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Quality Control Metrics to Assess MoS_2 Sputtered Films for Tribological Applications

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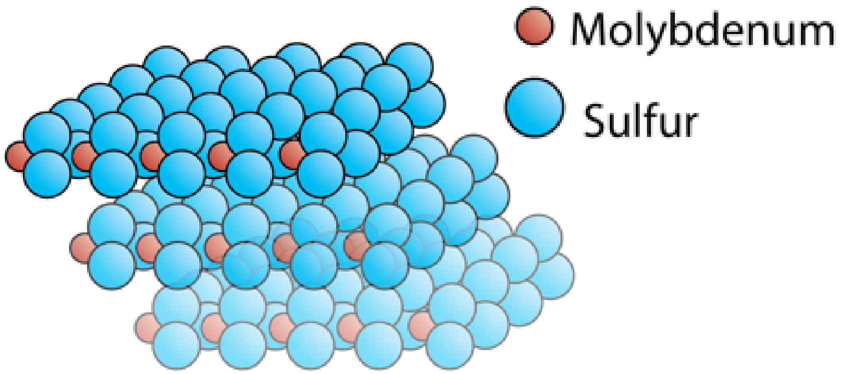
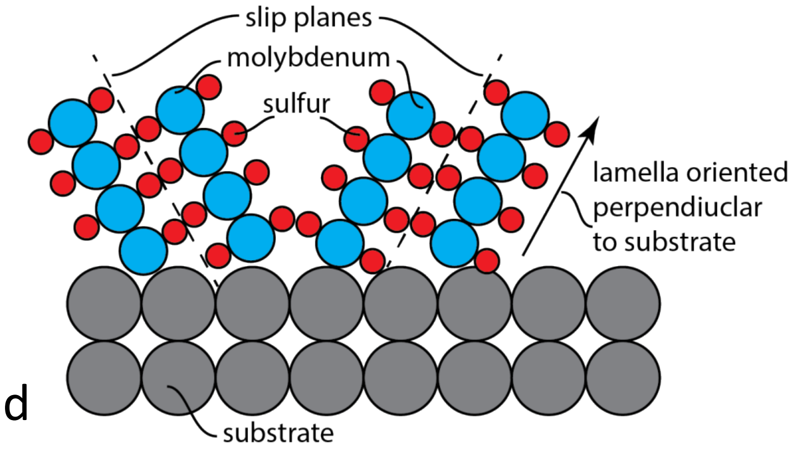
Introduction – MoS_2 Coating Morphology: Orientation

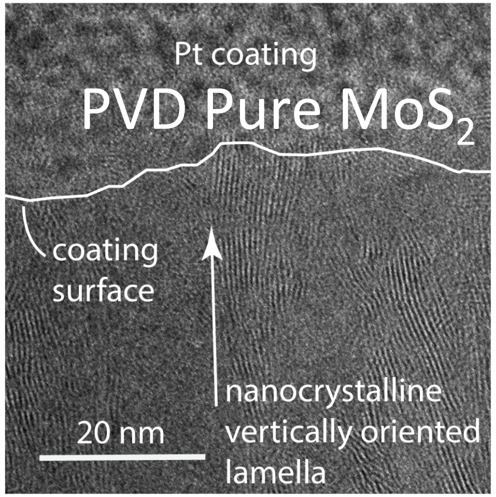
- MoS_2 – 2D lamellar lubricant, performance depends on structure
- Deposition methods affect coating morphologies:
  - Orientation
    - Columnar/Vertically-oriented (surface perpendicular)
    - Basally-Oriented (surface parallel)



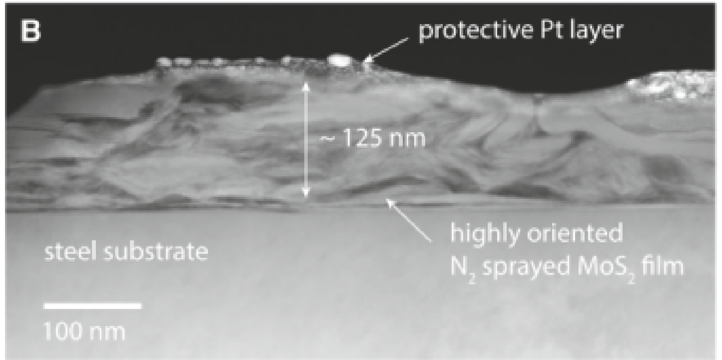
a) Type 1 MoS2 Coating: Vertically-Oriented



Nanocrystalline



Basally-oriented

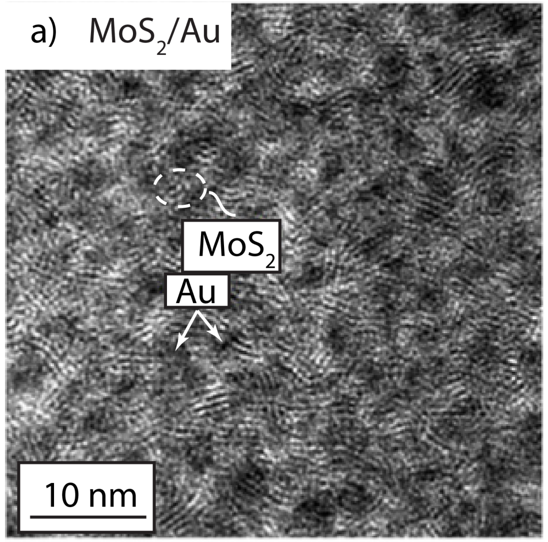


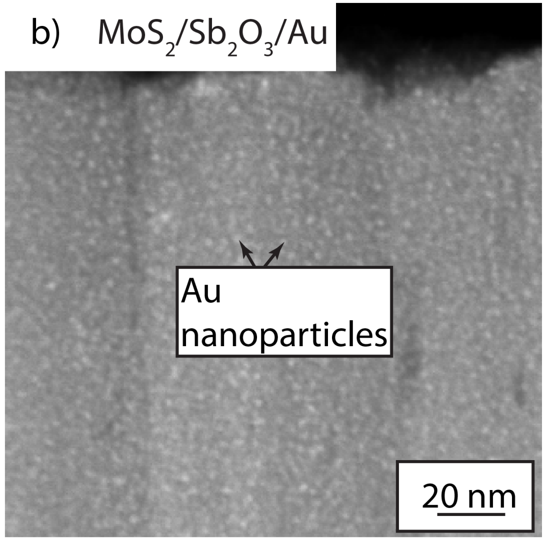
Introduction – MoS_2 Coating Morphology: Composition

- MoS_2 – 2D lamellar lubricant, performance depends on structure
- Deposition methods affect coating morphologies:
  - Composition
    - Pure
    - Composite – added dopants such as Au, Sb_2O_3, Ti etc.

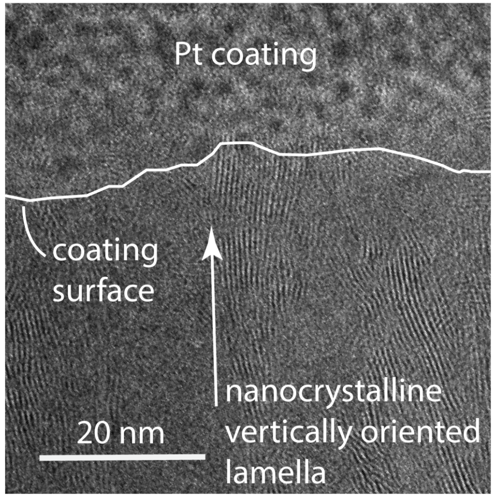
Question: *How does coating morphology affect tribological performance of pure films?*

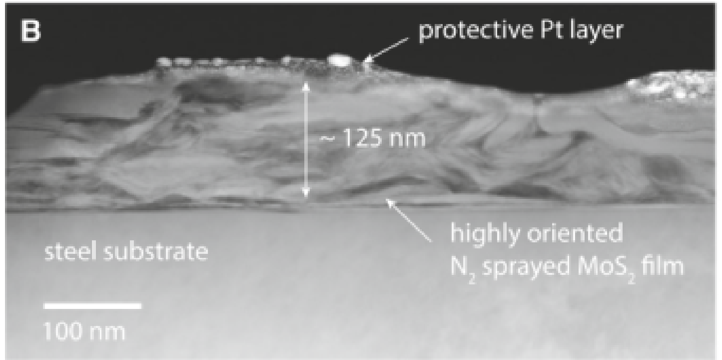
MoS_2 w/dopants





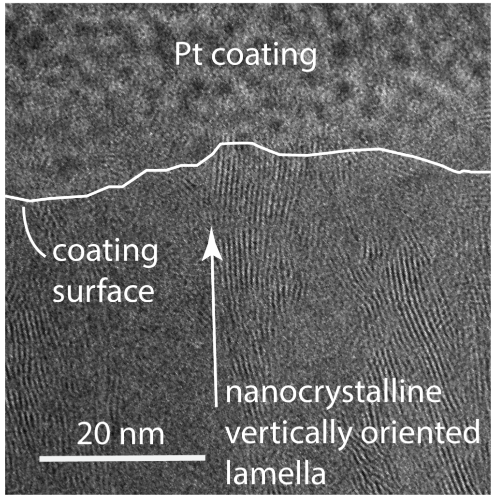
Pure MoS_2

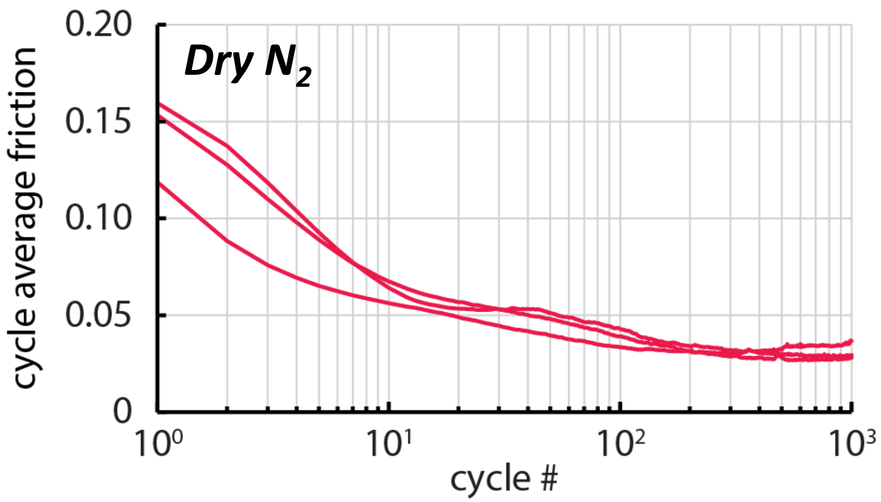




- Orientation and size of lamella impacts friction coefficient
- Basal orientation:
  - Decreases initial COF and run-in

Randomly-oriented/Columnar

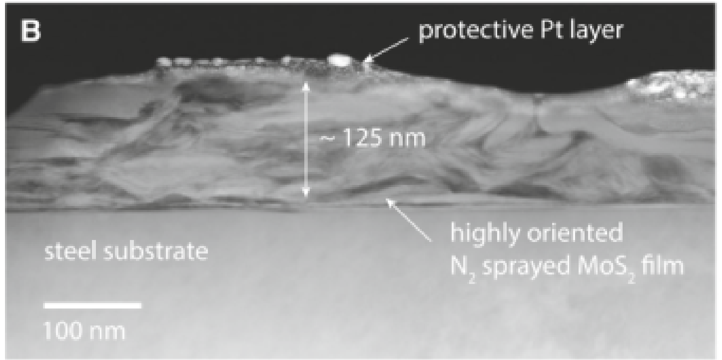


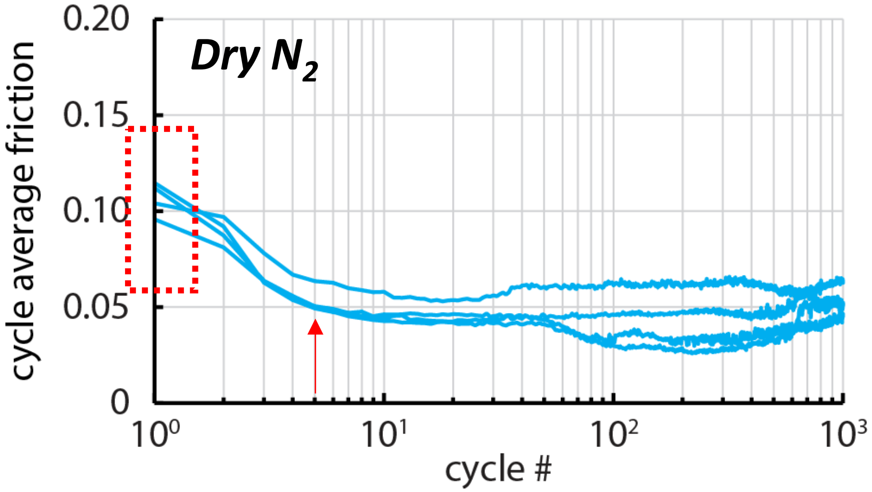


Tribological Behavior of a quality coating

1. *Low initial COF*
2. *Fast run-in*

Basally-oriented





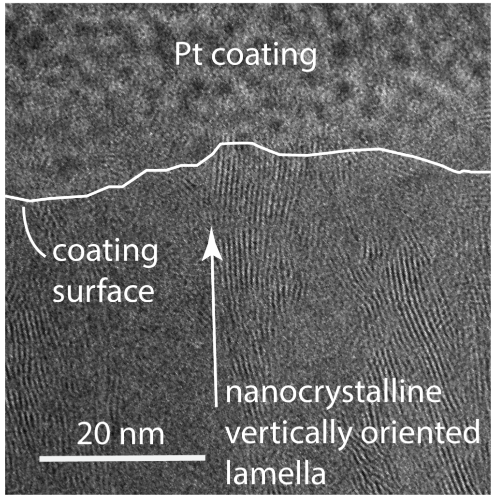
Introduction – Tribological Properties and Morphology

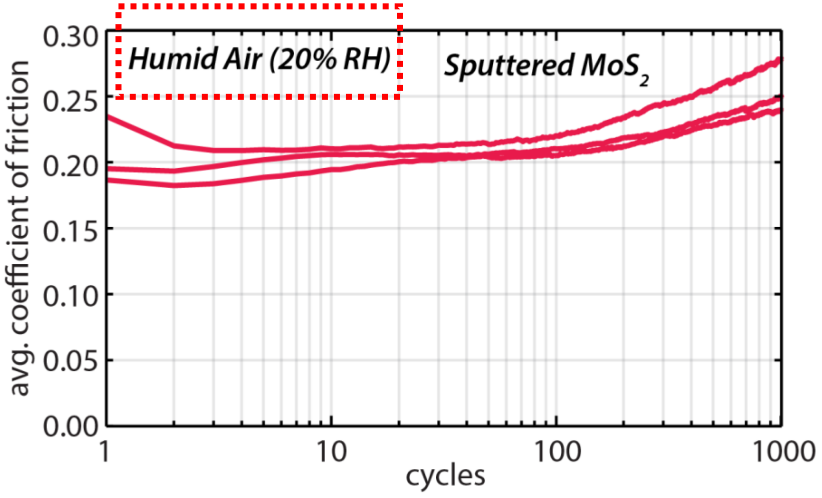
- Orientation and size of lamella impacts friction coefficient
- Basal orientation:
  - Decreases initial COF and run-in
  - Lower COF in humidity

Tribological Behavior of a quality coating

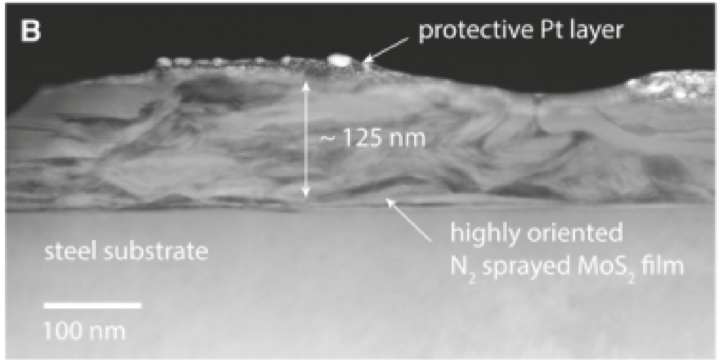
1. *Low initial COF*
2. *Fast run-in*
3. *Low COF in humid environments*

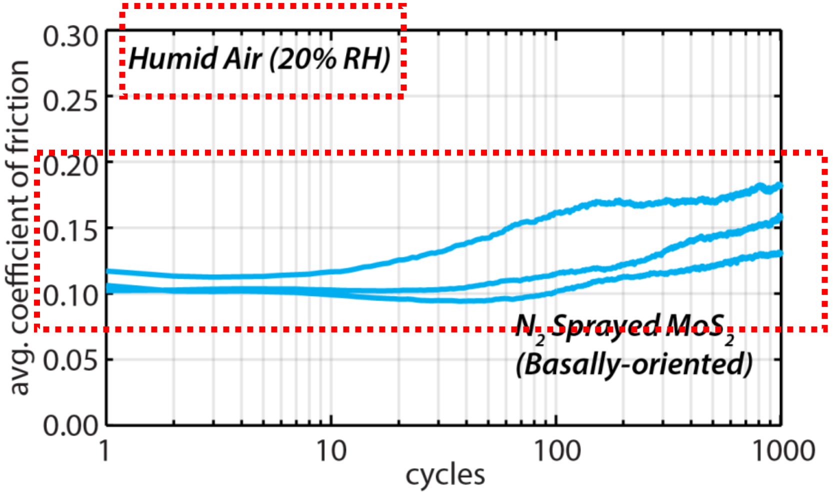
Randomly-oriented/Columnar





Basally-oriented



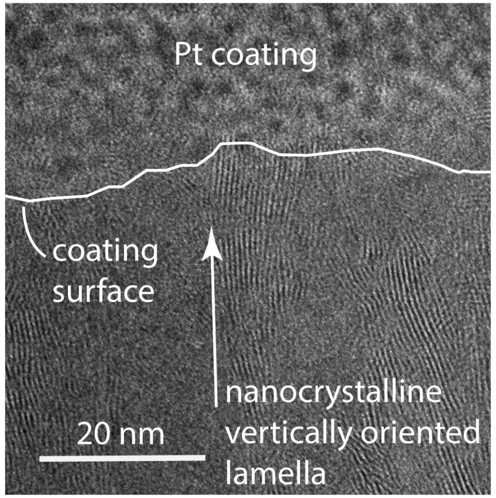


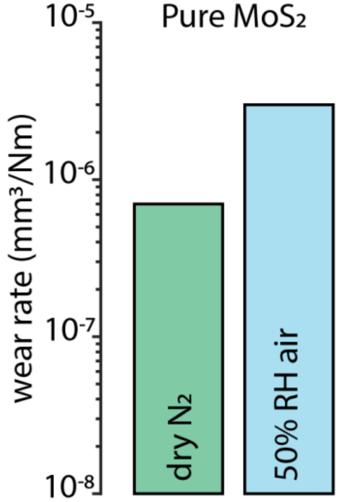
- Pure coatings are susceptible to high wear in humid environments
- Dopants improve wear resistance in humid and dry environments:
  - Densification of coating

Tribological Behavior of a quality coating

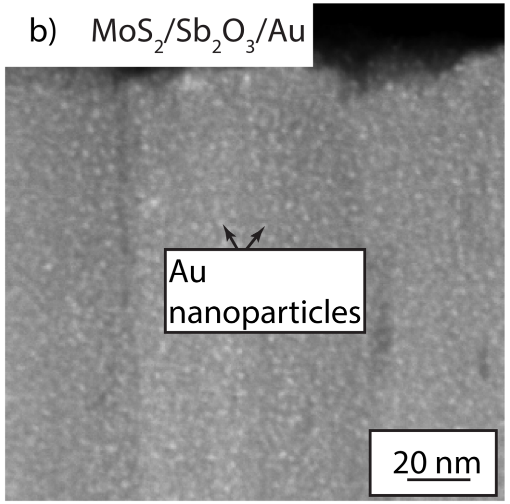
1. *Low initial COF*
2. *Fast run-in*
3. *Low COF in humid environments*
4. *Resistant to wear in different environments*

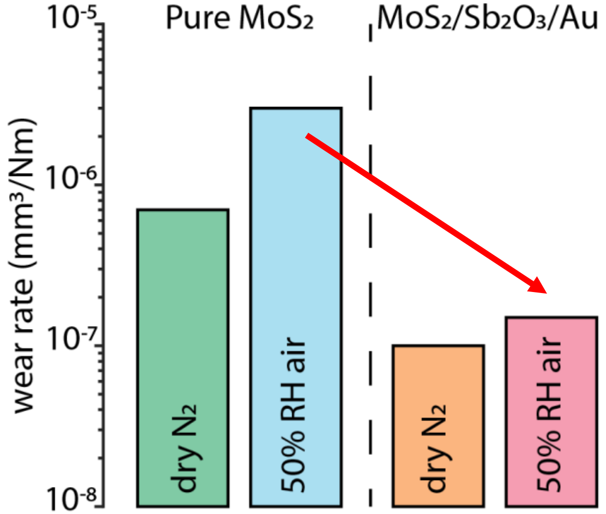
Pure MoS_2 Coatings





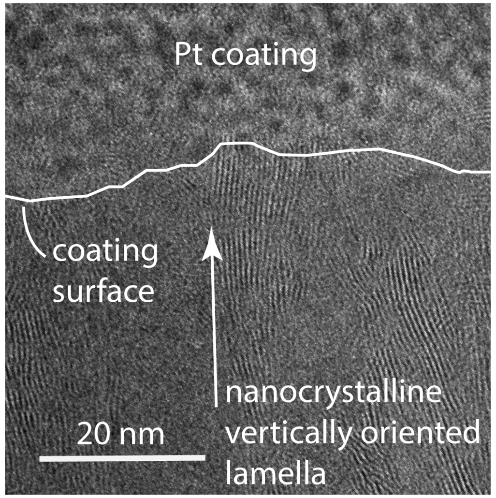
Doped MoS_2 Coatings

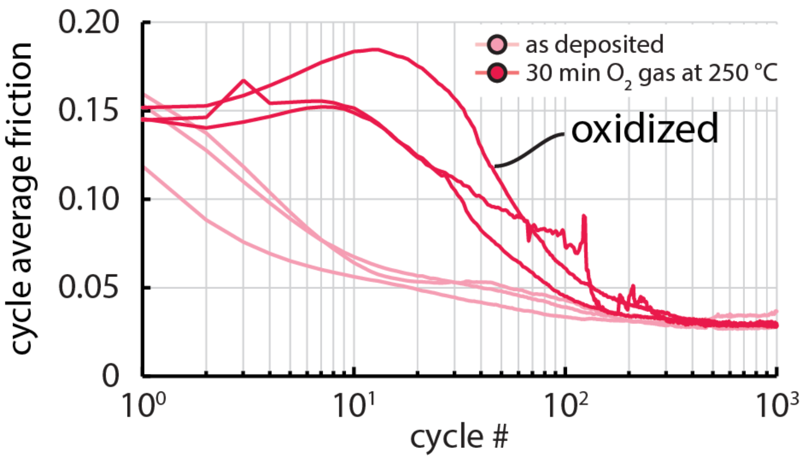




- Storage of MoS_2 coatings in water and oxygen (aging) changes performance
- Increased COF and prolonged run in to steady-state friction
- Decreased coating endurance (higher wear rate)

Randomly-oriented/Columnar

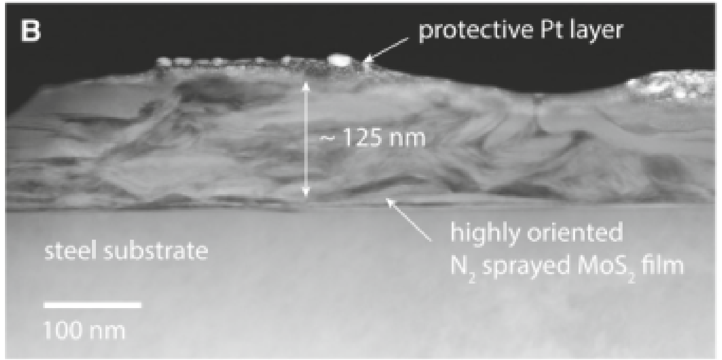


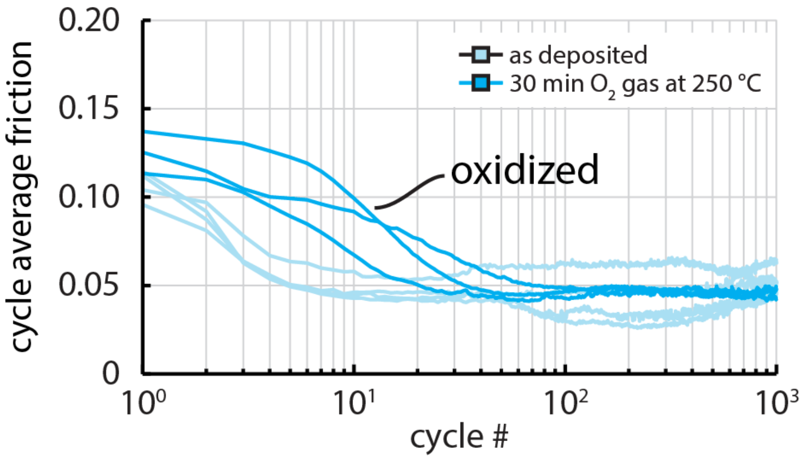


Tribological Behavior of a quality coating

1. *Low initial COF*
2. *Fast run-in*
3. *Low COF in humid environments*
4. *Wear resistant in different environments*
5. *Limits changes from oxidation (aging)*

Basally-oriented





Introduction – Oxidation Resistance and Morphology

- Orientation effects coatings resilience towards oxidation:
  - ***Columnar*** microstructures provide pathways exposed edge sites increasing oxidation
  - ***Oriented*** microstructures limit surface oxidation and depth

Tribological Behavior of a quality coating

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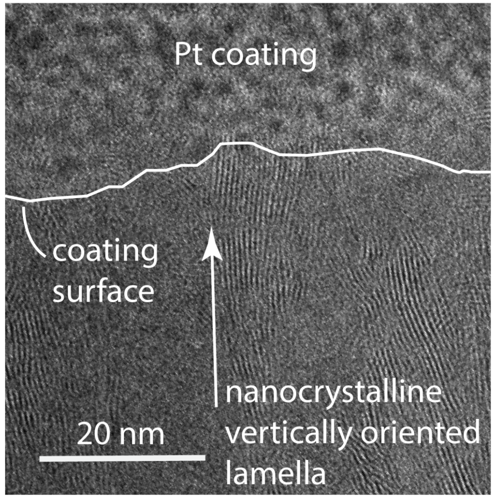
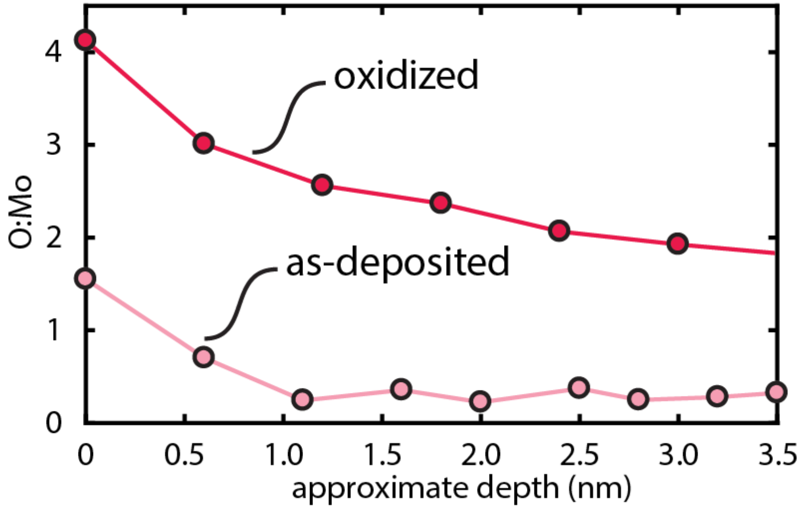
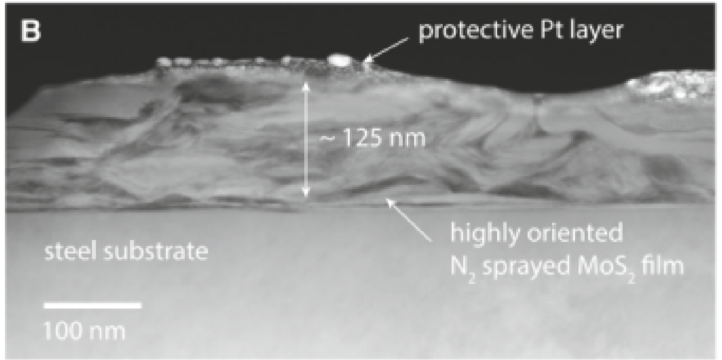
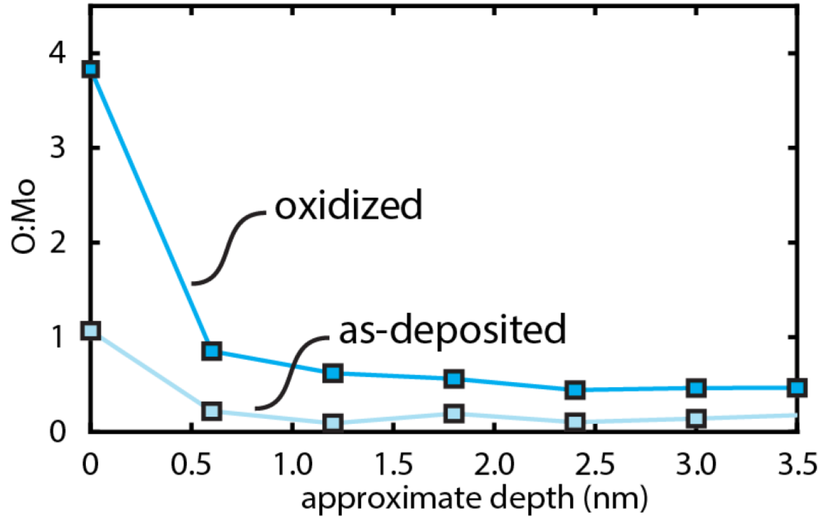
Randomly-oriented/Columnar





Basally-oriented





The Making of a Quality Pure MoS_2 Coating

Tribological Behavior of a quality coating

1. *Low initial COF*
2. *Fast run-in*
3. *Low COF in humid environments*
4. *Wear resistant in different environments*
5. *Limits tribological changes from oxidation*

Characteristics of a quality coating

1. *Basally-oriented (surface parallel lamella)*
2. *Nanocrystalline*
3. *Highly-dense (no voids or porosity)*
4. *Low in contaminants (oxygen, hydrocarbons)*

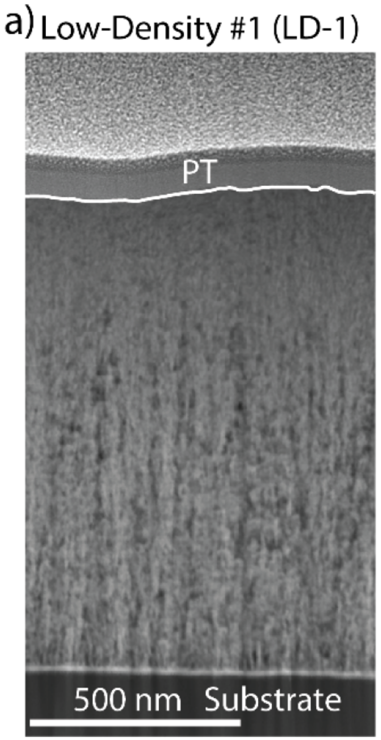
Time to deposit a coating with these characteristics! Easy right….

The Making of a Quality Pure MoS_2 Coating

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

Batch 1

Porous



Characteristics of a "quality" coating

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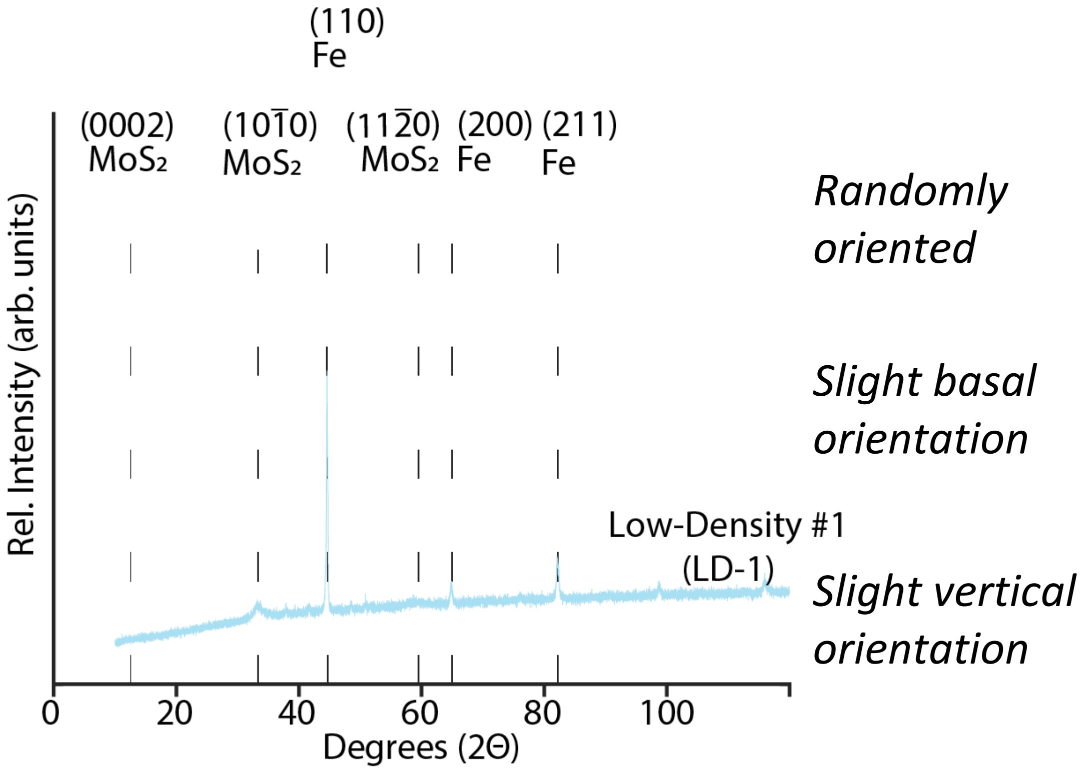


3 different coatings yet deposition parameters were identical!

Metrics to Determine Coating Quality: Density and Hardness

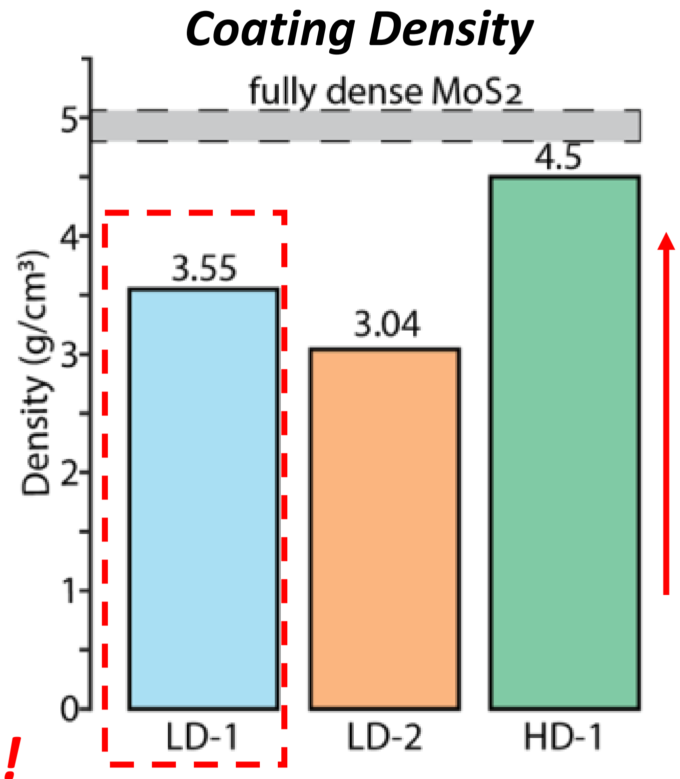
Requirement: Developing a metric to quantify coating "quality" will allow for screening of MoS_2 coating batches

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



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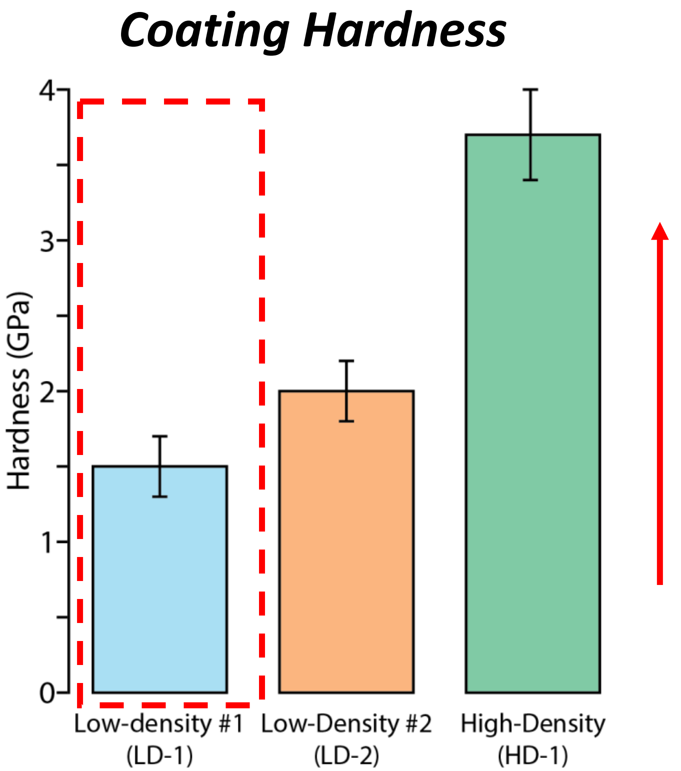


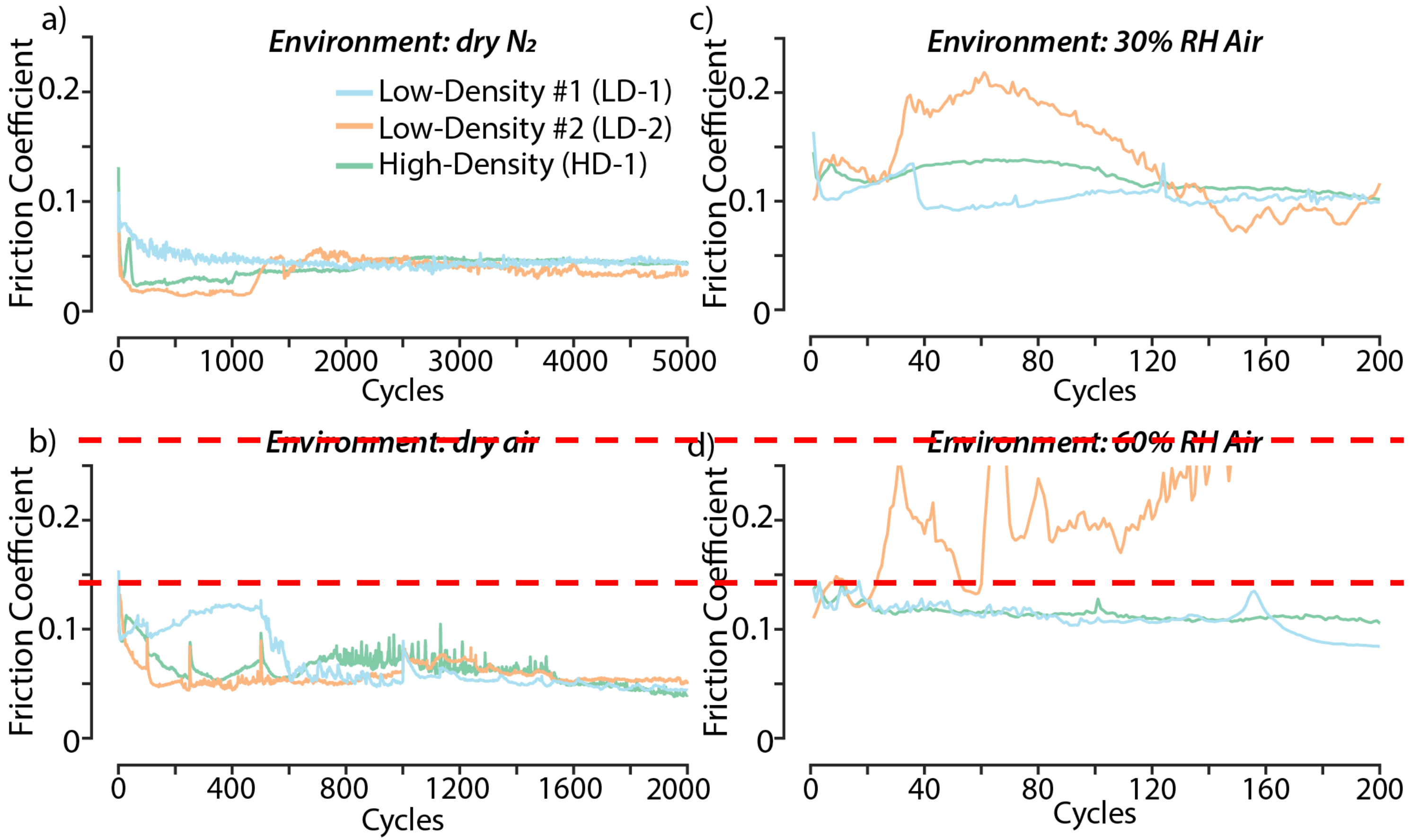
Dense coatings have higher hardness!

Metrics to Determine Coating Quality: Friction and Wear

Takeaways:

- ***High density coatings have lower wear rates in air***
- ***Meeting a threshold in wear rate can help define a quality coating***

- 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* (~$5x10^{-8}$ mm^3/Nm)
- 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



Role of Density on the Oxidation of MoS_2 using HS-LEIS

So far, we have studied the relationship between density, hardness, friction and wear

↑ density = ↑ hardness = ↓ wear rate

Tribological Behavior of a "quality" coating

1. *Low initial COF*
2. *Fast run-in*
3. *Low COF in humid environments*
4. *Wear resistant in different environments*
5. *Limits tribological changes from oxidation*

- Literature shows that basally-ordered films are more resistant to oxidation
- ***Question:*** How does density impact oxidation behavior?

Perform accelerated aging on coatings with different densities

- Use HS-LEIS depth profiling to measure oxidation
- Elemental info of top monolayer

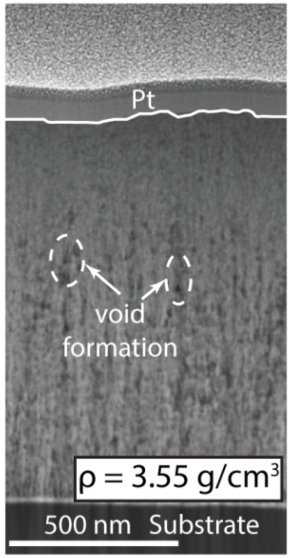
Coating Density is an Important Factor for Aging

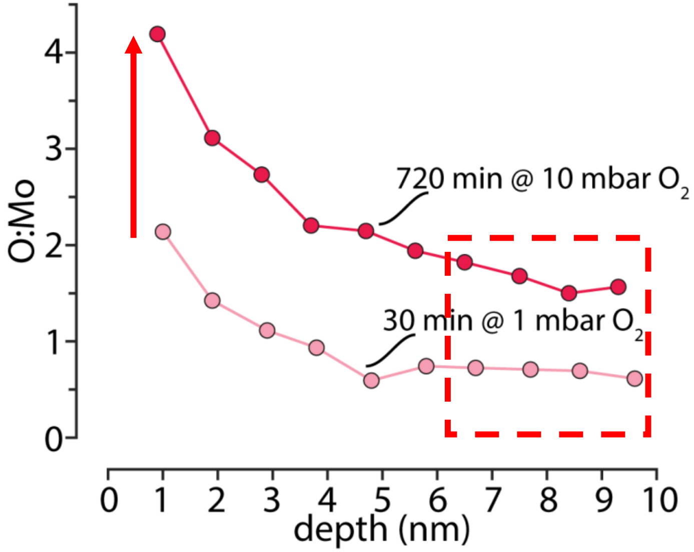
- High-temperature oxidation of coatings in HS-LEIS
  - 30 minutes at 250° 1 mbar O_2
  - 720 minutes at 250° 10 mbar O_2
- Depth-profiling of as-deposited coatings

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

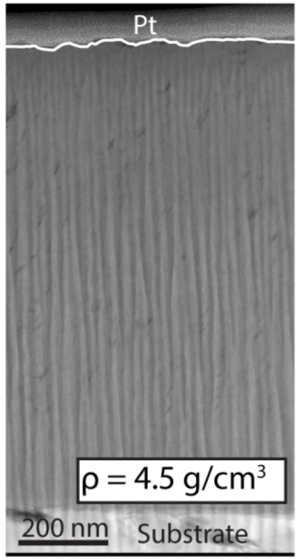
- After 720 min:
  - Surface of low-density coating highly oxidized
  - O penetrates low-density coating
  - High-density coating is unaffected

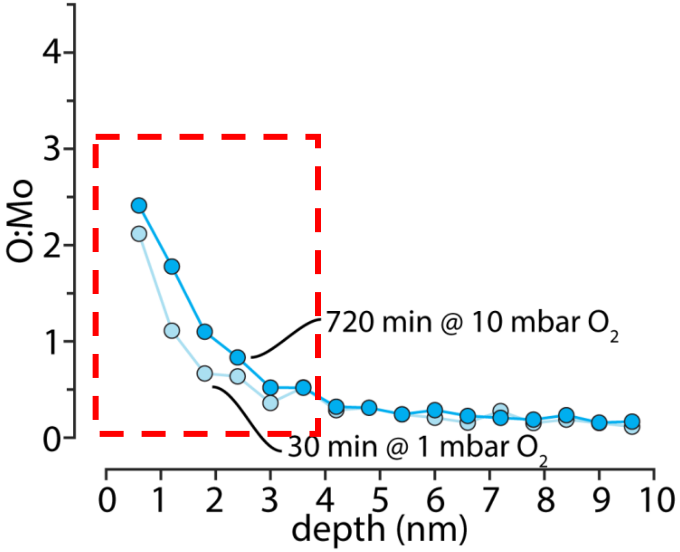
Low-Density Coating





High-Density Coating

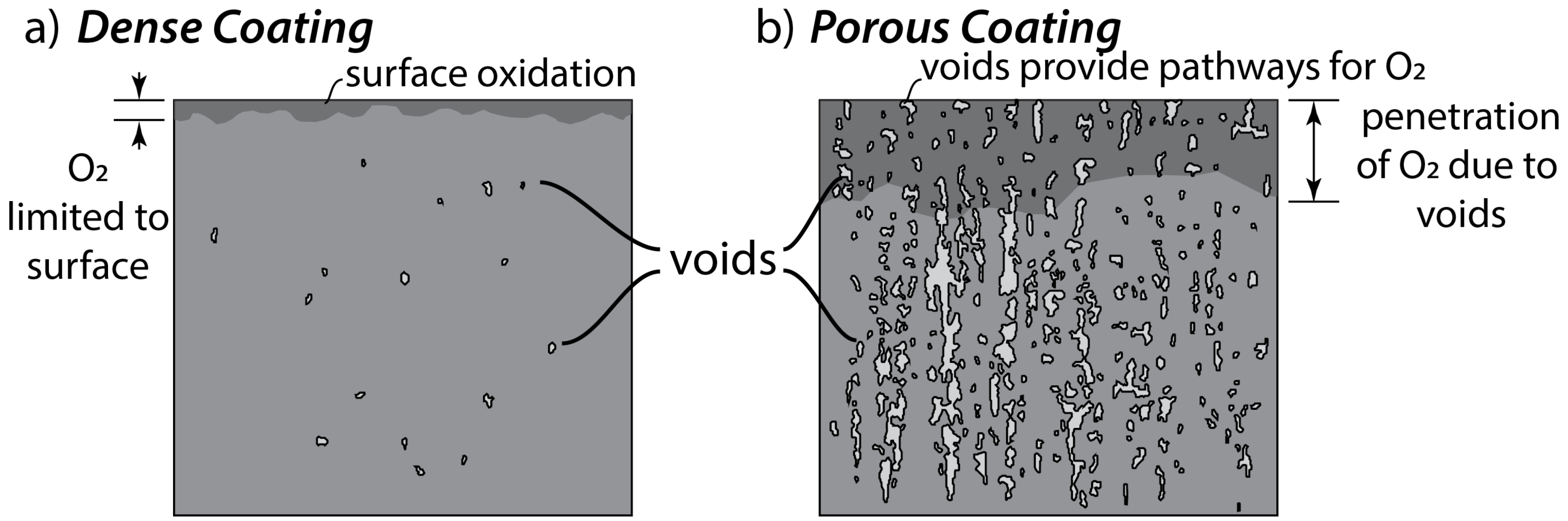




Dense Films Limit Oxidation to the Surface

Takeaways:

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



Summary and Future Work

- The tribological behavior of MoS_2 coatings is dependent on coating morphology
- We defined the ideal tribological properties and characteristics of a quality coating:

Tribological Behavior of a "quality" coating

1. *Low initial COF*
2. *Fast run-in*
3. *Low COF in humid environments*
4. *Wear resistant in different environments*
5. *Limits tribological changes from oxidation*

Characteristics of a "quality" coating

1. *Basally-oriented (surface parallel lamella)*
2. *Nanocrystalline*
3. *Highly-dense (no voids or porosity)*
4. *Low in contaminants (oxygen, hydrocarbons)*

- Deposition of "quality" MoS_2 coatings is difficult due to batch-to-batch variation causing changes in density and orientation
  - This variation is problematic for fundamental studies and hardware
  - Coating performance becomes unpredictable and unreliable

Summary and Future Work

- Started to develop easily measured metrics that could assign quantifiable values to coating batches for quality control
- High-density is an attribute of quality films
  - Increased density results in harder, more wear resistant coatings in inert and humid environments
  - Denser films are more resistant to oxidation from aging, limiting the severity and depth of oxide into the coating.

- Future work - Using metrics for quality, the next step is to develop in house deposition capabilities to develop process-structure relationships

Acknowledgments

Krick Career on Lamellar Lubricity: #2027029

NSF GRFP: #1842163

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