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Program: Cover Gas Seals

AEC Task: 11 - FFTF-LMFBR Seal-Test Program

Program Manager: R. W. Keaten

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DOE/SF/71011--T2

I. PROJECT OBJECTIVES

DE82 005376

The objectives of the general seal test program are to: (1) conduct static and dynamic tests to demonstrate or determine the mechanical performance of full-size (cross section) FFTF fuel transfer machine and reactor vessel head seals intended for use in a sodium vapor - inert gas environment, (2) demonstrate that these FFTF seals or new seal configurations provide acceptable fission product and cover gas retention capabilities at Clinch River Breeder Reactor Plant (CRBRP) operating environmental conditions other than radiation, (3) develop improved seals and seal technology for the CRBRP to support the national objective to reduce all atmospheric contaminations to low levels, and (4) publish a ~~Design Guide for reactor seals~~ along with Ordering Data.

Testing of FFTF Instrument Tree inflatable seal to demonstrate its performance is to be conducted. Also the IVHM Inflatable Seal is to be tested to CRPRP conditions to provide design information for the CRBRP.

Design analysis and testing of proposed CRBRP dip seal configurations to determine an acceptable configuration and generate performance data will be conducted.

An additional objective will be to develop advanced seal geometries and materials to overcome deficiencies in permeation rate, compression set and high temperature use of commercially available seals.

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II. MAJOR ACCOMPLISHMENTS DURING FISCAL YEAR 1975

Permeation and compression set data from the FFTF seal characterization tests have been reduced and plotted in graphs to provide a basis for making the recommendations in the Seal Design Guide which has been released as AI-AEC-13145.

Considerable data on the IVHM inflatable seal under CRBRP conditions were accumulated and published in Quarterly Report AI-AEC-13138. A simulated long term dwell (~ 4 mo) under operating conditions has been completed.

Procedures for installing the Instrument Tree inflatable seals have been developed, the seals were installed in the test rig, and two life tests were completed. A failure mode was identified, a seal rework process was devised and implemented on spare seals. The two times life test was repeated on the modified seals with no recurrence of the previous failures.

Several advanced seal geometries and materials that exhibit low compression set and low permeability have been identified and obtained for testing. Permeation and compression set tests have been started on some advanced materials. The test fixture required to test the effects of sodium vapor and aerosols on elastomers was fabricated.

Sub-scale tests of dip seal configurations have produced data for use in formulating a test program and in designing a larger size dip seal mockup. The metal dip seal fixture was designed and is in fabrication. Performance tests in a 30-in. plastic model have been completed, except for mixing tests.

III. MAJOR PROGRESS DURING THE REPORT PERIOD

A. GENERAL SEAL DEVELOPMENT

The compression set testing of elastomer O-rings was completed. The results are shown in Tables 1 and 2. Much of these data are plotted on the curves published in the quarterly report, AI-AEC-13143.

TABLE 1.0
COMPRESSION SET AND HARDNESS VS TIME AT TEMPERATURE
(0.210 in. dia, O Rings at 50% Initial Compression)
(Sheet 1 of 3)

Elastomer	Tem- perature (°F)	% Compression Set / Hardness, Shore A						Initial Hard- ness Shore A
		Weeks at Temperature						
		1	2	4	8	16	32	
Minn. Rubber Nitrile 366Y	100	6.2	8.9	14.3	20.5	27.7	32	73
		73	73	74	73	72	72	
	150	15	22.2	33.3	54.8	66.7	73.0	64
		64	66	69	73	78	82	
	200	43.5	47.3	59.5	65.5	72.7	81.8	65
		69	69	70	70	75	80	
Parker Seal Nitrile N741	100	5.7	6.1	13.6	16.6	20.1	21.9	75
		75	75	75	75	75	75	
	150	6.6	17.8	Seal Cracked				76
		74	74					
	200	11.2	23.8	Seal Cracked				76
		75	74					
		1	2	4	9	15	22	
Parker Seal Nitrile N219	250	74.9	81.8	S				-
		-	-					
	200	64.1	67.7	84.8	95.5	98.8	99.7	70
		-	78	81	89	92	94	
	150	25.3	35.6	45.8	55.8	64.7	S	70
		-	-	75	78	80		
100	17.4	20.3	26.3	30.5	40.0	S	70	
	-	-	72	74	74			
		1	2	4	8	16	32	
Minn. Rubber EPR 559N	200	33.2	36	42.2	43.2	68.5	84	67
		67	67	67	69	74	78	
	250	53.4	61.7	74.4	82.4	89.4	-	70
		68	70	68	66	72		

S = Stopped

TABLE 1.0
COMPRESSION SET AND HARDNESS VS TIME AT TEMPERATURES
(0.210 in. dia, O Rings at 50% Initial Compression)
(Sheet 2 of 3)

Elastomer	Tem- perature (°F)	% Compression Set / Hardness, Shore A						Initial Hard- ness Shore A
		Weeks at Temperature						
		1	2	4	8	16	32	
Parker Seal EPR E529	150	15.1 65	18.2 61	19.2 60	20.2 62	16.2 63	30.4 62	62
	200	41 62	30.7 63	40.4 63	47.1 63	50.9 63	71.2 72	65
	250	54.3 64	62.3 65	72.0 70	84.8 72	97.0 69	S	62
Parker Seal EPR E692	150	6.1 70	11.7 70	13.2 70	15.5 70	16.5 69	15.1 70	82
	200	17.7 67	21.9 68	29.9 69	33.6 71	52.8 72	59.3 74	79
	250	26.4 69	34.1 70	43.7 73	55.2 66	75.3 74	81.7 79	81
Minn. Rubber Silicone 71417	200	12.9 65	19.1 65	22.0 67	23.4 66	38.3 68	40.6 68	70
	250	26.5 66	32.3 60	45.2 68	50.7 68	67.7 74	77.5 77	71
Parker Seal Silicone S684	150	8.2 67	13.6 64	16.5 64	13.6 64	19.4 61	13.6 63	82
	200	16.2 67	21.1 67	25.6 66	31.5 66	48.2 64	64.0 71	82
	250	27.4 67	33.0 66	44.2 67	69.5 70	95.5 74	97.5 75	80
Minn. Rubber Urethane 522FX	100	17.1 70	20.9 69	21.0 67	26.3 59	32.7 62	37.5 61	68
	150	31.6 68	38.2 69	45.3 72	60.8 67	82.5 72	91.0 67	68
	200	81.5 72	90.0 72	S				68
Parker Seal Urethane P642	100	12.7 69	17.7 68	22.5 67	25.5 65	27.5 65	45.0 68	72
	150	29.8 72	36.5 71	49.0 70	63.5 72	79.8 72	91.5 68	72
	200	35.9 71	89.3 62	S				72

S = Stopped

TABLE 1.0
COMPRESSION SET AND HARDNESS VS TIME AT TEMPERATURES
(0.210 in. dia, O Rings at 50% Initial Compression)
(Sheet 3 of 3)

Elastomer	Tem- perature (°F)	% Compression Set/Hardness, Shore A						Initial Hard- ness Shore A
		Weeks at Temperature						
		1	2	4	8	16	32	
Parker Seal Urethane P648	100	70.5	72.4	71.5	81	80	83	91
		93	93	93	94	91	91	
	150	65.5	65.5	72.2	87	88	91	91
		92	92	92	94	96	96	
	200	83.8	87.6	94.2	S			91
		93	93	94				

TABLE 2.0
COMPRESSION SET AND HARDNESS VS TIME AT TEMPERATURES
(0.125 in., O Rings at 50% Initial Compression)

Elastomer	Tem- perature (°F)	% Compression Set／Hardness, Shore A						Pre-Test Shore Hardness
		Weeks at Temperature						
		1	2	4	8	16	32	
Parker Seal Butyl B278-7	250	96.7	100	S				82
		78	78					
	*250	100	100	S				75
		74	74					
	200	91.7	97.7	S				75
		82	75					
	*200	93.3	97.7	S				75
		81	73					
	150	71.2	84.0	-	98	S		79
		78	78		82			
	*150	73.5	88.0	-	93	S		77
		80	80		84			
	100		45.2	-	68.5	84.7		75
			77		77	85		
	*100		41.3	-	69.3	87.4		75
			81		80	83		

*35% Initial Compression

S = Stopped

Four compression set tests to provide supplemental information per a Westinghouse request were started. They consist of testing Parker Buna N N-741 and Minnesota Rubber polyurethane MR-522FX at 115°F under 20 and 30% compression over a 32-wk period. In response to that same request, preparations were started to measure the permeation of O₂ and H₂O vapor through the same two elastomers at 115°F.

The Seal Design Guide was completed and released as AI-AEC-13145.

B. INFLATABLE SEAL TESTS

1. CRBRP Rotating Plug Inflatable Seal

The high temperature storage test on the CRBRP inflatable seal was stopped after 140 days. During this time duration, the fixture had been maintained at 144 to 156°F. While in storage the seal inflation pressure, P_i , was maintained with argon at 15 psig; reactor pressure, P_r , was held at 10 in. H₂O; and the head compartment pressure, P_h , was held at ambient pressure. The buffer pressure which was initially set at 1.0 psig (P_b) was allowed to rise or fall because it was found that this rose to a maximum of 3.6 psig, probably due to permeation of the inflation gas. Throughout test whenever the buffer pressure rose to this value, it was reset to 1.0 psig. Following the storage the seal fixture assembly was relocated intact on the inflatable seal drive fixture and reheated to 150°F in preparation for the seal breakaway drag test. Pressures for the breakaway drag test will be as follows: a cover gas pressure of 10 in. H₂O, buffer gas pressure of 1.0 psig, seal inflation pressure of 7.5 psig, and the head access pressure at ambient. In addition to the initial breakaway drag force, seal drag will be measured at a speed of 15 feet/minute until a stable drag force is reached. Following the breakaway drag test, the fixture will be modified for a seal offset test.

Chemlok adhesive shear strength tests continue. Specimens exposed at 150°F for 4 months were pulled to separation on the Instron tensile test machine. Tests were previously completed on specimens exposed for 1 and 2 months. The four specimens exposed for 1 month all separated at the seal-Instron attachment joint.

Each of the four were returned to the oven and repulled after an additional month's soak. At that time three specimens separated in the seal body adjacent to the adhesive and the fourth at the adhesive-tab interface. Three of the specimens separated at a lower shear stress than previously and this indicated that the seal body or adhesive-tab joint had been weakened to some degree during the initial pull. Two additional specimens soaked for 2 months and the four specimens soaked for four months had not been previously pulled. After these latter six were tested, it was found that separation on 8 of the 10 specimens occurred in the seal body adjacent to the adhesive rather than in the bond area indicating a bond joint stronger than the seal itself. No effect of time at temperature is apparent in the data.

All separations occurred at shear stresses between 125 and 180 psi. The maximum shear stress exerted on the seals during CRBRP tests has been 4.8 psi (114 lbs/ft drag on two seals) with an inflation pressure of 25 psig. Nominal operating conditions are expected to be 7.5 psig inflation pressure and approximately 55 lbs/ft drag (2.3 psi shear stress).

2. Instrument Tree Inflatable Seal Test

After the original 2 times life test program was completed on the original Instrument Tree inflatable seals, a visual inspection of the seals revealed that the length of the striker bead separation noted during the visual inspection (performed half way through the 2 times life program) had increased from a total of 11.0 in. to 15.5 in. as summarized in Figure 1. In addition, complete separation of the striker bead on the lower seal had occurred for 3/4 in. Figure 2 is a photo of the separated striker bead. In discussions of this situation between ARD, B.F. Goodrich (supplier of the seal) and AI, it was agreed that AI should remove the damaged silicone seals, trim the striker beads of one new silicone and one new EPDM seal to reduce the tendency of the bead to flop, and install both in the test fixture for a second life time test.

This trimming procedure was first done on one of the partially damaged silicone seals used in the first 2 x life test. The seal was trimmed in place in the test fixture with good results. Figure 3 shows the stabilizing effect of

- (1) ALL SEPARATIONS OCCURRED ON TOP SIDE OF STRIKER/BODY INTERFACE
- (2) NO SEPARATIONS PASSED COMPLETELY THROUGH THE UNDERSIDE OF THE STRIKER BEAD FOLLOWING FIRST LIFE CYCLE; SEPARATION FOLLOWING SECOND LIFE CYCLE IS NOTED
- (3) MEASURED AT SEAL INFLATION PRESSURE OF 5.0 psig
- (4) DIMENSIONS ON OUTER PERIPHERY TAKEN FOLLOWING FIRST LIFE CYCLE (819 ft)
- (5) DIMENSIONS ON INNER PERIPHERY TAKEN FOLLOWING SECOND LIFE CYCLE (1400 ft)

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Figure 1. Instrument Tree Inflatable Seals Visual Inspection

Figure 2. Lower Seal Striker Bead Separation
After 2 x Life Cycle Test

Figure 3a. Silicone Seal Before 2 x Life Test,
5/32 in. Bead Height, 5 psig Inflation Pressure

Figure 3b. Same Seal After 2 x Life Test, Trim-
med to 1/16 Bead Height, 15 psig Inflation Pressure

Figure 3. Effect of Trimming Seal Striker Bead

trimming the bead. Both pictures are of the same section of the upper silicone seal and were taken through the plastic cylinder. Figure 3a was taken prior to the 2 x life tests at an inflation pressure of 5 psig. Figure 3b was taken at a pressure of 15 psig after the seal had been trimmed in place to a nominal bead height of 1/16 in.

Using the information gained in trimming the partially damaged seal, the two new seals were trimmed using the guide and technique shown in Figure 4. The two seals were trimmed from a bead height of 5/32 to 3/32 in. and installed in the test fixture. Observation of the striker bead against a plastic cylinder used to simulate the mast, indicated no tendency of the bead to flop under the most extreme operating conditions. These conditions had previously resulted in a bead flop of 75% of seal circumference on the upper seal and 50% on the lower seal.

The test fixture was reassembled and a two times life test was conducted on the trimmed seals at 150°F. Following completion of the first life sequence, approximately 20 days at 150°F with 730 feet of travel, the fixture was disassembled for visual inspection of the seals. No evidence of seal degradation was detected on the EPDM seal. A small depression was noted on the silicone seal adjacent to the seal bead prior to testing and this depression had elongated slightly. Figure 5 shows the striker bead and illustrates the smoothing effect of trimming the bead and absence of any bead wear.

Following completion of the second life test, the seals were removed from the test fixture and given a detailed examination. The EPDM seal appeared in excellent condition, while the silicone seal had three small irregularities. Figure 6 shows both seals prior to removal from the test fixture. The silicone seal irregularities are shown in Figure 7 a, b, and c. The small depression shown in 7a approximately doubled in length as a consequence of the 2 times life test. The depression does not extend into the fabric body of the seal. Figure 7b shows a small area of thinned fabric overlay probably due to formation of a bubble during layup of the seal. The third irregularity, 7c, lies within the straight segment of the seal mold and does not extend past the mold flash lines. The crazed area is along one side of the seal body.

Figure 4. Striker Bead Trimmer
Guide and Setup

a. SILICONE SEAL STRIKER BEAD OVER SEAL BODY LAP JOINT

b. TOP VIEW OF FIGURE 6 A

Figure 5. Trimmed Silicone Seal After 170 Cycles
(One Life Cycle in Silicone/EPDM Follow on Test)

Figure 6. Trimmed Seals After Second Life Test

Figure 7a. Trimmed Silicone Seal — Irregularities
After 2nd Life Test

Figure 7b. Trimmed Silicone Seal — Irregularities
After 2nd Life Test

Figure 7c. Trimmed Silicone Seal — Irregularities
After 2nd Life Test

Figure 8. Instrument Tree Inflatable Seal
Seal Drag for 2 X Life Test – Untrimmed Seals
Seal BFG Silicone 4C1425-2

Figure 9. Instrument Tree Inflatable Seal Seal Drag for 2 X Life Test - Trimmed Seals

Buffer cavity gas leakage was measured as a function of seal inflation pressure for each trimmed seal prior to the start of the life tests, after 89 operational cycles of the first life test, and at the end of the second life test. With one seal at 15.0 psig (to prevent leakage past it), inflation pressure of the second seal was slowly decreased until buffer gas leakage was detected and then further decreased to obtain leakage rates as a function of pressure. As shown in Table 3, once leakage was detected, in all cases only slight additional pressure reduction was required for gross leakage. Typical data from similar tests performed on the untrimmed silicone seals of the original two times life test are shown for comparison.

At the start of the first life cycle test the inflation pressure of the silicone seal was set at 6.0 psig and the EPDM seal at 10.0 psig. As a consequence of the noted changes in leakage, the inflation pressure of the EPDM seal was lowered to 7.0 psig for the second half of the first life cycle test. During the second life cycle test the inflation pressure of the silicone seal was varied between 6.0 and 8.0 psig and the EPDM between 8.0 and 10.0 psig. The higher inflation pressures were used based on a desire of WARD to perform structural checks on the seals.

Breakaway and dynamic drag for the 2 times life tests on the untrimmed silicone seals are shown in Figure 8 for comparison with the dynamic drag for the trimmed silicone and EPDM seals 2 times life test shown in Figure 9. Because the seal inflation pressure for the trimmed seals was varied during the test, the results of Figure 9 are normalized to 6.0 psig and 7.0 psig for the silicone and EPDM seals respectively. Breakaway drag was not measured for the trimmed seals. While drag for both the trimmed and untrimmed seals is initially similar, the trimmed seals exhibit a much greater rise in drag throughout an individual test sequence. Trimming of the seals removed the grooves in the bead and greatly increases the probability of wiping lubricant off the seal bead. It is likely that the greater loss of lubricant results in the higher drag. Static dwells between the test sequences appeared to permit some self relubrication of the bead which resulted in a

TABLE 3
BUFFER CAVITY LEAKAGE AS A FUNCTION OF INFLATION PRESSURE

Buffer Cavity Leakage Past Single Seal (SCCM)	Seal Inflation Pressure ~psig						
	Trimmed [†] EPDM Seal			Trimmed [†] Silicone Seal			Untrimmed Silicone Seal - Original Test Series
	Pre- life Test	Mid-Point 1st Life Test (89th Cycle)	End 2nd Life Test (314th Cycle)	Pre- life Test	Mid-Point 1st Life Test (89th Cycle)	End 2nd Life Test (314th Cycle)	
0	10.0	7.3	6.1	5.1	5.1	4.6	8.8
10	9.5	6.8	5.9	4.9	5.0	4.4	8.6
100	8.6	6.1	5.6	4.9	5.0	4.3	7.8
500	7.9	5.8	5.2	4.8	4.8	4.3	6.5

[†] 15.0 psig on 2nd seal insured leakage past test seal only

lower drag at the start of each test sequence. Maximum drag was generally reached in about four hours of the nominal 16 hour test steps. These minimum and maximum drag values established the magnitude of drag "ranges" plotted on the Figures.

Seal drag for the individual trimmed silicone and EPDM seals was measured at the start of the first life cycle as shown in Figure 10. The lower drag was measured under normal operating conditions. The higher drag was measured after one-half rotational cycle of seal operation at 15 psig inflation pressure to force grease out of the striker bead-mast contact zone. This approach was used to simulate loss of grease on the striker bead due to long term operation at normal inflation pressures.

Figure 11 shows drag for the individual trimmed seals at the end of the second life test at ambient and 150°F. The 150°F drag is somewhat greater than initial drag (Figure 11) but the sum of the drags at 150°F is comparable to the combined drags for the pair of trimmed seals as shown in Figure 12, when both seals are inflated equally. (Under actual test conditions, inflation pressures were based on zero leakage conditions from Table 3.) For comparison, the combined drag of two untrimmed silicone seals is shown in Figure 12.

A 20 hour gas pressurization sequence simulating a sodium fill cycle was conducted and leakage past the EPDM seal was measured as a function of inflation pressure while a pressure differential of 0.9 psi was maintained across the seal. Figure 13 shows the test sequence and results. From an initial inflation pressure of 15.0 psig, the pressure was increased to achieve a zero leak rate and then reduced to the point where seal leakage occurred. It is postulated that the high pressure, used to achieve zero leakage, wedged the seal in the seal to mast gap and consequently prevented the seal from relaxing and leaking as the pressure was relieved.

Figure 10. Instrument Tree Seal Test Silicone/EPDM
Seal Drag vs Inflation Pressure, P_i

Figure 11. Instrument Tree Seal Test Trimmed EPDM and Silicone Seal Drag versus Inflation Pressure

Figure 12. Instrument Tree Seal Test Drag Comparison Between 2 Untrimmed Silicone Seals and 1 Each Trimmed Silicone and EPDM Seal

Figure 13. Seal Leakage During Sodium Fill

Reactor overpressure tests at various seal inflation pressures were conducted and are shown in Figure 14. The data show leak rates as a function of reactor cavity pressure minus the individual seal inflation pressure for a given inflation pressure. For a specific leakage, reactor cavity to inflation pressure differentials are at least 2.0 psig greater for the trimmed silicone seal than for the untrimmed seal previously tested.

Permeation rates through the EPDM seal of 1.3×10^{-5} cc/sec-in-Atm ΔP for Hydrogen and 5.7×10^{-6} cc/sec-in-Atm ΔP for Xenon were measured after 187 hours at 150°F. These data show good agreement with values of 1.9×10^{-5} (H_2) and 1.3×10^{-5} (X_e) calculated from seal geometry (assuming a homogenous material) and basic EPDM slab permeation rate tests. Permeation rates through the silicone seal were measured during the original test program on untrimmed seals and were not repeated.

Permeation tests with hydrogen, argon, krypton, and xenon were made on a silicone elastomer sample reinforced with nylon fabric. This sample was manufactured by the B.F. Goodrich Company and is of the same compound as the seals, 91-14A. The permeation data for this sample were used to calculate the permeability of the gas through the elastomer based on the assumption that the test specimen was homogeneous. Therefore the permeability data should be used for comparative purposes only. These pseudo-permeability data are given in Table 4 and also in the form, $\log \bar{P}$ vs $1000/T$ (°K) in Figure 15.

Tests to determine DC731 RTV seal adhesive compatibility with Exxon 5182 grease and with silicone seal segments lubricated with 5182 have been completed through 82 days of planned 320 day tests at 150°F. Figure 16 summarizes weight gain of the test specimens normalized to account for weight loss of the unlubricated seal and adhesive control specimens. For both tests, the greatest weight increase (absorption of lubricant) occurred during the initial test period. Through the 82 days, actual weight gain of the greased DC731 RTV specimens has been 0.9% compared to a loss of 0.7% for the control specimens. For the greased seal segments, the weight gain was 2.8% compared to a 0.25% loss for the ungreased controls.

Figure 14. Instrument Tree Seal Reactor Over Pressure Tests
(Seal Leakage vs Reactor Cavity and
Inflation Pressure Differentials)

Figure 15. Permeability of Nylon Fabric Reinforced Silicone, Compound 91-14A

ADHESIVE: DC 731 RTV
 SEAL: BFG SILICONE 4C1425-2
 GREASE: EXXON 5182
 OVEN TEMP: 150 F AIR

HD 3-19-75

Figure 16. Instrument Tree Seal Test Adhesive in Grease and Seal Segments in Grease

TABLE 4
PERMEABILITY* OF SILICONE WITH NYLON
REINFORCING FABRIC TO H₂ Ar, Kr AND Xe

Gas	Temperature °F	$\bar{P}\dagger$
H ₂	102	3.44×10^{-7}
	202	4.16×10^{-7}
	310	9.20×10^{-7}
Ar	102	2.54×10^{-8}
	202	7.53×10^{-8}
	310	9.20×10^{-8}
Kr	102	1.87×10^{-7}
	202	1.62×10^{-7}
	310	2.73×10^{-7}
Xe	102	2.12×10^{-7}
	202	3.07×10^{-7}
	310	4.31×10^{-7}

*Permeability in this case was calculated from permeation data through this sample making the assumption that the elastomer sample was homogeneous.

$\dagger$ Permeability given in $\frac{\text{cm}^3\text{-cm}}{\text{sec-cm}^2\text{-cm Hg } \Delta P}$

Peel and tensile stress specimens of the silicone seal bonded to chrome plated metal tabs with DC731 RTV were thermal soaked for 1 month at 150°F. Shear stress on two specimens averaged 37 psi. Peel strength on one specimen was 1.8 lb/inch. A second peel specimen failed in the bond area during preparation for mounting. Moisture was noted in the bond area which indicated a faulty preparation of the bond between the adhesive and tab. Additional specimens will undergo pull and peel tests after 2, 4, 8, and 16 month thermal soaks. Details of the test specimens and their arrangement in the Instron Tensile Machine are shown in Figures 17 and 18.

a. SHEAR SPECIMEN

b. PEEL SPECIMEN

Figure 17. Adhesive Test Specimens

Figure 18. Peel Test Instron Setup

C. TOP SHIELD DIP SEAL CONFIGURATION CHARACTERISTICS

Fabrication of the sodium dip seal-deposition test article and the bellows system that will provide prescribed cover gas pressure oscillations in the test article have been completed. Installation of the test article has been initiated.

Fabrication and installation of the 30 inch long, plastic model of the prototype dip seal was completed. Figure 19 shows the cross section of the dip seal in the 30 in. long plastic model. Performance tests have been made with the following blade configurations:

- 1) Plain blade (shown in Figure 19)
- 2) Notched blade (90° V notches, 1/2 in. high, cut in the plain blade every 4 in.)
- 3) Beveled blade (shown in Figure 19)

In each run in these tests, the dip seal was initially in the incipient bubbling point condition with the cover gas channel filled to the bottom of the weir. The dip seal was then subjected to gas flow at various rates and for various time periods. At the end of each run, the water level loss in the cover gas channel was measured. Figure 20 shows the test results for gas flow time periods of 6 seconds and 30 seconds. It is seen that the water level loss is generally least for the beveled blade. A beveled blade will be installed in the sodium dip seal deposition test article.

The natural frequency of water oscillating in the 30 in. long plastic model dip seal was measured to be 2.3 Hz for the condition of 5.1 in. water depth in the dip seal and a gas volume of $0.381 \text{ ft}^3/\text{ft}$ in the buffer leg. The calculated natural frequency of a dip seal having channels of equal width, the same length of water and buffer leg gas volume as above, is 2.27 Hz.

The degree of mixing of the water in the dip seal trough, when subjected to gas pressure oscillations, will be determined by measuring the time required for a salt solution to travel across the dip seal. The required instrumentation for this mixing test has been assembled.

Figure 19. Cross-Section of Dip Seal in 30 in. Long, Plastic Model

Figure 20. Gas Flow Rate vs Water Level Loss

The analytical effort on the transport of radioactive gases through the sodium dip seal has been completed. It is concluded from this study that a sodium dip seal at 400°F is an effective barrier to xenon, krypton, argon, and the radioisotopes of these gases. Some results obtained in this study are as follows:

- 1) Figure 21 shows the calculated permeability of xenon, krypton, argon, and helium in quiescent liquid sodium.
- 2) Calculations were made for the transport of xenon, krypton, and argon through a sodium dip seal at 400°F and two inflatable, nitrile seals at 150°F, all in series. The results are shown in Figures 22, 23, and 24. The nomenclature used in these figures is as follows:

D = diffusion coefficient of the gas in sodium, $\frac{\text{cm}^2}{\text{sec}}$

D_{eff} = effective diffusion coefficient of the gas in sodium when the sodium is being mixed, $\frac{\text{cm}^2}{\text{sec}}$. Hence, the magnitude of the ratio, D_{eff}/D , is a measure of the degree of mixing in the sodium.

L = total thickness of the sodium in the dip seal, inch

P_1 = partial pressure of the gas upstream of the sodium dip seal, atm

P_2 = partial pressure of the gas downstream of the sodium dip seal, atm

q = gas flow rate, $\frac{\text{scm}}{\text{sec-cm}}$

$q_{L=0}$ = gas flow rate for no sodium dip seal, $\frac{\text{scm}}{\text{sec-cm}}$

w = effective width of the sodium in the dip seal, inch

θ = transport time for flow of the gas through the sodium, hour.

Figure 21. Permeability of Some Gases in Quiescent Liquid Sodium

Figure 22. Transport of Xenon Through Sodium Dip Seal - Inflatable Seal System

Figure 23. Transport of Krypton Through Sodium Dip Seal - Inflatable Seal System

Figure 24. Transport of Argon Through Sodium Dip Seal - Inflatable Seal System

Figures 22, 23, and 24, show, for the respective gases, the flow rate reduction effected by the sodium dip seal, the pressure decrease across the sodium dip seal, and the transport time in the sodium.

- 3) The results may be applied to radioisotopes of the respective gases if corrected for the radioactive decay that occurs during transit through the sodium dip seal. Specifically, P_2 and q_2 , the partial pressure and flow rate, respectively, of the radioactive gas downstream of the sodium dip seal are reduced by the amount of radioactive decay that occurs in the sodium dip seal. Thus,

$$\left(\frac{P_2}{P_1}\right)_{\text{isotope}} = \left(\frac{q_2}{q_{L=0}}\right)_{\text{isotope}} = \frac{P_2}{P_1} \exp \left[-\frac{0.69310}{\theta_{1/2}} \right]$$

where $\theta_{1/2}$ is the half life of the radioisotope.

The report, TR-707-230-002, "Phase I Dip Seal Tests - Screening and Model," on the test results obtained in the dip seal configuration screening device and the 3 ft long plastic dip seal model was issued.

Revision of the report, TI-707-230-009, "Dissolution of Na_2O Deposits by Flushing with Sodium," was completed.

D. ADVANCED SEALS AND GEOMETRY

EPDM and nitrile O-rings with a thin indium film deposited on approximately one-half of the seals' surface (circumferential coating on the outermost diameter) were received. Both seals are currently being tested for reductions in gas permeation rates as compared to uncoated O-rings. The tests are being run at 150°F and 20% compression in static permeation test fixtures for applications as face seals. Indium was chosen for the initial tests samples because of cost and processing availability despite a slight tendency for the metal to form an alloy on exposure to sodium.

Data indicates that krypton permeation rates are less for the test specimens than corresponding rates previously determined for unplated O-rings. For example, the leak rate for an EPDM O-ring at similar test conditions was

previously determined to be 60×10^{-8} cc of Kr/sec/linear in. of seal/ ΔP in. atm. at standard conditions. Recent values for the same O-ring with the indium film plating are in the 9×10^{-8} range. However, the rates determined for hydrogen leakage for the plated seals are up to 100 times greater than corresponding unplated seals. Inspection of the indium surface under a microscope indicates that during plating, agglomerations of molten metal made the surface irregular. The mating of the seal surface with the gland surface could thus be adversely affected accounting for the high hydrogen leak rates.

The magnetic fluid rotary seal test fixture was received. The permeation test is being run at 175°F with a 3 in. Hg pressure gradient and a 2500 ppm concentration gradient of H_2 , Kr, and Xe across the interface. Because the test is "proof of principle" in nature, the device was fabricated with a readily available fluid of low vapor pressure to extend the seal lifetime. Pending successful results of initial evaluations, additional tests of fluids with better physical and chemical resistance are required. The fixture is currently charged with a diester carrier fluid for the ferrite particles. The chromatograph response curves indicate no krypton leakage across the interface. The xenon leakage is somewhat less than that for corresponding O-ring rotary seal tests but is in the range of low permeability elastomers. Hydrogen leakage is, however, predominant and exceeds rates for low permeable elastomers. No explanation exists for this phenomena, but the responses of the low magnetic permeable gases will be examined in terms of variations to paramagnetic or diamagnetic characteristics at the test conditions.

The metal E-seals have been received and assembled for static permeation tests with either indium, EPDM, or nitrile gaskets applied to the seal interfaces. Tests are being conducted at 300, 300, and 200°F respectively.

Shipment of the V-shaped spring seals has slipped due to vendor spring die fabrication problems.

E. ADVANCED MATERIALS

1. Inorganic Backbone Elastomers

As previously reported, AI-AEC-13143, the gas permeation resistance of the original slab specimen of polyphosphazene was superior to EPDM and silicone but inferior to nitrile and urethane at 200°F. This data is included in Table 5 which shows permeation of a variety of standard and experimental compounds currently under evaluation.

Compression set tests on 1.0 inch diameter stacks of the polyphosphazene are being conducted at 250°F based on methods specified in ASTM specification D 395, "Constant Deflection Slab Compression Set." Each stack consists of 4 slabs of .080 nominal thickness material. Figure 25 includes the compression set for the polyphosphazene (identified as polyphosphazene "H") and other materials under test. As this sample was not compounded to optimize compression set resistance, a program was initiated to improve compression set resistance through filler loading and curing mechanism variations on the phenoxy, ethylphenoxy copolymer. The effort has revealed that silica, silane, and aluminum silicate loadings do not improve the set resistance of the elastomer. Varying loads of carbon black fillers do, however, significantly modify the compression set resistance. The performance for the optimum material processed to date is compared with the specified requirements as noted in Table 6. Samples of two preferred reinforcing carbon black loadings in the elastomer are being fabricated for shipment to AI for long term simulated set and permeation tests. If the permeation for such materials is high, the option for a terpolymer investigation program is being kept open.

A second form of polyphosphazene (containing phenoxy and ethyl phenoxy pendent groups on the phosphorus-nitrogen backbone) was obtained. This material was compounded by a second vendor and molded by a processing laboratory. Initial compression set data is shown on Figure 25, identified as polyphosphazene "F," and is substantially superior to the initial material. Permeation tests are currently being conducted, but results are not yet available.

TABLE 5

$\bar{P}$ PERMEATION COMPARISON OF SLABS @ 200°F							
TEST GAS	TEFLON DUPONT * @ 300°F	NITRILE PARKER N741 **	URETHANE PARKER P64B **	EPDM PARKER E692 **	PHOSPHA- ZENE HORIZON	SILICONE PARKER SP684 **	G.E. SILICONE 17 MOLE. % PHI NYL
H ₂	17.8	14.1	14.6	47	34.2	380	280
Ar	1.63	1.8	2.4	20	2.3	82	31.8
Kr	3.13	3.6	18.0	22	10.3	210	200
Xe	.71	4.9	11.2	37	8.7	260	710

$\bar{P}$ = PERMEABILITY IN $\text{CM}^3\text{-CM/SEC/CM}^2\text{/CM Hg } \Delta P \times 10^{-9}$

* TEMP. INCREASED BECAUSE OF LOW $\bar{P}$ @ 200°F

** PREVIOUSLY DETERMINED VALUES

Figure 25. SLAB COMPRESSION SET TESTS @ 250°F

TABLE 6
OPTIMIZED POLYPHOSPHAZENE PROPERTIES

Property	AI Requested	Vendor Obtained
Tensile Strength	1500 - 2000 psi	~2000
Elongation @ Break	>100%	150%
Hardness (Shore A)	60 - 80	63 - 65
Compression Set (70 hrs at 302°F, 25% Load)	<25%	15%

2. Thermoplastics

A vendor has tentatively agreed that it can fabricate and supply a thermoplastic bound-flat spring seal for dynamic permeation evaluations. An order was placed for composite seals of a filled polyphenylene sulfide thermoplastic. Rotary and reciprocating permeation tests will determine the effectiveness of the relatively rigid interface surface with the seal gland. Polyphenylene sulfide resin was selected because of processing ease, cost, and availability. The tests are, therefore, proof of principle in nature.

A set of gasket seals of polyphenylquinoxaline (PPQ) bound glass laminates was received. Since PPQ belongs to the aromatic heterocyclic polymer group, it has excellent heat and radiation resistance, but lacks flexibility. The glass laminate-PPQ composite was developed to compensate for this lack of flexibility. The samples are gasket face type seals and a special permeation test fixture is required.

3. R&D Elastomers

A slab sample of polycarborane-siloxane polymer was received. Simulated compression set tests are currently underway on the material at 250°F and 25% compression. Results, shown in Figure 25, indicate a substantial improvement over common elastomers in the test. Permeation tests on the slab could not be run because the organic peroxide used to initiate polymerization generated volatiles which made the surface porous. A pressure gradient

across the membrane could not be maintained. Another sample has been received and permeation tests will be rerun.

4. Common Elastomer Modifications

Two slab samples of silicone elastomers were obtained with increased phenyl group concentrations of 10 and 17 mole %. The higher phenyl group polymer has undergone permeation tests and shows no improvement over the methyl silicone slabs. Both data are shown in Table 5. Slab stacks of both materials are currently being tested for compression set resistance at 250°F and 25% compression with the 17 mole % phenyl group silicone also showing lower set resistance, Figure 25, than the original material.

F. SODIUM VAPOR TESTS

A test plan was prepared for testing of elastomer's resistance to degradation during exposure to sodium vapors and aerosols. The test fixture has been fabricated and installed on the sodium test loop. Based on program redirection to conduct the trimmed seal Instrument Tree Seal Tests and other priority items, the sodium vapor-elastomer test has been delayed until FY 1975.

The following is a listing of the materials which will be tested and descriptions of possible interactions with sodium for each.

- 1) Urethanes are susceptible to sodium induced degradation of both the backbone chain (causing fragmentation) and the cross link mechanism. Polyurethanes have active hydrogens and double bonds to oxygen along the chain making it extremely reactive to moderate sodium exposure.
- 2) Silicones are almost totally saturated elastomers but have polar monomer linkages with oxygen in the backbone making depolymerization or partial fragmentation possible on exposure to sodium. Side groups and cross-linkage interaction with sodium should be low.

- 3) Nitriles (or Buna N) are highly unsaturated polymers and are thus susceptible to substitution attack via active hydrogen atoms that are alpha to carbon-carbon double bonds. In addition, processing aids such as polar plasticizers are almost universally used in nitrile compounding. These are generally esters that are highly reactive to alkali metals. Nitriles, then, could also degrade on exposure to sodium. (NOTE: only 3-5% of the unsaturated butadiene bonds are used as cross-linkage sites during vulcanization.)
- 4) EPDM terpolymers do have some degree of cyclic unsaturation, but the carbon-carbon double bonds are pendent to the backbone, because the butadiene undergoes 1, 2 polymerization rather than 1, 4. Therefore, even if sodium attach occurs, the polymer should not degrade extensively. Also, EPDM contains extremely low quantities of processing aids.

The above descriptions are based on the chemical structures of the polymer units. It is concluded that EPDM will be relatively inert to sodium. Urethanes, nitriles, and silicones could degrade from severe to moderate levels, respectively.

IV. IMPACT ON LMFBR PROGRAMS

The initial phases of this program have guided selection of materials and seals for the FFTF and CRBRP from information on premeability, compression set and gland design published in progress and technical reports. FFTF designs of the IVHM inflatable and primary seals were verified during testing. Current phases have verified the Instrument Tree inflatable seal design and are evaluating the use of inflatable seals for CRBRP. Continued testing of materials and seals will give confidence to the data already collected, define long-term performance and develop materials and seals for long term use. Uncertainty as to the effect of sodium vapors and aerosols on elastomers will be resolved by tests designed to measure these effects. Advanced seal geometries and materials will overcome the commercial seal deficiencies of excessive compression set (short maintenance cycle) and permeation. Specifications and a design guide for use in the LMFBR industry will become available.

V. NEXT REPORT PERIOD ACTIVITIES

The CRBRP Rotating Plug Inflatable seal will be tested for breakaway drag following the 140 day static dwell test. Following short term leakage and drag tests the test will be disassembled for inspection of the seal and modification of the fixture for seal off-set tests. A final report will be written. The FFTF Instrument Tree Inflatable Seal Test Report will also be completed.

The Sodium Dip Seal mixing tests (model tests) will be completed. The Dip Seal test fixture will be assembled and tests initiated.

Static leakage tests of metal E seals will continue and static and dynamic tests of other advanced seal concepts, metal assisted configurations, will be initiated as the seals are received from suppliers. Testing of advanced elastomer compounds, polyphosphazene and polyphenylquinoxaline, will be initiated.

VI. SCHEDULE

The currently scheduled activities for the seal program in FY 1975 are shown on Figure 26.

Figure 26: Schedule and Milestones