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ELASTOMERIC SEALS AND MATERIALS AT CRYOGENIC TEMPERATURES

D. H. Weitzel, R. F. Robbins, R. N. Herring

NBS

U. S. DEPARTMENT OF COMMERCE
NATIONAL BUREAU OF STANDARDS
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Progress Report

Elastomeric Seals and Materials
at
Cryogenic Temperatures

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Foreword

This report was prepared by the National Bureau of Standards Cryogenic Engineering Laboratory under WADD (now ASD) 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.

Program Status

This research is being carried out in three overlapping areas. These are Static Seals, Moving Seals, and Elastomer Properties.

The work on static seals is in the advanced stages of testing optimum seal designs. We are also evaluating some of the more important side parameters such as flexing and stretching of flange parts.

The study of shaft seals consists of two parts: packings for slow-turning shafts and bellows type face seals for high speed rotating shafts. Tests of packings are in progress; background work for a high speed tester has been done and shop drawings are being prepared.

The work on low temperature properties of elastomers and plastics is being expanded. Two new physicists will join our group in June. They will contribute to all phases of the program but will primarily strengthen the properties effort. The Elastomers Section of Materials Central, ASD, is now supplying samples for properties and functional testing.

1. Static Seals

The most significant improvement in static cryogenic seals during the past year has been the substitution of simple flat flanges for the tongue and groove design. This development, together with the use of O-rings of smaller cross section, has reduced the required flange load by a factor of four and flange machining costs by a similar amount. The softest elastomers cannot be used in this way, and there may be some small sacrifice in reliability of the seal at high pressures, but these disadvantages are offset by simplicity and the great saving in weight which the new design makes possible.

A detailed study of the possibilities of flat flange seals was undertaken in connection with some requirements of the Centaur missile program. A number of flanges supplied or specified by Convair Astronautics were studied, using the method shown schematically in Figure 1. The flange and seal assembly was surrounded by a vacuum jacket which was maintained by a roughing pump and a helium leak detector. A stainless steel tube which passed from the seal assembly through the top plate of the vacuum can could be used to introduce either helium pressure or refrigerant (liquid N_2 or H_2)

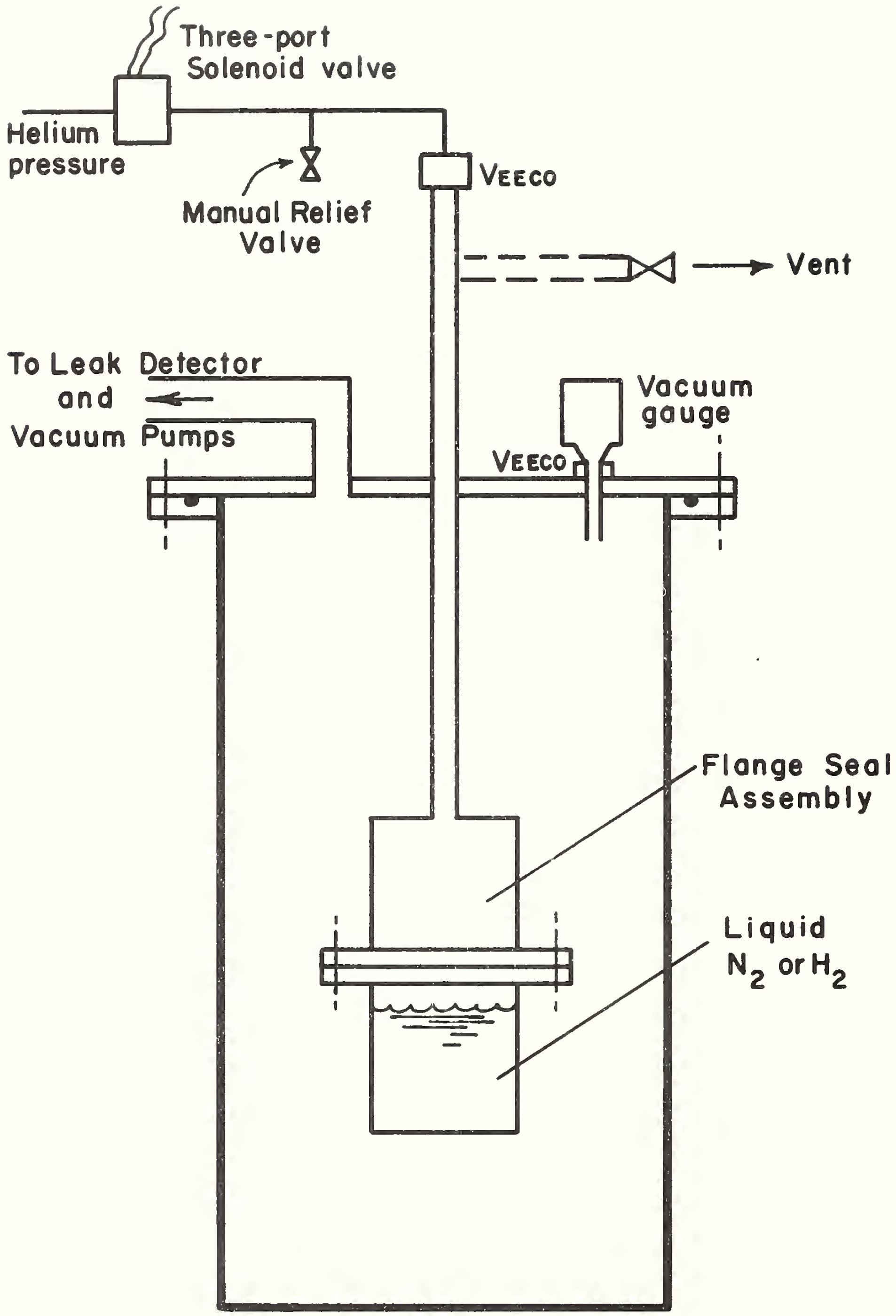


Fig. 1

Schematic of Seal Test Procedure

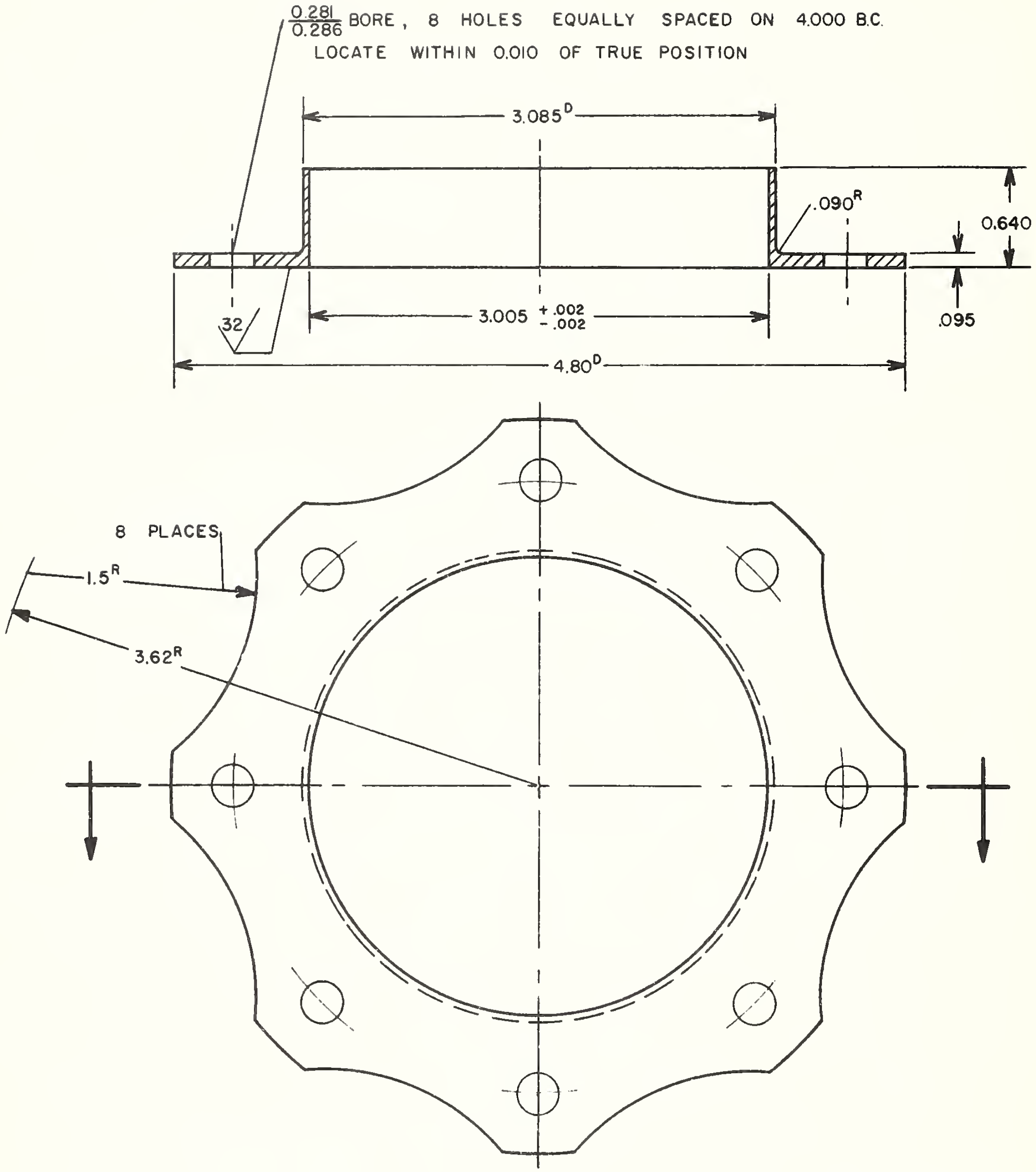
into the seal assembly. Thus the seal could be tested at any desired pressure, either before, after, or during cooldown, and any helium escaping into the vacuum space would be immediately observed by the leak detector.

Leak rates greater than 0. 5 cc per hour exceeded the capacity of the leak detector. These were evaluated by removing the vacuum jacket and submerging the flange assembly in water or liquid nitrogen. The inside of the assembly was then pressurized with helium gas, sealed off, and the leak rate determined by observing the rate of pressure decay.

Test procedure consisted of cleaning the flange faces with trichloroethylene and installing the O-ring as received from the manufacturer. Compression was by stepwise cross-tightening of the flange bolts with the aid of a calibrated torque wrench. The threads of the bolts were kept lubricated with a molybdenum disulfide aerosol dispersion. The assembly was then placed in the vacuum can and the latter evacuated. The seal was checked at room temperature by purging out the air and then pressurizing with helium gas. Success of the seal was determined by a zero reading on the most sensitive scale of the leak detector, which meant a leak of less than 3×10^{-4} standard cc of helium per hour. Higher leak rates were determined by reading the leak detector scales or by pressure decay as mentioned above. Some of the seals were subjected to a series of 25 temperature cycles by removing the flange assembly from the vacuum can and alternately submerging it in liquid nitrogen and hot water. The assembly was then placed in the vacuum can and again tested for leaks. Rapid pressure cycling was accomplished by means of an off-on switch which operated the three-port solenoid valve shown in Figure 1.

1.1 Extreme Lightweight Flange

One application involving airborne hardware for the Centaur missile required that weight be kept to an absolute minimum. The flange proposed by Convair Astronautics for this application is shown in Figure 2. The flange, which fits a 3" O. D. pipe, was made of type 304 stainless steel with faces only .095" thick. There were eight holes for 1/4" bolts, equally spaced on a 4" bolt circle, and further reduction in weight was made by cutting out scallops of material between the bolt holes. The various methods which were tested for sealing pairs of these flanges are listed in Table 1.



Scale: Full

Material: 304 S.S.

Note: Fractional tolerance ±1/64

Decimal tolerance ± .010, except where noted.

FIG. 2

Extreme Lightweight Flange

TABLE 1. Static Seal Tests Using Convair Astronautics Extreme Lightweight Flange

Test No.	Type of Seal	Size of Seal	Torque on Eight 1/4" N. F. Bolts	Comments and Results*
14.	Parco Hypalon[R] 921-50 O - Ring	3.480" I. D. .070" W	125 in-lb	O.K. warm. Pressure cycled 10 times. Leak rate cold approximately .5 cc/hr
23.	ditto	3.239" I. D. .070" W	125 in-lb	Flanges backed up by carbon steel ring under bolts. Used O-ring retaining sleeve. O.K. warm, leaked cold.
25	ditto	3.489" I. D. .070" W	125 in-lb	Flanges backed up. No sleeve. O.K. warm and at 1 atm cold. Leaked at 50 psig.
27	ditto	3.489" I. D. .070" W	125 in-lb	Flanges backed up and .005" shims under backing between bolts. O. K. warm, leaked cold.
31	ditto	3.239" I. D. .070" W	125 in-lb	Flanges given reverse bend before bolting. O. K. warm, leaked while cooling.

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* O.K. means leak rate less than 3×10^{-4} cc/hr of helium at 160 psig

(Table 1. Continued on next page)

Test No.	Type of Seal	Size of Seal	Torque on Eight 1/4" N. F. Bolts	Comments and Results
43.	Parco Hypalon 921-50 O-Ring	3.239" I. D. .070" W	150 in-lb	One lightweight flange vs. heavy aluminum. Used sleeve but O-ring loose on sleeve. O.K. warm, leaked while cooling.
45.	ditto	2.864 " I. D. .070" W	ditto	One lightweight flange vs. heavy aluminum. O-ring stretched over sleeve. O.K. warm and cold.
47.	ditto	ditto	ditto	Repeat of 45. Leaked cold. Increased torque to 165 in-lb. Still leaked at 76°K.
16.	ditto	3.237" I. D. .103 " W	125 in-lb	O.K. warm, leaked while cooling. At 76°K leaked approx. 180 liters/hr.
24.	ditto	3.237" I. D. .103" W	ditto	Same configuration as 23. O. K. warm, leaked while cooling.
26.	ditto	3.489" I. D. .103" W	ditto	Same configuration as 25. O. K. warm, leaked while cooling.
15.	Parco Neoprene 308-30 O-ring	3.237" I. D. .103" W	ditto	Soft - 30 durometer. Leaked warm.
28.	Parco Neoprene 307-50 O-ring	3.489" I. D. .070" W	ditto	Same configuration as 25. Failed warm. Non-uniform compression.

(Table 1. Continued on next page)

Test No.	Type of Seal	Size of Seal	Torque on Eight 1/4" N. F. Bolts	Comments and Results*
29.	Parco Neoprene 307-50 O-ring	3.239" I. D. .070" W	125 in-lb	Same configuration as 27. Failed warm. Non-uniform compression
30.	ditto	3.239" I. D. .070" W	125 in-lb	Same configuration as 27, but used sleeve. O. K. warm, leaked while cooling.
13.	.0075" Mylar[R] .065" Nichrome wire	Wire 3 1/4" I. D.	80 in-lb	Braced with .040" wire ring outside bolts. Leaked warm - no compression between bolts.
17.	.0075" Mylar .040" Nichrome	Wire 3 1/4" I. D.	60 in-lb	Same configuration as 13. Again leaked warm.
21.	.012 Mylar .050 Nichrome	Wire 3 1/8" I. D.	60 in-lb	Uneven compression. Leaked warm.
20.	ditto	Wire 3 1/4" I. D.	60 in-lb	Somewhat better compression. Leaked in H_2O at 10 Psig.
34.	.005" Mylar .040" Nichrome	Wire 3 1/4" I. D.	100-125 in-lb	Flanges backed up by carbon steel rings under bolts. Leak rate 0.2 cc/hr at 76°K, 50 Psig.

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As shown in Table 1, three seal materials were used. These were Parco Hypalon [R] Compound 921, durometer 50; Parco Neoprenes Compound 307, durometer 50, and 308, durometer 30; and Mylar [R] film. Hypalon and Neoprene were used as O-rings in two thicknesses, .070" and .103". On some of the O-ring tests a short stainless steel sleeve was fitted snugly inside the flange to hold the O-ring in position and prevent inward extrusion. A typical cross section of a flange assembly using such a sleeve is shown in Figure 3. Mylar film was used by making "sandwich" of two flat Mylar gaskets separated by an O-ring shaped compression ring made of nichrome wire. The ends of the wire were tapered and silver soldered to make a smooth joint.

No really satisfactory method for sealing these extremely light-weight flanges was found. Some of the seals held at room temperature, and one Hypalon O-ring used with the sleeve was leak detector tight at 76°K. But even this successful seal was probably borderline since a second attempt of the same kind failed when cooled to 76°K.

The reason for failure of these seals was lack of sufficient stiffness in the flange faces. Attempts to overcome this weakness by various schemes, as noted under "comments" in Table 1, were not successful. It is felt that the flange could be designed for greater rigidity with little or no increase in weight. In the present design there is minimum stiffness between bolts, which is precisely where maximum stresses occur. The price of a more carefully engineered flange design is likely to be higher machining costs, but a rib between the bolt holes, for example, would not add much to the cost and would greatly improve the flange characteristics.

1.2 Successful Flat Flanges

Additional flanges specified for the Centaur missile program are shown in Figures 4 and 5. These flanges, for 2.5" and 3" O.D. pipelines respectively, were made of stainless steel with flange faces .200" thick. The smaller flange was drilled for ten 1/4" NF 18-8 stainless steel bolts on a 3.345" bolt circle, and the inside corner was given additional beef at the expense of some reduction in pipe I.D. The larger flange lacked this reinforced corner and used only 8 bolts (same bolts as the other flange) on a 4" bolt circle.

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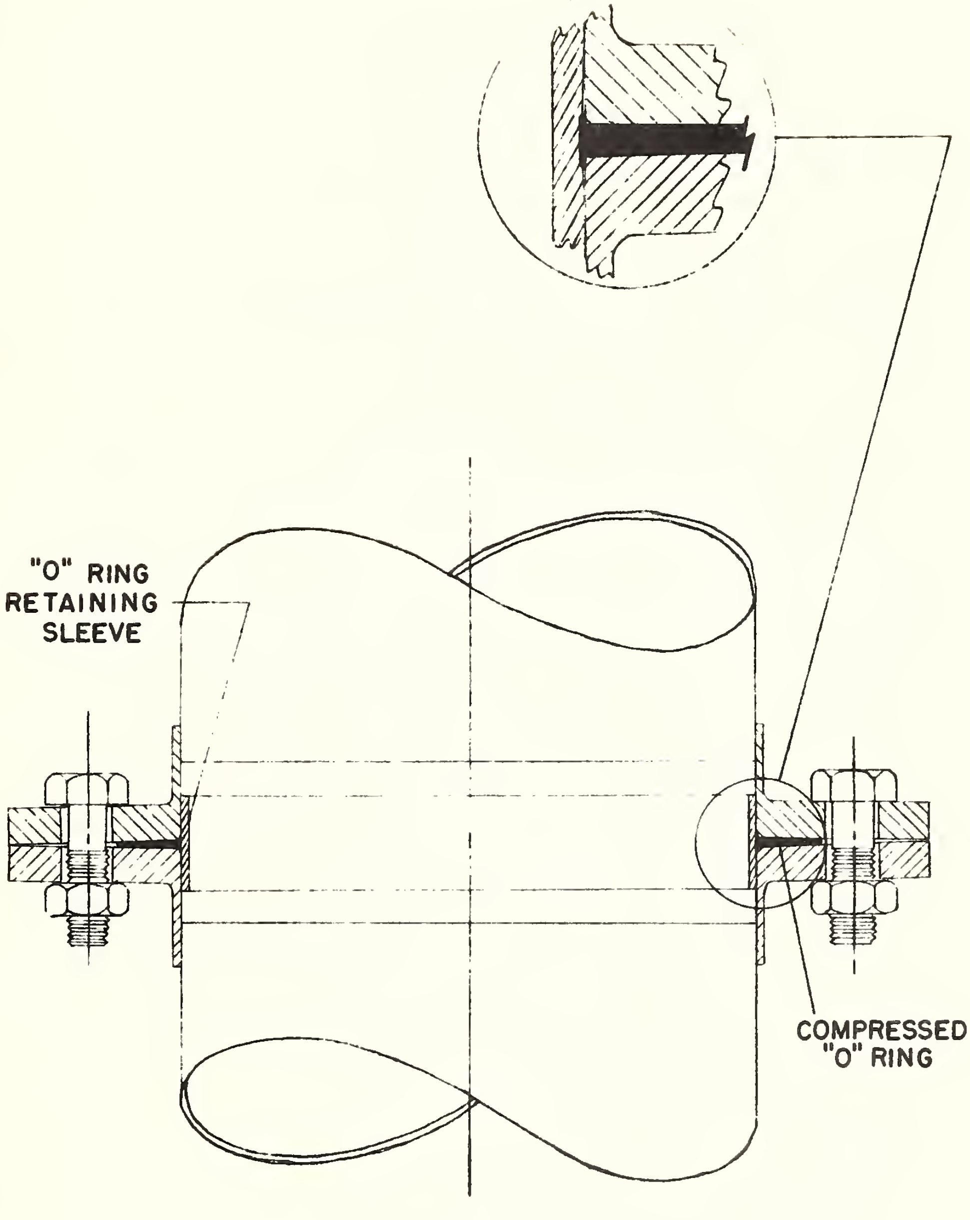


FIG. 3

Typical Flange Assembly
With "O" Ring Retaining Sleeve

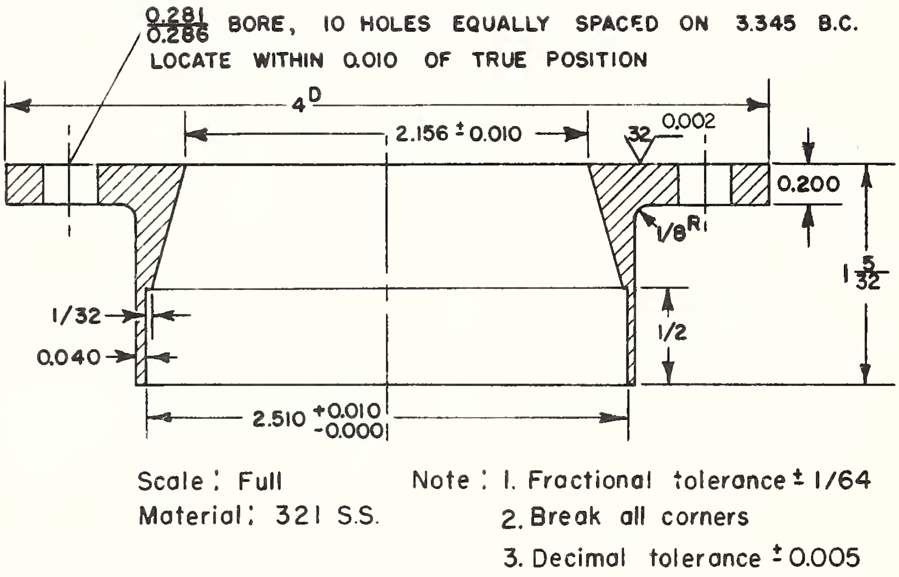
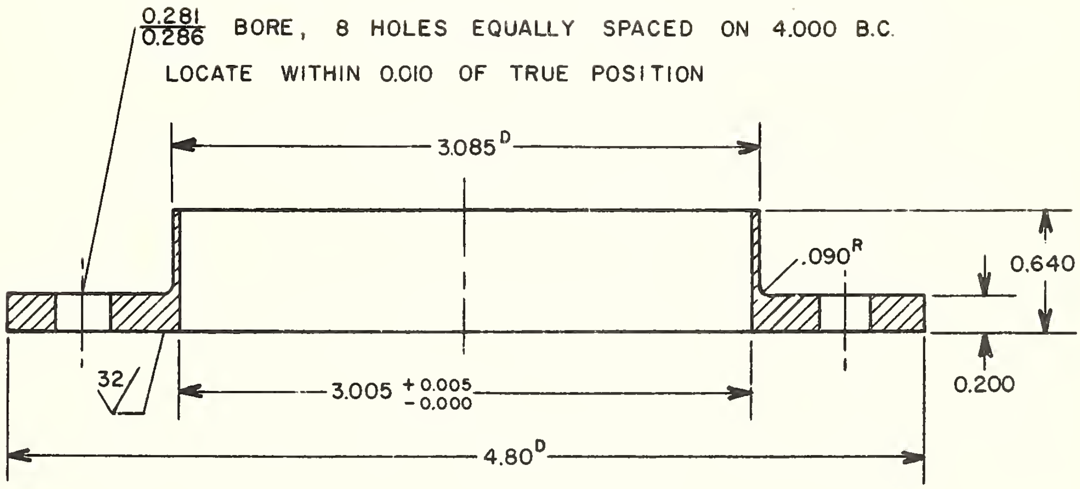


Figure 4. Centaur Flange for 2.5" O.D. Line



Scale: Full

Material: 304 S.S.

Note: Fractional tolerance ± 1/64

Decimal tolerance ± .010, except where noted.

FIG. 5

Centaur Flange for 3" O.D. Line

Certain parts of the Centaur fuel handling equipment call for a transition from stainless steel to aluminum. The flanges shown in Figures 4 and 5 were therefore duplicated from 6061, T6 aluminum, except that the flange faces were made .300" thick instead of .200". These were tested, both before and after anodizing to a black hardcoat finish, by mating with the corresponding stainless steel flanges. Except for the anodizing treatment, which was tested both polished and unpolished, all flange surfaces were used in the as-machined condition.

Table 2 gives the results of testing various combinations of these flanges with seals of Neoprene, Hypalon [R], Viton [R], Mylar [R], and Teflon [R].

Successful seals were obtained with all of the flange combinations. The Hypalon compound seemed to be somewhat more reliable than the Neoprene, and was tested more extensively. The Viton compound was successful in both of the tests for which it was used. The Mylar "sandwich" seal was very reliable when the proper combination of film thickness, compression ring cross section, and compressive force were used. In both types of seal a spacer ring to control the amount of compression would be advantageous. A machined step in one of the flange faces would also serve this purpose. The Mylar seals require only about half as much flange loading as the .070" W elastomers, but this advantage is somewhat offset by the fact that the elastomers can more easily adjust to flange irregularities. Another problem in the use of Mylar film is the possibility of cracking if the compressive stress is too concentrated or too high.

An important factor present in these tests was flexibility of the flanges. In every case there was appreciable flexing of the flanges which resulted in a more or less uniform spring loading of the seals. In the case of the extreme lightweight flange, flexing was so non-uniform that a reliable seal could not be achieved. The other flanges flexed into a fairly uniform cone shape (although some of the seals deformed into a flower pattern, showing some bowing between bolts). When the flexing was relatively uniform it served to reduce the initial compression required to maintain the seal during cooldown. Measurements indicated that good seals were being made with 65 to 70% compression of the O-ring, whereas 70 to 80% has been required in previous tests.

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TABLE 2. Seal Tests Using Convair Astronautics 2.5 and 3-inch Flat Flanges

Parco Neoprene O-Rings (Compound 307-50)

	Flange	O-Ring Size	Torque on Bolts	Comments and Results
5a.	Fig. 5 steel to steel	3.239'' I.D. .070'' W	75 in-lb	Flange edges just touched. OK warm. Leaked while cooling
5b.	ditto	ditto	180 in-lb	Additional squeeze after flange edges touched. Seal deformed inward to flower shape. Leaked warm.
6.	ditto	ditto	120 in-lb	OK warm. Leaked approx. 1 liter/hr when cold.
39.	Fig. 4 steel to Al.	2.614'' I.D. .070'' W	125 in-lb	Al. anodized, unpolished. Temp cycled twice, pressure cycled to 160 psig 5 times. No leak at 76°K.
44.	ditto	ditto	ditto	Al. anodized, polished. No leak at 76°K, 160 psig.

(Table 2. Continued on next page)

Parco Hypalon O-Rings (Compound 921-50)

	Flange	O-Ring Size	Torque on Bolts	Comments and Results
7.	Fig. 5 steel to steel	3.489" I.D. .070" W.	155 in-lb (Flange edges touch at 50 in-lb)	Pressure cycled 1 atm to 160 psig 40 times during cooldown, 25 times at 76°K. Temp cycled 25 times. No leak.
8.	ditto	3.350" I.D. .070" W	100 in-lb (Flange edges touch at 50 in-lb)	OK warm, held 1 atm at 76°K Small leak at 150 psig, 76°K
41 & 59	Fig. 4 steel to Al	2.614" I.D. .070" W	135 in-lb	Al. not anodized. OK warm. No leak at 76°K, 240 psig: No leak at 20°K, 150 psig.
56a.	Fig. 5 steel to Al	2.864" I.D. .070" W	125 in-lb	Al. not anodized. Used sleeve. OK at 150 psig warm. Leaked during cooldown.
56b.	ditto	ditto	150 in-lb	Same as 56a except torque. No leak warm or 76°K.
60.	ditto	ditto	ditto	Newly machined steel flange. Used sleeve. No leak warm or 76°K, 225 psig.

(Table 2. Continued on next page)

Parco Hypalon O-Rings (Compound 921-50)

	Flange	O-Ring Size	Torque on Bolts	Comments and Results
37.	Fig. 5 steel to Al	3.239" I.D. .070" W	150 in-lb	Al. not anodized. Temp cycled 5 times, pressure cycled 10 times. No leak at 76°K, 150 psig.
58.	ditto	ditto	ditto	Al. not anodized. Used retaining sleeve. No leak at 150 psig and room temp, 76°K, 20°K.

Parco Viton O-Ring (Compound 949-60)

	Flange	O-Ring Size	Torque on Bolts	Comments and Results
61.	Fig. 5 steel to Al.	2.864" I.D. .070" W	125 in-lb	Al. not anodized. Used retaining sleeve. No leak at 150 psig, room temp and 76°K.
62.	ditto	ditto	ditto	ditto

Double Mylar with Nichrome Wire Between

	Flange	O-Ring Size	Torque on Bolts	Comments and Results
9.	Fig. 5 steel to steel	.0075" Mylar .065" Nichrome 3-1/4" Ring. Diam.	100 in-lb	15 hot water to LN_2 cycles. 160°F overnight bake. 40 cycles, warm and cold, 150 psig to 1 atm. No leak at 250 psig room temp. No leak at 150 psig 76°K.

(Table 2. Continued on next page)

Double Mylar with Nichrome Wire Between

	Flange	O-Ring Size	Torque on Bolts	Comments and Results
11.	Fig. 5 steel to steel	.0075" Mylar .065" Nichrome 3-1/4" Ring. Diam.	75 in-lb	ditto Test 9 except no 160°F bake. No leaks warm or 76°K. 80 pressure cycles.
12a.	ditto	ditto	60 in-lb	Slight leak both warm and 76°K at 150 psig.
12b.	ditto	ditto	70 in-lb	No leak warm and 76°K at 150 psig. Leaked cold at 240 psig.
10.	ditto	.005" Mylar .065" Nichrome 3-1/4" Ring Diam.	75 in-lb	Leaked warm. Mylar cracked when increasing torque.
38.	Fig. 5 steel to steel	.0075" Mylar .050" Nichrome 3-3/16" Ring Diam.	100 in-lb	OK warm, leaked cold. Warmed and torqued to 125 in-lb, which cut through the Mylar.
40.	Fig. 4 steel to Al	.0075" Mylar .050" Nichrome 2-11/32" Ring Diam.	75 in-lb	Leaked warm. Torqued to 80 in-lb, which cut through the Mylar.

(Table 2. Continued on next page)

Miscellaneous Seals

	Flange	Seal Description	Torque on Bolts	Comments and Results
32.	Fig. 5 steel to steel	Teflon[R1] -coated Pressure actuating Metal core seal[(1)]	Low. Squeezed to spacer thickness	Seal supplied by Convair. Leak approx. 17 cc/hr at 150 psig, 76°K
46.	Fig. 4 steel to Al	ditto	ditto	Al. anodized and polished. Leak at room temp, 150 psig, was 390 cc/hr.
42.	ditto	ditto	ditto	Al. not anodized. Leak rate at 76°K, 150 psig, was 80 cc/hr.
64.	Fig. 5 steel to Al	Fluorogreen[R2] O-Ring 3" I.D. .070" W.	100, 125, and 150 in-lb	Used sleeve. Req'd 150 in-lb torque to make room temp. seal. Then no leak at 250 psig, 76°K.
66.	ditto	ditto	150 in-lb	Used sleeve. Leaked approx. .04 cc/hr at 250 psig, room temp. Leaked more than .5 cc/hr at 76°K, 175 psig

R 1 du Pont trademark
R 2 Trademark of John L. Dore' Co. for their modified Teflon product.
(1) Seal designated "Rayco", supplied to Convair Astronautics by the Fluorocarbon Co.

An attempt was made to study this spring loading effect with the aid of cone-shaped washers having a known spring constant. The .070"W O-rings of Hypalon compound 921-50 were loaded through 6 of these washers having a spring constant of 61.5 lb/mil. An apparent squeeze of 80% required to hold the seal without washers was reduced to 74% when loaded through the washers. Total force on the 1" diameter O-ring was 6300 pounds, or 2000 pounds per linear inch of original circumference.

Although heavy flanges and bolts were used for this experiment there was still some flexing of the parts, which complicated accurate measurement of O-ring thickness. Some very massive plates are presently being fabricated to continue this study. They will be used with oversize bolts and instrumented with strain gauges to measure the forces on the O-ring. This should make possible a more accurate comparison of O-ring seals made with and without the effects of spring loading.

The Fluorogreen[R] seal shown under Miscellaneous Seals in Table 2 holds some promise for applications which cannot use elastomers because of oxygen incompatibility. This modified Teflon[R] material made a good seal in our first test, when used in the same way as elastomer O-rings, even though the plastic was permanently deformed to the shape shown in Figure 3. A second identical test, however, was not satisfactory. The leak rate was not excessive, but it built up slowly at 76°K, and was still increasing when it exceeded the scale of the helium leak detector. It is of course possible that the second O-ring was defective, but at present the tests of this material are inconclusive.

In summary, the following recommendations are based on our present knowledge of the art of cryogenic sealing with elastomer or reinforced Teflon O-rings:

1. Use simple flat flanges with sufficient stiffness to prevent bowing between bolts, but a slight conical flexing of flanges or stretching of bolts (which does not exceed the elastic limit of the materials) is not objectionable and will tend to reduce the required flange loading. Design the flange and bolts

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for a load of about 2000 pounds of force per linear inch of original O-ring circumference. The insides of the bolt holes should not intersect the compressed O-ring, but should be kept as close to the O-ring as practical.

2. A normal machined flange surface is suitable. All traces of oil or grease should be removed from the flange faces with solvent.

3. Choose an O-ring (see Table 2 for suggested materials) having a cross section diameter of .070" and a mean ring diameter slightly less than the I. D. of the flanges.

4. Stretch the O-ring around a short thinwalled tube (same material as the flanges) which fits snugly inside the flange I. D.

5. Slip this retaining tube inside one flange, with the O-ring resting on the inside shoulder of the flange.

6. Bring the mating flange into position and cross-tighten the bolts until the O-ring is compressed to 75 to 80%.

7. The O-ring will flatten and spread outward at the same time that is is forced tightly against the retaining sleeve. Slight extrusion around the inside corners of the flanges is not objectionable.

8. A .014" thick spacer ring or an equivalent machined step in one of the flange faces will help control the O-ring compression, and will eliminate the need for taking bolt torque readings or O-ring compression measurements.

2. Properties Program

2.1 Compression Testing of Some Composite Inorganic Seal Materials

2.1.1 Introduction

The Armour Research Foundation materials testing laboratory has investigated the possibility of combining the structural properties of hard metals with the conforming properties of soft, ductile metals to be used primarily for high temperature seals. In coordination with this effort N. B. S. - C. E. L. has tested several compounds supplied by Armour to determine their low temperature behavior. Sealability tests have been reported previously; in brief, it was found that gaskets containing the metal indium as the soft impregnate made leak tight high vacuum, high pressure seals at temperatures as low as 20°K. The testing conditions and flange designs were similar to those previously used in the functional elastomer seal tests.

The promising results obtained from these tests prompted preliminary investigations of the mechanical and viscous behaviors of the composites, at room temperature and at 76°K. It is hoped that comparisons between results at various test temperatures will aid in understanding the composite structure and its possible use for low temperature seals.

TABLE 3 Armour Research Test Specimens

Sample	A	B	C	D	E
Skeleton	430 ss*	430 ss	304 ss	Molybdenum	430 ss
Skeleton bonded?	Yes	Yes	Yes	Yes	No
Skeleton density	23%	23%	22%	31%	23%
Impregnate	indium	indium	indium	silver	silicone resin slurry

* Stainless Steel

2.1.2 Materials and Apparatus

The five samples tested were supplied by Armour; they are designated A, B, C, D, E in Table 3 and were cylindrical approximately 1 in ^{2} x 1/2 in. long, before testing. Since only a limited number of tests could be performed on each sample, stress-strain curves in compression and load relaxation were chosen to describe the behavior of the materials. Tests were performed using an Instron universal testing machine and graphs of applied load vs. time were made using the Instron recorder. Crosshead speed was maintained at a constant rate of 0.002 inches per minute during all tests.

Figure 6 shows the compression cryostat and loading members. The overall deformation was calculated from the relative time rates of the chart and the crosshead. Deformation of the loading members was measured, and subtracted from the overall to determine the actual sample deflection. The loading members were responsible for more than 60% of the total deflection in most cases; consequently the measurements are not quantitatively accurate. This is particularly true at stresses less than 200 psi. In order to use the Instron machine directly in compression a load cell was mounted on the bottom of the moving crosshead, and the compression cryostat was placed on a large plate mounted on the base of the machine.

2.1.3 Testing Procedure

Two samples of the 430 ss + Indium composite and one each of the other three composites were available for testing; therefore more tests were performed on the 430 ss + Indium than on the others. The tests were performed and reported by T. F. Durham of the Mechanical Properties section of C. E. L. The deformations refer to total deflection. Thus "the sample was deformed 5%" means that the total deformation of the sample and the loading rods was 0.05 x 0.5 = 0.025 inches. Following is Mr. Durham's report of test procedure on each sample:

> Specimen A: Nominally 0.505" thick with a diameter of 1.130". All loads were applied at room temperature and the sample was deformed successively to 1%, 5% and 20% deflection with stress relaxation curves of 1 minute duration and unloading curves obtained for each test. Final thickness of the specimen was 0.417 inches. The sample was then reloaded to obtain a stress relaxation curve of 10 minutes at 20% deflection. No further measurements were made.
>
> Specimen B: Nominally 0.505" thick with a diameter of 1.130". All loads were applied at 76°K and the sample was deformed successively to 5% and 6%. Stress relaxation curves of 1 min. and 6-1/2 min. respectively, and unloading curves, were obtained. Deforming this sample beyond 6% would have involved loads much greater than 5000 lbs and this was considered undesirable with the particular loading apparatus being used. Also

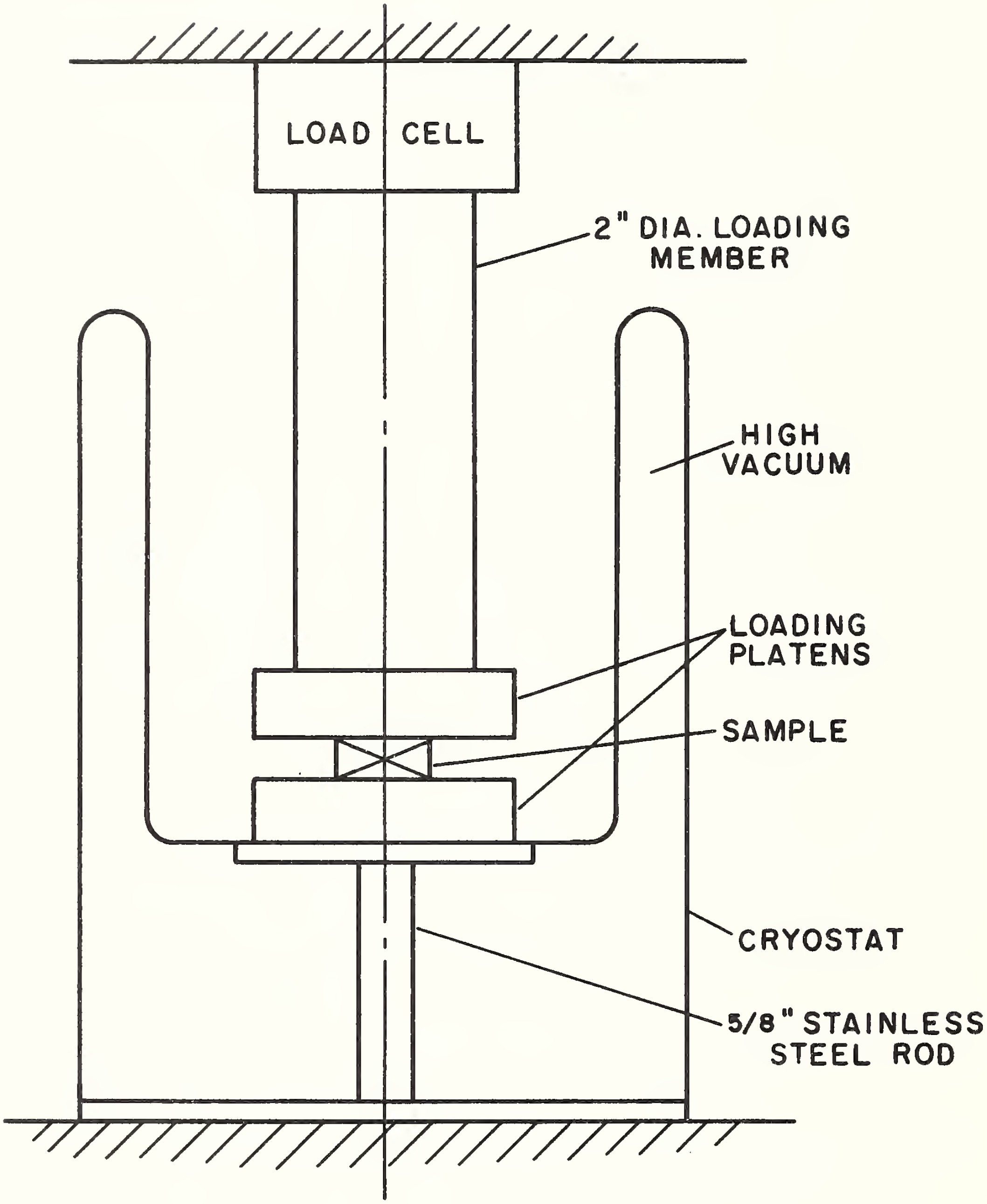

Figure 6. Compression Cryostat and Loading Members

the sample was thought to have been deflected to 1% but this amount was later found to be "take up" in the loading members. On all subsequent tests this "take up" was compensated for. The sample was permanently deformed 0. 012".

Specimen C: Nominally 0. 505" thick with a diameter of 1. 130". At room temperature the specimen was deformed 1%. It was then cooled to 76°K and again deformed 1%, and following this it was deformed 5%. Stress relaxation curves of sufficient duration to establish the behavior, and unloading curves, were obtained for each test. After warmup the specimen was found to have sustained only about 0. 001" permanent deformation.

Specimen D: Nominally 0. 502" thick with a diameter of 1. 130". At room temperature the sample was deformed 1%. It was then cooled to 76°K and deformed successively 1%, 5% and again 1%. Stress relaxation curves for periods up to 20 minutes, and unloading curves, were obtained for all tests. After warm-up the specimen was found to have sustained only about 0. 001" permanent deformation.

Specimen E: Nominally 0. 502" thick with a diameter of 1. 130". The specimen was deformed 1% at room temperature and then 1% and 5% at 76°K. Stress relaxation curves for periods of about 3 to 10 minutes and unloading curves were obtained. There was approximately 0. 003 inches permanent deformation.

2. 1. 4 Discussion of Results

Since the deformation of the loading members played an important part in the total deflection recorded on the original graphs, the data were translated to working curves and these corrected curves plotted in Figures 7 thru 12. In all cases the stress was calculated on the basis of the initial cross section area of the samples.

Figure 7 shows room temperature loading curves for each sample. Only sample A was loaded extensively at room temperature, and a complete curve for this test is shown in Figure 8. With reference to Figure 7, it should be re-emphasized that difficulties in measuring the exact deformation of the loading members in this range of loads cause some doubt as to the quantitative value of the curves. However, the shape of the curves should be valid. The steadily increasing slope was not observed in tests by Armour at room temperature shown in WADC

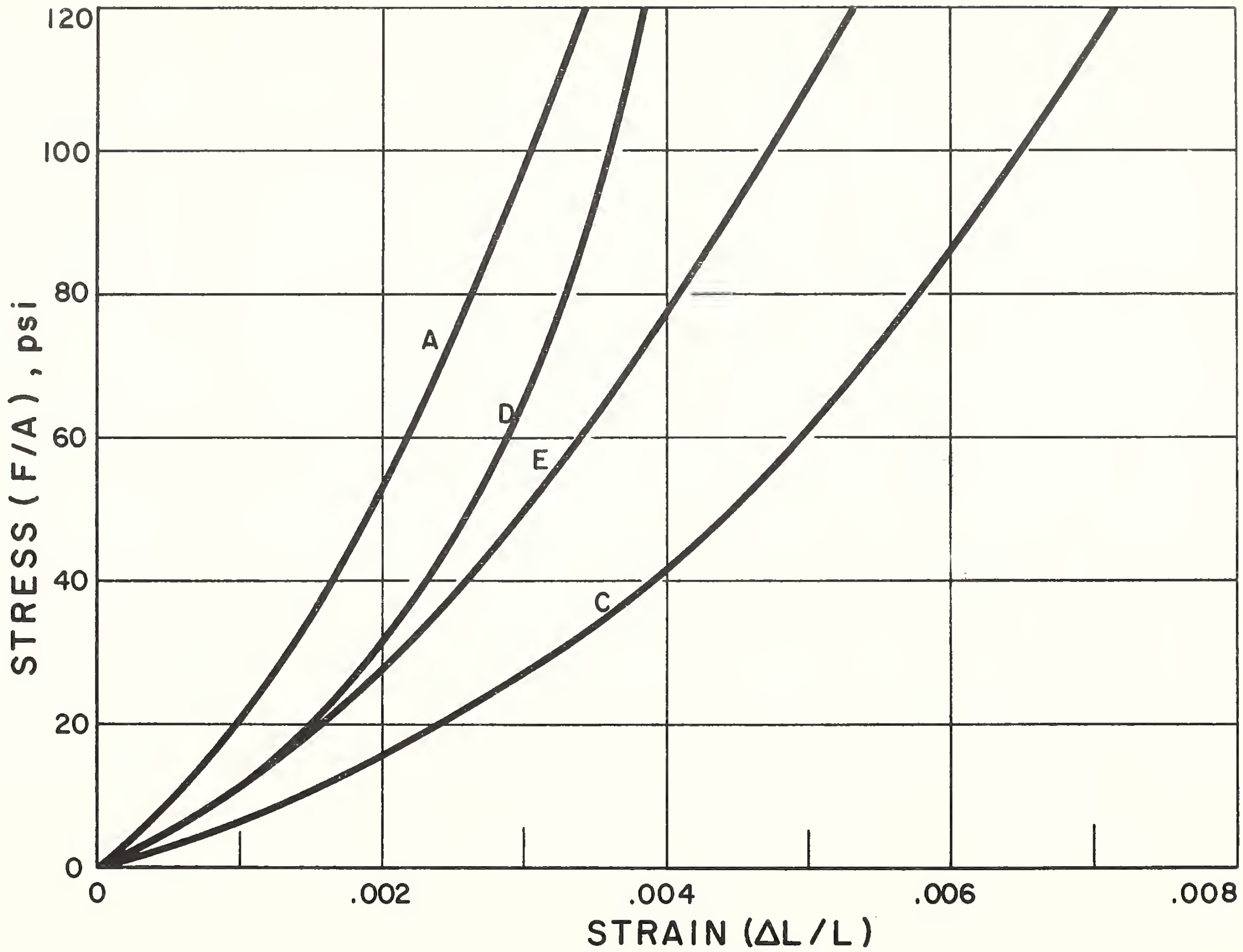

Figure 7. Room Temperature Compression Loading of Inorganic Composites

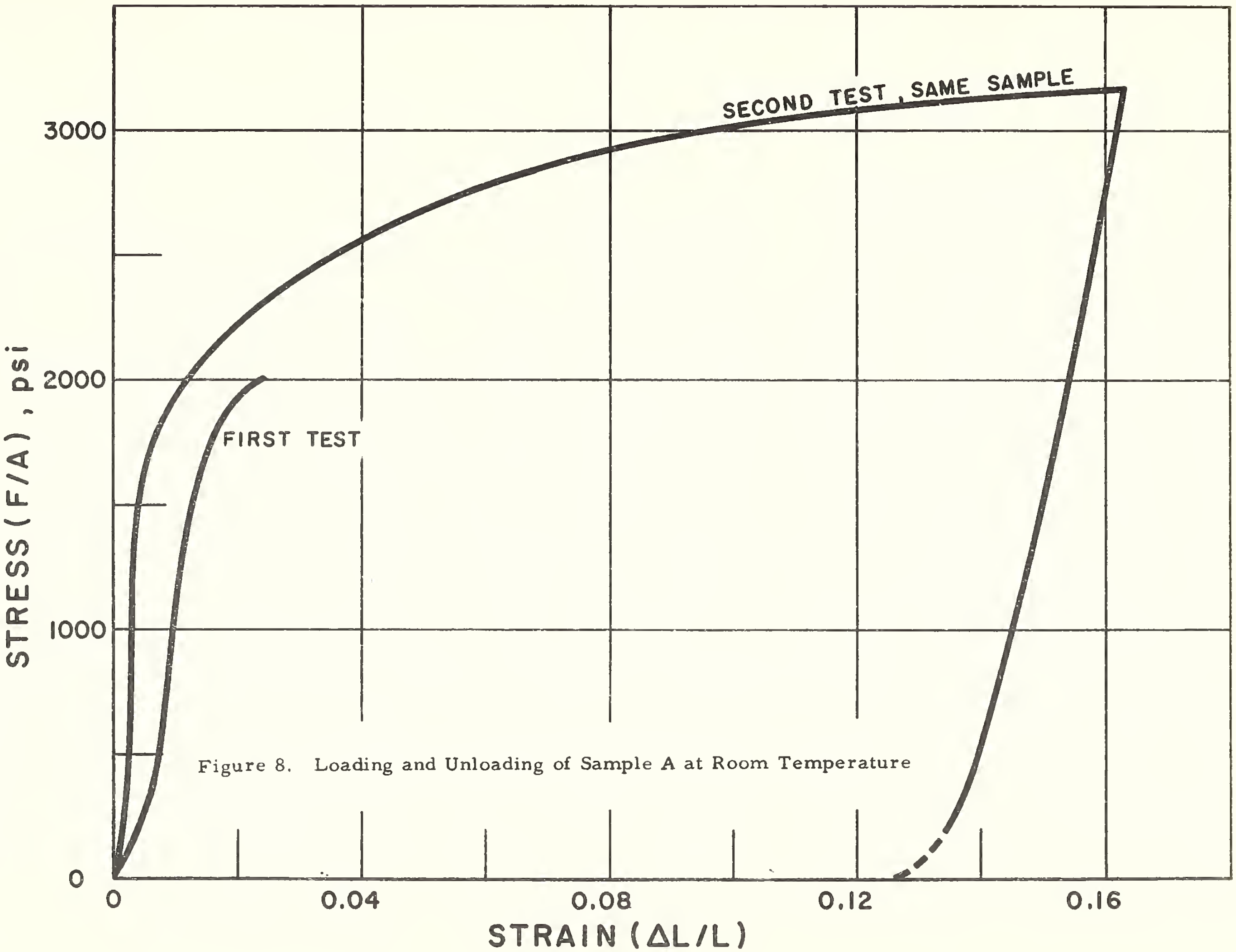

Figure 8. Loading and Unloading of Sample A at Room Temperature

Tech. Report 59-338, Part I. The increasing slope in the NBS curves (Figure 7) is probably due to two effects not representative of the materials as a whole. These are 1) initial compression of surface irregularities, which reduces the actual loading area; 2) compression of a film of the soft impregnant which is closest to the surfaces. In other words, the main body of the sample does not "see" the entire applied load until these initial effects have been overcome. However, they cannot be disregarded, since they are present each time a sample, or a gasket type seal, is compressed at room temperature. The strain rate also plays an important role in the measurements. The curves might have entirely different characteristics if the strain rate were increased or decreased by a factor of five. This could also be a reason for discrepancies between the NBS tests and the Armour tests. The Armour tests were taken with a constant rate of stress during the loading period, whereas the present testing used a constant rate of strain.

Figure 8 shows two tests run on sample A at room temperature, including the unloading portion of the second test. Hysteresis in the loading rods prevented accurate analysis of the unloading curves in general; however, in Figure 8 the deformation was much greater than that for other tests, and some analysis of the recovery was possible. Note that stress was calculated on the basis of the initial cross-section of the sample, not a good approximation for this test since the area increased significantly during the loading period. Note also the yield for both tests, and the non-recoverable plastic flow after yield for the second test.

Figures 9 and 10 show loading curves taken at 76°K. The curve for sample C, 304 ss + Indium, was actually the second test performed on the sample, and therefore can be compared with the second test performed on sample B, 430 ss + Indium. It can be seen that the stronger 430 skeleton caused a higher yield strength, but after yield the indium was the more predominant material and the slopes of the curves were similar. Sample D did not show a yield probably due to the greater skeleton density and higher strength of the impregnate. The non-bonded skeleton of sample E was not as strong, and behaved in a manner similar to the post-yield curves of samples B and C. Permanent deformation was somewhat greater for this sample, but even so most of the deflection was recovered.

After a seal has been assembled, it is important that most of the initial sealing force be maintained throughout the use of the seal.

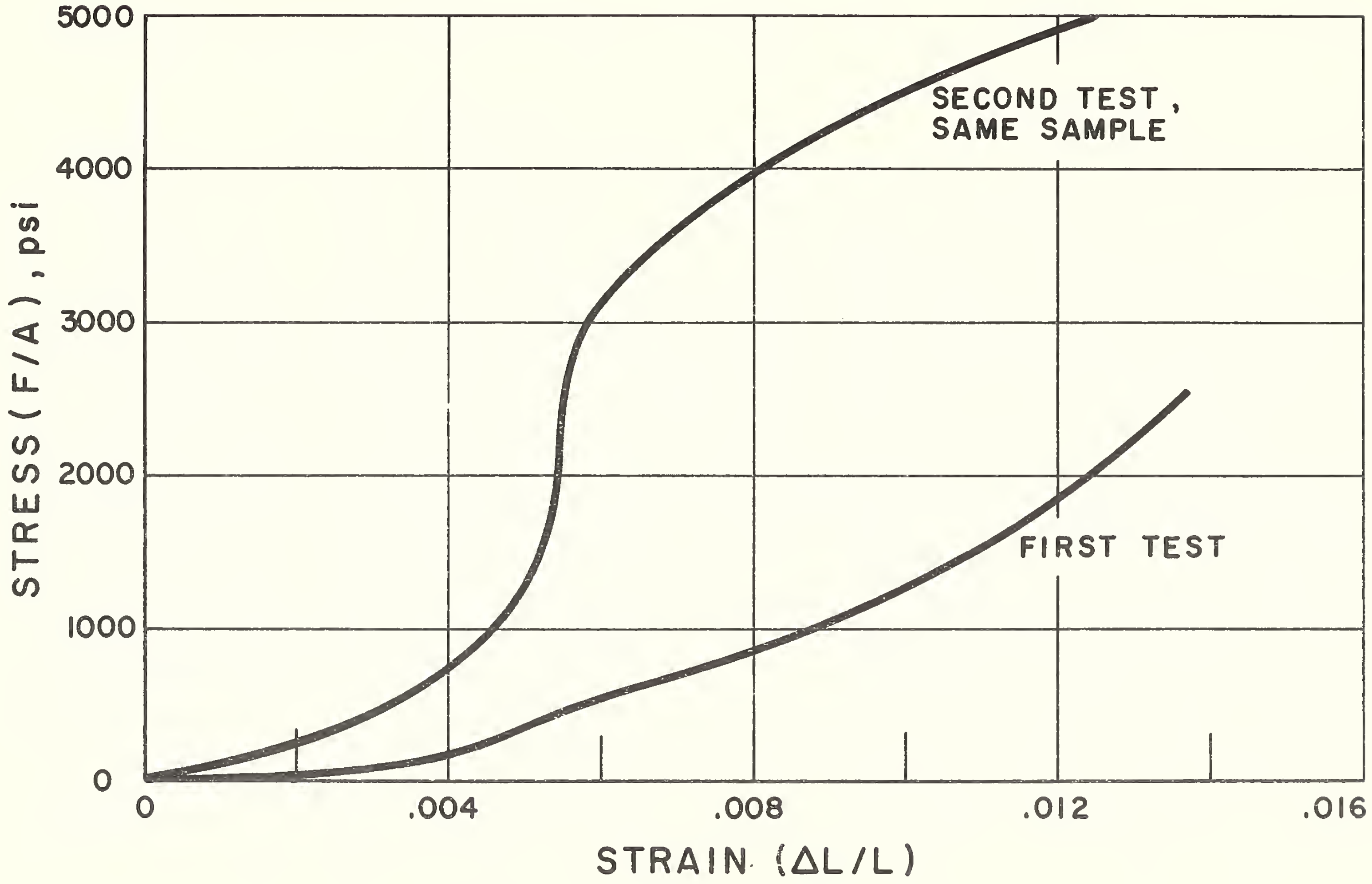

Figure 9. Loading Curve for Sample B at 76°K

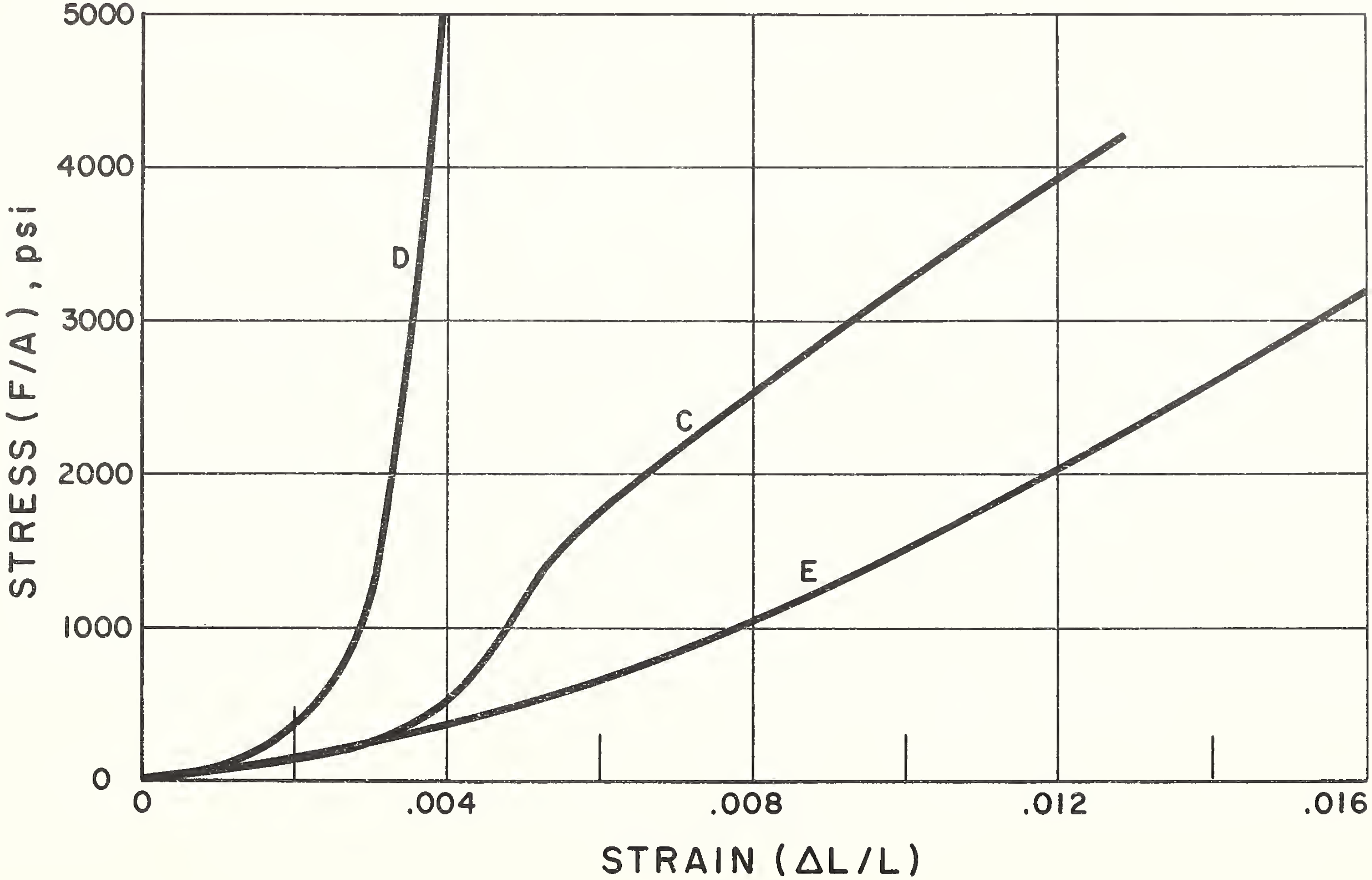

Figure 10. Loading Curves for Samples D, C. and E. at 76°K

Hence some knowledge of the viscoelastic behaviors of the composite inorganic seal materials is important to the design engineer. To this end stress relaxation at constant strain was measured as a function of time during the stress-strain tests described above. Figures 11 and 12 show the results of these tests at room temperature and at 76°K respectfully. Sample D was omitted from Figure 11 because no relaxation was measured from a stress below 200psi; however, the relaxation was very small - from 450 psi to 435 psi in 4 minutes, with a shape similar to that of sample E, Figure 11. Sample B was omitted from Figure 12 because irregularities in the testing procedure are believed to have affected the results.

During the summer months continuous expansivities of the composites from room temperature to 76°K will be measured using a continuously recording dilatometer now under development. Also the 304 ss+Indium composite, which exhibited some anomalies not presented here, will be investigated further if time permits.

2.2 Properties of Elastomers

2.2.1 Compression Dilatometer

Work with the compression dilatometer described in our March 1 Progress Report has continued during the present reporting period, and will be discussed briefly at this time. These experiments have as their objective the understanding of the mechanism involved when an elastomer is compressed at room temperature and subsequently cooled to a low temperature - in this case 76°K. The four variables pertinent to the mechanism are temperature, applied stress, strain, and time. A variation in any one of these produces changes in some of the others. The apparatus was designed to keep the applied stress constant during a given test, thereby allowing some discussion of the relations between the other variables.

The process begins when the constant stress is applied at room temperature. Even before the sample is cooled, a time-dependent deformation begins: as time increases, the sample contracts. Other investigators have shown deformations at around 300°K due to shear strain to be a linear function of log time in the range one minute to one week, and the same dependence is expected in compression. This continued deformation, or creep, is caused by re-orientation of secondary bonds between molecules, which continually seek a more relaxed

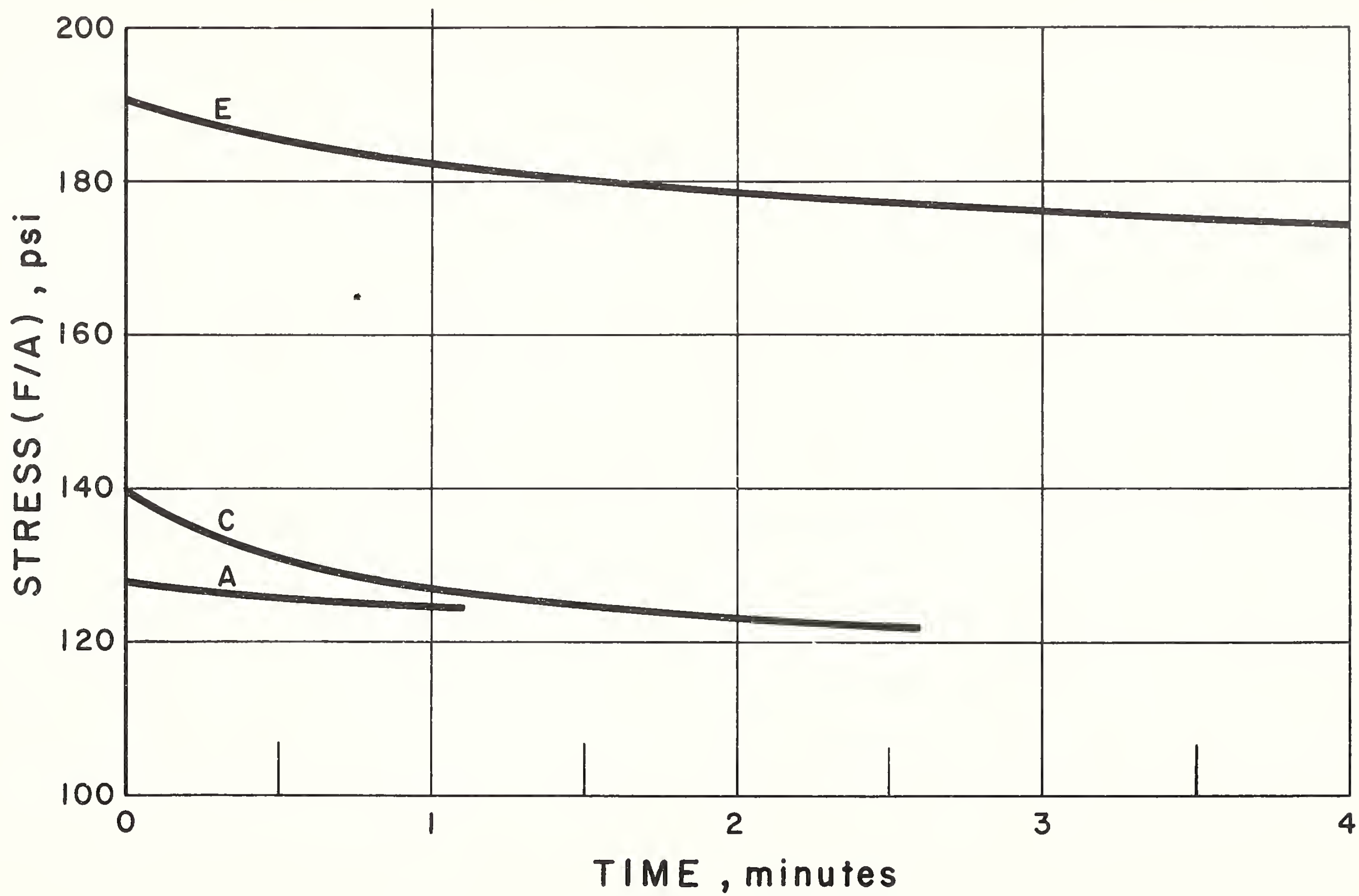

Figure 11. Relaxation of Compressed Composites at Constant Strain, Room Temperature

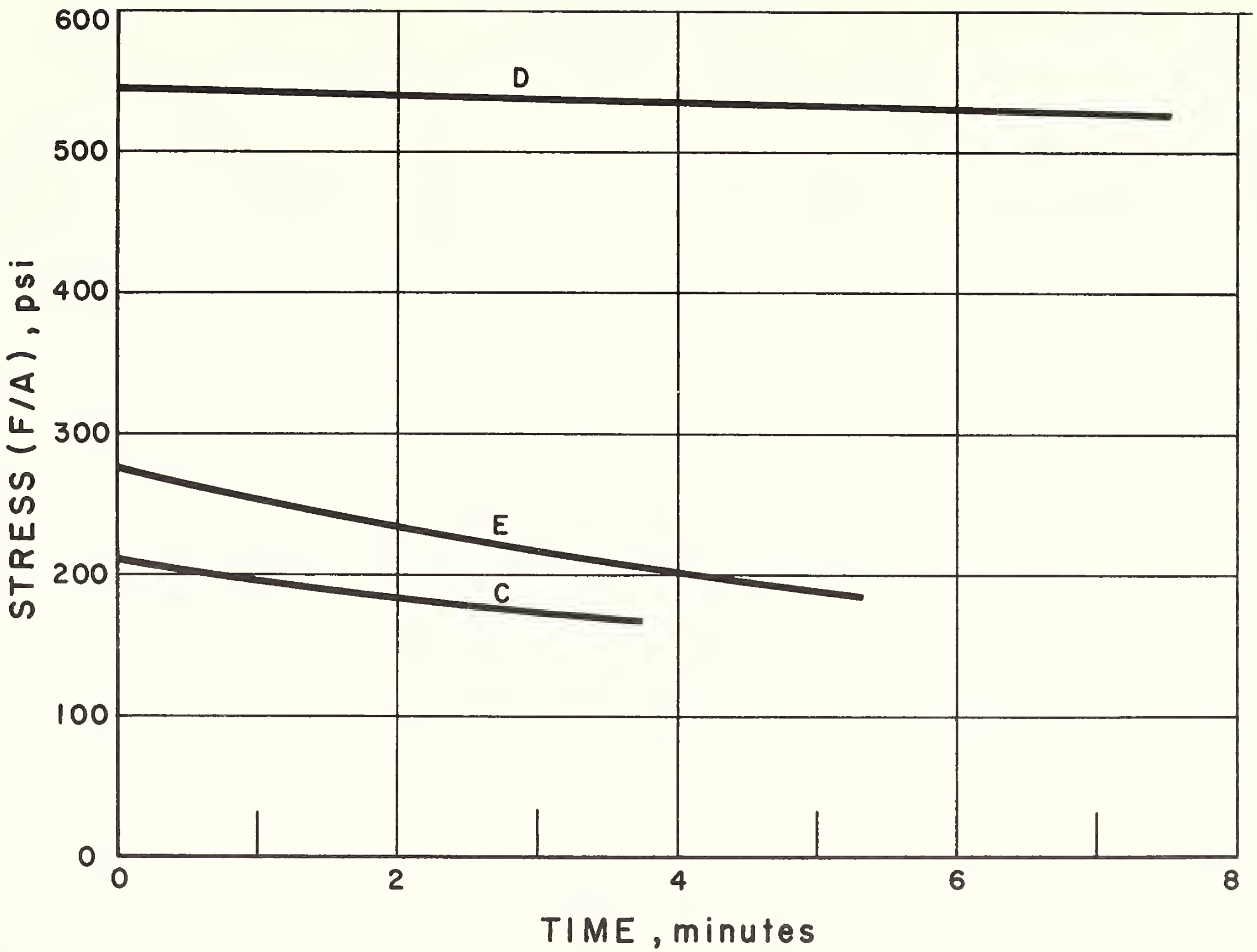

Figure 12. Relaxation of Compressed Composites at Constant Strain, 76°K

position. Cission of primary cross-links between the long chain molecules and re-orientation of chain entanglements also, to a lesser extent, cause the creep phenomenon.

As the elastomer starts to cool creep continues at a somewhat reduced rate, and at least two other effects add to the overall contraction. First, the normal thermal expansion causes shrinkage; it has not been determined whether this so-called normal expansion (or contraction, in our case) is changed by the configurational changes in the compressed sample. Secondly, the vibration of the atoms becomes of smaller amplitude, causing the molecular configuration to tend toward a more ordered state. Since, at any temperature, the equilibrium position with no force applied is the configuration of maximum entropy or highest disorder, it follows that the amount a sample contracts when subjected to a constant force increases with decreasing temperature. This effect is referred to as the Gough-Joule effect, and adds to the overall contraction of the sample.

The above effects change radically when the elastomer reaches its glassy-state transition temperature. At this point, random thermal motion of the chains is inhibited by the fact that free rotation of chain elements about single bonds will not take place at an appreciable rate, and the material becomes hard and rigid like a glass. At temperatures below this point (T_g) the behavior corresponds to that of an ordinary solid, and the entropy has a negligible effect on force-length-temperature relations. Therefore the Gough-Joule effect, as well as the creep effect, do not appear below T_g. Only the normal thermal expansion below the brittle point should be present.

Figures 13 and 14 show some results obtained using Viton[R] A, a co-polymer of hexafluoropropylene and vinylidene fluoride. It should be mentioned that the results are still preliminary in nature, and thus should be considered qualitative rather than quantitative. Figure 13 is a plot of the contraction of a 1 in^2 x 1/2 in compression set button as a function of time. Before cool-down the contraction was a linear function of log time, which agrees with the results in shear as previously mentioned. After cool-down was begun, the normal thermal contraction and Gough-Joule effect added to the already rapid contraction until the glassy-state transition was complete, after which only a small change in length was observed, not discernible on the scale used in this plot.

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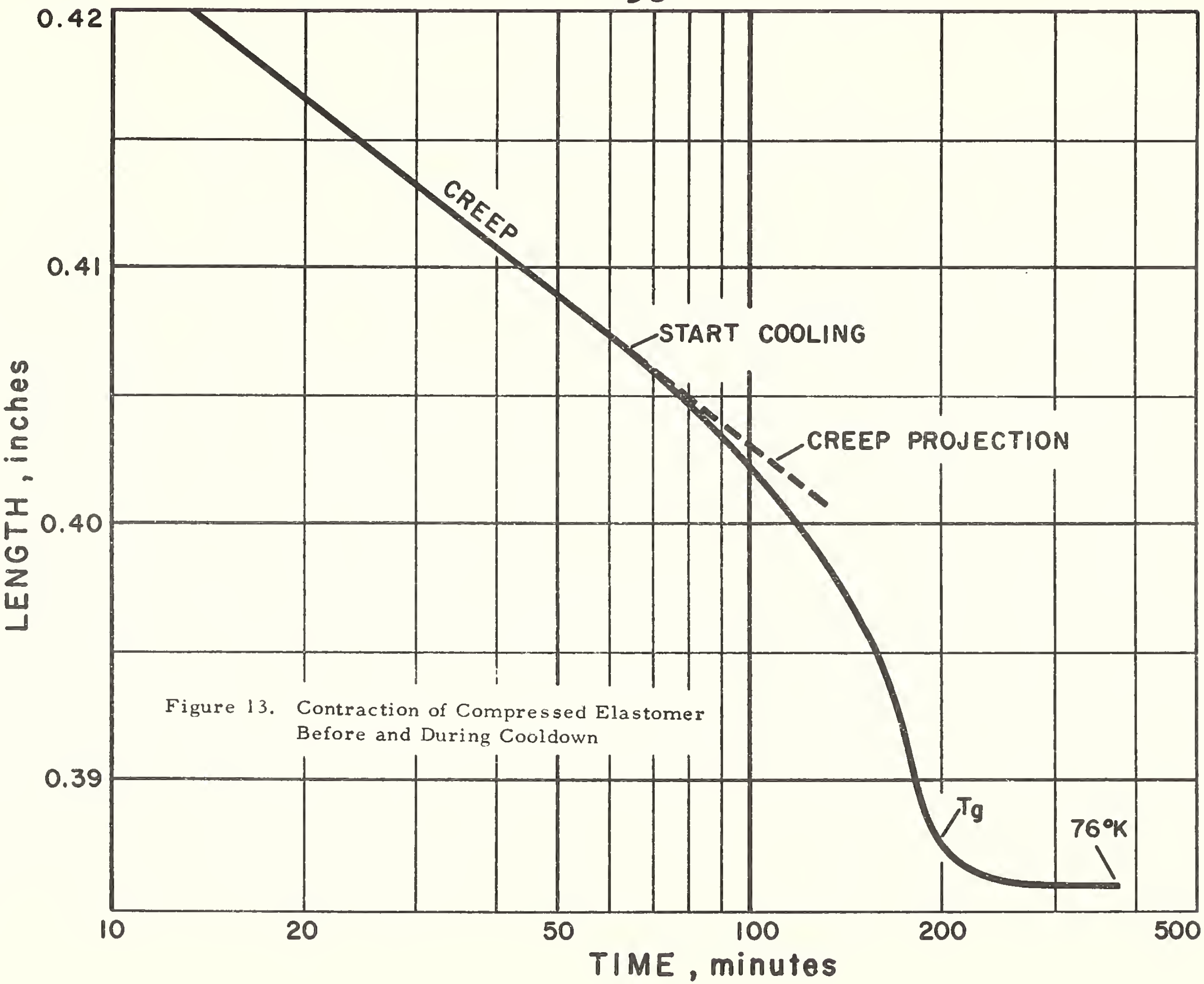

Figure 13. Contraction of Compressed Elastomer Before and During Cooldown

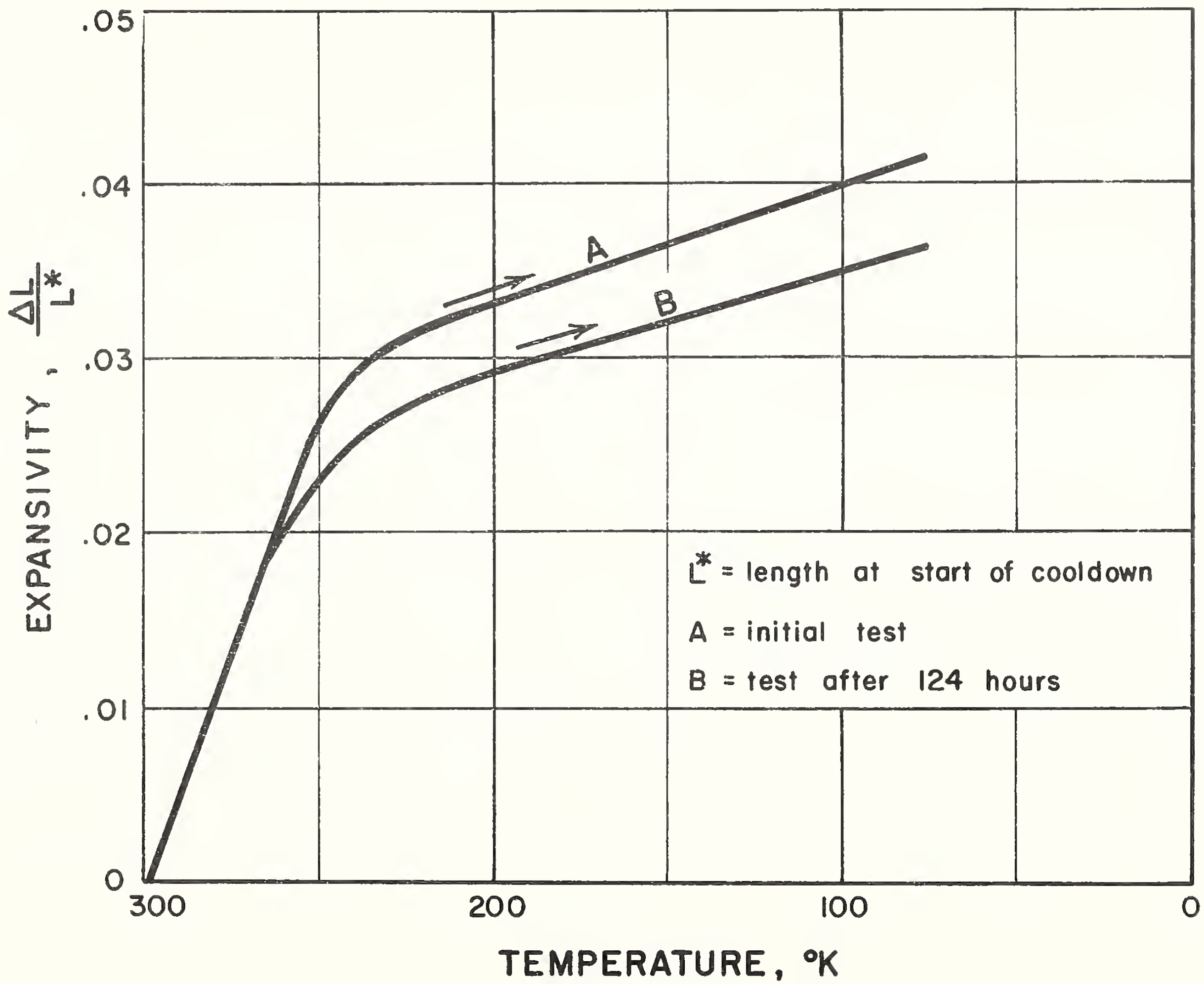

Figure 14. Effect of Conditioning by Sustained Strain Before Cool-down

Figure 14 shows how the overall contraction of the material can be reduced by keeping a small strain on the sample for several days. During the pre-stressing period at room temperature the strain was about 40 psi, and resulted in an appreciable reduction in contraction during cooldown. It is believed that this small strain could not have induced crystallinity, or otherwise affected the true properties of the elastomer. The reduction is attributed solely to diminishing the contribution of creep. The obvious conclusion is that if an O-ring were stressed prior to actual use (by assembling the seal ahead of time, for example) and then re-stressed just before using (by re-tightening the bolts) it would effect a satisfactory seal at lower temperatures than if the prestressing were not performed. Alternately, a smaller squeeze (and smaller bolt torque) would effect a good seal at some given temperature. This result was observed in a few of the functional seal tests, but has not been studied with specific reference to creep effects.

2.2.2 Properties Program Plans

Two sets of elastomer samples have been received from the Elastomer Section of Materials Central. Other elastomer and plastic samples will be compounded and supplied as needed. These are being made in the form of compression buttons, expansivity cylinders, and O-rings. Present plans call for continuation of the compression dilatometer and O-ring sealability studies, using these newly available samples. In addition, two new tests are under development.

The first of these, for which the apparatus is about 80% complete, is a dilatometer for continuously measuring and recording the temperature-length relation of a 1/2" x 2" long cylinder as it is slowly cooled from room temperature to 76°K and again warmed to room temperature. The slope of this temperature-length curve at any temperature will, of course, yield the linear coefficient of thermal expansion at the temperature. It is intended that this experiment will provide the basis for preliminary screening and comparison of the various elastomers and plastics.

The second test, for which the apparatus is about 50% complete, will use both O-rings and compression buttons. The object of these measurements will be a detailed study of the forces required to highly compress O-rings and test-buttons at room temperature, and the rate of decay of these forces as the temperature is lowered. The compressive force will be applied through temperature compensated and calibrated strain gauges, and temperature measurement will be by thermocouples. Both temperature and force will be continuously recorded by means of either an x, y recorder or a multipoint strip chart recorder.

3. Moving Seals

The apparatus for study of packings for slow-turning shafts is shown in its present form in Figure 15. It was necessary to go to the double seal arrangement shown in Figure 15 in order to eliminate end thrust on the shaft when pressure was applied to the seal. Attempts to oppose this thrust through a ball bearing were partially successful, but measurements of the torque required to turn the shaft were more difficult to interpret because the resistance of this bearing varied with pressure and temperature. Another important improvement in the present apparatus is lining the packing sleeves with molybdenum filled and reinforced Teflon. This has completely eliminated the siezing which would frequently occur when the stainless steel shaft and brass sleeve rubbed together.

The shaft is turned at 16 rpm through a variable clutch and flexible drive. These allow the tests to be performed with the jig clamped in a vice on the bench or, alternately, in a dewar of liquid nitrogen or hydrogen. Time has not yet permitted any extensive testing of packings with this improved apparatus, but preliminary tests with chevron type packings of Teflon indicate that the problem of bearing resistances has been solved.

Figure 16 is a semi-schematic sketch of the high speed rotating seal tester which is presently being detailed. Complete working drawings of this apparatus will be prepared, but machining and assembly of parts cannot be supported by our present budget.

Acknowledgement

We wish to acknowledge the important contribution of D. B. Chelton and L. E. Scott who planned and supervised most of the tests shown in Tables 1 and 2.

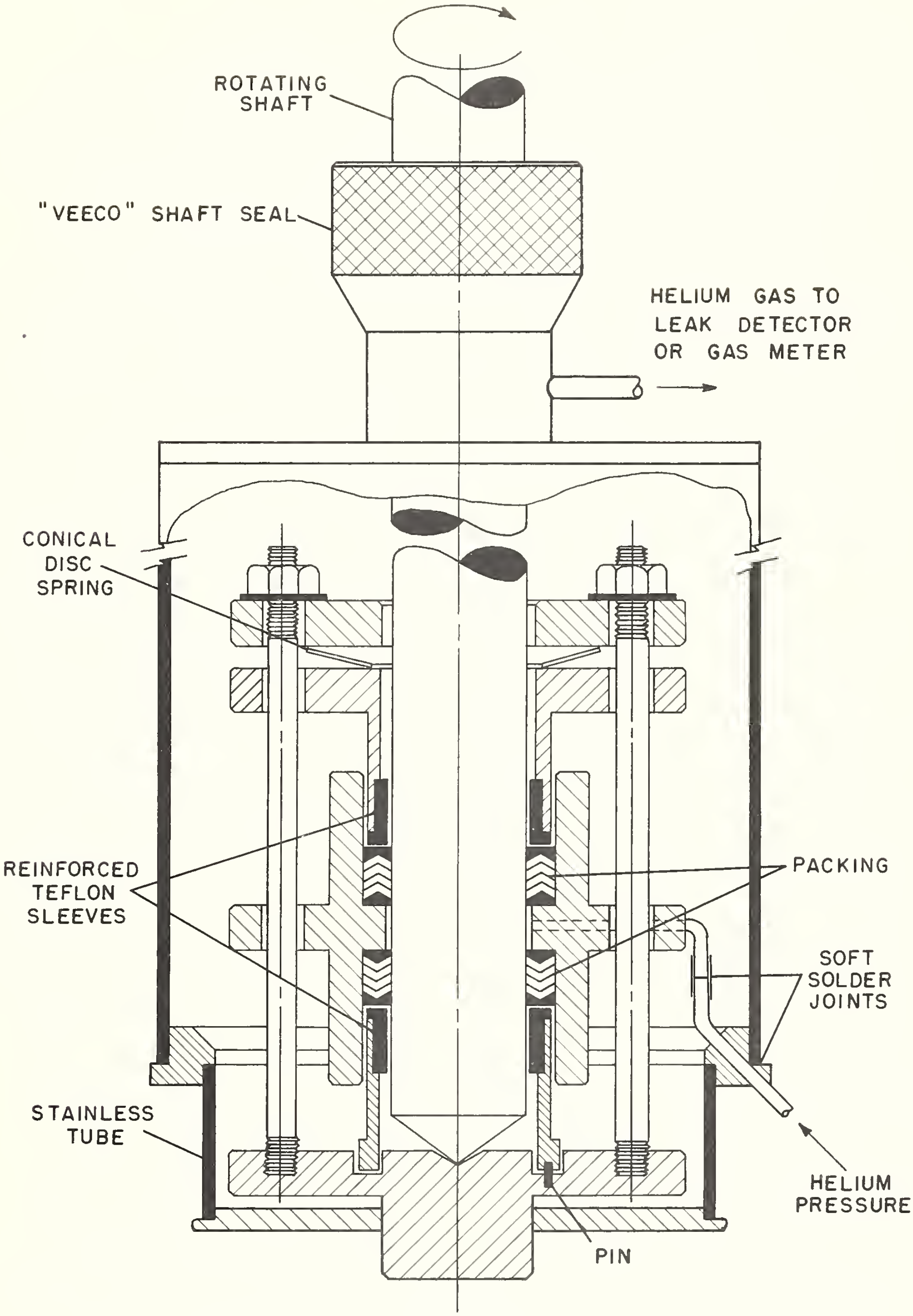

FIG. 15

Packing Tester for
Low-Speed Rotating Shaft

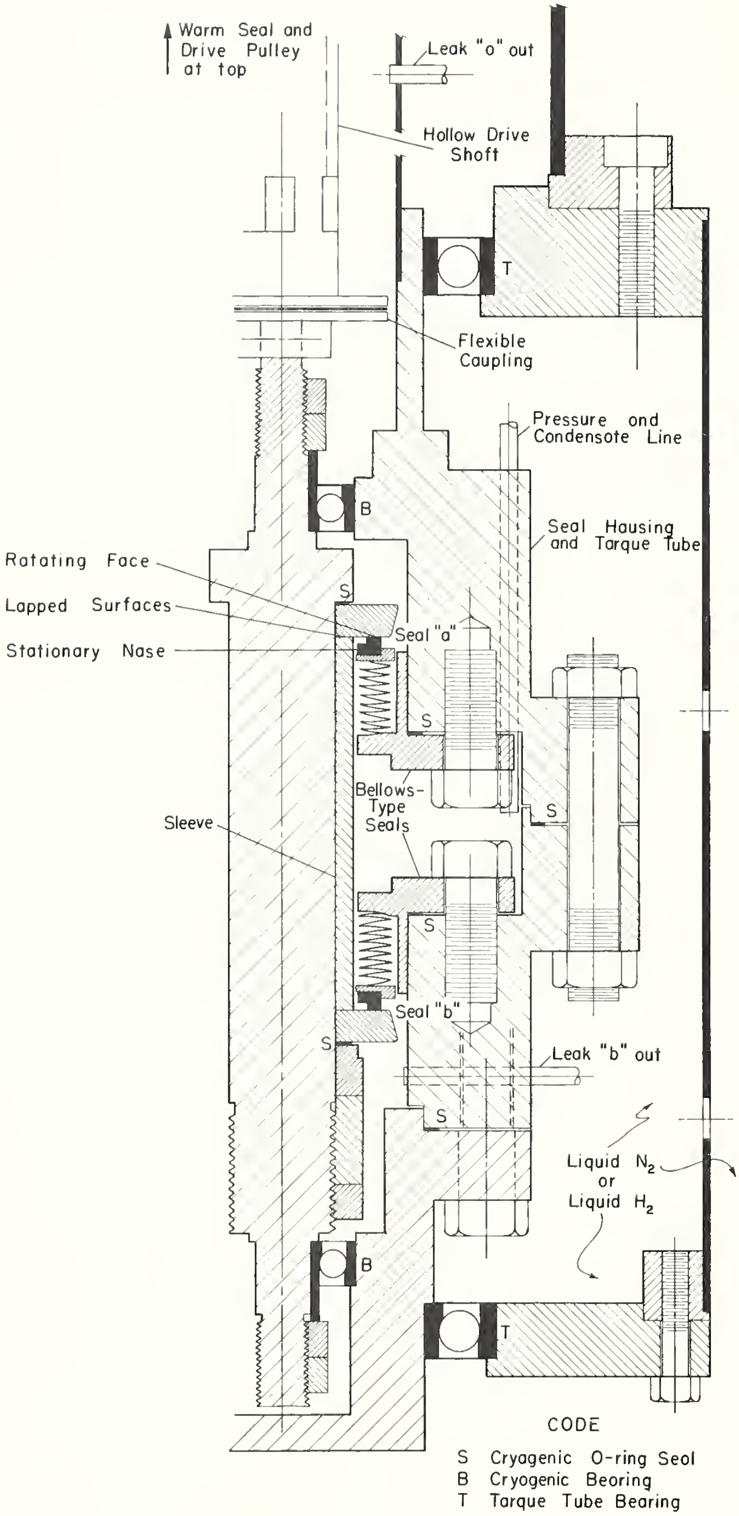

Fig. 16 High Speed Rotating Seal Tester

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