SAND2022-8490C

Unlocking the Potential of Pure MoS_2 Coatings

Tomas Babuska[1,2,3], John F Curry[3], Ryan Thorpe[2], Michael T. Dugger[3], Frank W. DelRio[3], Morgan Jones[3], Nicholas C. Strandwitz[5], Md Istiaque Chowdhury[5], Robert Chrostowski[6,7], Filippo Mangolini[6,7], Tomas Grejtak[1,2], Kylie Van Meter[1], Gary L. Doll[4], and Brandon Krick[1].

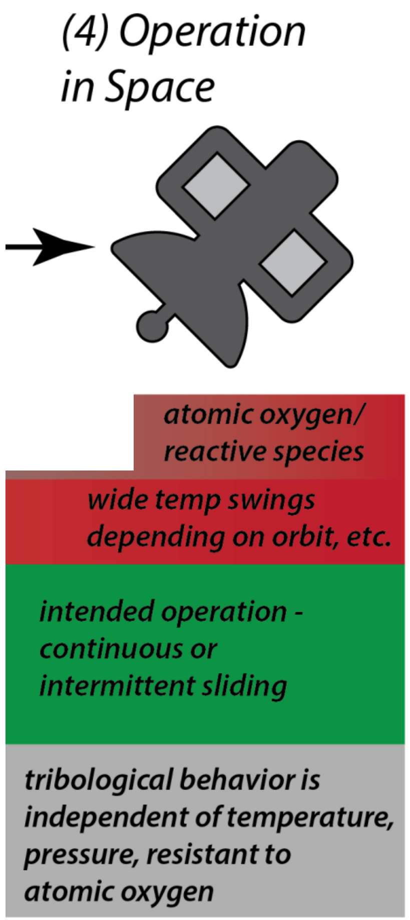
[1]*Florida State University, Department of Mechanical Engineering*
[2]*Lehigh University, Department of Mechanical Engineering and Mechanics*
[3]*Sandia National Laboratories, Material, Physical and Chemical Sciences Center*
[4]*The University of Akron, College of Engineering and Polymer Science*
[5]*Lehigh University, Department of Materials Science and Engineering*
[6]*The University of Texas at Austin, Walker Department of Mechanical Engineering*
[7]*The University of Texas at Austin, Texas Materials Institute*

Sandia National Laboratories is a multimission laboratory managed and operated by National Technology & Engineering Solutions of Sandia, LLC, a wholly owned subsidiary of Honeywell International Inc., for the U.S. Department of Energy's National Nuclear Security Administration under contract DE-NA0003525.

Designing Robust Coatings for Space Applications

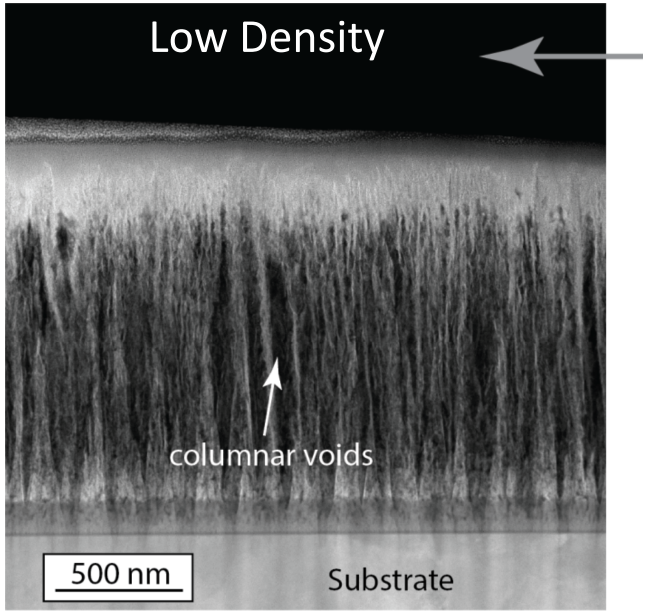
- There is a renewed interest in space ranging from tourism to exploration and colonization
- When designing a coating, we must think of operating environments beyond the intended end use
- Coatings must maintain their tribological performance from lab to space
- Molybdenum Disulfide (MoS_2) is an ideal solid lubricant for space
- Use of sputter deposited MoS_2 has been limited due to ***high variability*** in the tribological performance

Life Cycle of a Space Coating



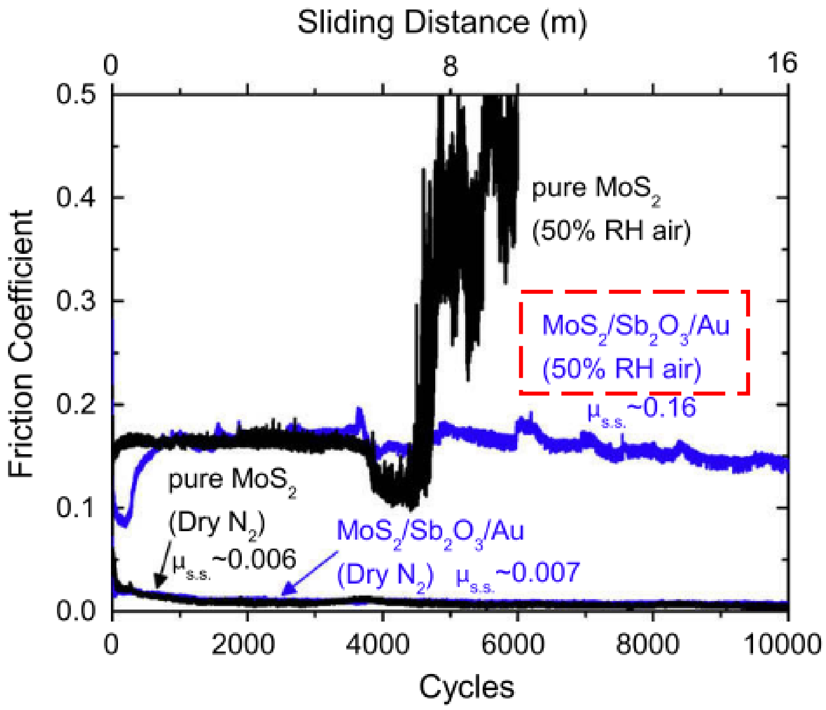
Pure MoS_2 Films and Porosity

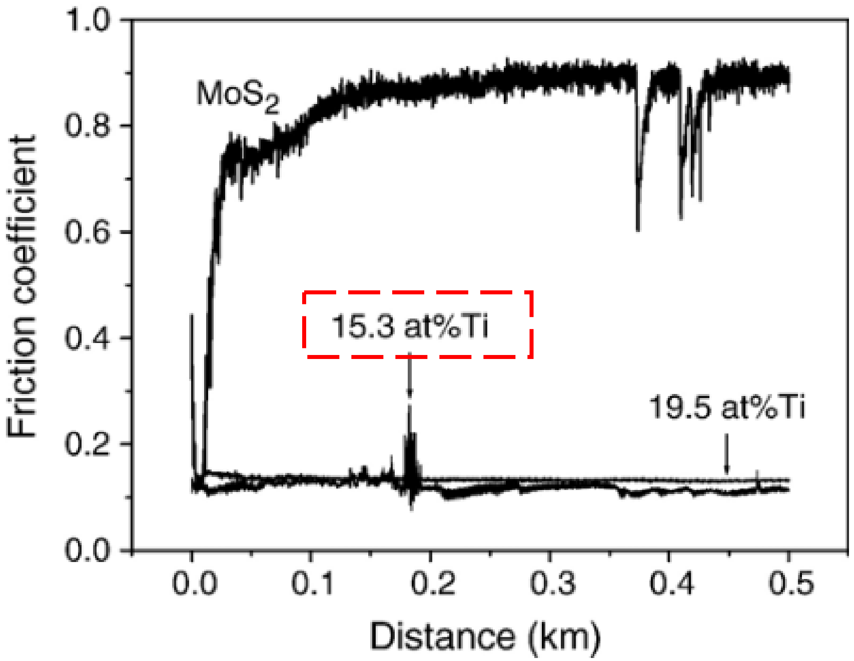
- Pure MoS_2 coatings deposited with PVD techniques are prone to forming voids

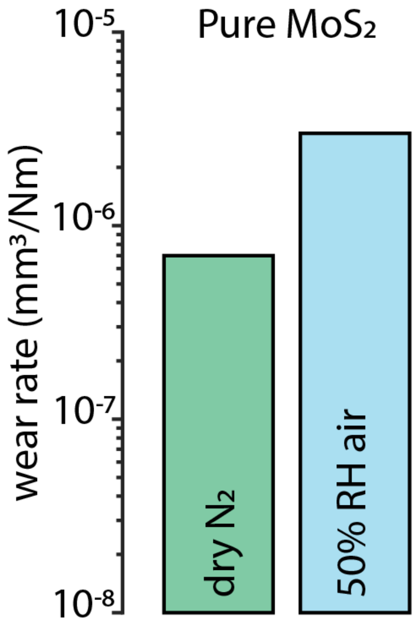


Overcoming Performance Issues with Dopants

- Pure MoS_2 coatings deposited with PVD techniques are prone to forming voids
- Low density pure MoS_2 coatings have high wear rates and can fail when exposed to water
  - Adding dopants to films such as Au, Sb_2O_3, Ti, Ni improve wear life in humid environments



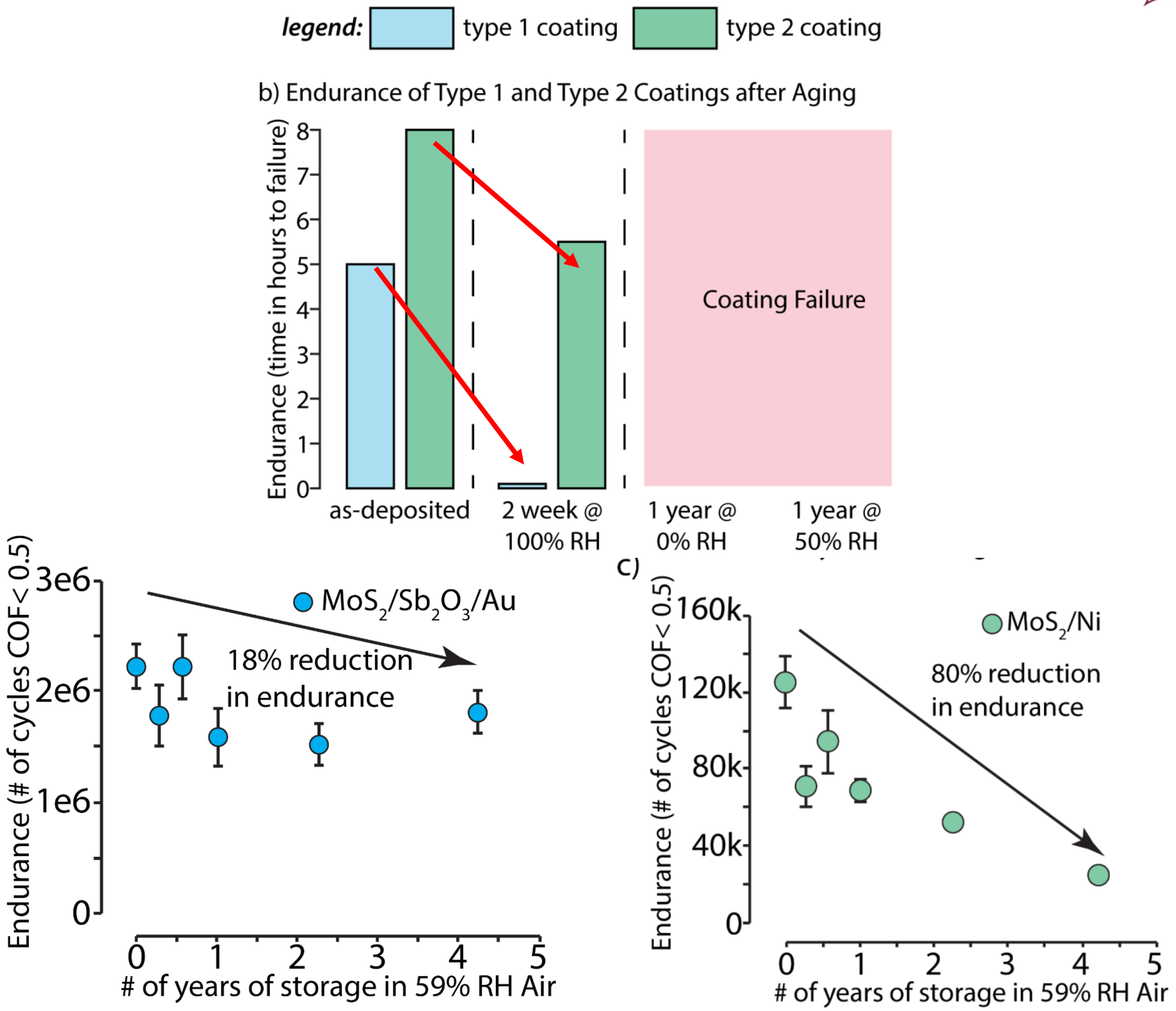




Role of Dopants on Aging Behavior

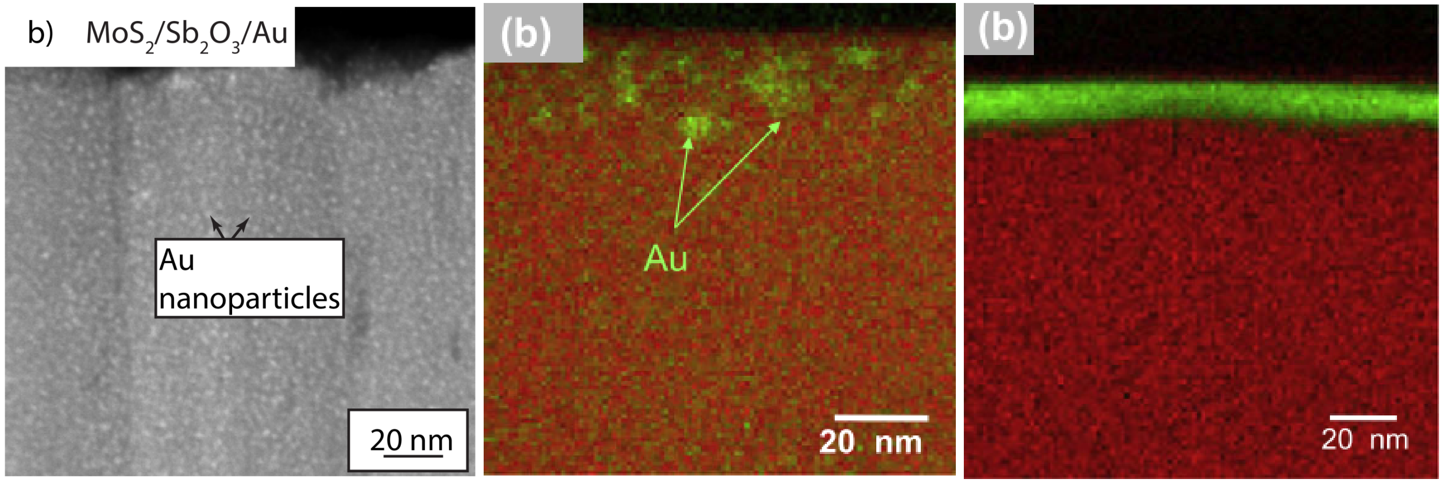
- Pure MoS_2 is susceptible to oxidation from water and oxygen over long periods of time
  - Oxidation decreases endurance and causes failure

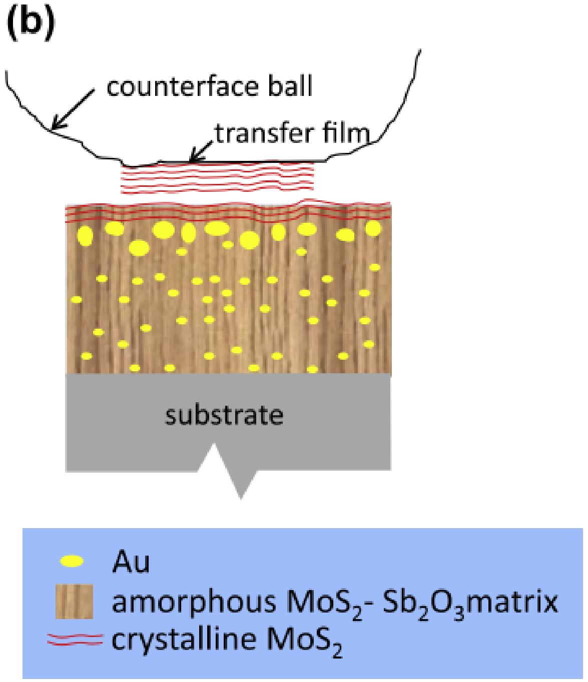
- Doped films can maintain performance after years of storage
  - Improvements depend on dopant used



Why are Doped Coatings more Wear Resistant?

1. Dopants densify coatings increasing hardness
2. Improve adhesion to the substrate
3. Sliding causes sub-surface coarsening of dopant nanoparticles
    1. Stabilize sliding interface
    2. Minimize interaction with H_2O and O_2 by preventing diffusion into the coating





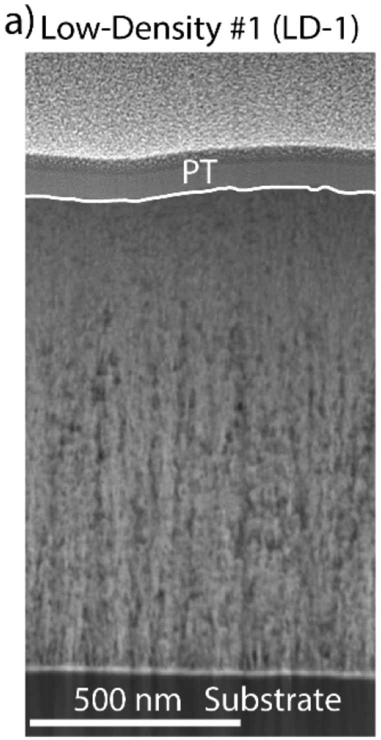
Can pure films achieve performance of doped films if highly dense?

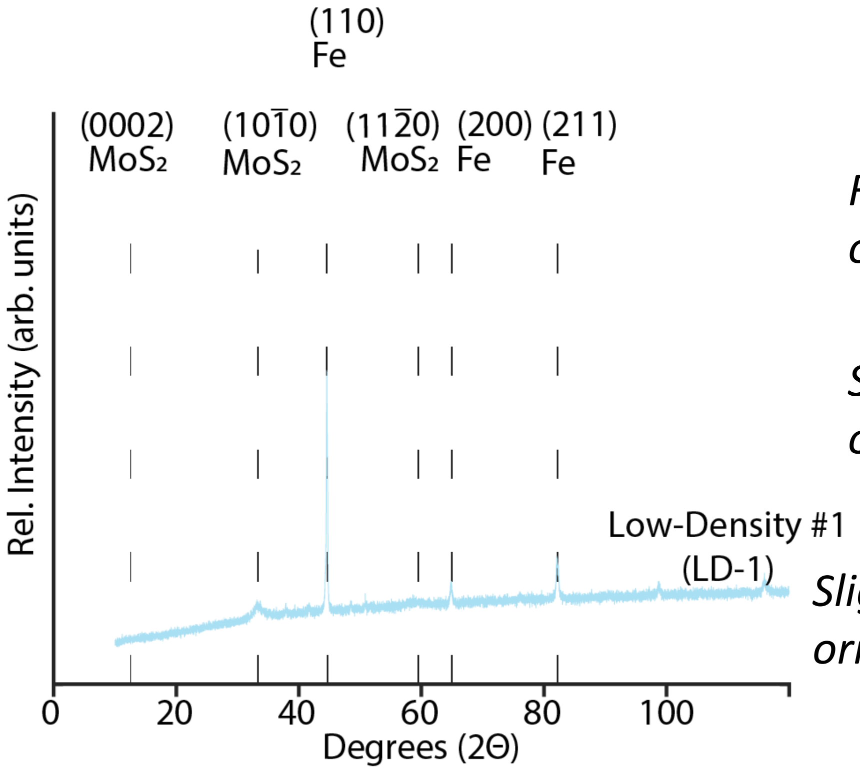
The Making of a Dense Pure MoS_2 Coating

Problem #1: ***Variation in coating morphology between deposition batches creates unreliable MoS_2 coatings***

Batch 1

Porous





Randomly oriented

Slight basal orientation

Slight vertical orientation

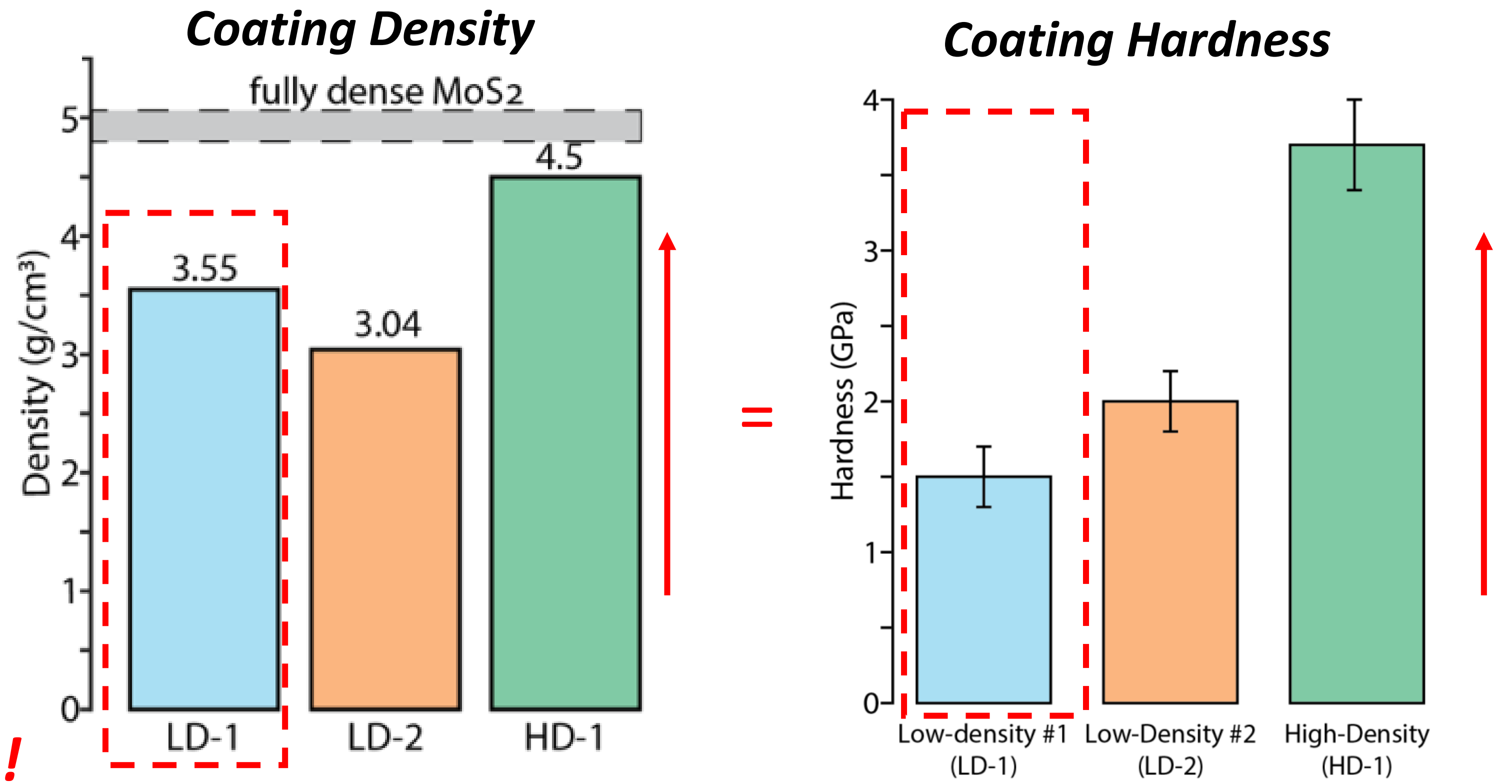
3 different coatings yet deposition parameters were identical!

Hypothesis: Density is a driving factor for the tribological/mechanical behavior of MoS_2 coatings

- Hardness and wear rate depend on coating density

- Increased density = increased hardness
  - Except for the low-density #1 coating

- Low-density #1 = columnar
- Low-density #2 = basally-oriented
- Hardness depends on orientation



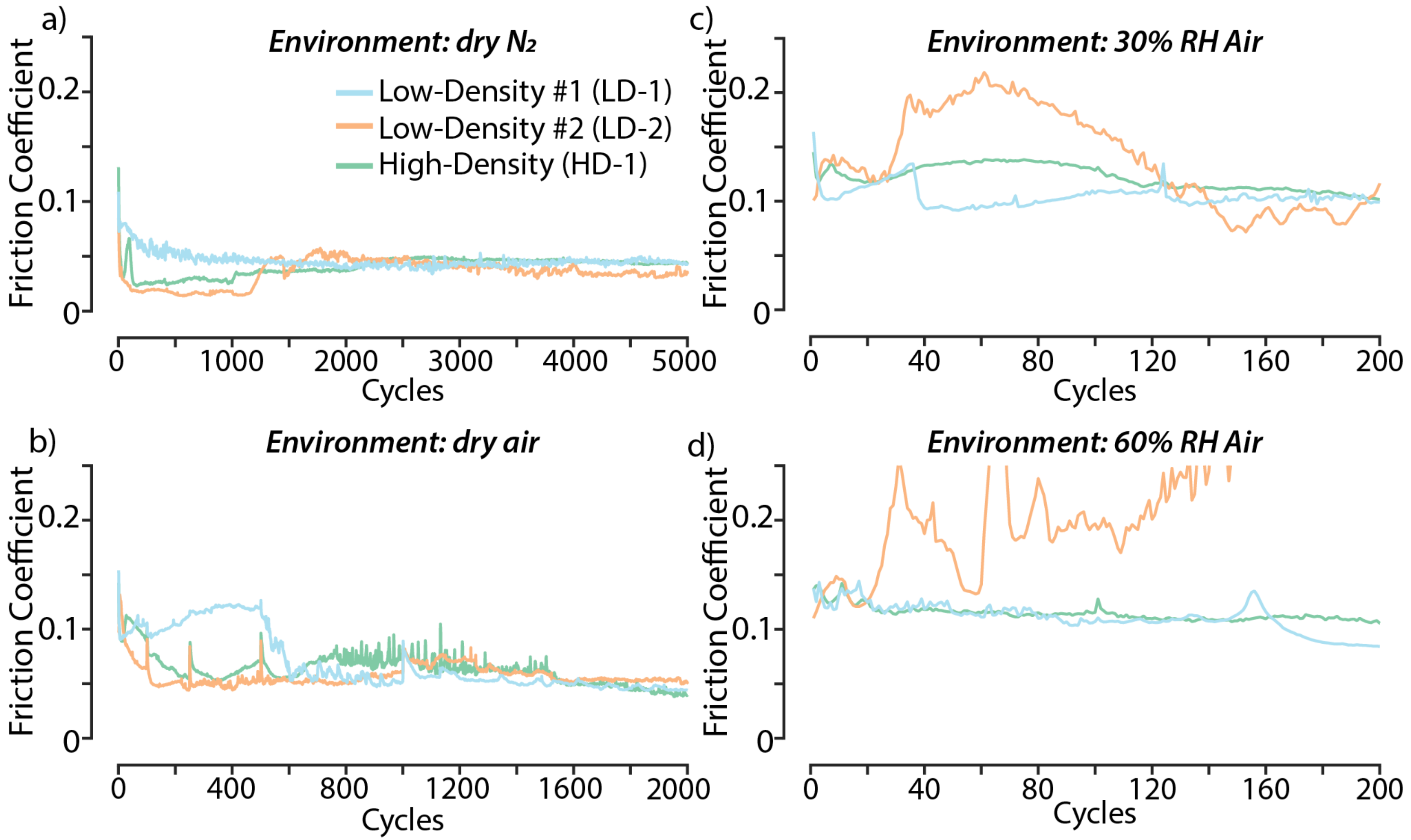
Dense coatings have higher hardness!

Impact of Coating Density on the Friction and Wear Behavior

Takeaways:

- ***High density coatings have lower wear rates in air and N2 than low-density coatings***
- ***In dry environments, dense pure films are comparable or better than composite films!***

- Overall, the COF does not significantly vary between coatings
  - Wear rate does, especially in different environments
- In dry N_2, *highest density* coating = *lowest wear rate* (~5×10^{-8} mm^3/Nm)
  - *As low or lower than composite films!*
- In dry air, 10-100x increase in wear rate for low-density films
  - Only 2x for high-density
- In 30% Rh air, large increase in wear rate for high-density film
  - ~10x lower than low-density coatings
- Highest humidity (60% RH) = highest wear rates



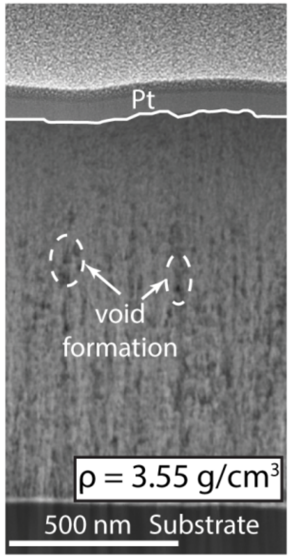
Coating Density is an Important Factor for Aging

- High-temperature oxidation of coatings in HS-LEIS
  - 30 minutes at 250° 1 mbar O_2
  - 720 minutes at 250° 10 mbar O_2
- Depth-profiling of as-deposited coatings

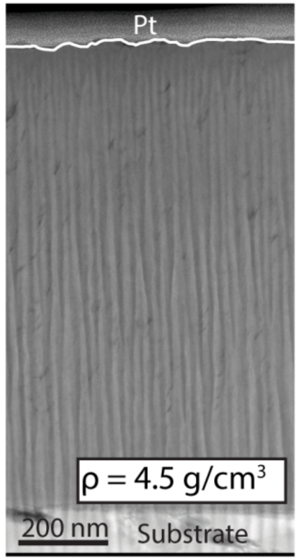
- After 30 min:
  - Mild surface oxidation ~5 nm into the low-density coating
  - Less O in top ~10 nm of the high-density coating

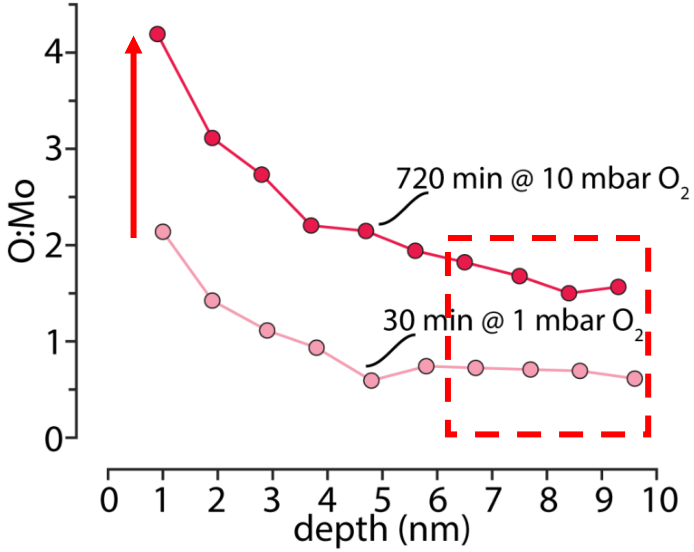
- After 720 min:
  - Surface of low-density coating highly oxidized
  - O penetrates low-density coating
  - High-density coating is unaffected

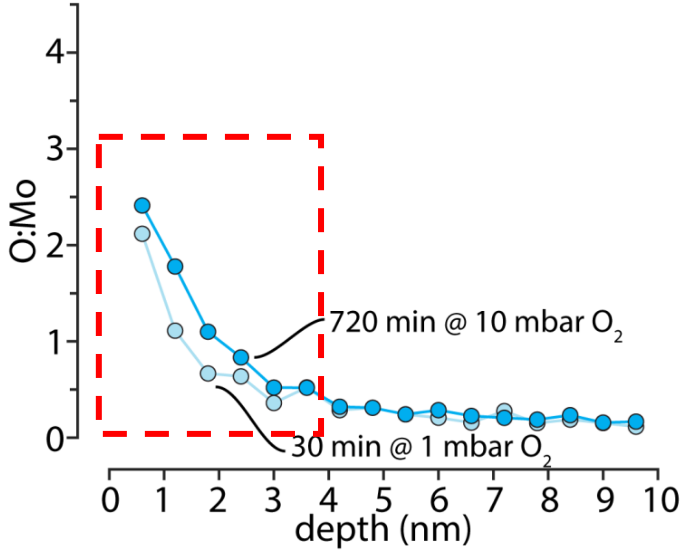
Low-Density Coating



High-Density Coating



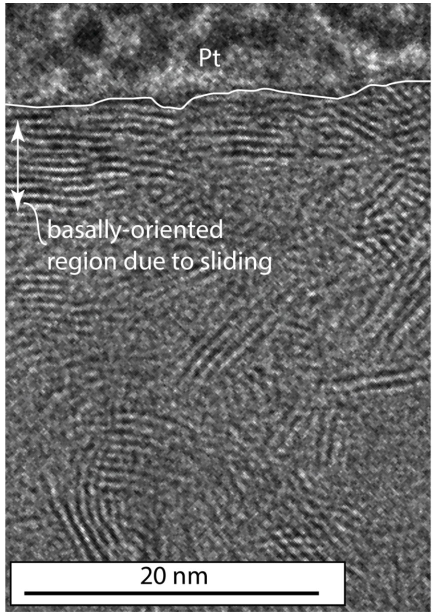




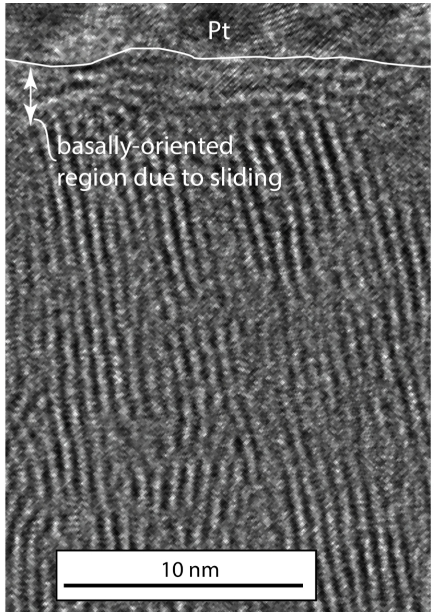
Sliding Helps Improve Aging Resistance for Low-Density Films

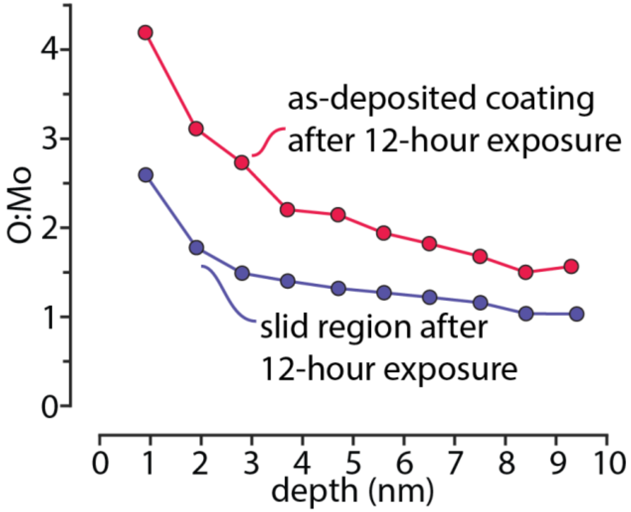
- Sliding results in a basally-oriented surface layer
  - Oriented layer forms on coating regardless of density
- Remember, low-density coatings are susceptible to oxidation
  - Voids provide pathways into the coating
  - Exposure of reactive edge-sites
- LEIS depth-profiles show that sliding on low-density coatings:
  - Limits severity of surface oxidation
  - Limits sub-surface oxidation

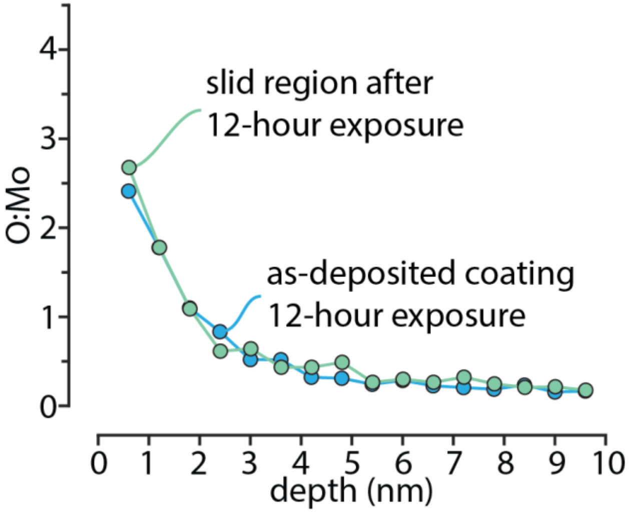
Low-Density Coating w/sliding



High-Density Coating w/sliding



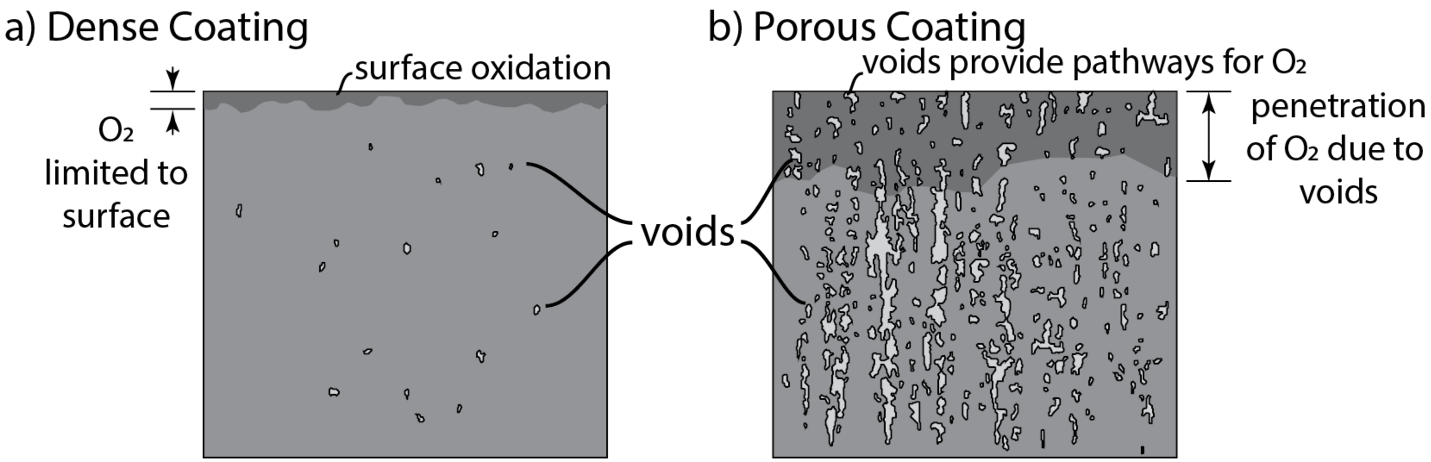




Dense Films Limit Oxidation to the Surface

Takeaways:

1. ***Density has a large impact on oxidation resistance***
2. ***Voids in low-density coatings provide pathways for oxygen***
3. ***Sliding on low-density coatings improves aging resistance***



Conclusions: Have we Unlocked the Potential of Pure MoS_2?

- Density is a key factor driving the tribological and aging behavior for MoS_2 coatings
  - Dense films can achieve ultra-low wear rates and limit oxidation from aging
- Wear rate and hardness are key indicators of film density that can be used to screen coating batches
  - TEM etc. can get expensive and is time-consuming
- We cannot repeatably and intentionally manufacture dense pure coatings with sputtering techniques
  - As tribologists, to often do we use third-party manufactures and do not have enough control over the processing side of the materials we make
  - This limits our ability to explore process-structure-property relationships
- To maximize the full potential of pure films, we need to make our own coatings!

Acknowledgments

Krick Career on Lamellar Lubricity: #2027029

NSF GRFP: #1842163

This work was funded by the Laboratory Directed Research and Development (LDRD) program at Sandia National Laboratories, a multi-mission laboratory managed and operated by National Technology and Engineering Solutions of Sandia, LLC., a wholly owned subsidiary of Honeywell International, Inc., for the U.S. Department of Energy's National Nuclear Security Administration under contract DE-NA0003525.