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Program: Cover Gas Seals

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

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I. PROJECT OBJECTIVES

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 LMFBR applications to support the national objective to reduce all atmospheric contaminations to low levels, and (4) publish and periodically update a Design Guide for reactor seals.

Activities in support of these objectives include demonstrating the performance of the FFTF Instrument Tree Inflatable Seal, providing CRBRP design information based on tests of the IVHM Inflatable Seal to CRBRP conditions, performing the required analyses and testing to select a CRBRP dip seal configuration and determine its performance characteristics, and delineating the effects of sodium and radiation environments on the efficiencies of various seal materials.

The primary direction of the general seal effort is to develop advanced seal geometries and materials to overcome deficiencies of commercially available seals — permeation rate, compression set, and high temperature operation — which restrict their use in LMFBR applications.

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II. MAJOR ACCOMPLISHMENTS DURING REPORT PERIOD

A. GENERAL SEAL DEVELOPMENT

Permeation of oxygen through Parker Seal Co. N741-75 nitrile and through Minnesota Rubber FX522 urethane elastomers was measured as a function of temperature on slabs previously measured for permeation of argon, krypton, xenon, and hydrogen. Figures 1 and 2 present the oxygen permeation rate data for the respective elastomers in addition to the argon, hydrogen, krypton, and xenon data previously reported.

The 32-week compression set tests at 115°F on O-ring seal forms of the same nitrile and urethane compounds listed above were completed at 20 and 30% initial compression. Figures 3 and 4 represent the data generated from these tests.

B. CRBRP ROTATING PLUG INFLATABLE SEAL TEST

Following completion of horizontal offset tests, at ± 0.05 and ± 0.095 in. offset, during FY 1975 (see AI-ERDA 13154), the seal blade assembly was relocated to the nominal ± 0.02 offset position. The seals were cleaned, inspected, relubricated with DC-55M grease, and the test fixture was reassembled. Static and dynamic leakage, seal drag versus rotating speed, and seal drag versus inflation pressure tests were conducted at ambient and 150°F. Figure 5 shows running drag at 150°F as a function of inflation pressure. The data is consistent with that taken during the offset tests which are included for comparison. Figure 6 presents the static seal leakage characteristics as a function of buffer cavity and seal inflation pressures for ambient and 150°F. The two curves presented are for leakage at 10 sccm. Zero leakage data points are included for the 150°F curve. For reference, a similar curve is shown for data taken at the conclusion of the first life test (87,567 ft of travel). The curve appears to be shifted such that a slight additional inflation is required to prevent leakage. If this is an actual shift, it is probably due to a very slight softening of the seal because of absorption of grease. Additional tests at the conclusion of the 1500-hr interim dwell will verify the shift of the curve or determine whether the apparent shift was due to data scatter.

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Figure 1. Log $\bar{P}$ vs $1/T$ ($^{\circ}K$) for Hydrogen, Argon, Krypton, Xenon, and Oxygen for Parker Seal Buna N (Compound N741)

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Figure 2. Log $\bar{P}$ vs $1/T$ (°K) for Hydrogen, Argon, Krypton, Xenon, and Oxygen for Minnesota Rubber Urethane (Compound FX522)

Figure 3. Compression Set of Parker Seal N741-75 Nitrile O-Ring Seals at 115°F; 20 and 30% Compression

Figure 4. Compression Set of Minnesota Rubber FX522 Urethane O-Ring Seals at 115°F; 20 and 30% Compression

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Figure 5. Running Drag vs Inflation Pressure

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Figure 6. Static Cross Seal Leakage as a Function of Pressure and Temperature

One interim dwell test of 1600 hr at 150°F has been completed on the rotating plug inflatable seal. Breakaway drag was measured to be 87.8 lb/ft for the two seals. Within one-quarter revolution, drag dropped to 37.6 lb/ft and stabilized at about 25 lb/ft. The breakaway drag was less than predicted, based on results of previous breakaway drag tests run under identical conditions but after 400 and 3200-hr dwells. The drag versus inflation pressure, drag versus speed, and leakage versus inflation pressure parametric tests have been completed at 150°F. This data is being analyzed and will be reported with results from similar tests currently being run at ambient temperature.

Two IVHM DC-140RTV adhesive pull-test specimens, thermally soaked at 150°F for 14 months, have been tested for adhesive bond shear strength. Bond failures occurred at 17 and 20 psi for the two specimens. This is consistent with previous specimens which separated at 34 psi (1 month soak), 15 and 28 psi (5 month), and 13 and 24 psi (9 month). Two final specimens will be tested after 18 months thermal soak. Nominal and worst-case shear loads for actual seal operation are 2.3 and 5.5 psi. The DC-140 RTV adhesive had previously been replaced with Chemlok 304 epoxy adhesive since the initiation of the 140 RTV shear strength tests, but tests will be completed as scheduled.

C. TOP SHIELD DIP SEAL CONFIGURATION CHARACTERISTICS

Checkout of the dip seal test article was completed, sodium was circulated and cleaned up in the low temperature loop, and the test article was charged with sodium.

Pressure recovery margin tests were run for an equilibrium gas pressure of 15.05 psia over 400°F sodium. Figure 7 shows test points for sodium equilibrium levels of 1.75, 2.75, and 3.75 in. plotted over a curve of the calculated performance. Pressure recovery margin is defined as the increase in cover gas pressure, above the nominal cover gas pressure, that can be sustained without the sodium level in the cover gas leg of the dip seal dropping below the dip seal blade. An additional pressure increase of about 0.2 psi generally resulted in bubbling gas across the dip seal. Figure 8 illustrates these conditions.

Figure 7. Sodium Dip Seal Pressure Recovery Margin

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NOTE: RECOVERY MARGIN PRESSURE (FIGURE 7) = $P_{CG \text{ ②}} - P_{CG \text{ ①}}$
COVER GAS PRESSURE AT LEVEL ② - COVER GAS PRESSURE AT LEVEL ①

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Figure 8. Sodium Dip Seal Configuration - Pressure Recovery Margin Tests

The scram pressure transient tests, at 400°F nominal sodium temperature, were completed on the sodium dip seal system (Figure 9). Tests were conducted as follows:

- 1) The sodium dip seal trough was filled to capacity (6-1/4 in.) and the argon gas pressure set at 15.65 psia in both cover gas and dip seal buffer gas legs. (The pressures were set 0.60 psi higher than design pressure to obtain the desired scram pressure transient by venting the cover gas to the atmosphere.)
- 2) A scram pressure transient, i. e., a reduction in cover gas pressure of 1.3 psi (from 15.65 to 14.35 psia) was imposed at an initial rate of -0.2 psi/sec. At the completion of the transient, the sodium level and gas pressure in each leg were recorded.
- 3) A scram recovery transient, an increase in cover gas pressure from 14.35 to 15.85 psig at an initial rate of +0.2 psi/sec, was then conducted and the sodium levels and gas pressures were again recorded.
- 4) Gas pressure was restored to 15.65 psia in both legs and sodium levels recorded.
- 5) Steps 2, 3, and 4 were repeated five additional times without refilling the dip seal with sodium.

These tests were conducted with the dip seal test article level and tilted to 0.35 and 0.70°. The tilts correspond to raising one side of the 41-in.-diameter dip seal blade 1/4 and 1/2 in., respectively.

The results of the scram pressure transient tests were:

- 1) No sodium was ejected from the dip seal trough after the sodium dropped to the indicated level, which occurred by the end of the fourth scram pressure transient.

<u>Tilt (degrees)</u>	<u>Sodium Level (in.)</u>
0	4-5/8
0.35	5
0.70	4-3/4

Figure 9. Dip Seal System

- 2) There was some gas flow across the dip seal blade from the dip seal buffer gas space to the cover gas space during each scram pressure transient.
- 3) There was no indication of gas flow across the dip seal blade during scram recovery pressure transients.
- 4) The minimum sodium level in the dip seal cover gas leg at the end of the scram recovery pressure transient was:

<u>Tilt (degrees)</u>	<u>Sodium level (in.)</u>
0	3-1/2
0.35	3-7/8
0.70	3-3/4

These results indicate that: (1) a sufficient amount of sodium will always be retained in the dip seal to prevent flow of the cover gas into the dip seal buffer gas space, and (2) tilting the test apparatus to raise one side of the 41-in. diameter blade 1/2 in. above the other side has no significant effect on the transient performance of the dip seal.

Mixing tests with water in the plastic dip seal model were completed. The plastic dip seal model (Figure 10) is a 30 in. length of a full size dip seal. The objective of the mixing test was to determine the effect of cover gas pressure oscillation of ± 0.2 psi at a frequency of 6 cycles/min on the transport rate of a dissolved gas across the sodium dip seal. The tests were conducted using a dip seal in which the fluid was water instead of sodium; this permitted a relatively simple and inexpensive test. The test method was to place a thin layer of ammonium hydroxide on the water surface in the cover gas leg of the dip seal (see Figure 10), and measure the time required for the dissolved ammonia to reach the buffer gas leg. The latter was detected by a thermal conductivity probe placed in the water in the buffer gas leg. The probe was provided with three pairs of electrodes: one pair at the bottom of the probe, a center pair 1/2 in. up from the bottom of the probe, and a top pair 1 in. up from the bottom of the probe. (Only two of the three pairs were hooked up to the recorder at any one time.) The bottom of the probe was $\sim 1/16$ in. above the bottom of the dip seal trough.

Figure 10. Mixing Test in Plastic Dip Seal Model

In all runs, the dip seal trough was initially filled with water to a level of 1-1/2 in. in both legs. Runs were made with both static and oscillating water conditions. During the static water run, the water level in the buffer gas leg was depressed to the 1-1/4 in. level by gas pressurization. During the oscillating water run, the water level in the buffer gas leg was cycled between the 1-in. and 1-1/2-in. levels at a frequency of 6 cycles/min, in the manner shown schematically in Figure 11. This amplitude ($\pm 1/4$ in.) is the calculated amplitude of the sodium level in the buffer gas leg when the cover gas pressure oscillates ± 0.2 psi at a frequency of 6 cycles/min.

The results of the mixing tests were as follows:

Run No.	Date	Condition	Electrodes	Transport Time (hr)
1	August 13, 1975	Static	Top Center	8 8-3/4
2	August 19, 1975	Oscillating	Center Bottom	8 7-1/2
3	August 25, 1975	Oscillating	Center Bottom	10-3/4 10-3/4

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Figure 11. Water Level Oscillation in Buffer Gas Leg

An anomaly exists in the results of Run 1 in that the transport time to the top electrodes should be longer than to the center electrodes (see Figure 10).

Comparison of the results of Runs 1 and 2 indicate that oscillating the water had negligible effect on the transport time. The conditions for Run 3 were the same as for Run 2, but it is seen that the transport time for Run 3 is about 25% longer than in Run 2. A possible explanation for this difference is that in Run 3, the run was not initiated until 3 days after the dip seal trough was charged with water, whereas in Run 2, and also in Run 1, the runs were initiated only 1 day after the dip seal trough was charged with water. In Runs 1 and 2, the water may not have reached thermal equilibrium, so that there may have been some convection. To check this explanation, a static run in which the run will be initiated 3 days after the trough is charged with water is planned.

Wall and gas temperature distribution data for the upper annulus have been obtained for analysis of argon convection in the upper annulus. This analysis is in progress.

Preparations for the radioactive gas attenuation tests have been initiated.

D. ADVANCED SEALS AND GEOMETRY

The static and dynamic leakage tests in Table 1 were initiated at the listed representative operation conditions to evaluate improvements in the sealing efficiencies of the selected geometries over common O-ring configurations.

Data from these tests are being reduced periodically to generate leakage versus time curves. Results will be issued after ~2000-hr test duration for each of the listed test operations.

The indium-plated nitrile and EPDM O-ring static leakage test is being run to verify the effects of seal gland surface finish and repeatability of the previous leakage data which showed a high leakage rate the first 1000 hr, and a greatly reduced rate the final 1000 hr. The initial test was run with fixture seal glands having 32 rm finishes for the nitrile O-rings and 8 rm finishes for the EPDM O-rings. In the current tests, this has been reversed.

The static leakage test on metal E-seals with nitrile sheet gaskets and no adhesive is being run to compare generated data with a previous leakage test in which an adhesive was used between the gasket and the seal mating surface.

The Ferrofluidic seal test fixture was returned to the vendor for disassembly, inspection, and recharge with a higher performance carrier fluid. No changes will be made in the pole block assembly or cavity design.

Shipment of spring assisted rigid thermosetting material seals was received. These seals are of EKKCEL C1000® (polyester) in reciprocating test sizes and VESPEL SP-1® (polyimide) in rotary test sizes. Drawings are being prepared for modification of existing test fixtures to allow for the installation of these articles in the leakage test rigs.

TABLE 1
ADVANCED SEALS AND GEOMETRY TESTS

Seal Description	Seal Material	Test Type	Test Motion	Initial Compression (%)	Test Temperature (°F)	Test Objective
Indium plated O-rings	EPDM	Static	—	20	150	Verify previous data and determine surface finish effects on leakage
	Nitrile	Static	—	20	150	
Metal E-seals	SS-Seal and Nitrile Gasket	Static	—	14.7, 20.6	200	Determine leakage with no gasket adhesive
	SS-Seal and Indium Plated Surface	Static	—	10.7, 17.9	300	Determine general sealing efficiency
U-shaped, wound stainless steel ribbon with elastomeric jackets	EPDM	Dynamic	Reciprocating	10	150	Determine efficiency of general seal design
			Rotary	10	150	
	Nitrile	Dynamic	Reciprocating	10	150	
			Rotary	10	150	
Stainless Steel canted coil, helical spring with elastomeric jackets	EPDM	Dynamic	Reciprocating	10	150	Determine efficiency of general seal design
			Rotary	10	150	
	Nitrile	Dynamic	Reciprocating	10	150	
			Rotary	10	150	

E. ADVANCED MATERIALS

Slab specimens of Union Carbide's improved carborane siloxane and Horizon's polyphosphazene elastomers were sent to the chemistry laboratory for permeation measurements. Permeation measurements were initiated on the polyphosphazene but the sample split near the vacuum flange clamping edge at 100°F with a pressure gradient of 200 torr across the membrane. The carborane siloxane sample later split under similar testing conditions. Previously run nitrile slabs were used to verify that the test fixture was not contributing to these failures. The various suppliers were notified of the material failures and additional slabs were ordered. A 0.125-in.-thick specimen of the carborane siloxane elastomer was received and has been delivered for a repeat of the permeability tests.

Test slabs of Dupont's Viton® and experimental ECD-006 perfluorinated elastomers were tested at 200°F for permeability. Results are listed in Table 2.

TABLE 2
PERMEABILITIES OF CHARACTERISTIC
HALOGENATED ELASTOMERS

Gas	Elastomer	Temperature (°F)	$\bar{P}^*$
H ₂	Viton	201	21.1
	ECD006	200	113
Ar	Viton	201	4.13
	ECD006	200	6.10
Kr	Viton	201	3.29
	ECD006	200	9.92
Xe	Viton	201	1.35
	ECD006	200	19.9

* $\bar{P}$ (std cc-cm/sec-cm²-cm Hg $\Delta P \times 10^{-9}$)

As an approximate comparison with previous slab tests at 200°F, the Viton and ECD006 permeabilities are comparable to urethane and EPDM permeabilities, respectively. Properties of these materials are being evaluated for preliminary characterization of high performance halogenated elastomers.

Simulated compression set tests were begun on slab stacks of the two forms of Horizon's polyphosphazene, the latest polycarborane siloxane formulation, Viton, ECD006, and Parker Seal silicone at ~25% initial compression and 250°F. Three additional silicone stacks are being tested at 15, 35, and 45% initial compression to determine the effect of compression on permanent set for this material. Preliminary results through 4 weeks show the compression set to be about 10% for the phosphazene and 20% for the halogenated elastomer.

The preliminary program to evaluate the feasibility of reducing elastomers permeabilities by depositing thin, highly crosslinked polymeric films on the sealing surface of elastomers has been initiated. The deposition technique is a radio frequency plasma polymerization process developed by the Rockwell Science Center, whereby ~1- μ -thick films of selected polymers are formed from monomeric gases in a contained system. The initial tests are being run on two slabs of silicone, EPDM, nitrile, urethane, and polyphosphazene which were coated with acrylonitrile or styrene. All slab samples have been coated and delivered for permeability testing. The program objective is to reduce the solubility of inert gases via this deposition so that gas permeation through the seals could be curtailed. Data from the first specimen (silicone with an acrylonitrile film) has been reduced and is represented in Figure 12. No significant improvement was noted in this sample, but it was reported that silicone had been the least receptive material in the test matrix. Permeability testing is currently underway on the remaining test articles.

The sodium vapor test fixture was loaded, checked out, and filled with ~4 in. of sodium. The expected temperature gradient between the two regions that contain the silicone-EPDM seals and the nitrile-urethane seals could not be attained. Coiling coils were added to the upper region and insulation re-

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Figure 12. Log $\bar{P}$ vs 1000/T(°K) for Hydrogen, Argon, Krypton, and Xenon for Parker Silicone (Compound S684) Coated With a Film of Acrylonitrile – Plasma Polymerization Study

moved from the argon recirculation lines in attempts to increase the gradient and to allow an increase in the sodium pool temperature. A gas line for oxygen content analysis was added and is being monitored along with the region temperatures.

F. GAMMA IRRADIATION

The test fixtures and cart assembly for eight fixtures have been designed and fabrication materials have been ordered. These fixtures were designed so that hydrogen evolution could be monitored on compressed, "as-installed" O-ring test specimens during irradiation. Such tests will also generate leakage and compression set data at dosages up to 10^7 , 5×10^7 , and 10^8 rad in the simulated installed seal state.

A fixture test location was selected in the gamma cell where eight cobalt pencils can be oriented around the test articles to provide the required doses.

The test plan is being written. The gas chromatograph system has been debugged and the residual gas analyzer is being checked out.

III. 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 permeability, 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. The effects of radiation on the sealing efficiency of elastomers will be delineated. 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 and will be updated as new information is generated.

IV. NEXT REPORT PERIOD ACTIVITIES

The CRBRP rotating plug inflatable seal will be subjected to a 150°F dwell, duplicating that completed this quarter, but breakaway drag will be measured at a "slow start" speed, duplicating CRBRP low speed capabilities.

Testing of the sodium dip seal test article will be completed.

Static and dynamic reciprocating tests on advanced seal geometry tests currently in progress will be completed.

Gamma irradiation tests on nitrile, urethane, EPDM and silicone elastomers will be initiated.

The currently scheduled activities for the seal program in FY 1976 are shown in Figure 13.

Figure 13. Schedule and Milestones