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Development of Nanocrystalline Metal Oxides and Sulfides for Extreme Tribological Applications using High Throughput Analysis

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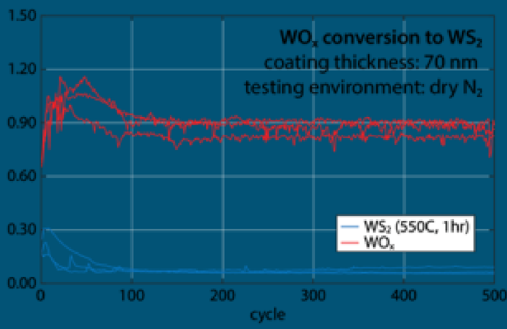
Introduction, Background, Method

Solid lubricants represent an area of significant opportunity both for Sandia's nuclear deterrence mission and the broader scientific community. New solid lubricant materials and improved processes have the potential to improve performance by mitigating long standing age-related degradation mechanisms, improving conformality and reducing the sensitivity of these materials to operating atmospheres. Current state of the art manufacturing processes for lubricant integration involve mechanical impingement or burnishing [1 - 3] which lack the control of thickness, uniformity and repeatability required for modern precision mechanisms. Physical vapor deposition (PVD) methods offer excellent thickness control and repeatability but have difficulty with high aspect ratio features. We propose to use plasma enhanced atomic layer deposition (ALD) and high throughput friction analysis to develop advanced solid lubricants.

Proof of concept was demonstrated using ALD coatings of tungsten oxide and molybdenum oxide and a 550 ºC sulfide conversion temperature. The figure below shows successful conversion of MoO_x and WO_x to MoS_2 and WS_2. The high throughput testing demonstrates that it's repeatable across multiple samples and wear tracks on the same sample. This provide to motivation to move towards lowering the thermal budget for conversion. The end goal being to develop a deeper understanding of the conversion kinetics and its influence on tribological performance and from an application standpoint reduce the thermal budget to allow for integration, of these materials and processes, on engineering substrate such as metals coupons, parts, and assemblies.

1) 

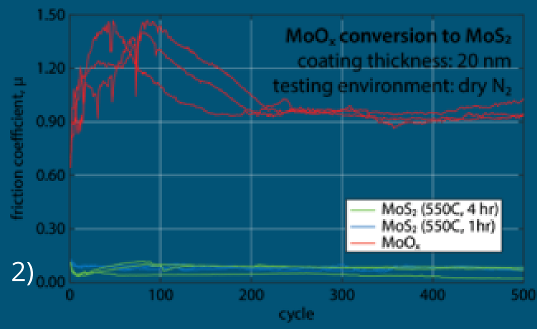
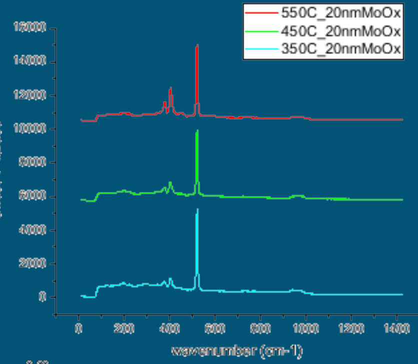
2) 

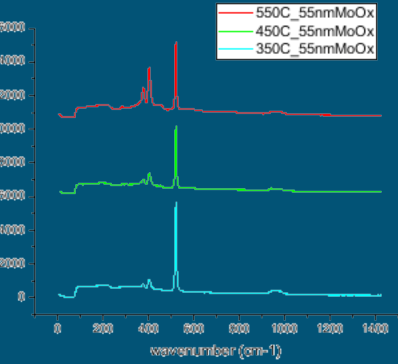
Figure 1 & 2 - Average cycle friction coefficients for (a) WOx converted to WS2 and (b) MoOx converted to MoS2 at 550C at two different annealing durations.

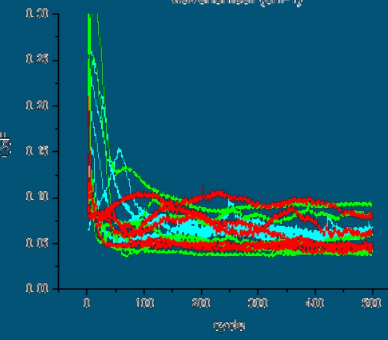
Reference:

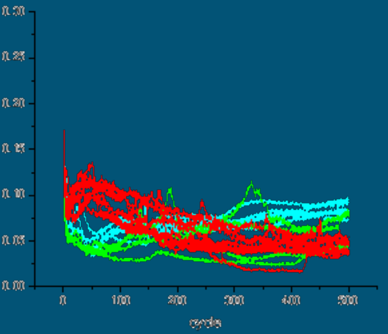
1. Jurca, Titel et al. 2017. "Low-Temperature Atomic Layer Deposition of MoS_2 Films." *Angewandte Chemie* 56 (18): 4991–4995. doi: 10.1002/anie.201611838.
2. Scharf, Thomas. W. et al. 2009. "Atomic Layer Deposition of Tungsten Disulphide Solid Lubricant Nanocomposite Coatings on Rolling Element Bearings." *Tribology Transactions* 52 (3): 284–292. doi: 10.1080/10402000802369747.
3. Mattinen, Miika et al. 2017. "Atomic Layer Deposition of Crystalline MoS_2 Thin Films: New Molybdenum Precursor for Low-Temperature Film Growth." *Advanced Materials Interfaces* 4 (18). doi: 10.1002/admi.201700123.

Results and Direction

3) 

4) 

5) 

6) 

Figures 3 & 4 show the Raman Spectra at three conversion temperatures and two thickness, the spectra are vertically shifted for better visualization. This data shows peak intensity grows at higher conversion temperature only with the 55nm thick sample which suggests that the 20 nm sample is fully converted at all temperatures.

Figure 5 & 6 show the Tribological performance is a function of coefficient of friction at three conversion temperatures and two thicknesses. In general the, 20 and 50 nm MoS2 converted samples exhibit similar behavior. Steady state friction decreases with increasing conversion temperature, inverse that of initial friction. And the conversion temperature dependence is only now emerging at the thicker (55nm) film relative to thinner (20nm) MoS2 converted sample. Which again suggests the thinner samples are fully converted.

Next we are looking at getting TEM/XRD sections to look a conversion depth and structure. And continue to drive toward lower thermal budgets.

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