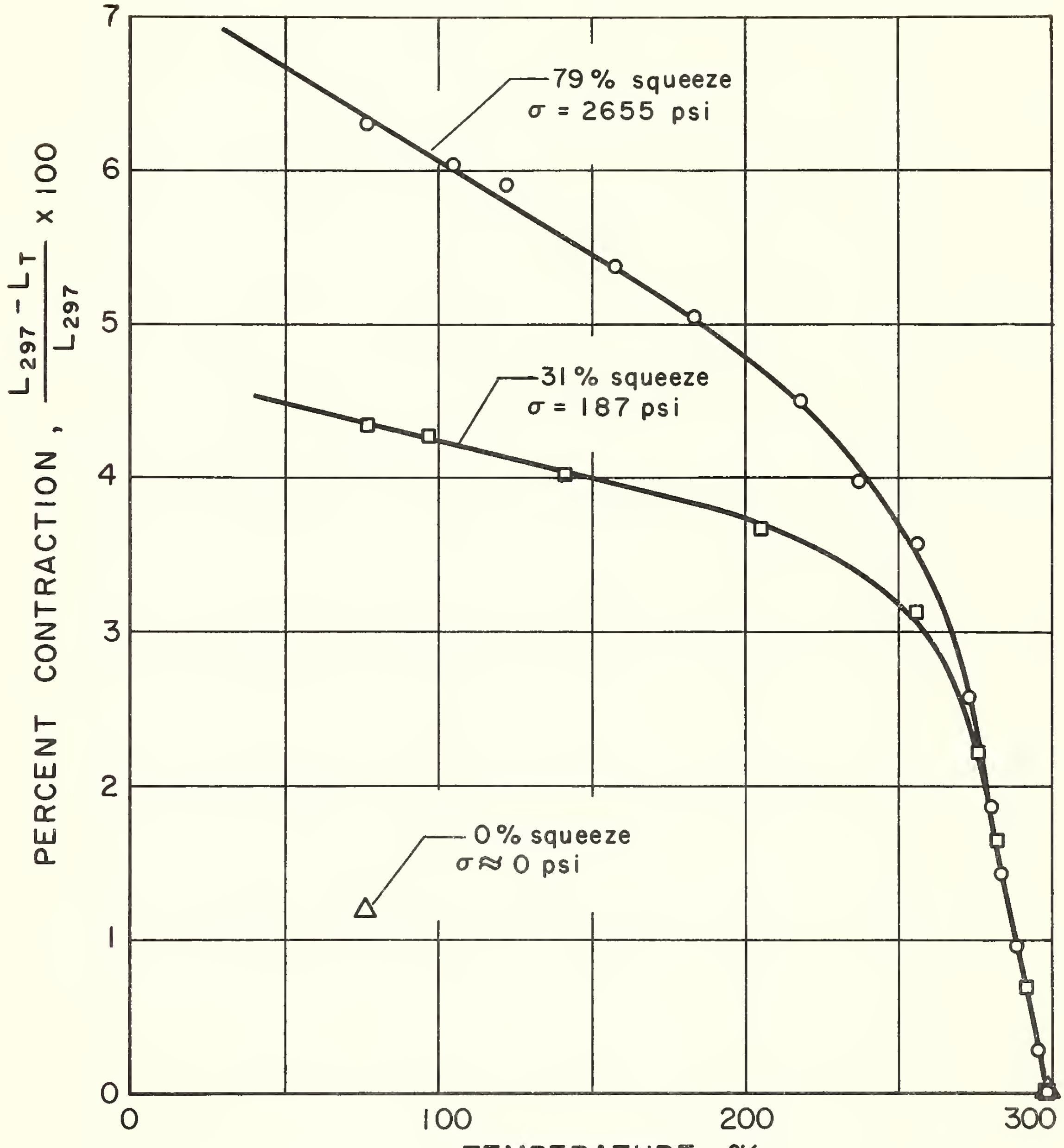


Fig. 10· Linear thermal expansion of a compressed Viton-A elastomer at constant stress.

If a constant compressive force is applied at room temperature and maintained while the material is cooled, the secondary bonds between molecules are continually shifting and seeking a more relaxed orientation causing contraction, independent of any temperature change, which is commonly referred to as creep. This time effect continues throughout any reasonable experimental testing period; Martin, Roth, and Stiehler[7] have experimentally found the elongation vs log time curve (in tension) to be linear over the range one second to one week for several common elastomers. Since the secondary forces become more important at temperatures near the glass transition[8], it stands to reason that the resulting creep (contraction, in our case) would be large between 250°K and 300°K, even though contraction was essentially zero at room temperature. It also follows that higher initial compressions result in greater unit contraction, since the larger internal stresses cause more re-orientation of the secondary bonds.

Thus, it is apparent that time effects greatly influence measurements of thermal expansion of elastomers taken at constant force. Variation in test time is probably responsible in part for the differences in slope below the brittle point for 31% and 79% squeeze. Since the definition of coefficient of linear expansion [$\alpha = \frac{1}{L} \left(\frac{\partial L}{\partial T}\right)_p$] assumes no independent time effects, the slopes of the curves in Figure 10 cannot be interpreted as true coefficients. Since the long chain molecules tend to align in the plane perpendicular to the applied force, the contraction due solely to shrinking in the chains probably is reduced by high compressions. It is not evident that the creep and shrinkage contributions to the overall contraction can be separated experimentally; however, a two-directional extension experiment equivalent to pure uni-directional compression is being considered, since the resulting data could possibly be handled in part by classical theory of elasticity[4]. If so, the test could be a valuable tool in the clarification of the mechanisms involved in low temperature elastomer seals. Work with the compression dilatometer will also continue, with tests of other promising seal materials.

4. Acknowledgement

The authors wish to acknowledge the very valuable contribution of B. W. Birmingham, D. B. Chelton, and L. E. Scott in planning and supervising the Convair flange studies described in section 1.1.1.

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