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Oxidation and Frictional Performance of Solid Lubricants Used in Weapon Stronglinks*

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Abstract

The oxidation and performance of the solid film lubricant used in a majority of the surety devices in the enduring stockpile have been investigated. Oxidation of this lubricant in air at 150°C produces a significant increase in the molybdenum oxide to sulfide ratio, indicative of degradation of the primary lubricating constituent of the composite lubricant. Oxidation is more extensive on samples that were burnished such that the substrate is exposed over a fraction of the surface, relative to those which were only lightly burnished. Friction results indicate that oxidation in air did not increase the initial or steady-state friction coefficient for lightly burnished surfaces. However, surfaces burnished to expose substrate material experienced a significant increase in both initial and steady-state friction. Oxidation of lubricated parts retrieved from "aged" stronglinks has also been demonstrated.

Introduction

Lifetime extension of nuclear weapons in the enduring stockpile may result in materials and components remaining in service more than 30 years *longer* than originally intended. Most surety devices employ solid lubricants to mitigate friction and wear at sliding, impact, and rolling contact surfaces. Changes in lubricant performance during long term storage can have adverse effects on the operation of electromechanical mechanisms. Proper operation of these devices is frequently dependent on precise timing of mechanical motion. This is particularly true of the MC2969 stronglink, which is present in the B61, W78, W80, B83 and W84. These systems represent more than half of the enduring stockpile.

The primary factors that determine the friction coefficient and wear rate of a solid lubricant are the amount of lubricant present, its chemical state, and the contact parameters. Gas sampling of stronglinks returned from the stockpile has shown that these devices contain some oxygen and/or water vapor, even after being backfilled with inert gases and sealed during production. The solid lubricant used most extensively in weapon electromechanical assemblies contains MoS_2 particles as the primary functional constituent. The dynamic steady-state friction coefficient of MoS_2 is increased by the presence of oxygen and water vapor. This material is also known to oxidize after long term *static* exposure to oxygen and water vapor, causing an increase in the friction coefficient and wear rate. While oxidation of crystalline MoS_2 and sputtered thin films has been extensively studied in a range of environments [1-4], the oxidation kinetics of MoS_2 in a resin-bonded composite, and the effect of the degree of oxidation on tribological performance, are not known.

The goal of this work is to develop tools for predicting the performance of solid lubricated contacts in stronglinks after long-term exposure to conditions in the stockpile. As a first step, we present the results of our initial investigation of the susceptibility to oxidation of MoS_2 particles in a resin matrix, and the concomitant effects on lubricant performance. We also present initial surface composition information on lubricated components returned from aged stronglinks.

Experimental Procedure

Disks of 15-5 PH stainless steel (H900 heat treatment) were fabricated, having 25 mm diameter and 1.5 mm thickness. The samples were lapped to insure that both surfaces were parallel. Samples were then wet blasted with an alumina slurry to produce a uniform matte finish for adhesion of the lubricant. Coupons were ultrasonically cleaned in alcohol followed by rinsing in deionized water, and then blown dry in nitrogen. The lubricant was manufactured at Allied Signal and consisted of a heat-curing resin matrix containing particles of MoS_2 and graphite. This material is identical to that used during the past 20 years in stronglink production, but was formulated in-house to improve quality control, and because the commercial formulation is no longer available due to environmental safety and health concerns. The Allied Signal formulation has been accepted for weapon use and meets or exceeds the performance of the commercially-supplied formulation. The lubricant was sprayed on the stainless steel coupons to a thickness of approximately 25 μm using an automatic spray device to insure uniform coverage and reproducibility. The lubricant was cured in an oven. Two burnishing techniques were performed on the cured lubricant, to duplicate old as well as current stronglink lubricant application specifications. A light burnish was performed using an acid solder flux brush, which removes only particles that do not adhere well to the surface. This process represents current lubrication practice, and serves to expose solid lubricant particles at the surface. A more aggressive ("medium") burnish typical of earlier production practice uses

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a wire wheel spun at 1750 RPM, and results in significant exposure of the underlying steel at the surface. In both cases, the surface can be processed to a well controlled end point, where the surface exhibits a uniform color and reflectivity. Coupons were kept in desiccated containers at all times when not in analysis or oxidation exposures. The coupons were exposed for different times to elevated temperatures in laboratory air.

Surface chemical analysis was performed on as-burnished and oxidized coupons using x-ray photoelectron spectroscopy (XPS). Survey scans were used to identify the atomic species present, and detailed scans for each element were used to determine chemical state and atomic concentration. In particular, details of the Mo3p, O1s, and S2p peaks were used to assess the oxidation state of the MoS_2 particles in the coating. Parts returned from two MC2969 stronglink escapements were also analyzed for evidence of MoS_2 oxidation. The units were opened and disassembled on a clean bench. The parts were immediately packaged with desiccant to minimize changes in the lubricant coating due to water vapor and atmospheric contaminants. XPS analysis of "aged" lubricant surfaces was similar to that performed for coupons.

Performance analysis was accomplished on coupons using pin-on-disk sliding friction tests in a dry (<1 ppm H_2O) nitrogen environment containing less than 65 ppm O_2. This environment is similar to that in which the weapon components were designed to operate, and minimizes effects of dynamic oxidation of the lubricant during sliding so that transients associated with an oxidized surface layer could be examined. Friction tests were conducted at a peak Hertzian contact pressure of 1 GPa and an interfacial velocity of 3-5 cm/sec. The test duration was 200 cycles in all cases, which was found to be long enough for steady-state sliding conditions to develop.

Results & Discussion

Results of surface chemical analysis of burnished and oxidized samples is shown in Figure 1. The figure shows the ratio of molybdenum oxide to molybdenum sulfide present as a function of lubricant condition. This ratio was chosen to evaluate the oxidative degradation of the lubricant because it indicates oxidation of the primary lubricating constituent of the composite coating. The figure shows similar amounts of oxidation for both burnishing conditions, as well as the lightly burnished sample exposed to 75°C deionized water. However, samples exposed to 150°C air experienced extensive oxidation compared to the as-burnished surfaces. More work is necessary to determine the oxidation mechanism for MoS_2 in an organic resin matrix, but these results demonstrate that MoS_2 particles in the composite are susceptible to oxidation, at relatively modest temperatures, when exposed to air.

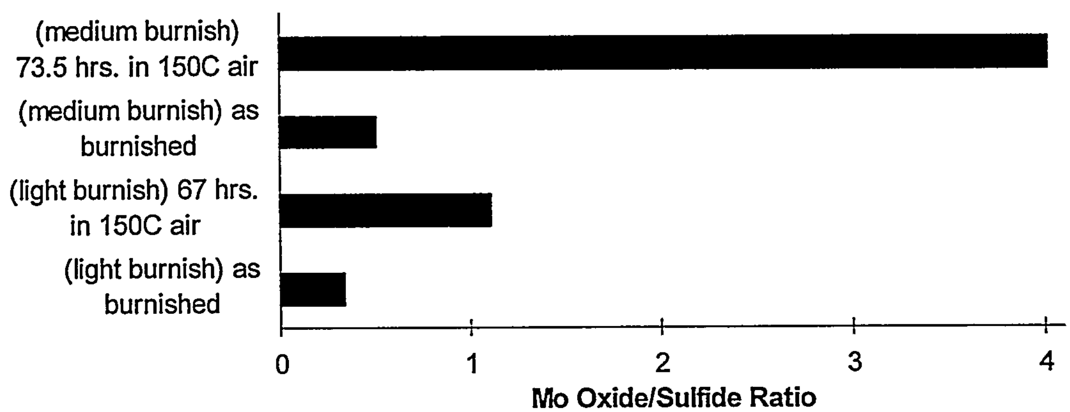

Figure 1. Molybdenum oxide to sulfide ratio for as-burnished and oxidized coupons.

The results of pin-on-disk friction tests on burnished and oxidized surfaces are shown in Figure 2. Initial and steady-state friction coefficient are shown for as-burnished coupons and coupons exposed to 150°C air for 48 hours. Lightly burnished surfaces experienced no degradation in friction after oxidation under these conditions. In fact, initial and steady-state friction decreased slightly after oxidation. This may be due to changes in the amount or composition of organic constituents on the lubricant surface. This phenomenon will be the subject of further research. However, exposure to these conditions produced a dramatic change in the friction of samples that were more heavily burnished.

These surfaces exhibited a significant increase in initial and steady-state sliding friction. This result is consistent with expectations based on experience with sputtered films, and the fact that the MoS_2 particles are probably highly defected from the burnishing process, providing ample sites and diffusion pathways for oxygen and water vapor. The exposed metallic constituents from the substrate may also catalyze oxidation of MoS_2. This will be investigated in future work.

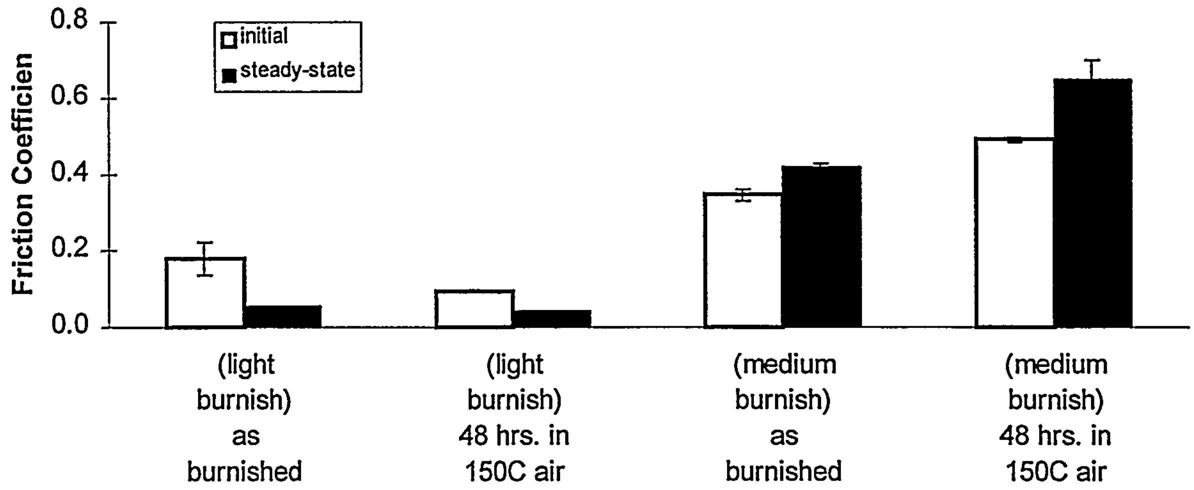

Figure 2. Friction coefficient of as-burnished coupons and those exposed for 48 hours to 150°C air.

Surface chemistry measurements on parts retrieved from "aged" stronglinks showed evidence of lubricant oxidation. In each case, a gear (part # 345895) from the escapement mechanism was analyzed. One stronglink was from an accelerated aging unit (AAU), which was believed to be hermetically sealed until the time at which the parts were retrieved (about 1 week prior to analysis). The other unit was D-tested 10 years ago. The D-test sequence is known to result in loss of hermeticity at ceramic to metal seals. Gas composition within the D-tested unit revealed atmospheric levels of oxygen and water vapor. The molybdenum oxide to sulfide ratios were 0.83 for the D-test unit, and 0.34 for the AAU. The results from the AAU unit are consistent with the composition of as-burnished coupon surfaces. The data from the D-test unit demonstrates that oxidation of lubricant inside sealed mechanisms is possible.

Conclusions

The oxidative stability of composite coatings used to lubricate electromechanical devices has received little attention. The present work demonstrates that the lubricating phase of these coatings can be oxidized at relatively modest temperatures in air. Burnishing conditions which expose substrate material make the lubricant susceptible to extensive oxidation. Oxidation of burnished surfaces with exposed substrate material produces a dramatic change in the initial and steady-state friction coefficients, compared with as-burnished surfaces. Although the temperatures used in this work exceed those expected in a typical stockpile-to-target sequence (STS), the exposure duration is much shorter than that in the stockpile. In addition, processing temperatures in excess of STS limits are frequently employed during production. The reactions observed here are therefore possible. Oxidation of lubricant from sealed stronglinks has also been demonstrated. This result suggests that lubricant oxidation under STS conditions is feasible. No adverse effects on device performance due to lubricant oxidation have been observed. The impact of lubricant oxidation on the operation of electromechanical mechanisms beyond their intended design life is, however, unknown.

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