



## The Role of Water on the Tribological Performance of Molybdenum Disulphide



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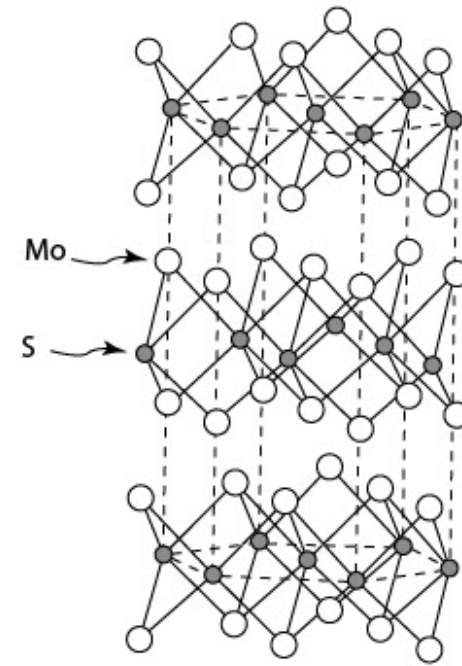
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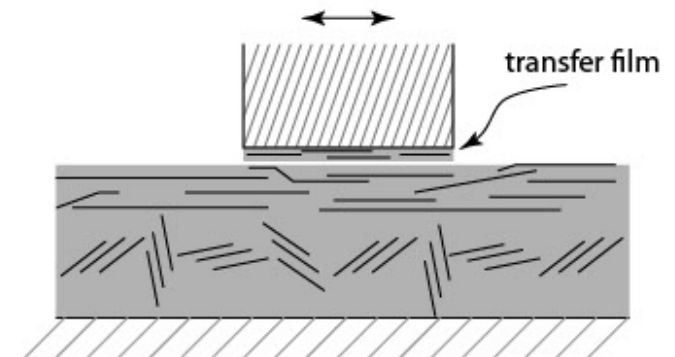
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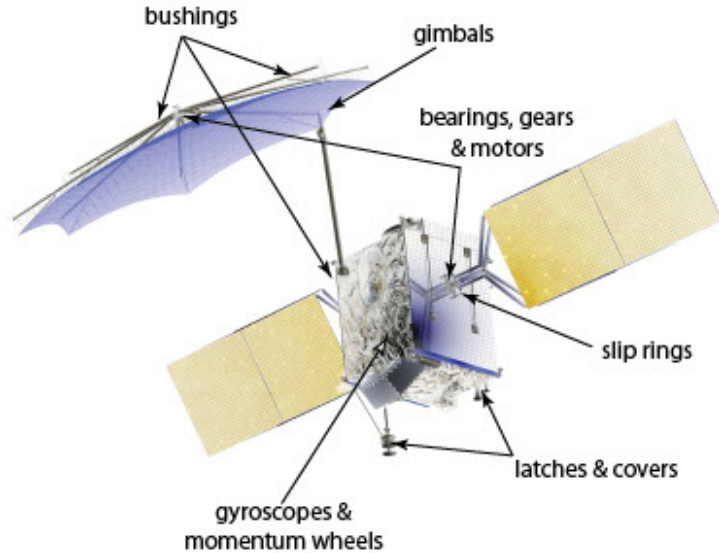
Molybdenum disulphide has a hexagonal, layer-lattice structure in which there is covalent bonding between the S-Mo-S, but van der Waals attraction between each tri-layer. This layer-lattice structure in conjunction with the van der Waals allows for the easy slip between the tri-layers providing the low friction properties.



Amorphous  $\text{MoS}_2$  coatings are known to form crystalline layers which orient themselves upon sliding leading to the low friction characteristics.



## Satellites



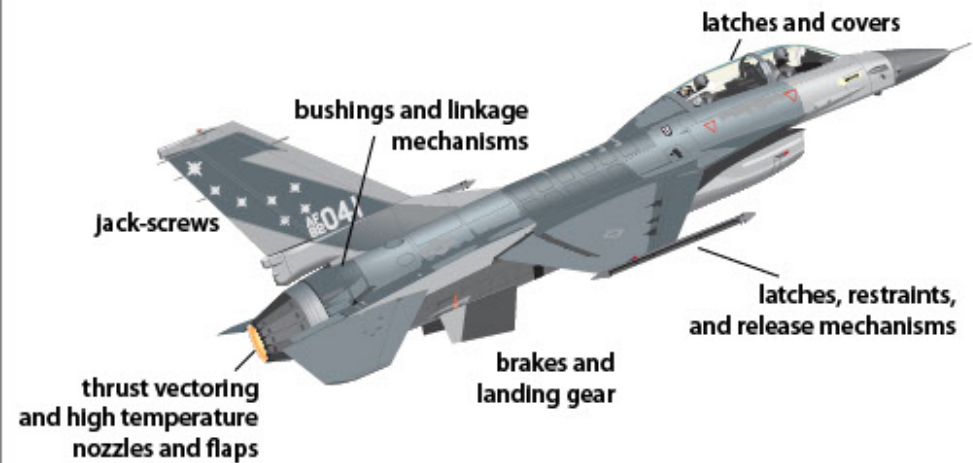
### Operating Environments

Environmental Extremes:

- extraterrestrial deployment (UH vacuum, -150°C to 100°C)
- terrestrial testing (ambient lab air)

Packaged antennas must survive extended cycled deployment in ambient conditions prior to deployment in space.

## Aircraft Actuators



### Operating Environments

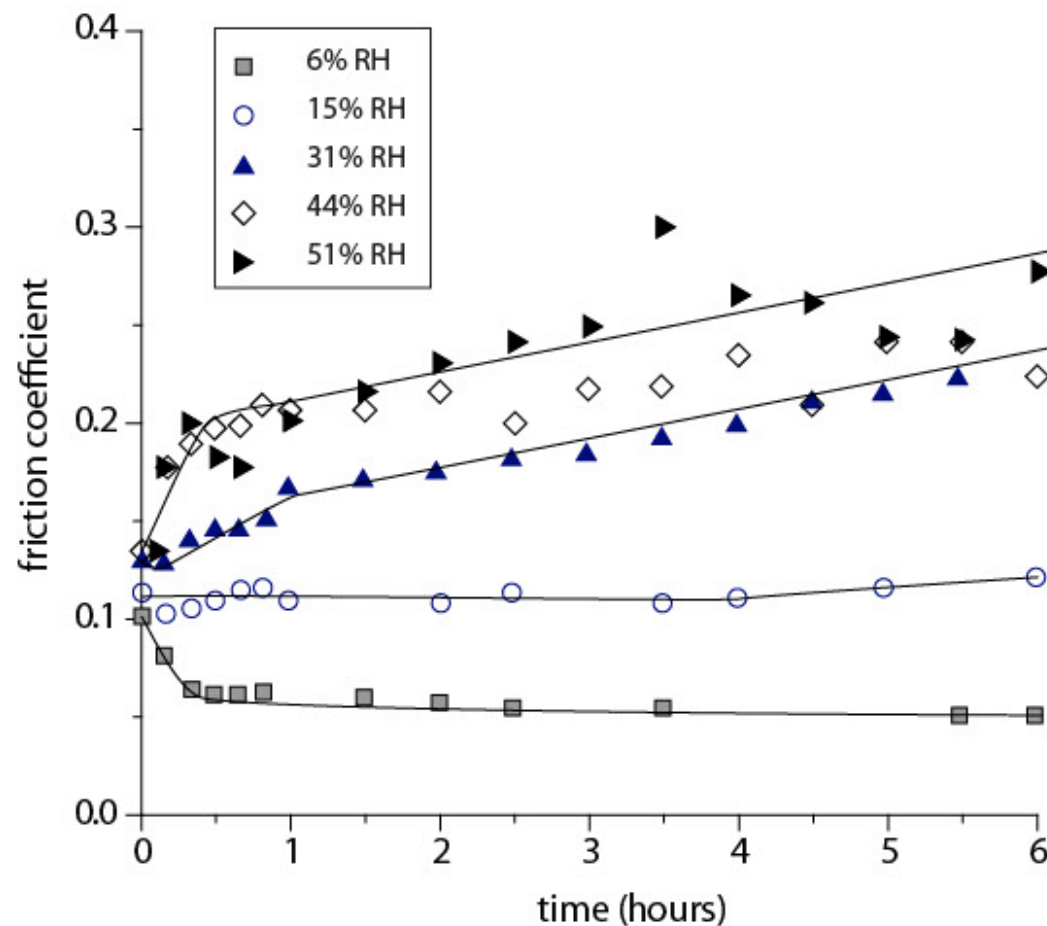
Environmental Extremes:

- high altitude dog fights (-50°C, low humidity)
- equatorial carrier operations (>35°C, >90% humidity)

Devices must function for 1000's of cycles between environmental extremes.

*Since 1951 over 1,000 papers have been published on the topic of MoS<sub>2</sub> Tribology.*

The first report on the effect of relative humidity on the friction and wear of MoS<sub>2</sub> is from a NACA publication in 1953 by M. B. PETERSON & R. L. JOHNSON.

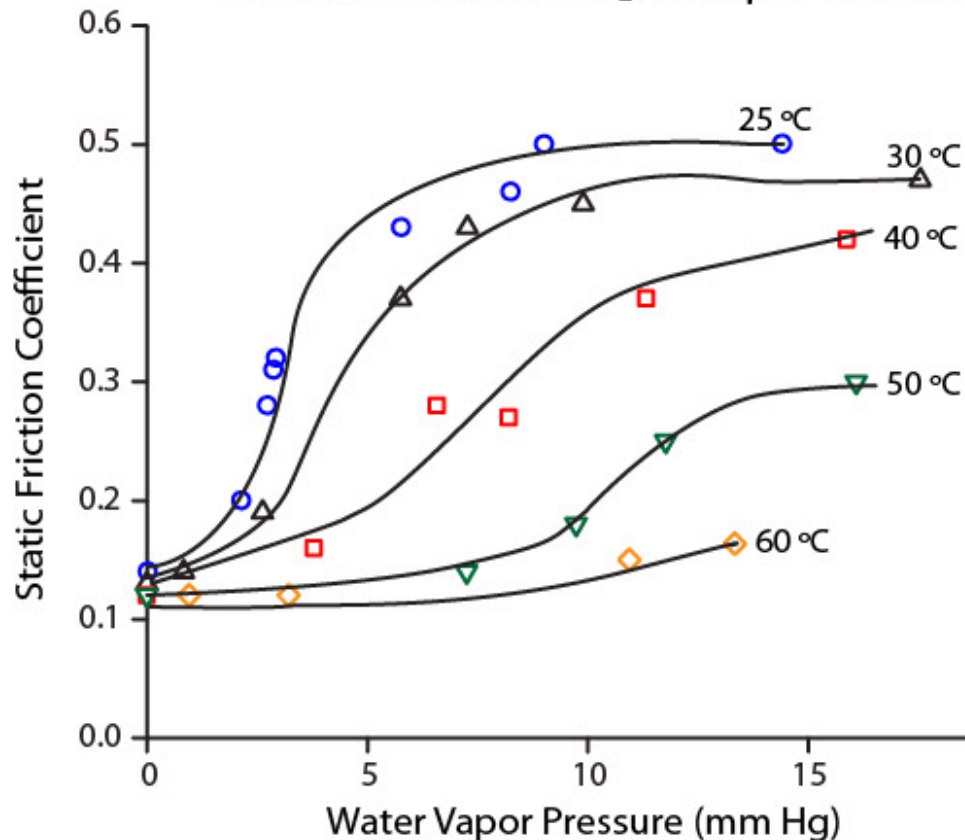


The effect of various relative humidities on the run-in of the coefficient of friction of steel specimens coated with molybdenum disulfide powder. Constant loads of ~180 N and ~30 mm/s.

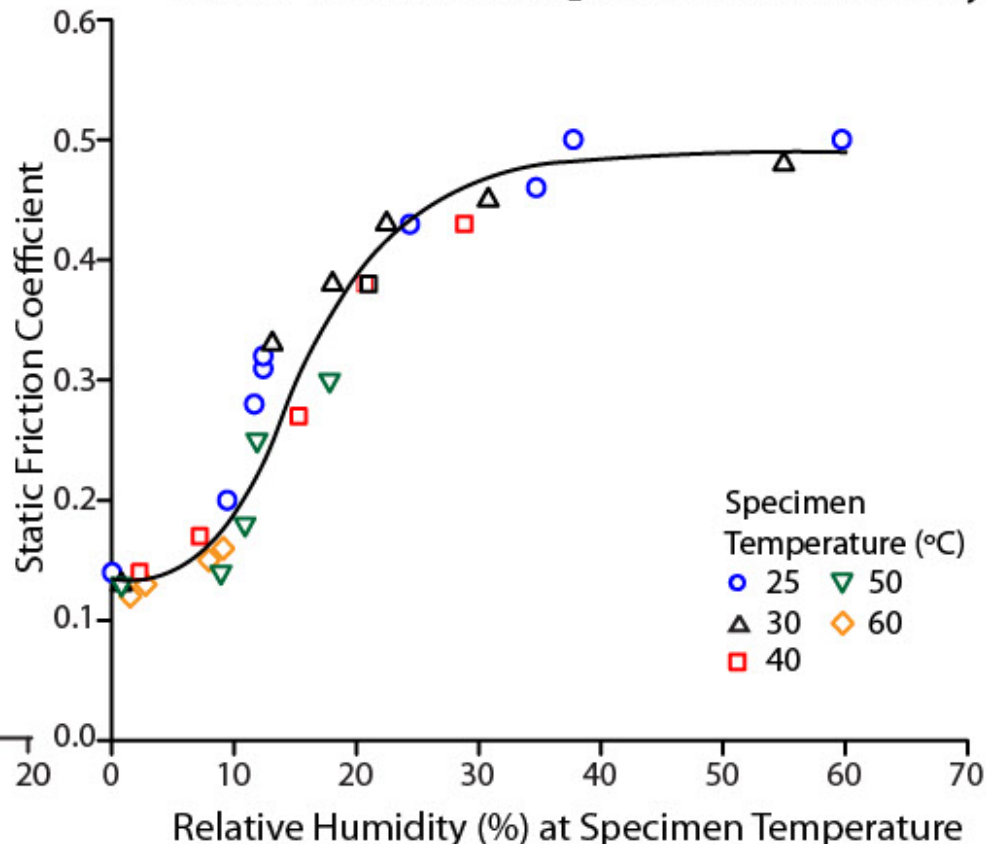
Data adapted from Peterson, M.B. and Johnson, R.L., Friction and wear investigation of molybdenum disulfide I - effect of moisture, NACA, 1953



## Static Friction of MoS<sub>2</sub> vs. Vapor Pressure

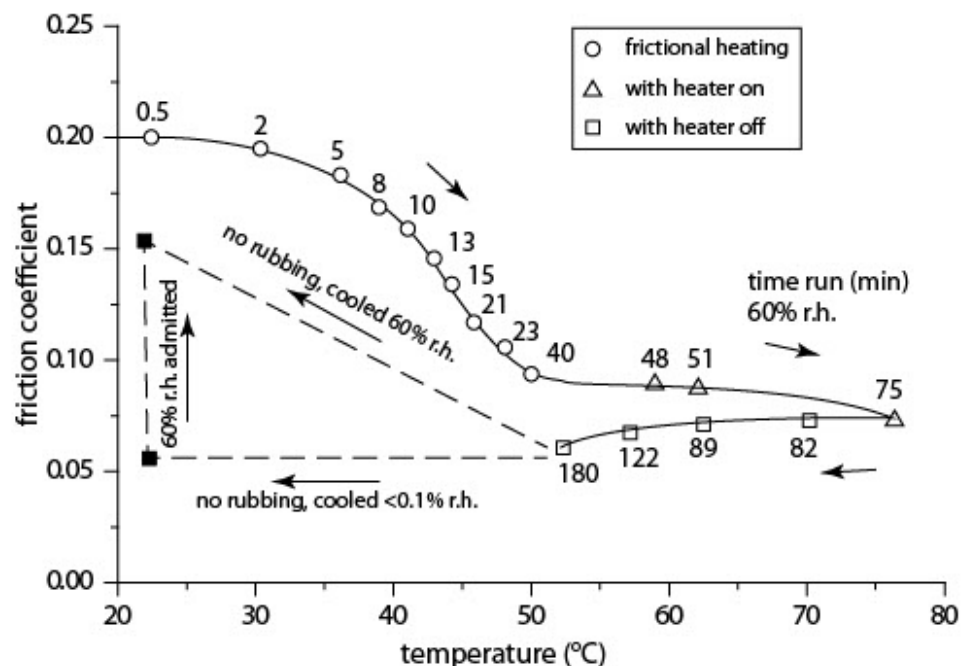


## Static Friction of MoS<sub>2</sub> vs. Relative Humidity



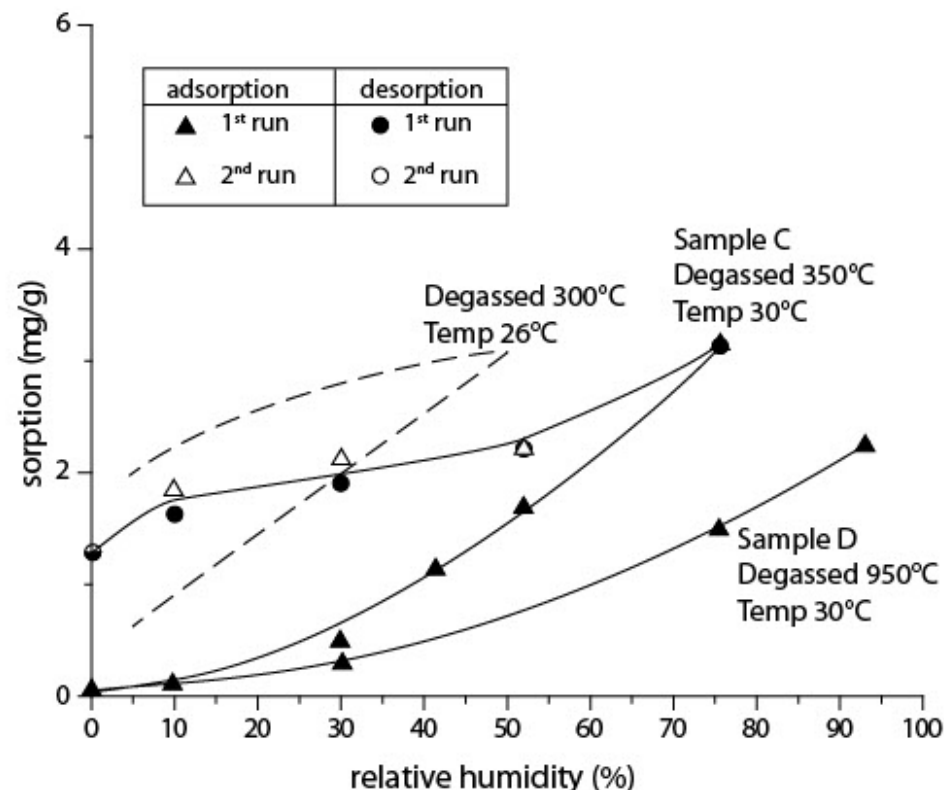
Water vapor pressure is increased while static friction is measured. The experiments were repeated five times at five different temperatures. The trend of low friction with high surface temperature indicates that water is a likely culprit.

Here the results are plotted as a function of relative humidity. The data points all adhere closely to one curve illustrating good agreement between experiments.



The reversible effect of moisture on the coefficient of friction for a rubbed molybdenum disulphide film.

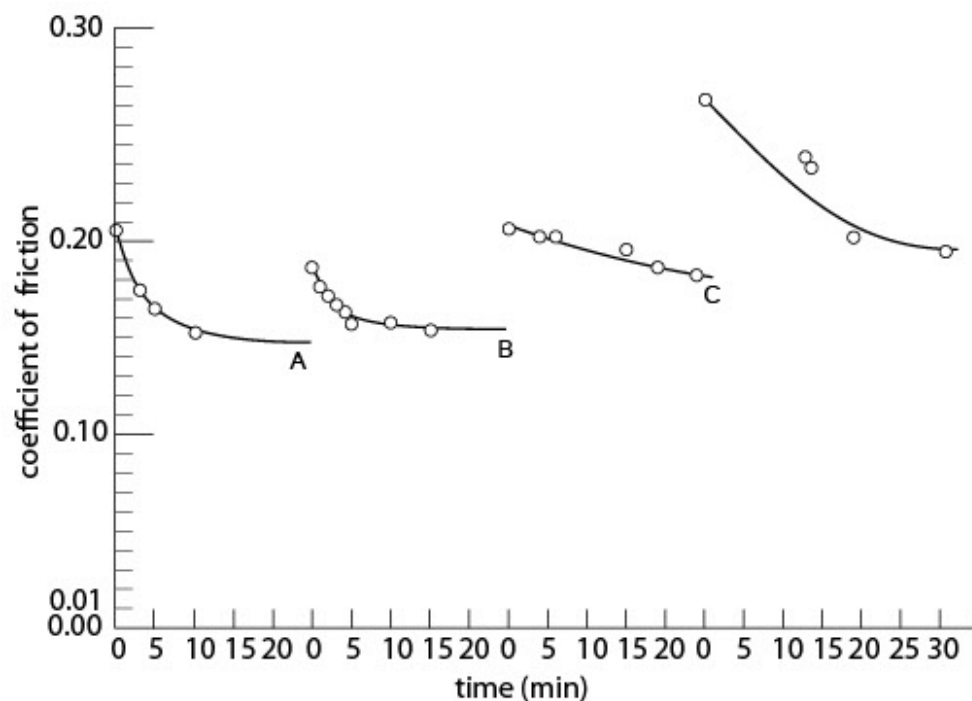
Midgley's results from Lansdown, A.R., Molybdenum Disulphide Lubrication, D. Dowson (ed.), Elsevier Press, Amsterdam, 1999



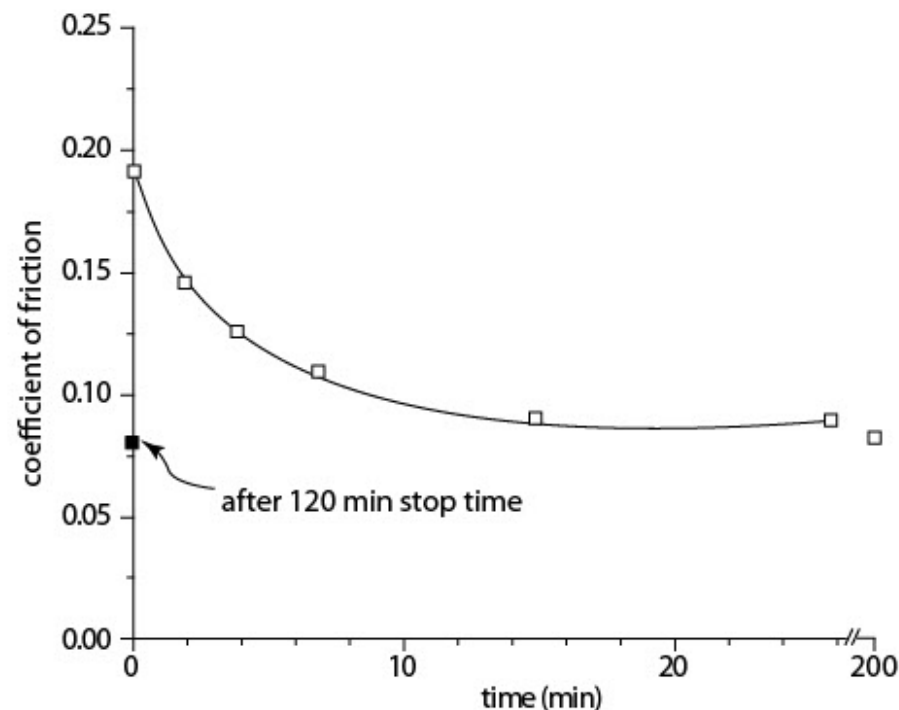
The reversible sorption and desorption of water vapor on MoS<sub>2</sub> after various degassing methods.

Johnston, R.R.M. and Moore, A.J.W., Water adsorption on molybdenum disulfide containing surface contaminants, Journal of Physical Chemistry, 1964, 68(11): p. 3399-3406

*In the late 1950's and early 1960's the response of MoS<sub>2</sub> to varying exposure time was explored. Haltner studied this effect in air and dry nitrogen environments.*



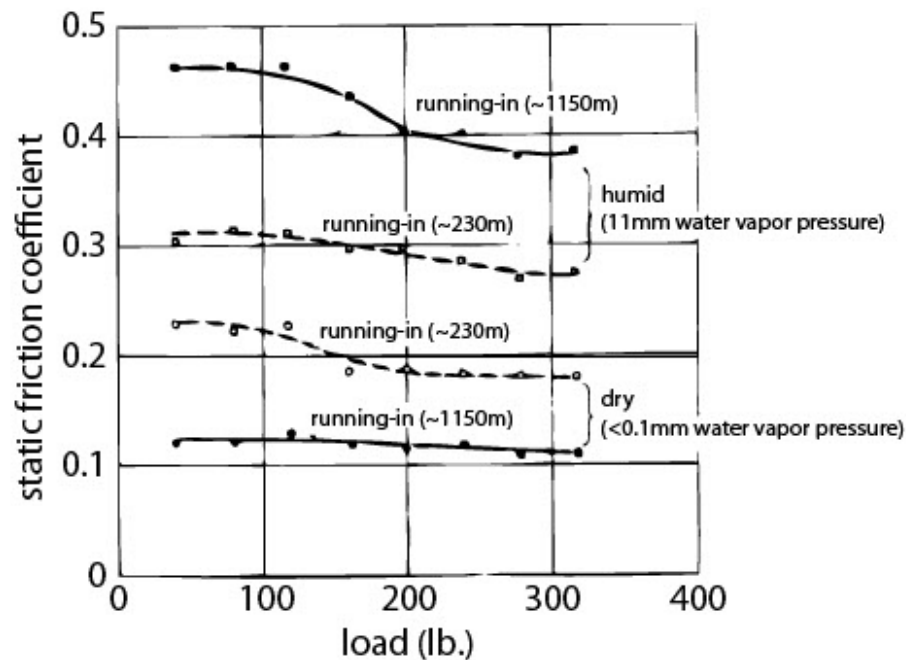
Transient frictional behavior of MoS<sub>2</sub> in air: ... After reaching  $\mu$  level at A, film and rider stood for 4 hours after sliding for 80 min; at B they stood overnight after sliding for 135 min; at C they stood overnight after sliding for 420 minutes.



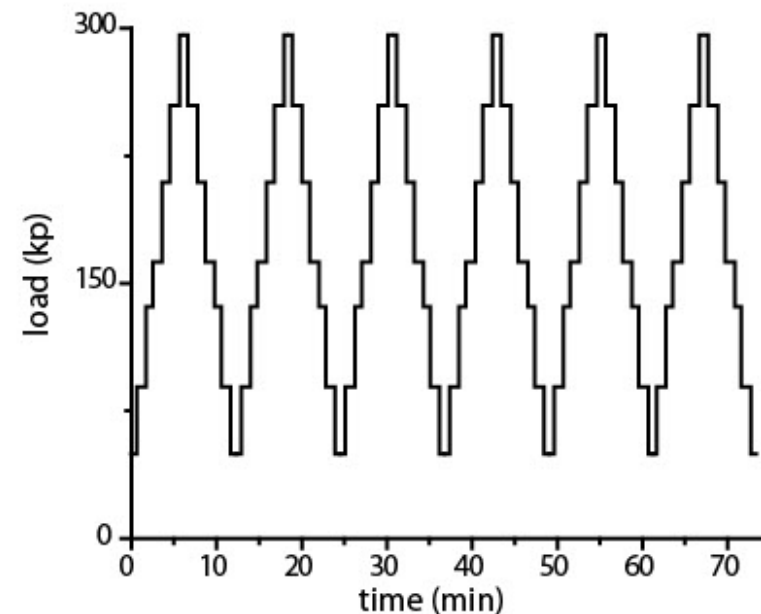
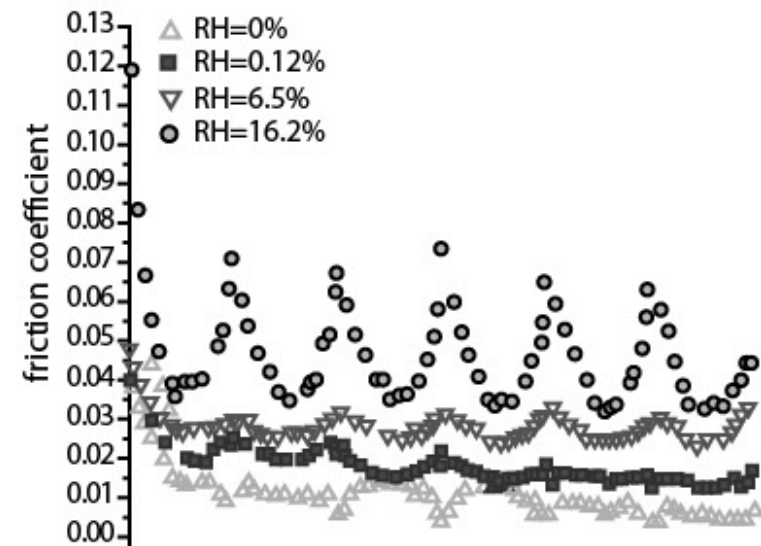
The friction coefficient of MoS<sub>2</sub> in a nitrogen environment at 175°C after 200 minutes of sliding is shown. The test was halted for 120 minutes and when sliding was resumed the friction value was unchanged.

The effect contact pressure has on the coefficient of friction of  $\text{MoS}_2$  has been studied since the 1960's. During this time frame, an understanding that moisture plays a role in the contact pressure effects came about.

Pritchard *et al.* and Gänsheimer analyzed the effect an increase of load had on the friction coefficient of a pure  $\text{MoS}_2$  coating at different humidities.



Adapted From Pritchard, C., and Midgley, J.W., The Effect of Humidity of the Friction and Life of Unbonded Molybdenum Disulphide Films. *Wear*, 1968. 13: p. 39-50



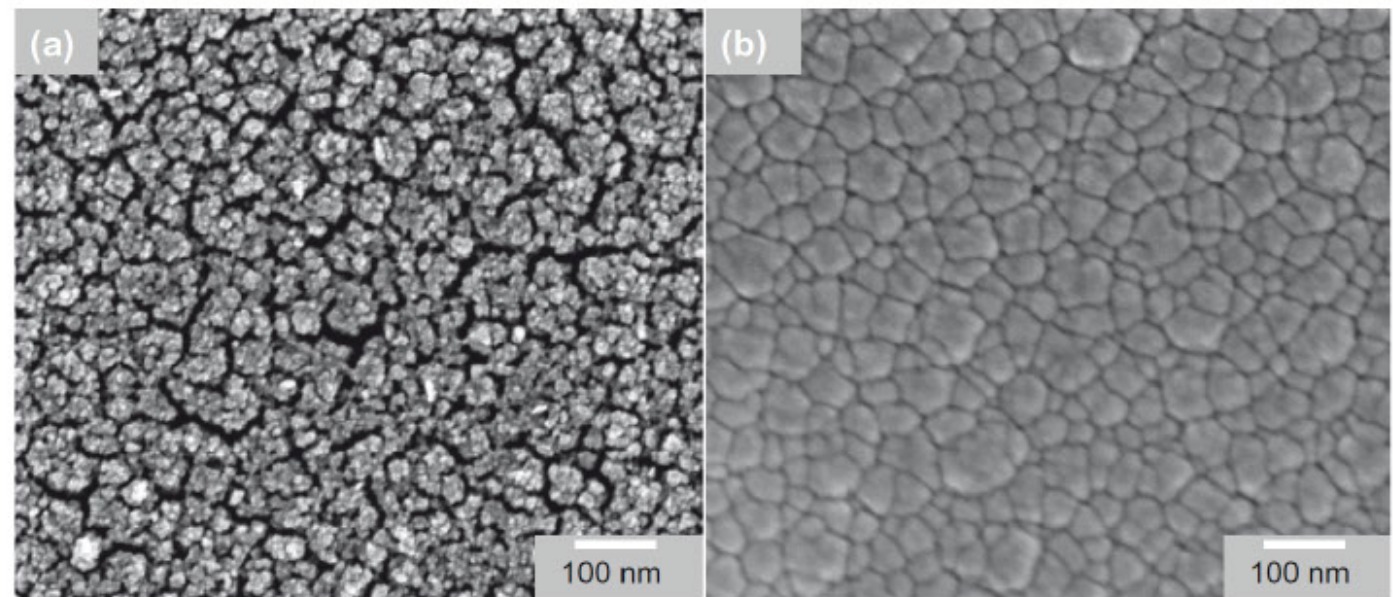
Gänsheimer's results adapted From Winer, W.O., Molybdenum disulfide as a lubricant: A review of the fundamental knowledge. *Wear*, 1967. 10(6): p. 422-452.



Pure MoS<sub>2</sub> has shown differences between its tribological behavior in dry and moist environments. A necessity for improvements of the performance in moist environments led to the development of composite MoS<sub>2</sub> coatings through various deposition processes. The addition of dopants was made to improve the friction, wear, and environmental stability of the coatings.

Examples of composite MoS<sub>2</sub> coatings in use today are:

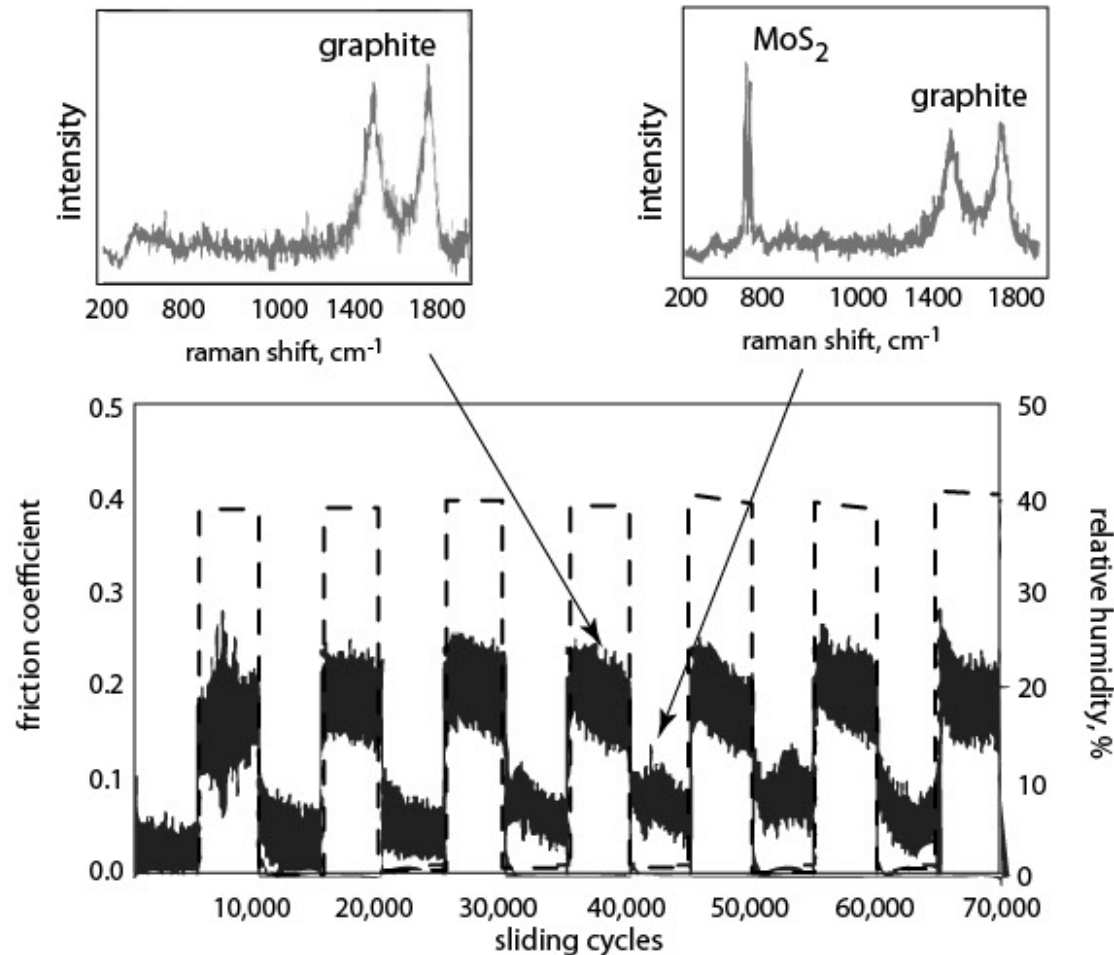
- MoS<sub>2</sub>
- MoS<sub>2</sub> + Ni
- MoS<sub>2</sub> + Ti
- MoS<sub>2</sub> + Sb<sub>2</sub>O<sub>3</sub>
- MoS<sub>2</sub> + Sb<sub>2</sub>O<sub>3</sub> + Graphite
- MoS<sub>2</sub> + Sb<sub>2</sub>O<sub>3</sub> + Au



HRSEM secondary electron images of (a) pure MoS<sub>2</sub> film and (b) nanocomposite MoS<sub>2</sub>/Sb<sub>2</sub>O<sub>3</sub>/Au coating

Images adapted from Scharf, T. *et al.*, Friction and wear mechanisms in MoS<sub>2</sub>/Sb<sub>2</sub>O<sub>3</sub>/Au nanocomposite coatings, *Acta Materialia*, 2010, 58: p. 4100-4109

"In recent years, the concept of adaptive "chameleon" tribological coatings was developed, where coatings were designed to self-adjust their tribological contact chemistry and structure depending on the operating environment and temperature."

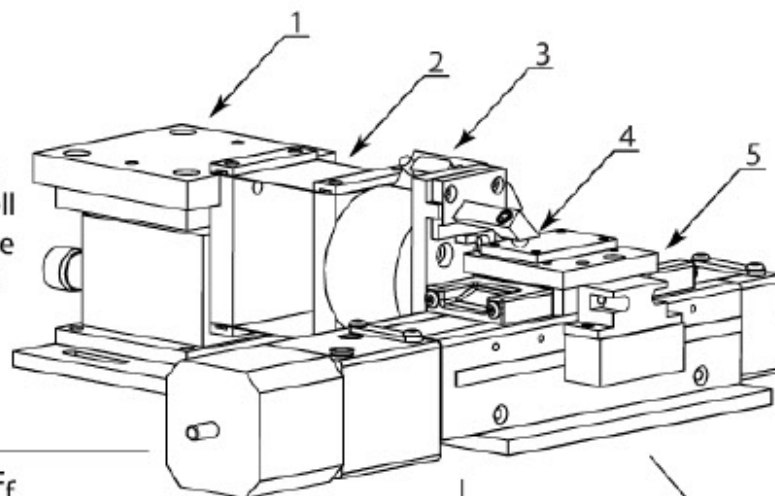


Variation of the friction coefficient (solid line) in sliding tests of a laser textured TiCN coating with a burnished MoS<sub>2</sub>/graphite/Sb<sub>2</sub>O<sub>3</sub> layer against a steel ball in conditions with a cycled environment humidity (dotted line). Insets show Raman analyses spectra taken from wear tracks with tests stopped during high and low humidity cycles.



①

1. vertical stage
2. parallel leaf flexure
3. six-channel load cell
4. pin and counterface
5. reciprocating table

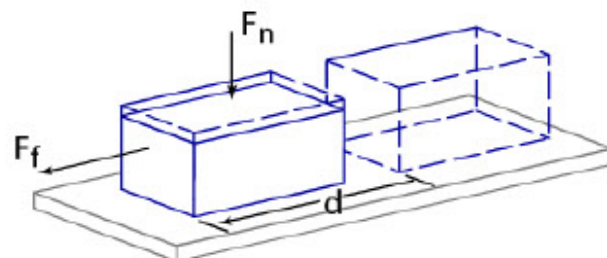


Accurate measurement of friction coefficient requires measurements of normal and frictional forces. The design philosophy is to make both measurements simultaneously with a multi-axis load-cell that is located directly in the force path to ground.

To make in-situ measurements of wear, we have implemented a scanning white-light interferometer that can measure relative displacements of surface features with sub-nanometer accuracy.

friction  $\mu = \frac{F_f}{F_n}$  ①

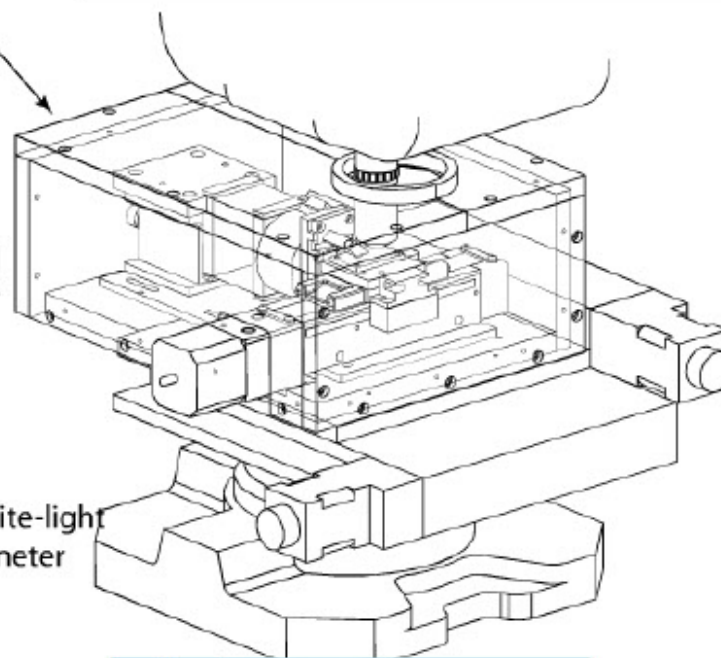
wear  $K = \frac{\text{Vol}}{F_n d} \left( \frac{\text{mm}^3}{\text{N m}} \right)$



Because the samples are consumed during tribological testing, there are no standard artifacts. Thus, following wear and progression of wear requires sensitive displacement measurements and interrupted measurements.

environmentally controlled chamber

scanning white-light interferometer



Properties



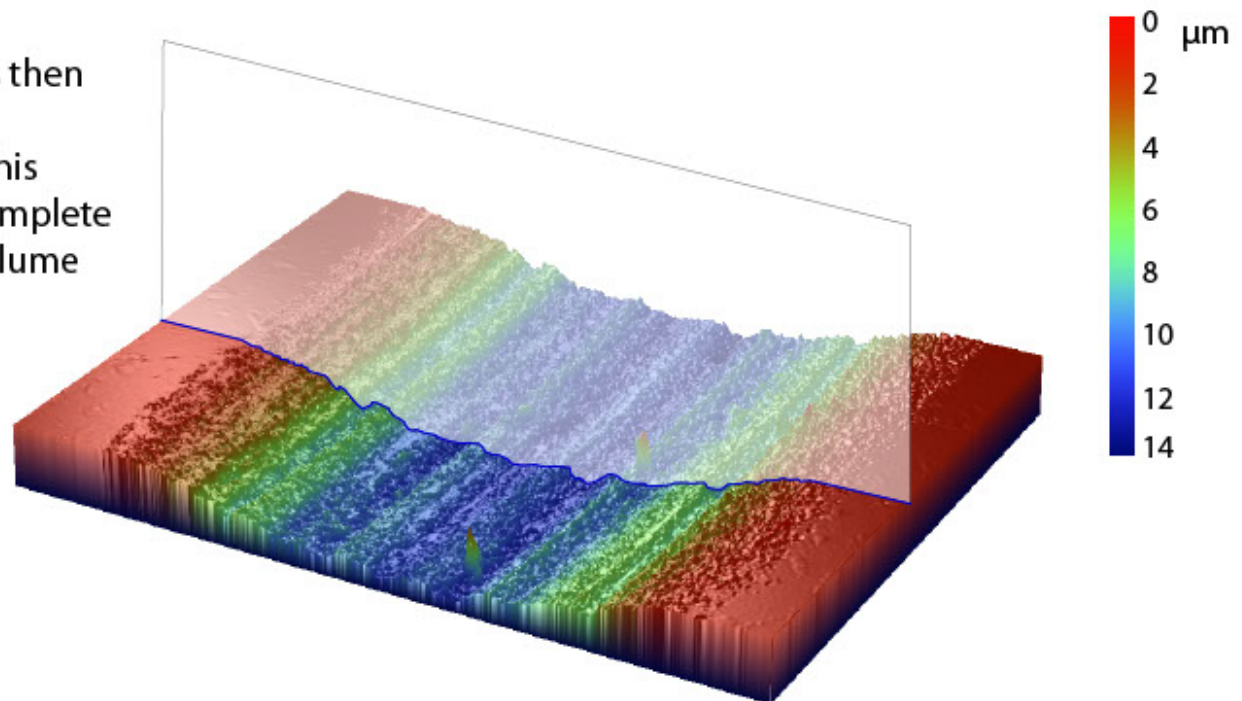
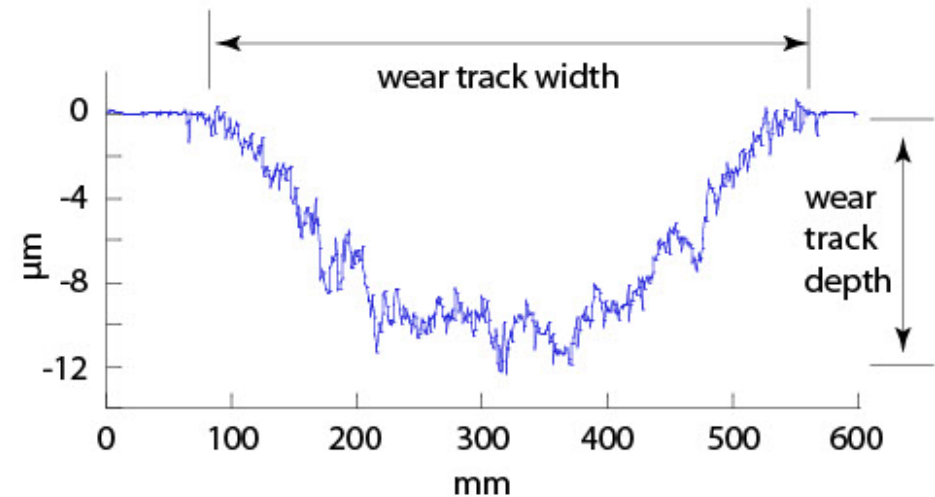
## Volume loss

Using scanning white light interferometry, a 3D surface scan of the wear track is taken.

A representative 2D slice through the wear track is taken and analyzed for the wear track cross-sectional area.

Creep of these thin film coatings is considered negligible.

The volume of material removed is then estimated by calculating the cross-sectional area of the scans. This average area is swept along the complete wear track path to calculate the volume loss.

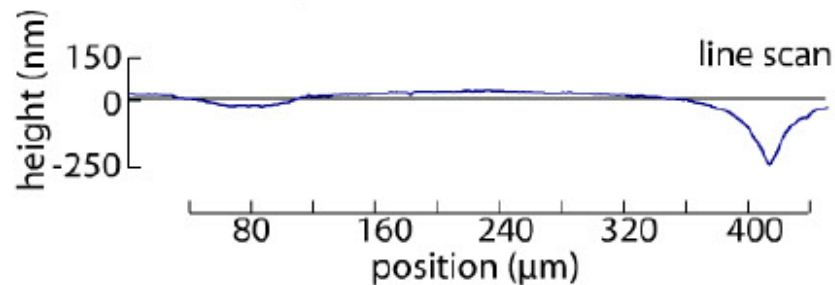




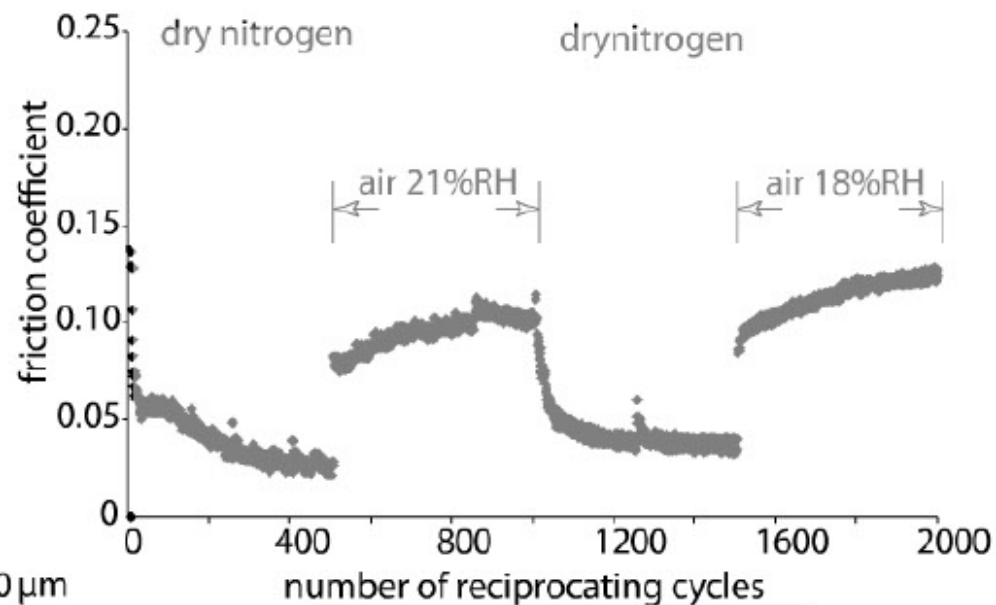
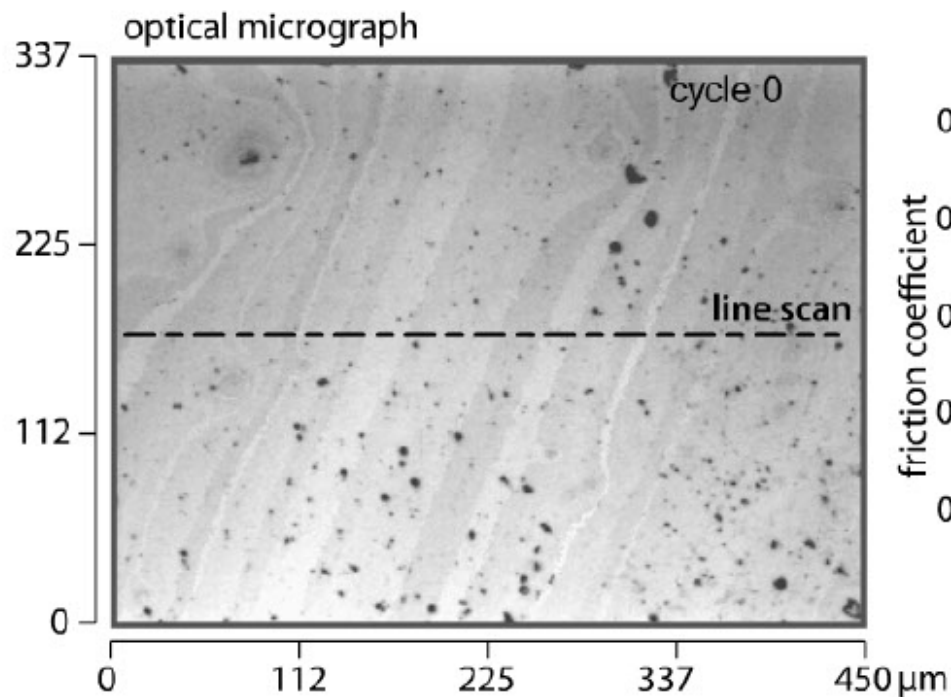
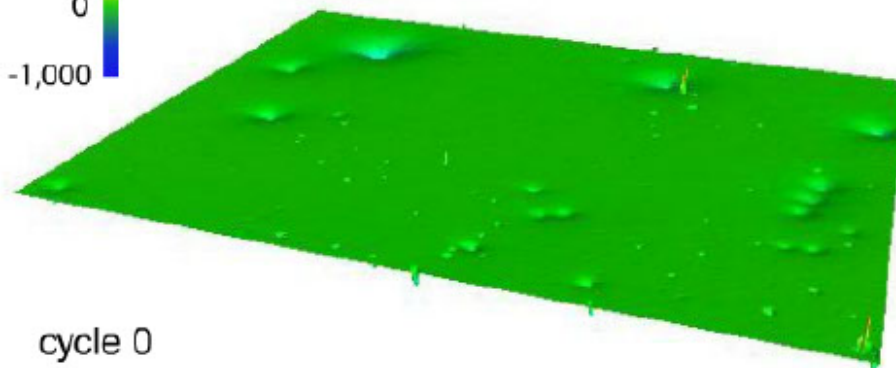
# Self-Mated $\text{MoS}_2/\text{Sb}_2\text{O}_3/\text{Au}$ in Dry Nitrogen and Air



$F_n = 5\text{N}$   
 $V = 10\text{ mm/s}$   
 $L = 10\text{ mm}$   
nitrogen  $< 1\% \text{ RH}$



height (nm)  
1,000  
0  
-1,000



# Self-Mated MoS<sub>2</sub>/Ti in Air and Dry Nitrogen



$F_n = 5\text{ N}$

$L = 10\text{ mm}$

$V = 10\text{ mm/s}$

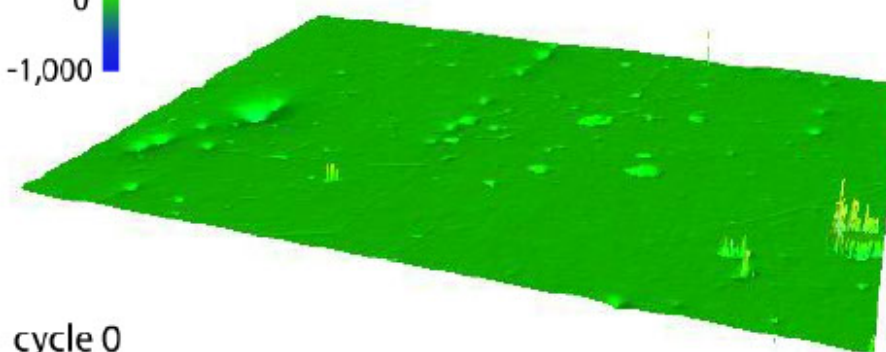
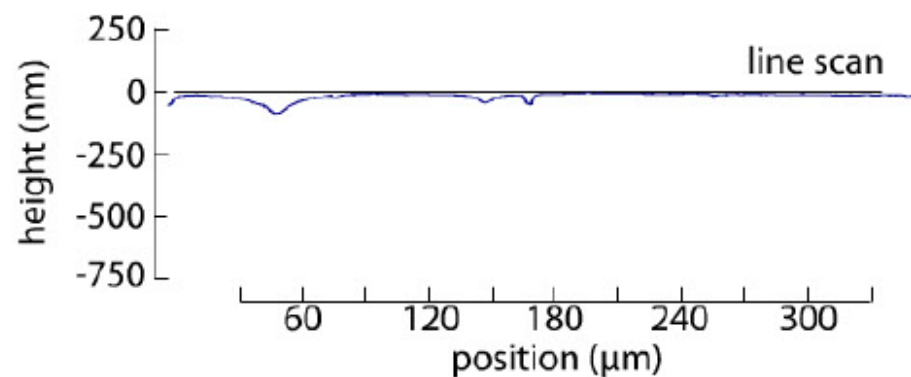
nitrogen <1% RH

height (nm)

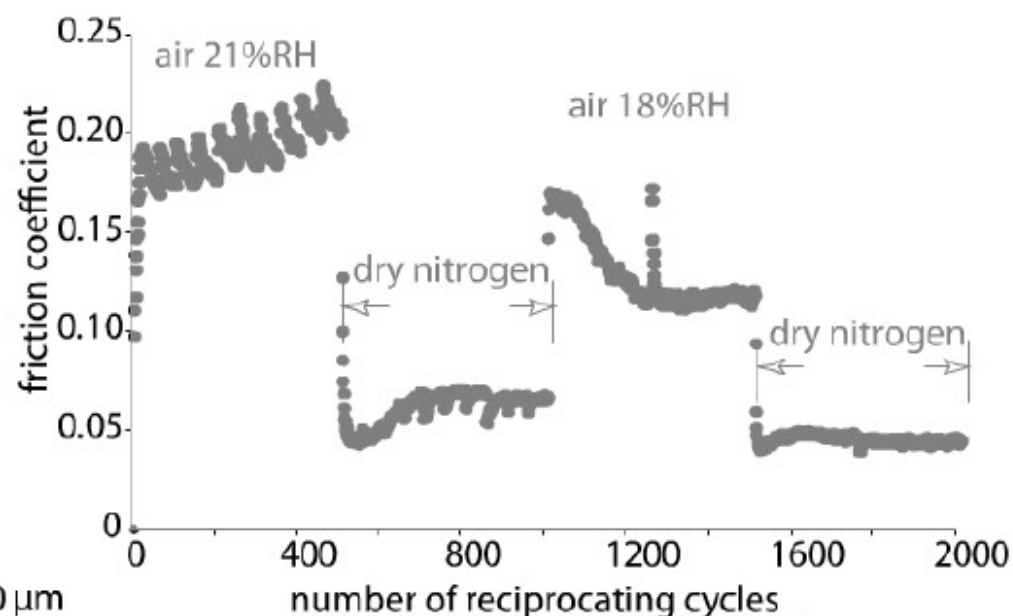
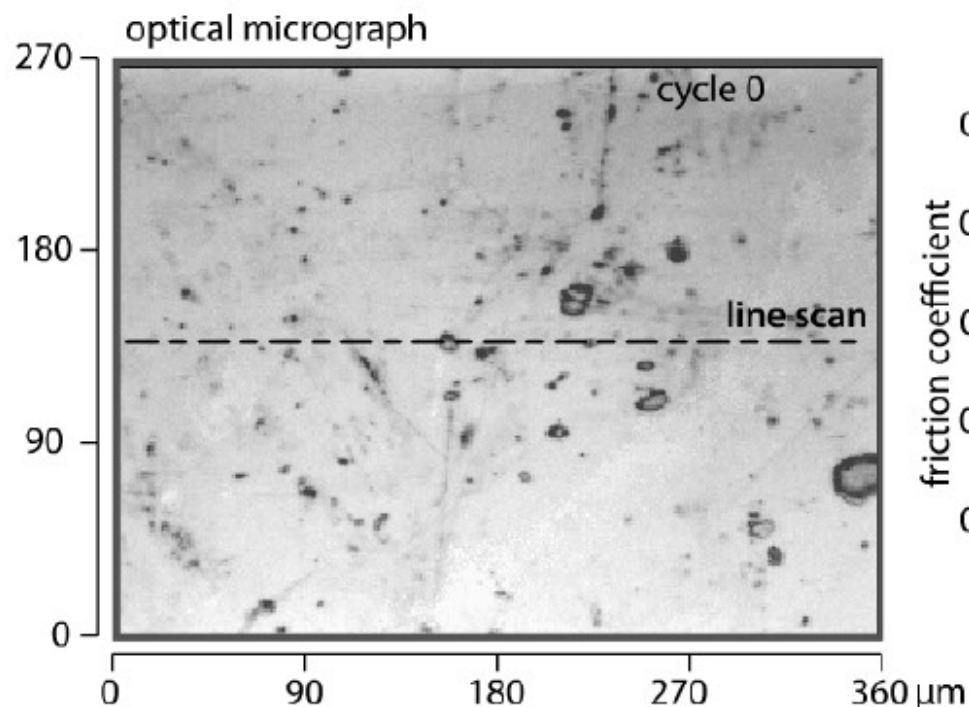
1,000

0

-1,000

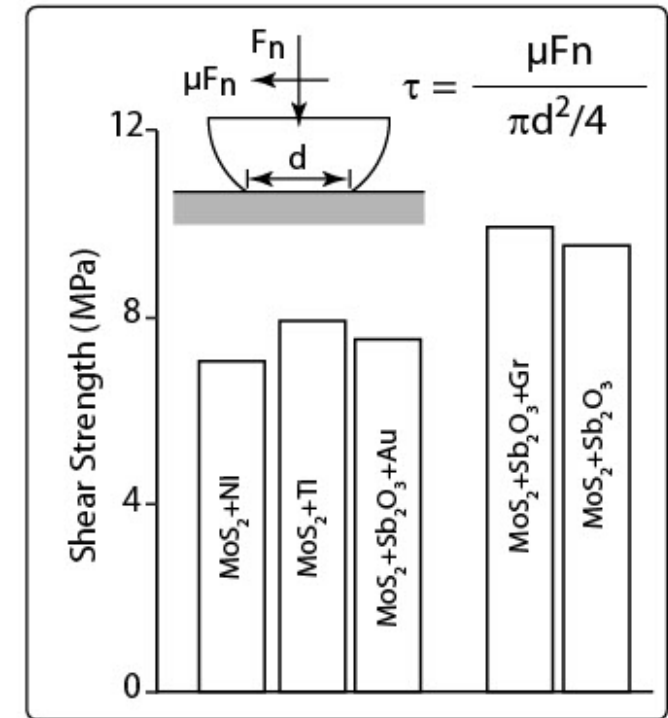
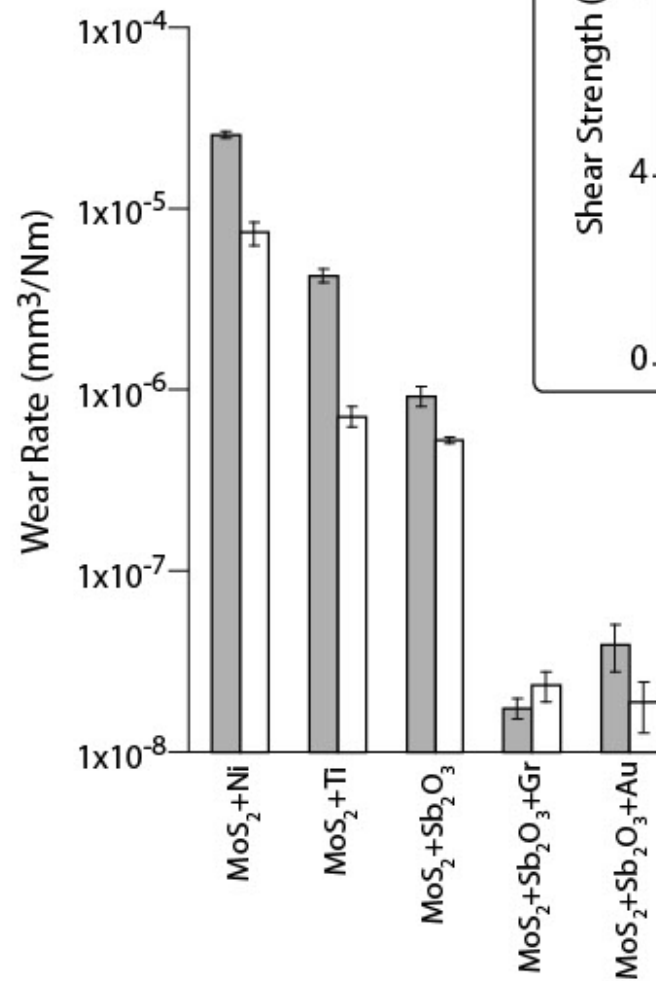
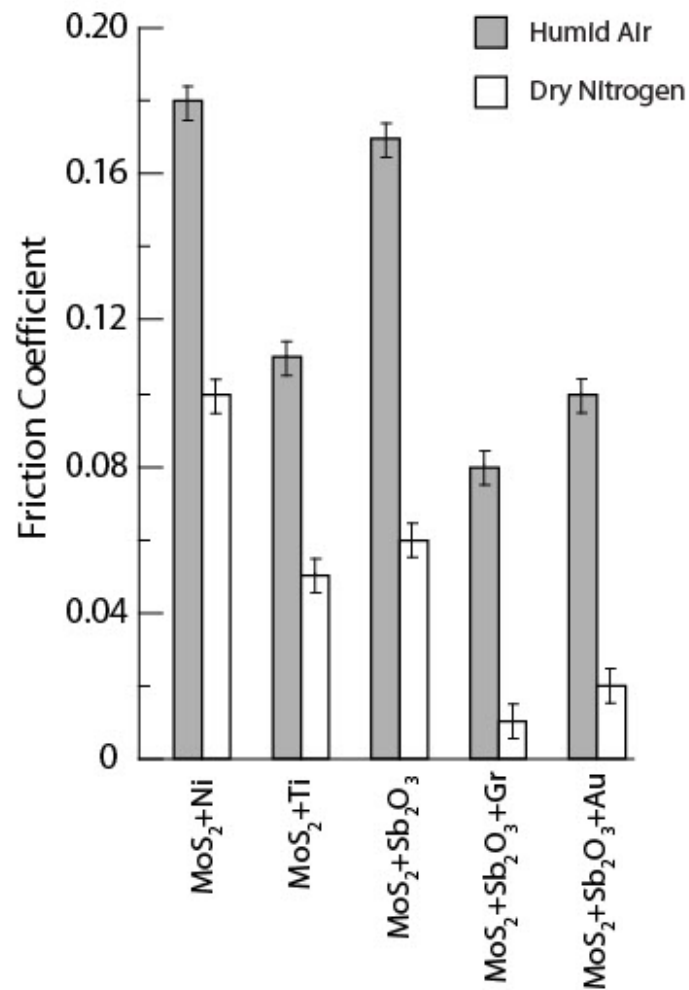


cycle 0



Properties

The friction coefficient and steady-state wear rates for the various coatings along with the experimental uncertainties in the measurements are given below. The wear rates of the films varied over 3 orders of magnitude. All tests were run self-mated at 5N load, 10 mm/s sliding speed, for 10,000 cycles; the total sliding distance was 100m.



*Under these experimental conditions at a wear rate of  $K=1 \times 10^{-7} \text{ mm}^3/(\text{Nm})$  approximately 1nm of material is removed each pass.*

### Summary of Trends

Friction coefficient decreases with a decrease in water content

Friction coefficient decreases with an increasing normal force, but only with water present

Initial run-in friction coefficient values increases with increased exposure to moisture without sliding

The water sorption of  $\text{MoS}_2$  is a reversible process once a portion of water is irreversibly sorbed

### Questions to be Addressed

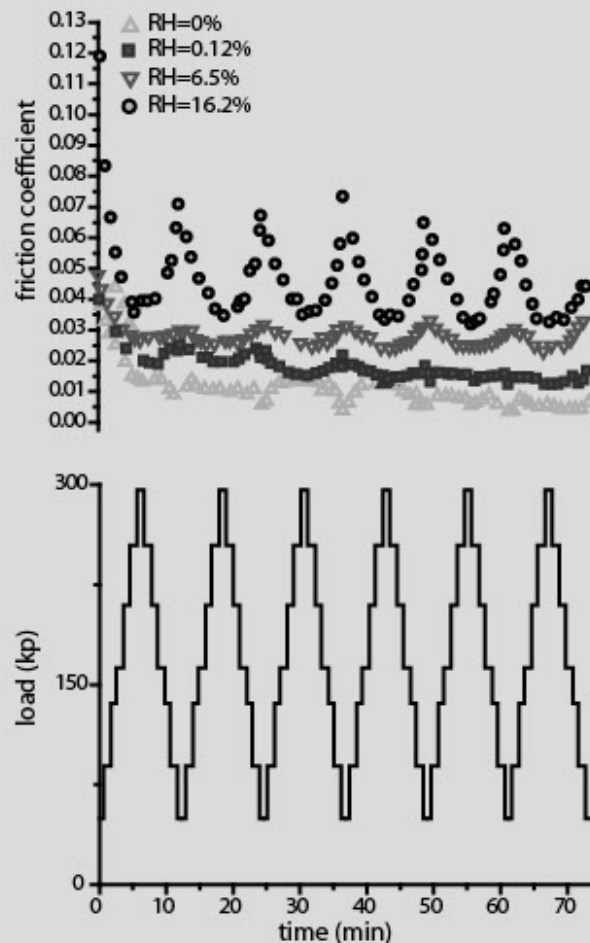
What is the amount of water being adsorbed into the films?

What is the critical moisture content which causes the increase in friction coefficient?

Is the water sorption affected by the density/porosity and thus surface area/volume ratio of the films?

Does orientation of the films play a role on the moisture effects?





Gänsheimer's results adapted From Winer, W.O., Molybdenum disulfide as a lubricant: A review of the fundamental knowledge. Wear, 1967. 10(6): p. 422-452.

$$\mu = \frac{F_f}{F_n} = \frac{A_{real} \cdot \tau_{shear}}{F_n}$$

There is a strong interdependence between friction coefficient and applied load at high humidity. That interdependence disappears at low partial pressure of water.

This indicates that the friction force is the factor influenced by moisture. As the friction force is defined as the real area of contact multiplied by the shear stress of the film, this would indicate that the shear stress is the variable of interest.

The friction force is hypothesized to be correlated water sorption.



Quartz Crystal Microbalance's (QCM) work using the piezoelectric effect from the quartz crystal. Alternating currents are applied to the crystal which creates a standing shear wave. The frequency of oscillation of the quartz crystal is dependent upon the mass of the crystal, thus if a change of mass occurs, the frequency will change correspondingly.

The Sauerbrey's Equation is used to calculate mass change per unit area:

$$\frac{\Delta m}{A} = -\frac{\Delta f}{2f_0^2} \sqrt{\mu_q \rho_q}$$

## QCM Studies

The mass of water sorption into a  $\text{MoS}_2$  film per exposed area at various partial pressures of water can be studied.

As the partial pressure of water is varied, the friction can be measured as well. Allowing a correlation between mass of the water on the film and the friction coefficient to be made.

*Image courtesy of Inficon*

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