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ELASTOMER COMPATIBILITY
WITH LIQUID SODIUM
AT 450 °F

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UNITED STATES ATOMIC ENERGY COMMISSION

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LIQUID SODIUM AT 450°F

E. F. Antal

JUNE 1971

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INTRODUCTION

The use of elastomer seals in the FFTF Closed Loop nozzle to isolate the Closed Loop Sodium/Sodium Vapor/Inert Gas from ambient air is of prime importance to reduce handling time and complexity of the closed loop handling system. A literature search showed no direct information on the use of elastomer seals in sodium systems. Elastomer seals are being used in systems where there is little or no chance of sodium or sodium vapor coming into direct physical contact with the elastomers. These systems generally use a purge gas to keep sodium vapors away from the elastomer seal.

A minimal test program was undertaken to investigate physical changes in elastomer O-rings and to establish their specific compatibility with liquid sodium at 450°F.

SUMMARY AND CONCLUSIONS

Three materials were suggested for compatibility testing with liquid sodium: (1) silicone rubber; (2) ethylene propylene; and (3) polyimide.

The ethylene propylene's recommended operational temperature of 350°F limited it to a brief compatibility test. Data on polyimides and a sample of a Dupont polyimide, "Vespel", showed that this material was a rigid plastic and could not function as an elastomer O-ring. Silicone O-rings with a ferric oxide heat stability additive are affected by liquid sodium at 450°F. Sodium vapor also affected the O-rings, but to a lesser degree. The strength loss noted in these tests is primarily caused by heat aging.

Silicone O-rings would have a limited useful life as an elastomer seal in contact with sodium/sodium vapor at temperatures of 450°F. The silicone apparently goes into solution in the liquid sodium at these temperatures. The effects of this contamination should be determined. Further tests should be undertaken with pressurized sodium systems at temperatures to determine the useful life of silicone O-rings.

Table 1 is a summary of the tests showing the parameters and data obtained.

TABLE 1
SUMMARY OF TEST RESULTS

Test No.	Material	Exposure (hr)	Exposure Temp (°F)	Exposure Material	Tensile Strength Loss (%)	Comment
1	Silicone Rubber Parker S604-7	70	450°	Sodium/ Sodium Vapor	30-40	Surface etched-10 mil dia loss
2	Silicone Rubber Parker S604-7	140	450°	Sodium/ Sodium Vapor	30-40	Surface etched-10 mil dia loss
3	Silicone Rubber Parker S604-7	140	450°	Sodium Vapor	---	Slight surface etch- 5-7 mil dia loss
4	Silicone Rubber Parker S604-7	96	450°	Air Oven	30	2-1/2% wt loss-3-5 mil dia loss
5	Ethylene Propylene Parker E515-8	1	450°	Sodium	---	Surface damage
6	Polyimide Dupont "Vespe1"	---	---	-----	---	Not Tested-Rigid Plastic

TEST PROGRAM

A test program was initiated to test the compatibility of silicone elastomer with liquid sodium.

An inert gas chamber with a sodium pot was utilized. Figure 1 shows the sodium pot with the test fixture and a silicone elastomer O-ring. The test fixture was designed to hold an O-ring diagonally in slight tension. Only a portion (three to four inches) of the O-ring was immersed in the liquid sodium. The top support plate of the test fixture fit snugly into the sodium pot to keep the sodium vapors contained. The sodium is introduced into the pot through an internal line from a storage tank. The pot is surrounded by external heaters to maintain the sodium at the temperature required.

Three materials were selected for their potential thermal properties: (1) silicone rubber; (2) ethylene propylene; and (3) polyimide. At the maximum operating temperature, these seals would be subjected to a temperature of 500°F.

Silicone rubber O-rings of Parker Compound S604-7 were acquired and used in the testing program. Silicone rubber consists of the following:

- 1) A silicone gum or polymer - the framework upon which the rubber is built.
- 2) Reinforcing and/or extending fillers - finely divided particles of such materials as synthetic or naturally occurring silicas, which give the rubber strength and body. The most widely used extending fillers are the diatomaceous earths, ground quartz, and clays.
- 3) Special additives - ingredients such as heat stability additives, coloring pigments, blowing agents, and additives that retard creep-hardening. The standard additives for heat stability are Ferric Oxide, Barium Zirconate and Carbon Black.
- 4) A vulcanizing agent - usually a peroxide that reacts when heated to form chemical cross-links within the polymer, giving the vulcanizate its rubbery qualities.

FIGURE 1. Elastomer Compatibility Test Fixture

The producers of silicone elastomer products consider their compounding recipes as proprietary information and are reluctant to give details.

The silicone elastomer O-rings used in compatibility tests were purchased from the Porter Seal Company, Glendale, California, and were certified to be compound S604-7. A personal communication with the manufacturer did disclose the fact the heat stability additive used in compound S604-7 was Ferric Oxide.

The recommended operating temperature of ethylene propylene elastomer is 350°F, which is substantially below the 500°F at which the elastomer seals must operate. For this reason it was subject only to a minimal test.

A brief review of the properties of polyimide (which has excellent high temperature compatibility) showed the material to be a rigid plastic without any elastomeric properties. A sample obtained of "Vespel", a Dupont polyimide, confirmed these properties.

TEST NO. 1

A silicone O-ring was installed on the test fixture and immersed in liquid sodium at a temperature of approximately 450°F for a period of 70 hours. This O-ring was broken when the test fixture was bumped against the side of the sodium pot during a visual inspection in the inert gas chamber.

The O-ring was removed from the inert gas chamber for examination.

Tensile strength tests were performed first on an unexposed control O-ring. These tests were performed on an Instron Tensile Testing Machine. As the samples tested were of uniform cross section throughout their length, the grip jaws weakened the specimen, and all specimens broke at the grip jaw.

The tensile strength consequently was lower than that reported by Parker in their tests (where they used a jaw-bone shaped specimen). The results of several tests using the control O-ring sections showed enough repeatability that we felt the data would be reliable enough to give us a percentage change in tensile strength.

Physical testing of O-ring No. 1 showed the following data:

Tensile strength	30 to 40% loss
Hardness	80 Shore A (+5 Change)

A loss of 10 mils in diameter was noted on the portion of the O-ring immersed in liquid sodium.

Visual examination of the O-ring portion immersed in sodium showed a roughened, serrated surface with penetrations of one to two mils deep (see Figures 2 and 3).

Figure 2 is a view looking at surface of a section of the O-ring near the cut end.

Figure 3 is a view looking at the cross section of the O-ring near the edge showing.

(105X)

FIGURE 2. Surface of O-Ring Near Cut End

(105X)

FIGURE 3. Cross-Section of O-Ring Near Edge

TEST NO. 2

Test No. 2 was a repeat of Test No. 1 except for the time duration. The O-ring was exposed to sodium/sodium vapor for 140 hours.

The results of physical testing and visual examination were very similar to the results of Test No. 1.

TEST NO. 3

Test No. 3 was run to determine the effects of sodium vapor on a silicone O-ring.

The O-ring was placed on the test fixture in such a manner that none of it was in direct contact with the liquid sodium pool. The temperature of the pool of sodium was maintained at 450°F.

The test was run for a period of 140 hours. Microscopic examination of the silicone O-ring surface again showed the groove pattern typical in Tests No. 1 and No. 2, but to a much lesser extent.

A loss of five to seven mils on the diameter was noted.

Figure 4 is a view of a surface section of the O-ring that was exposed to sodium vapor. The starting of deterioration of the surface is clearly visible.

Figure 5 is a view of the surface of an unused control O-ring for comparison purposes.

TEST NO. 4

A heat-soak test was initiated on a silicone rubber O-ring to determine physical change due to temperature. The test was conducted in an air oven at 450°F for a period of 96 hours. The first two hours of soaking produced smoke from the furnace when the door was opened. Figure 6 shows the weight loss of the O-ring during the heat soak. There was a loss of three to five mils in diameter measured at the end of the test. There was no visible surface erosion. Physical test showed a loss of 30% in tensile strength.

(105X)

FIGURE 4. Surface Section of O-Ring
Exposed to Sodium Vapor

(105X)

FIGURE 5. Surface Section of O-Ring
Used as Control

The smoke noticed in the first two hours of heat was unexpected, and could lead to system contamination. This is especially true if the O-rings were to be installed in a vacuum system that is to be heated. Pre-soaking at temperatures before using the O-ring in such systems would probably lead to longer equipment life.

TEST NO. 5

A portion of an ethylene propylene O-ring was immersed in a small quantity of liquid sodium at 450°F for one hour to determine if there was any rapid reaction or degradation of the elastomer. The O-ring was examined and showed a greenish mottled appearance on the surface. However, the seal operating temperature is above the recommended elastomer operational temperature, and no further testing was done.

FIGURE 6. Weight Loss at 450°F in Air Furnace

TEST NO. 6

A sample of polyimide, Dupont "VespeI", was obtained. This sample supported the analysis of physical properties that showed polyimide was too hard and brittle for use as an elastomer. Therefore, there were no sodium compatibility tests conducted with polyimide.

SODIUM CONTAMINATION

A limited amount of testing was done to determine the contamination of the sodium from exposure to the silicone elastomer. With the limited amount of sodium being used (approximately one quart) the silicone in the sodium was readily detected. In Figure 1, the dark specks seen in the sodium crud were sampled. Examination of these specks under a microscope showed that they were clumps of a translucent brown material. This material is primarily silica, with 1 to 3% occluded iron oxide.

BIBLIOGRAPHY

1. Parker Seal Company - Test Report No. 2908 - 5/20/70.
2. Compatibility of Several Plastics and Elastomers with Sodium, Potassium, and Rubidium, by L. Rosenblum and H. Putre, NASA TN-D-1284, 1962 NASA Cleveland, Ohio.
3. Monthly Technical Report for February 1958, PRDC-TR-8, Power Reactor Development Associates, Inc., Detroit, Michigan, December 1958.
4. Compatibility of Rubber and Elastic Materials in Sodium and Sodium Vapor, R. H. Jones, Test Memorandum No. 4, Atomic Power Development Associates, Inc., Detroit, Michigan, December 1958.

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