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ABSTRACT

Three solid-film lubricant formulations - PbS + MoS_2 + B_2O_3 , GD/FW Dynalube, and Almasol SFD-560 - have been exposed to reactor radiation and tested for wear-life on a Hohman A-6 wear tester. Rub shoes were of Rex AAA steel, load per shoe was 110 lb, and sliding speed was 128 ft/min. Substrate materials were T-l tool steel for GD/FW Dynalube and Timken standard test races (T-54148-3-233) for the other two lubricants. Several test temperatures up to $1150^{\circ}F$ were employed. The solid-silm lubricant specimens received an average radiation exposure of 1.7 x $10^{11} ergs/gm(C)$ of gammas and 3.7 x 10^{16} n/cm^2 (E>2.9 Mev) of neutrons.

Significant decreases in wear life occurred at 80° and 900°F for Dynalube and at 900°F for SFD-560; PbS + MoS₂ + B₂O₃ showed no change; and SFD-560 improved markedly at 80° F.

ACKNOWLEDGEMENTS

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REPORT SUMMARY

The irradiation and testing of three solid-film lubricants (PtS + MoS_2 + B_2O_3 , GD/FW Dyanlube, and Almasol SFD-560) are described.

The lubricants were applied to appropriate substrate materials and tested on a Hohman A-6 wear tester. Rub shoes were of Rex AAA, load was 110 lb per shoe, sliding speed was 128 ft/min, and machine cutoff was automatic at a coefficient of friction of 0.4. Several test temperatures up to 1150°F were employed.

The irradiation was performed with the Ground Test Reactor (GTR at General Dynamics/Fort Worth, in June 1963. The solid-film specimens were exposed to an average gamma dose of 1.7 x 10^{11} ergs/gm(C). The associated neutron exposure was 3.7 x 10^{16} n/cm² (E> 2.9 Mev). Based on the data obtained in the experiment, it was concluded that exposure to reactor radiation had no significant effect on the wear life of the PbS + MoS₂ + B₂O₃ film. However, detectable decreases in wear life were noted at 80° and 900°F on the GD/FW film and at 900°F on Almasol SFD-560. A marked improvement in wearlife properties was displayed by the Almasol film at 80°F after exposure to radiation.

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I. INTRODUCTION

Many lubrication problems are solved by the use of lubricants which are bonded to the bearing surfaces by various methods prior to assembly. These surfaces require no service lubrication during the life of the part. Lubricants thus applied are termed "solid film lubricants." Developmental and proposed UEAF Aerospace vehicles will, undoubtedly, rely upon solid-film lubricants as the most feasible means of lubrication in applications where extreme environments of temperature, vibration, pressure and nuclear radiation may be encountered. Therefore, the Air Force, along with certain other government agencies and private industry, is concentrating on research programs for development of high-temperature solid-film lubricants.

General Dynamics/Fort Worth (GD/FW) is under contract to the U. S. Air Force to evaluate the properties and performance of materials which have been exposed to high-intensity nuclear radiation. This work, performed at the GD/FW Nuclear Aerospace Research Facility (NARF), includes the investigation of the effects of irradiation on the wear-life properties of solid-film lubricants. These studies were initiated under the 1962 NARF program and are being pursued on a continuing basis. This report covers the work accomplished during the NARF-63 period (1 October 1962 to 30 September 1963).

The basic test conditions were established jointly with the ASD project monitor.^a. Three lubricant pigment-bondingagent combinations were selected for testing. They were:

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Lubricant	Nominal Formulation
A - Ceramic-bonded	$PbS + MoS_2 + B_2O_3$
B - Metal-matrix-bonded	Silver-matrix, electro-deposited film (formulation proprietory)
C - Resin-bonded	Pb0 + MoS ₂ + mcdified-silicone resin + proprietory lube pigment.

The developmental work on Lubricant A was done by Midwest Research Institute under Air Force contract. Lubricant B was developed by GD/FW and is referred to as "Dynalube." Lubricant C was developed by the Almasol Corporation of Fort Worth, Texas, and is commercially available as Almasol SFD-560.

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^aMr. B. D. McConnell, ASRCNL-2

II. DESCRIPTION OF TEST EQUIPMENT

2.1 Hohman A-6 Wear Tester

The Hohman A-6 machine was used for all wear-life determinations in this experiment. Figure 1 is a photograph of this machine, along with two other wear testers used in solidfilm-lubricant work at GD/FW.

The mechanics of the Hohman A-6 are shown diagramatically in Figure 2. The test specimen is rotated at the desired speed and two diametrically opposed rub shoes (blocks) are pressed against it under loading. Lubricant failure (metalto-metal contact) is indicated by a sharp rise in frictional torque. The machine stops automatically when this torque reaches a preset value. A cycles counter registers the wear life of the lubricant. Except for the basic drive unit and instrumentation, the tester may be enclosed in a sealed steel chamber. Heaters are included in the standard machine.

The basic capabilities of the Hohman A-6 include:

- . Specimen temperature of from -60° to 1500° F.
- . Loading to approximately 525 lb per rub shoe.
- . Maximum contact-area pressure of approximately 16,000 psi.
- . Rotation to approximately 550 rpm (\sim 200 sliding ft/min at the specimen surface).

Other test parameters, such as atmosphere and pressure, may be varied.



Figure 1. Solid-Film-Lubricants Test Area



Figure 2. Diagrammatic Sketch Shewing Essential Mechanics of Halman A-6 Wear Tester

2.2 Test Cups

Approximately 50 test cups for use with Lubricant B were machined from T-1 tool steel to the configuration specified in Figure 3. Test cups for Lubricants A and C were Timken standard lubricant test races, case hardened to R_c 58-63 (Timken Part No. T-54148-3-233).

2.3 Rub Shoes

A sufficient supply of rub shoes for conducting the tests were fabricated from Rex AAA steel and heat-treated as specified in Figure 4.



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Figure 3. Test Cup for Solid-Film Lubricants

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Fabrication Specifications

- 1. Except as noted, hold 2-place dimensions to $\pm .030$ and 3-place to $\pm .005$. 2. Except as noted in V symbol, surface finish
- to be 63 rms microinches.
- 3. Maintain surfaces determined by .400 ± .002 to within .001 T.I.R of parallel.
- 4. Break all sharp corners with .010 R or 45°
- chamber.
 5. Block specimen shall be REX AAA steel having a Rockwell C hardness of 60-65.
- 6 The .2500-inch dimension shall be parallel to the axis of the cup.

Figure 4. Rub Shoe for Solid-Film Lubricants

III. TEST-SPECIMEN PREPARATION

3.1 Lubricant A: PbS + MoS₂ + B₂O₃

Quantities of PbS and B_2O_3 were screened with a 325-mesh sieve. The ingredients were mixed in the following ratio:

4 parts of PbS (Fisher Scientific No. A-76)

8 parts of MoS₂ (Alpha Molykote, microsize)

1 part of B_2O_3 (F. S. No. L-173)

A sprayable slurry was obtained by mixing one gram of lubricant per five milliliters of an ethanol-water carrier (30% water). The lubricant was sprayed on at room temperature under 30-psi air pressure with a Paasche H-5 air brush. A spraying time was established which gave a film thickness of approximately 0.0005 inch. Bonding was accomplished by placing the specimens in a 200°F oven, increasing the temperature to 1150°F and curing for one hour under a nitrogen blanket. The test cups had been liquid-honed, scrubbed with levigated alumina, and rinsed with water then acetone prior to coating.

3.2 Lubricant B: GD/FW Dyralube

All specimens were prepared by the GD/Fw Engineering Materials Laboratory. Film thicknesses were approximately 0.0005 inch.

3.3 Lubricant C: Almasol SFD-560

All specimens were prepared by the Almasol Corporation. Film thicknesses were approximately 0.0005 inch.

IV. IRRADIATION PROCEDURES

4.1 Radiation Source

The Ground Test Reactor (GTR) served as the mixed-field radiation source. The GTR is a 3-Mw, water-cooled, watermoderated, thermal reactor utilizing MTR-type fuel elements. This reactor is used as the primary source for radiationeffects testing at the Nuclear Aerospace Research Facility. The irradiation of materials and assemblies by the GTR is accommodated by means of a shuttle system which transports the test items into and out of the high-flux region.

The NARF radiation-effects test facilities are described in detail in Reference 1. Figure 5 is a photograph of the overall transport and reactor system.

4.2 Irradiation of Test Specimens

Eight specimens of Lubricant A and 18 specimens each of Lubricant B and C were irradiated in June 1963. The 44 specimens were mounted equidistant from the center of a rack to ensure that all specimens would receive approximately equal exposure. The radiation exposure was monitored by gamma and neutron detectors interspersed with the specimens. Four each of the following detectors were used:

Cobalt foils:	Thermal-neutron detector ($E < 0.48 \text{ ev}$)
Aluminum foils:	Fast-neutron detector (effective threshold 8.1 Mev)
Nitrous-oxide:	Gemma dosimeter



Figure S. Radiation-Affects Testing System

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The details of nitrous-oxide gamma dosimetry are described in Reference 2. Standard foil-counting procedures were used for the neutron measurements, with data reduction by IBM 7090. The neutron flux in energy regions above or below those reported can be estimated by consulting the spectrum information contained in Reference 3.

The results of measurements made in this experiment indicate that the solid-film lubricant specimens received the following average radiation exposure.

Gamma dose	$1.7 \times 10^{11} \text{ ergs/gm(C)}$
Integrated neutron flux E > 2.9 Mev	3.7 x 10^{16} n/cm ² (converted from aluminum to the effective sulfur threshold)
E < 0.48 ev	3.1 x 10^{15} n/cm^2

V. TEST PROCEDURE AND RESULTS

5.1 Wear-Life Measurements

Standard procedures for using the Hohman A-6 tester were followed. A load of 110 lb per rub shoe (the minimum load attainable with the standard weights) was applied after the machine was started. A sliding speed of 128 ft/min (355 rpm) was maintained for each measurement. The elevated temperature runs were started about 100°F below the desired test temperature to compensate for frictional heating.

The machine was so adjusted that it would cut off automatically when the coefficient of friction reached 0.4.

5.2 Data Analysis and Results

As wear-life data were accumulated, averages and standard deviations were calculated for the performance of each lubricant under each set of conditions. Wear-life averages for the irradiated specimens were compared to control averages by the use of a standard "t" test. The following relationship was used for these analyses:

Difference =
$$\overline{X}_1 - \overline{X}_2 + t_\alpha \left[\left(\frac{1}{n_1} + \frac{1}{n_2} \right) - \overline{s}^2 \right]^{1/2}$$

where \overline{X}_1 , \overline{X}_2 = average wear-life for the two conditions under comparison,

- n1, n2 = the number of specimens under each condition,
 - \overline{s} = average standard deviation

$$= \left[\frac{(n_1 - 1) s_1^2 + (n_2 - 1) s_2^2}{n_1 + n_2 - 2} \right]^{1/2}$$
 and

 $t_{\alpha} = standard tabular value of t for n1 + n2 - 2 degrees of freedom.$

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The results of these analyses, given in Tables I and II, indicate that irradiation had no detectable effect on the wear-life of Lubricant A at the 90% confidence level. However, detectable changes were noted at 900°F on Lubricant E and at 80° and 900°F on Lubricant C.

Coefficient-of-friction charact_ristics of all the lubricants were essentially unchanged by irradiation. Typical friction coefficients (μ) of the films were as follows:

Lubricant A	Avg Coefficient of Friction
_@ 850 [°] - 1000 [°] F	0.20
Lubricant B	
@ 80 ⁰ F @ 900 ⁰ F @ 1150 ⁰ F	0.36 0.26 0.20
Lubricant C	
@ 80° - 170°F @ 600°F @ 900°F	0.07 0.13 Initially 0.07 with fairly rapid increase to 0.4

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WEAR-LIFE DATA FOR SOLID-FILM LUBRICANT A AND R

(Wear Life in Revolutions)

	1150 ⁰ F	d Controls Irradiated	21,003 46,394 28,854 16,427 ** 35,514 37,692 33,989 12,499 ** 29,265 18,033 **	Avg Avg 27,378 28,507) Difference (90% C.L.) =1,129 <u>+</u> 1,211.3	**Test specimen started slipping on shaft. Un- able to tighten - test discontinued.
ANT B	ч ^о 006 -	Irradiated	1,508 492 711 2,995 6,406	Avg 4,418	Difference 90% C.L.) =14,779 <u>+</u> 722.4	
LUBRICANT B	850° -	Controls	13,880 14,395 31,256 25,679 20,466	Avg 19,197	Differer =14,779	
	F**	Irradiated	929 9,944 9,944 10,279 3,885	Avg 5,867	se (90% C.L. 514.4	were started t temperature). Maxlumum ue to nal heat d 6250F.
	800	Controls	16,599 14,165 7,938 7,938 7,999	Avg 8,637	Difference (=2,770 <u>+</u> 514	*Tests wer at room te (~ 80°F). temp. due frictional averaged 6
ANT A	1000 ⁰ F	Irradiated	4,241 8,459 27,526 28,220 12,527 18,509 18,509	Avg 15,156	ce (90% C.L.) 843.1	
LUBRICANT	850° -	Controls	8,459 14,263 20,893 10,934	Avg 13,637	Difference (=1,519 <u>+</u> 843	

Test Conditions:

Hohman A-6 machine Load: 110 1b per shoe Speed: 355 rpm (128 ft/min) Substrate: Timken T-54148 steel for Lubricant A, T-1 tool steel for Lubricant B. Rub Shoes: Rex AAA

TABLE II

WEAR-LIFE DATA FOR SOLID-FILM LUBRICANT C

(Wear Life in Revolutions)

		LUBRICANT C	T C		
80° - 1	170 ⁰ F	600 ⁰ F	E	850° - 5	900 ⁰ F
Controls	Irradiated	Controls	Irradiated	Controls	Irradiated
60,987	59,364	16,280	11,317	3,954	1,217
80,801	49,840	344,445	14,556	4,068	1,950
80,311	186,700	16,307	17,462	3,914	1,490
28,742	51,253	18,145	14,054	2,817	696
57,931	120,977	18,497	17,270	3,790	762
47,345	106,700	18,066	5,210	2,656	956
Avg 59,353	Avg 95,806	Avg 16,957	Avg 13,312	Avg 3,533	Avg 1,224
Difference (90% C.L.) = 36,453 <u>+</u> 2600.8	00€ C.L.) = 00.8	D1fference 3645 <u>+</u> 329	Difference (90% C.L.) = 3645 <u>+</u> 329.6	Difference 2309 <u>+</u> 56	Difference (90% C.L.) = 2309 <u>+</u> 56.2

Test Conditions

Hohman A-6 machine Load: 110 1b per shoe Speed: 355 rpm (128 ft/min) Substraje: Timken T-54148 steel Rub Shoes: Rex AAA

VI. CONCLUSIONS

6.1 Lubricant A: PbS + MoS₂ + B₂O₃

Based on the data obtained in this experiment, exposure to reactor radiation had no significant effect on the wear-life of this lubricant. The same conclusion was drawn in an earlier experiment with this film on an Inconel X substrate (Ref. 4).

6.2 Lubricant B: GD/FW Dynalube

Lubricant B was tested over a wide range of temperatures and a sufficient quantity of data obtained to indicate good lubricating properties under the conditions imposed. However, there was a significant radiation induced decrease in wear-life at the intermediate test temperature of 900°F. Nevertheless, data from the 1150°F tests indicate that the lubricant performs exceptionally well at this elevated temperature. In fact, if the three cases in which the test race began slipping on the shaft were disregarded, the average of the three irradiated races which ran to failure would be 41,360 revolutions as compared to the average of 27,378 for the controls. However, this can be taken only as indication of possible improvement in wear life due to irradiation.

6.3 Lubricant C: Almasol SFD-560

Lubricating properties of Lubricant C were greatly improved by irradiation at the lowest test temperature. However, as can be expected with most resin-bonded solid-film lubricants, its lubricating properties were lacking at 900°F. Furthermore, a significant reduction in wear life was caused by irradiation at this temperature.

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