DYNAMIC FRICTION AND WEAR OF A SOLID FILM LUBRICANT DURING RADIATION EXPOSURE IN A NUCLEAR REACTOR

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The effect of nuclear reactor radiation on the performance of a solid-film lubricant was studied. The film consisted of molybdenum disulfide and graphite in a sodium silicate binder. Radiation levels of fast neutrons (E \geq 1 \text{ MeV}) were fluxes up to \(3.5 \times 10^{12} \text{n/cm}^2\text{-sec}\) (intensity) and fluences up to \(2 \times 10^{18} \text{n/cm}^2\) (total exposure). Coating wear lives were much shorter and friction coefficients higher in a high flux region of the reactor than in a low flux region. The amount of total exposure did not affect lubrication behavior as severely as the radiation intensity during sliding.
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SUMMARY

Coating wear life, friction, and wear were measured on a solid film lubricant composed of molybdenum disulfide and graphite in a sodium silicate binder. These data were obtained during irradiation in a nuclear reactor at various radiation levels and compared with data obtained with no radiation. Results indicate that the amount of total exposure (fluence) did not affect lubrication behavior as severely as the radiation intensity (flux) during sliding.

Coating wear lives were much shorter during exposure to high flux levels. Flux levels of fast \( E \geq 1 \text{ MeV} \) neutrons were \( 3.5 \times 10^{12} \text{ n/cm}^2\text{-sec} \) at high level and \( 4 \times 10^{11} \text{ n/cm}^2\text{-sec} \) at the low level. However, if specimens were first exposed to the high flux and then tested during exposure to the low flux level, wear lives were within the range obtained with unirradiated coatings. This was true even after fluence levels up to \( 2 \times 10^{18} \text{ n/cm}^2 \).

Friction coefficients during exposure to low levels of flux were also within the range obtained with unirradiated coatings. But as the flux level was increased, the friction coefficient increased. Specimen wear was generally lower during radiation exposure than during tests with no radiation exposure. One exception was observed: Wear was higher during high flux exposure after a high fluence level had been attained.

These data were obtained using a hemispherically tipped rider in sliding contact with a coated (lubricated) disk. Disk and rider were 440-C stainless steel (hardened to \( R_C 60 \)). Experiment conditions in a dry air atmosphere were disk temperature, \( 150^\circ \text{C} \); normal load, 1 kilogram; and disk rotation, 5000 rpm.

INTRODUCTION

Low friction and wear of sliding components are necessary for the efficient and reliable operation of power systems. This is especially important with nuclear power
because radioactive machinery is difficult to maintain. Furthermore, nuclear radiation effects could change the friction and wear properties of the lubricant.

The purpose of this experimental program was to determine how lubricating properties change due to nuclear radiation exposure. To do this, a unique capability was developed whereby fundamental sliding friction studies could be made during radiation exposure near a nuclear reactor. These dynamic studies were planned so that changes in lubrication due to radiation intensity (flux) could be detected in addition to changes due to total radiation exposure (fluence).

These studies involved the measurement of the friction coefficient between a hemispherically tipped rider in sliding contact with a coated (lubricated) disk and, by increases in friction, determining the time until coating failure. Specimen wear, measured after sliding, indicated the extent of coating failure.

The coating was composed of molybdenum disulfide and graphite in a sodium silicate binder. This coating is one of the better solid-film lubricants when considering its combination of low friction, long wear life, availability, and basic radiation stability. Because of its inorganic composition, this coating was expected to be affected more by the displacement effects due to fast neutron radiation than by the ionization effects due to gamma radiation.

When atoms are displaced from their normal lattice positions, vacancy-interstitial pairs called Frenkl defects are formed (ref. 1). The mobility of these defects depends on the material and its temperature. At normal temperatures, these defects can aggregate, forming voids and dislocation loops, or they can recombine causing annealing. Defect annealing could explain why no significant changes in lubricating properties of solids have been observed in postirradiation studies (see BACKGROUND).

Radiation produced defects are usually detrimental but can be beneficial in solid-film lubrication. The important properties of lubricating films are low shear strength and good adhesion. Shear strength of the film could be either decreased by lattice expansion or increased by dislocation pinning. Substrate bond strength and adhesive film transfer could be either increased due to increased chemical activity or decreased due to the enhancement of diffusion, decomposition, and oxidation.

BACKGROUND

The units of radiation measurement are described in table I (ref. 2). In general, displacement effects are reported in terms of fast neutron fluence and ionization effects in terms of gamma dose. Fluence describes the amount of incident radiation, and dose describes the amount of energy absorbed from the radiation. The relation between incident radiation and absorbed energy depends on the absorption characteristics of the
TABLE I. - DEFINITION OF RADIATION TERMS

<table>
<thead>
<tr>
<th>Term</th>
<th>Units and definition</th>
</tr>
</thead>
<tbody>
<tr>
<td>Flux</td>
<td>( \text{n/cm}^2 \cdot \text{sec} ); number of particles (or energy) incident per unit area per unit time</td>
</tr>
<tr>
<td>Fluence</td>
<td>( \text{n/cm}^2 ); time-integrated flux</td>
</tr>
<tr>
<td>Dose rate</td>
<td>( \text{rad}^9/\text{sec} ); energy absorbed per unit mass (of exposed material) per unit time</td>
</tr>
<tr>
<td>Dose</td>
<td>( \text{rad} ); time-integrated dose rate</td>
</tr>
</tbody>
</table>

\[ a \text{ rad} = 10^{-5} \text{ J/g} \ (100 \text{ ergs/g}). \]

irradiated material (equal energy) and on the sensitivity of its properties (equal damage).

Property Changes

Radiation stability limits for representative materials are shown in table II (ref. 4). With organic compounds the primary carbon and hydrogen bonds are ruptured by radiation, and gas is evolved, mainly hydrogen with some methane, ethane, etc. The free radicals formed usually cross-link causing increases in hardness and rigidity and decreases in compressibility and elongation.

TABLE II. - RADIATION STABILITY LIMITS OF MATERIALS

<table>
<thead>
<tr>
<th>Material</th>
<th>Gamma dose, ( \text{rad (carbon)} )</th>
<th>Neutron fluence, ( \text{n/cm}^2 ) (( \text{E} \geq 1 \text{ MeV} ))</th>
</tr>
</thead>
<tbody>
<tr>
<td>Elastomers</td>
<td>( 10^7 )</td>
<td>( a_{10^{15}} )</td>
</tr>
<tr>
<td>Organic fluids</td>
<td>( 10^8 )</td>
<td>( a_{10^{16}} )</td>
</tr>
<tr>
<td>Plastics</td>
<td>( 10^9 )</td>
<td>( a_{10^{17}} )</td>
</tr>
<tr>
<td>Ceramics</td>
<td>( b_{10^8 - 10^{11}} )</td>
<td>( 10^{17} - 10^{20} )</td>
</tr>
<tr>
<td>Metals</td>
<td>( b_{10^9 - 10^{13}} )</td>
<td>( 10^{18} - 10^{22} )</td>
</tr>
</tbody>
</table>

\[ a \text{ Fluence equivalent based on equal damage.} \]
\[ b \text{ Dose equivalent based on equal energy.} \]
Oils and greases rapidly degrade at radiation doses above $10^8$ rads (C), with increasing viscosity, acidity and gas evolution (ref. 4). Only select aromatics, such as polyphenyl ethers can be used up to $10^{10}$ rads (C) (ref. 5). In a review of static irradiation studies, Cosgrove (ref. 6) stated that these data are only approximate. Neely (ref. 7) reported lubricant failure of an oil before it received 20 percent of the static exposure to cause viscosity changes. Fainman and others (ref. 8) reported some beneficial effects due to radiation when lubricants are dynamically tested. Bolt and Carroll (ref. 5) reported that a sodium soap - mineral oil grease provided successful lubrication of a ball bearing during irradiation even after exceeding the static exposure which solidified the grease.

Inorganic solids are, of course, more stable than organics; but dramatic changes can occur, such as the dimensional changes in graphite due to lattice expansion (ref. 9). Metals are most stable but become harder, stronger, and more brittle when irradiated (ref. 1). Possible mechanisms of these changes are dislocation pinning by simple defects or dislocation slip by defect clusters.

**Radiation Levels in Reactor Applications**

The severity of radiation exposure to sliding components in nuclear power systems depends on time, distance, and shielding. Power systems for aerospace applications must be compact and light, requiring minimal distance and shielding from the reactor radiation. Possible reactor applications for space vehicles are a nuclear rocket engine and a nuclear power supply. The radiation stability required for lubricants in the components of these systems is typical for most nuclear aerospace applications.

A nuclear rocket engine uses a reactor to heat liquid hydrogen and expand the gas through a nozzle to generate thrust. Operation is cyclic, and some sliding components see a high radiation intensity, about $10^{12}$ n/cm$^2$-sec $(E \geq 1$ MeV) for a total time of about 10 hours, yielding a low level of total exposure (fluence) in the range of $10^{16}$ n/cm$^2$ $(E \geq 1$ MeV).

Nuclear power supplies use a reactor as the heat source in either a liquid-metal Rankine cycle or a high temperature helium Brayton cycle and expand gas through a turbine to generate electricity. Operation is continuous, and most sliding components see a lower radiation intensity, about $10^{10}$ n/cm$^2$-sec $(E \geq 1$ MeV) for more than a year, yielding a high level of total exposure, up into the $10^{18}$ n/cm$^2$ $(E \geq 1$ MeV) range.

**Solid-Film Lubricants**

Solid-film lubricants were developed for sliding components that could not be con-
ventionally lubricated because of environmental or operating conditions (ref. 10). The effectiveness of solid-film lubricants depends on how long they provide lubrication (coating wear life) as well as on how much they reduce friction and wear of sliding surfaces. Longer wear lives have been obtained when the lubricant (low shear material) is mixed in the proper ratio with an appropriate binder. In an optimization study with sodium silicate as a binder, the coating composition giving longest life in a ball bearing was molybdenum disulfide (71 wt.%), graphite (7 wt.%), and sodium silicate (22 wt.%) (ref. 11).

Increases in friction, after static irradiation of solid lubricants, have been reported by McConnell (ref. 12) for self-lubricating compacts and by McDaniel (ref. 13) for bonded films. After exposure to $10^{10}$ rads and $10^{17}$ n/cm$^2$ ($E \geq 1$ MeV), coating wear lives of the silicate bonded mixture of molybdenum disulfide and graphite were longer than for unirradiated coatings (ref. 13).

The wear life of solid-lubricant coatings has been determined during gamma irradiation in a cobalt-60 source (ref. 12). In one case a 25-percent reduction in wear life occurred, and in another, a 25-percent increase. These changes were within the data scatter, however. No data were found for solid-film lubricant evaluation during reactor radiation exposure.

**APPARATUS**

A rider-on-disk apparatus was designed for use in a horizontal beam hole of a 60-megawatt (thermal) research reactor at the NASA Plum Brook Reactor Facility. The design of the experiment capsule was described in detail at the NASA Symposium on Irradiation Testing Technology (ref. 14).

Three of these capsules were used to obtain the data presented in this report. A perspective drawing of the capsule shows its three main parts (fig. 1). The outer tube is sealed from external cooling water by a front end cap. Utility connections are made on flanges on the back of the tube. The inner assembly contains the drive systems for disk rotation and rider loading. Location of these systems minimizes their radiation exposure. The friction module contains the required instrumentation for measuring temperatures and the frictional force between the rider and disk.

Two loading cables are attached to the friction module. The large arrows (fig. 1) show loading of the upper rider against the disk at a radius of 25.4 millimeters (outer track) by the lower cable. The lower rider is alternately loaded at a radius of 19.1 millimeters (inner track) by the upper cable.

Each cable is attached to a proving ring and a pneumatic loading piston. A linear variable differential transformer (LVDT) inside the proving ring measures the ring distortion, which is proportional to cable tension.
Figure 1. - Apparatus for dynamic friction measurements during radiation exposure in nuclear reactor.
The apparatus was cooled internally with dry air. The small arrows (fig. 1) show the path of air flow over the specimens and through the hollow shaft. The baffle plate prevented direct flow across the disk surface under study.

The rider holder (fig. 1) is held by two flexure pivots in a yoke, which is attached to the base plate by four leaf springs. Friction force causes a proportional displacement of the yoke arms, which is measured by two LVDT units. This redundancy increases the reliability of the friction force measurement. The LVDT coils are attached to the base plate and the LVDT cores are attached to each yoke arm.

Lubrication data on unirradiated coatings was obtained using an apparatus that was identical to the radiation apparatus from the ball bearing up to the friction module (fig. 1). Disk temperature was maintained by induction heating from a radiofrequency coil, located about 6 millimeters from the uncoated side of the disk. The disk, coil, and module were enclosed to maintain a controlled atmosphere.

PROCEDURE

Specimen Preparation

Disk specimens were 63 millimeters in diameter by either 2.5 or 7.6 millimeters thick. The thicker disks were used for irradiations at the lower levels of radiation intensity (flux). Rider specimens were 6.3 millimeters in diameter by 9.6 millimeters long, having a hemispherical tip with a 4.8-millimeter radius. Disk and riders were 440-C stainless steel, finish ground to less than 0.2 micrometer (centerline average), and heat treated to maximum hardness (about 60 Rockwell C). The disks were sandblasted to produce a surface roughness of 1.2 to 1.6 micrometers (cla). Specimens were cleaned with ethanol, moist levigated alumina, and then distilled water.

Coatings of the lubricant mixture in an aqueous carrier were sprayed onto one side of the disk to a thickness of about 25 micrometers (1 mil). The disk coating was cured for 2 hours at 80° C followed by 2 hours at 200° C. After curing, the coating thicknesses were 20±10 micrometers.

Two chromel-alumel thermocouples were tack-welded to the disk, 180° apart. The junction was located inside the disk rim (6 mm deep) about 1 millimeter from the coated surface.

Friction and Wear Measurement

Dynamic friction measurements were made during radiation exposure. After the
exposure time yielded the desired fluence level, the disk was rotated at 5000 rpm. Two consecutive friction experiments were then made: first at a 25.4-millimeter radius and then at a 19.1-millimeter radius. The resulting rider sliding speeds were 13.3 meters per second at the larger radius and 10 meters per second at the smaller radius. This speed difference was not significant in this study.

The 1-kilogram rider load was pneumatically applied by increasing the pressure differential across the loading piston. Friction force was continuously recorded. Total sliding time depended on coating wear life but was maintained for a minimum of 15 minutes.

Coating wear life was defined as the sliding time until coating failure. The failure criterion was a friction coefficient increase either to an average value of 0.10 or to an instantaneous value of 0.15. These friction increases were most likely due to increasing metallic contact through the coating.

Specimen wear was measured after radiation exposure. Because of induced radioactivity, all handling of the irradiated specimens was done remotely in a hot cell. Photographs were made at ×20 magnification of both the rider wear scar and the disk wear

![Figure 2. - Typical wear data from friction specimens used to evaluate solid-film lubricant coatings.](image-url)
track. Also, a surface profile trace of the wear track was made at ×20 horizontal and ×1000 vertical magnification.

Typical wear data are shown in figure 2. A calculated rider profile is shown at the same distorted view as the disk track profile trace, depth equals 50 times the width. This shows how the rider is somewhat supported by the coating on the edges of the wear track.

Rider wear volume was calculated from the rider wear scar diameter, measured on the photograph (fig. 2). Because of the different sliding speeds (13.3 and 10 m/sec), the sliding distance in the same time, say 15 minutes, was different (12 and 9 km). Therefore, rider wear data was reduced to rider wear volume per unit distance of sliding.

Also, after irradiation, the riders were weighed, and hardness measurements made on the disks and riders. Weight and hardness were compared with pre-irradiation measurements. No significant changes were observed.

Coating Irradiation

The irradiation program was planned for dynamic friction studies to be made at three levels of radiation intensity (flux) after three levels of total exposure (fluence). Since fast neutron radiation was of main interest in this study, it was chosen to characterize the desired exposure levels:

\[
\text{FLUX} = 0.3, 1, 3 \times 10^{12} \text{n/cm}^2\text{-sec (E \geq 1 MeV)}
\]

\[
\text{FLUENCE} = 0.1, 1, 10 \times 10^{17} \text{n/cm}^2 \text{ (E \geq 1 MeV)}
\]

Flux was controlled by varying the proximity of the friction apparatus to the reactor core. Fluence was controlled by varying the exposure time at the estimated flux level before starting the dynamic friction study. Typical irradiation times were from 2 hours to 3 days.

Dosimetry

The fluence levels of both fast and thermal neutrons were measured with dosimetry wires. These wires (0.8 mm diam, 12.7 mm long) were installed in the front end cap in four places, 90° apart. Cobalt, nickel, and stainless steel wires were used and replaced after each irradiation. The estimated uncertainty in the dosimetry calculations,
at the 95-percent confidence level, was ±25 percent for fast neutrons \((E \geq 1 \text{ MeV})\) and ±10 percent for thermal neutrons \((E \leq 0.025 \text{ eV})\).

Gamma dose rate was estimated from the internal heat generation rate. This rate was determined by temperature measurements in the front flange of the outer tube. An uncertainty of about ±50 percent was estimated in this gamma dose rate determination.

The complete radiation environment can be characterized by the fast neutron flux \((E \geq 1 \text{ MeV})\), fluence divided by time. This is shown by figure 3 where the radiation levels are given as a function of reactor condition and irradiation position. The normal reactor power cycle is 15 days, starting at 60 megawatts (thermal) power, with the last 10 days at 50 megawatts. Constant power is maintained by control rod changes, which produce an increase in radiation intensity inside the beam hole. Thus, the reported flux levels are average values.

Radiation exposure positions of about 3, 6, and 9 inches from the reactor core were used to obtain high, medium, and low levels of flux. The 3-inch position was with the friction apparatus fully inserted into the reactor beam hole.

The neutron energy spectrum in the reactor beam hole had been determined at two positions: 2 and 8 inches from the reactor core (fig. 4). Flux levels of fast neutrons defined by different energy ranges can be determined from the spectrum. Approximate

![Figure 3. Flux as function of irradiation position in NASA Plum Brook reactor during power cycle of 15 days.](image-url)
Temperature Control

During irradiation the friction apparatus was cooled by a closed, recirculating air loop. The disk temperature was maintained near 150° C by balancing the heat transferred to the air flow with the heat generated inside the disk due to the radiation energy absorption. Temperature control by a variable air flow was augmented by using two disk thicknesses. At low intensities, around 0.5 watt per gram, thick disks (7.6 mm) were used; at high intensities, around 1.5 watts per gram, thin disks (2.5 mm) were used.

Direct air flow across the coated surface of the disk was prevented by a baffle plate. Thus, the effects of flow variations were not considered to significantly affect the coating lubricating characteristics.

Due to a gradient in radiation intensity, a temperature gradient occurred on the disk surface. At high flux, the bottom of the disk was 10° C hotter and the top 10° C cooler than the middle of the disk. During disk rotation, this temperature gradient was assumed to disappear.

Environment Control

The cooling air loop contained a synthetic air mixture of 95 percent nitrogen and
5 percent oxygen with less than 40 ppm water vapor. The pressure at the disk location was maintained at 4 atmospheres (60 psia). Thus, the oxygen partial pressure was 0.2 atmosphere, the same as normal air at 1 atmosphere. Accelerated chemical oxidation reactions at higher partial pressures of oxygen could have masked any effects on lubrication due to radiation exposure.

An added benefit of using synthetic air was its lower argon content: less than 100 ppm compared with 10 000 ppm in normal air. Thus, gaseous radioactivity due to argon-41 was considerably reduced.

Unirradiated Coating Evaluation

The lubrication characteristics of unirradiated coatings were also determined, following the same procedures in specimen preparation and friction and wear measurement. Disk temperature was maintained at 150° C by induction heating during rotation. Temperature was measured with an infrared pyrometer focused on the rim of the disk. A dry air atmosphere at standard pressure was supplied from a bottle of compressed air (20 ppm of water vapor). After purging at a flow of a volume change per minute, flow was decreased to a volume change per hour, during sliding.

RESULTS AND DISCUSSION

Dynamic lubrication studies on bonded lubricant coatings were made during reactor radiation exposure. The coating was composed of molybdenum disulfide and graphite in a sodium silicate binder. Data were obtained on coating wear life, friction coefficient, and rider wear. These data indicated that the intensity of radiation exposure adversely affected lubricant performance only during dynamic testing.

Radiation intensity (flux) and total radiation exposure (fluence) were both designated by three arbitrary levels called low, medium, and high. Because of the basic radiation stability of the lubricant coating, these levels were characterized by fast neutrons with energies equal to or greater than 1 MeV. The symbols for these levels and the ranges that they designate are listed in table III.

<table>
<thead>
<tr>
<th>Radiation level</th>
<th>Flux, n/cm²·sec</th>
<th>Fluence, n/cm² (E ≥ 1 MeV)</th>
</tr>
</thead>
<tbody>
<tr>
<td>Low</td>
<td>2×10¹¹ - 4×10¹¹</td>
<td>2×10¹⁵ - 3×10¹⁵</td>
</tr>
<tr>
<td>Medium</td>
<td>9×10¹¹</td>
<td>6×10¹⁶ - 7.5×10¹⁶</td>
</tr>
<tr>
<td>High</td>
<td>3×10¹² - 3.5×10¹²</td>
<td>7.5×10¹⁷ - 2×10¹⁸</td>
</tr>
</tbody>
</table>

12
Coating Wear Life

Coating wear life data are shown in table IV for six combinations of the flux and fluence exposure levels. These data were compared with the wear lives obtained with unirradiated coatings (19±11 min). Coating wear life was reduced during high flux exposure. This reduction occurred at two fluence levels: medium fluence (high flux exposure for 8 hr) and high fluence (high flux exposure for 72 hr). Coating wear life was either unaffected or slightly increased at the other four irradiation levels.

A comparison of the wear life data obtained at high fluence (table IV) shows that radiation intensity (flux) during sliding had a significant effect. The five wear lives were

<table>
<thead>
<tr>
<th>Flux exposure level</th>
<th>Fluence exposure level</th>
<th>Coating wear life, min</th>
</tr>
</thead>
<tbody>
<tr>
<td>High</td>
<td>Low</td>
<td>60, 60, 70</td>
</tr>
<tr>
<td></td>
<td>Medium</td>
<td>40</td>
</tr>
<tr>
<td></td>
<td>High</td>
<td>35, 40, 17, 20</td>
</tr>
</tbody>
</table>

aLife = minutes of sliding time before failure.
bCoating life with no radiation exposure, 19±11 min.
cNo failure.

all determined after exposure to a high flux for 72 hours. But two wear lives were determined after repositioning the coating into a low flux level. The longer wear lives at low flux show that no significant changes in lubricant performance resulted from the prior high flux exposure.

This flux effect implies an interaction between the mechanical stresses of the sliding process and the defects produced by irradiation. At high flux the defect concentration depends on exposure time and the equilibrium between defect production rate and defect annealing rate. When the irradiated specimen is repositioned into a lower flux level,
the defect production rate decreases, and the defect concentration probably decreases, depending on time and the equilibrium process. Thus, the effect on wear life of the defects present during high flux exposure would be detected only by applying the mechanical stresses of sliding during high flux exposure.

**Friction Coefficient**

Sliding friction before coating failure was higher during high flux exposure than for other conditions. The average friction coefficient for the five high flux experiments (see table IV) was 0.13±0.02. For the other irradiations, the average friction coefficient was 0.07±0.03.

A comparison of the friction data obtained at medium fluence shows the effect of flux on friction as well as wear life (fig. 5). The average friction coefficient before coating failure was 0.06 for low flux, 0.09 for medium flux, and 0.12 for high flux. These data also imply an interaction of radiation-induced defects with the mechanical stresses existing at the lubricated sliding contact.

**Rider Wear**

An important function of a bonded lubricant coating is to prevent direct contact be-
tween sliding metal surfaces. The amount of wear to a rider sliding against a coated disk often depends on the amount of metallic contact that occurs through the coating. Thus, a good indicator of coating performance is rider wear rate. This is defined as rider wear volume per unit distance of sliding.

Data of table V show that coating performance was better during radiation exposure than when unirradiated. The only exception was during exposure to a high flux level at a high fluence level, where higher rider wear rates occurred.

<table>
<thead>
<tr>
<th>Radiation exposure level</th>
</tr>
</thead>
<tbody>
<tr>
<td>High flux - high fluence (3 riders)</td>
</tr>
<tr>
<td>All other irradiations (10 riders)</td>
</tr>
<tr>
<td>No irradiation (18 riders)</td>
</tr>
</tbody>
</table>

<table>
<thead>
<tr>
<th>Average rider wear rate, m³/m</th>
</tr>
</thead>
<tbody>
<tr>
<td>(29±9)×10⁻¹⁶</td>
</tr>
<tr>
<td>(3.5±3)×10⁻¹⁶</td>
</tr>
<tr>
<td>(16±14)×10⁻¹⁶</td>
</tr>
</tbody>
</table>

Wear data obtained during exposure to the high flux level are compared at two levels of fluence, medium and high (fig. 6). Both coatings were evaluated for 15 minutes of sliding on the 25.4-millimeter-radius wear track. The lower amount of wear at the high flux - medium fluence level indicates that the coating prevented significant metallic contact. Thus, the reduced coating wear lives at this level (table IV) were probably due to the increased friction at high flux (previously discussed).

Flux Effect at Low Fluence

Lubrication characteristics of a coating were determined first at the outer wear track (25.4-mm radius) during low flux exposure. Then, after readjusting the air flow and repositioning the disk, these characteristics were determined at the inner wear
Medium fluence, $0.17 \times 10^{17}$ n/cm² (E $\geq$ 1 MeV)

High fluence, $9.4 \times 10^{17}$ n/cm² (E $\geq$ 1 MeV)

Figure 6. - Effect of fluence on wear; flux, $3 \times 10^{12}$ n/cm² -sec (E $\geq$ 1 MeV); sliding duration, 15 minutes; rotative speed, 5000 rpm; load, 1 kilogram; temperature, 150°C, dry synthetic air. Coating was sodium silicate bonded molybdenum disulfide and graphite, on 440-C stainless steel.

**TABLE VI. - FLUX EFFECT AT LOW FLUENCE ON THE LUBRICATION CHARACTERISTICS OF SODIUM SILICATE BONDED MOLYBDENUM DISULFIDE AND GRAPHITE**

[Load, 1 kilogram; rotative speed, 5000 rpm; temperature, 150°C; dry synthetic air.]

<table>
<thead>
<tr>
<th>Condition of coating</th>
<th>Flux level</th>
<th>Wear life, min</th>
<th>Friction coefficient before failure</th>
<th>Rider wear rate, m³/m</th>
<th>Total sliding time, min</th>
</tr>
</thead>
<tbody>
<tr>
<td>Irradiated</td>
<td>Low&lt;sup&gt;a&lt;/sup&gt;</td>
<td>23</td>
<td>0.08</td>
<td>$1.2 \times 10^{16}$</td>
<td>60</td>
</tr>
<tr>
<td></td>
<td>High&lt;sup&gt;b&lt;/sup&gt;</td>
<td>28</td>
<td>0.08</td>
<td>1.7</td>
<td>60</td>
</tr>
<tr>
<td>Unirradiated</td>
<td>-----</td>
<td>19±11</td>
<td>0.07±0.03</td>
<td>$(16±14) \times 10^{16}$</td>
<td>30±15</td>
</tr>
</tbody>
</table>

<sup>a</sup>Low flux = $0.3 \times 10^{12}$ n/cm² -sec, low fluence = $0.8 \times 10^{16}$ n/cm².

<sup>b</sup>High flux = $3.7 \times 10^{12}$ n/cm² -sec, low fluence = $2.5 \times 10^{16}$ n/cm².
track (19.1-mm radius) during high flux exposure. The fluence level was low, although slightly different, for both flux levels.

The lubrication characteristics for low fluence exposure (table VI) are essentially the same at high flux as at low flux. In addition, when compared with unirradiated coating characteristics, wear lives and friction coefficients are unchanged. The only significant difference is the lower rider wear rates during irradiation, which has been discussed.

These results suggest that a certain time of exposure at a given flux level is required before changes occur in lubrication characteristics. In other words, there appears to be a fluence threshold before the radiation-produced defect concentration is sufficient to cause an interaction with the mechanical stresses of sliding.

**Effect of High Fluence at Low Flux.**

A coated disk was exposed to a high level of flux for 210 hours in order to obtain a high level of fluence ($2.1 \times 10^{18} \text{ n/cm}^2, E \geq 1 \text{ MeV}$). This was an intermittent exposure over a 10-week period. Coating evaluation was then made 6 months later during exposure to a low level of flux in which the disk temperature was $105^\circ \text{C}$. The following lubrication characteristics were obtained:

- Coating wear life, min: 35
- Friction coefficient (before failure): 0.07
- Rider wear rate, $m^3/m$: $2.8 \times 10^{-16}$

When compared with the data obtained at low fluence (table VI), these data show that high fluence levels alone cause no significant changes in lubricant performance.

**Unlubricated Specimen Friction and Wear**

The effect of irradiation on the sliding characteristics of the base metal was determined with uncoated disks with a surface roughness of 1.2 to 1.6 micrometers. The friction coefficient and rider wear rate obtained during irradiation are compared with those obtained with no irradiation (table VII). No significant differences are apparent. The low rider wear from irradiated coatings (previously discussed) was therefore probably a result of radiation effects on the lubricant coating and not a result of changes in the base metal wear rate.
TABLE VII. - FRICTION COEFFICIENT AND RIDER WEAR RATE FOR UNLUBRICATED SPECIMENS

[Rider and disk material, 440C stainless steel; load, 1 kilogram; rotative speed, 5000 rpm; temperature, 150° C.]

<table>
<thead>
<tr>
<th>Radiation exposure</th>
<th>Friction coefficient</th>
<th>Rider wear rate, m³/m</th>
<th>Total sliding time, min</th>
</tr>
</thead>
<tbody>
<tr>
<td>During irradiation a</td>
<td>0.23±0.02</td>
<td>200×10^{16}</td>
<td>10</td>
</tr>
<tr>
<td>Unirradiated</td>
<td>0.21±0.02</td>
<td>170</td>
<td>20</td>
</tr>
</tbody>
</table>

aExposed to a high level of flux (3.5×10^{12} n/cm²-sec, E ≥ 1 MeV) for 25 hr, resulting in a medium level of fluence (3.7×10^{17} n/cm², E ≥ 1 MeV).

Effect of Temperature

A reduction in coating wear life with increasing temperature is typical with solid-lubricant coatings. This effect is significant because, during some coating irradiations, the temperature of the disk was below the desired range, 150±20° C. The effects of temperature variation on coating wear life were therefore considered.

The lubrication characteristics were determined with unirradiated coatings at two additional temperatures, 25° C (unheated) and 300° C. These data (table VIII) show that increases in temperature caused increased friction coefficient, and increased rider wear rate, as well as decreased coating wear life.

All wear life data from unirradiated coatings are shown in figure 7 as a function of disk coating temperature. At 150±20° C, the coating wear lives ranged from 8 to 30 minutes, with an average of 19 minutes. A line was drawn through the average wear lives at 25°, 150°, and 300° C. The slope of this line shows that coating wear life is reduced by 50 percent for every 85° C increase in temperature.

Wear life data from irradiated coatings are shown in figure 8 as a function of disk temperature. The dashed line represents the average wear life from unirradiated coatings. The reduction in coating wear life can not be accounted for by a temperature effect.
TABLE VIII. - EFFECT OF TEMPERATURE ON UNIRRADIATED COATINGS

[Load, 1 kilogram; rotative speed, 5000 rpm; dry synthetic air; coating, sodium silicate bonded molybdenum disulfide and graphite.]

<table>
<thead>
<tr>
<th>Coating temperature, °C</th>
<th>Wear life, min</th>
<th>Friction coefficient</th>
<th>Rider wear rate, m^3/m</th>
<th>Total sliding time, min</th>
</tr>
</thead>
<tbody>
<tr>
<td>25</td>
<td>56</td>
<td>0.04</td>
<td>3×10^{-16}</td>
<td>70</td>
</tr>
<tr>
<td></td>
<td>47</td>
<td>0.04</td>
<td>5</td>
<td>55</td>
</tr>
<tr>
<td>300</td>
<td>5</td>
<td>0.16</td>
<td>190×10^{-16}</td>
<td>8</td>
</tr>
<tr>
<td></td>
<td>6</td>
<td>0.10</td>
<td>110</td>
<td>9</td>
</tr>
</tbody>
</table>

Figure 7. - Temperature effect on unirradiated coating wear life. Coating, sodium silicate bonded molybdenum disulfide and graphite; disk and rider material, 440-C stainless steel; load, 1 kilogram; rotative speed, 5000 rpm; dry air.

Figure 8. - Temperature effect on irradiated coating wear life. Coating, sodium silicate bonded molybdenum disulfide and graphite; rider and disk material, 440-C stainless steel; load, 1 kilogram; rotative speed, 5000 rpm; dry synthetic air.
Relative Effects on Flux and Fluence

Flux level, the radiation intensity during sliding, was more significant than fluence level in causing changes in coating wear life. This is seen when the wear life data are normalized and evaluated in two ways: first as a function of flux and second as a function of fluence.

Normalized wear life was calculated by dividing the measured wear life by the average wear life (unirradiated) at the measured temperature (figs. 7 and 8). The range for normalized wear life obtained with unirradiated coatings was 1±0.58. This was based on the range of coating wear lives at 150° C, that is, 19±11 minutes.

Normalized coating wear life is shown in figure 9 as a function of fast neutron flux in a log-log plot. Apparently, wear life was severely reduced at high flux (above \(3 \times 10^{12}\) n/cm\(^2\)-sec, \(E > 1\) MeV) and was slightly increased at low flux (below \(3 \times 10^{11}\) n/cm\(^2\)-sec, \(E \geq 1\) MeV). These data indicate that doubling the flux level reduced the coating wear life by 50 percent.

![Figure 9. Effect of flux on coating wear life. Coating, sodium silicate bonded molybdenum disulfide and graphite; load, 1 kilogram; rotative speed, 5000 rpm; temperature, 150° C, dry synthetic air.](image-url)
Normalized coating wear life is shown against fast neutron fluence in a log-log plot (fig. 10). Two lines are required to represent the data: The solid line shows that, during high flux exposure, coating wear life was reduced after the exposure level exceeded a fluence threshold. The dashed line shows that, during medium and low flux exposure, an increase in fluence by a factor of a thousand was required to reduce wear life by 50 percent. It also shows that a fluence level above $10^{19} \text{ n/cm}^2 (E \geq 1 \text{ MeV})$ would be required to cause coating wear lives to be reduced to below that obtained for unirradiated coatings.

![Figure 10 - Effect of fluence on coating wear life. Coating, sodium silicate bonded molybdenum disulfide and graphite; load, 1 kilogram; rotative speed, 5000 rpm; temperature, 150° C; dry synthetic air.](image)

**SUMMARY OF RESULTS**

Dynamic lubrication studies on bonded lubricant coatings were made during reactor radiation exposure. The coating was composed of molybdenum disulfide and graphite in a sodium silicate binder. Data were obtained on coating wear life, friction coefficient, and rider wear. These data were obtained at 5000 rpm and a 1-kilogram load at 150° C in dry air. The results were

1. Coating wear life was changed by the intensity of radiation exposure during sliding. Wear lives were severely reduced at high flux and slightly increased at low flux, when compared to unirradiated coatings.

2. Apparently, the exposure time had to exceed a certain value (fluence threshold) before coating wear life was reduced at high flux.
3. The total amount of radiation exposure (fluence) did not affect lubrication behavior so severely as the radiation intensity (flux) during sliding.
4. Friction coefficients tended to increase with increasing levels of flux.
5. Rider wear during exposure was less than rider wear against unirradiated coatings. The only exception was during high flux exposure at the high fluence level, where more rider wear occurred.
6. These results suggest an interaction between the mechanical stresses of sliding and the defects produced by radiation. Thus, the effect of radiation on the lubrication characteristics of bonded coatings can adequately be determined only during radiation exposure.

Lewis Research Center,
National Aeronautics and Space Administration,
Cleveland, Ohio, June 7, 1972,
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— National Aeronautics and Space Act of 1958

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