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6799

Third Progress Report

on

ELASTOMERIC SEALS AND MATERIALS AT CRYOGENIC TEMPERATURES

by

D. H. Weitzel, R. F. Robbins, P. R. Ludtke and Y. Ohori

U. S. DEPARTMENT OF COMMERCE
NATIONAL BUREAU OF STANDARDS
BOULDER LABORATORIES
Boulder, Colorado

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Foreword

This report was prepared by the National Bureau of Standards Cryogenic Engineering Laboratory under ASD Order No. (33-616) 61-04. It was administered under the direction of the Non-metallic 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

There has been progress on the development of a dilatometer for measurements of thermal expansions covering the range from room to cryogenic temperatures. The equipment is now operational and expansivity curves have been obtained for several elastomers. These measurements will continue and should give new insights into the behavior of polymeric materials as they are cooled through and below their brittle point temperatures.

A study of elastomer O-ring seals has been made under conditions of high compression unaffected by spring loads resulting from stretching of bolts or flexing of flanges. The results help to point up the importance of these factors in practical seal designs. The heavy fixture used for these tests is now being instrumented with strain gauges, and will be used to obtain basic data on the strain behavior of pre-stressed elastomers during cooldown to cryogenic temperatures. When samples are in the form of O-rings, the sealing characteristics will be obtained along with the strain-temperature information.

A torsion testing apparatus which was constructed earlier in this program has been used to make some measurements from which shear modulus can be obtained. The results should be useful in the comparison of low temperature characteristics of the materials supplied by Aeronautical Systems Division.

The rotating seal effort has been temporarily shelved pending clarification of funding for the coming year.

1. Heavy Plate Seal Tests

Shown in Figure 1 is a heavy plate fixture which was designed to test O-ring seals without the complications introduced by stretching of bolts or flexing of flanges. The flanges were made of 2 inch thick stainless steel, and compression was by means of six 3/4 inch diameter stainless steel studs. A machined depression in the center of one flange allowed the studs to be pulled up tight for metal-to-metal contact after the O-ring had been compressed the desired amount. The amount of squeeze (percent compression) of the O-ring was increased by placing thin metal discs in the bottom of the depression, or decreased by placing a thin metal insert between the flanges at the bolt circle. All surfaces were given a normal machine finish.

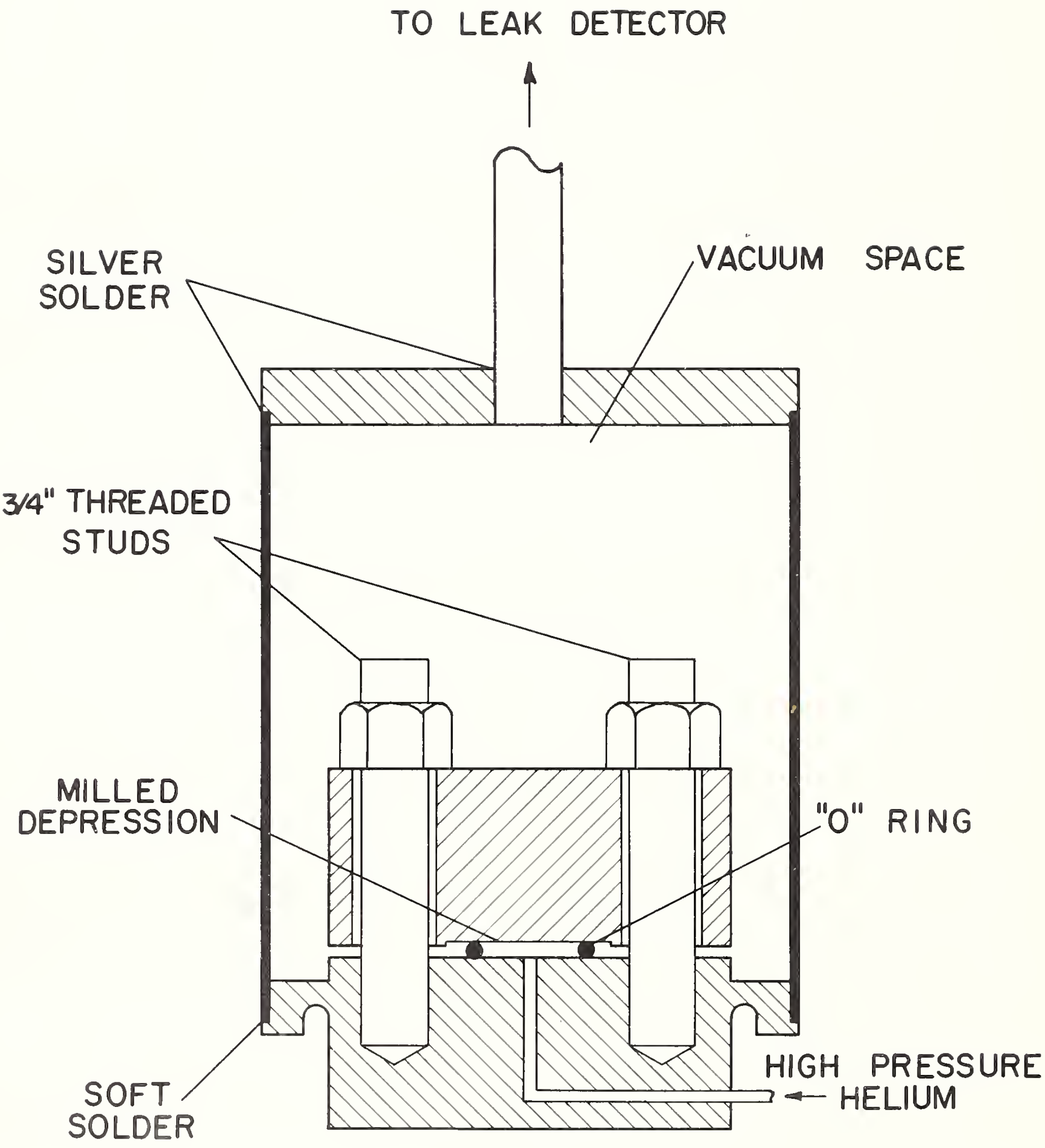

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Fig. 1

Heavy "O" Ring Seal Test Jig

With this heavy fixture, flexing of flanges was reduced to a negligible level through overdesign, and stress due to stretching of bolts was absorbed by metal-to-metal contact at the bolt circle. There remained the spring loading which results from compression of the steel flange faces. For high squeezes compression of metal above the O-ring was sufficient to result in a slight permanent depression showing that the elastic limit of stainless steel had been exceeded in the region immediately adjacent to the ring. There appeared to be no easy way to design around this effect, and it is present in all our tests. The amount of spring loading obtained in this way is very small, however, in comparison with that experienced with flanges of practical thickness.

It should be emphasized, of course, that spring loading inherent in most flange designs contributes to the effectiveness of O-ring seals for cryogenic use and thus should be utilized rather than eliminated. Since this has been a largely unknown and variable parameter, however, it has complicated the study of O-ring seal performance. Results with the oversize jig, even without strain gauge instrumentation, has been quite informative. These results are summarized in Table 1.

Commercial and Aeronautical Systems Division compounds* of chlorosulfonated polyethylene (hereafter called c.s.p.), natural rubber, vinylidene fluoride/perfluoropropylene (hereafter called v.f.p.), and neoprene, all of which have been good seal materials in previous functional tests, were tested in the heavy jig. The O-rings, in .070 and .140-inch thicknesses, were tested at progressively higher compressions until either a satisfactory low temperature seal was achieved or severe material failure resulted. Neither v.f.p. nor c.s.p. performed well under the conditions of this test. Apparently a certain amount of spring loading is required before either of these materials, as compounded in our samples, will make satisfactory cryogenic seals. The compounds based on natural rubber and neoprene, on the other hand, performed quite well without spring loading. In .070-inch width the latter two materials showed little or no material failure at 88% true compression, and made reliable high vacuum seals at temperatures from room temperature to between 76 and 93°K, and at pressures to 1250 psig. Satisfactory seals were also obtained with these compounds in .140-inch width O-rings, but only after compression reached the point of severe material failure.

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*For compounding recipes and original physical properties of A.S.D. materials see Table 2.

Compound	Shore A Hardness	O-Ring Width (in)	Percent Compression	Maximum Pressure (psi)	Material Failure	Pressure Cycles	Temp. Cycles	Seal Failure	Final or Failure Temp. (^{o}K)	Test Number
Commercial c.s.p.[(1)]	65	.070	70	100	None	0	0	Yes	153	1-H
ditto	65	.070	84	100	Severe	0	0	Yes	295	3-H
Commercial v.f.p.[(2)]	80	.070	70	100	None	0	0	Yes	280	7-H
ditto	80	.070	78	100	None	0	0	Yes	185	8-H
ditto	80	.070	88	1250	Severe	20	2	No	81	13-H
Commercial Natural	50	.070	84	100	None	1	1	Yes*	93	4-H
ditto	50	.070	88	1250	Slight	50	2	No	76-93	9-H
ditto	50	.070	91	1250	Severe	50	3	No	76-93	5-H
Commercial Neoprene	50	.070	84	100	None	0	0	Yes	128	12-H
ditto	50	.070	88	1250	Slight	20	2	No	76-93	11-H
ditto	50	.070	91	1250	Severe	50	2	No	76-93	10-H
Natural Rubber										
A.S.D. Comp. 8A	65	.140	70	100	None	0	0	Yes	153	3-P
ditto	65	.140	75	100	None	0	0	Yes	128	4-P
ditto	65	.140	80	100	Slight	0	0	Yes	122	5-P
ditto	65	.140	85	1250	Severe	0	0	No	76-93	13-P
Commercial Natural	50	.140	75	100	None	0	0	Yes	142	9-P
ditto	50	.140	80	100	None	0	0	Yes	142	10-P
ditto	50	.140	87	1250	Severe	30	2	No	76-93	11-P
Neoprene										
A.S.D. Comp. 8B	85	.140	70	1000	None	10	0	Yes**	93	1-P
ditto	85	.140	75	1250	Severe	30	2	No	76-93	2-P
Commercial Neoprene	50	.140	70	100	None	0	0	Yes	173	6-P
ditto	50	.140	75	100	None	0	0	Yes	135	7-P
ditto	50	.140	80	100	None	0	0	Yes	138	8-P
ditto	50	.140	85	1250	Severe	30	2	No	76-93	12-P
Commercial c.s.p.	65	.140	85	100	Severe	0	0	Yes	138	2-H
A.S.D. Comp. 8D(v.f.p.)	80	.140	70	100	Severe	0	0	Yes	173	16-P
ditto	80	.140	75	100	Severe	0	0	Yes	153	14-P
ditto	80	.140	80	100	Severe	0	0	Yes	210	15-P

(1) chlorosulfonated polyethylene

(2) vinylidene fluoride/perfluoropropylene

* tested cold after 3 days compressed at room temperature

** upon removal from bath and jarring on floor

Table 1. O-RING SEAL TEST DATA

A.S.D. No.	Polymer	Recipe		Original Properties: Tensile Strength (psi)	Original Properties: Ultimate Elongation (%)	Original Properties: Hardness (Shore A)	Original Properties: Compression Set* (%)	Polymer Trade Name
8D	Vinylidene fluoride and perfluoropropylene	Polymer Magnesium Oxide Hexamethylene Diamine Carbamate MT Carbon Black Cure 20 min at 280°F Post cure 16 hr at 400°F	100 20 1.3 25	2900	190	80	50	Viton[R] A
12B	ditto	Additives ditto 8D Cure 20 min at 310°F Post cure 16 hr at 400°F		2840	330	80	73	Viton A-HV
8C	Chlorosulfonated polyethylene	Polymer Magnesium Oxide Hexamethylene Diamine Carbamate Pentaerythritol Magcarb W HMF Carbon Black Cure 20 min at 310°F	100 10 1 5 10 5	2080	150	90	36	Hypalon[R]
8A	Natural Rubber	Polymer (smoked sheet) Stearic Acid Zinc Oxide N-cyclohexyl-2-benzothiazole sulfenamide Sulfur HAF Carbon Black Polymerized trimethyl-dihydroquinoline Cure 15 min at 310°F	100 3 5 0.6 2,75 50 1	2200	350	65	10	Coral Rubber
8B	Chloroprene	Polymer (WRT) Stearic Acid Zinc Oxide Magnesium Oxide (ELC) HMF Carbon Black Na 22 Cure 20 min at 310°F	100 5 5 4 50 0.5	2200	175	85	11	Neoprene

[R] Registered trade-mark E. I. DuPont de Nemours & Co., Inc.

* 50% compression, 90 hrs at room temperature

Table 2. Recipes and Properties of Materials Supplied by Aeronautical Systems Division

From these tests we can conclude that standard compounds of neoprene and natural rubber will make satisfactory seals for use at cryogenic temperatures, with little or no spring loading required, if .070-inch O-rings are compressed to just short of material failure. Compression beyond material failure does not necessarily mean that the seal will leak but this requires more flange loading than necessary and of course destroys the O-ring.

The pressure and temperature cycles indicated in Table 1 were introduced to provide a more severe test of seal reliability. Pressure cycling was done after the seal was cold, and consisted of 10 cycles between 1 atmosphere and 100 psig followed by additional cycles between 1 atmosphere and the maximum pressure shown in Table 1. The jig was cooled in liquid nitrogen for 2 hours at 100 psig, which brought the temperature of the seal to about 97°K. After this the pressure was raised to maximum and cooling continued for 30 to 60 minutes. If the seal was tight the pressure was reduced and the jig warmed in hot water, which completed one temperature cycle. The second cooldown was with 1000 psig on the seal, and this was raised to 1250 psig after 2 to 3 hours in liquid nitrogen.

In general, neither pressure nor temperature cycling caused an initially satisfactory seal to leak. The seal in test 4-H leaked when cooled down a second time after being assembled for a week-end. This indicates that relaxation may be an additional factor which should be investigated. In test 1-P the seal was tight through 10 pressure cycles and while cold at 1000 psig, but began to leak when removed from the bath and jarred against the concrete floor. It is interesting to note that every seal which held at 100 psig was also satisfactory at 1000 or 1250 psig.

Perhaps the greatest value of this series of tests is that it makes possible some further differentiation among materials and O-ring sizes which have passed all previous tests. Thus it appears now that compounds of natural rubber and neoprene may make reliable cryogenic seals under more severe conditions than compounds of v.f.p. or c.s.p. It is also quite clearly indicated that the .070 inch O-ring width is to be preferred over the larger .140 inch width. The larger size offers a wider seal and more material to conform to flange surface imperfections, but a given compression results in greater final thickness and therefore more chance for shrinkage and leaking during cooldown. Since there is more material to be compressed the required bolt loading is higher for a wider O-ring and material failure of the elastomer is more likely to occur.

2. Thermal Expansion of Elastomers

Figure 2 shows the dilatometer which is being used to measure thermal contraction and expansion of elastomer samples in the temperature range 76 to 300°K.

A 2-inch long by 1/2-inch diameter sample is surrounded by a heavy-wall copper tube or shield which helps maintain uniform sample temperature. The sample and tube are supported by three invar rods connected to a brass top plate. On top of the sample rests an invar rod with brass ends, which applies a constant force of 98 grams to the sample. The top plate and sample support assembly can be easily lifted out of the cooling system in order to change samples.

Around the sample support assembly is a second copper shield which has soldered around it a coil of copper tubing which carries a small flow of helium gas. This gas is discharged inside the shield, near the bottom, and serves as the heat transfer medium which cools the sample. The shield is supported and surrounded by expanded polystyrene which serves as insulation to slow down the cooling rate. The polystyrene is enclosed by a flanged metal can which is immersed in a bath of liquid nitrogen. The entire assembly is supported and stabilized by placing the dewar in a wide container and filling the intervening space with sand.

Both length and temperature are measured electrically, and continuously recorded by a 6-point millivolt recorder. Length measurement is achieved by means of a sensitive differential transformer which is activated by a hinged finger. The free end of the finger rests on a lever which moves up and down as the length of the sample changes with temperature. Sensitivity can be adjusted by moving the transformer to different positions along the length of the lever. Calibration is achieved by slipping the leaves of a thickness gauge between the lever and the top of the rod which rests on the sample.

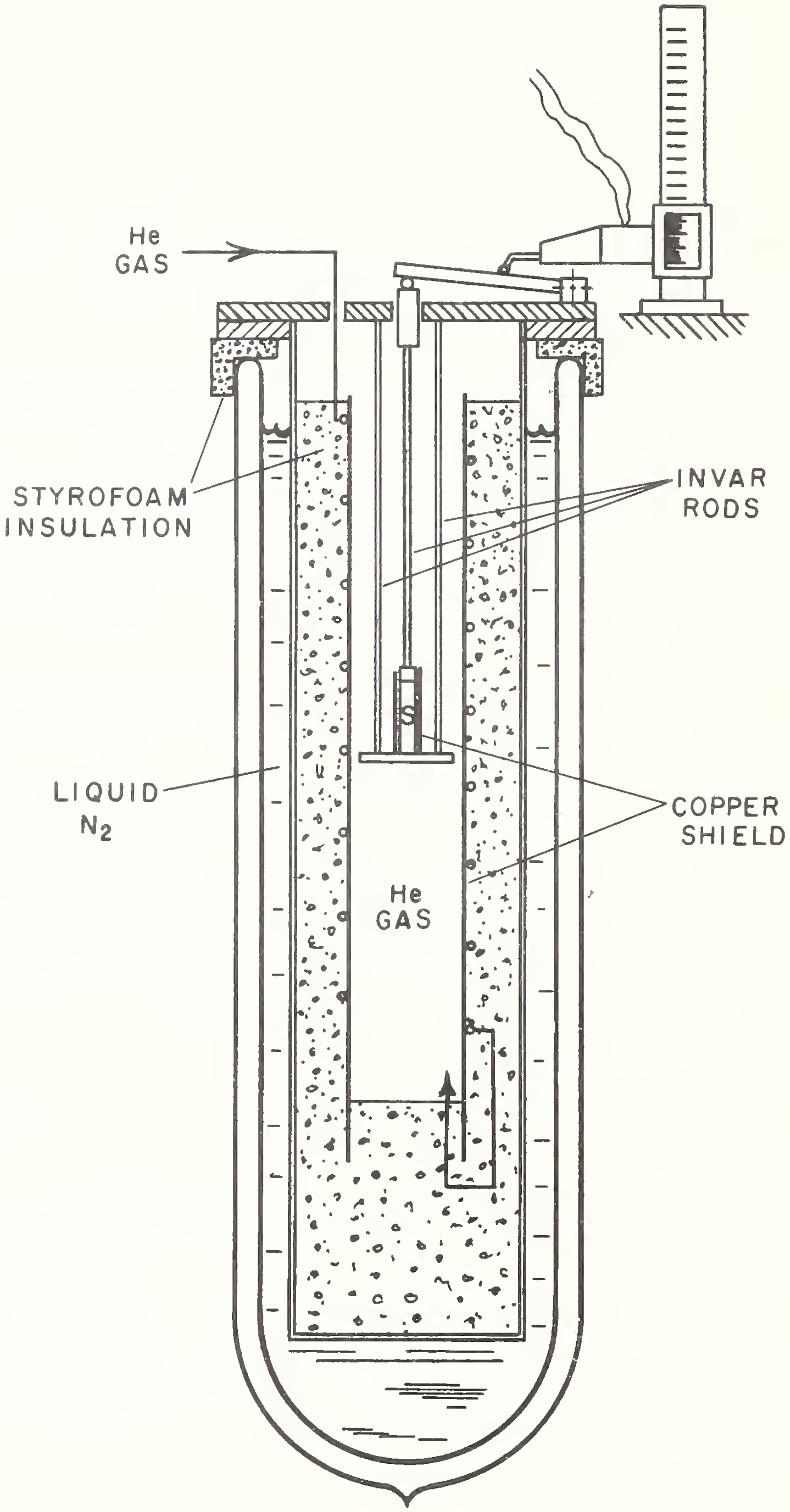

Fig. 2

CRYOGENIC DILATOMETER

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A check on the accuracy of the entire system was made by using a sample of pure aluminum having known expansivity. Overall accuracy appears to be about ±2.5%, with various factors still subject to improvement. Temperature is determined within 2° near 76°K, and within 1° or less from about 140°K to room temperature. It is anticipated that expansion measurements having an overall accuracy of ±1% can eventually be made.

Figure 3 shows results obtained using four samples of materials supplied by Aeronautical Systems Division. Continuous curves showing the relationship between length and temperature during cooldown from room temperature to 76°K were obtained as shown. For neoprene the results below 216°K were uncertain but the probable shape of the curve is shown dashed. The curves for natural rubber, v.f.p.-A, and neoprene are drawn through points from two entirely separate runs for each material, showing that reproducibility is quite good. The brittle point transition temperature is fairly well defined by the change in slope of the curves, and all are almost linear above and below the brittle transition temperature.

Note that neoprene and natural rubber, which performed best as cryogenic seals, have higher expansion rates than v.f.p. throughout the entire temperature range. Evidently the unfavorable expansion rate is easily outweighed by other properties for this particular application.

3. Torsion Testing

In addition to thermal expansion, force decay, and sealability it is desirable to compare stress-strain behaviors of the various elastomers throughout the temperature range of interest. With this in mind the torsion testing apparatus shown in Figure 4 was constructed. Similar equipment has been used to make measurements of plastics and metals[1, 2]. The apparatus shown in Figure 4 accommodates standard tensile specimens having rectangular cross section approximately 0.5 inch by 0.084 inch.

According to Timoshenko[3], shear modulus (G) can be approximated for a rectangular bar from measurements of the twist

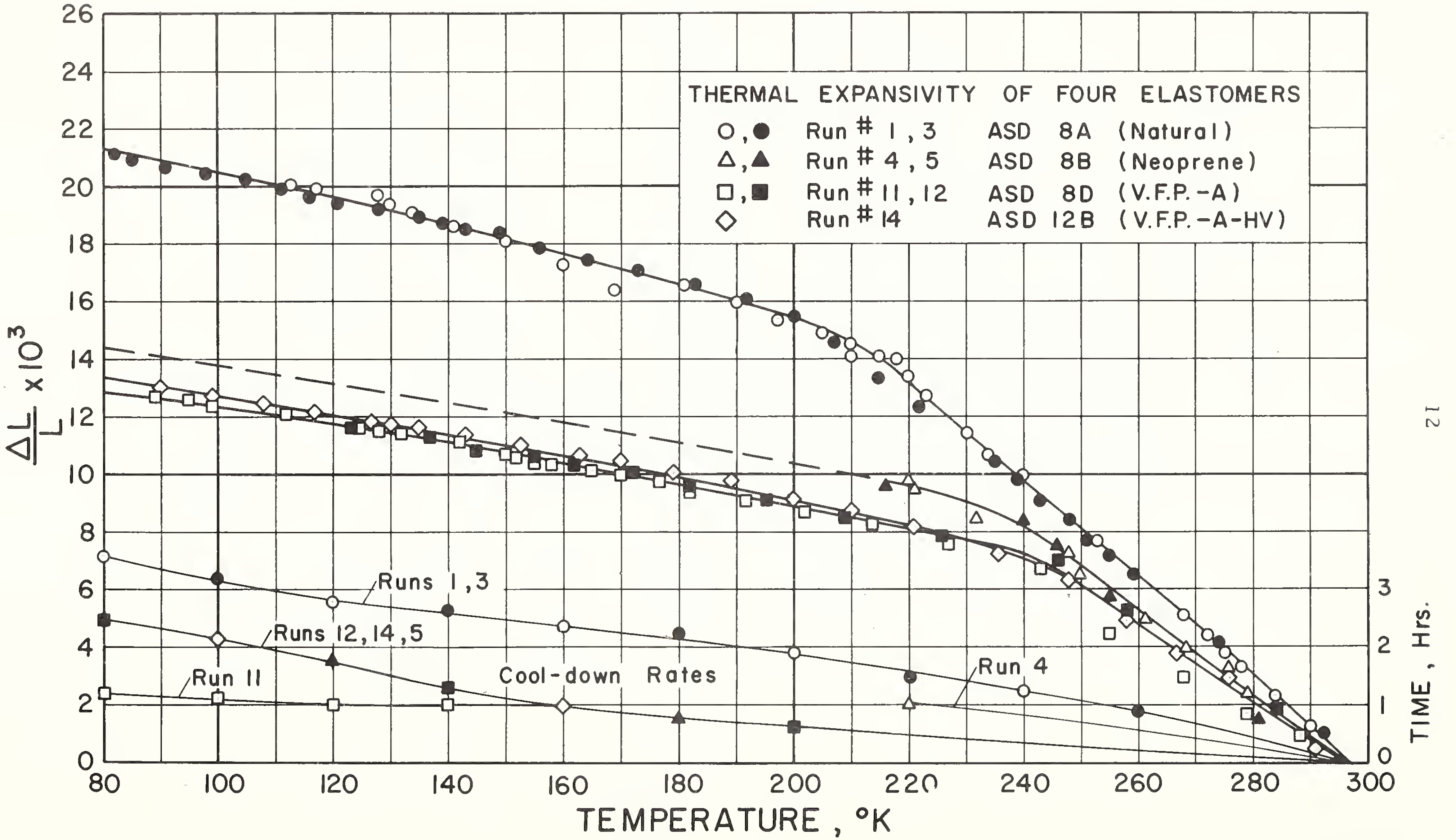
THERMAL EXPANSIVITY OF FOUR ELASTOMERS
○, ● Run # 1, 3 ASD 8A (Natural)
△, ▲ Run # 4, 5 ASD 8B (Neoprene)
□, ■ Run # 11, 12 ASD 8D (V.F.P.-A)
◇ Run # 14 ASD 12B (V.F.P.-A-HV)
$\frac{\Delta L}{L} \times 10^3$
TEMPERATURE, °K
TIME, Hrs.
Runs 1, 3
Runs 12, 14, 5
Run 11
Cool-down Rates
Run 4
0
2
4
6
8
10
12
14
16
18
20
22
24
26
80
100
120
140
160
180
200
220
240
260
280
300
1
3

Fig. 3

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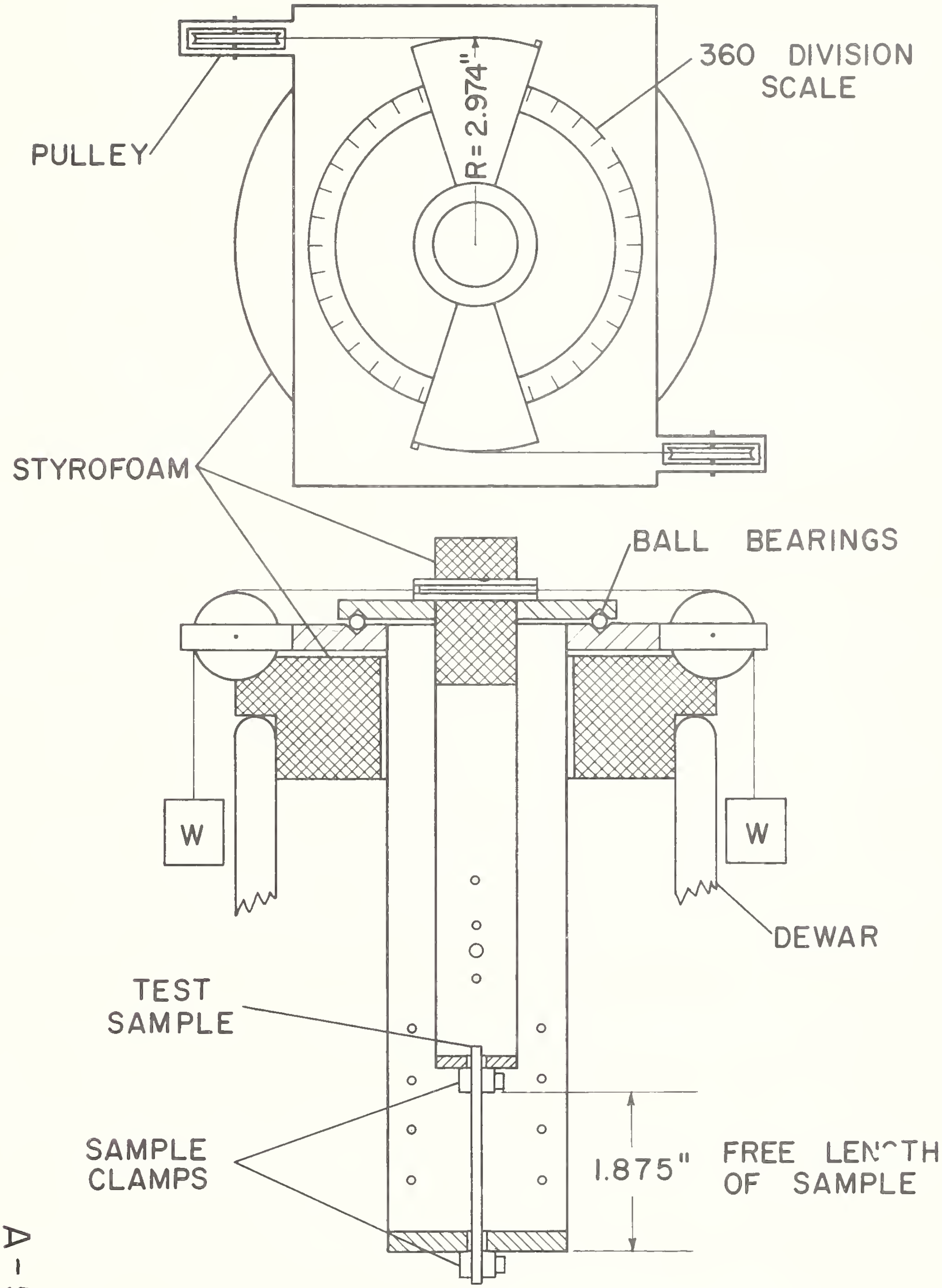

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Fig. 4

TORSION TESTING SCHEMATIC

angle per unit length (θ) and the applied torque (M_t) by use of the relation

$$G = \frac{M_t}{k_1 \theta (2a)^3 2b} .$$

Here k_1 is a numerical factor determined by the ratio of the cross section dimensions a and b.

A preliminary test was performed using a tensile sample of v.f.p. cooled to 76°K. The calculated value of G was 3.4×10^4 psi. If a Poisson's ratio (ν) of 0.25 is assumed, the value of Young's modulus (E) can be obtained from the definition of shear modulus[3]

$$G = \frac{E}{2(1+\nu)} .$$

This gives a value of 8.5×10^4 psi for E, which is about one-fourth as high as the value obtained by direct measurement of stress and strain in compression at 76°K[4]. A possible explanation of this discrepency is the high frictional forces which were present at the faces of the compression test button. One advantage of the torsion test is virtual elimination of frictional forces on the sample.

A continuation of the torsion testing is planned, using samples supplied by ASD in either round or rectangular cross section. This test should be quite useful as a quick and uncomplicated method of observing the relationship between modulus and temperature below the brittle point.

4. Cryogenic Engineering Conference

A paper entitled "The Application and Behavior of Elastomers at Cryogenic Temperatures", paper H-6, 1961 Cryogenic Engineering Conference, by Robbins, Weitzel, and Herring was presented at the Conference in Ann Arbor on August 16. The text of this paper as it will appear in the Conference Proceedings (Advances in Cryogenic Engineering, Vol. 7, Plenum Press, Inc.) has been designated NBS Report 6787 and is attached herewith.

5. References

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3. S. Timoshenko and J. N. Goodier, Theory of elasticity, 2nd Ed, McGraw-Hill Book Co., Inc. (1951).

4. F. W. Tipton, et al, Elastomers for cryogenic sealing, Proc. 6th Ann J. A. N. A. Conf. on Elastomer Research and Development, 2, 409 (1960).

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July 25, 1961

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The Application and Behavior of Elastomers

at Cryogenic Temperatures*

by

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

* This work was sponsored by Aeronautical Systems Division, Wright Patterson Air Force Base

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IMPORTANT NOTICE

1. INTRODUCTION

Elastomers offer the cryogenic engineer a dramatic demonstration of how the properties of some materials change radically when cooled to cryogenic temperatures. These materials have room temperature properties of high extensibility and forcible quick retraction but, without exception, become hard and glass-like when sufficiently cooled, and thereafter behave like crystalline solids. Around this so-called glass transition temperature the modulus of elasticity may increase by a factor of 400-1000, and the coefficient of expansion may decrease by a factor of 3 or more[1]. Because of these changes elastomers are not commonly used at cryogenic temperatures to solve problems for which they would be used at room temperature. Where an elastomer O-ring would perform the function of a static seal at room temperature, countless designs are now being used to seal cryogen transfer lines, fuel and oxidizer missile components, experimental apparatus, and so on. However, none of these has met with universal approval, and all exhibit at least one of the disadvantages of high cost, poor performance, or complexity.

To augment the efforts of industry in this area, NBS is investigating low temperature seals, with emphasis on use and behavior of elastomers. Early work was reported at last years Cryogenic Engineering Conference and elsewhere[2, 3]. In brief, it was found that elastomeric O-rings, molded from several compounds, could be used for high vacuum seals at cryogenic temperatures with system pressures up to 1000 psig. The present paper describes modifications of the earlier designs which result in substantial reduction in required sealing force, greater simplicity, and no apparent loss of reliability. An apparatus designed to measure the contraction of compressed elastomers in the temperature range 300°K-76°K will be discussed, together with some test results.

2. O-RINGS BETWEEN FLAT PLATES

Previous successful designs(2) incorporated mating tongue and groove flanges, which were dimensioned to closely confine the O-ring at the end of a linear compression of about 80%, 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 seals were very reliable, it has now been found that satisfactory seals can be made without lateral 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 with no close tolerances; and (3) the surface finish of the flanges is not critical - in fact a somewhat rough machine finish is advantageous since it helps to prevent excessive extrusion of the otherwise unconfined elastomer.

Since minimum bolt loading should be required to make the seal if an O-ring of minimum cross section is used, a number of seals were tested by applying 80-90% compression to one inch diameter O-rings 1/16-inch thick between flat plates. (All previous tests used 1/8-inch O-rings.) Figure 1 shows the apparatus for these tests. The test jig was immersed in a suitable cryogen, and internal helium pressure was maintained constant during cooldown, at values from 50 to 1000 psig. A dynamic vacuum of around 10^{-5} mmHg was effected on the exterior side of the seal, and this was connected directly to a consolidated mass spectrometer leak detector throughout each run. Leak rates measured with this detector ranged from 0.5 to 0.0003 standard cubic centimeters of helium per hour. Leak rates greater than 0.5 cc/hour were considered failures in our tests and were not monitored as a general rule, even though for many applications leak rates of hundreds of cc/hour can be tolerated.

It was found that O-rings molded from commercial compounds of Viton-A(R), Hypalon(R), and neoprene, one inch in diameter and 1/16-inch thick, made excellent seals at all temperatures between 297°K and 20°K when compressed between flat plates to about 1/5 their initial thickness (80% compression). Furthermore, the bolt torque required was considerably reduced compared to previous designs. The exact reduction was somewhat difficult to determine, but one can expect about 1/4 the bolt loads previously reported, depending upon thread lubrication, flange flexing, rate of compression, and other variables, some of which may vary each time a seal is assembled.

3. FLANGE SEALS FOR AIRBORNE CRYOGEN TRANSFER LINES

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, flexibility of the flanges, which can be used to advantage in overall seal design. With proper choice of materials and proper positioning of the bolts and O-ring, flexing of the flanges results in a very efficient spring loading which greatly reduces the rate of force decay during cooldown of the assembly.

To test the practicality of the NBS designs in airborne equipment, a number of flanges designated for airborne use were studied, using the method shown schematically in Figure 2. The flange and seal assembly was surrounded by a vacuum which was dynamically maintained by the diffusion pump of the helium leak detector. A stainless

(R)Trademark of E. I. du Pont de Nemours, Inc.

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 the refrigerant (liquid N_2 or H_2). The level of the liquid cryogen was kept below the seal, and helium pressure was applied above the liquid. Thus the ultimate temperature of the test was determined by the boiling temperature of the refrigerant at the pressure of the test. During cool-down rapid boiloff of the refrigerant hampered leak detection, but helium pressure was applied during cool-down and the detection seemed reliable enough. Leak rate limits of the leak detector were the same as in previous tests. Leak rates greater than 0.5 cc/hour were estimated by removing the vacuum jacket and submerging the flange assembly in the cooling media. 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.

Examples of the flanges tested are shown in Figures 3 and 4. The flange shown in Figure 3, which fits a 3" O.D. pipe, was made of 304 stainless steel with faces only 0.095 inches thick. There were eight holes for 1/4 inch bolts equally spaced on a 4-inch bolt circle. Further reduction in weight was made by cutting out scallops of material between the bolt holes. Commercial O-rings molded from the same materials which had been successful in previous tests were purchased 1/16-inch thick and in several diameters. Some tests were performed with the O-ring stretched slightly over a short stainless steel retaining sleeve which fit snugly inside the flange to hold the O-ring in position and prevent inward extrusion.

No satisfactory method for sealing these extremely light weight flanges was found. Some seals held at room temperature, and one Hypalon(R) O-ring used with the sleeve was tight at 76°K. Even this test was inconclusive, however, since a second seal assembled in the same manner failed at 76°K. This design was unsuccessful because the flanges lacked the stiffness necessary to compress the O-ring in the area between bolt holes. Attempts to overcome this weakness by various schemes, such as carbon steel back-up rings between the bolts and the flanges, wire rings outside the bolts between the flanges, and combination of one light weight flange with a heavier duty flange, were not successful. (In these designs there is minimum material between the bolts, which is precisely where additional strength is needed.) It is our opinion that lightweight flanges could be designed which would seal successfully with little or no additional weight at a small sacrifice in flange machining costs.

Figure 4 shows a flange which was successfully tested using the apparatus of Figure 2. Designed for use with a 2.5 inch pipeline, the flanges were made of 321 stainless steel with flange faces 0.200 inches thick, ten 1/4-inch steel bolts equally spaced on a 3.345 inch bolt circle, and additional reinforcement on the inside of the corners. Two other designs, one using 0.300 inch thick aluminum flanges and one designed for a 3 inch pipeline, were also tested successfully. O-rings 1/16-inch thick of Viton A$^{(R)}$, Hypalon$^{(R)}$, and neoprene of 50 to 60 durometer were employed. Most tests were run with 150 psig in the seal interior.

An important factor present in these tests was flexibility of the flanges. In the case of the extremely lightweight flanges previously discussed, flexing was so non-uniform that a reliable seal could not be achieved. However, the flange shown in Figure 4 flexed into a fairly uniform cone shape which actually served to reduce the initial compression required to maintain the seal force during cool-down. Measurements indicated that good seals were obtained with 55-70% compression of the ring, whereas about 80% was required in the tests mentioned in sections one and two of this paper.

The above results indicate that an optimum stainless steel flange thickness somewhere between 0.200 inches and 0.095 inches exists which would result in minimum compression, and consequently minimum load requirement and minimum flange weight. This thickness would depend upon system pressure requirements, spring constant of the flanges, and other parameters which will be discussed in the next section.

4. PROPERTIES AND THEIR RELATION TO SEALABILITY

When an amorphous rubber-like material composed of long chain molecules randomly distributed is compressed, the chains tend to become more oriented. For most co-polymers and polymers with complex side chain constituents this orientation does not lead to crystallization, but it does result in anisotropy. To investigate the behavior of elastomers compressed highly and subsequently cooled to some low temperature, the apparatus shown in Figure 5 was designed and built. Specifically, the contractions of 1/2 in thick by 1 in^2 cross section compression set buttons when compressed 20-50% and cooled to 76°K, are being studied. To measure contraction for a wide range of initial applied forces, a bellows capable of translating 3000 lbs. force to the sample was obtained. The actual bellows pressure used varies from 20 psi to 500 psi, and is kept constant for the duration of each test. Length measurements are made using a lever which translates vertical movement of the sample and part of the stainless steel compression platens into horizontal movement outside the cold area. Several compression platens are used, each having a different length; accordingly, the same lever can be used for widely varying values of initial contraction. It should be noted that contraction of the long vertical portion of the stainless steel lever has a very small effect on the measurements, but calibration of the lever was necessary due to contraction between the fulcrum and the lever pointers touching the top compression platen. The bellows top plate is free to move with the sample and is aligned by the three rods connecting the stationary brass plates. Two thermocouples are attached to the sample just before cooldown, and temperatures recorded continuously during the tests. The dilatometer is cooled by continuously transferring cold nitrogen (gas initially, and liquid during final cooldown) into the dewar which surrounds the dilatometer. Since large masses of brass and stainless steel are in contact with the sample, no further means is provided to insure uniform cooling. Cooling rate is controlled by regulating the pressure over the supply dewar, and rates as low as 0.5°K/min can be attained in this manner.

As was previously mentioned, this apparatus was developed to measure contraction of compressed elastomers during cooldown. Before preliminary results are presented, a brief discussion of what causes the contraction is in order. 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[4], and the same dependence is expected in compression.

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 volume contraction causes linear shrinkage; it is expected that the amount of this contraction is changed by the configurational changes in the compressed sample, but experimental verification has not been possible. Secondly, an appreciable linear contraction at constant volume is caused by the applied force when the sample is cooled. This effect, due to molecular configuration considerations, is referred to as the Gouge-Joule effect by Payne and Scott (5) and predominates at temperatures above the glassy state transition.

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 is similar to that of any ordinary solid. Therefore the Gough-Joule effect, as well as the creep effect, does not appear below T_g. Only the normal thermal expansion below the brittle point should be present.

The results shown in Figures 6 & 7 were obtained using commercially available samples of Viton(R) A, a co-polymer of hexafluoropropylene and vinylidene fluoride. It should be emphasized that the results are still preliminary in nature, and thus should be considered qualitative rather than quantitative. Figure 6 is a plot of the contraction of a 1 in^2 x 1/2 in elastomeric 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.

Figure 7 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 stress was

* This sample was compressed 24%, corresponding to a pressure of 150 psig.

about 40 psi, and resulted in an appreciable reduction in contraction during cooldown. It is believed that this small stress could not have induced crystallinity, or otherwise affected the true properties of the elastomer.[6] 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 pre-stressing 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.

5. THEORY AND CONCLUSIONS

In the final section of this paper we suggest a method for analytically treating the system where an O-ring is compressed between two flat plates and cooled and will conclude with some design recommendations derived from our experience in making simple, reliable low temperature seals.

When an O-ring is compressed by bolts between two metallic flanges, the effectiveness of the resulting seal depends solely on the existence of a force normal to the surfaces of the flange faces. During cooldown this force must remain above a minimum value determined by the system pressure required in the specific application. To determine how the force varies with temperature the following analysis is offered to relate experiments such as those previously described with sealability.

Considering only the changes in force on the sample due to temperature change, the relation between the stress (σ_{T_1}) at T_1 (initial) and that at T_2 is

$$\sigma_{T_1} - \sigma_{T_2} = \alpha E(T_1 - T_2)$$

(see Timoshenko and Goodier [7]). The coefficient of expansion α, and E, the modulus of elasticity in compression, are assumed constant. If α and E vary their product must be integrated over the temperature range in question, giving,

$$\sigma_{T_1} - \sigma_{T_2} = \int_{T_1}^{T_2} \alpha E dT \tag{1}$$

for the change in force with temperature after pre-loading at T_1. In the actual situation, length changes due to bolt and flange shrinkage during cooldown also affect the stress, and they may be taken into consideration in the following way. Since all force changes are a function of either temperature or sample length, L we may write

$$\sigma = \sigma(T, L)$$

which upon differentiation gives

$$d\sigma = \left(\frac{\partial \sigma}{\partial T}\right)_L dT + \left(\frac{\partial \sigma}{\partial L}\right)_T dL \tag{2}$$

A common identity gives us

$$\left(\frac{\partial \sigma}{\partial T}\right)_L = -\left(\frac{\partial \sigma}{\partial L}\right)_T \left(\frac{\partial L}{\partial T}\right)_\sigma \tag{3}$$

Multiply the right side of equation 3 by L/L:

$$\left(\frac{\partial \sigma}{\partial T}\right)_L = -L\left(\frac{\partial \sigma}{\partial L}\right)_T \quad \frac{1}{L}\left(\frac{\partial L}{\partial T}\right)_\sigma$$

By definition:

$$L\left(\frac{\partial \sigma}{\partial L}\right)_T = E, \text{ and } \frac{1}{L}\left(\frac{\partial L}{\partial T}\right)_\sigma = \alpha$$

Substituting in (2) gives

$$d\sigma = -\alpha E dT + \frac{E}{L}\, dL \tag{4}$$

The first term is identical with that given by Timoshenko, and the second term takes into consideration all length changes from "external" sources, such as bolt shrinkage. Integrating (4) we get:

$$\sigma_2 = \sigma_1 + \int_{L_1}^{L_2} \frac{E}{L}\, dL - \int_{T_1}^{T_2} \alpha E dT \tag{5}$$

d L is a function of T also, due to shrinkage in the bolts and flanges, so the temperature function could be used to evaluate this integral. If only α and T affect the stress, equation (5) shows that if volume changes and end effects are small the stress at any temperature T_2 may be estimated by knowledge of: 1) the initial stress; 2) continuous curves of E vs T and α vs T for the sample; and 3) the bolt shrinkage. However, if the flanges have flexed during the room temperature preload, changes in the flex angle will help maintain σ. Consequently less initial stress is needed to achieve a required stress at some low temperature if flange flexing is a factor.

In conclusion, we would like to present the following recommendations based on our present knowledge of the art of cryogenic sealing with elastomer 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 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. A 0.014" 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. A normal machined flange surface is suitable. All traces of oil or grease should be removed from the flange faces with solvent.

2. Choose an O-ring having a cross section diameter of .070" of material previously mentioned here and a mean ring diameter slightly less than the I. D. of the flanges.

3. Stretch the O-ring around a short thin-walled tube (same material as the flanges) which fits snugly inside the flange I. D. Slip this retaining tube inside one flange, with the O-ring resting on the inside shoulder of the flange.

4. Bring the mating flange into position and cross-tighten the bolts until the O-ring is compressed to 75 to 80% or less if there is some flexing of the flanges. The O-ring will flatten and spread outward at the same time that it is forced tightly against the retaining sleeve. Slight extrusion around the inside corners of the flanges is not objectionable.

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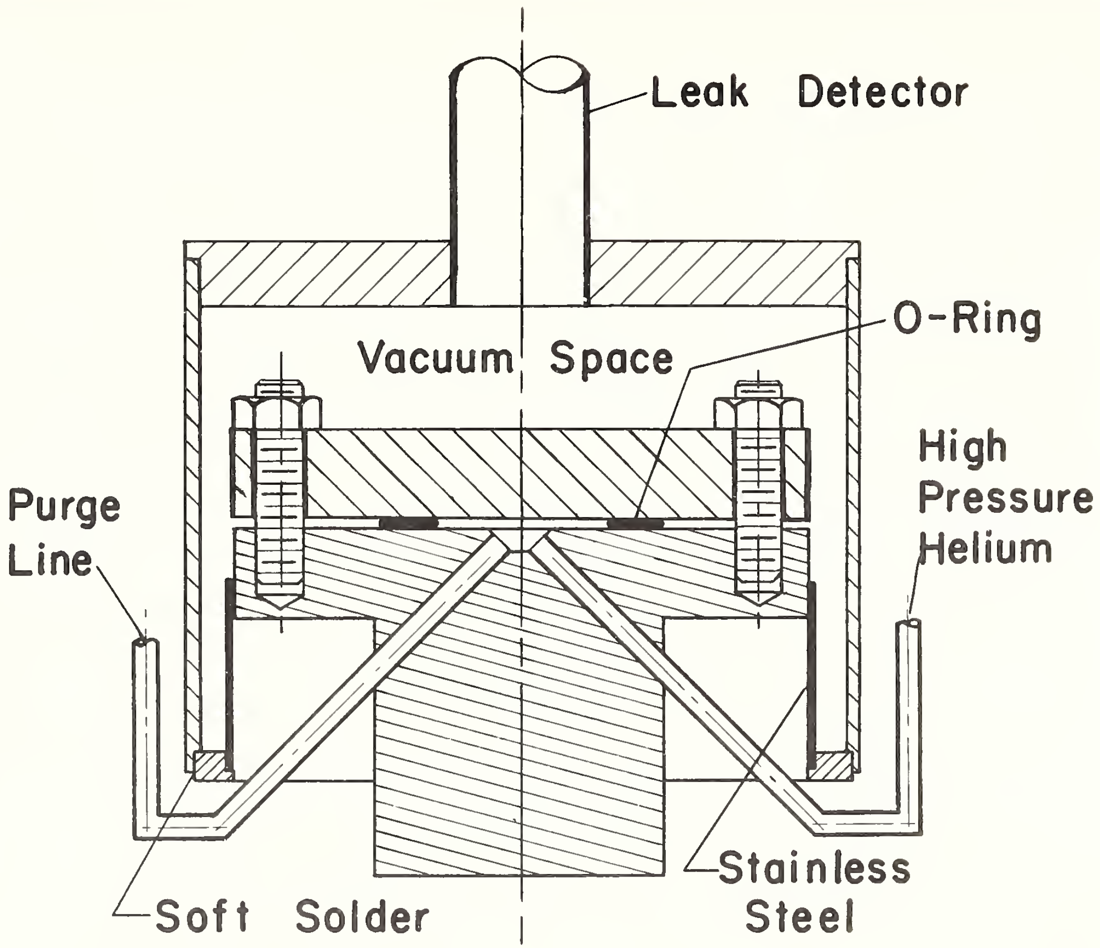


A-4745

Fig. 1· O-Ring seal test jig.

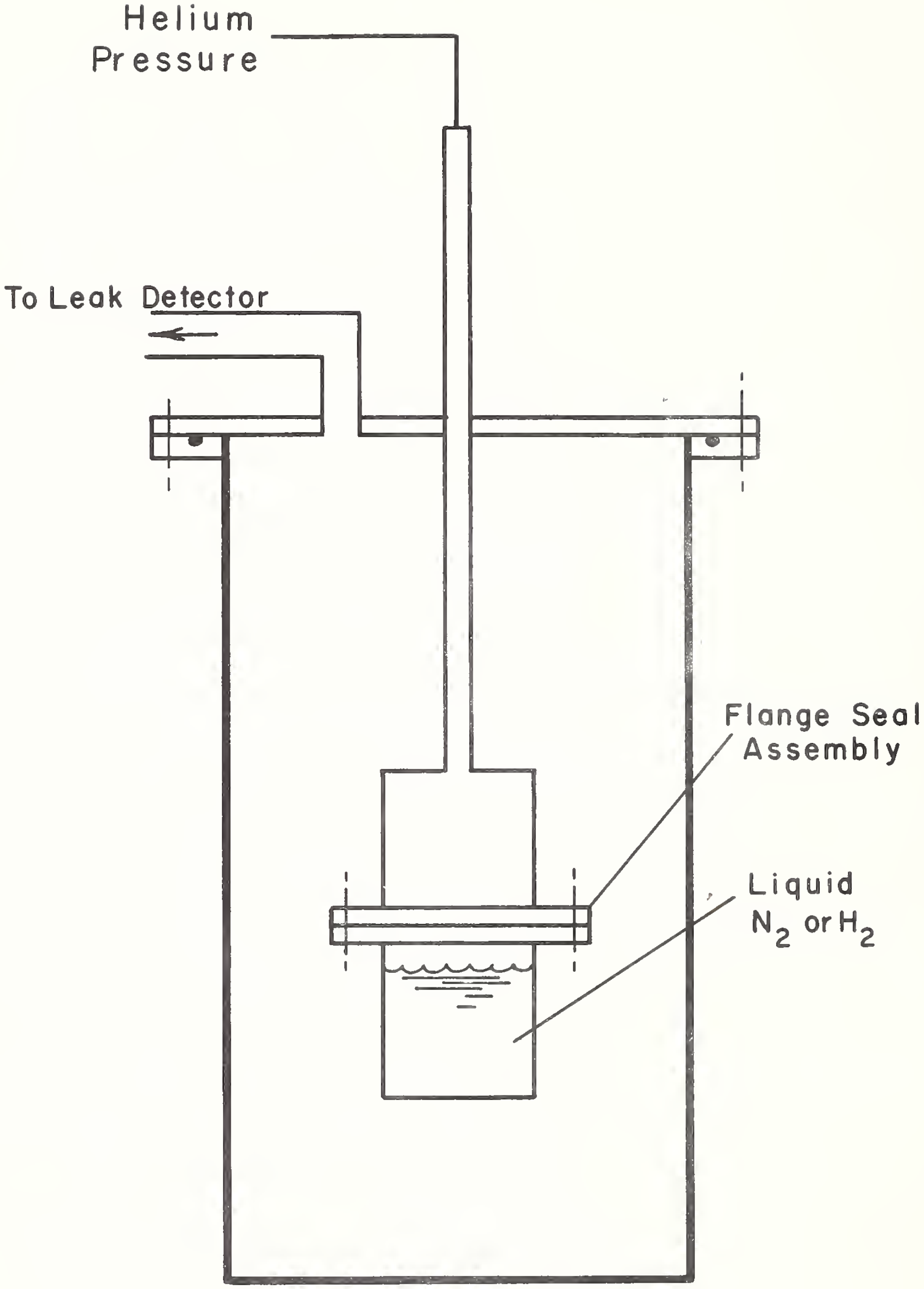
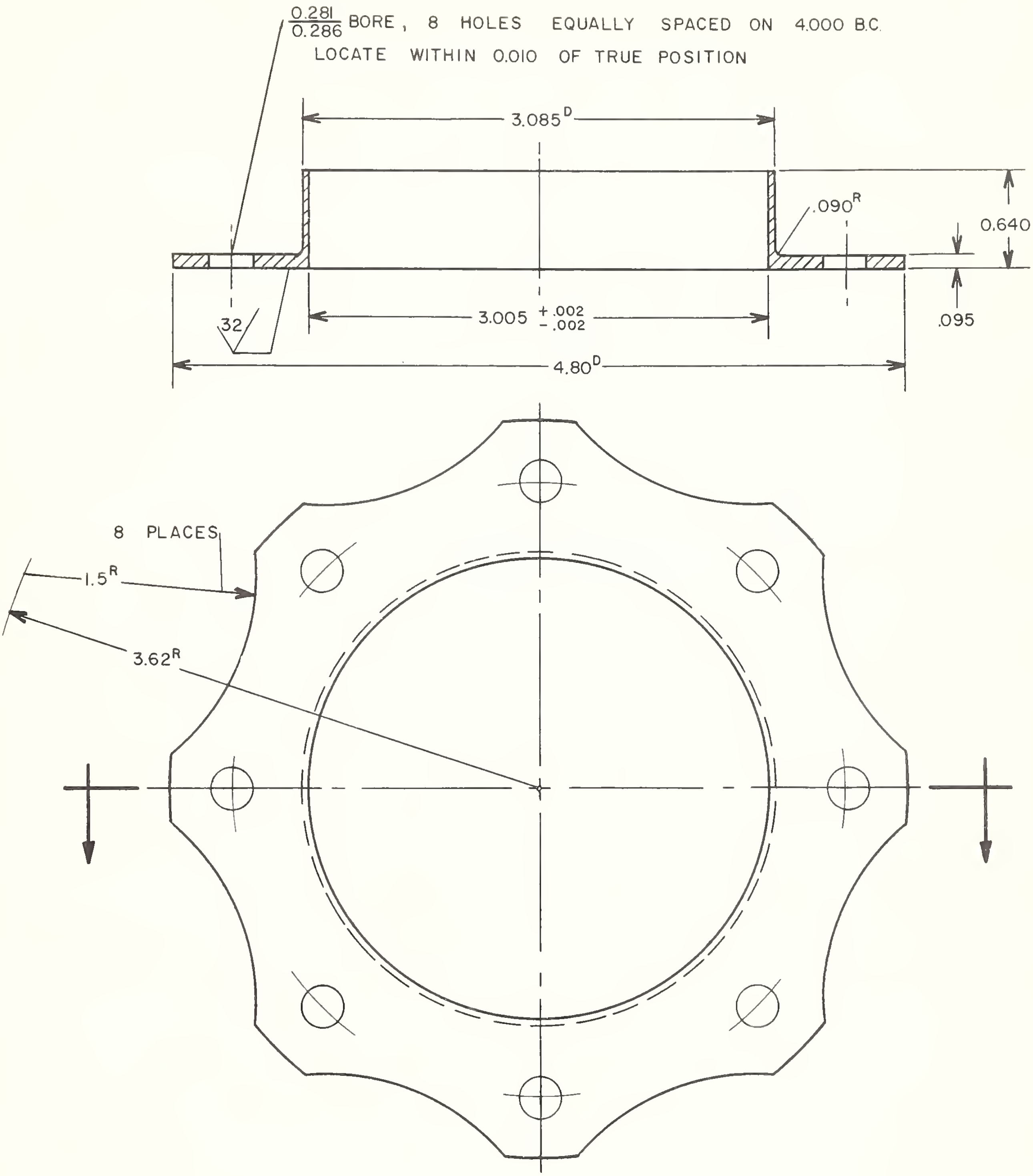


Fig. 2

Schematic of Seal Test Fixture

A-4933



Scale: Full

Material: 304 S.S.

Note: Fractional tolerance ± 1/64

Decimal tolerance ± .010, except where noted.

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FIG. 3

Extreme Lightweight Flange

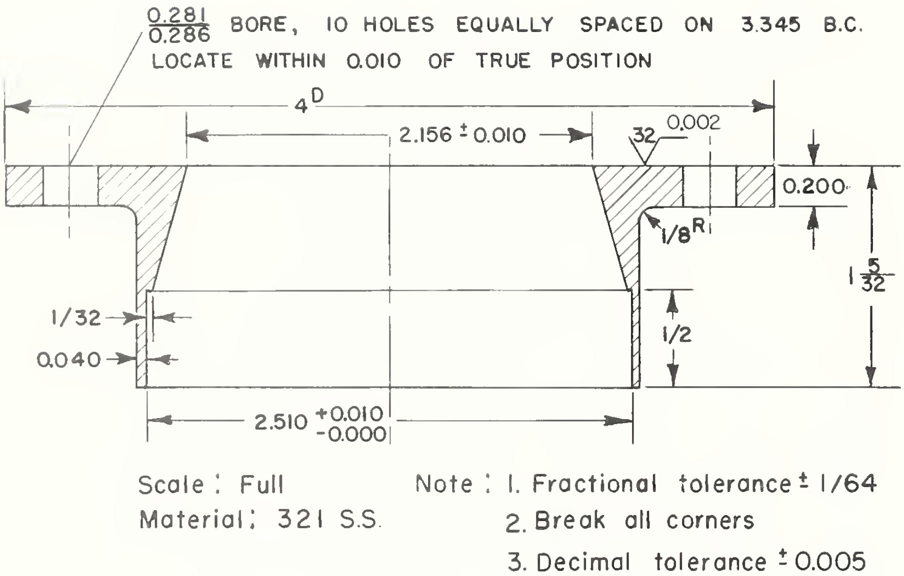

Fig. 4· Flange for 2.5" O.D. line

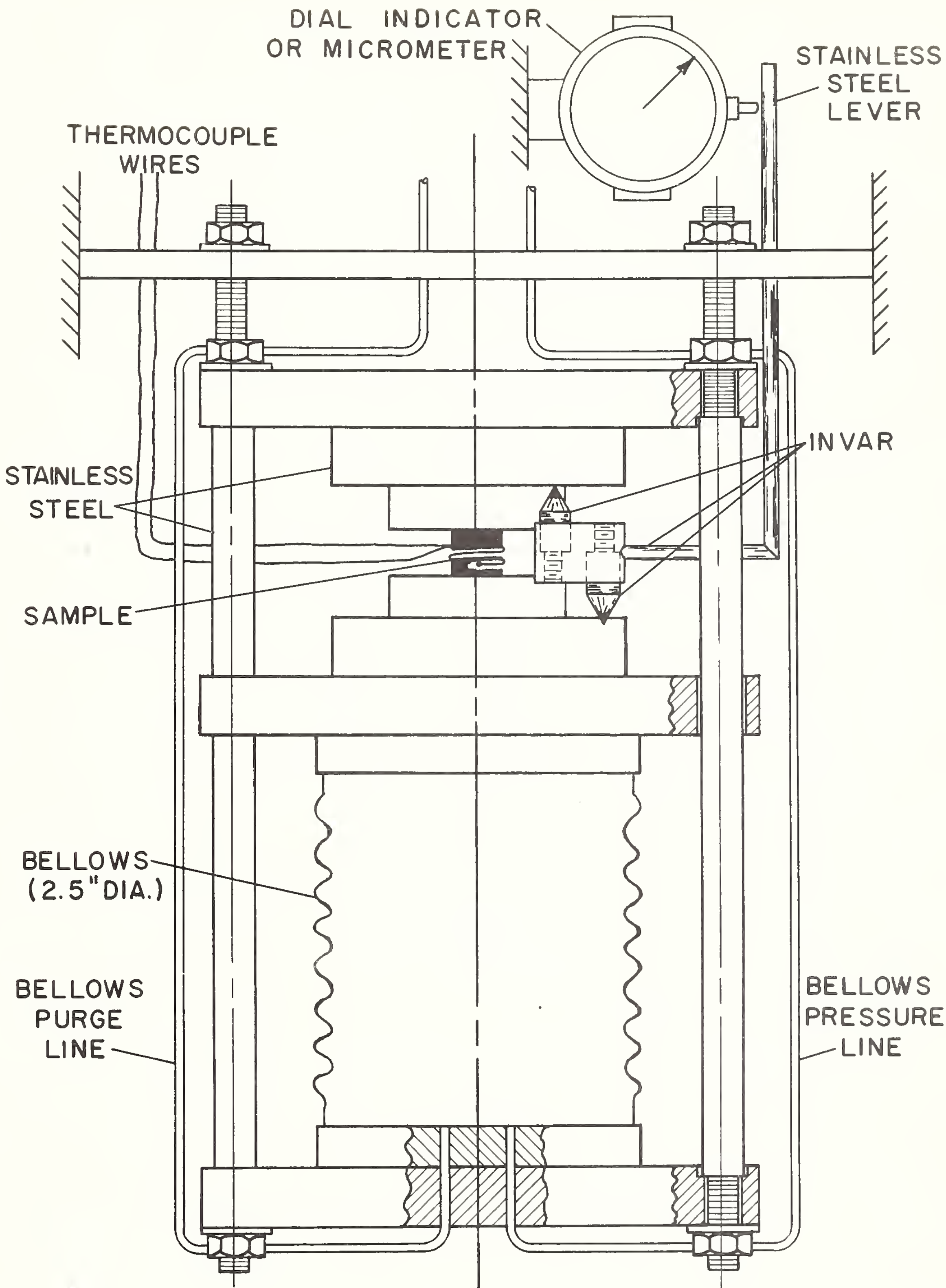

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Fig. 5 Compression dilatometer.

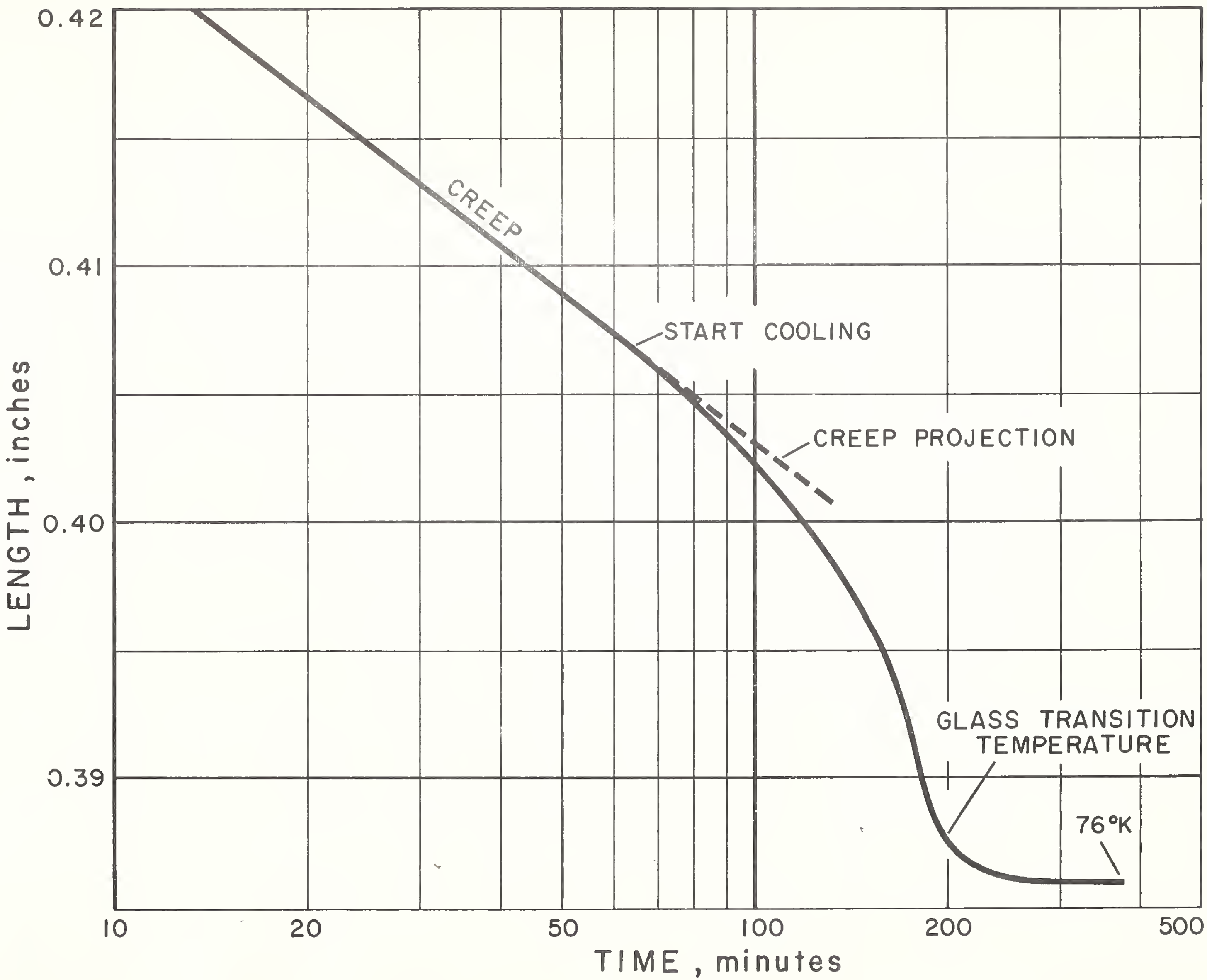

Fig. 6· Contraction of compressed elastomer before and during cooldown

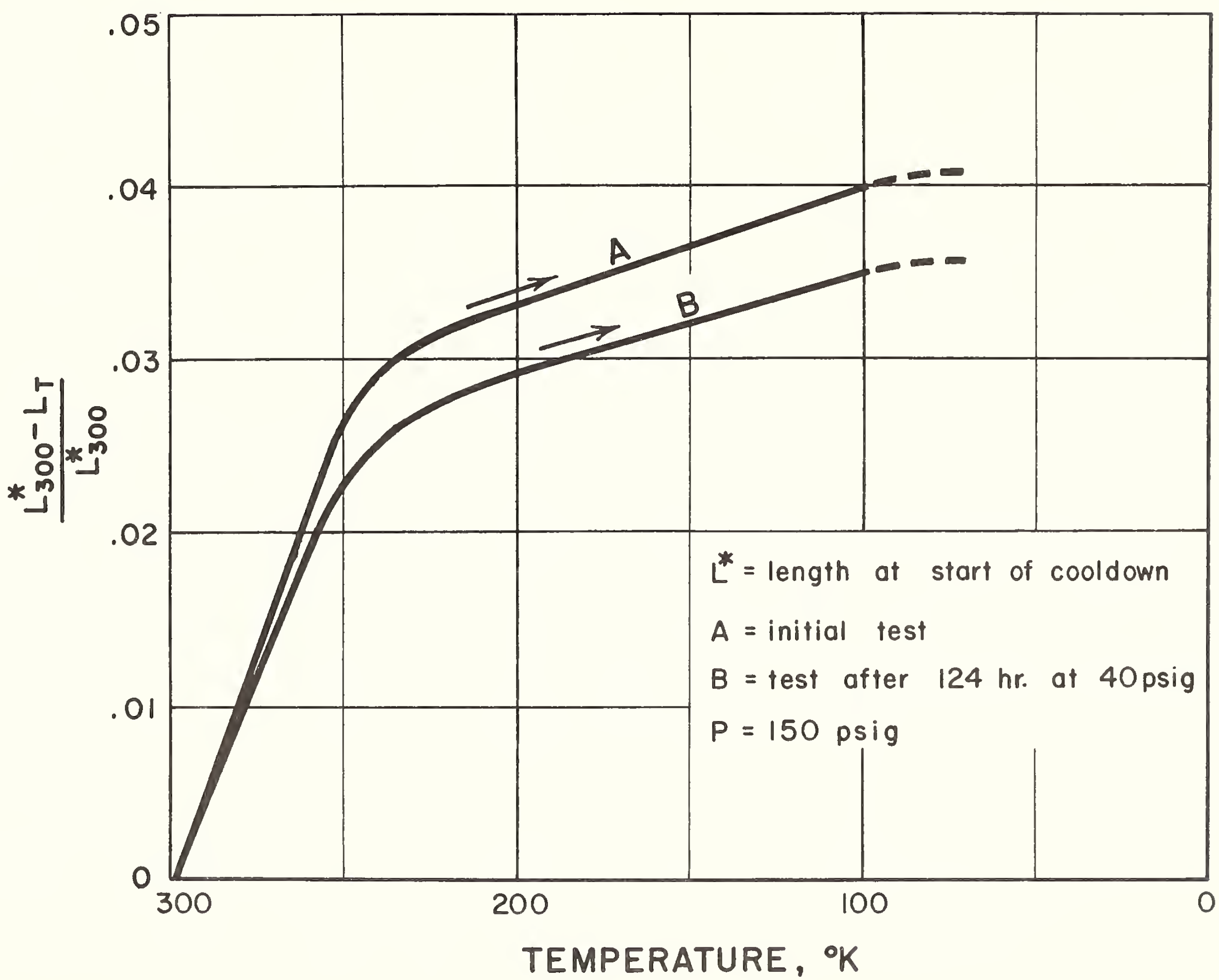

Fig. 7· Thermal expansion of Viton-A

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