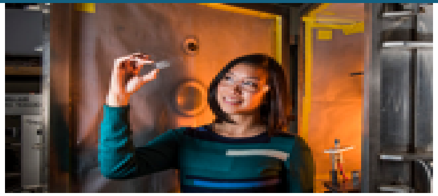
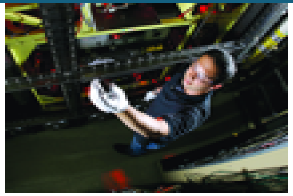
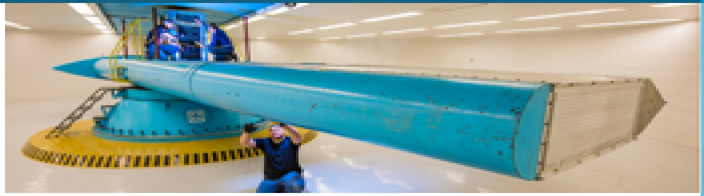
SAND2022-8989C

Aging and Run-In of Shear-Oriented Sputtered MoS_2

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World Tribology Conference
11-15 July 2022, Lyon France

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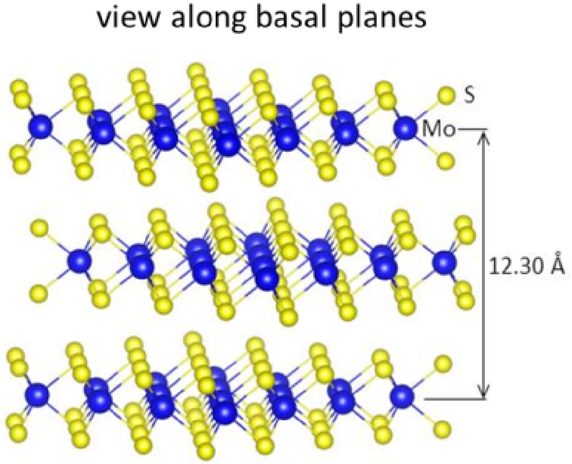
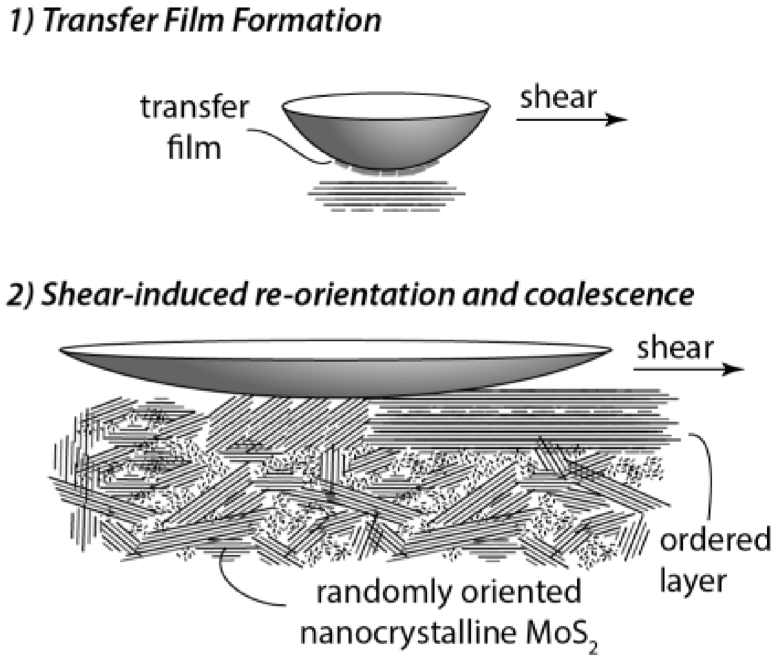
MoS_2 Films for Extreme Environment Lubrication



Run-In Processes:



Aerospace / precision mechanisms share similar concerns

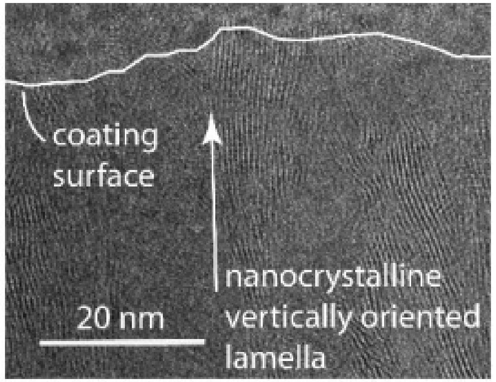
- operate in vacuum (+atomic oxygen in low earth orbit), or
- inert gas near P_{atm}, trace O_2, H_2O, outgassing species
- store months – years before use; generally non-serviceable
- operating temperatures from 50 – 300K, depending on location
- huge investments of time and money

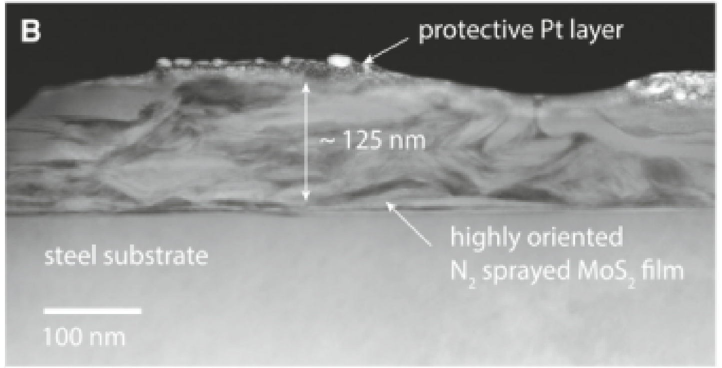
MoS_2 Structure-System Relationships

Material Microstructure

PVD Coating

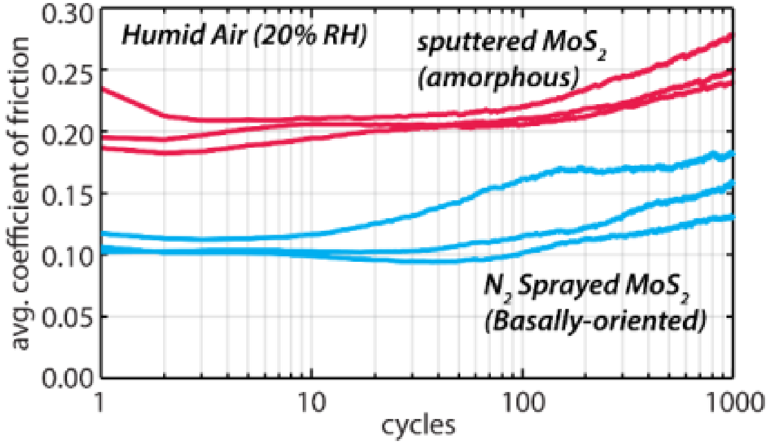
N_2 Spray Coating



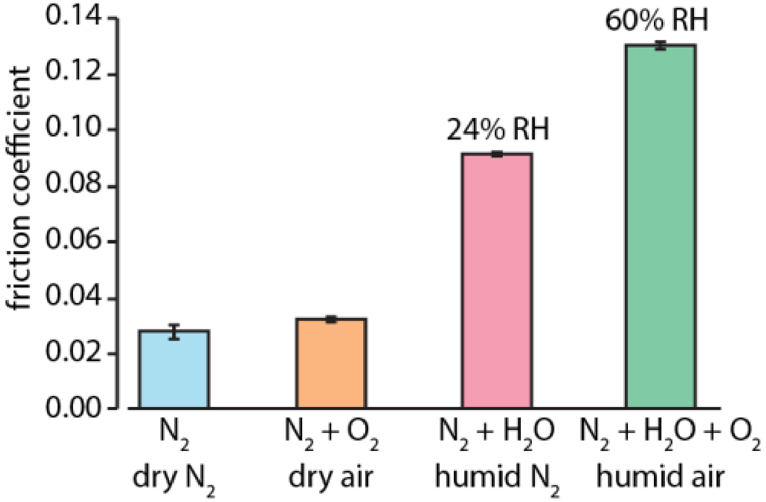


Tribological Properties are System Properties

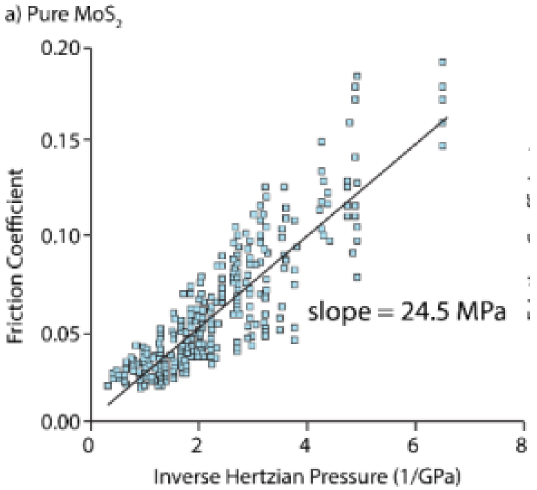
Defines Material Properties (i.e., mechanical, chemical etc.)



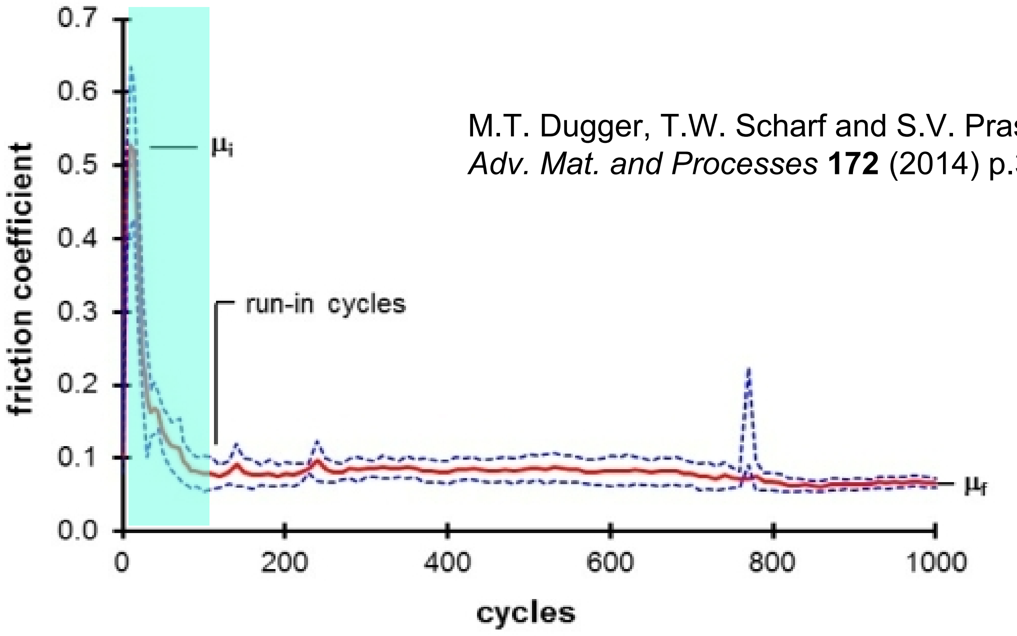
Tribological Properties Depend on: ***Environment***



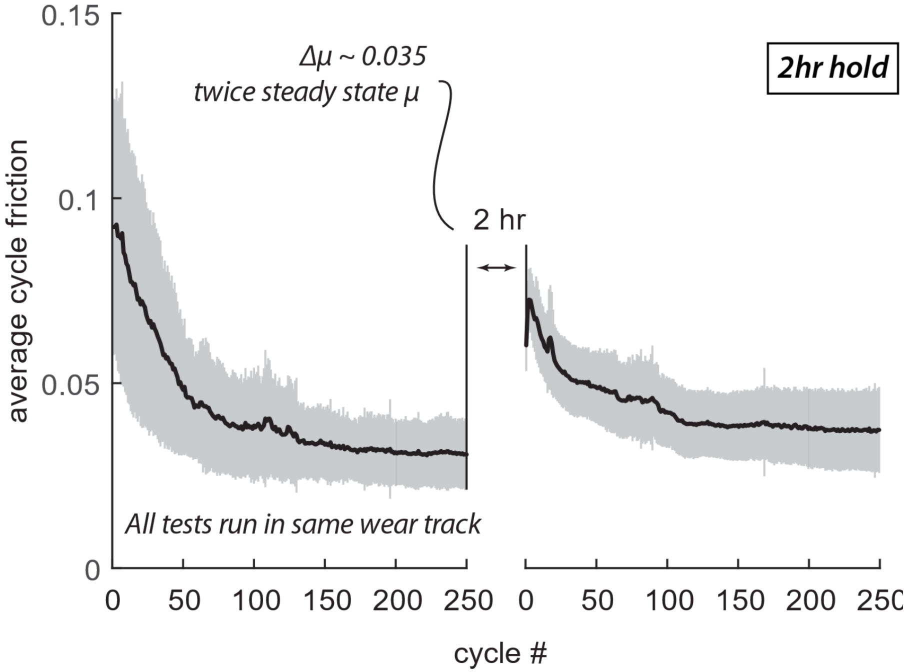
Tribological Properties Depend on: ***Contact Conditions***



Long- and Short-Term Aging Effects



M.T. Dugger, T.W. Scharf and S.V. Prasad, *Adv. Mat. and Processes* **172** (2014) p.35-38



- ❑ Surface oxidation can dramatically increase the initial friction coefficient
- ❑ in this example, atomic oxygen reacted with top 100 nm of film

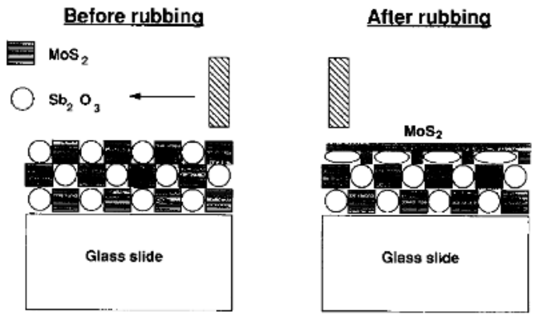
- ❑ Significant friction increase after dormant period
- ❑ Increase in initial friction increases with dwell-time
- ❑ Dwell-time effects are also observed in vacuum
  - monolayer adsorption and/or diffusion from bulk of film

Mitigation of Dynamic Friction Effects

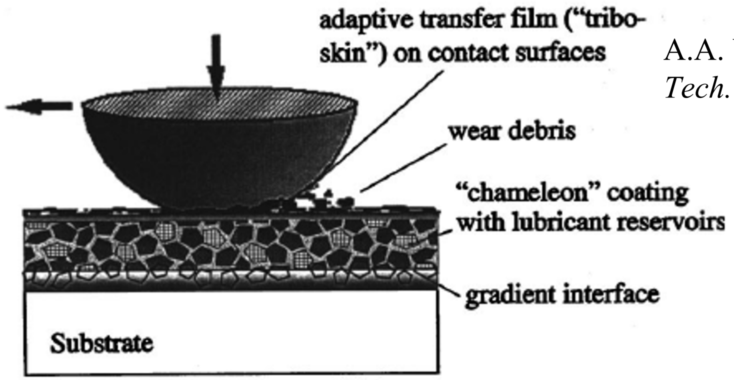
Strategies
- dopants (Ni, Ti, Au, …)
- compositing - multilayers, multiple phases (Sb_2O_3, Ni, AuPd, …)
- ion bombardment during growth

Proposed Mechanisms
- densification
- increased hardness
- preferential orientation
- sacrificial oxidation of dopants
- passivation of MoS_2 edge sites
- crack arresting

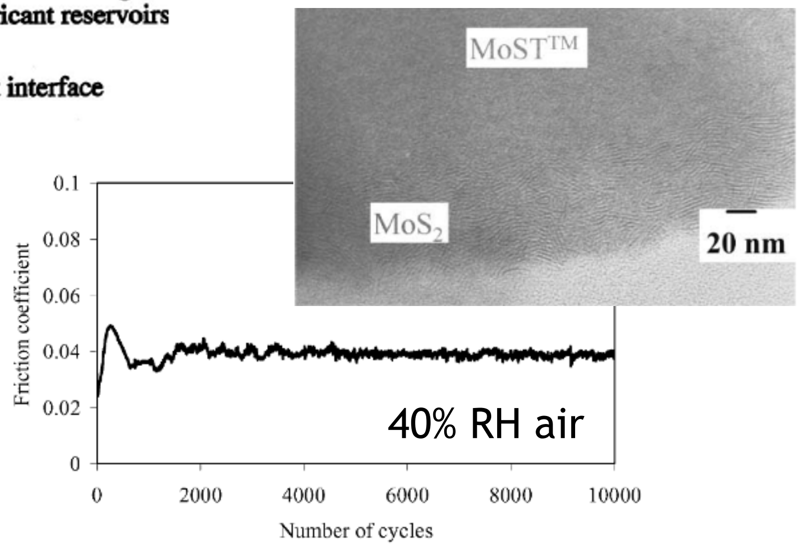
How do dopants/composite phases influence aging?



J.S. Zabinski et al, *Wear* **165** (1993) p. 103

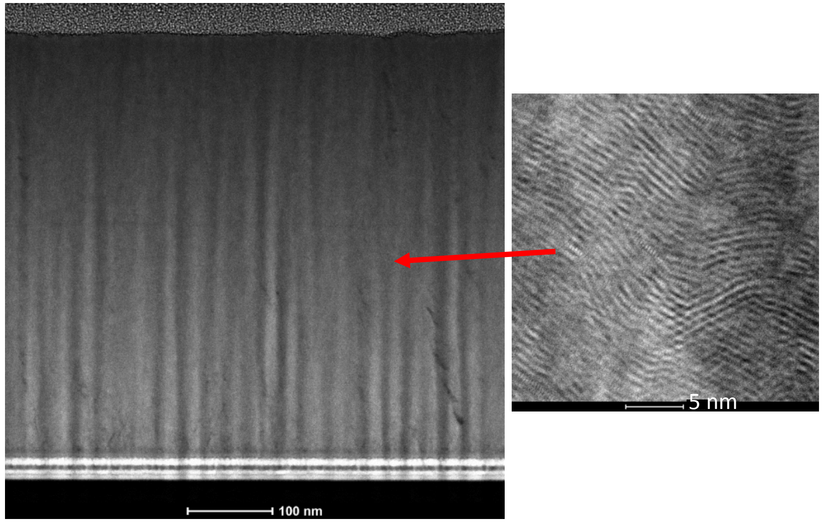


A.A. Voevodin et al, *J. Vac. Sci. Tech.* **A20** (2002) p. 1434

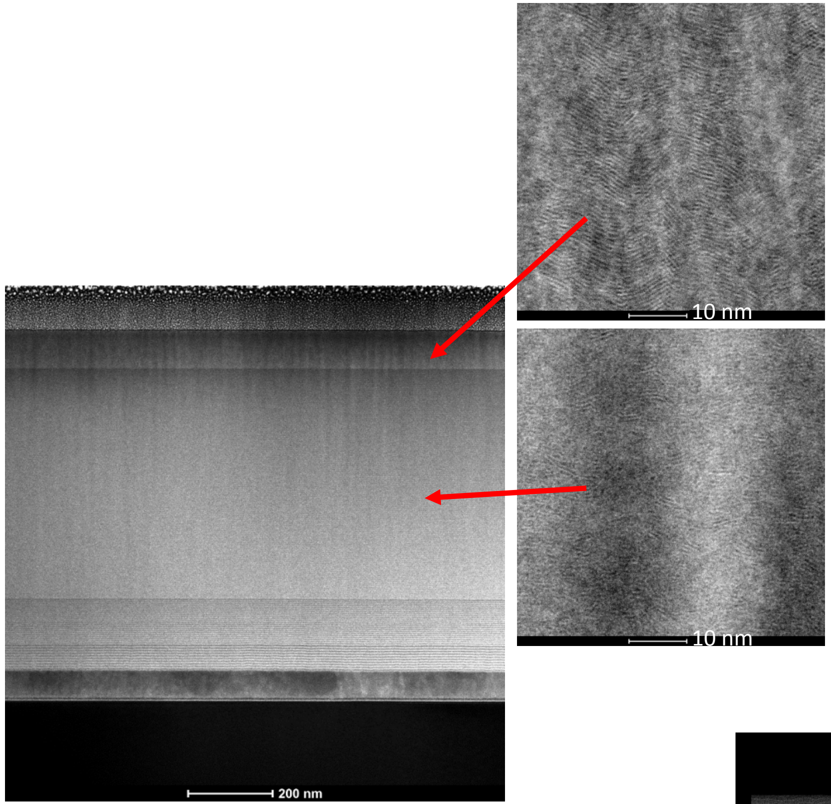


D.G. Teer, *Wear* **251** (2001) p. 1068

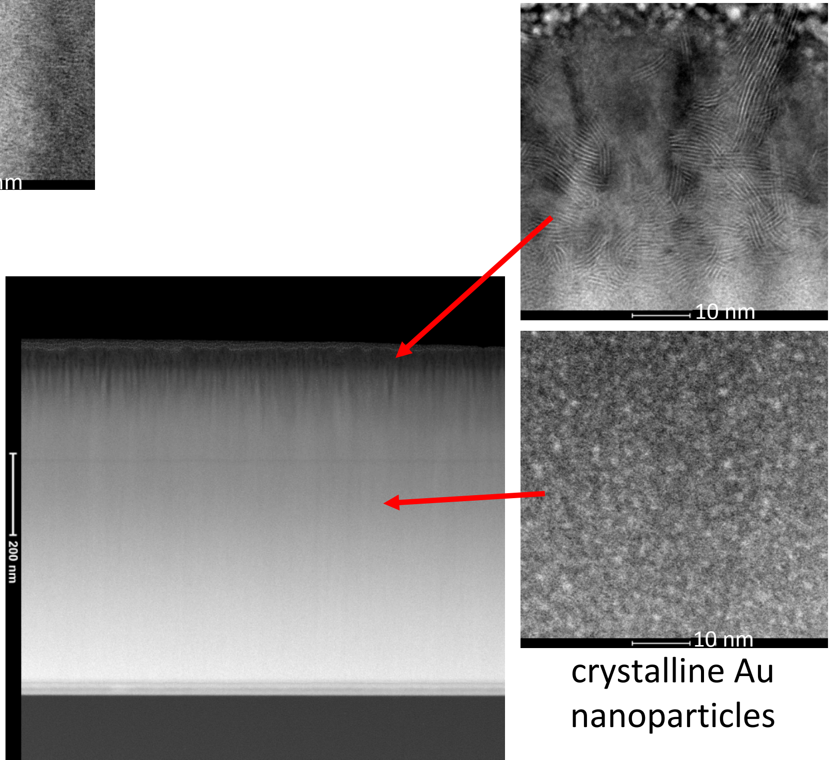
Sputtered MoS_2 Films Investigated



MoS_2 (NC=nanocrystalline)



MoS_2 + Ti (short range order)
MoS_2 + Ti + NC cap

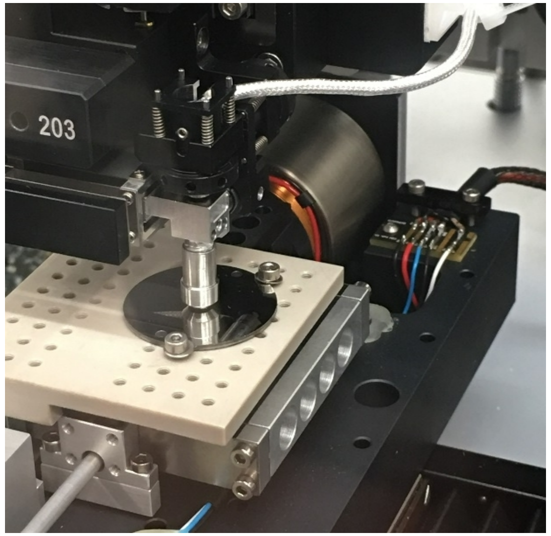


crystalline Au nanoparticles

$MoS_2 + Sb_2O_3$ + Au (amorphous)
$MoS_2 + Sb_2O_3$ + Au + NC cap

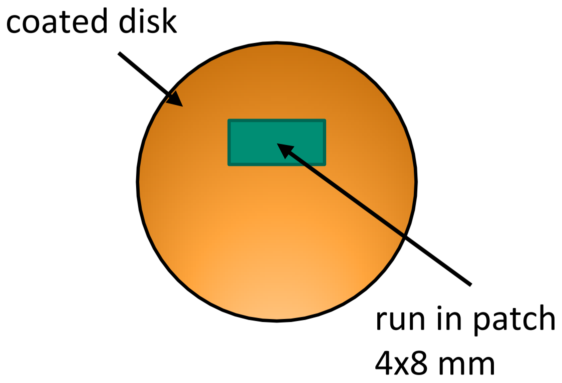
Experimental Setup

Friction Test



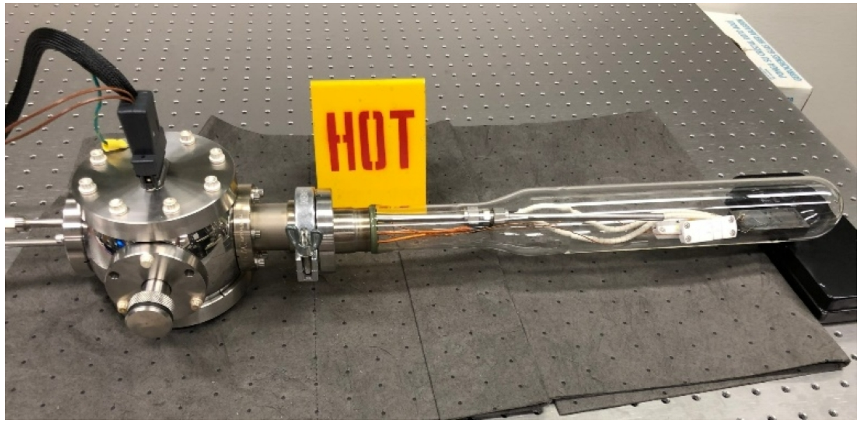
- 440C ball, 3.2 mm dia.
- 1 mm/s sliding speed
- 530 MPa

Run In



- 13-8PH or 440C stainless steel disks
- 50 passes, overlapping areas

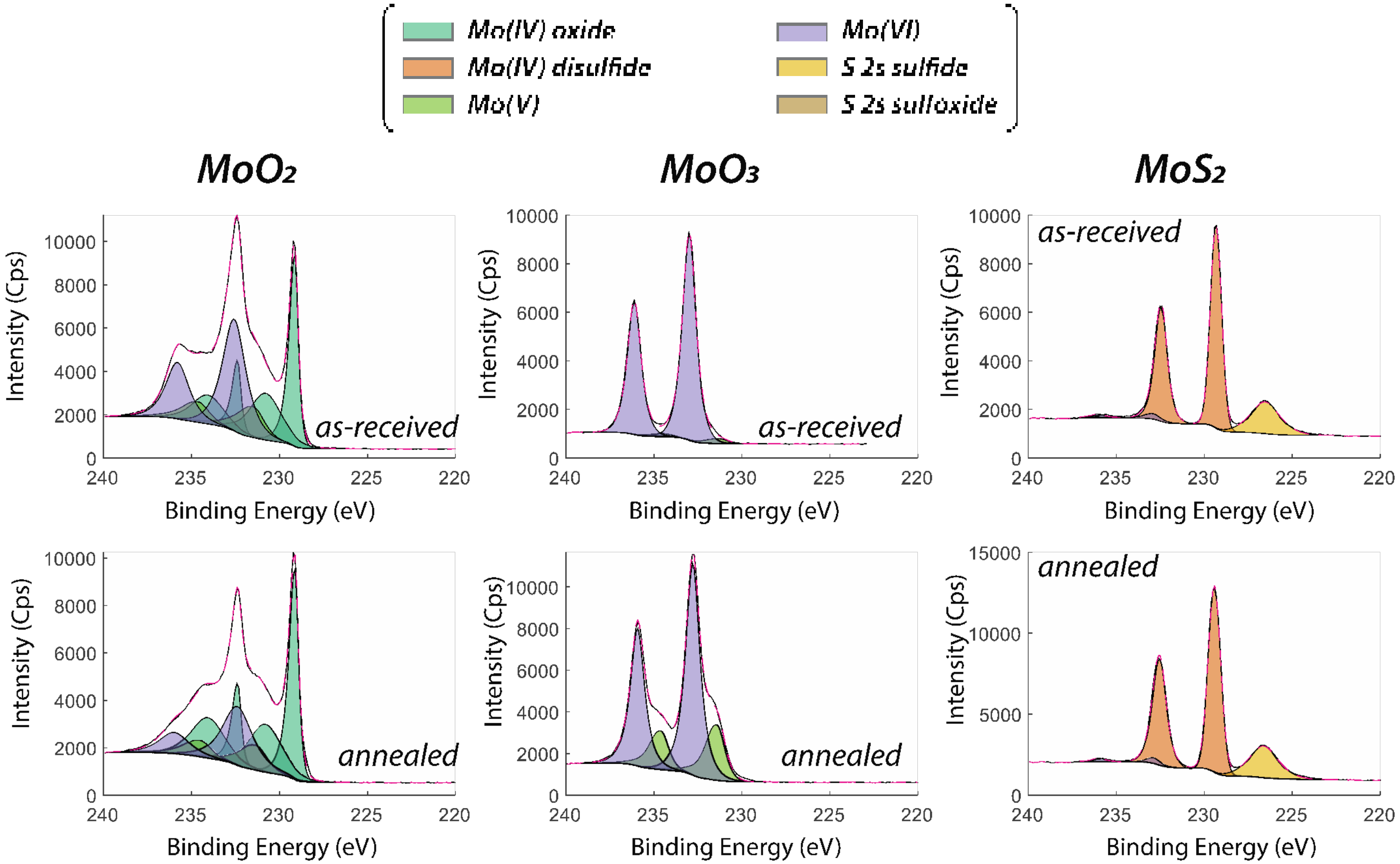
Accelerated Age



- 200°C, 12 hours, P_{atm} at 2.4 L/min.
  - Dry Air (DP < -60°C)
  - Humid Air (50%RH at 20 °C)

- The run-in area provides a region large enough for XPS of surfaces restructured by contact

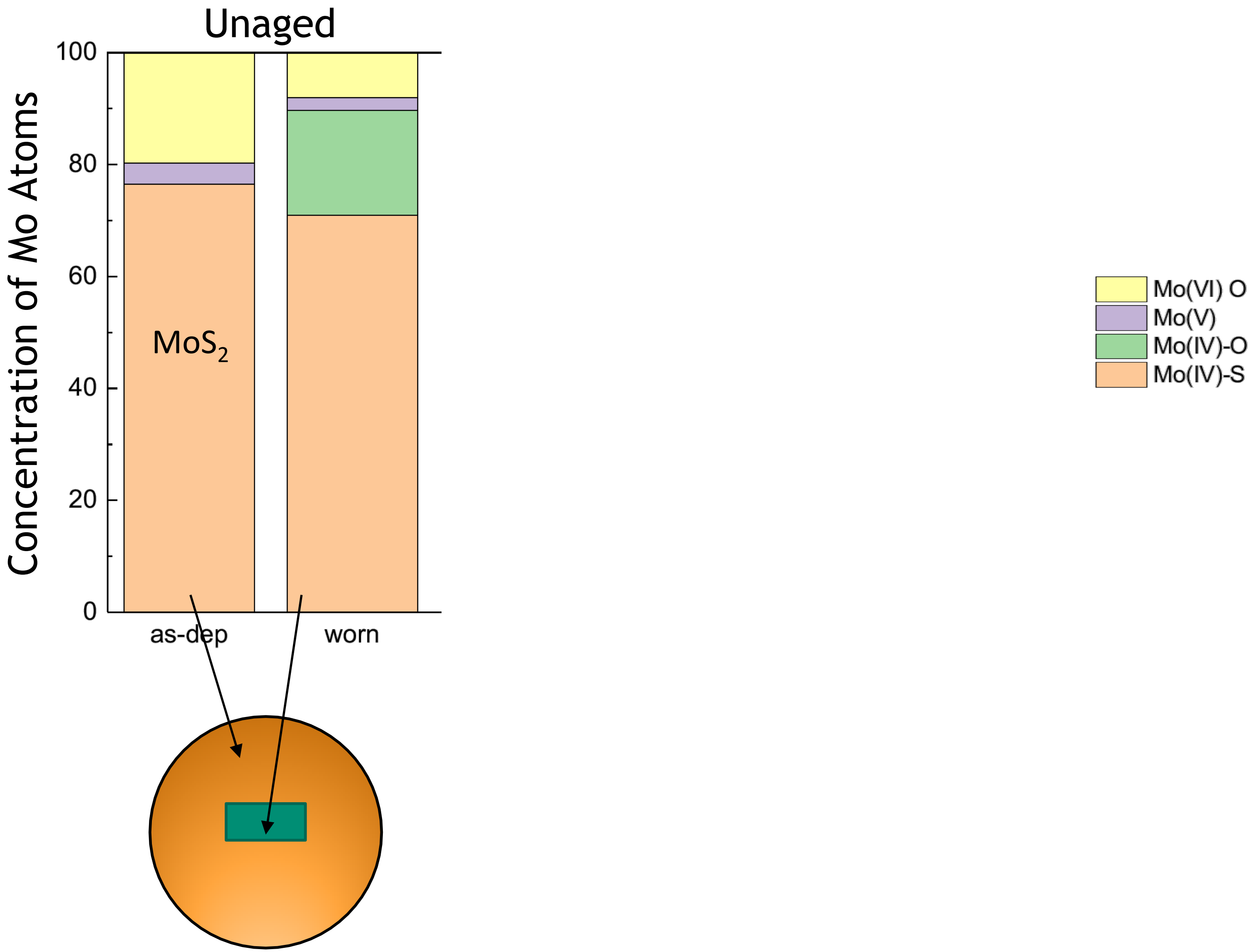
Determining Mo oxidation state using XPS



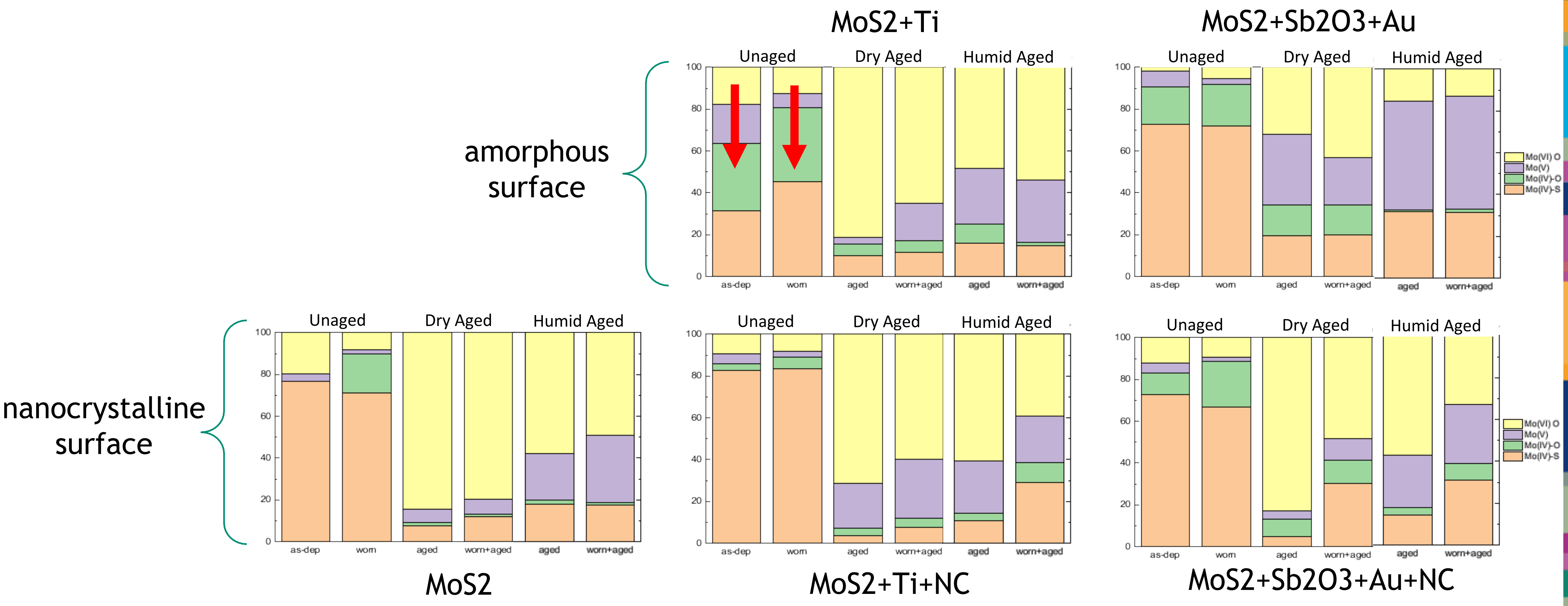
Anneal Reference Compounds at 160°C for 20 hours:

- Thermal reduction of Mo(VI) to Mo(V)
- Final state influences the line shape of Mo(IV) (well-screened and unscreened final states)
- Linear combination of Mo 3d spectra of MoO_2, MoO_3 (unannealed), and MoO_3 (annealed) allowed for the determination of line shape models for each Mo oxidation state
- Results of the fitting procedure were corroborated by quantitative XPS analyses

Tracking oxidation of MoS_2 in aged films

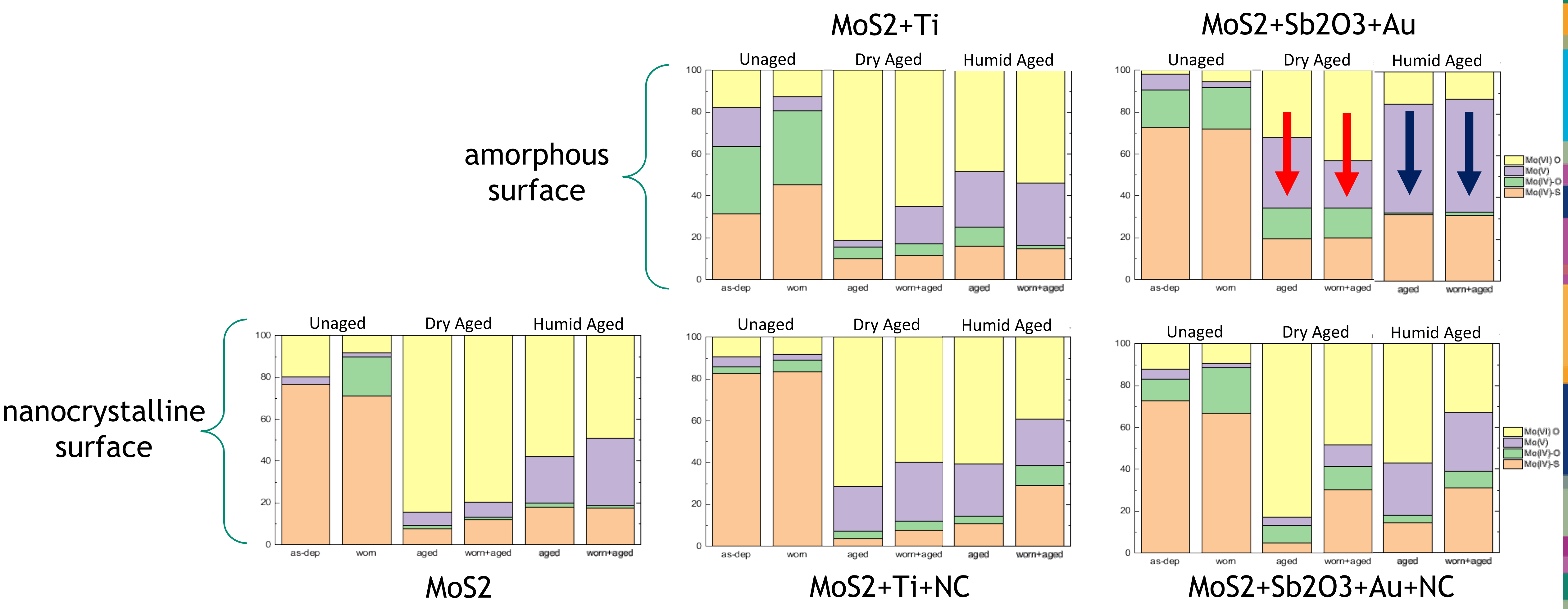


Tracking oxidation of MoS_2 in aged films



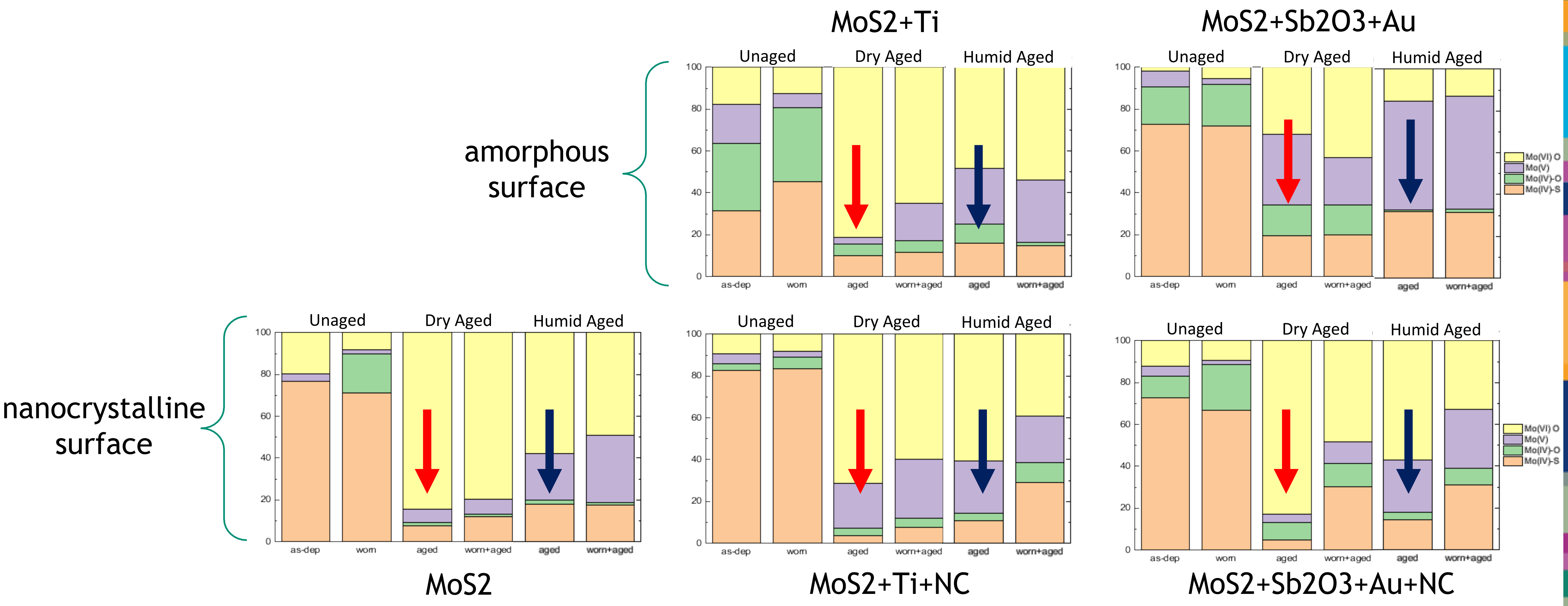
- The Ti-doped film has the lowest MoS2 fraction compared to the other film types

Tracking oxidation of MoS_2 in aged films



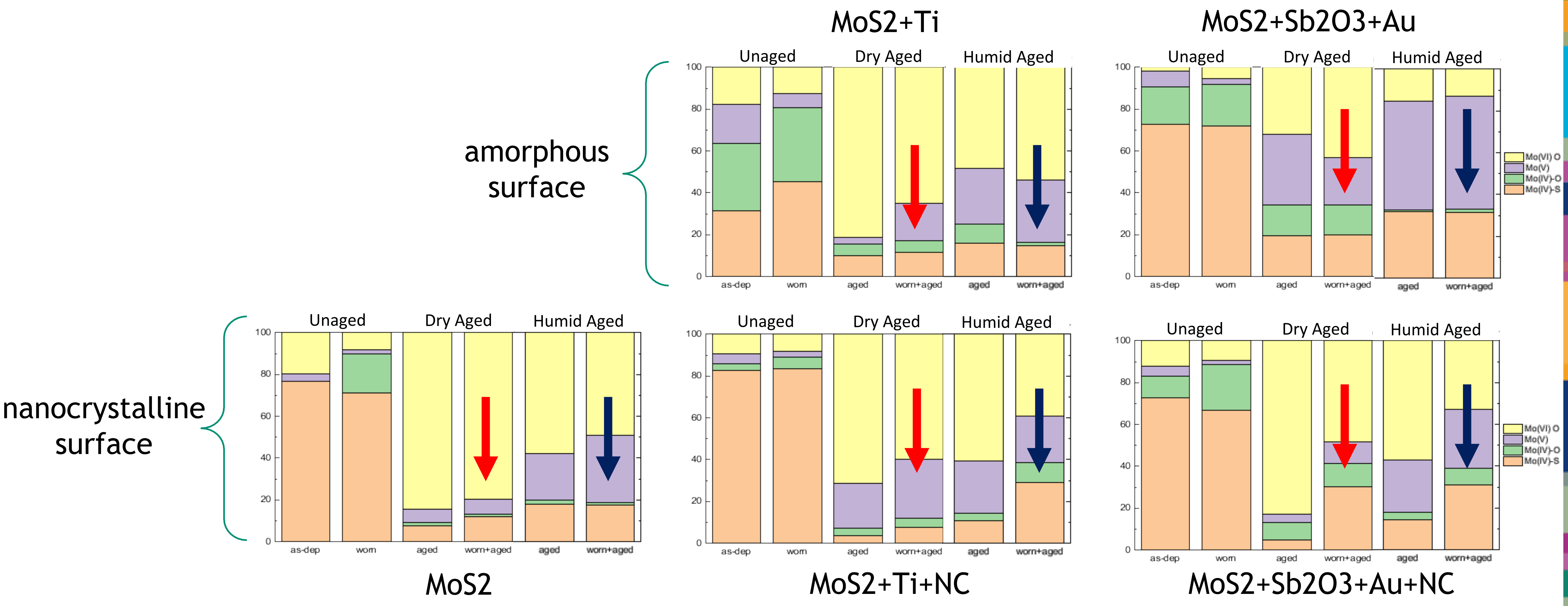
- The Sb2O3+Au doped film has improved oxidation resistance compared to the other film types, in both dry and humid atmospheres

Tracking oxidation of MoS_2 in aged films



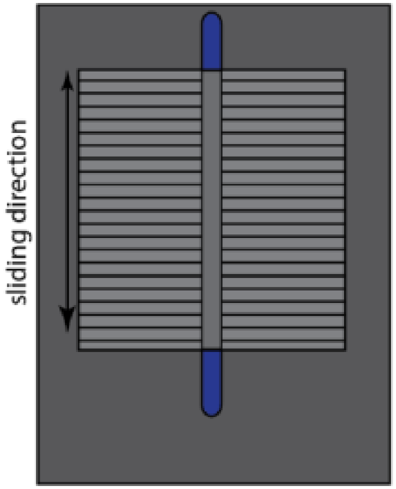
- Aging in dry air generally produced more oxidation than aging in humid air

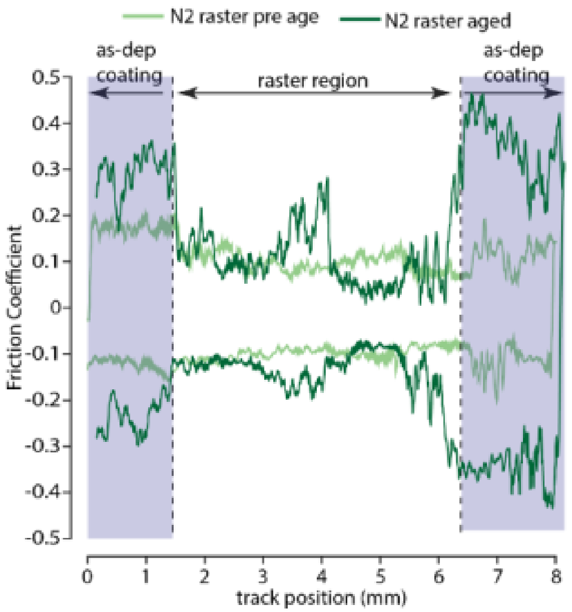
Tracking oxidation of MoS_2 in aged films

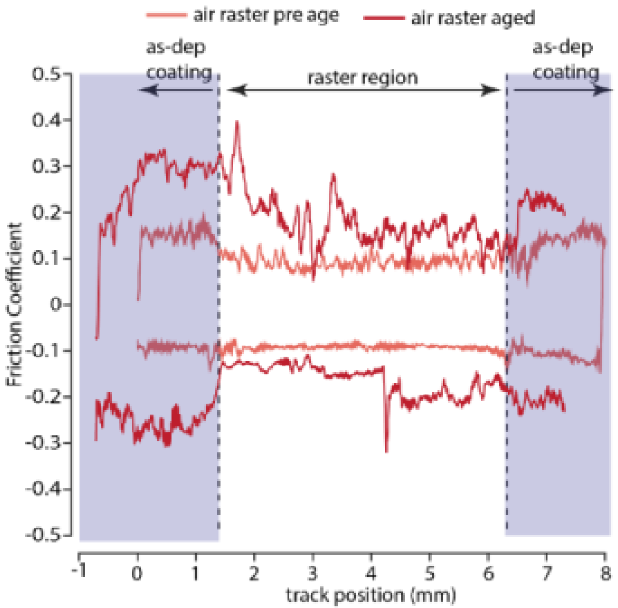


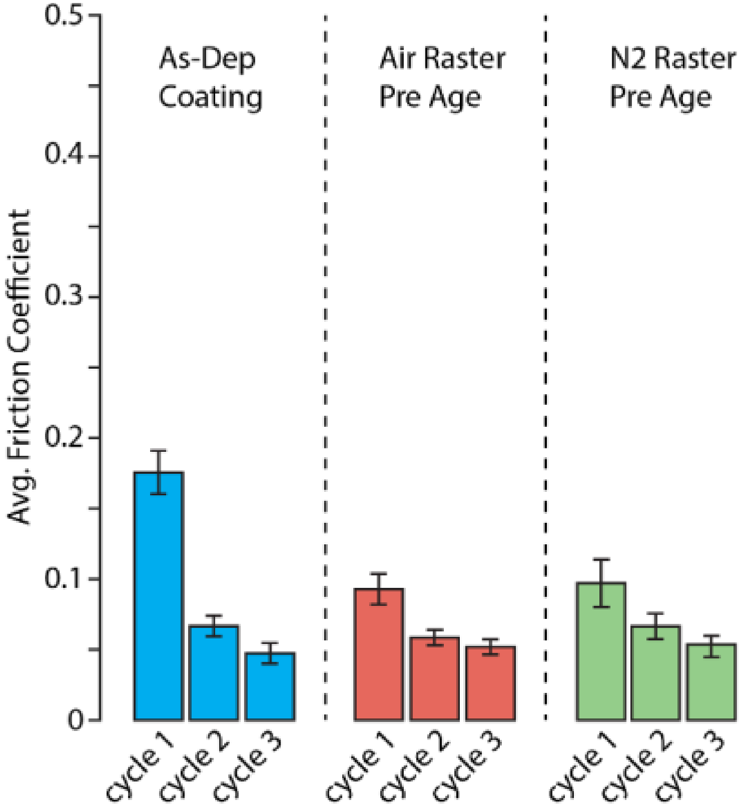
- Surfaces worn prior to aging generally exhibited less oxidation than unworn surfaces
  - this effect is modest, and most pronounced in the nanocrystalline surfaces

Aging studies on run-in surfaces - baselines





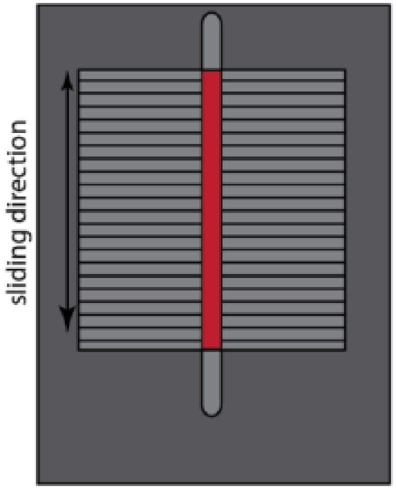




Environment:
Dry n2

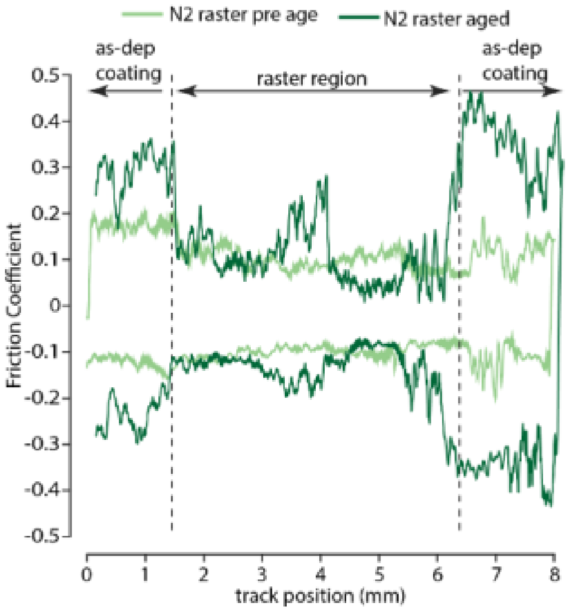
Coating:
Oxidized as-deposited

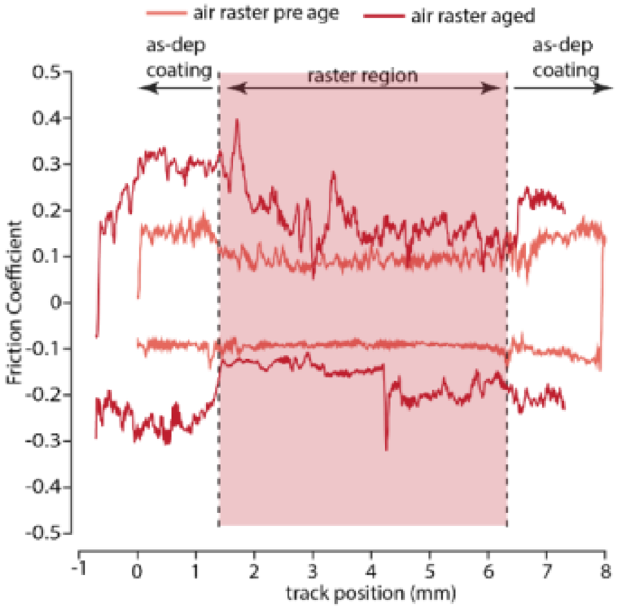
Aging studies on run-in surfaces – aged in air

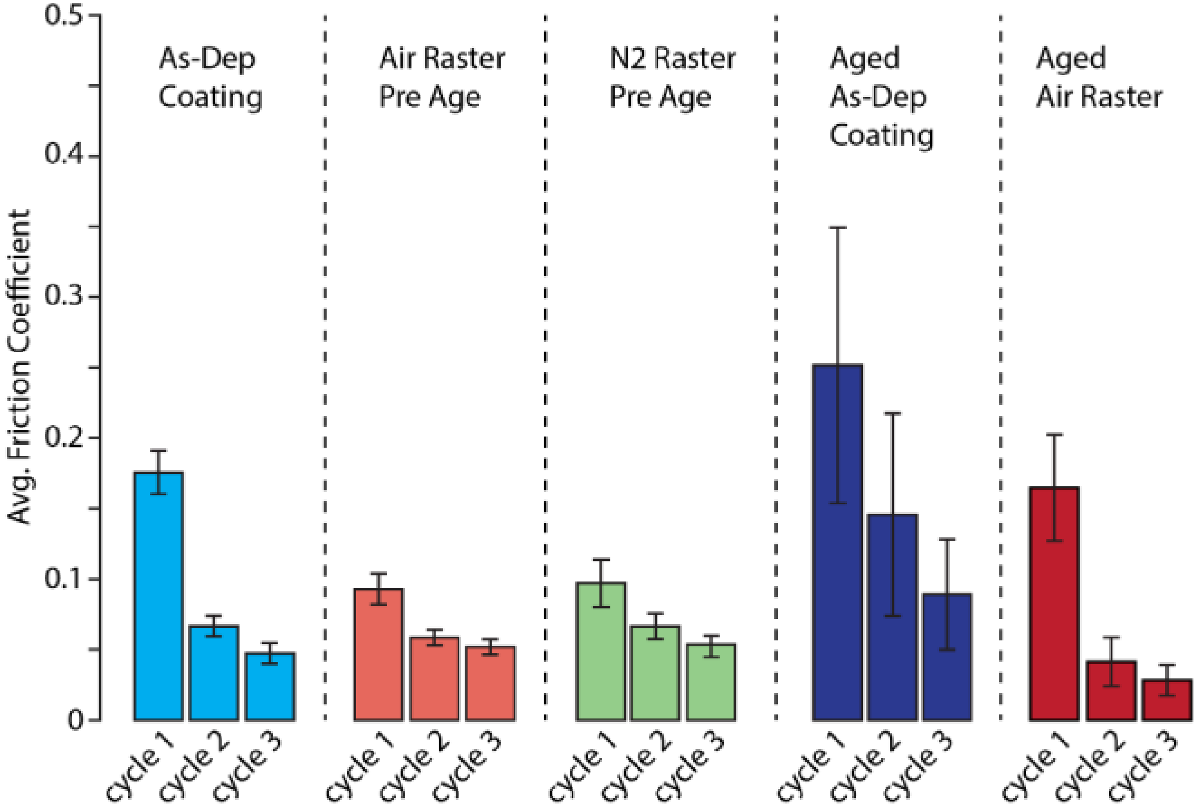


Environment:
Dry n2

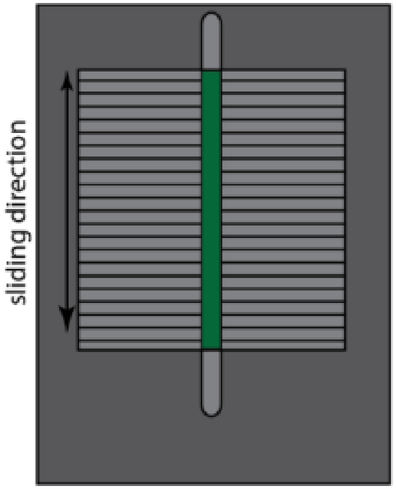
Coating:
Oxidized raster
Slid in air





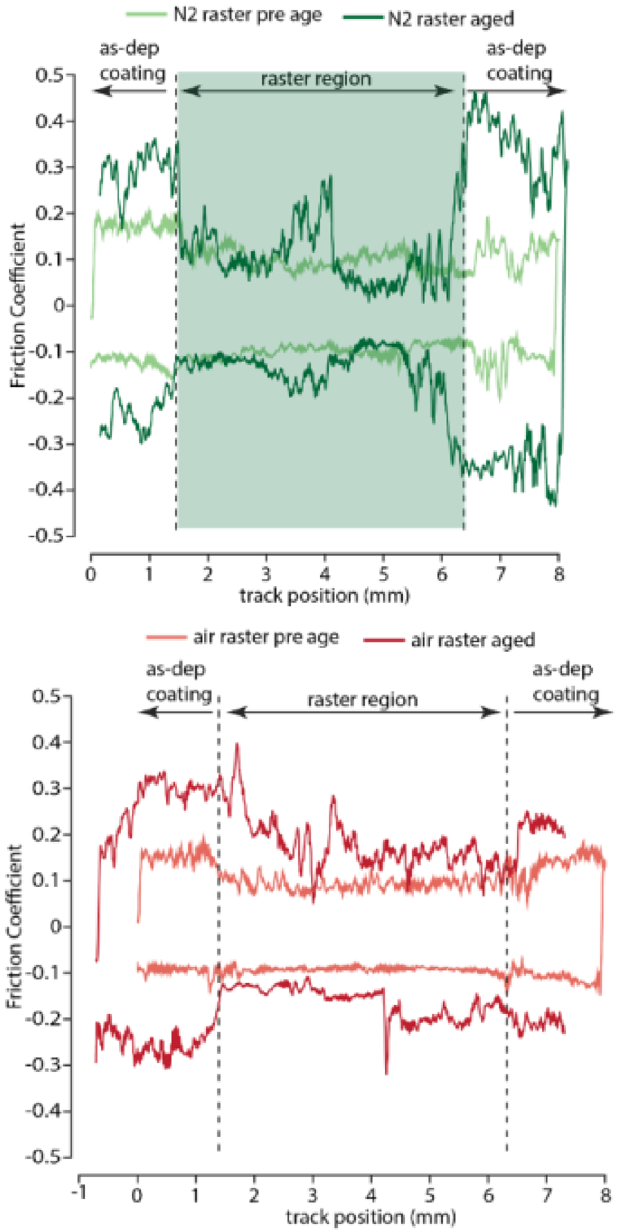


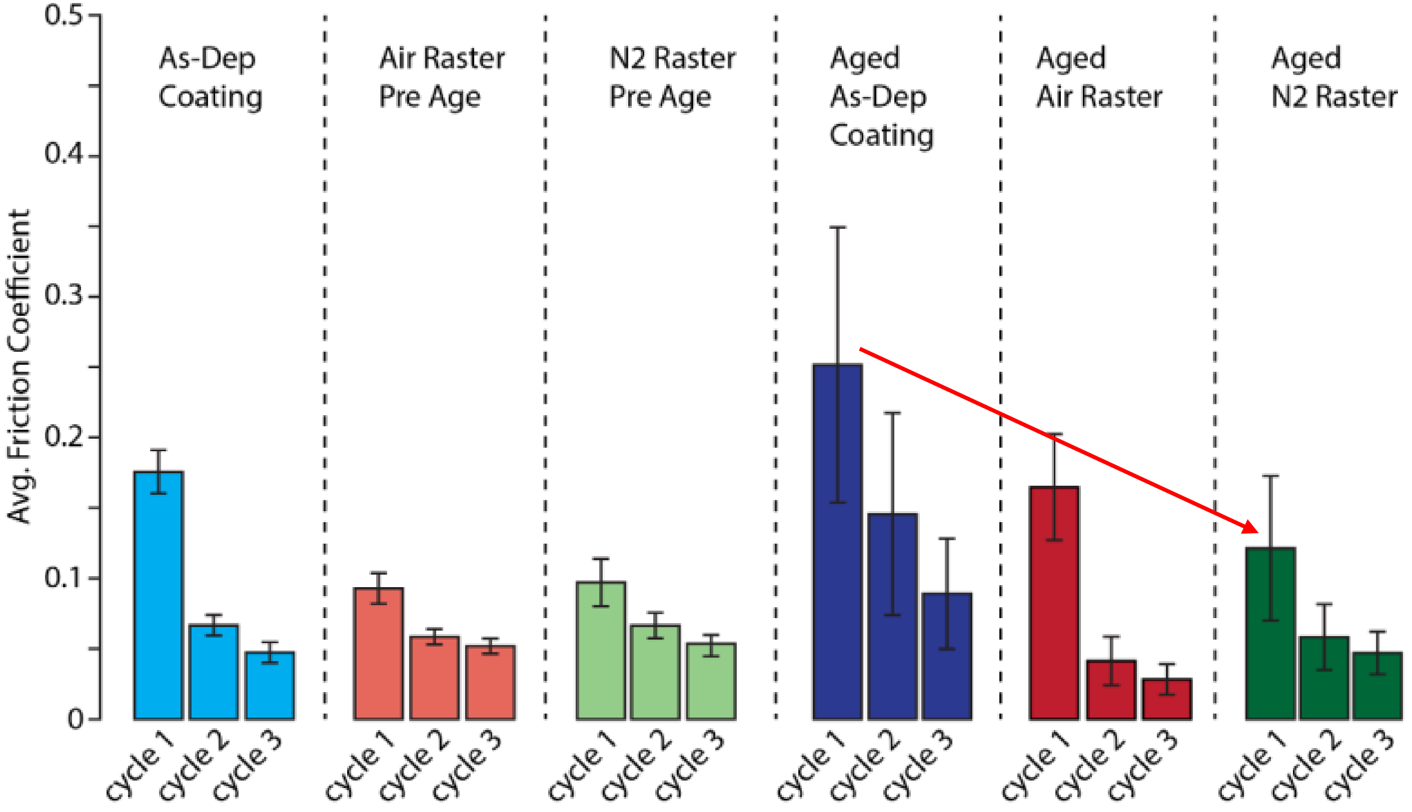
Aging studies on run-in surfaces – aged in N_2



Environment:
Dry n2

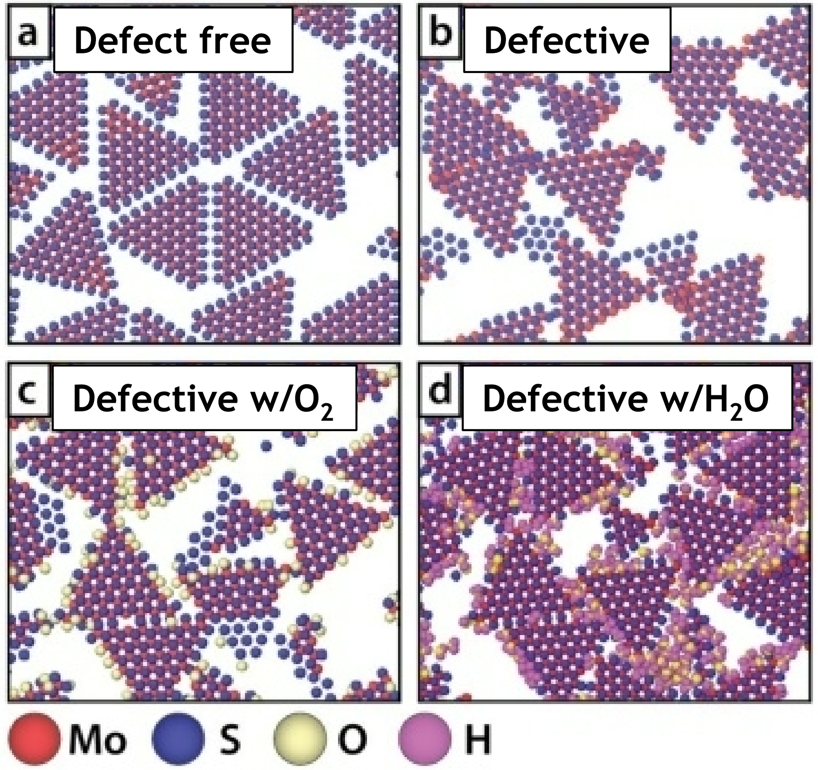
Coating:
Oxidized raster
Slid in dry n2

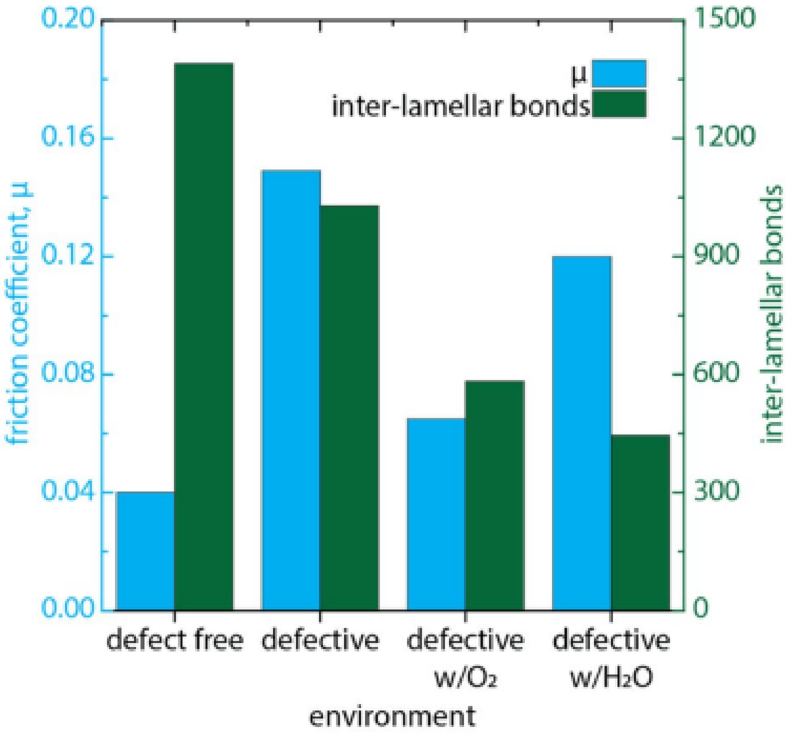




Basally-oriented MoS_2 surfaces provide reduced run-in after aging

MD Suggests Structural Mechanism at Play





MD Conclusions

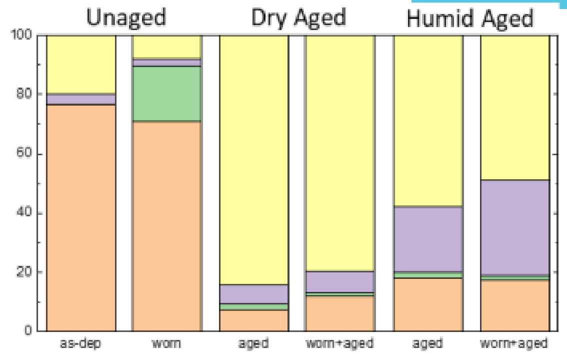
- Sliding on pristine MoS_2 orders lamella and increases lamella size – low friction
- O_2 and H_2O passivate edge sites preventing coalescence of lamella

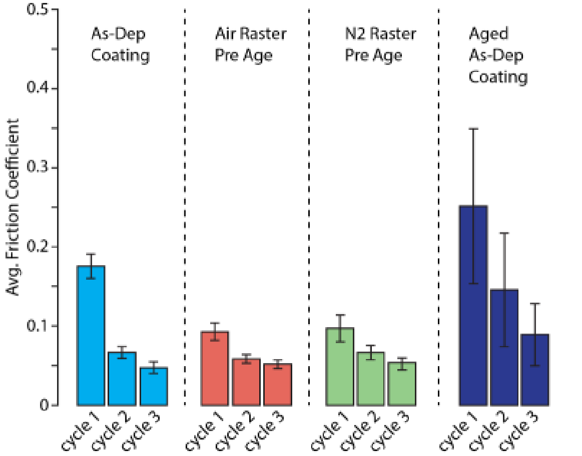
MD suggests environmental species inhibit long range order of lamellae, preventing restructuring to low friction interfaces

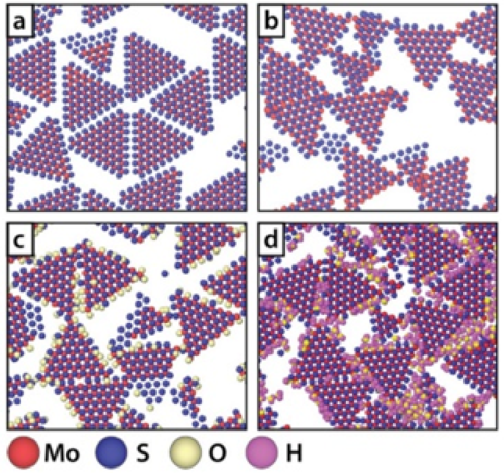
Curry, J. F. *et al. Tribol. Lett.* (2021)

Conclusions

- **Accelerated aging of composite MoS_2 film structures:**
  - Ti-doped film has lower Mo-S than other films, pure or doped
  - Sb2O3+Au-doped film resists oxidation better than Ti-doped film in both dry and humid atmospheres
  - Aging in dry air generally produced more oxidation than aging in humid air
  - Surfaces worn prior to aging generally exhibited less oxidation than as-deposited surfaces
- **Run-in of surfaces restructured by sliding contact:**
  - Higher degrees of crystallinity and lower defect densities at the surface are favorable for reducing friction and oxidation
  - Surface re-ordering can even occur in non-inert environments to afford similar protections
- **The role of water and oxygen on dynamic recrystallization during sliding contact:**
  - MD simulations suggest water interaction/agglomeration with edge sites prevents formation of long range order MoS2
  - Structural degradation (smaller flake size, higher defect density) due to environmental interactions leads to increased friction







Acknowledgments

- Samantha Rosenberg for XPS measurement and deconvolution of MoS_2 oxidation states
- Brendan Nation and John Wellington-Johnson for tribology testing and coating aging

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