Characterization of Nanoscale Surface Films in Solid Lubricants

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INTRODUCTION

Extreme environments often preclude the use of conventional lubricants in moving mechanical systems. Molybdenum disulphide (MoS_2), a lamellar solid lubricant exhibiting extremely low friction in vacuum, is often used to lubricate sliding interfaces in space applications.

Solid lubricants (particularly lamellar solids like MoS_2) accommodate motion by forming sliding-induced low shear-strength surface films (called tribofilms). Though the conventional wisdom suggests a liquid-like bulk shear process, recent studies suggest that the films and shear are confined to within 10 nm of the surface.[1] These films have proven difficult to detect and even more difficult to probe mechanically.

The tribofilm is widely regarded as a critical element of low friction sliding. Understanding the tribology of solid lubrication requires an improved understanding of the tribofilm. The aims of this research are to (1.) investigate the effect of environment on evolution of a stable sliding interface and (2.) characterize the properties of these films at relevant length-scales to understand their contributions to macroscale friction and wear.

EXPERIMENTAL

Transient Wear

Transient wear and friction were measured using a custom-built tribometer with an optical interferometer (SWLI) positioned above the wear track (*see Figure 1*) to allow periodic wear measurements.

Six wear-rate measurements were made on one MoS_2 coating (sputtered by Tribologix). The results are shown in Figure 2. The low initial wear rate of 2 x 10^{-8} $mm^3/(Nm)$ increased by an order of magnitude after two subsequent tests in dry air (<500 PPM water). The wear rate in lab air (after dry testing) was higher by nearly another order of magnitude and tended to increase with subsequent testing over time.

We have consistently observed increased wear and friction (not shown) over time which suggests a time-dependent uptake of moisture into the bulk of the coating. To test this hypothesis, the sputtered coat-

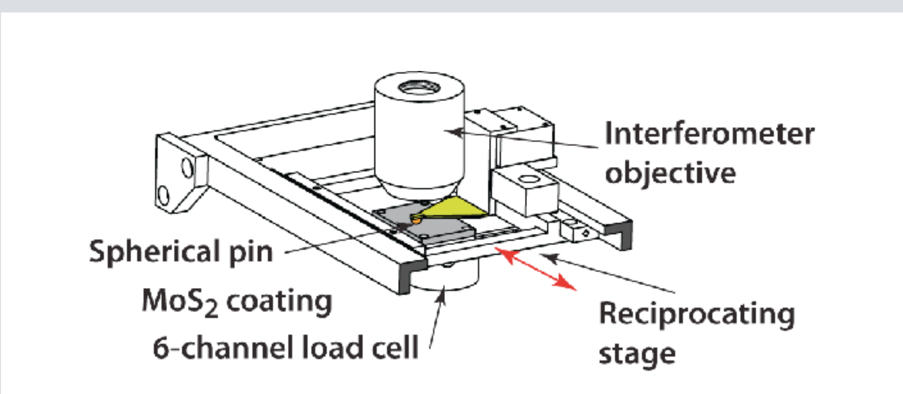

Figure 1 | **Custom-tribometer used for measuring transient wear.**

ing was annealed at 100°C for different durations and friction was measured after allowing the MoS_2 sample to cool to room temperature each time.

The friction coefficient (50 sliding cycles) decreases significantly with increased annealing time in between tests (*see Figure 3*). When the coating was annealed at 100°C for an hour, the friction coefficient in humid air at room temperature was comparable to those found in dry conditions. Similar measurements performed on hydrogenated diamond-like carbon (DLC) do not lead to any significant reduction in friction. During cooling, water adsorbs to the DLC surface and quickly reaches equilibrium with the environment. Conversely, friction of the MoS_2 coating remains low for several minutes in a humid environment after most of the water has been driven from the bulk. While DLC friction appears to be driven purely by coating surface adsorption (fast kinetics), bulk diffusion appears to play a significant role in the tribology of MoS_2 (*see Figure 4*).

Nanotribology

To characterize tribofilms and their contribution in macroscale friction, a direct, yet non-invasive probe at the rel-

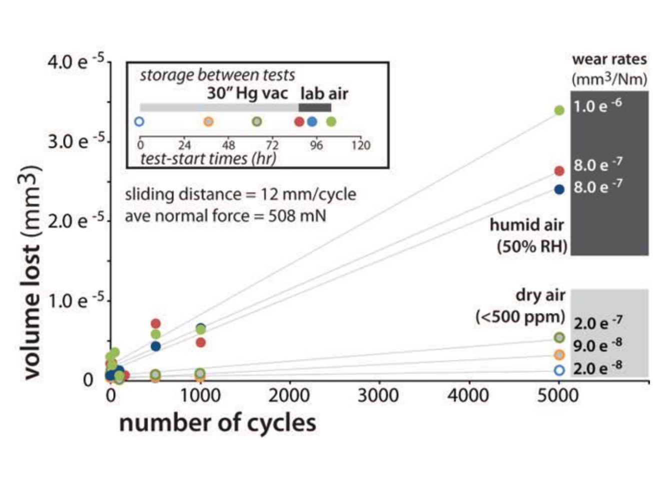

Figure 2 | **Wear rates based on intermittent wear volume measurements for three dry sliding and three humid air wear tracks. Inset shows wait times between tests and corresponding coating storage condition.**

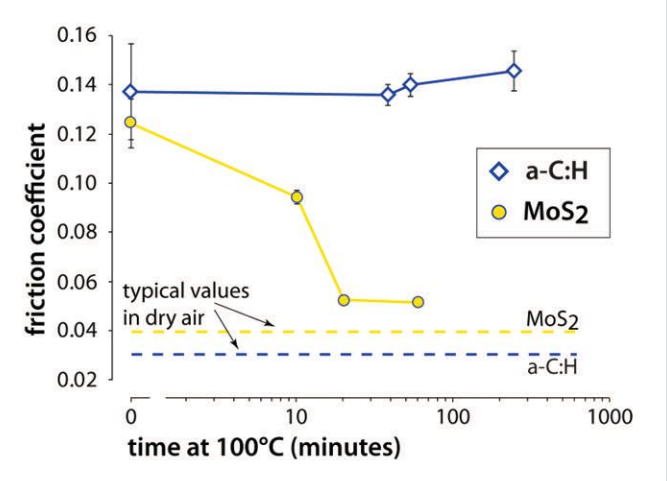

Figure 4 | **A comparison of change in friction as function of annealing time for DLC and MoS_2 sputtered coatings in lab air. Intervals of heating between tests had no effect on DLC friction. Numbers within bars indicate number of measurements.**

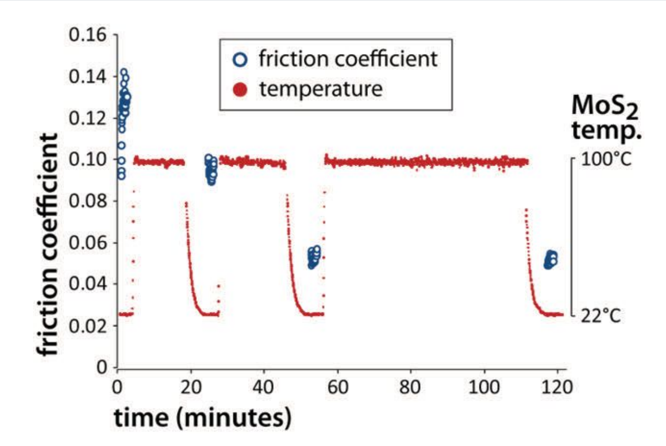

Figure 3 | **Friction coefficient and temperature for annealing experiment. Each friction measurement of 50 sliding cycles (440 C against MoS_2) was made in lab air at room temperature. Intervals of heating to 100°C in between tests reduced the amount of water in the coating, which led to reduced friction.**

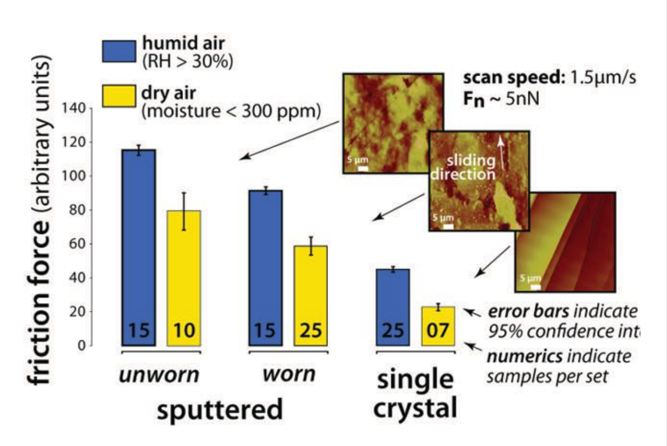

Figure 5 | **Results for LFM measurements on sputtered and single crystal MoS_2 show both environmental and spatial correlation.**

evant length-scales is required. Single-load lateral force microscopy measurements were performed with a silicon nitride tip on worn and unworn sputtered MoS_2 coatings and compared with single crystal MoS_2.

As illustrated in Figure 5, nano-friction (quantified here by the width of friction loops) bore a direct correlation with the presence of moisture in the surrounding environment, irrespective of the substrate structure. Further, for similar environments, friction within the wear track is seen to be lower than for unworn MoS_2, with the friction of single crystal MoS_2 being lowest in both environments.

These preliminary results illustrate the variation in nano-scale friction between worn and unworn sputtered coatings, presumably due to formation of ordered tribofilms and how these compares with the idealized single-crystal structure.

CONCLUSIONS

- Transient wear measurements show moisture-dependence of MoS_2 wear across two distinct time-scales: the immediate environment and the coating 'exposure history,' suggesting effects of storage conditions on prevailing coating wear rate.
- Sensitivity of MoS_2 friction to annealing is seen as indicative of the tendency of MoS_2 to act as a sink for gradual moisture uptake over time, which during annealing is thermally driven out.
- Preliminary single-asperity measurements demonstrate that sliding-induced surface structure affects the frictional response and moisture sensitivity.

ACKNOWLEDGMENTS

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