Aging Model for Solid Lubricants Used in Weapon Stronglinks: Tribological Performance and Hardware Review

Michael T. Dugger*, Diane E. Peebles*, Elizabeth H. Sorroche*, Kenneth S. Varga* and Robert N. Hemley*

* Sandia National Laboratories, P. O. Box 5800, Albuquerque, NM 87185-0340
+ Allied Signal, Federal Manufacturing & Technology, P. O. Box 419159, Kansas City, MO 64141-6159

Abstract
The solid lubricant used most extensively in stronglinks throughout the enduring stockpile contains MoS₂, which is known to react with oxygen and water vapor resulting in a change in the material's friction and wear behavior. We have examined the frictional behavior of this lubricant as a function of oxidation, in support of efforts to quantify the impact of changes in the material on the dynamic behavior of the MC2969 stronglink. Our results show that the friction response of oxidized lubricant is strongly influenced by the amount of burnishing performed on the lubricant after deposition. Low levels of burnish leave a thick film, of which only the near surface degrades during oxidation. Rapid wear of the oxidized material leaves a surface whose properties are the same as non-oxidized material. Higher levels of burnish leave a thinner film of lubricant such that the entire film may be oxidized. The friction coefficient on this surface reaches a steady state value greater than that of non-oxidized material. In addition to these fundamental differences in steady state behavior, we have shown that the initial friction coefficient on oxidized surfaces is related to the amount of sulfide converted to sulfate, regardless of the oxidation conditions used. Measurements on parts returned from the stockpile show that the friction behavior of aged hardware is consistent with the behavior observed on controlled substrates containing thin lubricant films.

Introduction
Prediction of age-related changes in weapon component performance requires an understanding of materials aging mechanisms and the associated kinetics, as well as an understanding of the impact of the material degradation on the overall performance of the component. Changes in the friction performance of the solid lubricant used in stronglinks can affect the reliability of the devices. Stronglinks contain a mechanical timing circuit known as an escapement, which responds to application of the correct sequence of input signals by operating within a specified time window to move a block of electrical contacts. Changes in the friction between parts can change the operating time of the escapement, and hence cause the stronglink to fail to discriminate between input signals. Although lubricated parts may still move freely after significant oxidation, changes in the friction coefficient can cause failure of the device. Since stronglinks are designed to fail safe, failure of this device renders the entire weapon system inoperative.

The MC2969 stronglink is present in more than half of the weapons in the enduring stockpile. Our goal is to develop a model for the performance of solid lubricants as a function of aging conditions, and integrate this materials model into a performance model of the MC2969 in order to assess the impact of stockpile life extension on its reliability. In support of this modeling effort, we have completed a series of experiments designed to understand the friction behavior of the solid film lubricant used on the MC2969 escapement.

The solid lubricant used throughout the MC2969 escapement, as well as other stronglink applications, consists of a resin matrix with particles of graphite and MoS₂. The dynamic friction coefficient of MoS₂ is increased by oxygen and water vapor in the environment, and films of MoS₂ are known to undergo surface oxidation that causes an increase in the initial friction coefficient, even when sliding takes place in an inert environment or vacuum. This surface oxidation may be produced by long term static exposure to O₂ or H₂O vapor, or by much shorter duration exposures to atomic oxygen. While oxidation of crystalline MoS₂ and sputtered thin films has been extensively studied in a range of environments, the oxidation kinetics of MoS₂ in a resin-bonded composite, and the effect of oxidation of the composite film on tribological performance, are not known. The subject of this work is the dependence of the friction coefficient of resin-bonded solid lubricants on the degree of oxidation of the films, the type of substrate on which the lubricant was deposited, and the environments in which oxidation took place.

Experimental Procedure
Disks of 15-5PH stainless steel (H900 heat treatment) and Cu-2%Be alloy (hard) 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 MoS₂ and...
DISCLAIMER

This report was prepared as an account of work sponsored by an agency of the United States Government. Neither the United States Government nor any agency thereof, nor any of their employees, make any warranty, express or implied, or assumes any legal liability or responsibility for the accuracy, completeness, or usefulness of any information, apparatus, product, or process disclosed, or represents that its use would not infringe privately owned rights. Reference herein to any specific commercial product, process, or service by trade name, trademark, manufacturer, or otherwise does not necessarily constitute or imply its endorsement, recommendation, or favoring by the United States Government or any agency thereof. The views and opinions of authors expressed herein do not necessarily state or reflect those of the United States Government or any agency thereof.
graphite. This material is identical to that used during the past 20 years in stronglink production. It was formulated inhouse to improve quality control, and because the commercial formulation is no longer available due to environmental safety and health concerns. The lubricant was sprayed on the coupons to a thickness of ~25 µm using an automatic spray device. The lubricant was allowed to air dry, then oven cured at 191 to 204°C, for 75 to 90 minutes. 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 a fiber 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 a stainless steel wire rotary brush at 1750 RPM, and results in significant exposure of the underlying substrate. 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 oxidized at temperatures of 60 to 200°C, in dry (3.8 ppm H₂O) air or air containing 3.7 volume percent water. After burnishing and oxidation exposures, the sample coatings were analyzed by x-ray photoelectron spectroscopy (XPS) to determine the elemental composition of the near surface layer and the oxidation state of the chemical species present.

Pin-on-disk friction experiments were performed in laboratory air and pure nitrogen environments. The mating surface in all cases was a ball bearing of hardened 440C stainless steel, either 3.18 or 1.59 mm in diameter. Applied forces were adjusted to give the desired peak Hertzian contact stress, which was in the range of 0.4 to 1.6 GPa for these experiments. Sliding speed was 2±1 cm/s, and test duration was typically 1000 disk revolutions.

Hardware was also analyzed from dismantled MC2969 stronglink escapement assemblies to determine the performance characteristics of lubricants present in the stockpile. After electrical and functional tests, the internal atmosphere of the stronglinks was determined by residual gas analysis (RGA), using small volumes at controlled pressure to minimize effects due to water adsorption on surfaces of the RGA analysis chamber. This method was evaluated on MC2969 units that were modified so that the moisture could be measured with a chilled mirror hygrometer as well as the mass spectrometer. The RGA analysis of moisture agreed well with hygrometer measurements. The units were then opened on a clean bench, and the parts immediately packaged with desiccant to minimize changes in the lubricant due to reaction with the atmosphere. XPS analysis was performed on five parts from each device, and friction measurements made on escapement gear assemblies. Parts from stronglink escapements from 9 to 20 years of age have been analyzed.

Results and Discussion

The solid lubricant coating was found to exhibit distinct differences in friction response depending upon the degree of burnishing. These differences in behavior are represented in Figure 1, from tests conducted in laboratory air on lubricant applied to 15-5PH stainless steel substrates. Films that were lightly burnished had a friction coefficient whose initial value was high, and which dropped after several cycles of contact to a steady state value which was independent of the amount of surface oxidation. Similar behavior has been observed in sputtered films with surface oxidation,1 and corresponds to rapid wear of the oxidized material at the surface to expose non-reacted material. More aggressively burnished films had friction coefficient that started at a higher value than the lightly burnished material, but did not decrease with sliding contact. In fact, the friction coefficient increased steadily throughout the test, to a final value 10-20% greater than the initial value.

Figure 1. Friction coefficient in air as a function of lubricant oxidation. The starting and final friction coefficient values are indicated by the tail and head of the arrow, respectively.
Older hardware in the stockpile is expected to be more heavily burnished. This type of surface was found to oxidize more rapidly. This result, combined with the insensitivity of the friction of lightly burnished surfaces to oxidation, led to additional investigations of the aggressively burnished material. The effect of substrate material, water vapor, and adsorbates on MoS₂ oxidation kinetics were examined. The friction behavior of these surfaces was examined after oxidation and compared to the surface chemistry results. The starting and final friction coefficient were not strongly affected by the contact stress, substrate material, or total oxidation as measured by conversion of MoS₂ to MoO₃. However, the initial friction coefficient in a nitrogen environment was sensitive to the amount of sulfur present as sulfate, as shown in Figure 2. These friction values are generally lower than those in Figure 1 because the tests represented in Figure 2 were conducted in a pure nitrogen atmosphere. The Cu-2%Be alloy produced more variation in sulfate concentration than did the stainless steel substrate. For both alloys, the starting friction coefficient is observed to increase with the amount of sulfur as sulfate, independent of whether oxidation took place under dry or moist air conditions. The amount of sulfate produced is directly related to the amount of sulfur removed from the MoS₂ phase, which has low shear strength. Friction coefficient is therefore expected to increase as sulfate is produced by oxidation. The data show that changes in starting friction coefficient of 50% are possible for highly oxidized surfaces.

Parts returned from the stockpile exhibit friction coefficients between 0.2 and 0.4 in nitrogen. The friction coefficient on parts typically remains near the starting value or increases slightly throughout the test, similar to the behavior observed with films having some exposed substrate. These results are consistent with those obtained from oxidized sample coupons, validating the use of aggressively burnished surfaces to develop the lubricant performance model.

Conclusions
An integrated program has been developed to predict the effects of aging on the performance of the MC2969 stronglink escapement. The performance model is based on the oxidation kinetics of the lubricant, and the impact of oxidation on lubricant performance. The friction response of oxidized lubricant is strongly influenced by the degree of burnishing after deposition. Thick lubricant films resulting from light burnishing are oxidized only near the surface, so that wear of the oxidized material leaves a surface whose properties are the same as non-oxidized material. Thin lubricant films resulting from more aggressive burnishing exhibit a friction coefficient that reaches a steady state value greater than that of non-oxidized material. The initial friction coefficient on oxidized surfaces is related to the amount of sulfide converted to sulfate, corresponding to loss of the low shear MoS₂ lamellar structure. The impact of such changes in friction on device performance are being evaluated in dynamic simulations of the MC2969, as well as instrumented functional tests of MC2969 escapements having controlled oxidation.

References

Acknowledgments
Sandia is a multiprogram laboratory operated by Sandia Corporation, a Lockheed Martin Company, for the United States Department of Energy under Contract DE-AC04-94-AL85000. The authors wish to thank Rand Garfield for help with sample preparation.