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SEALS AND PACKING MATERIALS FOR MOLTEN FLUORIDE SALTS

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SEALS AND PACKING MATERIALS FOR MOLTEN FLUORIDE SALTS

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ABSTRACT

An investigation was made of packing materials, to be used in a conventional type of stuffing box with a rotating shaft, for sealing at elevated temperatures a fused-salt liquid fuel being circulated by a centrifugal pump. The results of 26 tests are reported in which the fluoride NaF-ZrF_4-UF_4 (50-46-4 mole %) was sealed, with acceptable leakage rates, for periods up to 3500 hr. However, the investigation has not progressed to the point where these materials have been applied successfully to a full-size pump.

Various graphites (or graphitic mixtures) were used in the majority of the tests because of their apparent resistance to wetting by the liquid fuel. Of the types of graphite tested, the ones containing large amounts of amorphous carbon appeared to be the most promising even though they exhibited greater frictional properties. In some tests additives were mixed with the graphite powders as lubricants and/or binders.

Although the powdered materials indicate promise as a packing, the main difficulty encountered during the investigation was the retention of the packing itself within the stuffing box.

INTRODUCTION

The design of the pumps for circulating the heat transfer medium of a circulating-fuel reactor becomes a problem at high temperatures, in that the pump must have seals and/or bearings that can be operated in the pumped liquid. A seal for this application must provide minimum leakage and utmost reliability, since any repair or maintenance is complicated because of radiation. A similar problem encountered before in securing a leak-tight seal led to the development of (1) an electromagnetic type of pump for use with conductive fluids such as liquid metals and (2) a canned-rotor type of pump. When temperatures are too high for motor windings and when the pumped fluid is not a conductor, neither of these pumps is suitable. Consequently, an investigation was initiated on the study of seals and packing materials, and this report covers the work performed between September 1952 and March 1954. The problem was to seal reliably the shaft of the centrifugal or axial flow pump which would circulate the fuel through a reactor. The fluoride-salt fuel is corrosive to all but a few known materials, and one of the more resistant materials is graphite, which also appears to resist wetting by the fluoride salt. These two characteristics were the basis for emphasizing the testing of graphitic materials. In some tests additives such as MoS_2, BN, BeF_2, and BaF_2 were mixed with the graphite powder to act as a lubricant and/or binder.

This investigation is a continuation of some earlier work done by the Experimental Engineering Group of the Aircraft Reactor Engineering Division in connection with valves for liquid metals[1] and fluorides.[2]

TEST PROCEDURE

Types of Graphite Used

The several types of graphite investigated can be divided into "natural" or "artifical." Natural graphite is a mineral form of graphitic carbon, crystallized in the regular hexagonal system with rhombic symmetry. There are three physically distinct varities of natural graphite, depending on its mode of formation: disseminated flake, vein (also called crystalline or plumbago), and amorphous.[3] Artifical graphite, or electrographite, is

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[1] W. C. Tunnell, *ANP Quar. Prog. Rep. Sept. 10, 1951*, ORNL-1154, p 24.

[2] H. R. Johnson, *Valve and Pump Packing for High Temperature Fluoride Mixtures*, Y-F17-28 (Sept. 15, 1952).

[3] S. B. Seeley and E. Emendorfer, *Natural Graphite*, reprint from *Encyclopedia of Chemical Technology*, The Interscience Encyclopedia, New York, 1949.

made from carbon by heating it to a high temperature, and once converted to graphite does not revert to its original state. Graphitic particles can be formed from carbon to which has been added a binder, usually a petroleum pitch, and which has been subjected to high temperature and pressure; the solid shapes exhibit characteristics which are dependent upon the treatment.

Natural graphite from the following sources was readily available for test: Joseph Dixon Crucible Co., Jersey City, N.J.; Asbury Graphite Mills, Inc., Asbury, N.J.; American Graphite Co., Jersey City, N.J.; and J. T. Baker Chemical Co., North Phillipsburg, N.J. Table 1 gives a summary of the properties determined for some of the natural graphites. The artificial graphite used in the test was manufactured by National Carbon Co., Cleveland, Ohio, and the United States Graphite Co., Saginaw, Mich., and was obtained from the Y-12 Carbon Shop as machining dust. This material was essentially 100% graphite. Different particle sizes were obtained by the use of sieves.

Packing Penetration Tests

Materials under consideration were first screened in a packing penetration test for (1) their retentive characteristics or their capacity to resist passage or penetration of the liquid through the material and (2) their compatibility with the liquid at 1500°F. The packing-penetration-test apparatus, shown in Fig. 1, was first packed with the material to be tested, and then pressure was applied by turning the screw with a torque wrench. After the open end was welded shut, the unit was installed in a tube furnace and heated to 1500°F. When the unit reached the desired temperature, a small amount of liquid fluoride was introduced into the top of the test apparatus, and inert-gas pressure was applied. Application of pressures up to 30 psig tended to force the liquid around the washer and through the test material. A constant check was made on the temperature and on the period of time before leakage of the fluoride occurred. Table 2 summarizes results of the packing penetration tests.

Table 1. Properties of Graphite Test Samples

	Type of Natural Graphite				
	Dixon No. 2 Flake[a]	Dixon No. 635[a]	Dixon Microfyne[a]	American No. 620[b]	Baker Technical Powder[c]
Graphite, % (remainder amorphous)	90+	90+	50+	25 to 50	25 to 50
Impurities, % (spectrographic analysis)					
Al	0.02	0.02	<0.04	0.6	0.3
B	<0.03	<0.03		0.06	0.06
Ca	<0.08	<0.08	<0.08	0.06	0.1
Fe	0.2	0.2	0.2	0.6	0.6
K	0.05	0.004	<0.01	0.3	0.15
Mg	0.2	0.02	<0.04	0.2	0.2
Na	0.01	0.01	<0.01	0.08	0.15
Si	0.6	0.3	0.6	5.0	2.0
Ti	<0.04	<0.04	<0.04	0.1	<0.08
Particle size					
Per cent passing 100-mesh sieve	~75	100	100	100	100
Per cent passing 200-mesh sieve	~30	80 to 90	100	100	100
Per cent passing 325-mesh sieve	~10	50	95	30	

[a]Joseph Dixon Crucible Co.

[b]American Graphite Co.

[c]J. T. Baker Chemical Co.

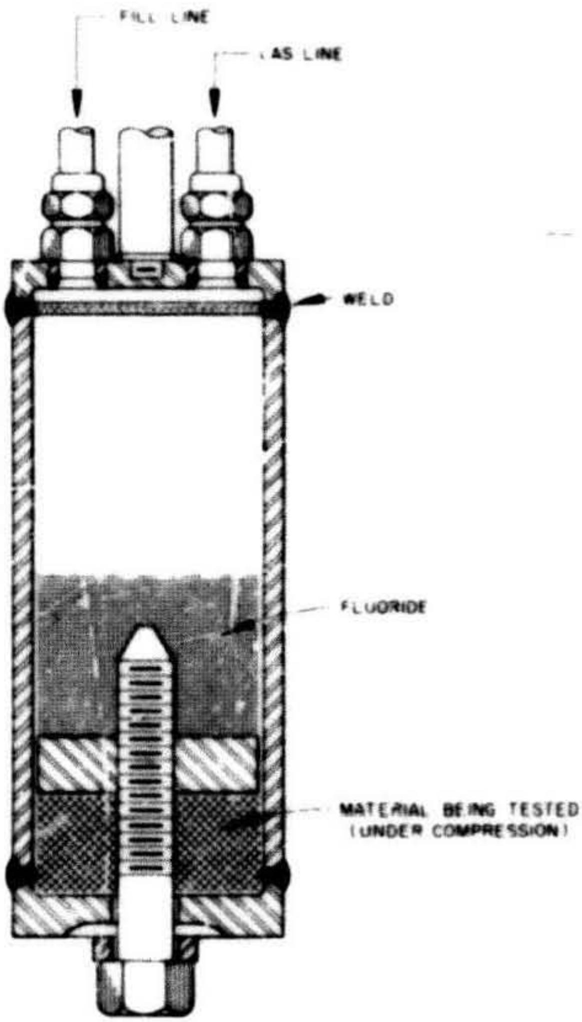

Fig. 1. Packing-Penetration-Test Device.

Seal Tests

The materials showing promise in the packing penetration tests, that is, resisting penetration and leakage and having no reaction with the fluoride, were then tested with a rotating shaft in a conventional stuffing box (Fig. 2). Two sizes of shafts were used, $1\frac{3}{16}$ in. in diameter and $2\frac{1}{2}$ in. in diameter, and each was operated horizontally and vertically. A $\frac{1}{4}$-in. annulus was used with the $1\frac{3}{16}$-in.-dia shaft, and $\frac{3}{8}$- and $\frac{3}{4}$-in. annuli were used with the $2\frac{1}{2}$-in.-dia shaft. As shown in Fig. 2, fuel was introduced and stored in the bottom pot in a molten condition. To start a test using the fluoride, liquid was raised by gas pressure through the riser leg into the seal pot and on into the surge pot until it touched the middle-length spark-plug electrode extension. With the fuel touching this center probe, heat was removed from the flanges on the bottom riser leg, allowing the fluoride to freeze in this region. This frozen material acted as a valve and held the liquid in the apparatus so that inert-gas pressure could be applied to the surge pot.

A series of tests with and without fluoride was conducted. At the beginning of the test the material was given a dry run – the seal was packed and put under compression and the shaft rotated in air to see whether the packing material would leak or vibrate out of the stuffing box. If leakage was apparent at either end of the seal, either it was repacked or more packing material was added.

Several different retaining materials were used with these powdered packings: solid close-fitting graphite rings, copper foil, copper wire braid, Inconel braid, stainless steel braid, stainless steel wool, and silicon-bronze wool. Some of the materials would not retain the powder during the dry-run tests; consequently, they were not tested with fluorides. Generally speaking, retaining materials made from small, flat, ribbon-like metal threads worked better than the foils or braids made from round wire-like threads, with the silicon-bronze wool being best.

The shaft was rotated by means of V-belts driven by a U.S. Electrical Motors, Inc., Varidrive motor. The bearing housing and support were salvaged from old Worthington or Duriron pump housings. The shafts were machined from stock and used plain or with a Stellite or Colmonoy hard facing material on the surface. Lack of concentricity of the seal components when they were at operating temperatures was thought to be a source of vibration; this is in addition to the vibration inherent in equipment with a double belt drive. The vibration could certainly have contributed to some of the difficulties encountered.

Heat was applied to the apparatus by means of Calrod-type heaters or by tube-furnace elements. The seal region was operated at various temperatures, and in some tests cooling coils in addition to the heaters were placed around the seal region.

Table 2. Results of Packing Penetration Tests

Test No.	Material	Fuel Type*	Time (hr) at Indicated Pressures							Remarks
			<2 psi	5 psi	10 psi	15 psi	20 psi	30 psi	Total	
1	Powdered graphite, technical grade	14		76		2		160	238	Did not leak
2	Boron nitride	27		26					26	Leaked when pressure was raised to 30 psi
3	Molybdenum disulfide	27	0						0	MoS_2 not sufficiently compressed; leaked on filling
4	Stainless steel braid	27	1½						1½	Leaked slightly on filling, then stopped when pressure was raised to 10 psi
5	Stainless steel braid impregnated with MoS_2	30	½						½	Leaked when pressure was raised to 5 psi
6	Stainless steel braid impregnated with MoS_2; heated to 800°F in air before filling	30	0						0	Leaked on filling
7	Boron nitride; close-fitting assembly	30		18	24		74	118	234	
8	Flake graphite, Dixon's No. 2	30		2				20	22	
9	Powdered graphite, American Graphite Co. No. 620	30			26		22	15	63	
10	Flake graphite, Dixon's Microfyne	30		21	44			10	75	
11	Stainless steel wool impregnated with MoS_2	30	0						0	Leaked on filling
12	Powdered graphite, technical grade	30						690	690	Did not leak
13	MoS_2 in copper sheathing	30						½	½	
14	Graphite from Y-12 Carbon Shop	30		2				260	262	Slow leakage detected at 96 hr
15	National Carbon Co. graphite 2301	30						191	191	Slow leakage detected at 72 hr
16	Graphite from Y-12 Carbon Shop 55%, BeF_2 45%							736	736	Did not leak; operated at 1150°F
17	Graphite from Y-12 Carbon Shop 50%, MoS_2 50%							40	40	Slow leakage detected at 30 hr

Table 2. (continued)

Test No.	Material	Fuel Type*	Time (hr) at Indicated Pressures							Remarks
			<2 psi	5 psi	10 psi	15 psi	20 psi	30 psi	Total	
18	National Carbon Co. graphite BB-4	30		1/2					1/2	Leaked when pressure was raised to 30 psi
19	Asbury graphite 805	30						1660	1660	Did not leak
20	National Carbon Co. brush-type graphite	30	1/2						1/2	
21	Norton Co. boron nitride	30		2				1273	1275	Did not leak
22	Carborundum Co. boron nitride	30		2				138	140	Slow leak detected at 36 hr
23	Barium fluoride	30		1/2					1/2	
24	Zirconium oxide	30		1/2					1/2	Leaked when pressure was raised to 15 psi
25	Asbury graphite 90%, barium fluoride 10%	30		4				624	628	Slow leak detected at 520 hr
26	Asbury graphite and Fel-pro C-5 high-temperature thread lubricant	30				2		1026	1028	Did not leak

*Fuel No. 14: $NaF-KF-LiF-UF_4$ (10.9-43.5-44.5-1.1 mole %).
Fuel No. 27: $NaF-ZrF_4-UF_4$ (46-50-4 mole %).
Fuel No. 30: $NaF-ZrF_4-UF_4$ (50-46-4 mole %).

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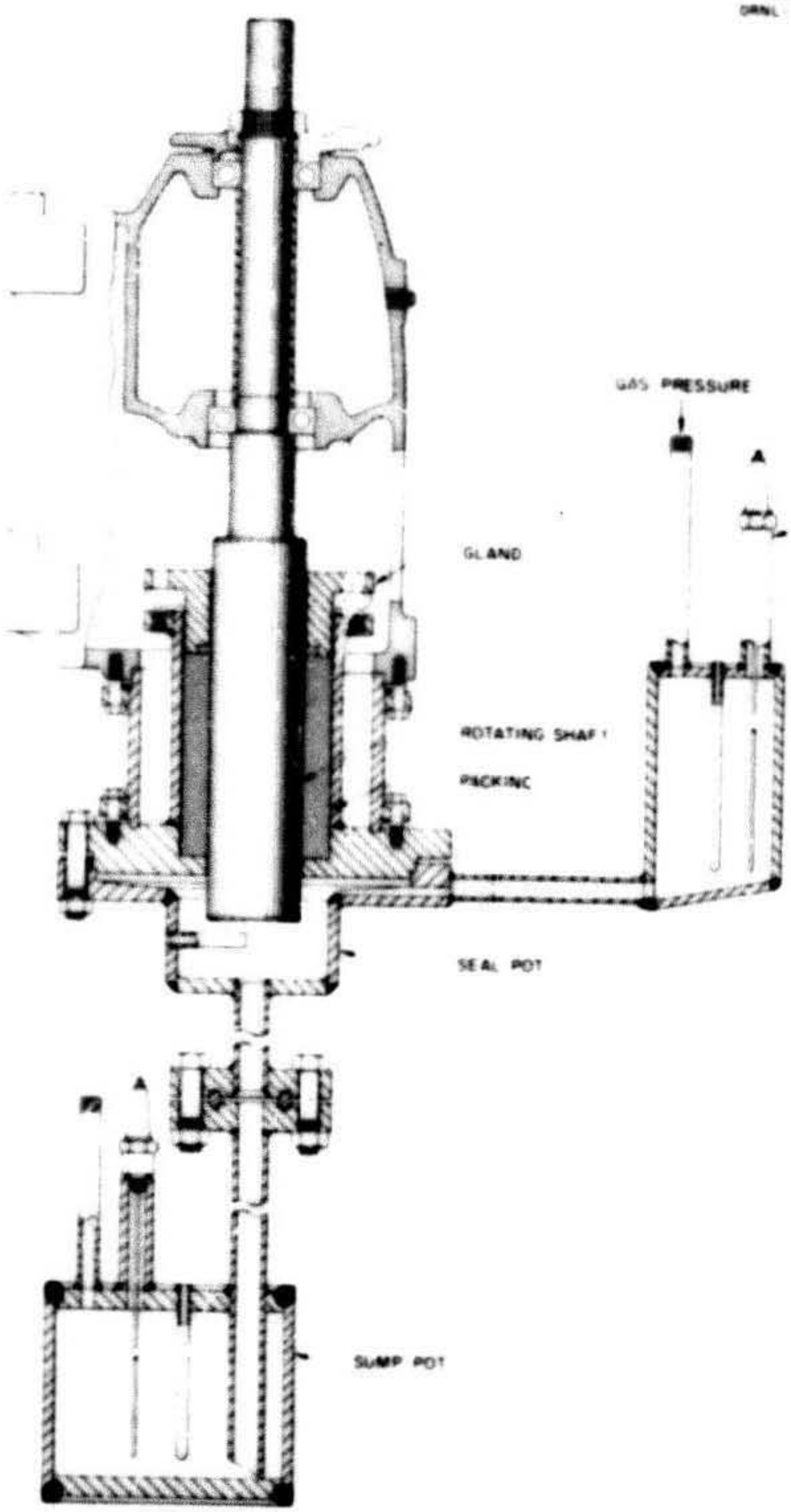

Fig. 2. Packed-Seal Tester.

The sealed-liquid temperature was varied from 1050 to 1500°F.

Continuous-reading instruments were used to record the various temperatures throughout the apparatus, as well as to record the electrical power supplied to the motor. No-load characteristics were established for the motor, V-belts, and bearings so that any change in power requirements could be attributed to friction within the seal region. No attempt has been made to determine the error of temperature measurement in the wells, but thermocouples were also located on the surface of the rotating shaft. Generally speaking, when thermocouples were adjacent to each other, for example, one outside the packing and one on the rotating shaft, the temperature readings differed by only a few degrees.

Operational characteristics of the apparatus were relatively consistent, in that power surges, as indicated on the recording wattmeters, normally showed up simultaneously with increases in temperature, and, by observing the various thermocouple readings, some estimate of other operating characteristics could be obtained. One unusual phenomenon was encountered several times during different tests. When the temperature within the seal region was between 450 and 550°F, instantaneous high-power requirements were noted. This condition was observed on heating and also on cooling.

It was originally intended to develop a seal that would operate above the melting point of the fluoride so that there would be no freezing of the leakage material within the seal region, but most of the tests failed when temperatures above 960°F were reached. Other tests were conducted with the seal below this temperature, and a so-called "frozen-packed seal" existed. The disadvantage of the frozen-packed or other frozen type of seal is that, if the shaft or pump rotation is stopped by accident or is intentional, the heat of friction is lost, and any fluorides within the seal will freeze and seize the shaft. Heating is normally required to melt the frozen fluoride before the shaft can be broken loose, and any excess heat contributes to the possibility of liquid leakage. This condition would probably not exist in a seal that remained above the melting point of the fluoride, so that stopping of the pump would not be critical, and temperature adjustments would not be necessary for a restart. In substance, a frozen-packed seal is one in which the temperature gradient is such that the sealed material freezes within the packing and tends to prevent leakage. Such a seal is characterized by variable and sharply fluctuating power requirements.

RESULTS AND DISCUSSION

The 26 tests reported can be divided into five different classifications: (1) powdered graphite packing, (2) other powdered packings, (3) metallic-type packings, (4) shafts with grooved surfaces, and (5) packing with solid-type materials. Results of individual tests are reported in detail in the Appendix.

Powdered Graphite Packing

It was recognized from the outset of the investigation that human error in packing powdered materials would be encountered. While it was agreed that using an "off-the-shelf" variety of packing would be desirable, such an item is unavailable. As mentioned previously, graphite apparently resists being wetted by the fluorides, and therefore it was thought that this material would form an effective seal. It was necessary that the graphite could be packed sufficiently tight around a rotating shaft to prevent leakage and still remain sufficiently "fluid" or resilient to prevent binding of the shaft. The investigation indicated that such conditions can be obtained and that the material will resist passage of the liquid so as to provide a seal.

During the powdered-graphite packing tests certain other problems were encountered. The retention of the powdered materials within the stuffing box was difficult and unpredictable. It is conceivable that any eccentricity of the shaft that resulted from machining inconsistencies or from warping was the basis for most of the difficulties encountered in attempting to retain the powder. Any eccentricity would tend to vibrate the packing or "wallow" out a large annulus around the shaft, contributing to leakage of the graphite out of the stuffing box and/or to liquid leakage through the seal. Another difficulty involved the limit to which the powder could be compressed, because it was confirmed that graphite, when packed too tightly, is not a lubricant but an abrasive. When it was packed properly, no leakage occurred nor was excessive friction encountered. When the packing was made very tight, the shaft would rapidly score and abrade, and the resulting friction would cause high temperatures.

Sealing the $1\frac{3}{16}$-in.-dia shaft was more successful than sealing the $2\frac{1}{2}$-in.-dia shaft, and one possible explanation is that the smaller shafts may have been partially restrained by the packing so as to reduce any eccentricity caused by machining inaccuracies or by thermal warpage.

As may be noted in the Appendix, most of the tests resulted in a frozen seal, or a frozen-packed seal, although test No. 6 involved a packed seal, and there was no detectable fluoride leakage or penetration into the packing. It is believed that a seal for hot fluoride, with an acceptable leakage rate, can be constructed for laboratory use or where there is access for inspection and maintenance.

Additional work must be done before a seal can be developed which will consistently and reliably operate on a rotating shaft for extended periods of time without maintenance.

Other Powdered Packings

The same comments might be made regarding this classification of packing that were made above, except that MoS_2 and MoS_2 mixtures were even more difficult to retain within the stuffing box. There is some evidence that there is a reaction between MoS_2 and the fluoride, and exposure to oxidizing conditions is more severe than with graphite. There appears to be some virtue in having MoS_2, or some other high-temperature lubricant, within the stuffing box in order to reduce friction and heat generation, as well as to reduce abrasion. In a protective atmosphere MoS_2 apparently remains quite "fluid," even at high temperature, and does act as a lubricant under these conditions. There are several grades of this material with varying degrees of purity, but little difference between them could be detected during the investigation.

Lubrication properties of boron nitride at high temperatures were unacceptable, based on dry-run tests.

Metallic-Type Packings

Advantages of metallic-type packing are that it can be retained within the stuffing box and that it can be selected so as to be resistant to corrosive attack. However, all materials tested, which included Inconel and monel braid, nickel and copper foil, stainless steel and bronze wool, and stainless steel ribbon, were sufficiently abrasive to cause severe scoring of the shaft. Therefore, use of metallic-type packings is not recommended.

Grooved Shafts

Seals consisting of a grooved shaft packed in graphite were tested. Two different groove configurations were used; the first consisted of machined annular grooves around the shaft, and the other was a spiral or screw-conveyor groove which was machined into the shaft. These grooves and the annular space between the shaft and the housing were packed with graphite. It was found that such a seal design will apparently resist leakage. In the most successful tests, left-hand spiral grooves were machined in a $1\frac{3}{16}$-in.-dia Stellite-coated shaft and operated with the shaft rotated in a right-hand direction. This groove arrangement apparently provided a constant and more uniform packing of the graphite. One test with this type of seal operated for over 3500 hr with no detectable fluoride leakage, and other tests were made that indicated no penetration of the fluoride into the packing, even when the temperature of the entire seal area was above 1000°F (see Appendix, test No. 20). It is believed that a seal incorporating similar features will operate satisfactorily if some reliable method can be developed to restrain the powdered graphite from leaking from the seal.

Solid Packings

In another attempt to exploit the apparent nonwetting characteristic of graphite and to eliminate the difficulty of retaining the powdered material, a packing was used which consisted of solid Graphitar rings as shown in Fig. 3. In order to compensate for the difference in the thermal expansion of the shaft and the stuffing box, the seal was constructed of solid Graphitar No. 14 rings with a triangular cross section and of mating rings of copper or copper-MoS_2 compacts which would expand against the stuffing-box wall. The Graphitar rings were designed so that they would contact the shaft when at the normal operating temperature. These tests were conducted on the $1\frac{3}{16}$- and $2\frac{1}{2}$-in.-dia shafts (see Figs. 3 and 4). This design appears to be promising but has the disadvantage of being temperature sensitive (see Appendix, tests Nos. 21, 23, and 25). The seals as tested would apparently be successful with helium at 1100°F; when tested with fluoride, there were times when no detectable leakage occurred. In these tests the

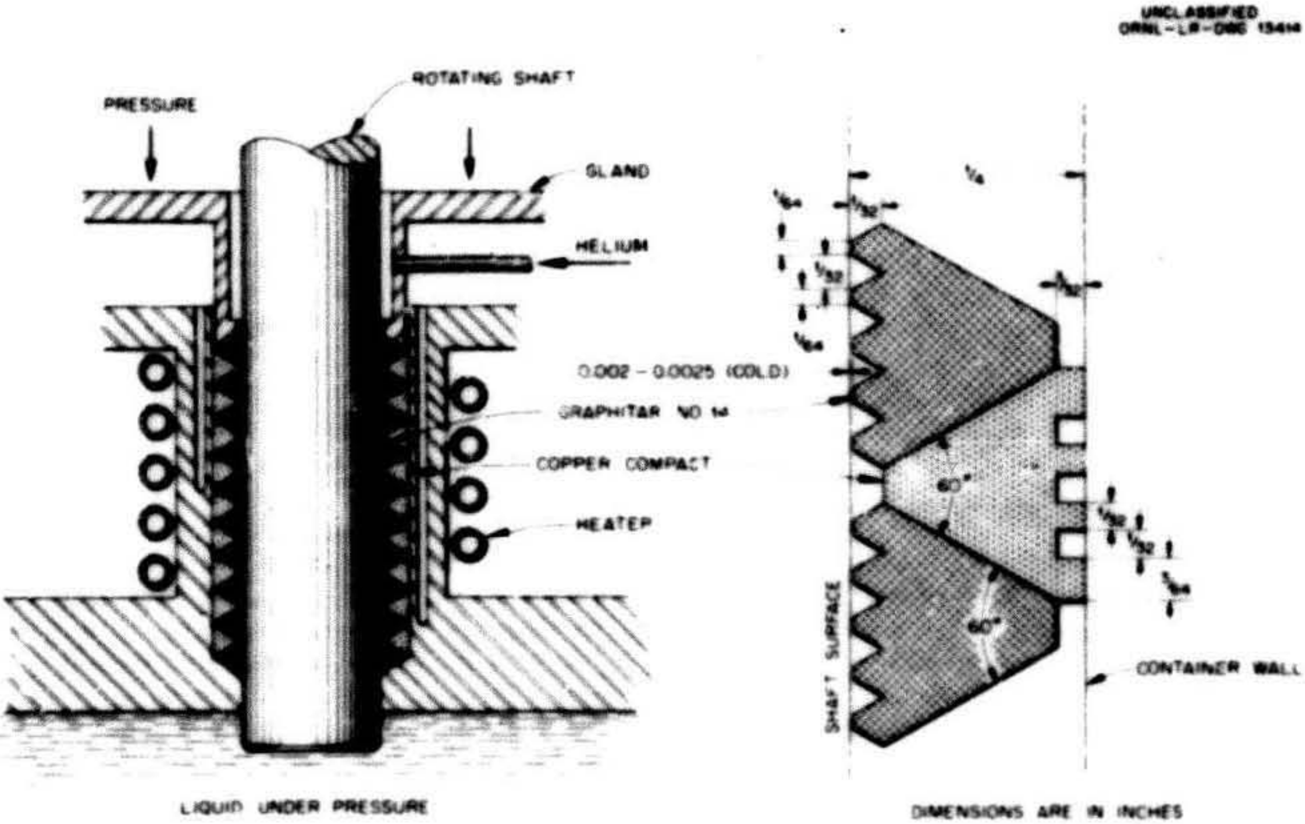

Fig. 3. V-Ring Seal for $1\frac{3}{16}$-in. Shaft.

temperature of the seal area was above 1000°F so that no freezing of the fluoride could occur.

A second design, which used solid packing, consisted of a solid Graphitar No. 14 sleeve that was held in the stuffing box and a solid Graphitar No. 14 bushing that was fastened to the shaft so that a graphite-to-graphite interface existed. The sleeve and the bushing were supported by tapered copper rings to compensate for thermal-expansion differences (see Fig. 5). This arrangement shows some promise (see Table A.1, test 24) and should be given consideration in subsequent investigations of fluoride seals. The design should be less sensitive to temperature changes than the concentric-ring design but has a disadvantage in that it is difficult to reliably mount and support the graphite.

The tests in this series were true packed seals, in that the temperature of the entire seal region was above the melting point of the fluoride. Leakage rate was found to be a function of temperature.

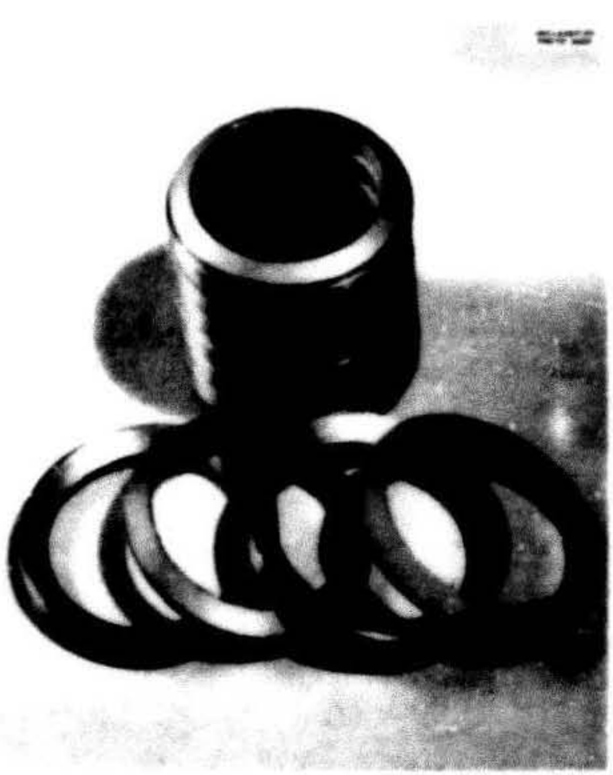

Fig. 4. V-Ring Seal for $2\frac{1}{2}$-in. Shaft.

CONCLUSIONS

Although this investigation did not result in the development of a successful seal, the progress

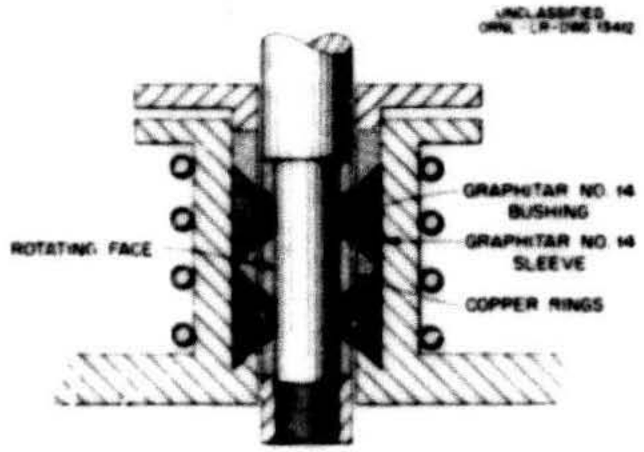

Fig. 5. Stair Seal with Graphite-Graphite Interface.

made was sufficient to indicate that successful seals of this nature can be developed.

One limitation that was imposed on the stuffing box around the $2\frac{1}{2}$-in. shaft was that the ARE pump forgings, the application for which these packed seals were originally intended, were made before any seal tests had been conducted and, consequently, before the annulus diameter and length were established. The $2\frac{1}{2}$-in.-dia seal test equipment was made to conform with the ARE pump dimensions, and there was some evidence that results from the tests would have been more successful if the annulus could have been made larger.

It is believed that a successful packed seal could be designed at this stage of development if a successful method of retaining the graphite were available. Such a seal would require that the shaft have a corrosion-resistant, hard-surface coating that would remain concentric at elevated temperature. The annulus surrounding the shaft would have to be large enough so that the compressive action of the pressure, as applied by the gland, would be fairly uniform throughout the length of the seal; also, some method should be employed which establishes a graphite-to-graphite interface, such as annular or spiral grooves. However, to better understand this type of seal or other seals designed to prevent leakage of the fluoride, it is felt that more theoretical and experimental data are needed.

Included herein are the individual seal tests within the various classifications and a summary of the operating conditions (see Table A.1). The test number corresponds to the order in which the tests were conducted.

At the beginning of this series two pieces of test apparatus utilizing a vertical $1\frac{3}{16}$-in.-dia shaft and a short-length stuffing box were available. It was evident after a few tests that a longer stuffing box might be advantageous, so these two test stands were modified accordingly. Further tests indicated the need for additional apparatus, so two more rigs were fabricated. After using these test stands for a period of time with encouraging results, it was decided that seals incorporating full-size shafts, that is, ARE pump dimensions, should be investigated; consequently, two test rigs using shafts of this size were fabricated. In all the above tests the shafts operated vertically. In order to further simulate the ARE pump configuration, the $2\frac{1}{2}$-in.-dia shaft rigs were modified so that the shafts operated horizontally (see test No. 18). The modifications required to change from vertical to horizontal operation did not materially affect any components or instrumentation.

Powdered Graphite Packing

Test No. 1. – After careful consideration of the results of the work on seals by Johnson *et al.*,[1] it was decided that graphite was the most helpful constituent in these packings, and a test with only graphite as the packing material was devised. In this test a $1\frac{3}{16}$-in.-dia shaft, hard-faced with Stellite, was used in a $1\frac{3}{4}$-in.-long stuffing box with a $\frac{1}{4}$-in. annulus. Technical-grade graphite was packed in the stuffing box, and close-fitting Graphitar No. 14 rings were used as retainers of the powder. The gland was tightened by means of bolt pressure. Liquid at 10 psi and at 1050 to 1075°F was introduced against this seal, and there was some leakage. The power requirement was variable from 2 to 3.5 kw at 1500 rpm. The seal temperature varied from 750 to 975°F at the hot end and from 500 to 850°F at the cooler end, so that a frozen seal resulted. This test continued for 49 hr before termination for inspection. After disassembly there was no visible evidence of wear or corrosion. The fluoride used in this test was fuel No. 27 (NaF-ZrF_4-UF_4, 46-50-4 mole %).

Test No. 2. – The components used in test No. 1 were reassembled for test No. 2, but the gland was modified to be spring-loaded instead of bolted, so that any differential expansion of the packing material, shaft, and stuffing box would not alter the force on the packing.

This test operated for 74 hr at a seal-pot temperature between 1060 and 1175°F, a speed of 1500 rpm, a power input of approximately 1.4 kw, and a liquid pressure up to 30 psi. During this time some leakage occurred. After the 74-hr operation period, leakage collections were made every hour, with the seal temperature at the hot end being varied from 870 to 1250°F at approximately 50°F increments and then reduced back to 870°F in the same manner. The liquid pressure was 16 psi. This procedure was repeated with a liquid pressure of 30 psi and with 100°F increments. The results of the leakage test were neither consistent nor conclusive. The leakage rates varied from 0.15 (1135°F) to 2.3 g/hr (1005°F) at 16 psi, with an average of approximately 1 g/hr during the test. At the higher pressure the variation was greater but the average was only slightly higher. The test was terminated after 315 hr of operation, when the liquid was heated to 1500°F and the temperature at the cooler end of the seal exceeded the melting point of the fluoride, resulting in excess liquid leakage. The fluoride used in this test was also fuel No. 27. At disassembly no visible evidence of scoring of the shaft was noted.

Test No. 3. – The test apparatus was the same as that in test No. 2. Operation continued for 725 hr, at which time the unit was terminated according to schedule. The leakage was similar to that in tests Nos. 1 and 2 for approximately 525 hr, but after this time the temperatures were sufficiently low to permit the fuel to freeze within the graphite-packing area, making the leakage practically nil. Any attempt to operate at still lower temperatures so that the fuel was frozen outside the graphite packing resulted in unstable power requirements and fluctuating temperatures.

Test No. 6. – In order to be able to definitely establish a freezing zone within the graphite area, a tester with a stuffing box 5 in. long and with a $\frac{1}{4}$-in. annulus was fabricated. To obtain a more accurate concept of the temperature at the rubbing

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[1] H. R. Johnson, *Valve and Pump Packing for High Temperature Fluoride Mixtures*, Y-F17-20 (Sept. 15, 1952).

Table A.1. Test Data

Test No.	Type of Packing*	Shaft Diameter (in.)	Shaft Orientation	Shaft Surface Material	Retainer Material	Packing Material	Fuel Type No.	Liquid Temp (°F)	Shaft Speed (rpm)	ΔP Across Seal (psi)	Leakage	Time of Operation (hr)	Reason for Termination	Power Requirements	Remarks
1	A	$1\frac{3}{16}$	Vertical	Stellite	Graphitar rings	Tech grade graphite	27	1050–1075	1500	10	Not collected	49	Modification	2–2.5 kw	Short stuffing box; frozen seal
2	A	$1\frac{3}{16}$	Vertical	Stellite	Graphitar rings	Tech grade graphite	27	1060–1175	1500	16, 30		2159	Liquid leak	1.4 kw	Short stuffing box; leakage tests at various temperatures
3	A	$1\frac{3}{16}$	Vertical	Stellite	Graphitar rings	Tech grade graphite	27	to [illegible]	1500	16	Same except last 200 hr	725	Scheduled	1.5 kw	No apparent leakage for last 200 hr
4	B	$1\frac{3}{16}$	Vertical	Cr plate	Graphitar rings	MoS_2	27	1200	1500	5	Low	4	Packing leak	Low	Unable to contain the MoS_2
5	C	$1\frac{3}{16}$	Vertical	Stellite	Inconel braid impregnated with MoS_2		30	1100	1750	10	Low	925	Scheduled	Low, surges	Short stuffing box; frozen seal; shaft lightly scored
6	A	$1\frac{3}{16}$	Vertical	316 SS	Graphitar rings, Inconel braid	Dixon No. 2	27	to [illegible]	to 1680	10	None	~50	Shaft binding	High, surges	Long stuffing box; packing area to 2200°F by friction
7	B	$1\frac{3}{16}$	Vertical	Stellite	Copper braid sleeve +	MoS_2	30	1200–1250	to 2000	5–7	1–2 g/day	122	Shaft binding	900 w	Loss of MoS_2; Cu braid corroded; frozen seal
8	B	$1\frac{3}{16}$	Vertical	316 SS	Graphitar ring, graphite particles	MoS_2	30	1200–1300	1480	10	50–75 g/day	271	Heater burnout	300–800 w	
9	B	$1\frac{3}{16}$	Vertical	Stellite 1	Bronze wool layers	MoS_2 layers	30	1200–1275		10	Negligible	78	Excess leakage	100–200 w	Liquid leakage when heated above 960°F
10	A and B	$1\frac{3}{16}$	Vertical	Stellite 1	Bronze wool	50–50 MoS_2-graphite	30	1150–1300	1500	10	None 1st 200 hr av 3.5 cm^3/day	1652	Scheduled	~200 w	Was stopped for $10\frac{1}{2}$ hr; motorpower start
11	C	$1\frac{3}{16}$	Vertical	Stellite 1	Copper rope and MoS_2		30	1200–1250	to 2800	10	High	>1000	Scheduled	Low, surges	Typical frozen-seal leakage; Cu particles welded to shaft; shaft scored; temperature sensitive
12	D	$1\frac{3}{16}$	Vertical	316 SS	Inconel braid	Dixon No. 2	30	1050–1225	1500	7.5	Not detectable	92	Bearing failure	Low, surges	Seven $\frac{1}{8} \times \frac{1}{8}$ in. grooves; shaft eroded; 5-min stop-start
13	D	$1\frac{3}{16}$	Vertical	Stellite	Inconel braid	Dixon No. 2	30	1100–1200	1000	10	Not detectable	53	Scheduled	Low, surges	Four $\frac{3}{16} \times \frac{1}{8}$ in. grooves
14	D	$1\frac{3}{16}$	Vertical	Stellite	Inconel braid	Dixon No. 2	30	1200–[illegible]	1050	10	Low	~150	Liquid leak	500–650 w	Liquid leak when heated above 960°F; 10-min stop-start
15	B	$1\frac{3}{16}$	Vertical	Stellite	Graphitar ring, Ag-graphite particles	MoS_2	30	1200		10	High	53	Excess leakage	High, surges	
16	D	$1\frac{3}{16}$	Vertical	Stellite	Bronze wool	Y-12 graphite, 5% MoS_2	30	to [illegible]	2890	20	None detectable for 158, low for 675		Scheduled	Low	Spiral groove; 3 threads/in.; shaft corroded
17	A	$2\frac{1}{2}$	Vertical	316 SS	Bronze wool	50–50 MoS_2-graphite	30	1200–[illegible]	1400	10	High	3 runs, 200	Excess leakage	High, surges	$\frac{3}{4}$-in. annulus
18	A	$2\frac{1}{2}$	Vertical and horizontal	Stellite	Bronze wool	Asbury 5%, MoS_2	30	1200	1400	10	~7 g/day	240 vert, ~1000 hor	Modification	Low, ~constant	$\frac{3}{4}$-in. annulus
19	D	$1\frac{3}{16}$	Vertical	Colmonoy	Bronze wool	Asbury 5%, MoS_2	30	1200–[illegible]	to 1275	10	Excessive	106	Liquid leak		
20	D	$1\frac{3}{16}$	Vertical	Spiral-groove stellite	Bronze wool	Asbury 5%, MoS_2	30	1250–[illegible]	1500	10	None detectable	~3500	Heater failure	~200 w	Three heater failures resulted in partial disassembly and then satisfactory restarting
21	E	$1\frac{3}{16}$	Vertical	Stellite	Graphitar and Cu-MoS_2 V-rings		30	1250	1780	5	0.5 g/hr	100	Inspection	~200 w	Seal area above 1050°F
22	A	$1\frac{3}{16}$	Vertical	Colmonoy	Bronze wool	80% graphite 20% BaF_2	30	1200	to 2400	10	~4 g/day for 1st 1000 hr	>4000		Low, ~constant	Negligible leakage after 1000 hr
23	E	$2\frac{1}{2}$	Horizontal	Colmonoy	Graphitar and Cu-MoS_2 V-rings		30	1050–1150	1240	Low	Apparently none at times	114	Inspection	Variable	Seal area above 1050°F
24	E	$1\frac{3}{16}$	Vertical	Graphitar	Graphitar bushing and sleeve		30	1200–[illegible]	to 1000	to 10	4 g/day	~200	Oxidation	Low, steady	Seal area above 1050°F
25	E	$2\frac{1}{2}$	Horizontal	Colmonoy	Graphitar and Cu V-rings			None – dry run		Low				Low, steady	Seal area ~1100°F
26	A	$2\frac{1}{2}$	Horizontal	Colmonoy	Bronze wool	Graphite-20% BaF_2	30	1075–1175	550	5	High	505	Excess leakage	100–500-w surge	

*A: Powdered graphite packing material, B: other powdered packing material, C: metallic-type packing material, D: grooved shafts with powdered packing, E: solid-material-type of packing.

surface of the shaft, a $1\frac{3}{16}$-in.-dia type 316 stainless steel shaft was fabricated in which four thermocouples were located just below the shaft surface. The thermocouple leads were fed through a hole in the center of the shaft, and the signal was taken off by means of a slip-ring arrangement.

The stuffing box was packed with Dixon's No. 2 flake graphite, which was retained by means of Graphitar No. 14 rings, but leakage occurred at the top of the shaft, so Inconel braid was added as the retainer material. The graphite was packed as tightly as possible, and it was necessary that a 5-hp motor be used to rotate the shaft so that the motor would not be overloaded. Fuel 27 was introduced against the packing, and from the start of the test the power and the temperature fluctuated to extremes. As indicated by the thermocouple readings, the shaft surfaces reached 2200°F as a result of friction. After 50 hr of operation the run was terminated because of binding of the shaft. Postrun examination showed that the shaft was severely scored and that the graphite was in caked annular layers. Figure A.1 is a photograph of the stuffing box, cut through the center line, showing the caked graphite and a section of the shaft that extended through the seal area. The type 316 stainless steel shaft was carburized in places to a depth of 0.070 in. and had a Rockwell C hardness of 54. There was no leakage of fluoride during this test, and postrun examination showed no evidence of any fluoride penetration into the graphite-packed region.

Test No. 10. – In this test a Stellite-coated shaft $1\frac{3}{16}$ in. in diameter was rotated in a 5-in.-long × $\frac{1}{4}$-in.-annulus stuffing box. The packing consisted of alternate layers of silicon-bronze wool $\frac{1}{4}$ in. thick and about $\frac{1}{2}$ in. of a 50-50 mixture of Dixon No. 2 flake graphite and MoS_2. The

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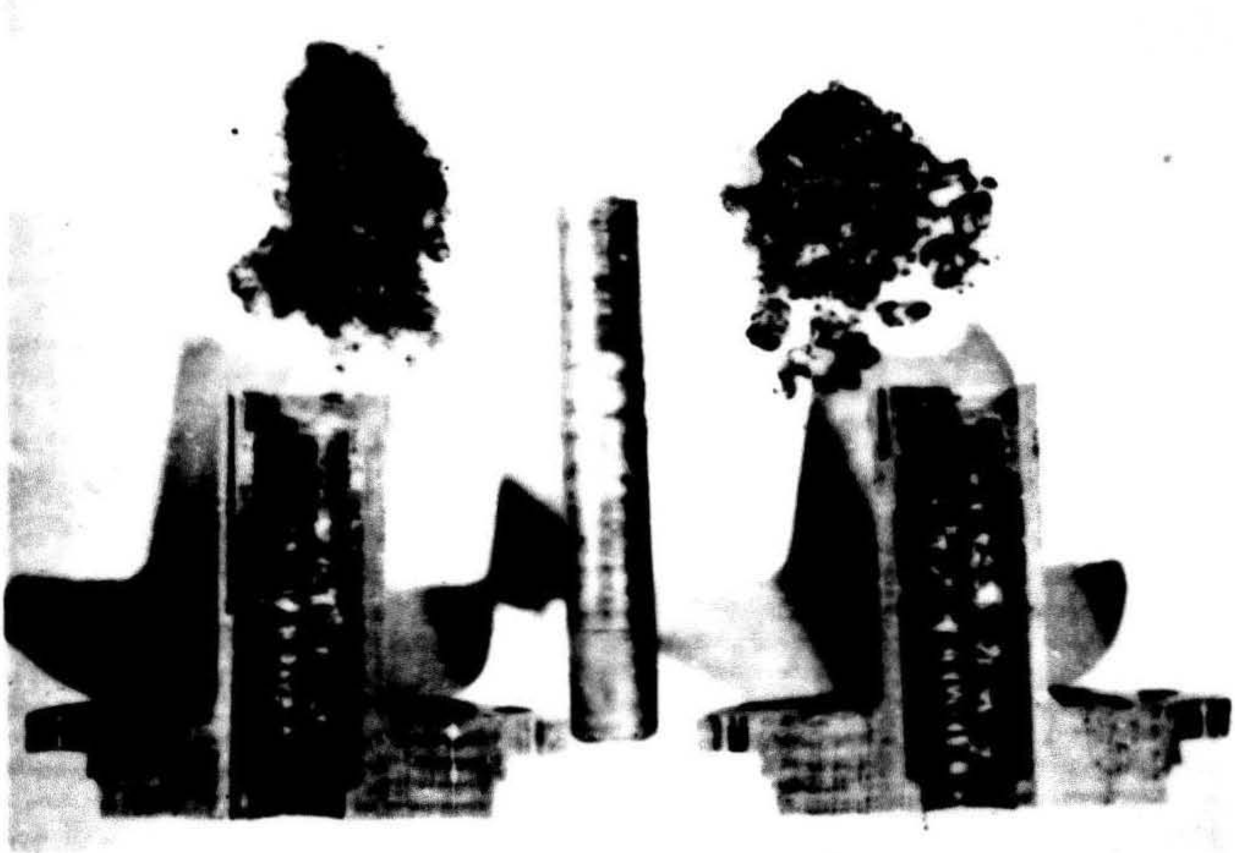

Fig. A.1. Stuffing Box and Scored Shaft – Test No. 6.

liquid used was fuel No. 30 ($NaF-ZrF_4-UF_4$, 50-46-4 mole %) at 10 psi, with the shaft turning at 1500 rpm.

It is believed that, during the first 200 hr of operation of this test, the freezing-point temperature existed within the braid and packing, inasmuch as there was no leakage and the power requirements were low and constant. Several successful stop-start tests were made without trouble of any kind; however, the location of the freezing-point temperature apparently moved outward to the gland, and a small leak developed. Leakage during the run varied and averaged about 3.5 cm^3/day over 1652 hr of operation. The test required a minimum of attention and maintenance after it was in operation and was terminated in order to make the equipment available for other tests.

Test No. 17. – As might be expected, tests conducted here verified that there is considerable difference in sealing around small shafts, as compared with sealing around large shafts. Seal constitutents and configurations that looked promising when used with the $1\frac{3}{16}$-in.-dia shaft failed or were not so successful when used with the $2\frac{1}{2}$-in.-dia shafts. The first seal tested with a vertical $2\frac{1}{2}$ in.-dia shaft consisted of alternate layers of bronze wool and a 50-50 mixture of graphite and MoS_2, as was used in test No. 10. There was a difference, however, in that the $2\frac{1}{2}$-in. shaft was not hard-surfaced. In operation, the power requirement was high, there were erratic surges, and liquid leakage occurred which seemed to wash some MoS_2 out of the packing, thus leading to additional leakage. This test was terminated after a short period of operation because of excessive leakage. Disassembly showed that the shaft had been slightly scored.

Test No. 18. – The seal employed in the second test with the $2\frac{1}{2}$-in.-dia shaft consisted of alternate layers of bronze wool and an Asbury graphite and 5% MoS_2 mixture. At startup the leakage consisted of an excessive amount of fluoride, together with some packing material; so after a short period of time the unit was stopped, and additional graphite mixture and bronze wool were added to the stuffing box. During this 1-hr shutdown the shaft remained free and turned easily, so that when the unit was reassembled it restarted by motor power alone. Operation with the shaft vertical and with small leakage continued for 240 hr, at which time the apparatus was modified for horizontal operation. During the modification the seal and the shaft were not disturbed. The unit was restarted without trouble and continued in operation with the shaft horizontal for over 1000 hr. There was no detectable difference in vertical or horizontal operation. The leakage rate varied; it ranged from as much as 7.5 cm^3/day to as little as 1 cm^3/day, averaging about 3.5 cm^3/day for the 1000 hr. The seal operated with a shaft speed which was varied from 800 to 1400 rpm, holding a 1200°F liquid against a pressure of 10 psi. The power requirement was erratic on occasions but for the most part was fairly constant and low. The test was terminated in order to modify the stuffing box, changing from a $\frac{3}{4}$-in. annulus to a $\frac{3}{8}$-in. annulus, to correspond with the ARE pump configuration.

Test No. 22. – Apparently, graphite can be quite abrasive under these test conditions, and because of this it was felt that it might be advantageous to provide a lubricant for the powder. Now BeF_2 is like glass in that it has no true melting point, and it is believed that any reaction of the fluorides with BeF_2 will form a lower-melting eutectic; so this material was investigated. An 80% graphite–20% BeF_2 mixture was used as a packing, and bronze wool was used as the retainer in this test. Before operation the mixture was packed in place and the assembly heated to 1000°F before the packing was compressed. This procedure was repeated until it was thought that the fluoride had flowed into the voids in the graphite powder. Fuel 30 was introduced against the seal at about 1200°F and at 10 psi. The seal temperature gradient varied from 500 to 1000°F, and the power requirement was slightly lower than with the seal using graphite alone. Shaft speeds to 2400 rpm produced a leakage of slightly over 4 g/day for the first 1000 hr of operation, but the leakage was reduced to practically nil during the next 3000 hr of operation. This test continued in operation for over 4000 hr.

Other Powdered Packings

Test No. 4. – It is claimed that MoS_2 has good high-temperature lubricating properties, and it was thought that it might also be used as a packing material. A test was made on a seal consisting of this material retained by Graphitar No. 14 rings in a short stuffing box. The fluoride leakage was slight, operation was smooth, and there were no power surges; however, the MoS_2 rapidly leaked out of the stuffing box, and so the test was terminated. The "fluidity" of this material was verified later when an attempt was made to retain it in a packing penetration test (see, "Seal Tests," this report).

Test No. 7. – In an attempt to retain the MoS_2, a braided copper sleeve was filled with MoS_2 and this "sausage" was used to pack the stuffing box. This approach did not solve the retention problem, since the material had again leaked out of the packing at the end of 120 hr of operation. Seizing of the shaft resulted in termination of the test. Postrun examination indicated that there was a large loss of MoS_2 and possibly some reaction with the liquid, since the sheathing was worn and corroded.

Test No. 8. – Another attempt to retain the MoS_2 involved the use of Graphitar rings to retain graphite which, in turn, was to retain the MoS_2. The packing consisted of the rings and 1 in. of Dixon No. 2 flake graphite on each end of a 2-in. section of MoS_2. This worked only fairly well as a packing material, and fluoride leakage occurred; however, the seal operated without excessive leakage for a short period of time with the complete seal-region temperature above the melting point of the fluoride. In general, the fluoride leakage rate was high and was as much as 50 to 75 g/day. The test was terminated after 271 hr of operation because of a gasket leak which, in turn, caused a heater to fail.

Test No. 9. – Still another attempt to retain the MoS_2 consisted in the use of alternate $\frac{1}{4}$-in.-thick layers of bronze wool and MoS_2. There was negligible loss of packing during a 12-hr dry run, so fuel 30 at 10 psi was introduced against the seal. This test continued in operation for 78 hr with slight leakage at 1200 to 1275°F. Apparently, there was penetration of fluoride into the packing, since the shaft would freeze during stop-start tests. Termination resulted when an intentional heating of the whole seal above the melting point of the fluoride caused an excessive liquid leakage.

Test No. 10. – This test was similar to test No. 9 except that the packing was a 50-50 mixture of graphite and MoS_2. Test No. 10 is reported in more detail in the section of the Appendix, "Powdered Graphite Packing."

Test No. 15. – A test similar to test No. 8 was conducted, using a silver-impregnated graphite powder (machine turnings) instead of graphite as the retainer, but it was unsatisfactory. There was some packing leakage, and the amount of fluoride leakage began to increase, so the test was terminated after 53 hr of operation.

Several dry runs were attempted in which BN was used as a packing because this material is reported to be a high-temperature lubricant; the tests did not verify this characteristic. The power requirement was high and erratic, and some shaft scoring was noted during the postrun examination.

Metallic-Type Packings

Test No. 5. – A short stuffing box was used in this test, and the seal consisted of commercial-type Inconel braid, impregnated with MoS_2, packed around a Stellite-coated shaft. This test resulted in a frozen seal in which the metal braid provided the resistance to the flow of the fluid leaking through the packing. The seal was temperature sensitive in that variations in temperature produced extreme power surges and changes in the leakage rate. When the seal temperature was properly adjusted, operation was fairly satisfactory, but leakage was evident and power surges continued. The test continued for 925 hr, sealing fuel 30 at 10 psi with the shaft rotating at 1750 rpm.

Test No. 11. – This test was conducted with the use of a copper rope composed of fine round wire that was dipped in MoS_2 suspended in a volatile carrier. Again, the temperature within the seal was such that a frozen region was established within the packing. The test operated for over 1000 hr and exhibited typical frozen-seal characteristics, that is, power surges, solid-chip fluoride leakage, and generally unstable operation. At termination, the hard-faced shaft was found to be scored, and the copper rope was worn and corroded.

Several dry runs were made on commercial-type packings which included copper rope, copper foil, nickel foil around monel braid, etc. All tests resulted in high friction and severe scoring of the rotating shafts, and these packings would not seal water or water-glycerine mixtures against very low pressure.

Grooved Shafts

Test No. 12. – In order to investigate the apparent nonwetting characteristic of graphite, a test was conducted with a seal in which a graphite-to-graphite interface might be established. A type 316 stainless steel shaft was machined so that seven annular grooves ½ in. wide × ⅛ in. deep × ¼ in. in pitch would be within the seal area. Inconel braid impregnated with MoS_2 was used to retain a packing of Dixon No. 2 flake graphite.

The test was operated for 92 hr with the sealed liquid at 1050 to 1225°F and at 7.5 psi without apparent leakage. Several stop-start tests were made, and there was evidence that the fluoride had penetrated the packing, since some sticking occurred after a 5-min stop. The run was terminated for examination because of excessive noise, and at disassembly it was discovered that one support bearing had failed. The shaft had been worn and scored severely, resulting in about half the groove lands being completely worn away (see Fig. A.2).

Test No. 13. – A test similar to test No. 12 was set up with new bearings and a Stellite-coated shaft, which was machined with four grooves $^3/_{16}$ in. wide × ⅛ in. deep × $^5/_{16}$ in. in pitch. The liquid temperature was varied from 1100 to 1200°F and the pressure was 10 psi. During the 53 hr of operation no fluoride leakage occurred. The test was terminated for inspection, and examination showed the shaft to be in good condition.

Test No. 14. – The equipment used in test No. 13 was reassembled and operation continued. The liquid temperature was varied between 1200 and 1350°F with a pressure of 10 psi. Stop-start tests were successful through 10-min periods. After 150 hr of operation the seal-area temperature was raised to above 960°F to see whether the seal would operate as a "packed" seal. Unfortunately, during this heating-up period an appreciable amount of graphite leakage occurred, and shortly after reaching 960°F liquid leakage occurred which resulted in the termination of the test. Leakage before the heating-up period was small, and power to the seal was about 500 w, with occasional surges of 150 w.

Test No. 16. – Another test to investigate the graphite-to-graphite interface involved the use of a spiral-grooved shaft. This shaft was machined with a left-hand V-thread about $^3/_{16}$ in. wide, with a pitch of three threads per inch. The shaft was rotated in a right-hand direction so that the spiral groove acted as a screw conveyor and compressed the packing material toward the liquid end. Several arrangements were tried as a means of applying the pressure to the gland, but an air cylinder appeared to be the best, in that the pressure to the gland was constant and easily controllable. With this arrangement it was determined that the packing pressure should be less than 30 psi in order to prevent excessive friction in the seal area.

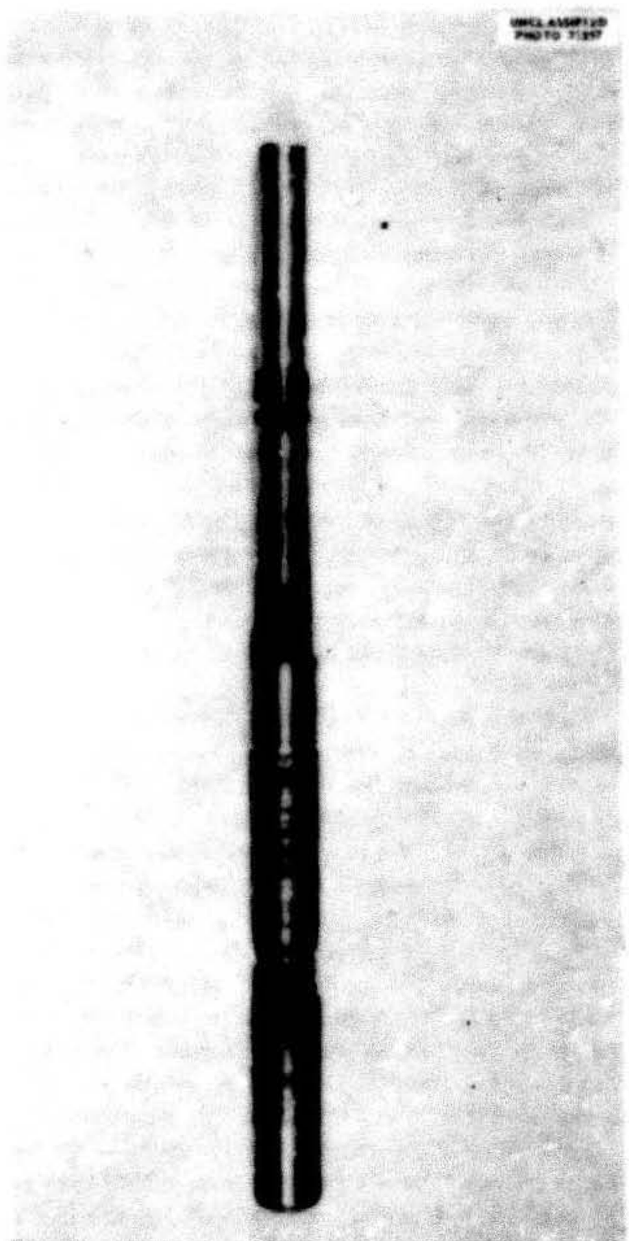

Fig. A.2. Shaft ($1^3/_{16}$-in.) with Seven Annular Rings After 92 hr of Operation.

With bronze wool as the retainer at the liquid end only and with artifical Y-12 graphite plus 5% MoS_2 as the packing, this seal held helium at 10 psi. Fuel 30 was introduced against the seal, and satisfactory operation was apparently obtained, as there was evidence that fluoride did not penetrate the seal area. The shaft was stopped and was free to rotate after a lower temperature equilibrium was established, indicating that the stopping time was not limited. The shaft was stopped and started by motor power after heating of the whole seal to above 960°F; then both operations were performed after cooling and with no heat on the seal area. One significant indication was that the power requirement for starting was less than that for operation. During these operations there was no sign of fluoride leakage, and this condition remained the same for 158 hr of operation. At this time the pressure to the gland was inadvertently left off overnight, and the next day some leakage had appeared that was typical of frozen-seal operation. This test continued in operation as a frozen seal for an additional 675 hr before it was terminated. The liquid sealed was fuel 30 at 1200°F and 20 psi pressure. Figure A.3 is a photograph of the shaft after disassembly.

Test No. 19. – This test was similar to test No. 14, except that the shaft was coated with Colmonoy instead of with Stellite, and the packing consisted of a mixture of Asbury graphite and 5% MoS_2 retained by bronze wool. The test operated for a period of 106 hr, but the leakage rate exceeded that of the previous test. Termination was due to leakage of molten fluoride which resulted from overheating of the seal by friction within the seal area. The machined grooves were not appreciably worn in this case.

Test No. 20. – This test, which was similar to test No. 16, was probably the most successful of the whole series. The only difference between these two tests was that the spiral groove in test No. 20 terminated in an annular ring instead of tapering to a point. The operating conditions were about the same, with the liquid temperature between 1250 and 1300°F and with 10 psi pressure across the seal. The power requirement fluctuated at times during the early part of the run but was fairly consistent and low during the rest of the operation. The seal operated for 3489 hr with no detectable leakage of fluoride and with only a slight amount of graphite leakage during the early part of the run. Stop-start tests which were conducted during the first 1000 hr were similar to those mentioned in test No. 16 and included heating of the seal area above 960°F. In general, all feasible methods were tried, short of disassembly, in an attempt to prove that no fluoride had penetrated into the packing area. During this period of

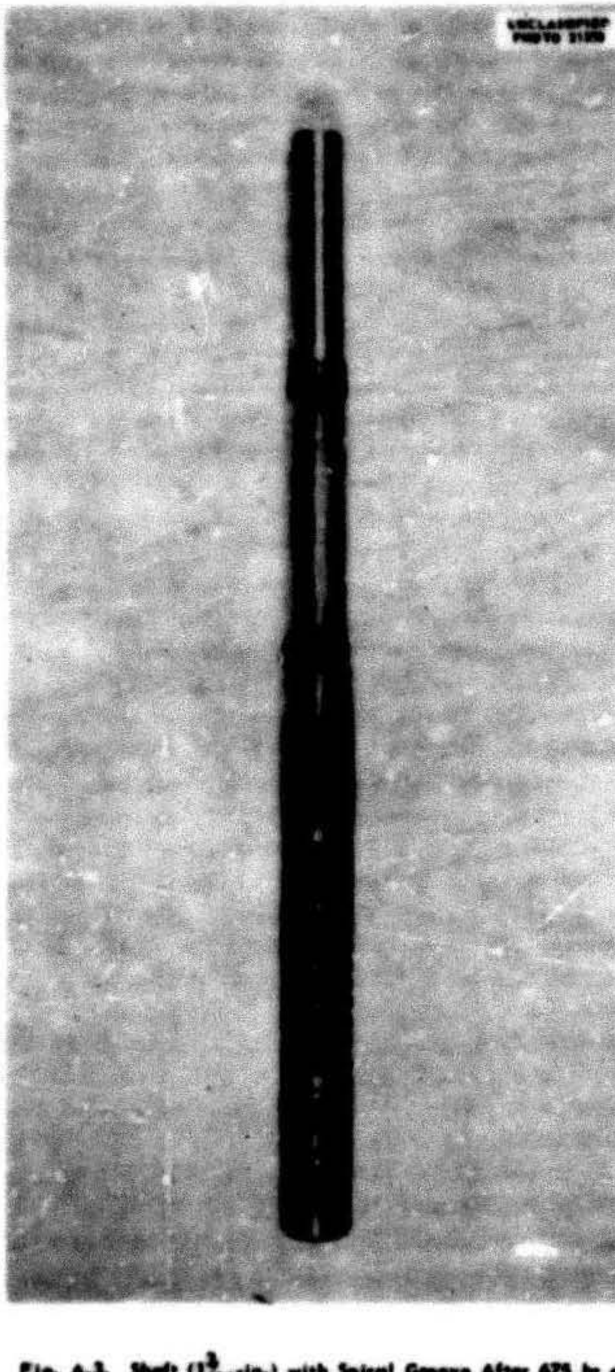

Fig. A.3. Shaft ($1\frac{3}{16}$-in.) with Spiral Groove After 675 hr of Operation.

operation, the seal pot, or container, holding the liquid around the rotating shaft leaked and permitted fluoride to pass through the thermocouple well on three occasions, causing the heater to burn out and resulting in shutdown of the apparatus. The first two shutdowns occurred at 1342 and at 2082 hr of operation. In these instances, the liquid was dumped, the rig was allowed to cool, and a new seal pot and surge tank were installed. After reheating of the system and raising of the liquid into the surge pot, the motor was able to start the rotation of the shaft without mechanical assistance. Termination of the run was due to the third leakage and heater burnout at 3489 hr, at which time the liquid froze in the pot, and the system could not be dumped. Postrun examination showed that the grooves were partially worn from the shaft (see Fig. A.4) and that fluoride had penetrated the packing. When the shaft was pressed from the seal assembly, there was fluoride within the grooves, and the packing was glued together by the material so that it was hard, and chipping was required to remove it from the stuffing box.

Solid Packings

Test No. 21. – In another attempt to determine the nonwetting characteristics of graphite, a seal was used which consisted of solid rings of graphite arranged to provide a close-fitting annulus around the rotating shaft. The V-shaped rings (see Fig. 3) that contacted the shaft were of Graphitar No. 14, and the other rings that fitted between the Graphitar rings were of a copper-MoS_2 compact. A test without fluorides was run by using this arrangement, and it apparently sealed helium at 30 psi and at 1100°F. When fluorides at 5 psi were introduced against this seal, a leakage rate of 0.5 g/hr occurred for 100 hr, at which time the test was terminated for inspection. The coldest part of the seal region varied between 1050 and 1100°F during this period of time, so there was no frozen liquid. No damage was apparent at disassembly.

Test No. 23. – The seal used in this test was similar to the seal in test No. 21, but heaters on the outside of the seal region were used to feed heat into the shaft; it might be expected that such a seal would be temperature sensitive. This was true to some extent when a $1\frac{3}{16}$-in.-dia shaft with a $\frac{1}{8}$-in. annulus was used, but the condition was even more critical when the same design was tried on a $2\frac{1}{2}$-in.-dia shaft. There were periods when the leakage was apparently nil and the power requirements were low, but a slight temperature change had a cummulative effect. A slight rise in

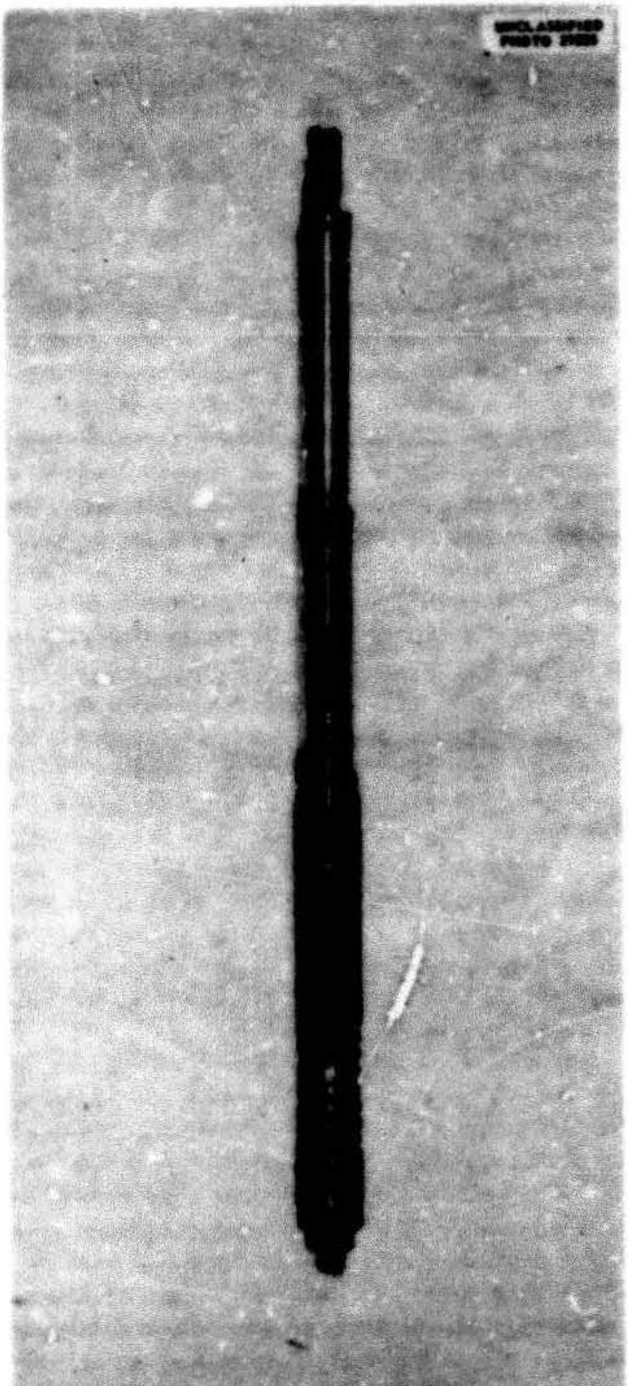

Fig. A.4 Shaft ($1\frac{3}{16}$-in.) with Spiral Groove After 3489 hr of Operation.

the temperature of the shaft would expand the shaft, causing less clearance and more friction and thereby making the shaft still larger, when binding of the shaft and the packing would occur; slight cooling would have an opposite effect, in that the shaft would get smaller, providing greater clearance in the annulus and a resulting leakage. Total operating time was 114 hr, with the whole seal region above 1050°F.

Test No. 24. – Another test was designed to eliminate the metal shaft which might provide a wetted surface for passage of the fuel. In this arrangement, $1\frac{3}{16}$-in.-OD Graphitar No. 14 bushings were mechanically fastened to the shaft, and similar sleeves were employed within the packing area so that these materials were adjacent (see Fig. 5). This seal operated for about 2000 hr under pressure differentials up to 10 psi and with the seal temperature above the melting point of the fuel. Power requirement was low and constant, and the maximum leakage observed was approximately 4 g/day. The test was terminated for inspection.

Test No. 25. – As mentioned above, the apparatus in test No. 23 was quite temperature sensitive during operation. In test No. 25 the rotating shaft was modified so that a heater could be installed within the shaft and adjacent to the seal area. Another difference was that the seals used were solid copper rings instead of the MoS_2-copper compact. The equipment was set up to check the leakage characteristics when helium was sealed at 1050°F. During this dry-run test it was possible to adjust the temperature so that the shaft expanded against the Graphitar rings and reduced the helium leakage through the seal to about 6 cm^3/min. A gas leakage of this magnitude converted to fluoride leakage would be negligible and would result in less than 10 g/day. However, while this test was being conducted the helium supply inadvertently became exhausted, and probably some oxidation of the seal occurred. It was impossible to obtain good sealing after this accident took place, and upon disassembly the outer ring was found to be about 80% oxidized, and the inside diameter of some of the inner seal rings had increased from 0.0005 to 0.0015 in. Graphitar will begin to oxidize in air at about 750°F, and this experience indicates the necessity of complete gas protection on this type of material at all times.

END