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A SUMMARY OF LABORATORY TESTING PERFORMED TO CHARACTERIZE AND SELECT AN ELASTOMERIC O-RING MATERIAL TO BE USED IN THE REDESIGNED SOLID ROCKET MOTORS OF THE SPACE TRANSPORTATION SYSTEM

By J.E. Turner

Executive Staff

February 1993

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An elastomeric O-ring material is used in the joints of the redesigned solid motors (RSRM's) of the National Space Transportation System (NSTS). The selection of the O-ring material used in the RSRM's was a very thorough process that included efforts by NASA's Marshall Space Flight Center and the Langley Research Center, and the Thiokol Corporation. One of the efforts performed at MSFC was an extensive in-house laboratory test regime to screen potential O-ring materials and ultimately to characterize the elastomeric material that was chosen to be used in the RSRM's. This report summarizes those laboratory tests performed at MSFC.

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A SUMMARY OF LABORATORY TESTING PERFORMED TO CHARACTERIZE AND SELECT AN ELASTOMERIC O-RING MATERIAL TO BE USED IN THE REDESIGNED SOLID ROCKET MOTORS OF THE SPACE TRANSPORTATION SYSTEM

INTRODUCTION

Each of the redesigned solid rocket motors (RSRM's) used in the space transportation system (STS) contains three field joints and one nozzle-to-case joint. These are joints located between segments of the solid motors that are mated during the assembly of the booster motors at the Kennedy Space Center (KSC). Figure 1 shows the location of these joints and an expanded view of a typical field joint. The seals used in the RSRM field joints have an internal diameter of approximately 144 in, while the cross section of the O-ring is 0.290 in. The O-rings are constructed by first centerless grinding the molded O-ring cord stock and then using a proprietary splicing technique to make a complete O-ring. Rigorous testing is then performed on the O-ring to assure that it meets all of the requirements imposed for a high-quality O-ring.

One phenomenon associated with these joints is that, upon ignition of the motor, internal pressure in the motor rises very quickly and then peaks at approximately 0.6-s after ignition. This very quick pressure rise (from nominal air pressure to 920 lb/in^2) causes the surface against which the O-ring is sealing to experience elastic deformation and move radially away from the O-ring (maximum

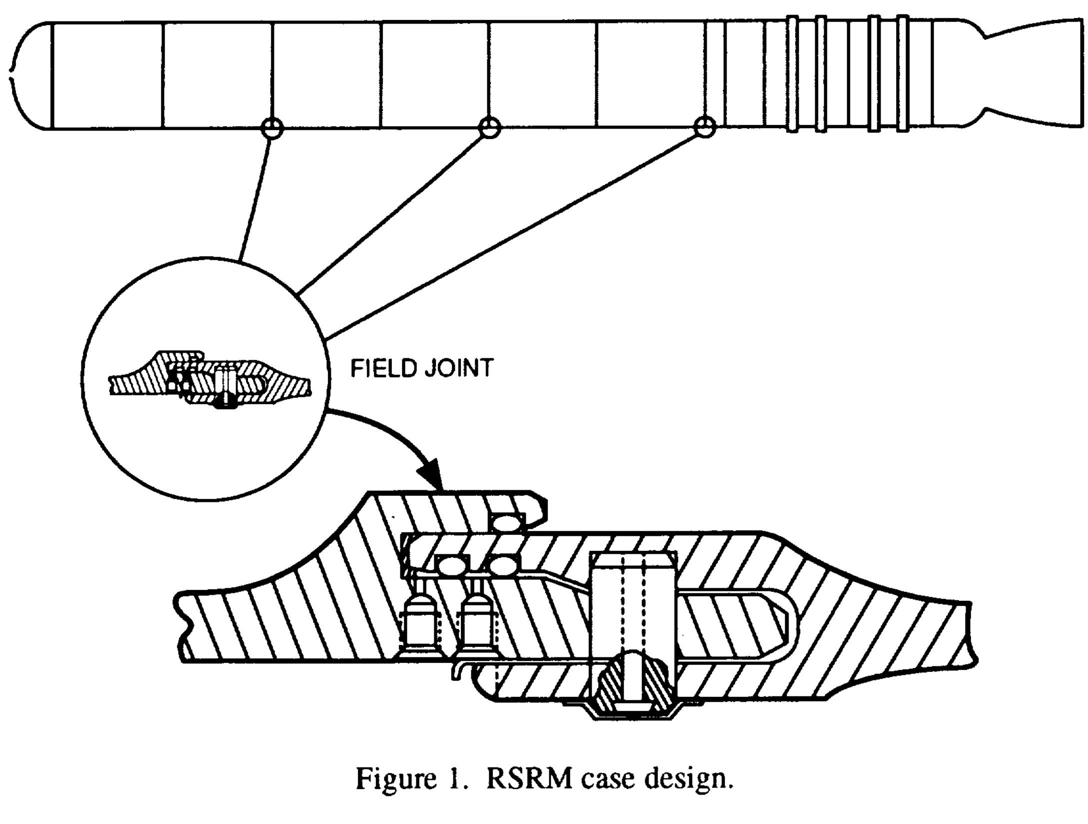


Figure 1. RSRM case design.

predicted movement was 0.009 in) as shown in figure 2. Also, during this short transient, the load is removed from the pins used in mating the cases, thereby allowing the sealing surface to move axially in relation to the O-ring seal. The combination of these two dynamic motions induces an unusual sealing environment for the joint seals. Due to this unusual environment, experimental test data needed to predict the performance of seals in this type of environment were extremely limited. This report attempts to describe the laboratory testing that was performed in support of the process of choosing an O-ring material to be used in the shuttle RSRM's and then characterizing that material fully.

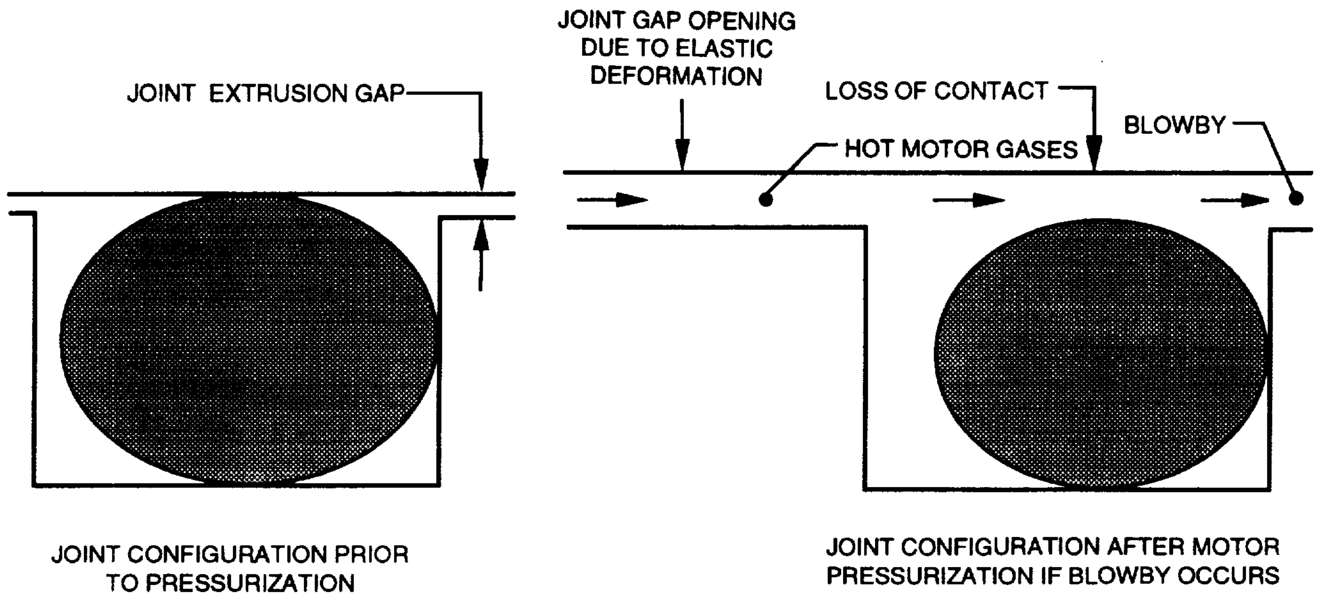


Figure 2. Joint gap opening.

Initial efforts in the selection of a safe and suitable O-ring material began with an engineering assessment of those tests needed to address those critical parameters which would affect the proper sealing action of an elastomeric seal in an RSRM environment. The most basic, yet critical, parameters were the elastomer's toughness (due to possible assembly damage when joints were mated), the elastomer's compatibility with the lubricating grease in the joints, and, finally, the most critical parameter of all, the material's ability to functionally perform so that a seal was maintained at all times in the joint. Once these required performance properties had been established, a set of tests was defined that would determine a material's suitability for each of these parameters.

These tests are defined in figure 3. This figure also indicates the two-tier screening approach that was used in the interest of time and manpower. Initially, over 15 commercially available materials were chosen for testing as possible candidate seal materials. These materials are shown in table 1. Materials that were not commercially available at the time of testing were not considered as possible candidate materials due to the extreme urgency under which this test program was conducted. Since there was such a large number of possible candidate materials, it was determined that an initial screening level 1 would be used to eliminate as many materials as possible at the beginning of the test phase. This would allow for more extensive testing required in screening level 2. Viewing figure 3, it should be noted that the tests shown did not occur in series, but instead were conducted in parallel in order to perform as many different tests as possible in the shortest amount of time.

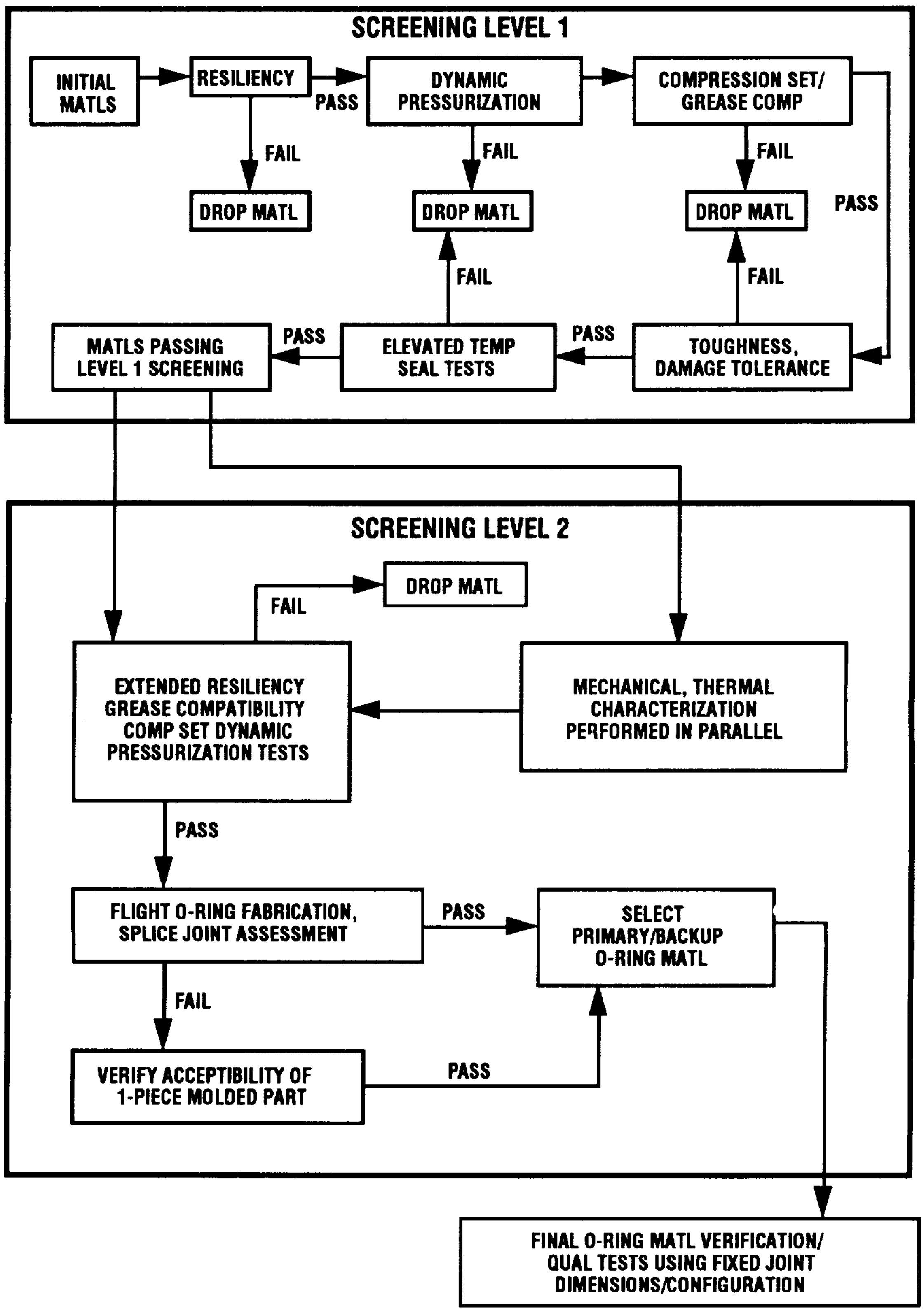


Figure 3. O-ring material screening logic.

Table 1. Candidate seal materials.

Material	Type	Supplier
V747 (V1115)	Fluorocarbon	Parker
V835	Fluorocarbon	Parker
S383	Silicone	Parker
S604	Silicone	Parker
S650	Silicone	Parker
Composite Seal	Fluorocarbon/silicone	Hunger
L677	Fluorosilicone	Parker
E515	Ethylene/propylene	Parker
E592	Ethylene/propylene	Parker
E692	Ethylene/propylene	Parker
N304	Nitrile	Parker
N602	Nitrile	Parker
N1084	Nitrile	Parker
Arctic Nitrile™	Nitrile	Cameron
Eypel-F	Polyphosphazene	Ethyl

In figure 3, the tests called resiliency and dynamic pressurization were aimed at determining the materials' performance characteristics in simulated RSRM environments. The tests defined as a compression set were performed in order to ascertain how much permanent set the O-ring material would retain after a compressive load was released. The tests defined as "elevated temp seal tests" were performed to determine the material's performance at elevated temperatures (>500 °F). The tests labeled splice joint assessment dealt with the inherent integrity of the splice joints that are used to manufacture the RSRM O-rings. All other tests listed in figure 3 are self-explanatory. All tests listed in this figure will be discussed in the following pages except for the work performed in assessment of the splice joint and molded O-rings. These will not be discussed due to the proprietary data involved with each.

All of the above testing can be broken down into three main areas: (1) characterization of inherent physical properties of the materials, (2) testing to determine the O-ring materials' sealing ability using high-pressure testing apparatus, and (3) testing to determine the resiliency of the material. Each of these three areas will be addressed with additional details given on the specific testing performed and some results of these tests. All of the work cited herein was performed by personnel from the Nonmetallic Materials Division at the Marshall Space Flight Center (MSFC). A parallel testing effort was also performed by Morton Thiokol, Inc. However, due to the importance of the results of these tests, all tests were run independently.

PHYSICAL PROPERTIES OF MATERIALS

One of the very first areas of consideration in investigating the properties of seal materials is the inherent physical properties that the materials possess. Testing deemed to be important in this test program were thermal characterization, mechanical properties, and grease compatibility.

Testing on the thermal characterization of the materials was done to determine the glass transition point and the temperature of decomposition of 13 materials. Also included in a type of thermal characterization were the high-temperature capabilities of several materials.

Glass transition point testing was performed utilizing both DSC and TMA test techniques. Table 2 lists the glass transition points for several of the seal materials.[1] Table 3 lists the onset of thermal decomposition.[1]

Table 2. Glass-transition temperature for alternate seal materials.

Materials	Glass-Transition (°F)		
	Method		
	DSC	Expansion Probe	Penetration Probe
Fluorel/V747-75	7	12	–
MFC/V835-75	–18	–17	–15
Silicone/S383-70	–155	–168	–155
Silicone/S604-70	–40	–	–36
Silicone/S650-70	–143	–	–141
FS/L677-70	–83	–89	–74
Nitrile/N304-75	–62	–65	–58
Nitrile/N602-70	–53	–58	–45
Arctic Nitrile™	–60	–67	–58
Camlast	–4	–9	–6
EP/E515-80	–63	–76	–62
EP/E529-60	–63	–76	–62
EP/E692-70	–58	–33	–62

Table 3. Decomposition onset temperature for alternate seal materials.

Materials	Onset Temperature, °C (°F)
Fluorel/V747-75	466 (871)
MFC/V835-75	456 (853)
Silicone/S383-70	516 (961)
Silicone/S604-70	497 (927)
Silicone/S650-70	504 (939)
FS/L677-70	483 (901)
Nitrile/N304-75	444 (831)
Nitrile/N602-70	447 (837)
Arctic Nitrile™	442 (828)
Camlast	446 (835)
EP/E515-80	458 (856)
EP/E529-60	458 (856)
EP/E692-70	456 (853)

One important consideration for any potential O-ring material is its ability to be compatible with the lubrication used on the seal itself. In the RSRM's, Conoco HD-2 grease is used as both an anticorrosion agent for the D6AC steel cases, as well as a lubricant for the elastomeric seals. This Conoco grease offers extremely good corrosion protection to the RSRM cases, but is a detriment in leak checking the elastomeric seals due to its very high viscosity at room temperature. A search was made to find a replacement for this grease; however, no suitable replacement was found. Thus, any elastomeric seal used in the RSRM's had to be compatible with this petroleum-based Conoco HD-2 grease.

To establish this grease compatibility, or lack thereof, a series of tests was performed. Typically, the test methodology utilized grease exposure per ASTM D471-79 to determine such parameters as volume swell, dimension change, and Shore A hardness change. Each test was performed with at least three samples. A small length of O-ring or a small button of rubber sheet stock was used for testing. The sample was coated with grease by hand or was totally immersed in the grease. Samples were stored at either 75 or 120 °F for periods of 14 days up to 180 days. Figures 4 and 5 show the results for some of the materials tested in this test series.[2] In general, the test results indicate that the candidate materials generally fell into three categories: (1) those greatly affected by the grease—the ethylene/propylenes, (2) those that were still compatible but displayed measurable property change—the silicones and nitriles, and (3) those that were only slightly affected—the fluorocarbons and the fluorosilicone materials. The gross incompatibility of the ethylene/propylene materials with the Conoco grease led to their being dropped from any further consideration as potential replacement materials.

It was also found that the grease absorption associated with silicone S650-70 and Arctic Nitrile™ materials resulted in the accumulation of a sticky, nonlubricating residue on the O-ring surface. Further investigation into this residue revealed that it was caused by the lower molecular weight fractions of the grease preferentially migrating into the O-ring, thereby leaving the higher molecular weight fractions and other additives to collect on the surface.

Testing for mechanical properties was performed on six elastomeric seal materials. Desirable mechanical properties for an O-ring in the RSRM included high tensile strength, low modulus, insensitivity to both low and high temperatures, high contact loading in compression, and high damage tolerance.

Tensile testing was performed on 2-ft lengths of the elastomer cord stock and was performed at 10 in/min until the rubber ruptured. Testing was performed on virgin material and on rubber pieces in which a 0.01-in deep cut has been purposefully placed. Compressive load-deflection tests were performed on O-rings made from the rubber cord stock. Each specimen was deflected at 0.05 in/min until a metal-to-metal condition was obtained. Loads were then calculated as load per linear inch. Tear strengths were determined according to ASTM D624. Tests were run at 10 in/min until rupture and test strengths were calculated as ultimate load divided by specimen thickness.[3]

The results of the tensile strength testing are shown in figure 6.[3] The tested materials seem to congregate into two groups. The fluorocarbons and nitriles have a significantly higher tensile strength than the silicones and fluorosilicones.

Load-deflection testing of the materials at 75 °F and 0.052 in of deflection indicated that the fluorocarbon materials and the Arctic Nitrile™ material were the stiffest materials (fig. 7).[3] At 25 °F, the harder materials exhibited the greatest loads, and all materials showed greater stiffness at the lower temperatures. One interesting test point to note is the very large increase in compressive load of the V747-75

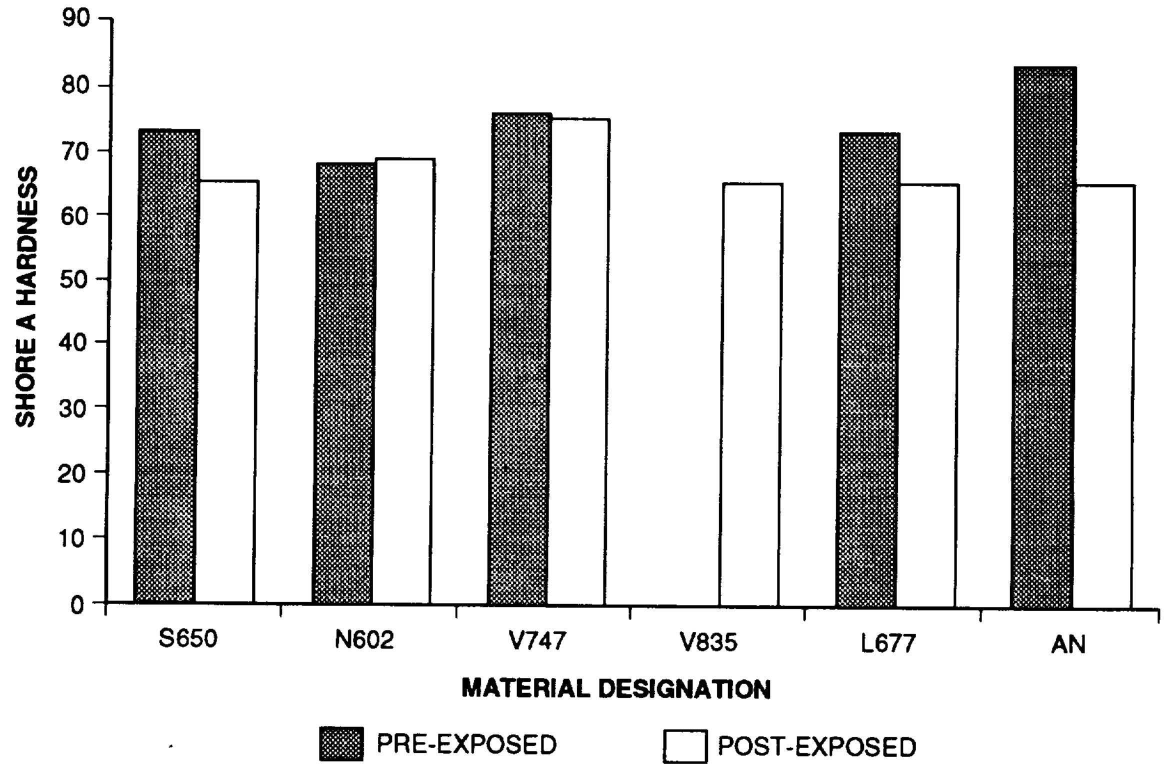


Figure 4. Grease compatibility, alternate O-ring materials, exposed for 60 days/120 °F.

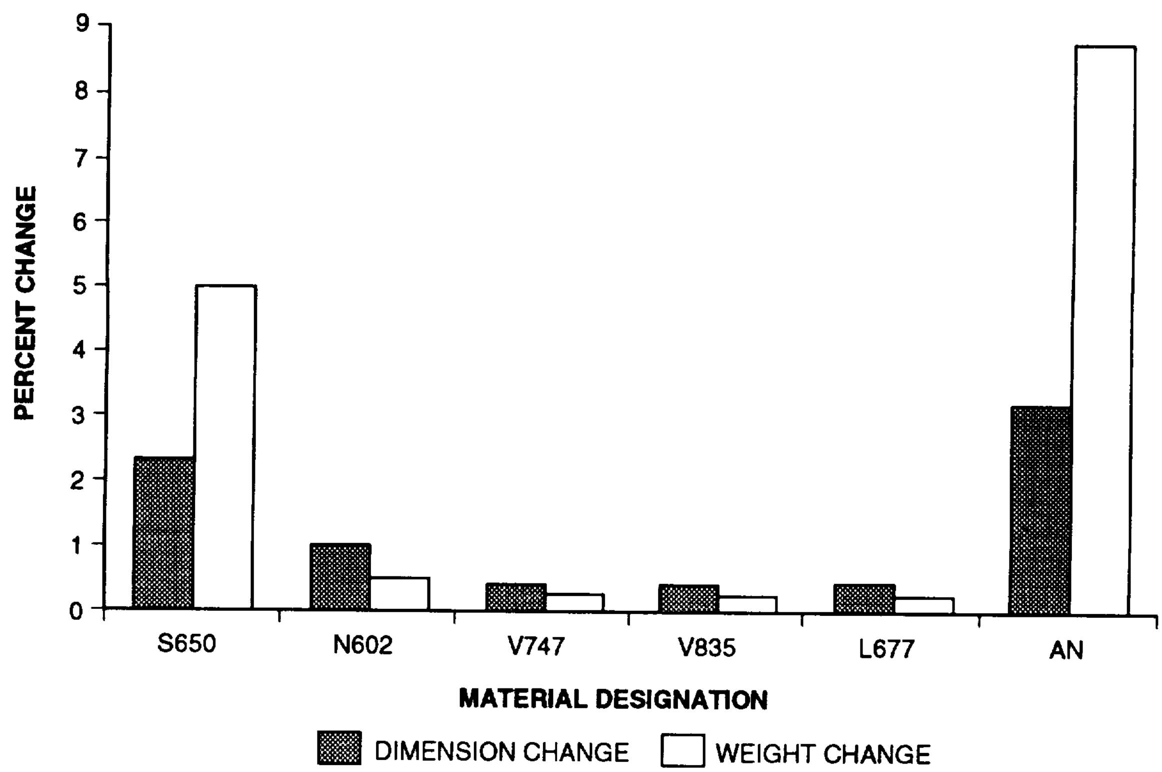


Figure 5. Grease compatibility, alternate O-ring materials, exposed for 60 days/120 °F.

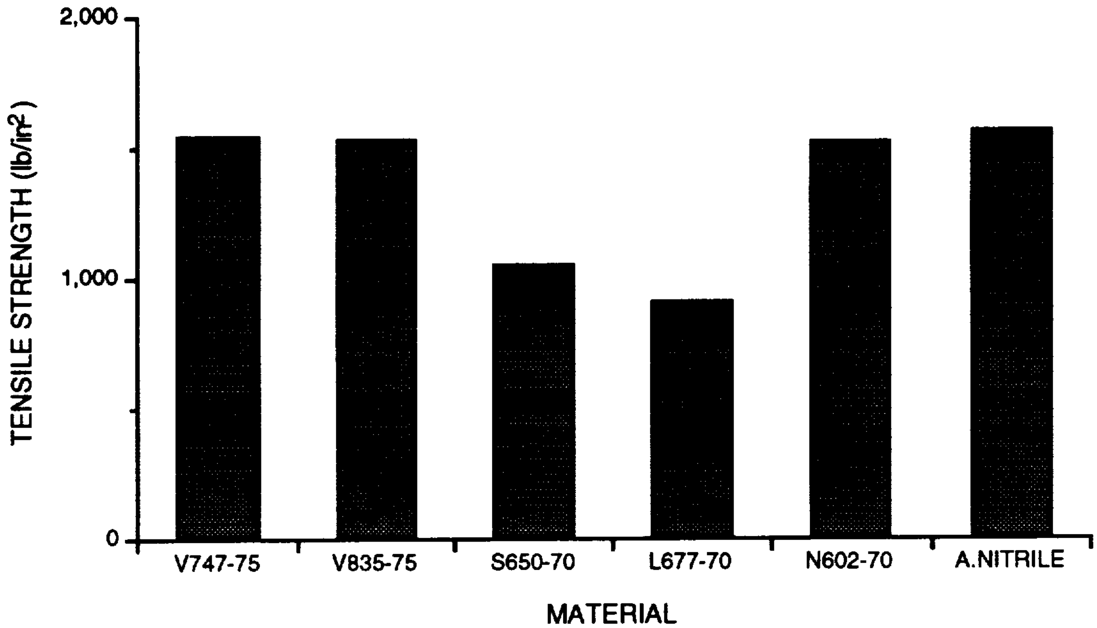


Figure 6. Ground cord stock tensile strengths.

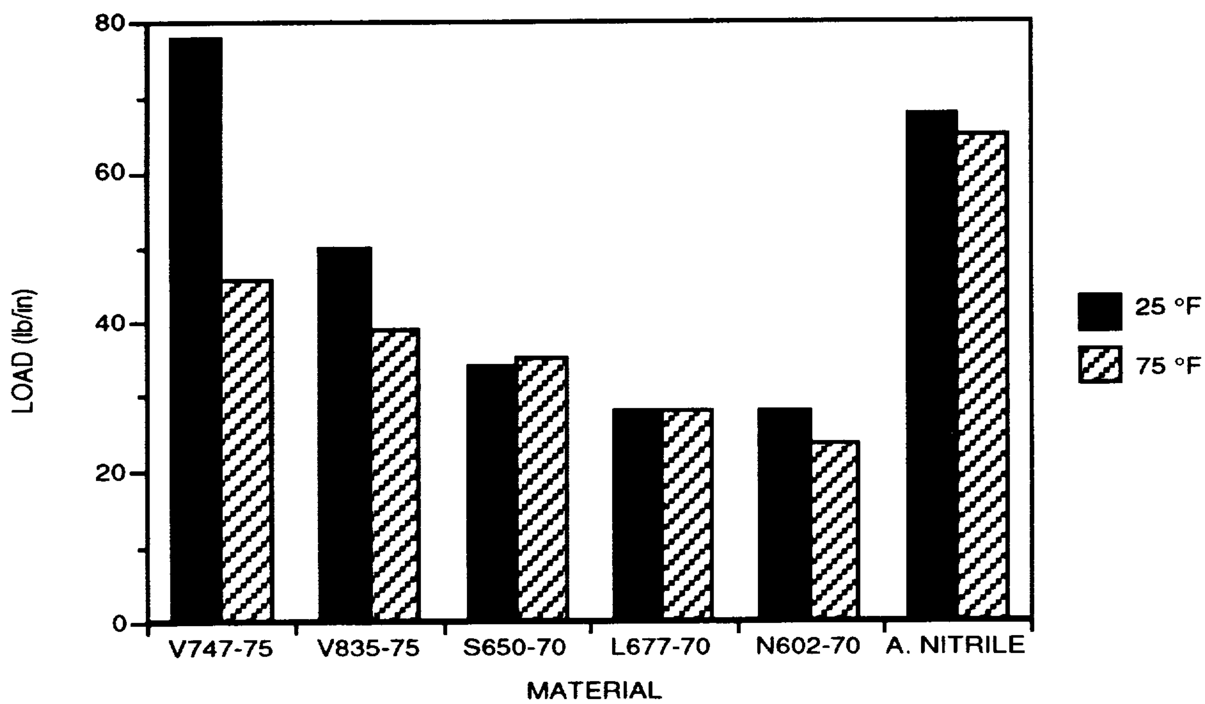


Figure 7. Compressive loads at 0.052-in deflection.

fluorocarbon material at the lower temperature. This can be attributed to the fact that the test temperature (25 °F) is close to this material's glass transition temperature.

The damage susceptibility of the O-ring materials can be assessed by the results of the three tests of (1) tear strength, (2) flawed tensile strength, and (3) assembly damage. Figure 8 shows that the fluorocarbons and nitriles have greater tear resistance than the silicone or fluorosilicone materials.[3] The tear strength of the silicone S650-70 material is higher than what is usually noted for commercially available silicone materials.

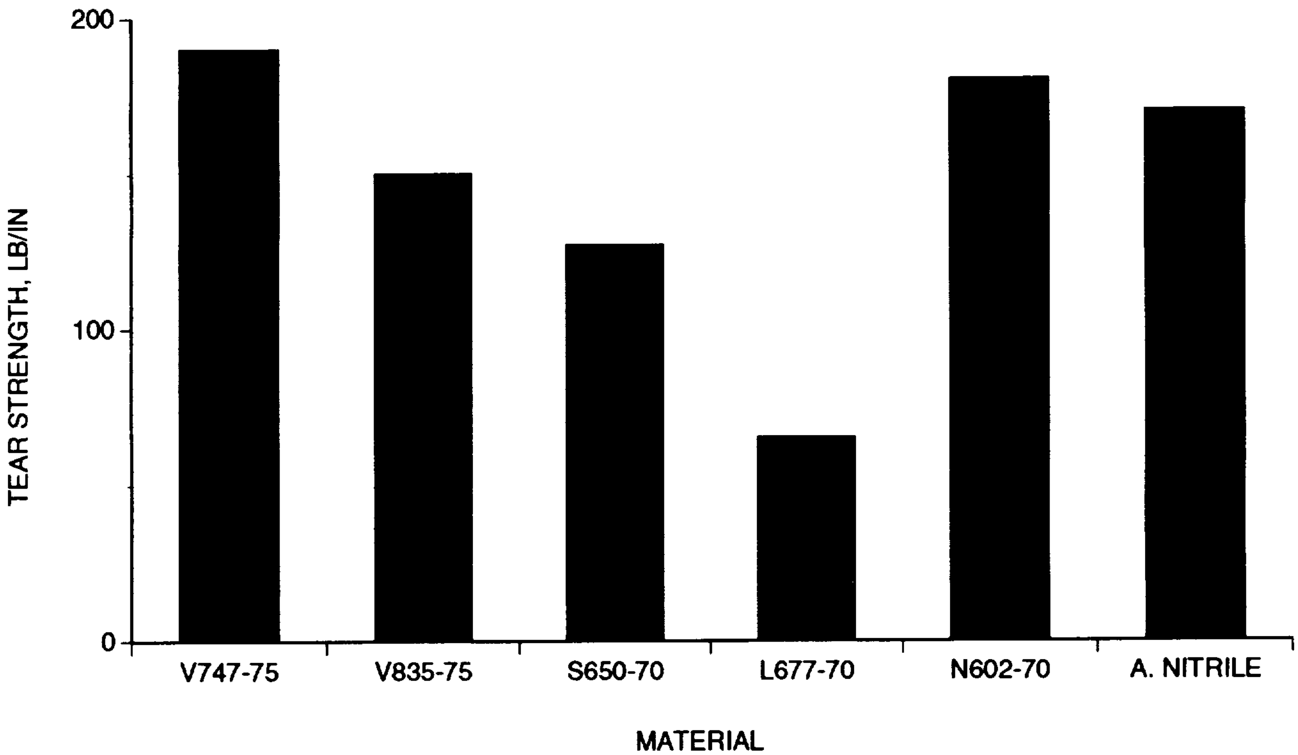


Figure 8. Tear strengths at 75 °F.

Testing was performed on O-ring cord stock material to assess the tear strengths of the material. Tests were performed per ASTM D624 methodology. Tests were run at 10 in/min until rupture, and tear strength was calculated as ultimate load divided by specimen thickness. The results of the flawed tensile strength are shown in figure 9.[3] The nitrile N602-70 materials performed best, while the fluorosilicone material had the least flawed tensile strength.

The O-ring materials were also tested for installation damage tolerance utilizing the test fixture, designed by Dr. Jerry Patterson as seen in figure 10, that attempts to simulate an RSRM field joint during installation operations.[3] The simulated tang was forced into the simulated clevis at 0.05 in/min, both with and without the Conoco HD-2 grease present.

The results of the assembly damage testing showed the importance of lubrication on the O-ring materials during assembly. All of the tests (except one) performed with ungreased materials resulted in damage to the material during assembly. All of the tests (except one) performed with greased materials did not yield any damaged materials. Both of the exceptions were with the modified fluorocarbon V835-75 materials.[3]

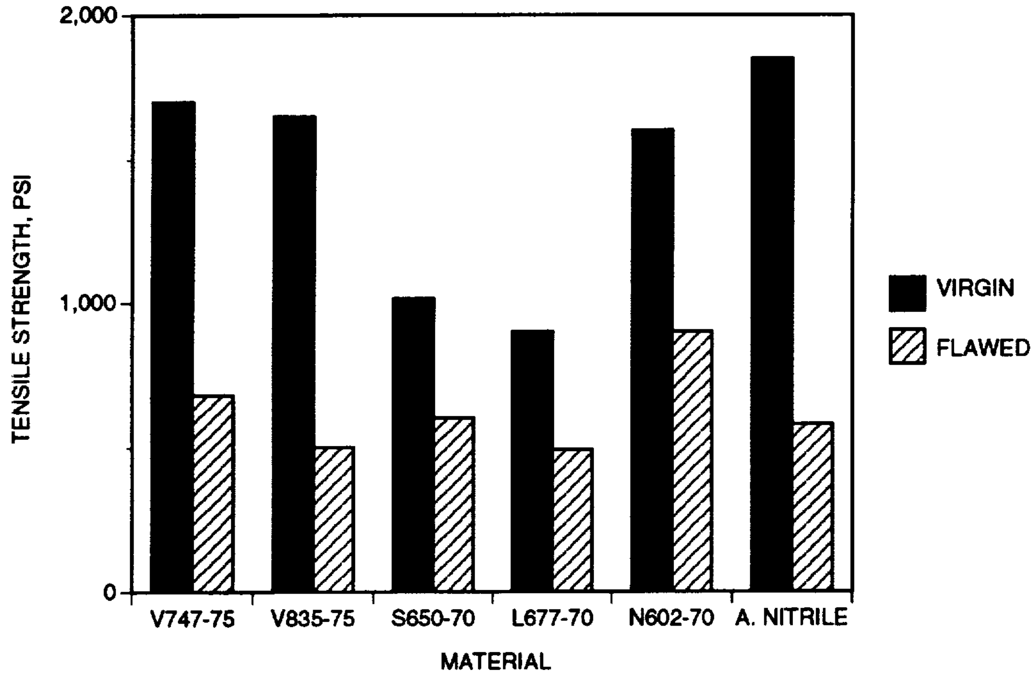


Figure 9. Virgin and flawed tensile strengths at 75 °F.

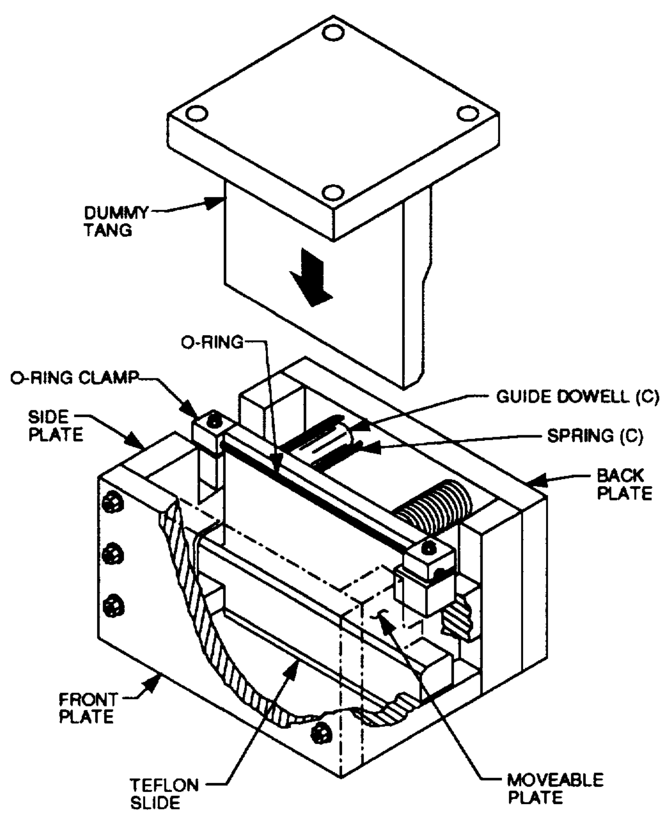
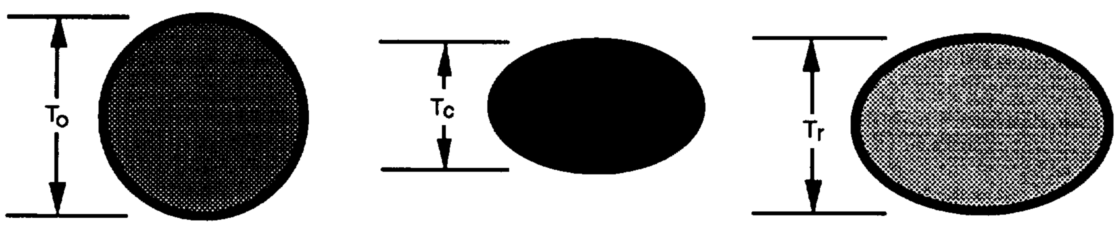


Figure 10. Installation damage tester.

Another area of testing involved the performance of compression set testing. One of the basic viscoelastic properties of elastomer or rubber materials is a significant relaxation with time in response to an applied stress. In the RSRM joint configuration, the O-ring is under constant strain for a period of up to 180 days until a gap opening occurs during motor pressurization. During this constant strain (compression), the O-ring will slowly relax, resulting in a decrease in the stress state. A portion of this deformation is nonrecoverable, and this portion is referred to as compression set. This factor of compression set has a direct effect on the rate of dimensional recovery of the seal, particularly in a condition such as that in an RSRM field joint.[4]

The testing performed on several materials for the compression set utilized ASTM D385 test techniques. The methodology for determining compression set (as a percentage) is shown in figure 11.[4] While this testing did give an indication of the overall compression set that a material might acquire over a long period of time, it was not sufficient to determine the adequacy of the material's response to RSRM joint opening conditions (<0.600 s). Other test methods were devised to evaluate the material's response over this short time transient and will be discussed later.



T_o = ORIGINAL THICKNESS

T_c = COMPRESSED THICKNESS

T_r = RECOVERED THICKNESS

PERCENT SET = $(T_o - T_r)/(T_o - T_c) \times 100$

DEFLECTION = $T_o - T_c$

COMPRESSION SET = $T_o - T_r$

Figure 11. Compression set test methodology.

One unique set of tests that was performed on several of the elastomeric O-ring materials was to test the materials to their upper temperature limits. The five materials tested were:

(1) Fluorocarbon V747-75

(2) Arctic Nitrile™

(3) Modified fluorocarbon V835-75

(4) Silicone S650-70

(5) Fluorosilicone L677-70.

In this test series, the hardware consisted of a steel plug with two O-ring grooves, a pressure port, and four thermocouple ports. The O-rings were installed into their grooves and then the cylinder was placed into a cylinder housing with four thermocouple ports. A carbon steel plug with a 1/2-in hole was added to the bottom of the plug to minimize the amount of flame impingement on the inside of the inner cylinder. This provided more uniform temperature readings between the cylinder thermocouples and the housing thermocouples.

Two types of tests were performed using this fixture. The first test series was performed as a "heat-to-failure" type of test. In this test series, a light coat of Conoco grease was applied to the O-rings and O-ring glands. The O-rings were then installed into the fixture. The fixtures were assembled so that a 0.02-in initial gap existed at the two O-rings (approximately 17-percent squeeze on the O-rings). Thermocouples and pressure lines were then connected to the test hardware. A leak check was then performed on the system using 1,100 lb/in^2 gauge. After the successful leak check, a pressure of 1,000 lb/in^2 gauge was placed between the O-rings. The fixture was then heated from the bottom with an oxygen/acetylene torch at a rate of approximately 5° per second. The pressure between the O-rings was monitored for signs of leakage past the O-rings during heating. Heating was continued until the 1,000 lb/in^2 gauge began to leak past the O-rings.

The failure temperature of the O-rings was determined by analysis of the thermocouple readings closest to the point of failure visually noted in the bottom O-ring. The bottom O-ring was the site of the failure in all tests. Also, the metal temperature on the inner side of the gland was cooler than the metal temperature on the outside of the gland. Therefore, in an effort to be conservative, the failure temperature was noted as the cooler, inside metal temperature in relation to the thermocouple nearest the failure point. If the failure did not occur precisely at a thermocouple location, the failure temperature was derived from interpolation between the two closest inner thermocouple locations.[5]

The second test series was a "constant temperature" type test. The fixture was heated to 500, 600, or 700 °F and held for 260 s. The oxygen/acetylene flame was extinguished before the test temperature was reached. However, the fixture temperature continued to climb until the test temperature was reached. The test temperature (±10 °F) was then maintained for 260 s. After the end of the 260-s hold, the fixture was cooled with nitrogen gas. If a material did not fail at the first test temperature (500 °F), it was then tested at the next test temperature (600 °F, etc.).

In the heat-to-failure testing, three tests were conducted with each material. Heating rates and failure points were repeatable within these three runs. Testing revealed two distinct events during the temperature rise. The first point noted was at an elevated temperature (420 to 575 °F) at which the first sign of fire from the fixture was noted. This was believed to have possibly been the Conoco grease igniting. The second point was the temperature at which the pressure blew past the seals. Typically, there was a 10- to 20-s time delta between the two events, corresponding to a temperature rise of 50 to 100 °F.

In order to test the theory that the grease was serving as an ignition source, two tests were conducted using ungreased fluorocarbon O-rings. Results from these two tests did not differ significantly from the other tests. The possibility exists that once the O-rings reach a certain elevated temperature and pressure, volatiles are driven off from the O-rings. The volatiles are then ignited by the oxygen/acetylene flame.

An approximate failure temperature was found by visually noting the point of failure on the O-ring and correlating this to the nearest thermocouples. However, this is only an approximate failure temperature. To more clearly ascertain the true O-ring temperature at failure, hypodermically injected thermocouples were used. These thermocouples were inserted into several fluorocarbon V747-75 test O-rings and the test-to-failure tests were repeated. Results from these tests indicate that the O-ring temperature at failure was in fact bounded by the inner and outer thermocouples at the location of failure. Results from this test series are summarized in table 4.[6]

Table 4. Hot O-ring test data (average of three runs per material).

Material	Time to First Flame (s)	Temperature to First Flame (°F)	Time to Blowout (°F)	Temperature at Blowout (°F)	Heating Rate (°F/s)
Arctic Nitrile*	86	506	103	590	5.3
Nitrile N602-70	88	510	99	562	4.9
Silicone S650-70	77	462	93	546	5.5
Viton V747-75	106	575	111	601	5.5
Fluorosilicone L677-70	81	421	96	513	6.3

*2 runs

In the series of tests called "constant temperature," more emphasis was placed on investigating the degree of damage that occurred to the elastomeric seal material during exposure to elevated temperatures. Effects studied were extrusion, internal/external damage, permanent set, and sealability. Also, the temperatures at which the pressure blew past the O-rings were noted. Tables 5, 6, 7, and 8 detail the results of these tests.

Table 5. Test temperature, 500 °F, 260 s.

	N602	V835†	V747	AN	L677	S650
Extrusion (mils)	40	None	None	5	25	35
Internal cracking	No	Yes*	No	No	Yes	No
Surface damage	No	Yes*	No	No	Yes	Yes
Permanent set (percent)	69	9	3	42	2	1
Seal	Yes	Yes	Yes	Yes	Yes	Yes

*Occurred at the Hydrapak joint.
†Two tests.

Table 6. Test temperature, 600 °F, 260 s.

	V747	AN	L677‡	S650†	V835§
Extrusion	None	30	50†	50†	None
Internal cracks	Yes	Yes	Yes	Yes	Yes
Surface damage	Yes*	No	Yes	Yes	Yes
Permanent set (percent)	12	100	10	32	16
Seal	Yes	Yes	No	No	Yes

*Occurred at the Hydrapak joint
†Due to extreme damage, exact extrusion amounts not determined
‡Two test
§One test

Table 7. Test temperature, 700 °F, 260 s.

	V747†	AN	V835*
Extrusion (mils)	50‡	40‡	50‡
Internal cracks	Yes	Yes	Yes
Surface damage	Yes	Yes	Yes
Permanent set (percent)	81	100	46
Seal	No	No	No

*One test
†Two tests
‡Due to extreme damage, exact extrusion amounts not determined

Table 8. Approximate failure points.

Silicone S650	T = 635 °F	66 s into 600 °F hold
Fluorosilicone L677	T = 645 °F	55 s into 600 °F hold
Modified fluorocarbon V835	T = 660 °F	Before 700 °F hold reached
Arctic Nitrile	T = 663 °F	Before 700 °F hold reached
Viton V747	T = 712 °F	178 s into 700 °F hold

At 500 °F, all the materials tested sealed for 260 s. The two nitrile materials, N602 and Arctic Nitrile, showed very high amounts of permanent set. Due to grease absorption problems discovered in parallel with this test series, the N602 was dropped from further testing. The silicone, fluorosilicone, and modified fluorocarbon materials all exhibited signs of external damage. Internal cracking was also noted in the fluorosilicone material. External and internal damage that occurred in the modified fluorocarbon material was limited to the area of the splice joint. The tested O-rings all had one splice joint made by Hydrapak, Inc. Due to the problems associated with the splice joint in the modified fluorocarbon material in this test and other tests, the splicing process was improved.

At the 600 °F test temperature, the fluorosilicone and silicone materials failed approximately 55 to 60 s into the test. Both materials exhibited large amounts of extrusion, as well as internal and external damage. The silicone material showed a moderate amount of permanent set, while the fluorosilicone exhibited only a small amount of set. The two fluorocarbon materials and the Arctic Nitrile material did seal throughout the 260-s hold. All three materials, however, did show signs of internal damage. The Arctic Nitrile material exhibited an almost 100-percent permanent set, while the two fluorocarbon materials showed only small amounts of set.

For the testing performed at 700 °F, none of the materials successfully sealed throughout the 260-s hold period. The modified fluorocarbon and Arctic Nitrile material failed at approximately 660 °F (during the temperature rise up to 700 °F). The fluorocarbon V747 material failed 178 s into the 260-s hold at 700 °F. All three materials showed significant extrusion and internal/external damage. The Arctic Nitrile material once again showed 100-percent set. The baseline fluorocarbon V747 material also exhibited very high set, while the modified fluorocarbon exhibited only a moderate amount of set. The

Shore A hardness of the two fluorocarbon materials showed little change after testing. The hardness of the Arctic Nitrile material, however, increased approximately 20 percent.[7]

DYNAMIC TESTING

In addition to the basic material properties type testing, additional tests were being performed to determine the sealability of various O-ring materials in simulated RSRM field joint conditions. In order to screen the candidate O-ring materials, test fixtures were designed that would, as practically as possible, duplicate the actual sealing environment of an RSRM field joint. The fixtures that were manufactured had to be capable of varying the amount of gap opening that was imposed on the test O-rings and had to be able to apply pressure to the O-rings at various times. United States Patent 5,000,033 has been assigned to this fixture design.

The basic design that was chosen to test the various O-ring materials is shown in figure 12. The fixture consisted of three basic parts: a bottom housing (piston), an inner "cone," and a top "hat." The piston walls and the cone walls were cut so that both were at a 15° angle but were parallel to each other when assembled. The cone portion of the fixture had three O-ring glands in which O-ring materials

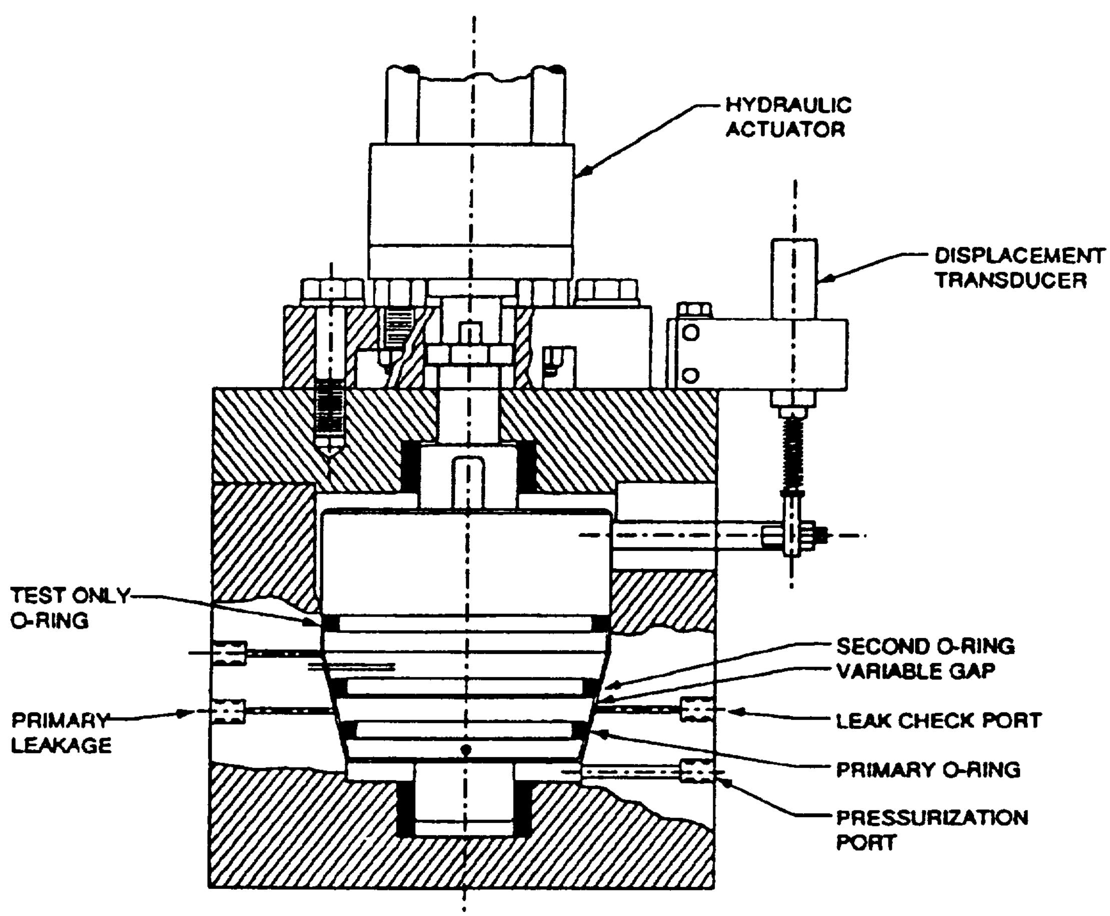


Figure 12. Dynamic cold gas pressurization test fixture.

were installed. The test O-ring was installed to hold any pressure that went past the first two O-rings. The first two O-ring glands were manufactured such that the bottom of the gland was parallel to the sealing surface. The cone was attached to a hydraulic cylinder. The cylinder was used to actuate the piston, thereby producing a radial gap opening at the O-rings, as well as an axial gap movement. The total magnitude and rate of the gap opening was controlled by a Cyber Systems profile generator. Pressure application could be varied in terms of magnitude, rate, and placement with respect to the gap opening. Pressure transducers monitored supply pressure and any blow-by past the first O-ring. The fixture contained five thermocouples to monitor temperatures of the O-ring during thermal conditioning and during actual testing.[8]

By controlling the hydraulic cylinder, several different gap opening conditions were examined. As more knowledge was gained from actual RSRM hardware measurements, more refined versions of gap opening curves were used. Pressurization curves used were head-end motor pressure traces taken from actual flight motors. Toward the end of the testing program, a more conservative 3-sigma pressurization curve was employed.

The fixture was designed to the O-ring gland dimensions that were being used at the time of the fixture construction. Consequently, the first test fixture was constructed with the primary O-ring gland 0.209–0.216 in deep, by 0.360–0.370 in wide, with a 20° slope on the front side of the O-ring gland. Small "scallops" were manufactured circumferentially on the cone ahead of the primary O-ring gland. These scallops were to allow pressure to reach the first O-ring more readily.

As the redesign effort continued, a new plug was made to test the gland designs. This new plug was manufactured, and it eliminated the scallops, decreased the front side gland angle from 20° to 0–5°, and increased the gland width to 0.375–0.380 in.

As testing progressed, a larger fixture (fig. 13) was built using the same basic features as the one described above. This new fixture had a diameter of approximately 12 in compared with the smaller 4.5-in fixture described above. This fixture further updated the gland design such that the gland width was reduced to 0.355–0.360 in. This change was made to minimize any effects of circumferential flow in the gland while at the same time not allowing the gland to be more than 90 percent filled by the O-ring. This 90-percent fill requirement was imposed to assure that a basic contractual requirement was met. This requirement says that the O-ring and sealing system must accommodate pressure assistance. The terminology of pressure assistance was coined to describe the physical act whereby, if pressure reaches the seal, the pressure serves to aid the O-ring in its ability to seal. To meet this requirement, the O-ring cannot touch on all three sides of the gland walls and the sealing surface simultaneously. Analysis has shown that if the O-ring is touching on all four sides and pressure reaches the O-ring during the gap opening sequence, the O-ring will be inhibited from sealing due to the pressure acting on the O-ring. On the other hand, if the gland is designed such that the O-ring is only touching on three sides (one of which is the surface that is moving radially away from the O-ring), then the pressure will serve to actuate the O-ring into a sealing position.

Although there were many variations on the subject parameters of testing, some of the most basic test procedures remained constant from test to test. Prior to O-ring installation, the test fixture was cleaned with a solvent and allowed to dry. The fixture was then inspected to insure cleanliness. A light coat of Conoco HD-2 grease was applied to the O-ring gland, and the O-rings were lightly greased. Following O-ring installation, the plug/hydraulic cylinder was installed into the housing, facility plumbing connected, and transducers and thermocouples installed.[9]

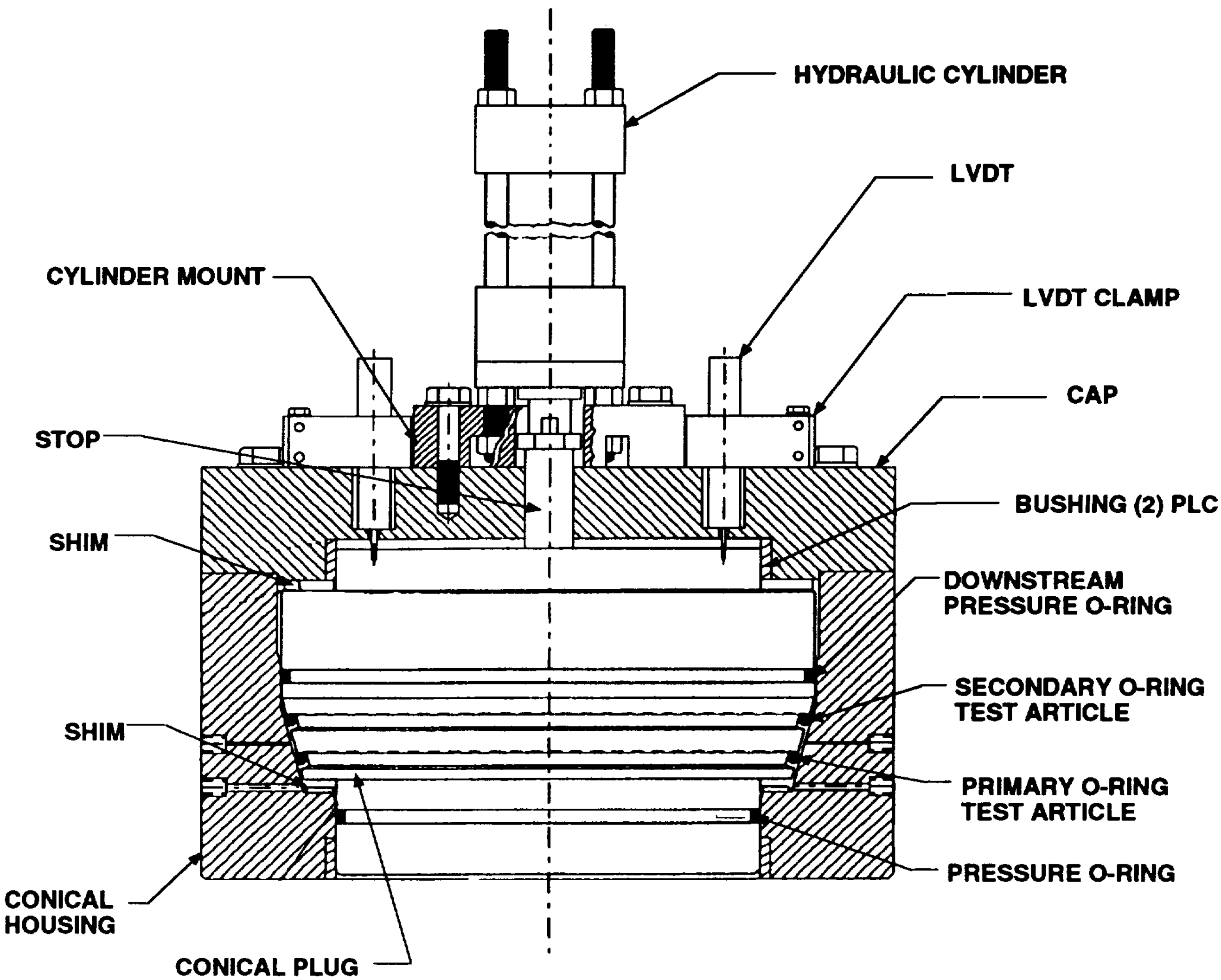


Figure 13. Dynamic cold gas pressurization test fixture.

After installation of the O-rings and assembly of the fixture, the plug was adjusted to verify that the O-ring gap was the same as specified by the test requirements. A leak check/seating procedure was then performed. This procedure allowed the system to be checked for any leakage that would represent an O-ring that had been damaged or the presence of any large contaminants. This procedure could also be altered to place the first O-ring in different positions in the gland.

Following this leak check procedure, the O-rings were conditioned to the required test temperature. For subambient conditions, liquid nitrogen was used to cool the fixture/O-rings. For elevated temperatures, a resistance type heating strip was used to warm the fixture. Typically, the fixture/O-rings were maintained within ±2 °F of the specified temperature for 15 min after the temperature of the fixture had stabilized at the required temperature. Once these conditions had been met, a test was performed.

The initial tests of the various O-ring materials were performed to ascertain the materials' performance as a function of decreasing temperatures. The O-ring materials were subjected to two types of testing. The first condition assumed that the gap opening at the O-ring occurred at the same time as the

O-ring was being pressurized. The second condition took into account the phenomenon called "case rounding" that was thought to exist in the original solid rocket motor (SRM) design. This phenomenon assumed a certain amount of ovality in the SRM cases before motor ignition. As the internal motor pressure reached approximately 50 to 150 lb/in^2 gauge, the motor cases suddenly became concentric. This motion created a very rapid gap opening at the O-rings.

The first set of tests that was performed dealt with the O-ring's ability to track a gap opening condition under "case rounding" conditions. In these tests, 50 lb/in^2 gauge was placed on the front side of the primary O-ring. After approximately 100 ms of exposure to 50 lb/in^2 gauge, the hydraulic piston was commanded to raise the cone, thereby opening the gap. Gap opening was performed as quickly as the equipment would allow (approximately 15 to 20 in/min). The initial gap at the first O-ring was essentially zero (metal-to-metal), while the final gap was a test parameter and varied from 2, 6, 10, 14, and 18 mils. Figure 14 shows the results from the tests in which the final gap was 18 mils.

All of the materials tested demonstrated an ability to seal the gap opening at ambient temperatures (70 to 80 °F). As the test temperature decreased, some of the materials could not effectively seal. Testing on the ethylene/propylene E692 material was discontinued during this test series due to severe grease absorption problems discovered in other test efforts. Testing was also discontinued on the silicone

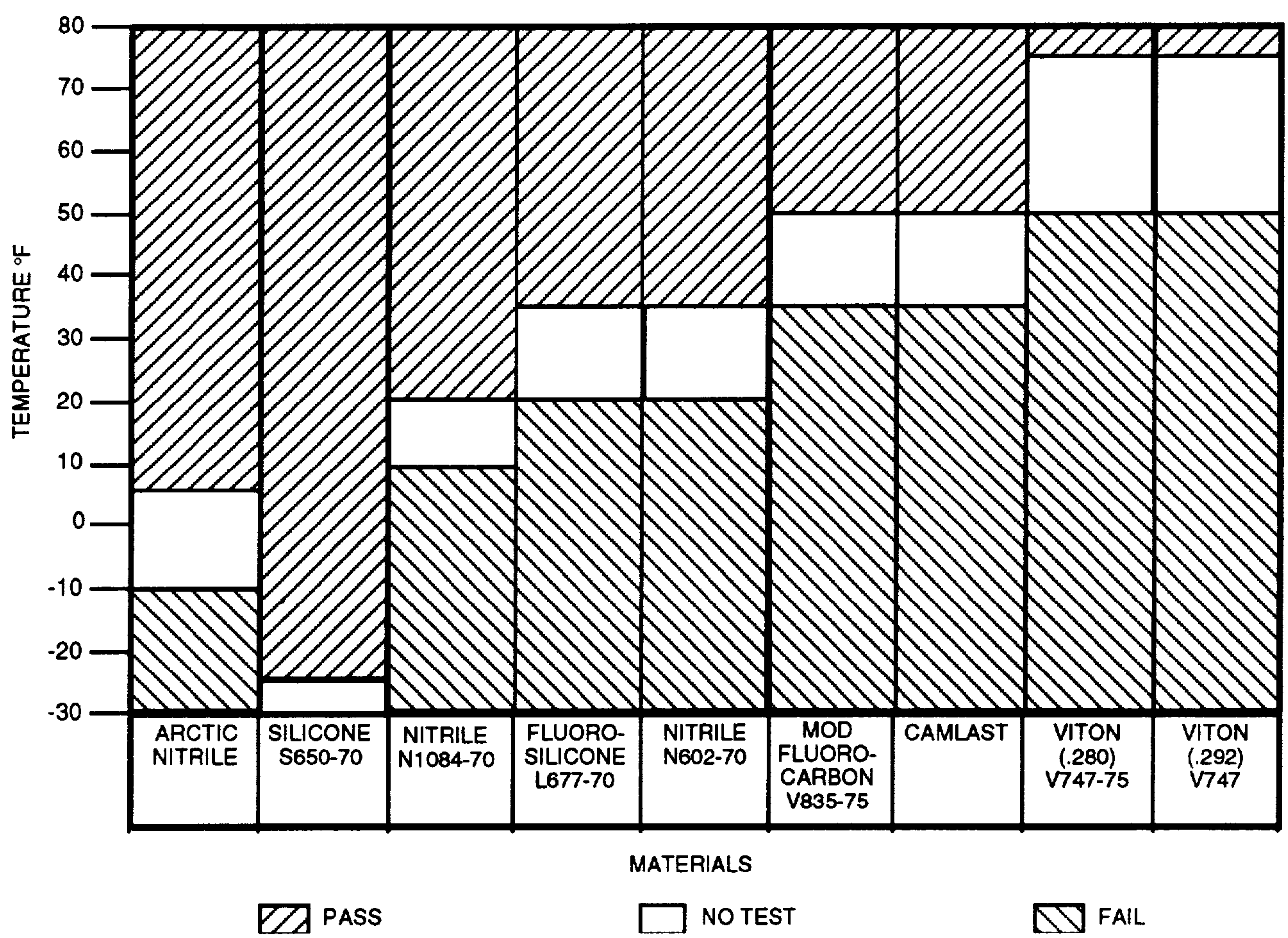


Figure 14. Rounding test results.

S604-70 due to the O-ring being susceptible to damage caused by O-ring extrusion into the gap during testing.

The second set of tests involved simultaneously pressurizing the O-ring as the gap was opened. Figure 15 shows the typical curves used for these tests. Results from these tests are shown in figure 16. These results indicate that, in this pressurization mode, all materials effectively sealed down to temperatures of 35 °F.

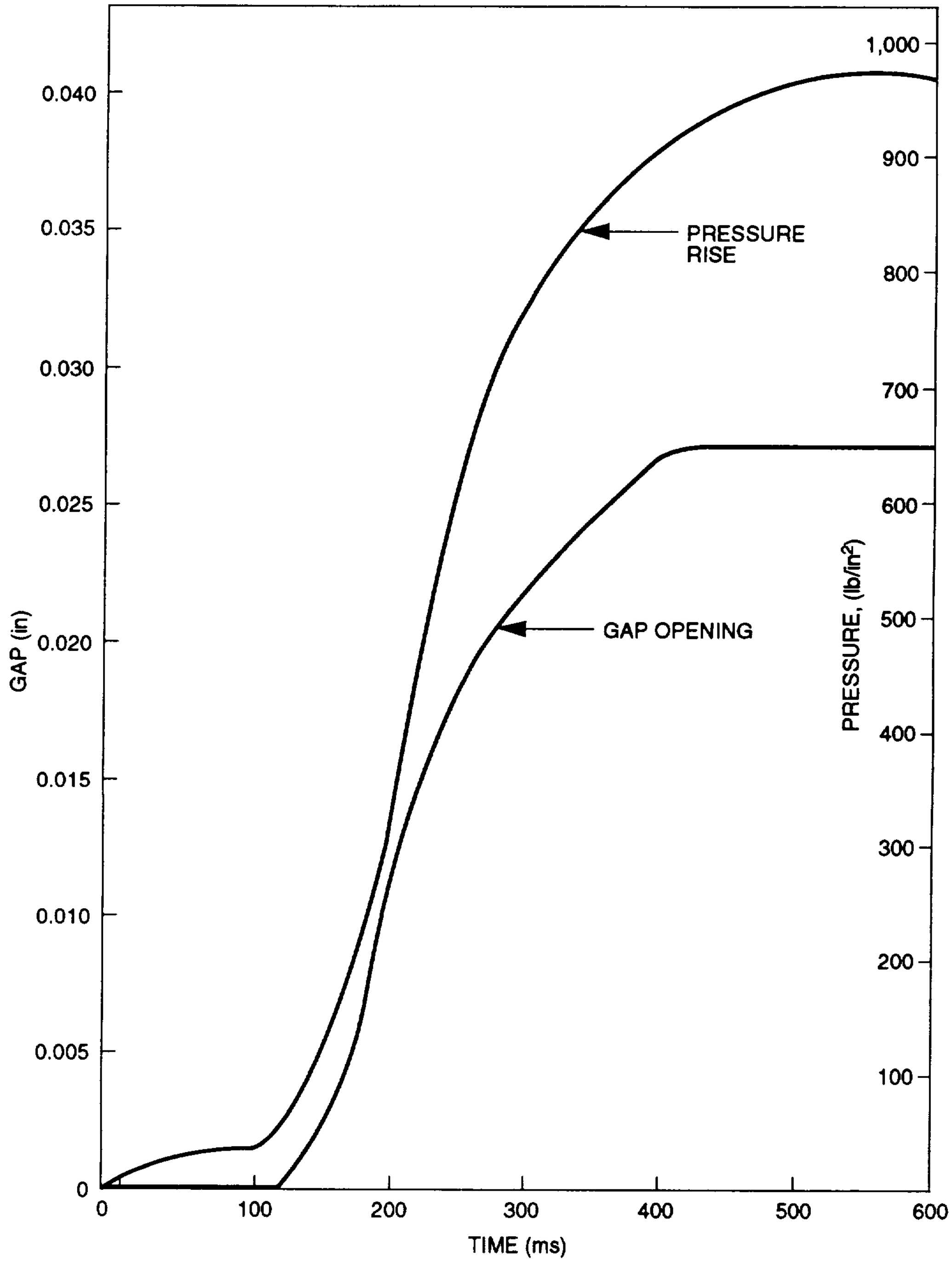


Figure 15. Gap opening and pressure rise versus time.

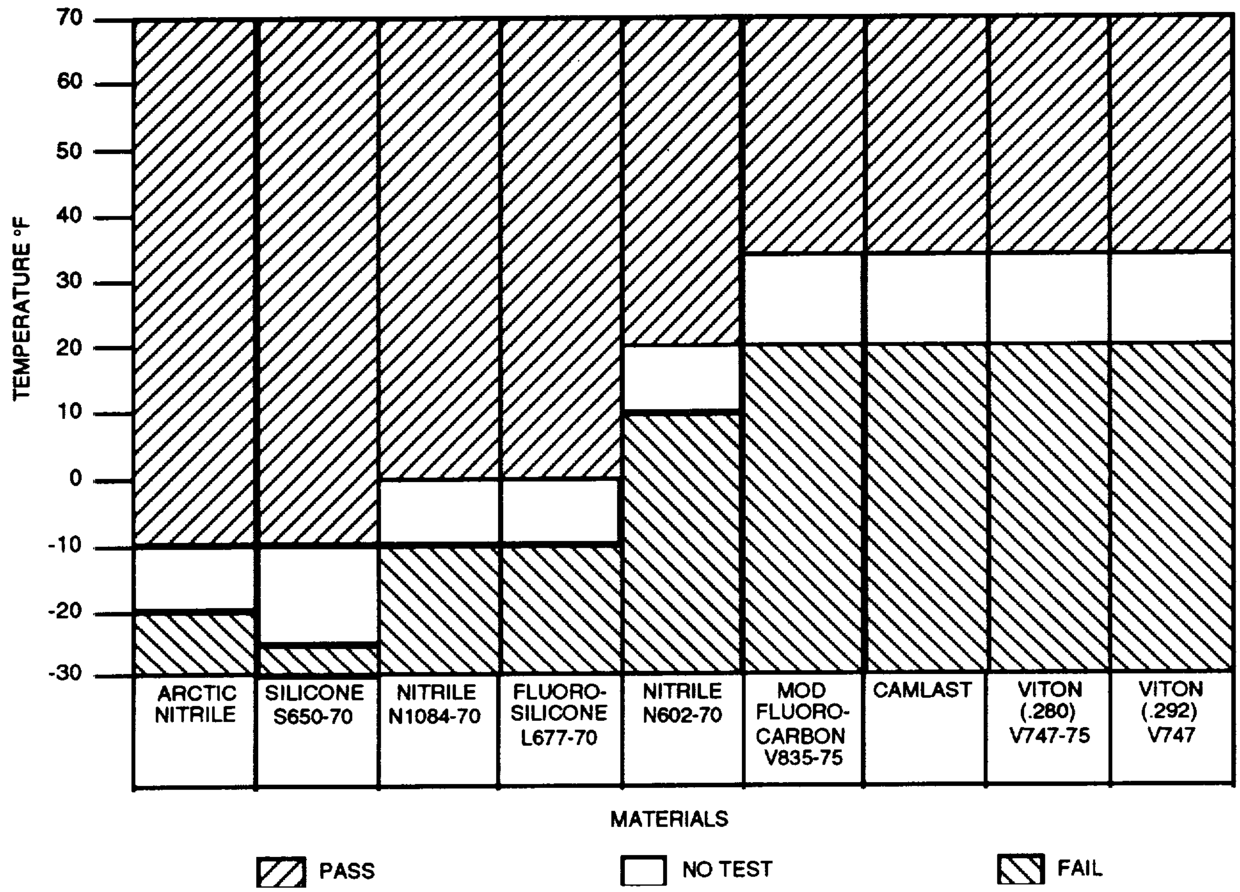


Figure 16. Pressurization test results.

Additional testing continued with the implementation of larger test fixtures. Tests were performed to evaluate parameters such as the effects of delayed pressurization on the O-ring sealing ability and the effect of the O-ring position in the gland on the sealing ability of the O-ring. One stringent requirement that the O-ring had to meet was that it had to seal at twice the maximum expected gap opening rate. This requirement was used to formulate the gap opening curves for both dynamic testing as well as all resiliency testing.

The next series of tests was performed using the larger piston cone fixture. The first two series of tests were performed to investigate if there were any effects of scaling up from the 4.5-in fixture to the larger fixtures. The first set of tests performed was the "rounding" tests as described previously. Results seen from these tests essentially duplicated the results seen in the 4.5-in rounding tests. The next set of tests performed was the pressurization type tests also previously described. Once again the results from the tests using the larger fixture were basically the same as those for the smaller fixture.[10]

The emphasis of testing then shifted to the determination of the effects of the O-ring position in the gland prior to pressurization. In this series, the O-ring was either placed at the correct "in position" or at the "out of position." These terms describe the O-ring's position in the gland relative to the pressurization direction. Figure 17 shows the orientation of the O-ring in the gland in both of these positions. When the O-ring is "in" position, it is at the same side of the O-ring gland before pressure acts on the O-ring as it will be after pressure acts on the O-ring.

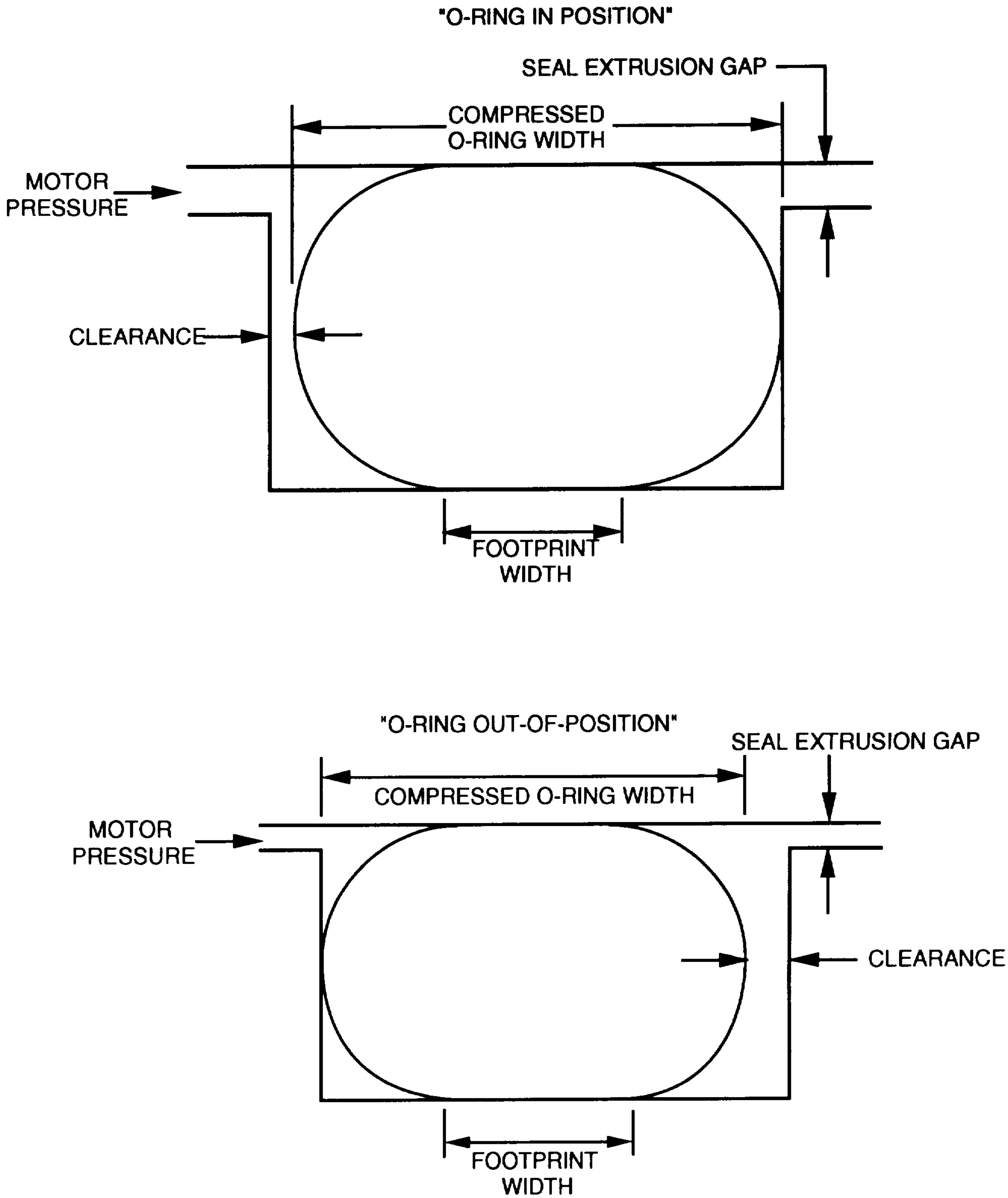


Figure 17. O-ring position.

An "in" position test was defined as first introducing 100 lb/in^2 gauge between the O-rings, holding this pressure for 5 min, then reducing pressure to zero. Next, 100 lb/in^2 gauge was introduced upstream of the primary O-ring, held for 5 min to once again check for leakage, and then reduced to zero. An "out-of-position" test was defined by the same methods as for the "in" position except that, following the venting of the 100 lb/in^2 gauge between the first and second O-rings, no pressure was introduced upstream of the primary O-ring. This left the first O-ring in the "incorrect" sealing position in the O-ring gland.

Since the volume behind the first O-ring was being monitored for any sign of blow-by, it was decided to investigate what effects would be seen by the first O-ring, "out-of-position," moving the gland as it was pressurized, and what effects the gap opening would have on this volume.

In the first series of tests, the gap at the first O-ring was held constant, and the first "out-of-position" O-ring was slowly pressurized so that the compression of the volume between the O-rings could be quantified. The results of these tests, seen in figure 18, indicate that, on average, the pressure rise between the O-rings was approximately 1.5 to 2.0 lb/in^2 gauge. These tests also showed that at approximately 20 lb/in^2 gauge the O-ring had begun to move across the gland. Recent tests performed by Morton Thiokol with full-scale RSRM hardware have shown that approximately 30 to 50 lb/in^2 gauge is needed to actuate the O-rings.

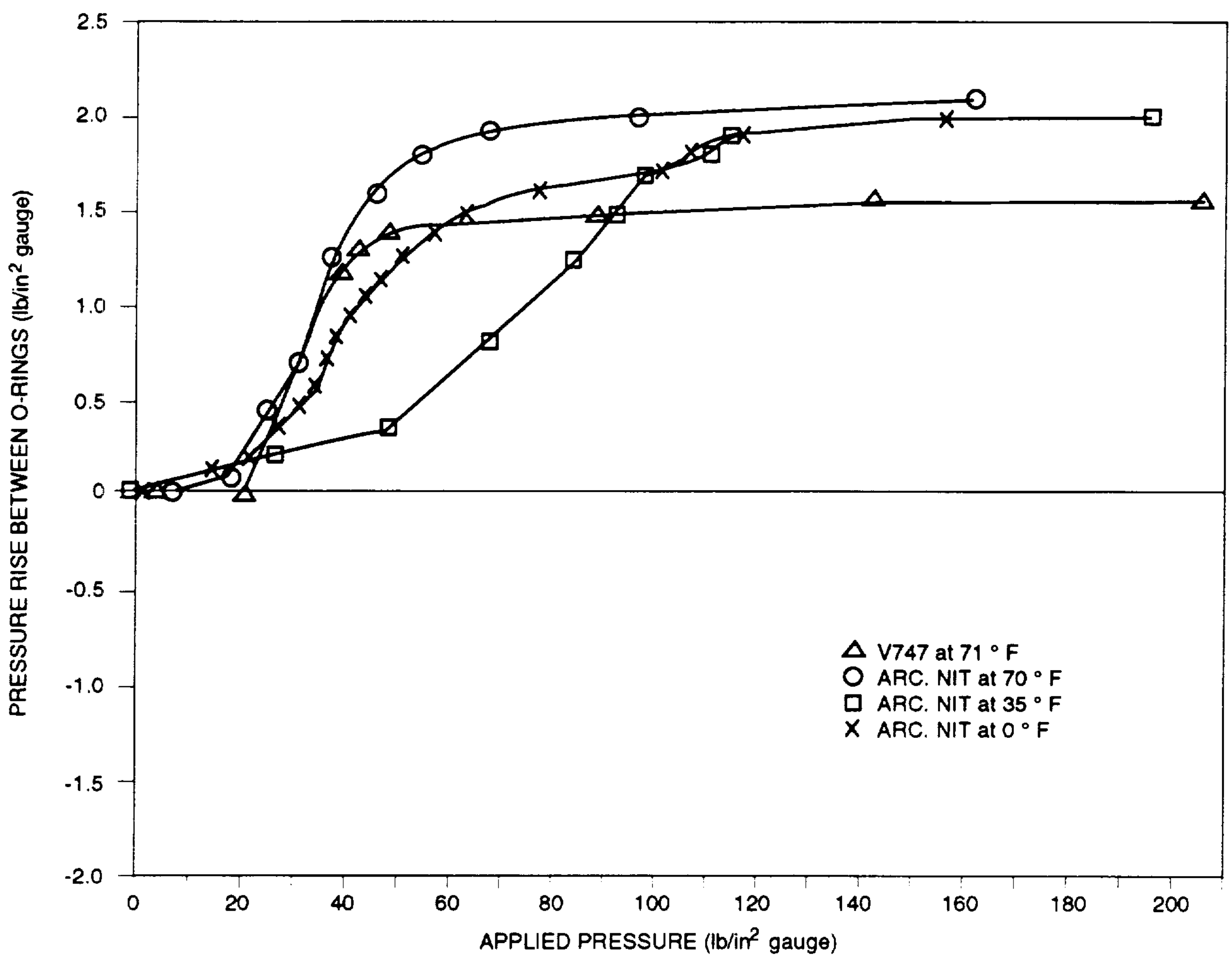


Figure 18. Pressure rise between O-ring with applied pressure and constant gap.

The next series of tests was performed whereby the gap was opened with no pressure applied to the O-rings, and the pressure in the volume between the first and second O-rings was monitored. Figure 19 shows the linear pressure drop versus gap opening (initial gap 0.004 in, final gap 0.016 in).

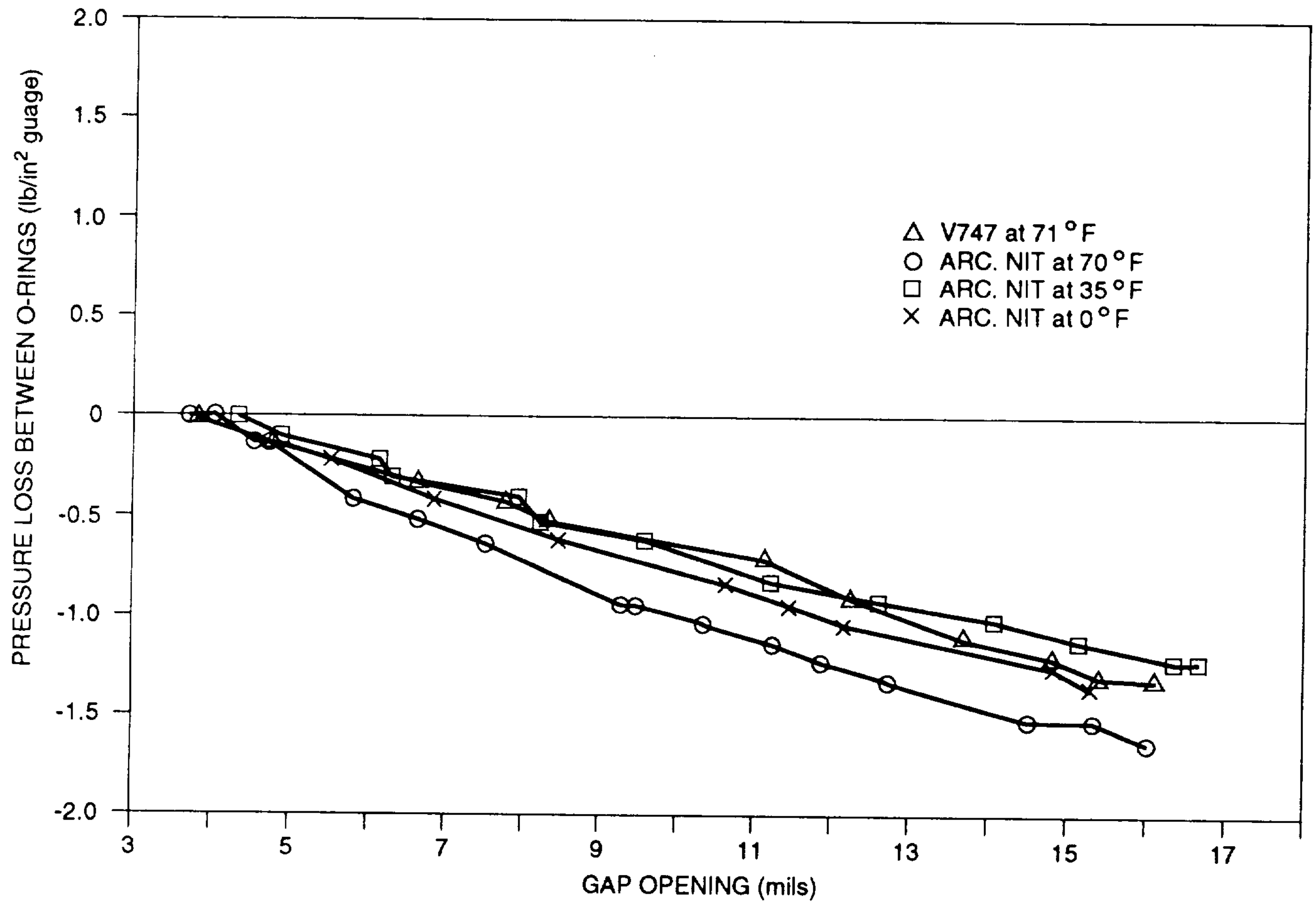


Figure 19. Pressure loss between O-rings with gap opening and constant pressure.

Testing for the effects of O-ring placement in the gland was performed using three different gap opening conditions: (1) gap opening from 14 to 27 mils at 9 in/min, (2) gap opening from 4 to 16 mils at 9 in/min, and (3) gap opening from 4 to 16 mils at 18 in/min. Testing was performed with the fluorocarbon V747-75, Arctic Nitrile, and silicone S650-70 materials. A pressure of 30 lb/in^2 gauge was placed in front of the primary O-ring 100 ms prior to gap movement. A standard pressurization curve was then applied simultaneously with the specified gap opening conditions.

The results of these test series were that the position of the O-ring in the gland did not significantly affect its sealing ability. This is not to say that the sealing ability could not be affected by position. It can be hypothesized that, if the sealing surface had an imperfection or if a piece of contamination existed in the sealing system, it could be possible that the O-ring could move over this flaw (or contaminant) and leak as it was translating across the gland. General results indicated that the silicone material successfully sealed at –25 °F, the Arctic Nitrile successfully sealed at 0 °F, and the fluorocarbon V747-75 material did seal at ambient (70 to 80 °F) conditions but failed some of the tests at 50 °F.

After the tests described previously had been completed, standardized gap opening and pressurization curves were formulated. The gap opening curve that was derived was based on the expected gap opening at the primary O-ring in the RSRM field joint. Using the expected gap opening, a safety factor of 2 was applied to both the total magnitude of gap opening and to the gap opening rate (fig. 20). Thus, the O-ring was required to seal twice the expected gap opening at twice the expected gap opening rate. This gap opening curve is referred to as the "2X,2X gap opening curve." Analysis indicated that the maximum gap opening the O-ring might experience was 0.009 in. Therefore, the total magnitude of gap opening was 0.018 in. The largest initial gap was predicted to be 0.014 in. Thus, in the next series of tests, the gap was opened from 0.014 in to 0.032 inches in 0.6 s. Pressurization occurred simultaneously with gap opening. Initial testing began at 69 °F, at which point the O-ring successfully sealed. The test temperature then decreased to 50 °F. Testing continued at decreasing temperature increments of 5 °F until the O-ring began to fail. The first indications of blow-by occurred at 40 °F. The test temperature was then raised back to the last passing temperature, 45 °F, and the tests repeated. At 45 °F, the O-rings did successfully seal.[11]

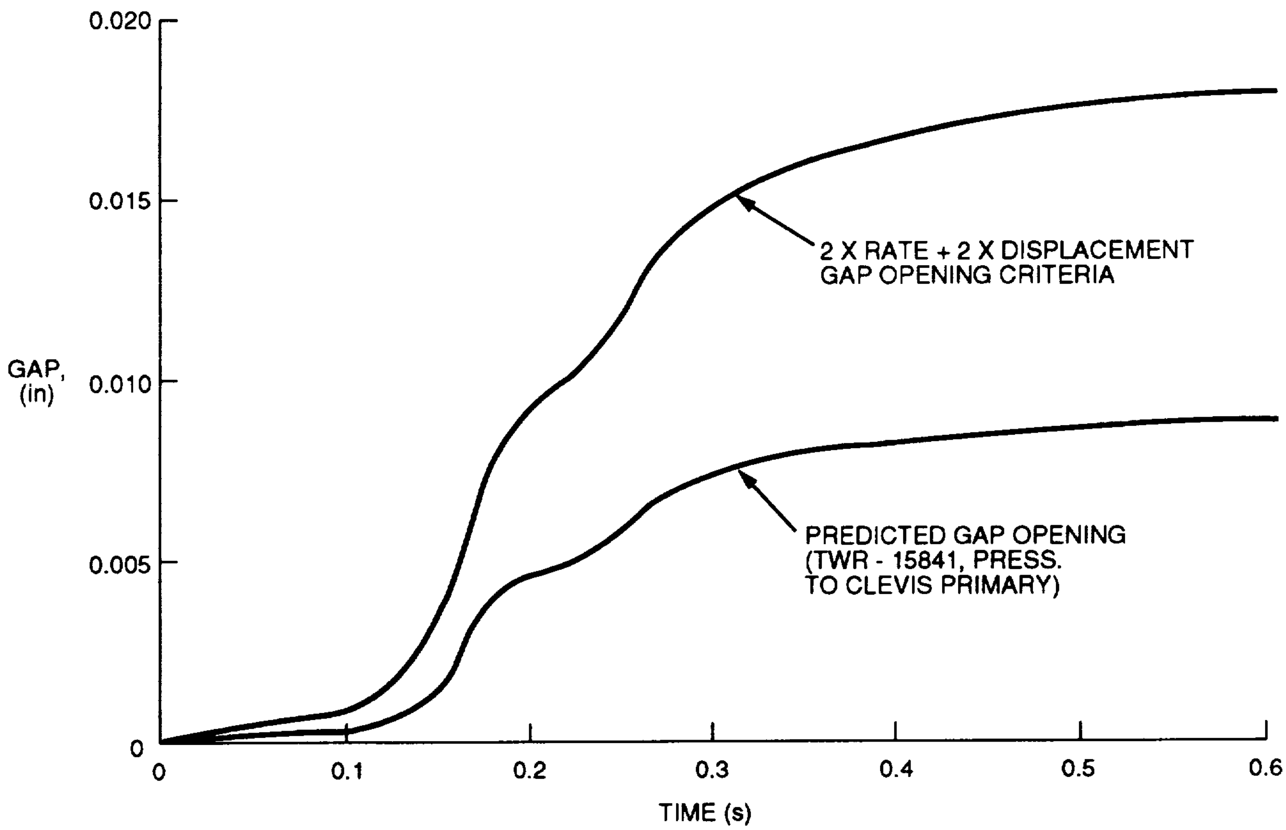


Figure 20. Predicted SRM field joint gap opening versus time.

To assess the effects of pressure assistance, several tests were performed where only 10 lb/in^2 gauge was placed in front of the O-ring instead of the full 1,000 lb/in^2 gauge used in the simultaneous pressurization tests. Under these conditions, the failure point increased from 40 to 45 °F. Evidently, the higher pressure was slightly assisting the O-ring sealing ability.

A final group of tests was performed to assess the effects of delayed pressurization on the sealing ability of the fluorocarbon O-rings. In the current RSRM field and nozzle-to-case joint designs, it is possible that the O-rings could be pressurized after the initial pressure transient. Testing was restricted to a temperature range of 65 to 130 °F. A slight modification was made to the gap opening curve to accommodate the worst case 3-sigma pressurization curve. A small number of tests were also performed with the modified fluorocarbon V835, fluorosilicone L677, and a composite silicone/Viton O-ring.

The minimum initial gap predicted at the O-rings was 0.004 in. The first round of testing utilized this minimum initial gap along with the new 2X,2X gap opening curve and test temperatures of 75 to 120 °F. The point of pressure initiation was at first simultaneous with the beginning of the gap opening. The effects of delayed pressurization were then investigated by delaying the pressure application points until finally the application of pressure was approximately 1 s after the gap opening began. The fluorocarbon O-rings sealed under all test conditions. The other materials tested also sealed successfully under a reduced testing schedule.

The next round of tests involved the maximum initial gap, 0.014 in, and the 2X,2X gap opening curve. Once again, pressure application was delayed up to 1 s. The fluorocarbon O-rings and the other materials tested, excluding the composite O-ring, sealed successfully.

The composite O-ring had an inner core of silicone (Shore A hardness of 50) and an outer cover of Viton (Shore A hardness of 78). The thickness of the Viton cover was 0.043 in. The first test performed on this O-ring at the conditions described directly above was successful. However, in the next three tests, blow-by past the O-ring was noted. Upon disassembly of the fixture, the outer Viton cover was found to be split open. The inner core of silicone had become debonded from the outer cover. The elevated temperature of 120 °F apparently had a detrimental effect on the bonding agent between the two materials. Testing was discontinued on this material after these tests.[12]

One of the last test series involved the determination of the combined effects of temperature, pressure delay, and O-ring position in the gland on the sealing ability of the fluorocarbon O-rings. Approximately 500 tests were performed to complete this matrix. Test temperatures ranged from 65 to 130 °F. Pressure application points ranged from simultaneous with gap opening to a 1-s delay after gap opening initiated. Once again, the effect of an O-ring out of position versus in position was investigated. Under all test variables, the fluorocarbon O-rings were able to seal with no blow-by.

The final set of tests conducted addressed the factor of extended amount of compression time that an O-ring seal might experience. Extended compression times allow the full effects of compression set to be realized and any possible effects of aging or grease interaction.

To determine the effects of long-term compression on O-ring performance, specialized test fixtures were manufactured (90M08700) and long-term testing was performed with the fluorocarbon V747-75 material. The test fixtures manufactured were of the same general design as those mentioned previously. However, some changes were incorporated that allowed the O-rings to be under compression for extended periods of time. Figure 21 shows the general design of these fixtures.

The fluorocarbon O-rings were placed in the test fixtures and stored for 180 to 200 days. After leak checking the O-rings, they were then subjected to a field joint test. The field joint gap opening curve was based on the maximum expected gap opening at the primary/secondary O-ring seals in the RSRM field joint. Using the expected gap opening curve, a safety factor of 2 was applied to the total magnitude of gap opening and to the gap opening rate. This curve also incorporated the worst-case effects of a 3-sigma head-end motor pressurization rise.

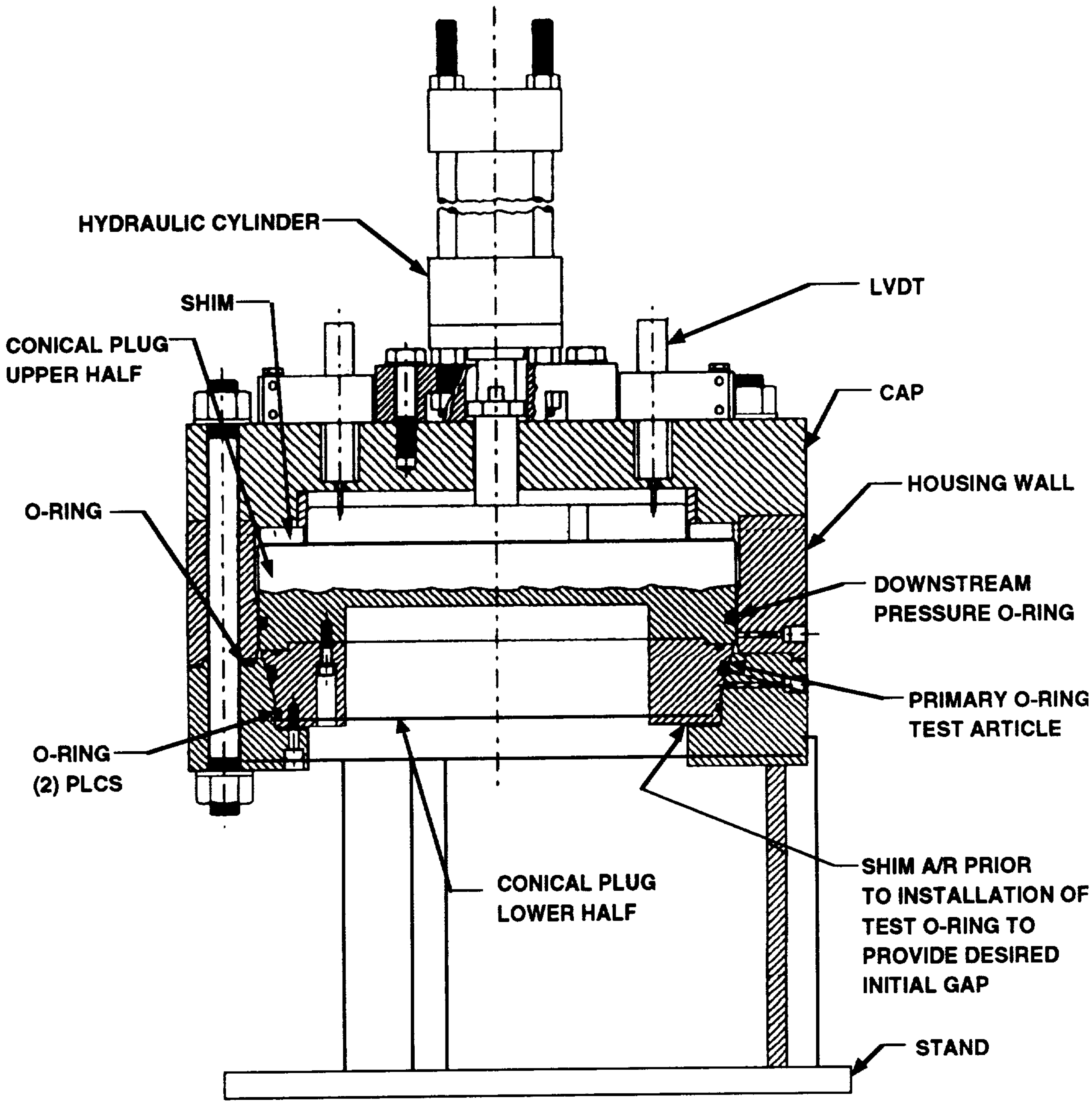


Figure 21. Long-term compression test fixture.

The fluorocarbon O-rings were tested at 75 °F after compression time of approximately 55, 92, and 200 days. The O-rings used in this testing were under approximately 19.7- to 21.7-percent squeeze and were stretched approximately 1.0 to 3.4 percent when seated in the O-ring gland. All of the O-rings tested under these conditions sealed successfully.

Additional testing that was performed with these fixtures centered on the effects of small flaws on the O-ring's performance. Three types of flaws (contaminants) were used, all with diameters of approximately 0.002 in; a copper wire, a felt fiber, and a human hair. All contaminants were placed perpendicular across the O-ring prior to assembly of the fixtures. Six contaminants of each type were used in the test, for a total of 18 tests. Nine fixtures were assembled and compressed for approximately 90 to 110 days with the three types of flaws. Another nine fixtures were assembled and compressed for 180 days. The amount of compression on the O-rings ranged from 19.6 to 21.6 percent. A hard type of flaw, such as a 2-mil copper wire, was readily detected during leak checks and also caused the O-rings to leak during pressurization tests. Softer types of flaws, such as a human hair, yielded more ambiguous results.[13]

RESILIENCY TESTING

During the redesign of the SRM's, it became apparent that there were no useful data available regarding the performance of elastomeric seals in very short time periods (<1 s) for applications in which the O-ring was sealing a surface that was moving radially away from the seal. In order to provide this critical data, a test series was established to determine the elastomeric seals' resiliency properties. This concept of resiliency is attached to the seal's ability to dimensionally recover in both magnitude and rate from an initial compressive strain.[14]

Two types of resiliency testing were performed under the direction of Dr. R.G. Clinton. The first was known as "free resiliency." This testing involved compressing the elastomer seal a known amount for a known length of time and then very quickly removing the compressive load. The recovery of the O-ring was then tracked as it freely recovered. Although this testing did provide very useful data for initial screening tests, a more sophisticated type of test was developed. This testing was referred to as controlled release testing wherein the sealing surface was moved through a controlled gap at a controlled rate, accompanied by measurement of residual sealing load. With this type of testing, a very defined amount of residual sealing force could be determined to exist after the O-ring experienced a simulated field joint movement. The effects of long-term storage, amounts of initial compression, and temperature on the O-ring's ability to seal could also be studied.

Controlled resiliency testing was performed to verify that the seals tested could meet the requirements that (1) the seals would accommodate any structural deflection that occurs in the RSRM joint, and (2) that the seals would be able to maintain sealing capability with twice the expected joint displacement applied at twice the expected gap opening rate. The initial phase of testing began with the O-ring compression set at 18 percent. This value was established through analysis using a combination of worst-case conditions: the minimum O-ring cross-sectional diameter, the deepest allowable O-ring groove, and the largest initial extrusion gap. Additional testing was also performed at 16.5-percent squeeze in order to address the possibility of having reduced squeeze at the O-ring seals due to possible rework areas within the O-ring gland or in the joint assembly itself. Further testing was also performed at even smaller squeeze amounts to determine possible margin in the sealing performance. Also tested were the effects of natural environments on the seal's ability to function properly. In all, the test program was conducted using over 200 O-rings with compression times from 2 h to 360 days.

The list of candidate materials had been narrowed to three by the time this testing commenced. The three materials were fluorocarbon V747-75, silicone S650-70, and modified fluorocarbon V835-75. All O-rings tested were manufactured by Hydrapak, Inc., to RSRM specifications, and all included at least one splice joint. The O-rings manufactured had an inside diameter of 3.625 in and a cross-sectional diameter of 0.290 in. Each O-ring's cross-sectional diameter was dimensionally analyzed using a Zygo laser micrometer to ensure proper dimensions were met.

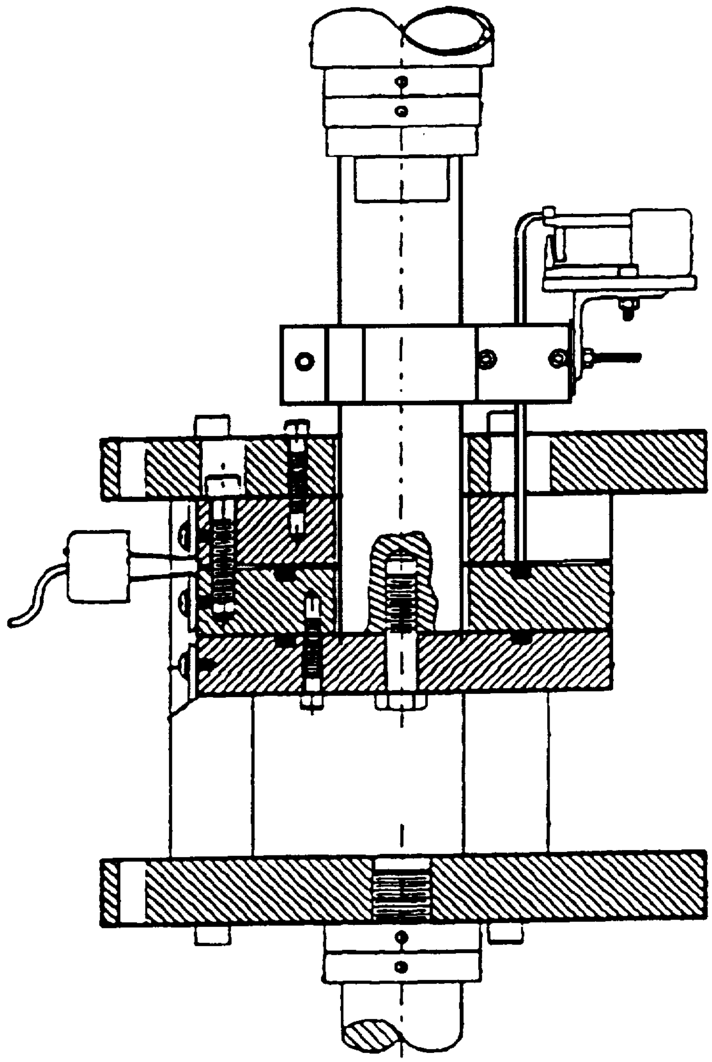


Figure 22. Resiliency test fixture.

The O-ring test fixtures that were built to perform the controlled release testing are depicted in figure 22. Two primary assemblies comprised the complete test fixture. The first was the storage fixture in which the O-ring was compressed, stored, and finally tested. The second was the test frame which was connected to the MTS machine, and into which the storage fixture was mounted.

The storage fixture consisted of four basic parts: base plate, top plate, shims, and a face place ("donut"). An O-ring groove of precise RSRM dimensions was machined into the base plate and contained the actual test O-ring.

A light film of Conoco grease was applied to the O-ring, groove, and the face of the donut. The O-ring was then placed in the groove and the donut placed on the O-ring. Shims, as needed, were then installed, and the top plate was bolted to the base plate. The fixtures were then stored in the prescribed environment for the requisite length of time.

Prior to testing, a calibration procedure was performed to assure the accuracy and consistency of the test. The key to the procedure was to verify that parallelism was maintained in the setup. Extremely tight manufacturing tolerances of each element in the test setup helped to ensure parallelism, but a pretest to double check the parallelism postassembly was deemed necessary due to the criticality of the fixture alignment on test results.

The test fixtures were instrumented with an extensometer, two clip gauges, and two thermocouples. The clip gauges monitored the gap opening, and the extensometer measured the O-ring dynamic recovery during the test. The DEC PEP 11/23 computer system was used for control and data acquisition.

The most critical result of this test series was the determination that the material baselined for usage in the RSRM (the fluorocarbon V747-75) was able to maintain a positive sealing load when tested at 75 °F after 360 days of compression (and an initial compression of 18 percent). Figure 23 is a plot of the data recorded during this testing that supports this conclusion.[14] Each data point is an average of three individual controlled load tests at the conditions described directly above. A logarithmic equation of the form $Y_i = a^*(X_i^b)+$ error was used to fit the experimental data.[14] Extrapolation of the curve to a 720-day end point indicates that retention of a positive sealing load after 2 years of compression could be expected.

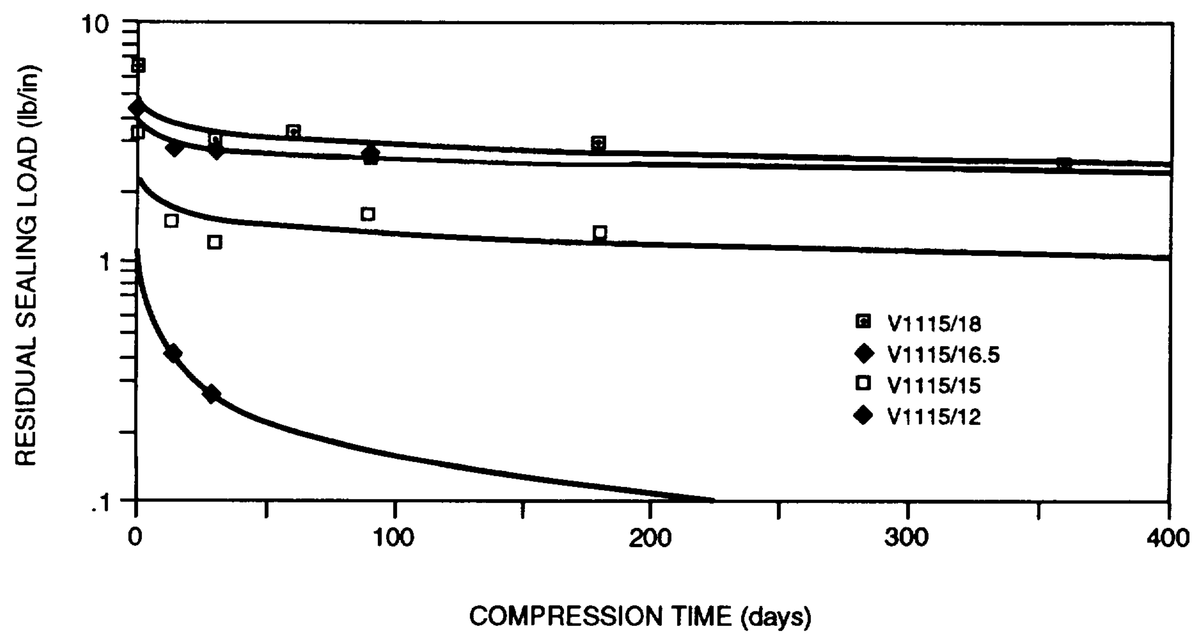


Figure 23. Long-term load/recovery for V1115, 75 °F, various percent compressions.

Testing was also performed at initial compression levels of 16.5, 15, and 12 percent. Results indicate that at 16.5- and 15-percent initial compression, some positive sealing loads were recorded after compression times of 180 days. Data from tests conducted with 12-percent initial compression are somewhat erratic, thus it could not be predicted reliably that a positive sealing load would be seen after 180 days of compression.

Testing was also performed with the fluorocarbon O-ring material to determine any possible adverse effects that natural environments might have on the material's ability to seal. Two separate sets of environments were used. The first was the natural environment at MSFC. The fixtures with their compressed O-rings were exposed to the outside ambient environment but were sheltered from direct sunlight and rain for 360 days. During this year-long period, the maximum temperature that the fixtures experienced was 101 °F, while the minimum temperature was 7 °F. Results from these O-rings undergoing the same controlled release testing as mentioned previously showed that the seals did maintain a positive sealing load after 360 days of compression.

The second set of environmental testing involved simulating the worst-case environments that could be expected at KSC. The three hottest months and the three coldest months were chosen as test conditions. Based on historical weather data and statistical analysis, a model was derived that simulated 99-percentile environment temperatures for the 6 months. This model led to thermal excursion ranging from 61 to 95 °F for the hot months and 28 to 84 °F for the cold months.

Results from the controlled load testing of the O-rings that had undergone this 6 months of conditioning revealed that after 180 days, the O-rings still maintained positive sealing loads.

Testing performed on the other two seal materials, the silicone and modified fluorocarbon, showed that both materials also retained positive sealing loads after compression times of 180 days (initial compression 18 percent). Figure 24 shows a comparison of the test results of the three materials.[14]

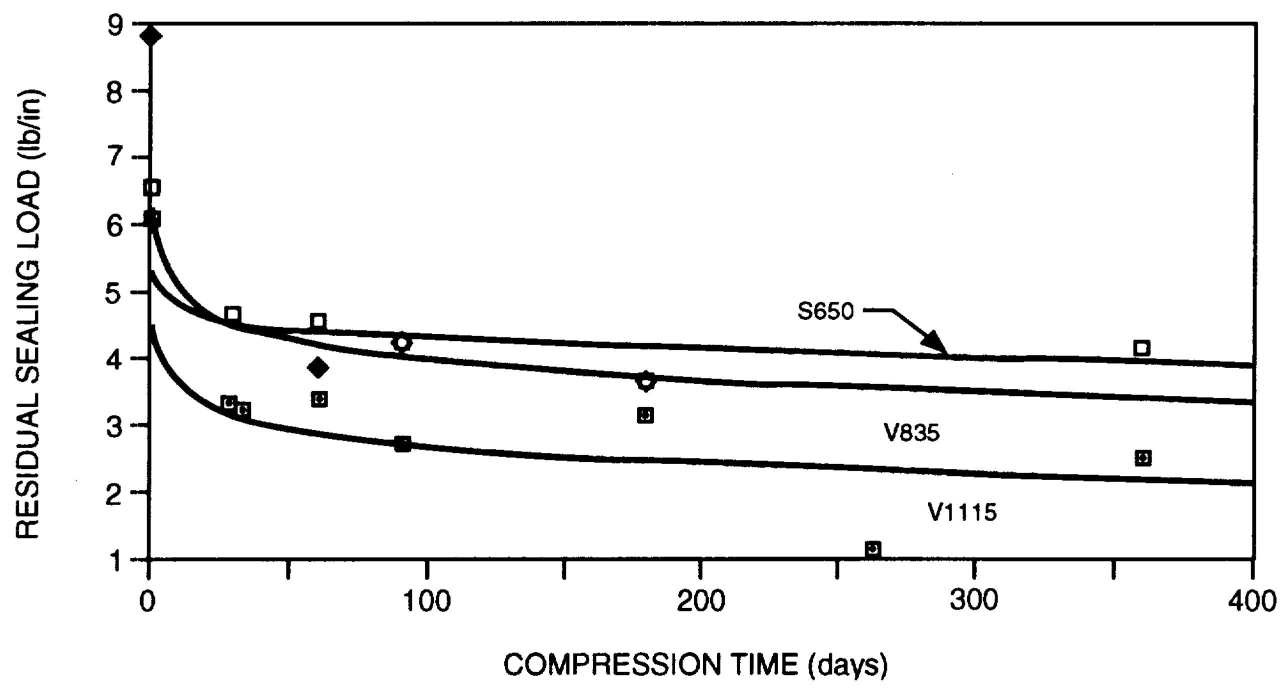


Figure 24. Long-term load/recovery comparisons for alternate seal materials, 75 °F, 18 percent.

CONCLUSIONS

The testing described in this report was performed so that the best performing materials would be chosen to be in the RSRM joints, and that the chosen material would be highly characterized. Also, sufficient test data had to exist so that it could be proven that the O-ring seals made from this material would always maintain a seal during motor operation. Due to the vast amount of testing described here, and also that performed by the contractor, ample data existed to support the choosing of the fluorocarbon V747-75 material as the O-ring seal material. The designation of this fluorocarbon material was changed to V1115-75 due to the stringent process quality control measures that were imposed on the manufacturer of the O-ring seals. No change was made to the material itself.

This report attempts to synopsize the testing that was involved in order to choose a material and then qualify the material for usage. However, not all testing was described. Additional testing, such as leak checking of the seals and the performance of the seals in full-scale and subscale motor firings, was not covered. The testing described covers a time period of approximately 30 months and many thousands of tests conducted at MSFC. Independent testing by Morton Thiokol also covered the same timespan and included thousands of tests. The final result of all of this extensive testing is an O-ring seal material that is fully qualified to be used in the RSRM's and has demonstrated its ability to perform successfully under many worst-case conditions that might occur.

Very important and useful engineering knowledge was gained as a result of the testing described. The most important fact learned from this testing was that the performance of an elastomeric seal in a dynamic environment such as the RSRM is influenced by a very large number of parameters, all of which must be considered in the design of the sealing system. Such apparently innocuous factors as the anticorrosion/seal lubricant used in the sealing system may have adverse effects on the seal material itself, as well as the ability of the sealing system to be effectively leak checked for seal system integrity. The natural or induced environments in which the seal is expected to perform can drastically affect the performance of the seal system.

When all of the factors that could possibly affect O-ring sealing performance are considered, it can be realized that the only way to assure an effective seal performance is by testing the sealing system, or as close as is possible, under the worst-case anticipated performance environment. This is especially true in sealing systems where seal failures are totally unacceptable.

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APPROVAL

A SUMMARY OF LABORATORY TESTING PERFORMED TO CHARACTERIZE AND SELECT AN ELASTOMERIC O-RING MATERIAL TO BE USED IN THE REDESIGNED SOLID ROCKET MOTORS OF THE SPACE TRANSPORTATION SYSTEM

By J.E. Turner

The information in this report has been reviewed for technical content. Review of any information concerning Department of Defense or nuclear energy activities or programs has been made by the MSFC Security Classification Officer. This report, in its entirety, has been determined to be unclassified.

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