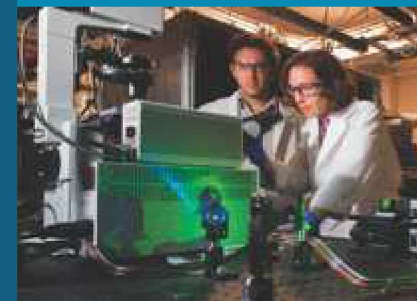
SAND2019-6722C

Run-In Behaviors of Solid Lubricants



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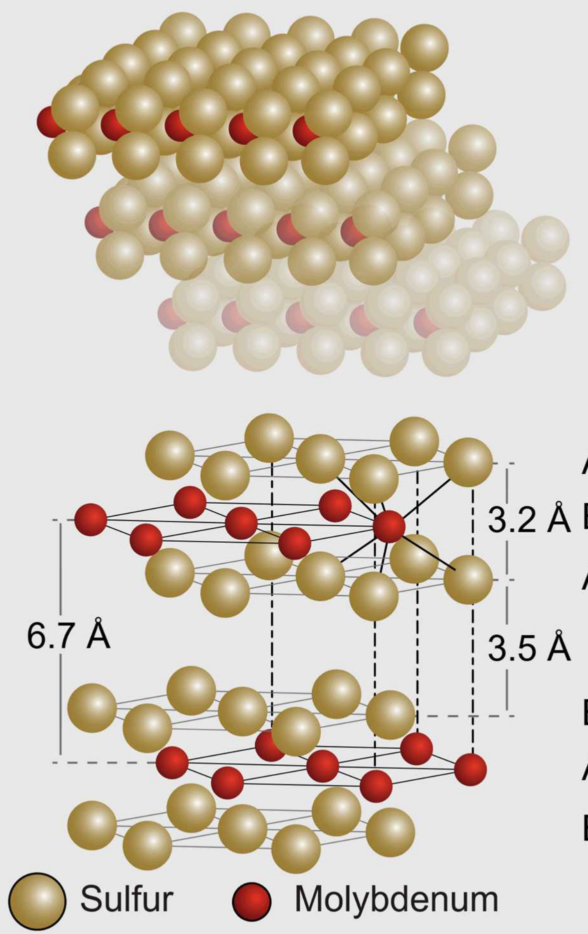
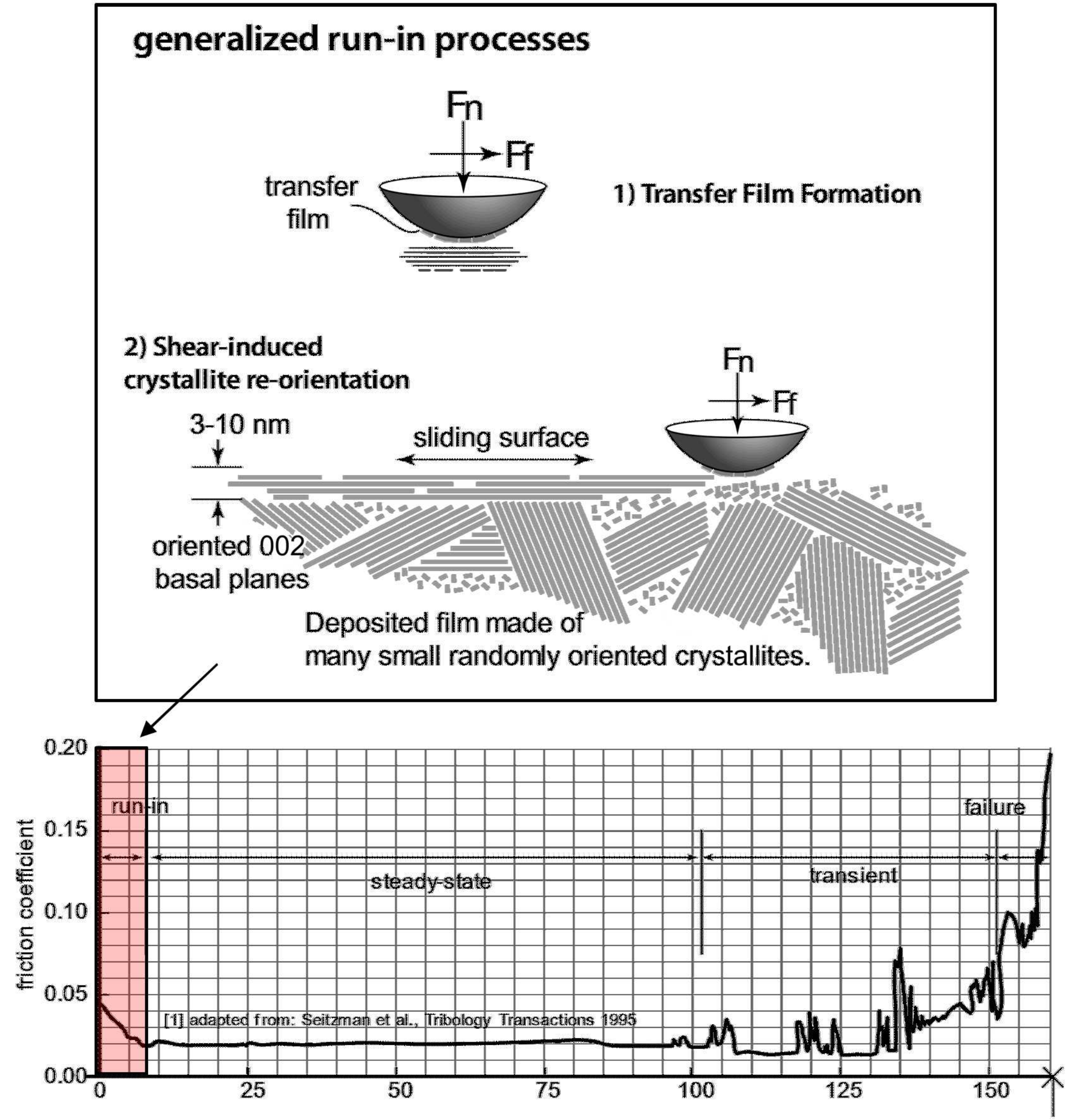
MoS_2 – How it Works

molybdenum disulphide

μ = 0.02 - 0.06 (inert @ 1N)

μ = 0.15 - 0.25 (humid air @ 1N)





Typical friction trace for MoS2 lubricated contact. Initial run-in is followed by steady state low friction which ultimately transitions to high friction before failure [1]

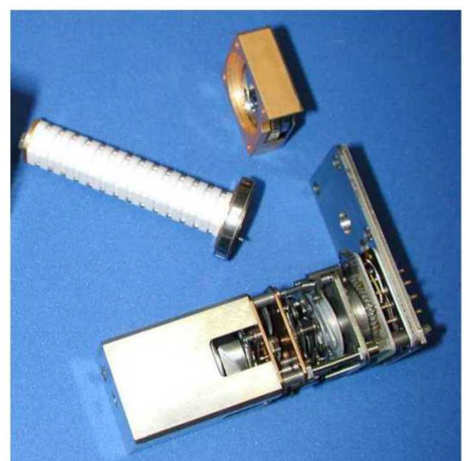
Motivation – Environmental Sensitivity & Aging

Space:

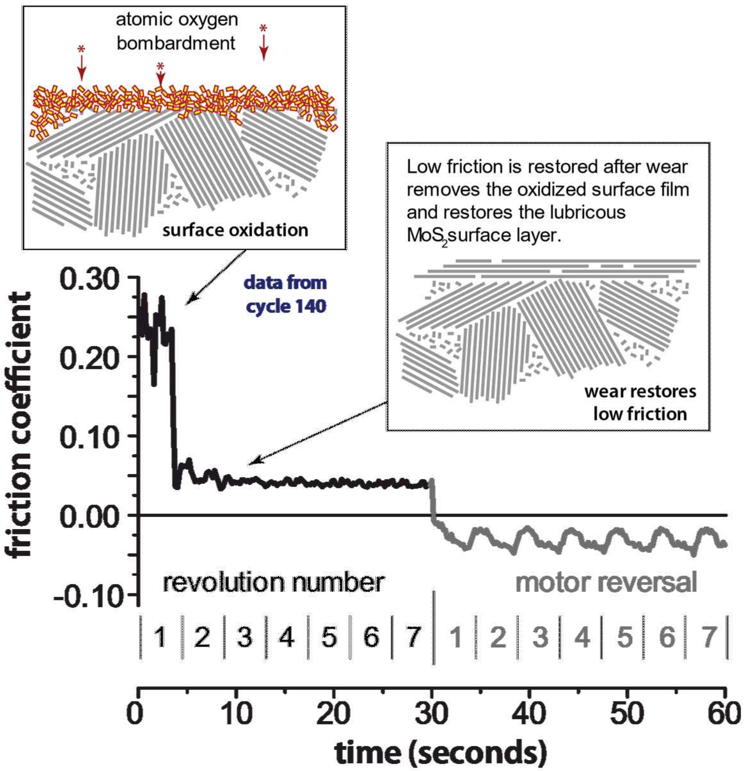
- operate in vacuum (+atomic oxygen in low earth orbit)
- store months – years before use; generally non-serviceable
- operating temperatures from 50 – 300K, depending on location
- large investments of time and money

Precision Mechanisms:

- inert gas near P_{atm}, trace O_2, H_2O, outgassing species
- store for decades; non-serviceable
- operating temperatures 200 – 350K
- large investments of time and money
- consequences (political, societal) of failure are unacceptable



Bad Actors – Environment & Aging



Krick et. al, unpublished

- Oxidation can occur in space (AO - fast), air at high temps (O_2 – fast) and room temp (H_2O – slow)

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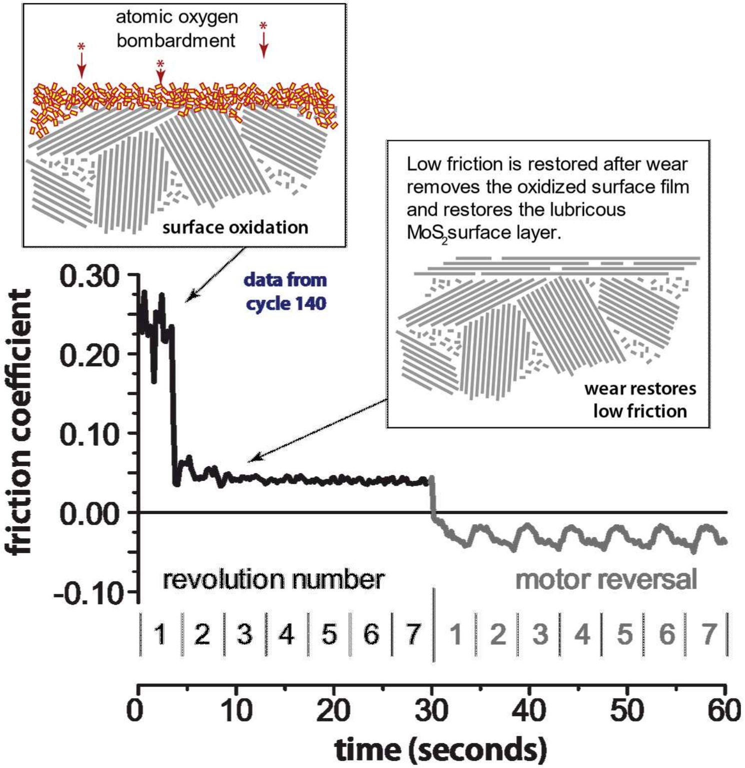

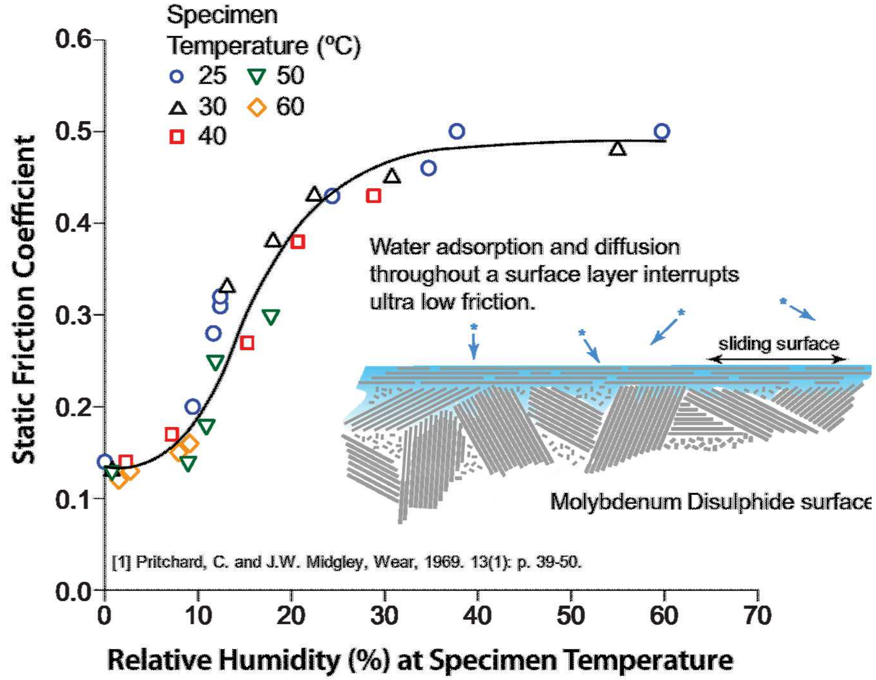

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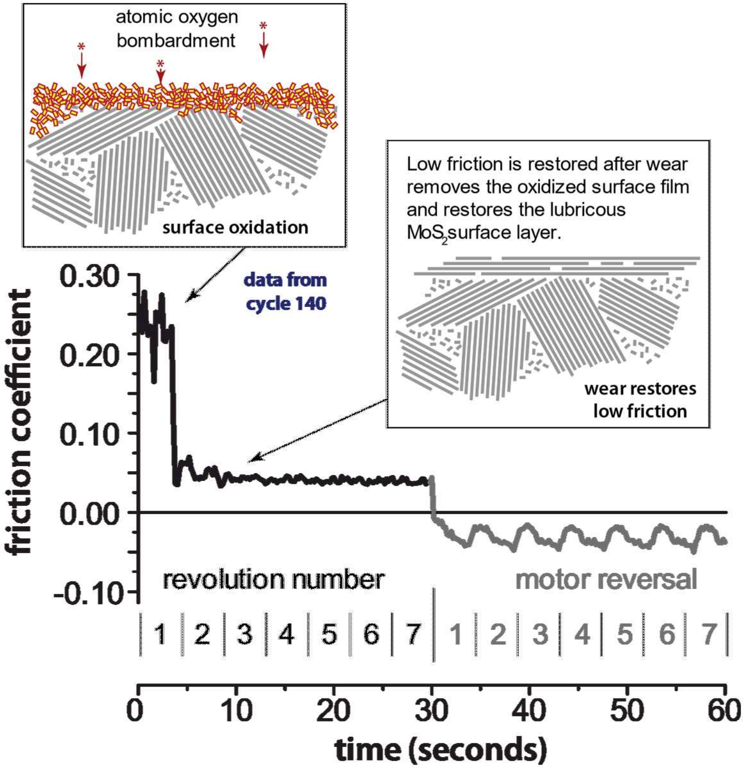

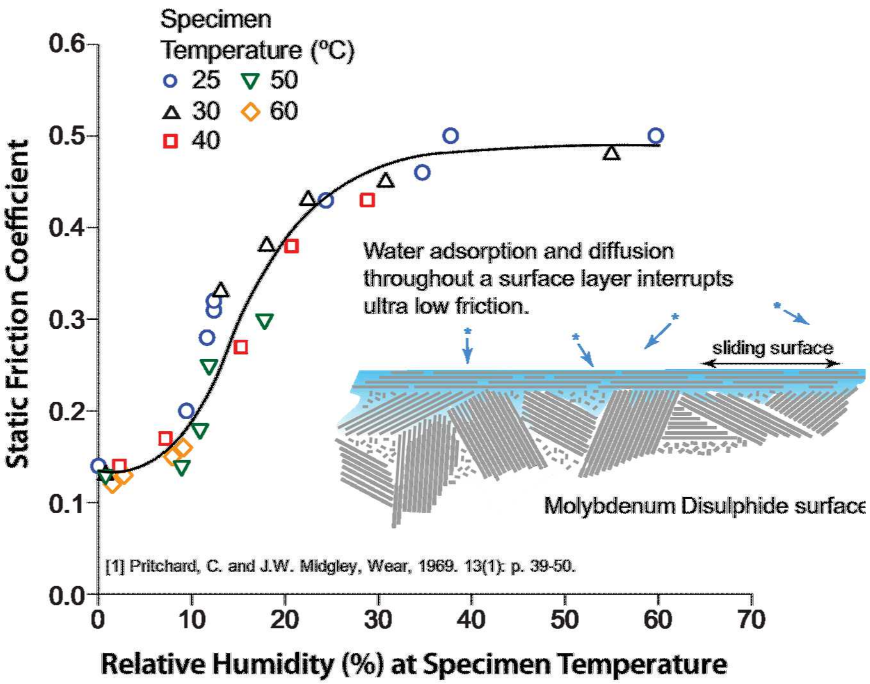

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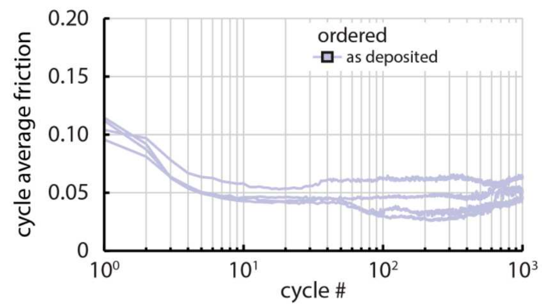
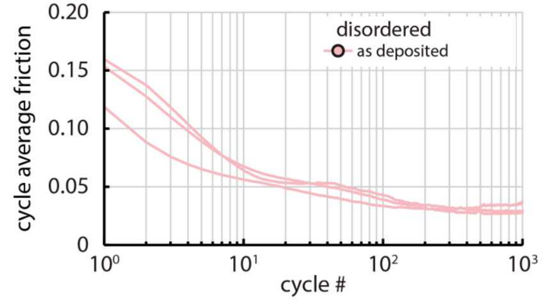
- Water enhances static and kinetic friction behaviors via increased shear between layers

Many components operate infrequently and for very few cycles – ***effectively living in the run-in regime***

Co-Influencing Factors: Microstructure & Oxidation

Ordered films **Disordered films**





- Basally oriented or burnished (ordered) films lessen need of reorientation, reducing friction

Curry et al., ACS AMI 2017

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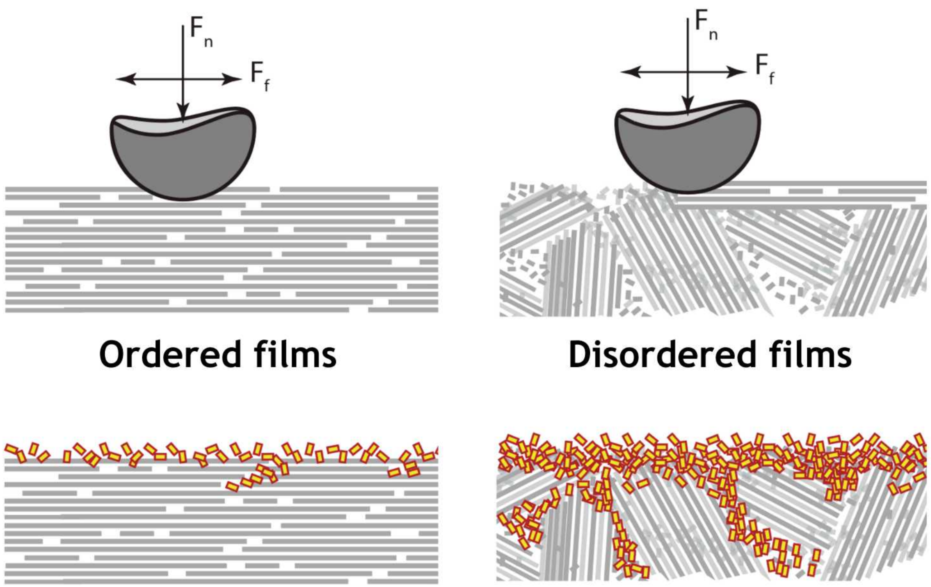
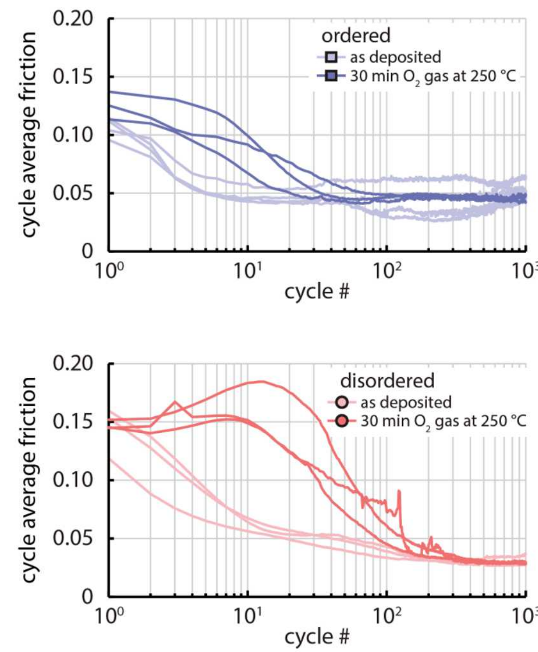
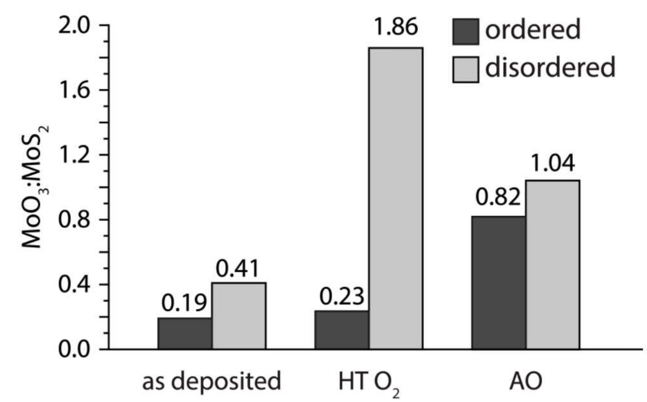




- Basally oriented or burnished (ordered) films lessen need of reorientation, reducing friction
- Also reduces edge:basal surface ratio, reducing run-in effects from oxidation
- Problem solved!

Mo 3p signal - MoO_3:MoS_2 ratio



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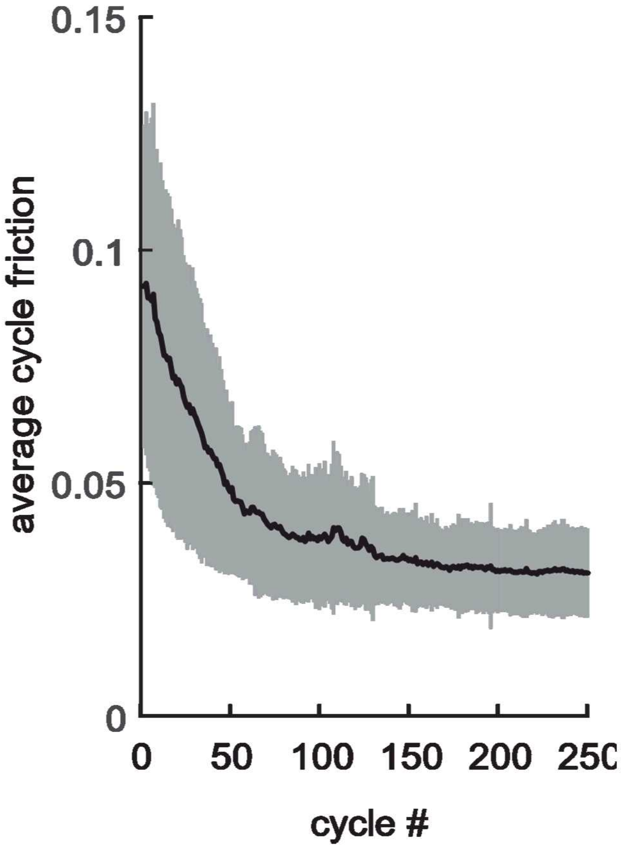
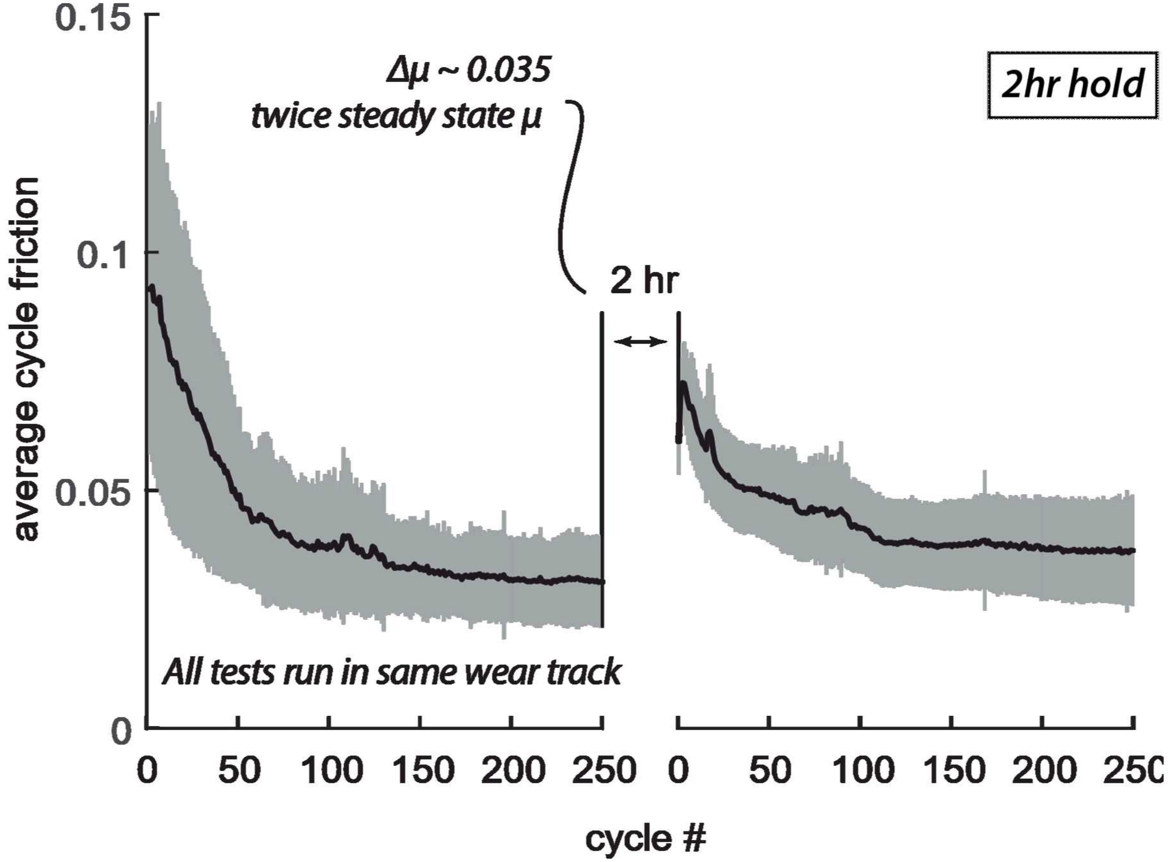
Run-In Solves Everything

- Recipe for success: run film in to steady state



Run-In Solves Everything… Except Time…

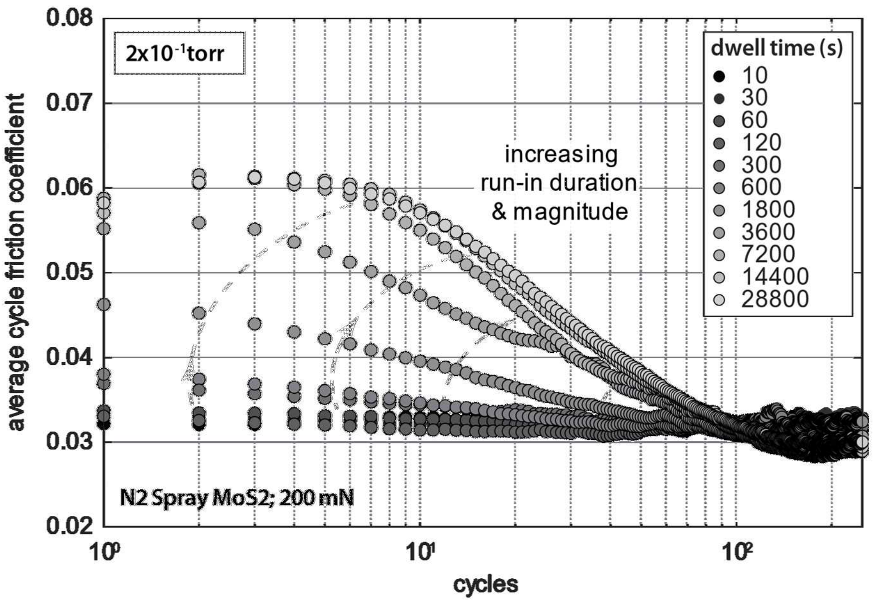
- Recipe for success: run film in to steady state… and watch friction increase upon return



Re-Run-In Time Matters

- Increase in initial friction is monotonic, depending on time in between; run-in duration also affected

- This "stop-time" effect is observable in vacuum



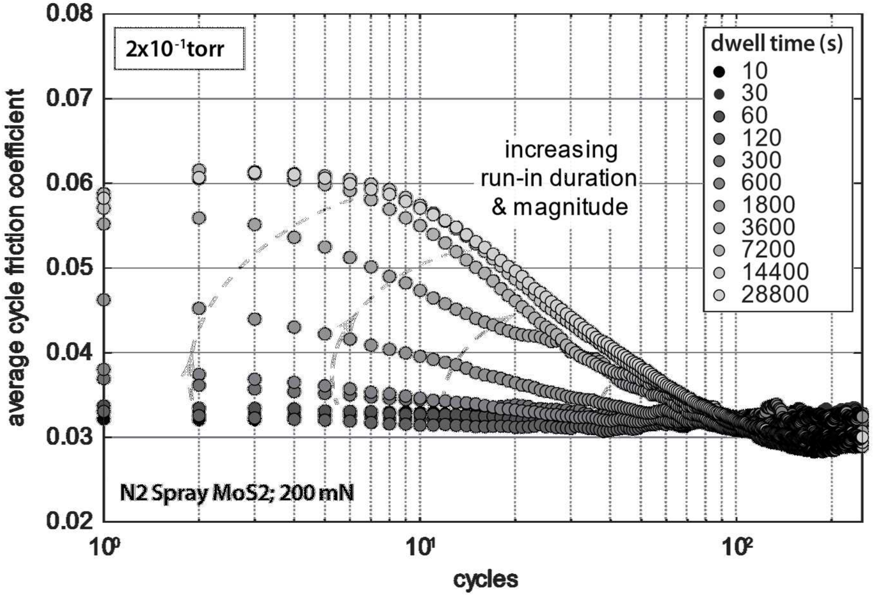
Re-Run-In Time Matters

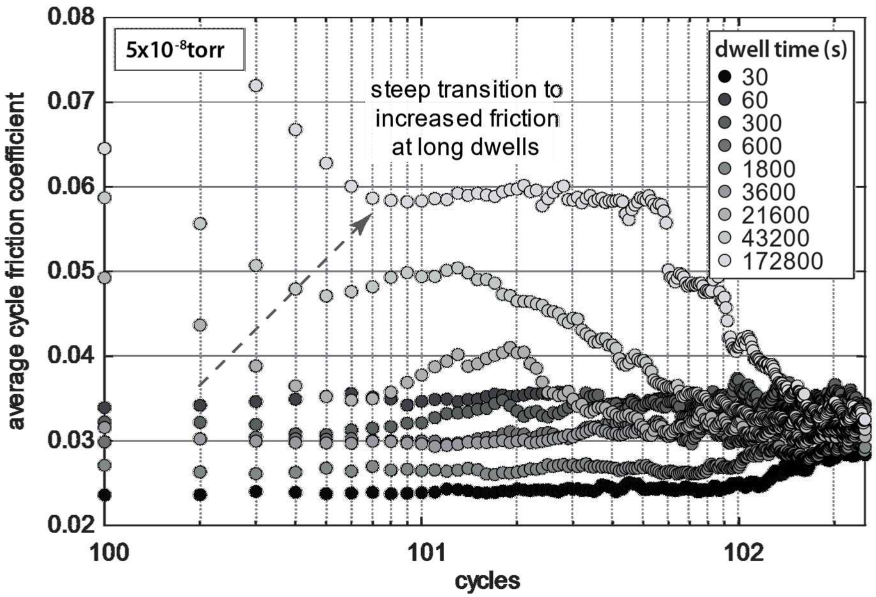
- Increase in initial friction is monotonic, depending on time in between; run-in duration also affected

- This "stop-time" effect is observable in vacuum

- Higher vacuum levels also exhibit this behavior; more abrupt transition at longer dwell times

- Run-in is longer and has a consistently different shape, along with steady state variation at high vacuum





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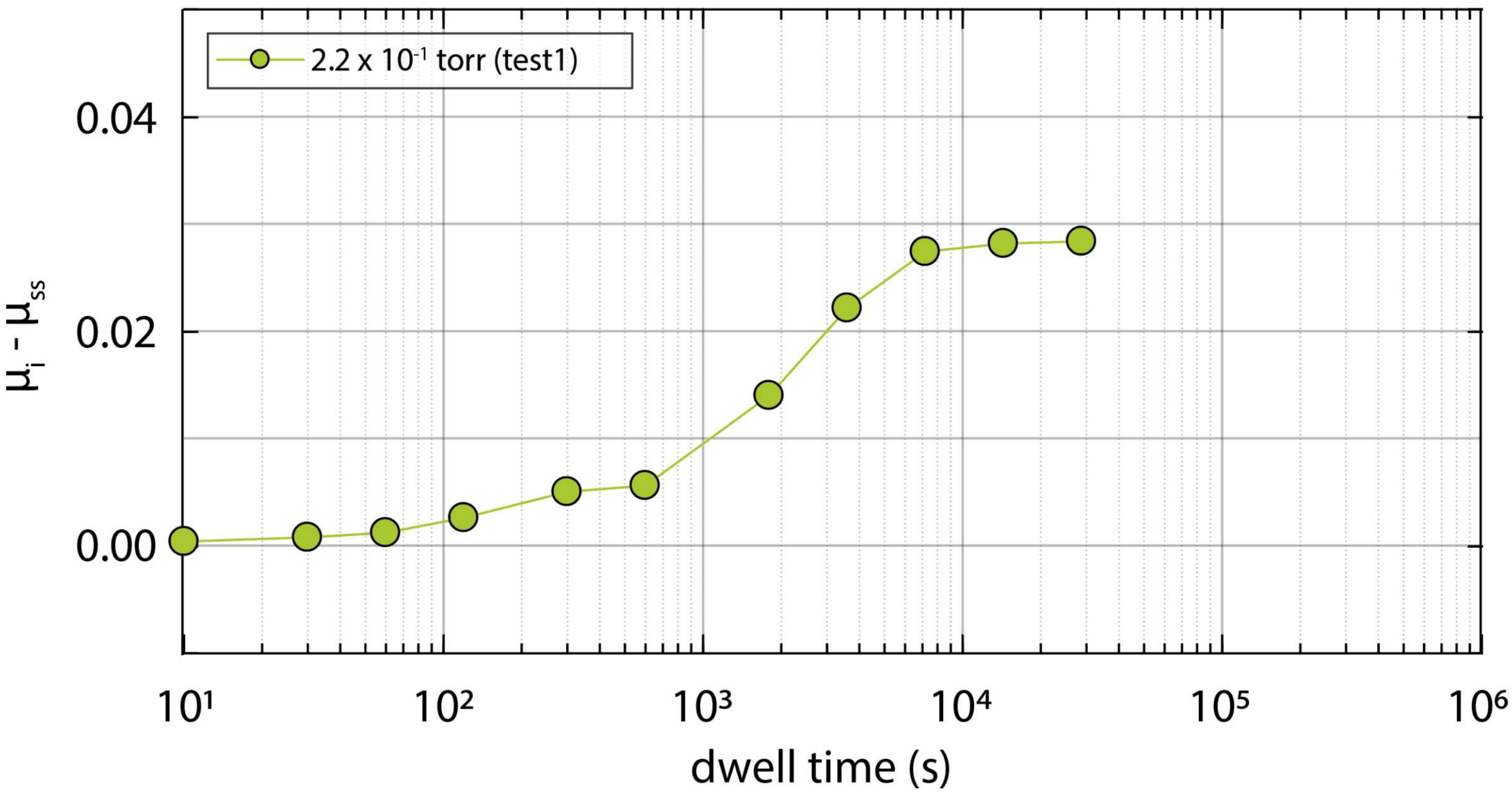
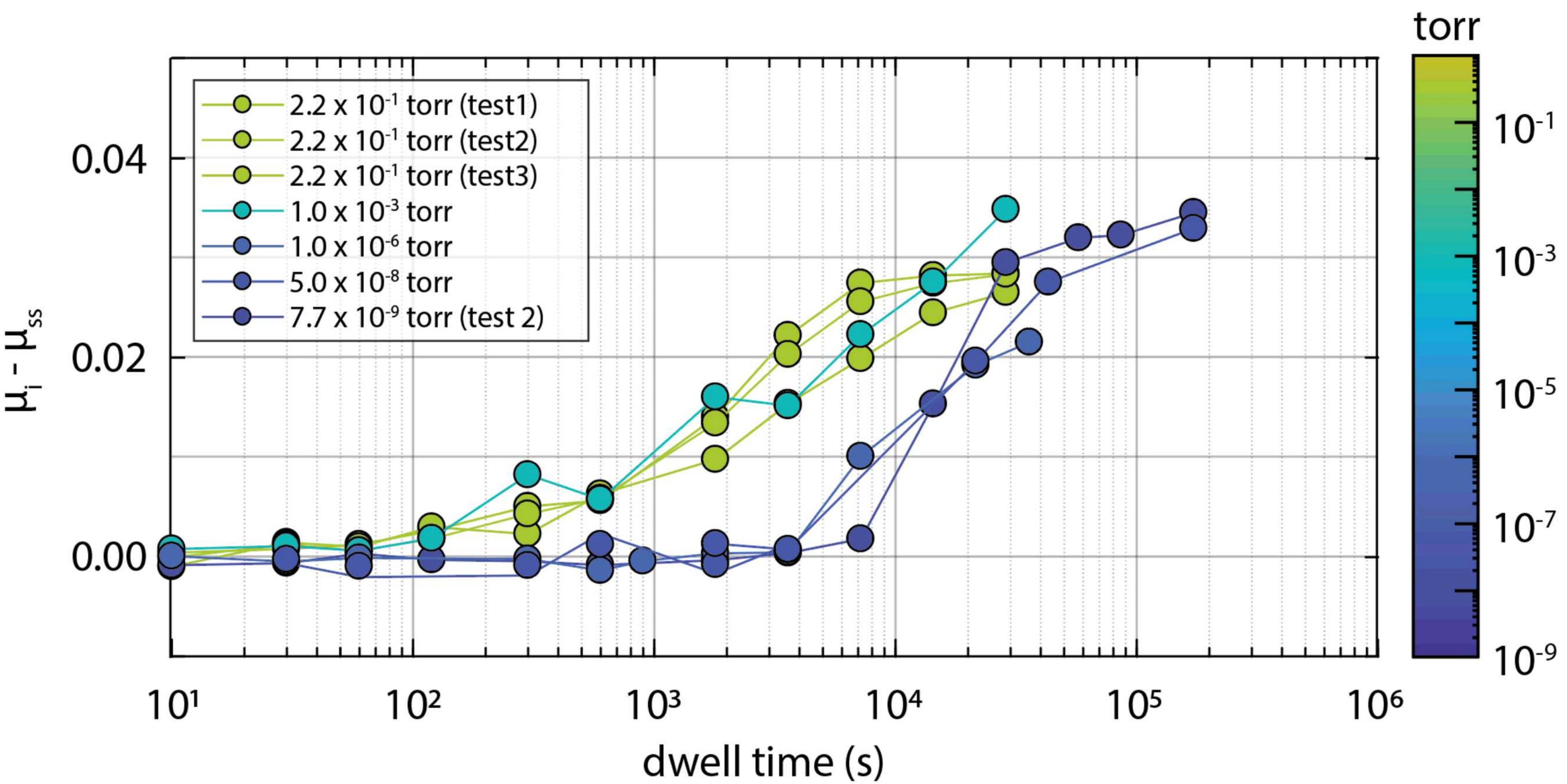


- View data as deltas - can plot the difference between previous steady state and returning initial friction

Re-Run-In Pressure Matters too!



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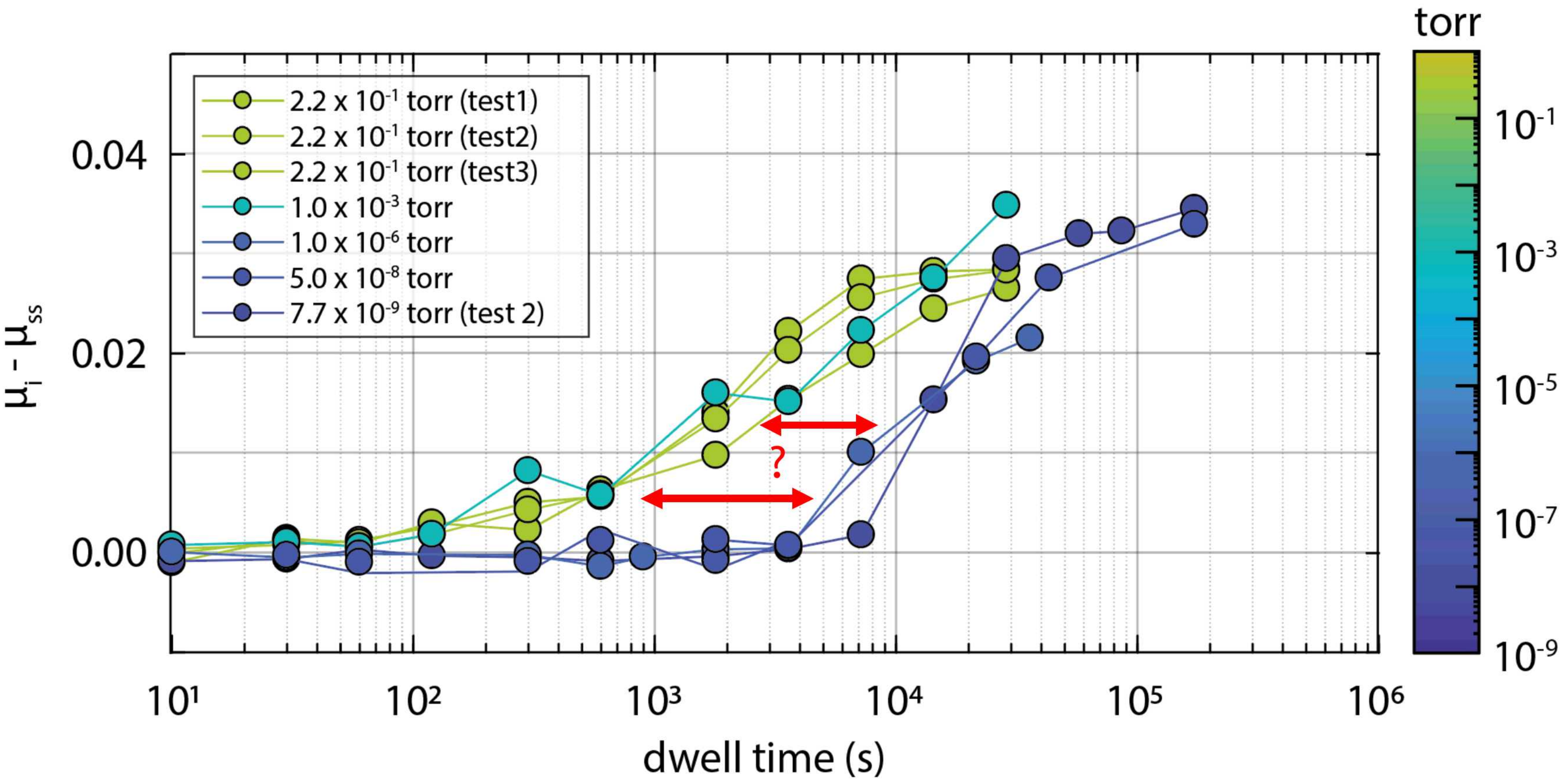

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- Observe stop time across a range of pressures, from $2x10^{-1}$ to $7x10^{-9}$ torr

- Low and high pressures exhibit distinct behaviors over 2-3 orders of magnitude… Friction traces also distinct… suggests different mechanisms responsible

What's Happening: Competing Mechanisms of Re-Run-In

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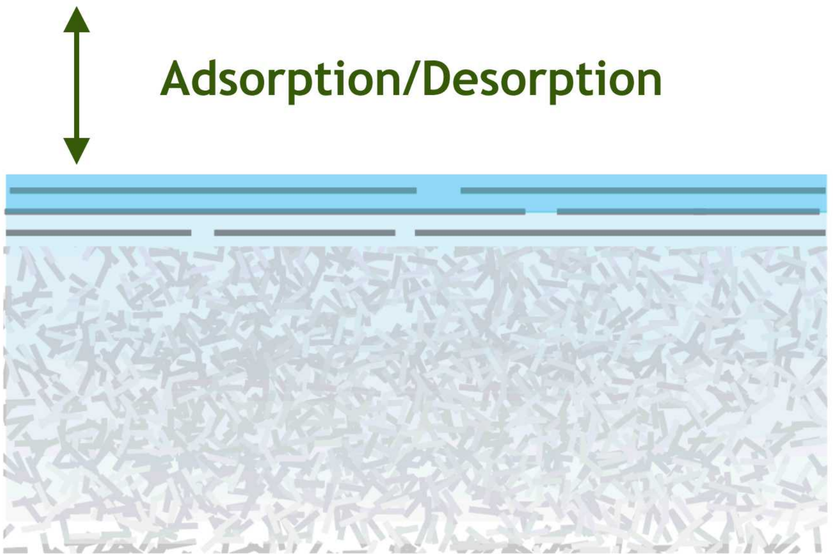

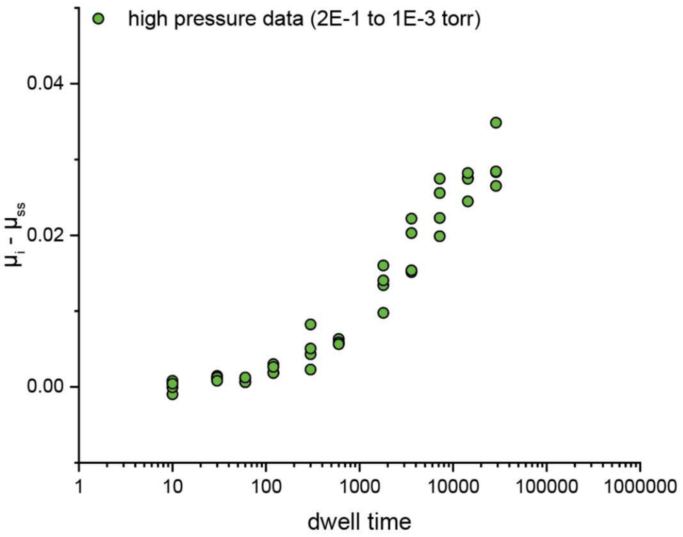

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- **Obvious Theory:** Concentration gradient of contaminants at surface & through bulk affecting friction

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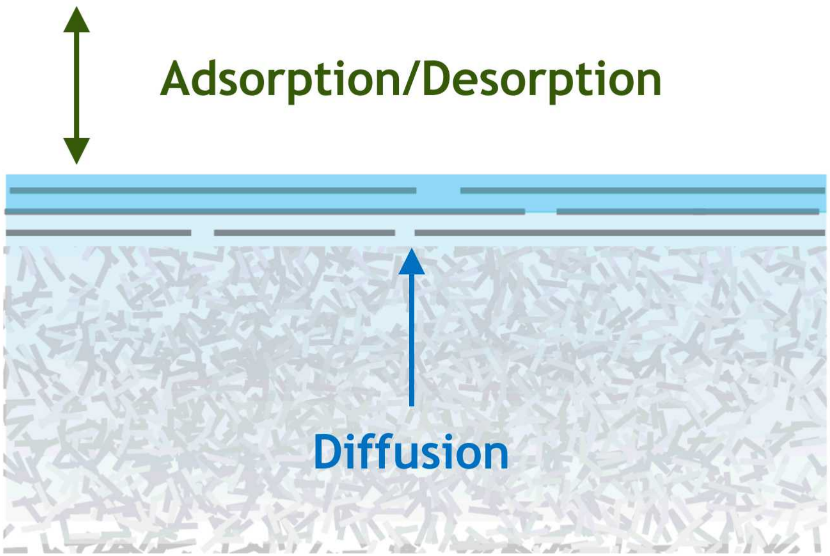
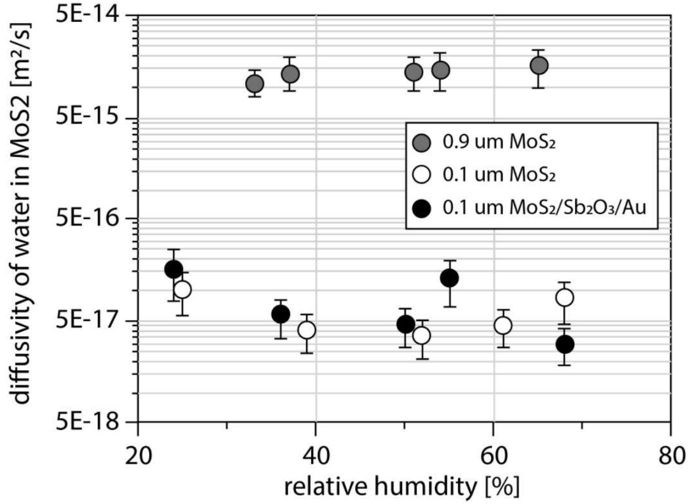
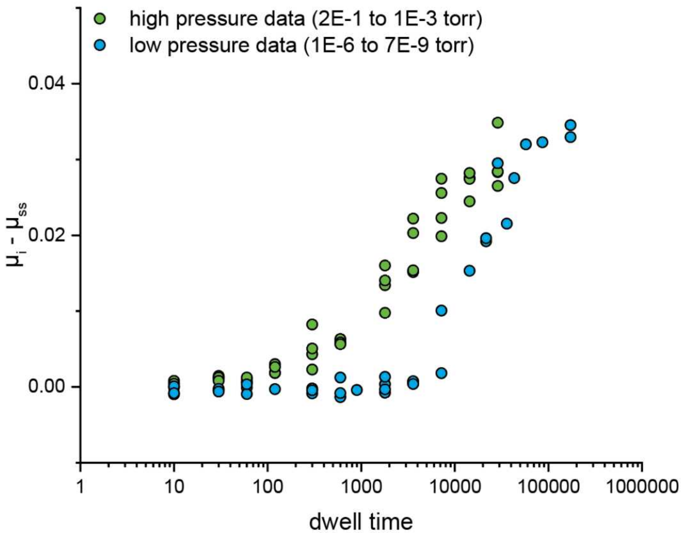






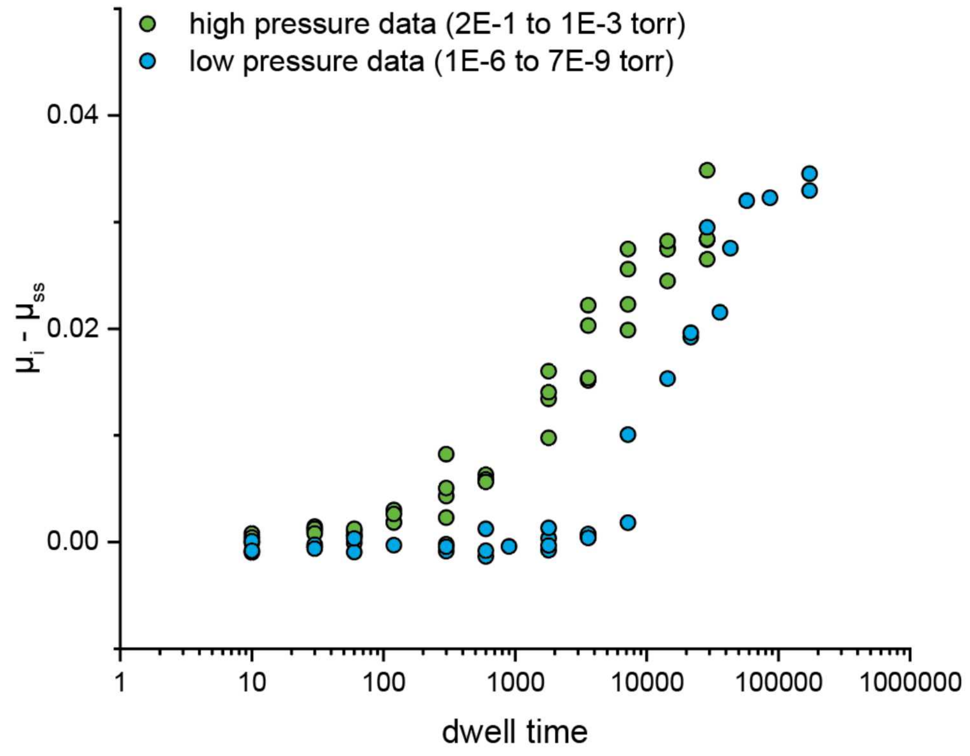
Simple Coverage Model

$$\frac{d\theta}{dt} = k_s(1-\theta) = k_s - k_s\theta$$

$$\theta = 1 - \frac{1}{k_s} e^{-k_s t}$$

- Can simple fractional coverage model help?

- K & s help account for diffusion and sticking (adsorption)



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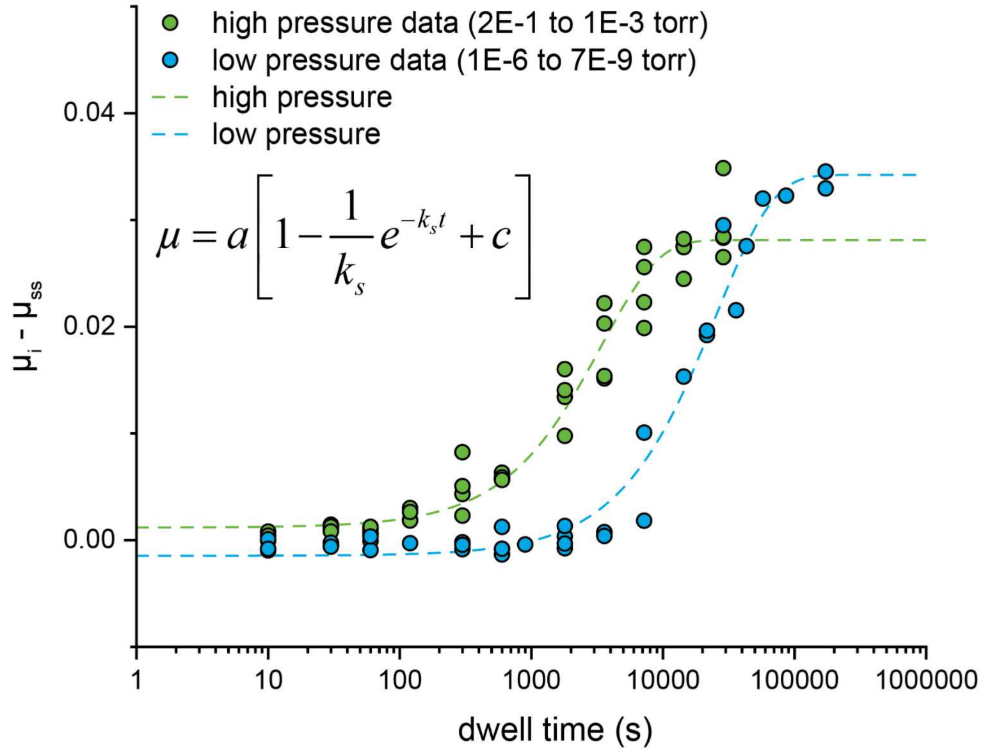

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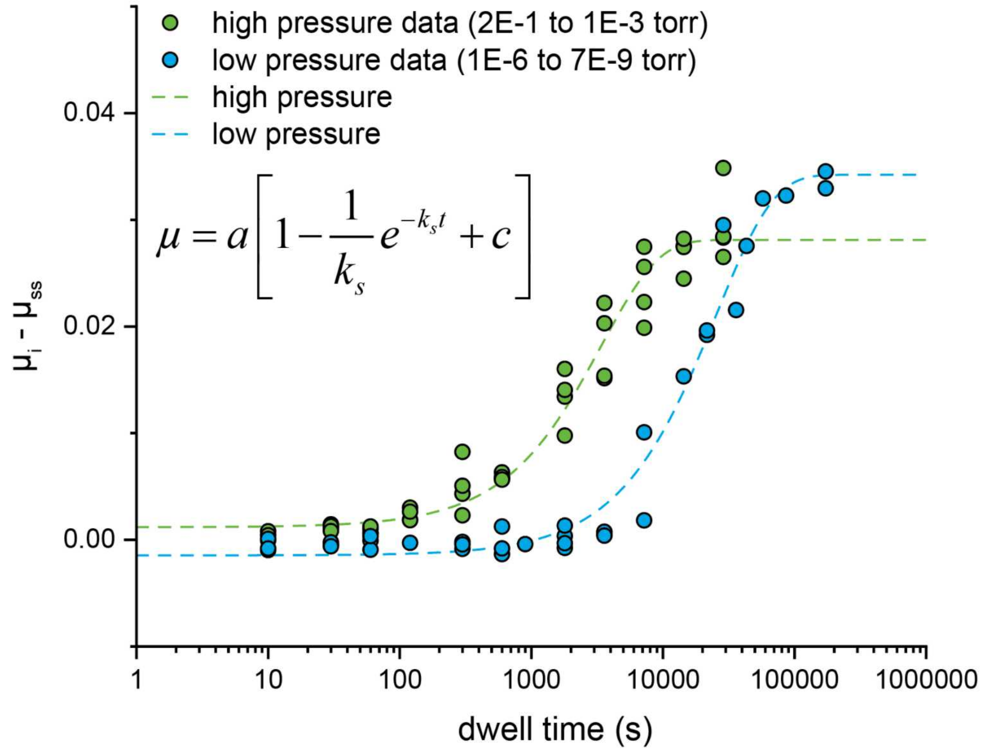
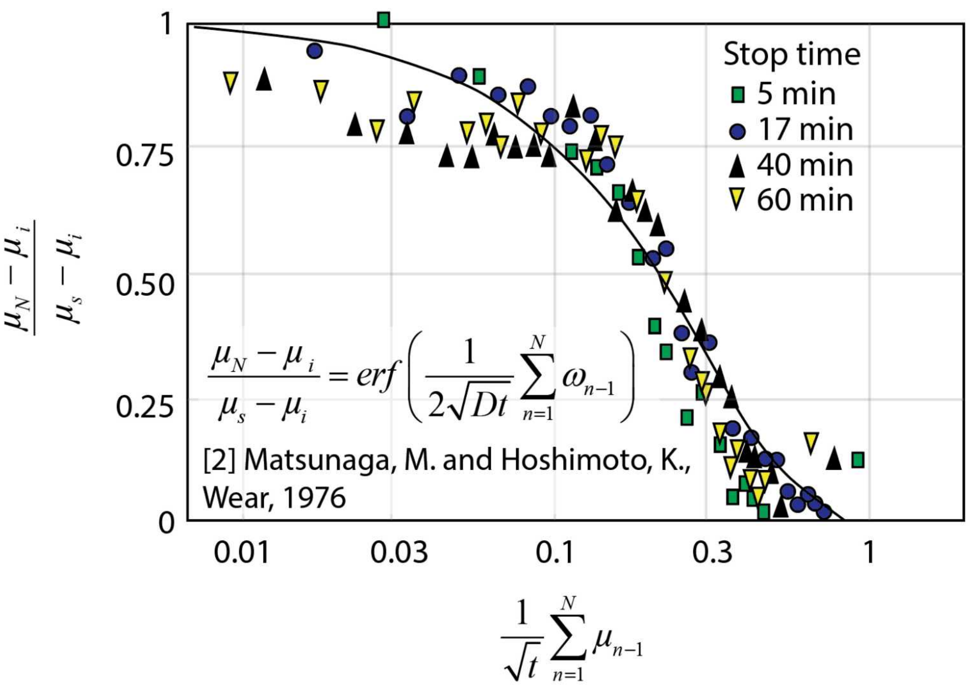
- Adsorbate competition? Different sites? Vapor pressures?



"All models are wrong, but some are useful"
- George Box

Not Alone: Re-Run-In Modeling in Literature



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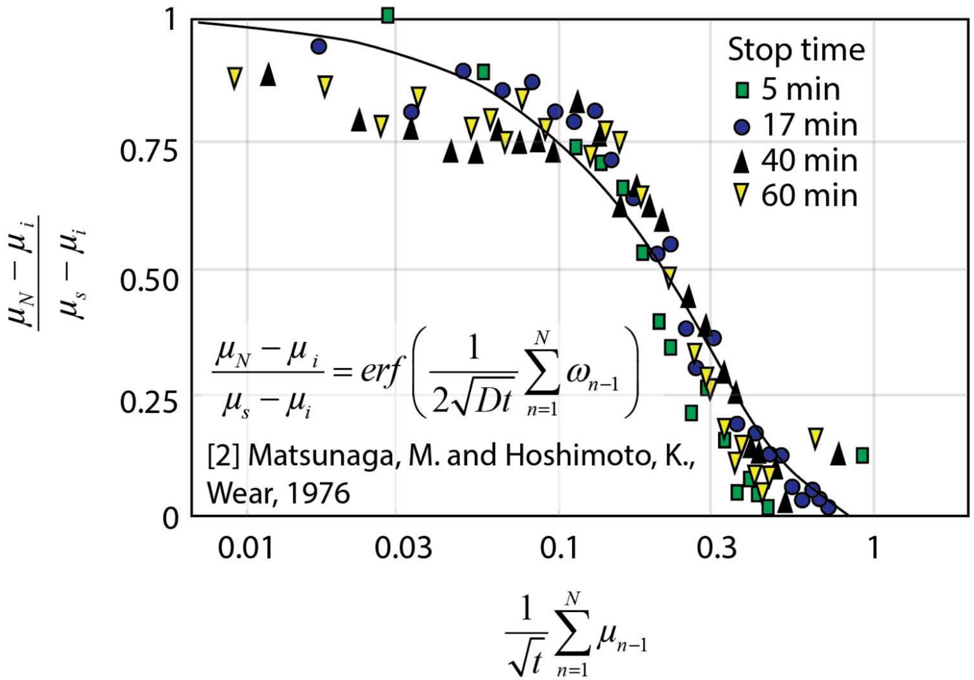

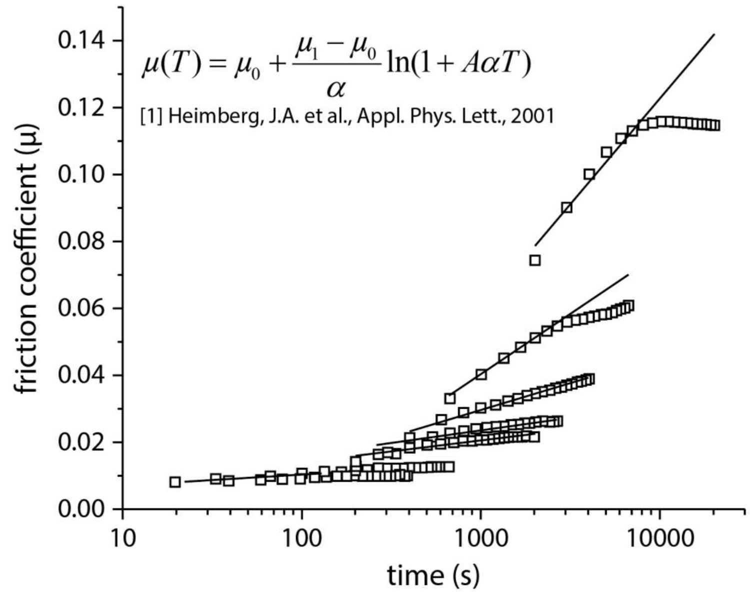

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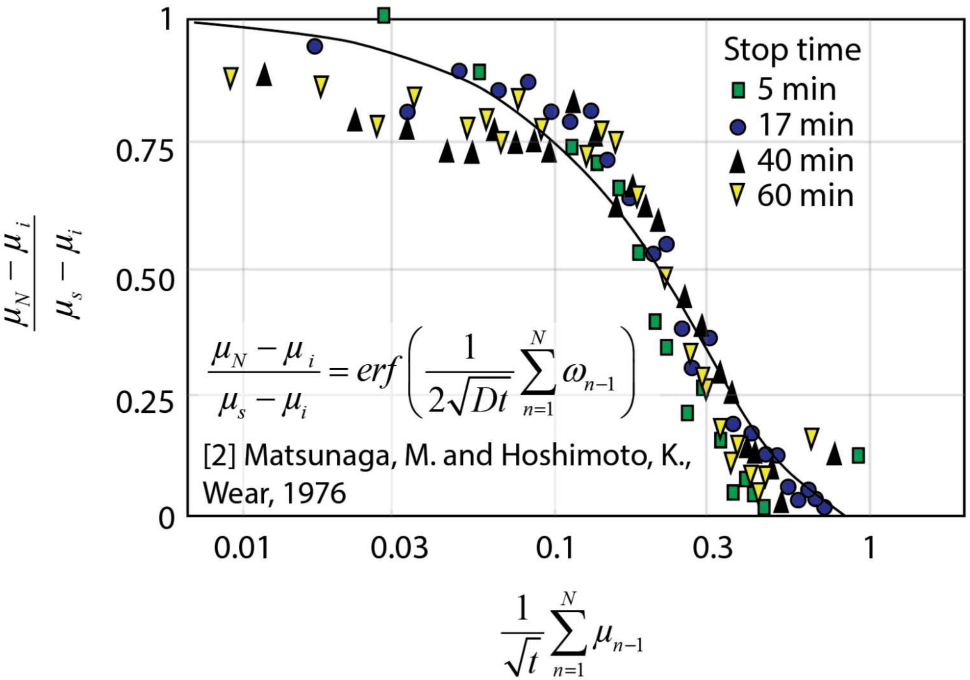

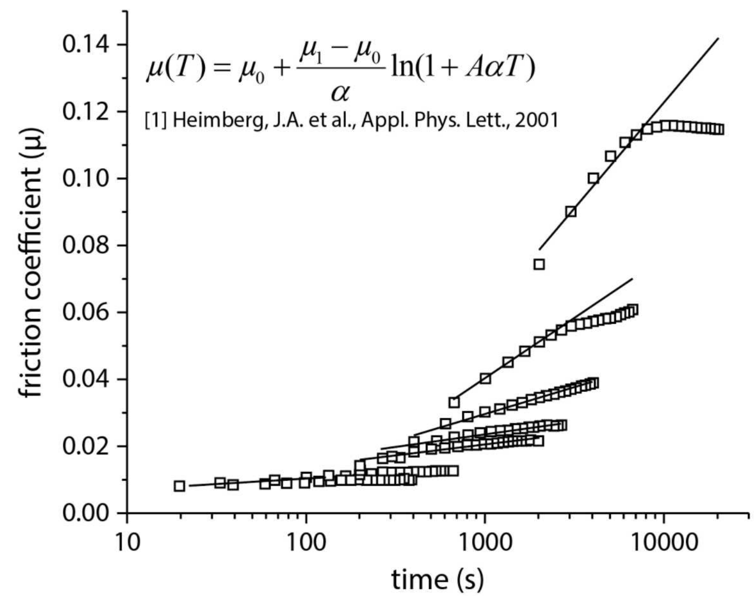

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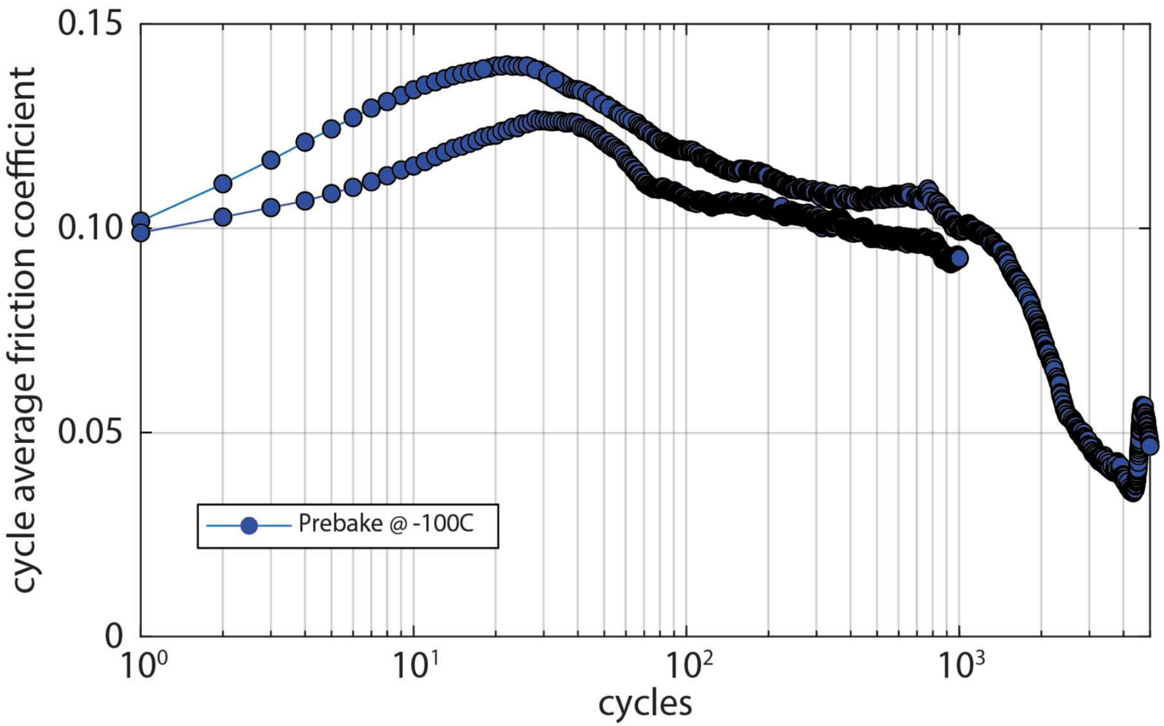
In all cases (two) either the interpretation/physics didn't match – or the fit didn't.

Testing the Mechanism – Playing with Temperature



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- Noticed prolonged (5K cycle) run-in at -100 C (5E-9 torr) on samples prior to baking out

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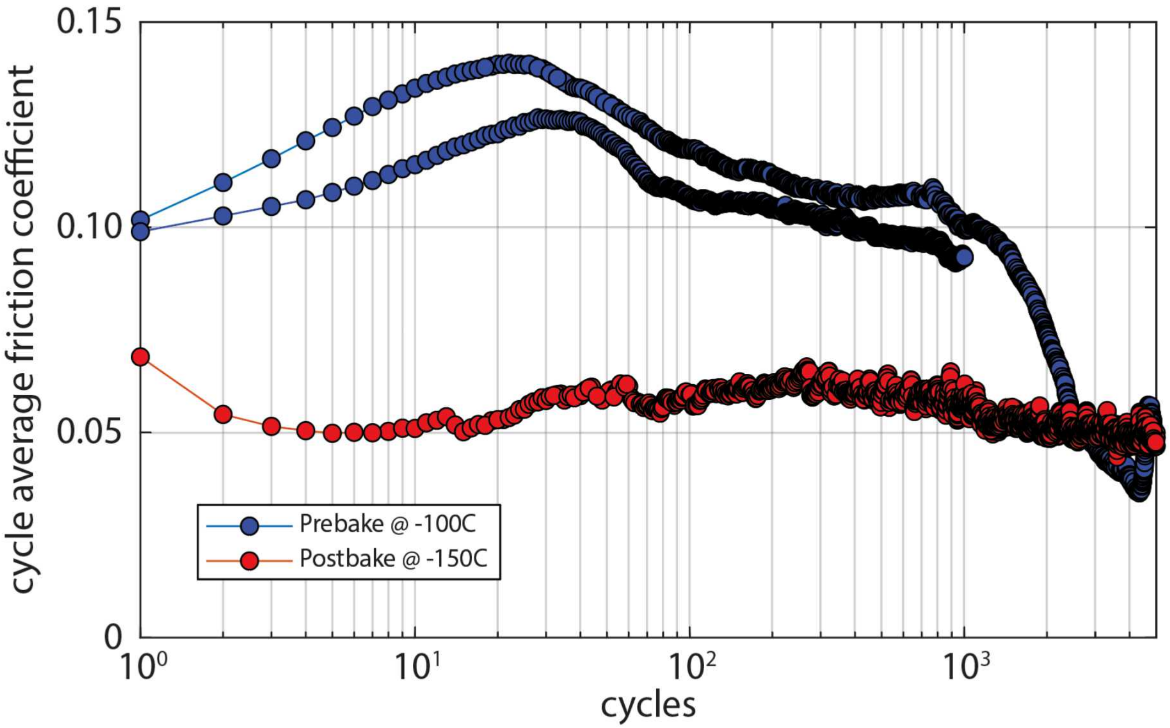
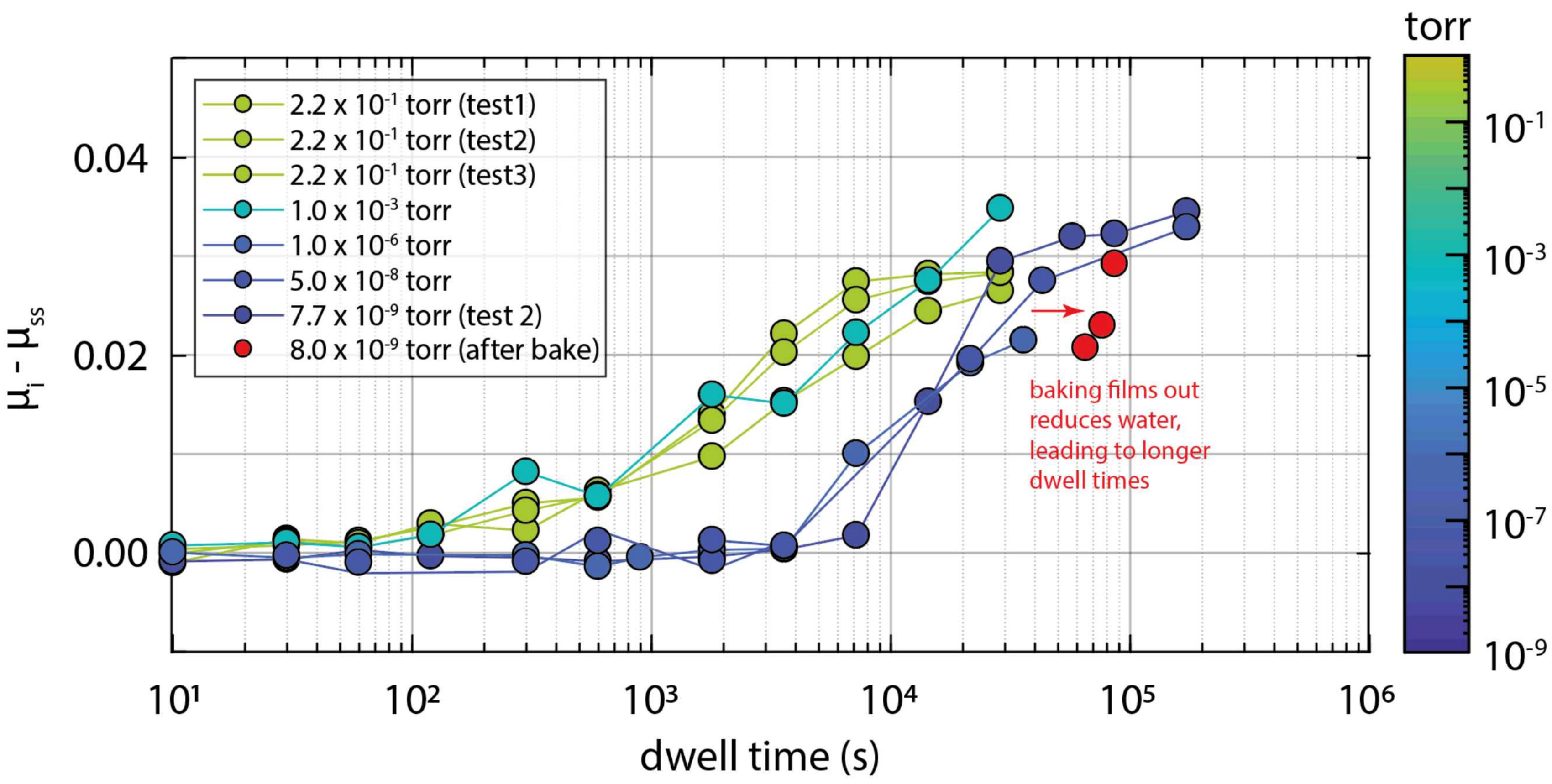


- At low temperature, likely ice or water retained in subsurface
- Noticed prolonged (5K cycle) run-in at -100 C (5E-9 torr) on samples prior to baking out
- Baking films out at 145C for 24 hours greatly diminishes resulting run-in at low temps
- Water likely diminished at surface/subsurface & diffusion much slower at low temps

Does baking out shutdown re-run-in?



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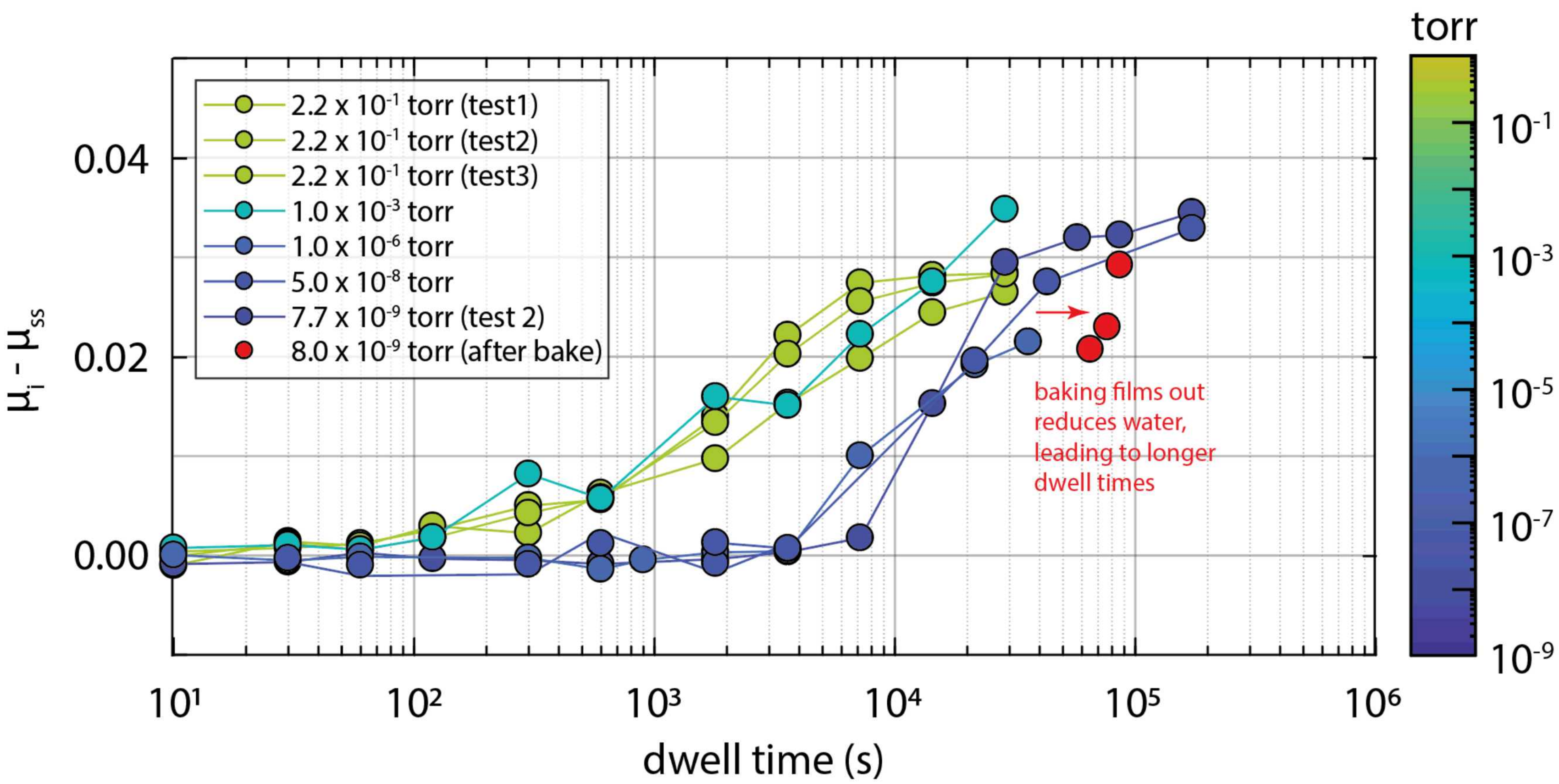
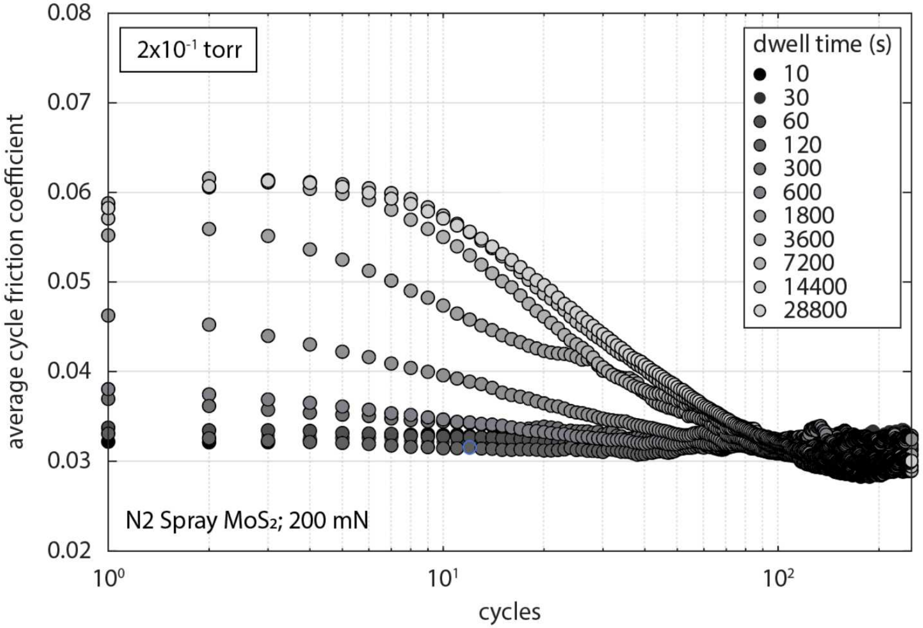
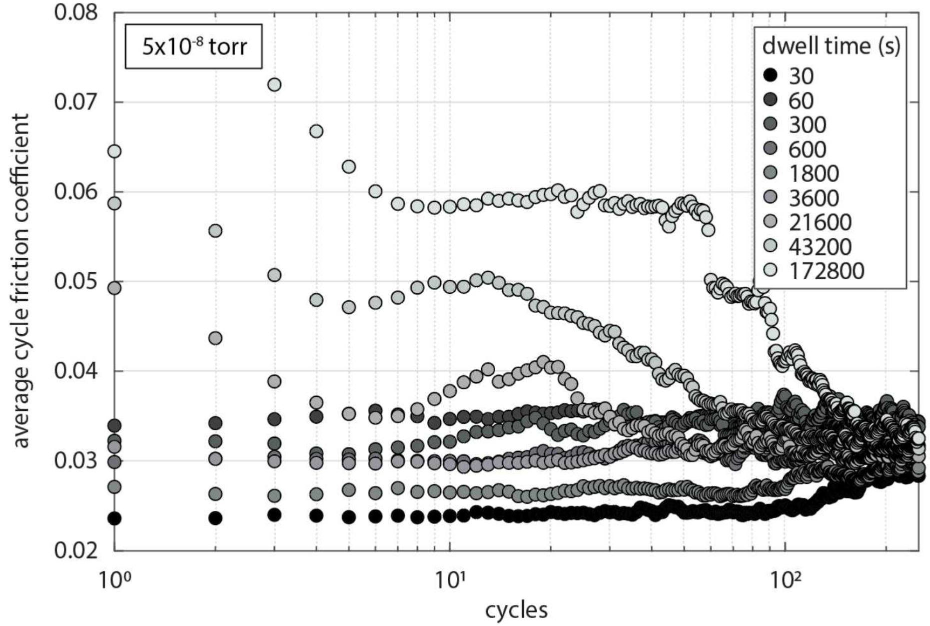


- Dwell times still persist, even after baking out, albeit at longer dwells – lots of water remains

- Likely removing water from the surface of the film – reducing concentration and slowing diffusion… more testing necessary

Comparisons to "Initial" Run-in





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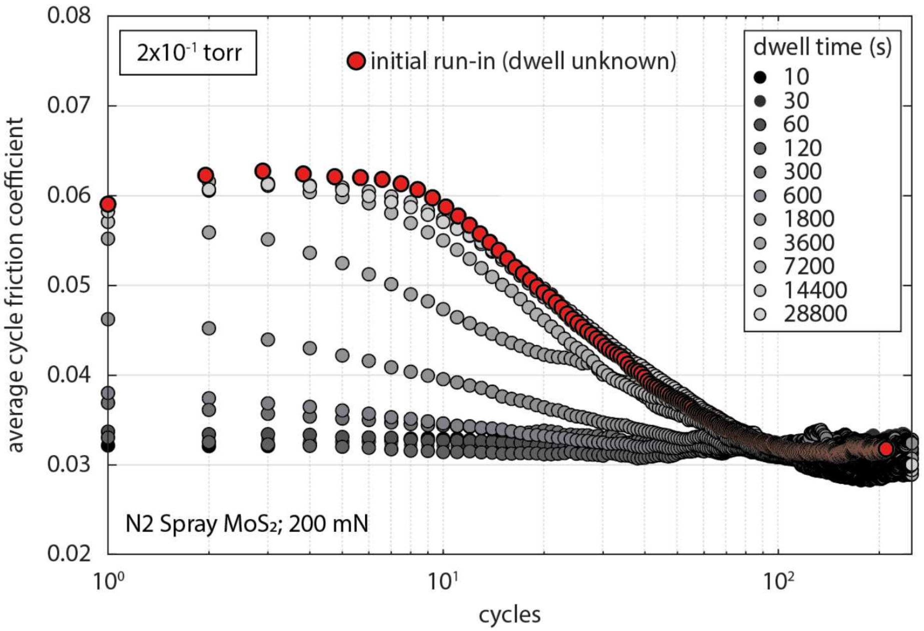
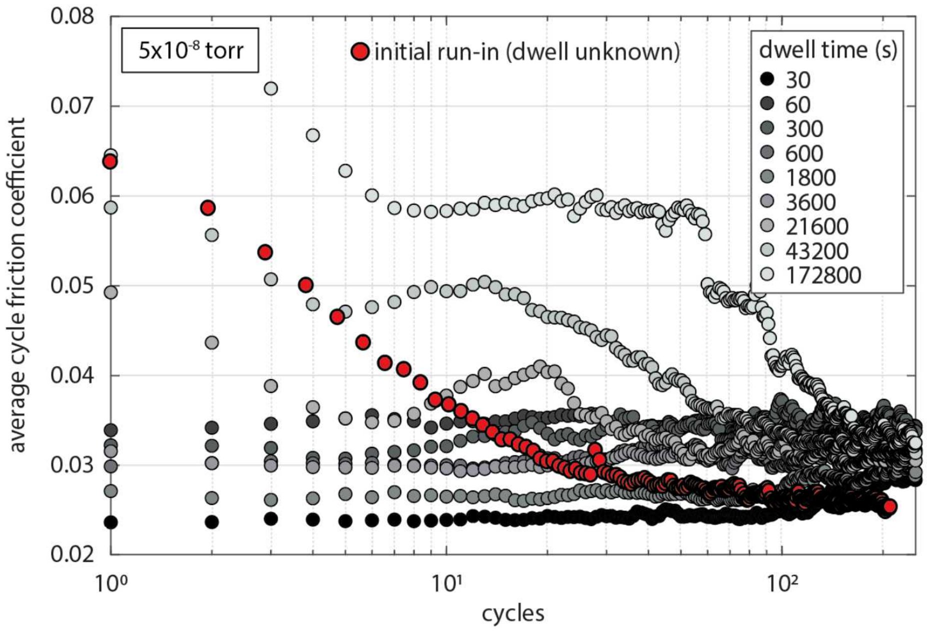




- Can look back at initial run-in to see how much mechanisms of latent/adsorbed water contribute

- High pressure case very convincing – initial run-in very similar to longest dwell re-run-in;
  - Suggests run-in on impinged films may be mostly adsorbate driven, not microstructure

- Low pressure case exhibits same initial friction, but no "shoulders" typically seen
  - Interpretation still incoming; may be initially adsorbed water

Conclusion

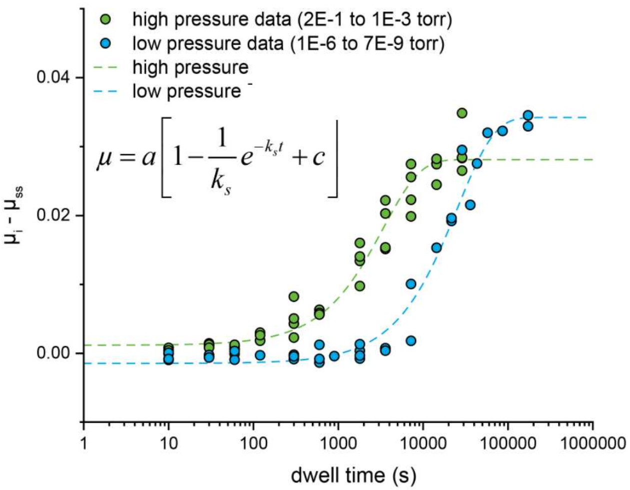
- Re-run-in strongly influenced by adsorbed/latent water, changes with time and pressure and diminished but not eliminated by baking out

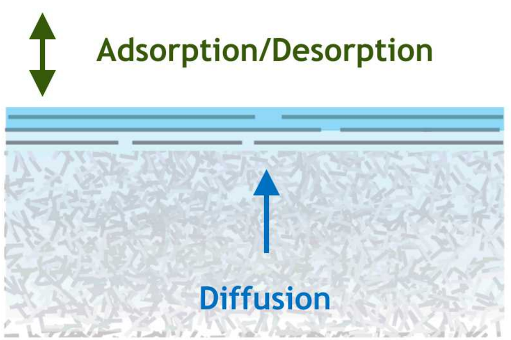
- Competing mechanisms in re-run-in behavior at high pressures (adsorption) versus low pressures (diffusion)

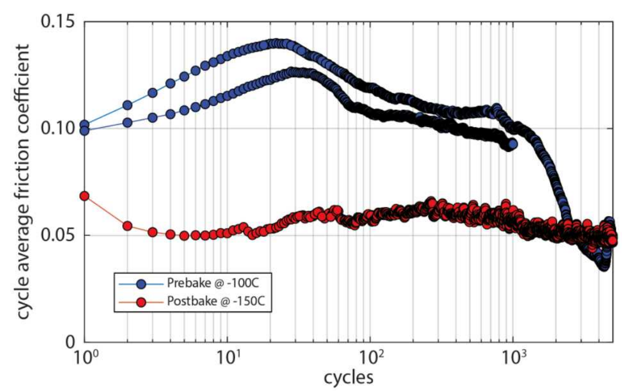
- Simple coverage model fits data well, requires tuning and use of relevant materials parameters governing behavior

- Baking out films helps remove water and prolong re-run-in – much water still remains in MoS2 films

- Future work
  - How temperature/microstructure change diffusivity/sticking prob of contaminants to in re-run-in
  - Tune coating design to aid in minimizing latent water







The Team

SNL

Nic Argibay (experimental staff)

Mike Dugger (experimental staff)

Mike Chandross (simulation staff)

Adam Hinkle (simulation postdoc)

Mark Wilson (simulation postdoc)

Brendan Nation (technologist)

John Johnson (technologist)

Morgan Jones (technologist)

Lehigh U.

Brandon Krick (Professor in Mech. E.)

Tomas Babuska (grad student/intern)

TAMU

James Batteas (Professor in Chem.)

Quentarius Moore (grad student/intern)

Sandia National Laboratories

NNSA National Nuclear Security Administration

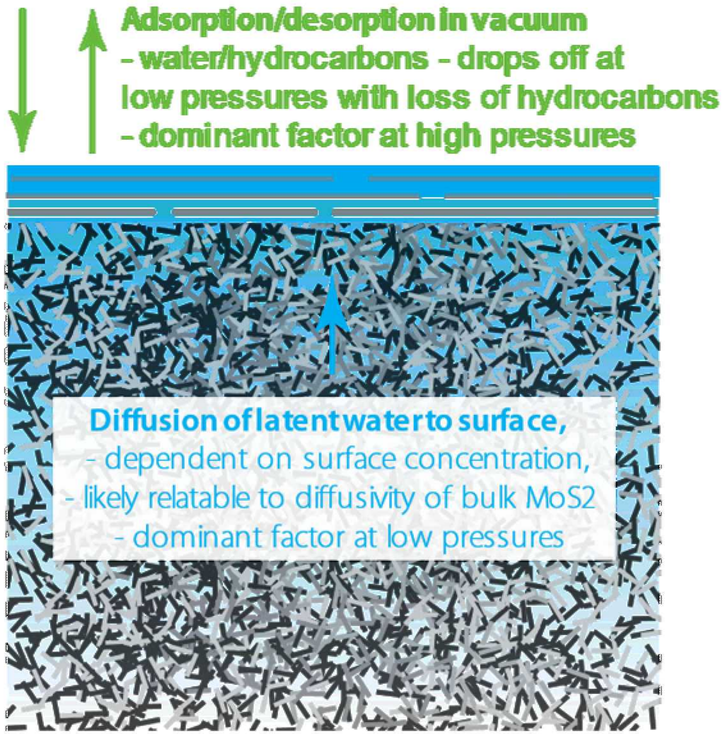
U.S. DEPARTMENT OF ENERGY

LEHIGH UNIVERSITY
SURFACE INTERFACES AND MATERIALS TRIBOLOGY LABORATORY

Thanks!

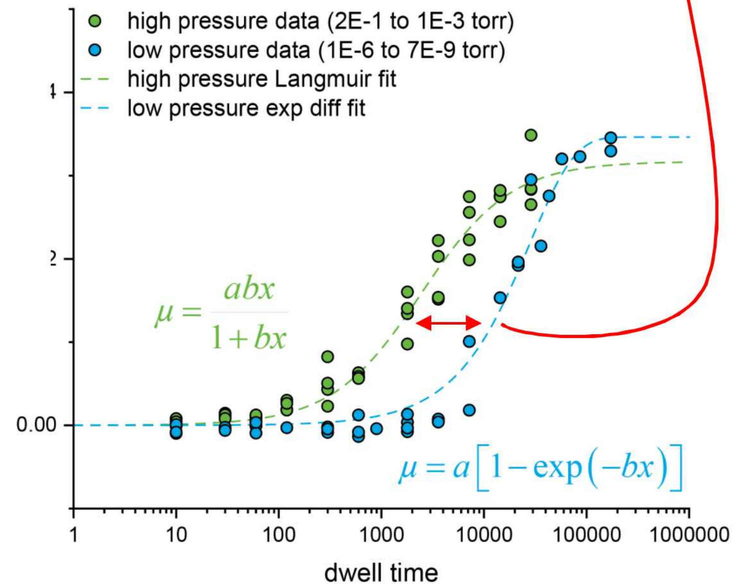
APPENDIX

What's Happening: Proposed Mechanisms of Re-Run-In



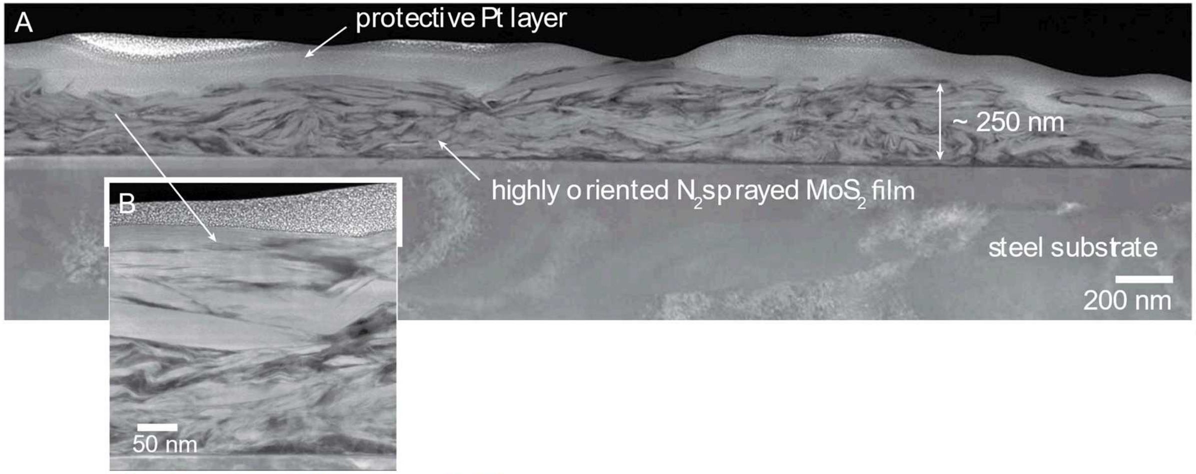
Oriented surfaces help trap water in the run-in surface/sub surface. More tortuous path providing slower diffusion

Concentration of contaminants in vacuum do not decrease linearly; different vapor pressures & sticking coefficients

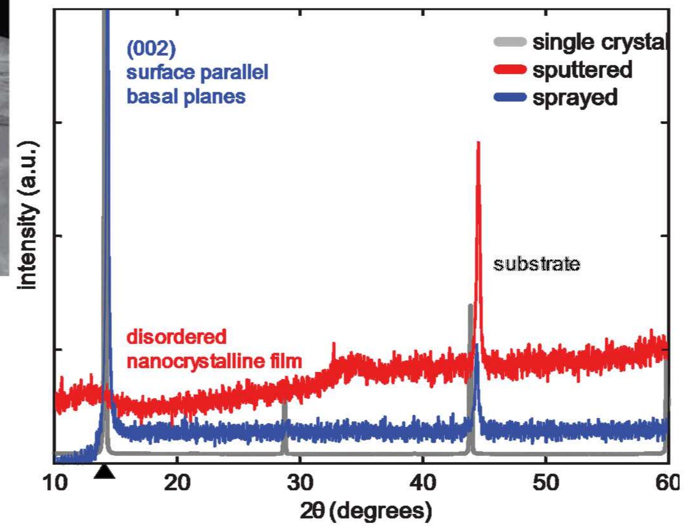


need more data & functional forms that make physical sense for this application

Run Films In before use! Industry Standard! Shows Over!

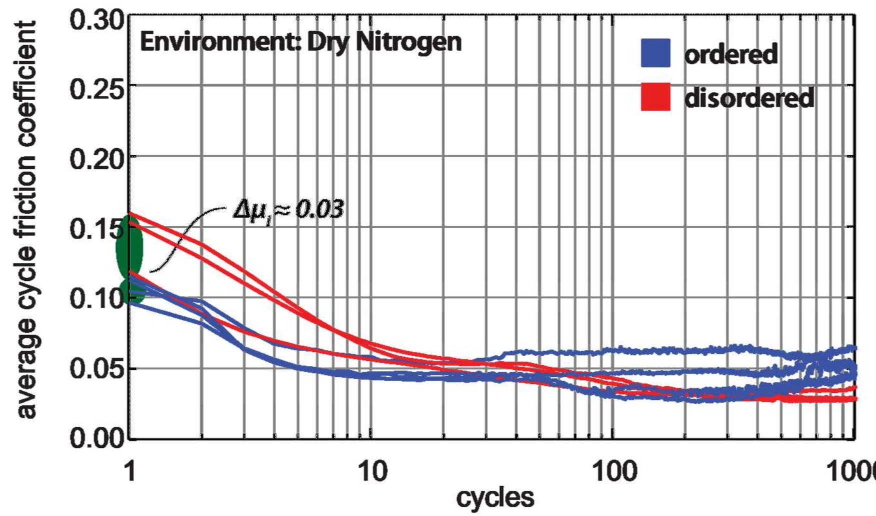


highly ordered (N2 sprayed) [1]



- To make things simple, we focused on ordered, impinged films

- Little to no reorientation required – exhibit lower initial friction coefficients
- Less susceptibility to environment (Krick)

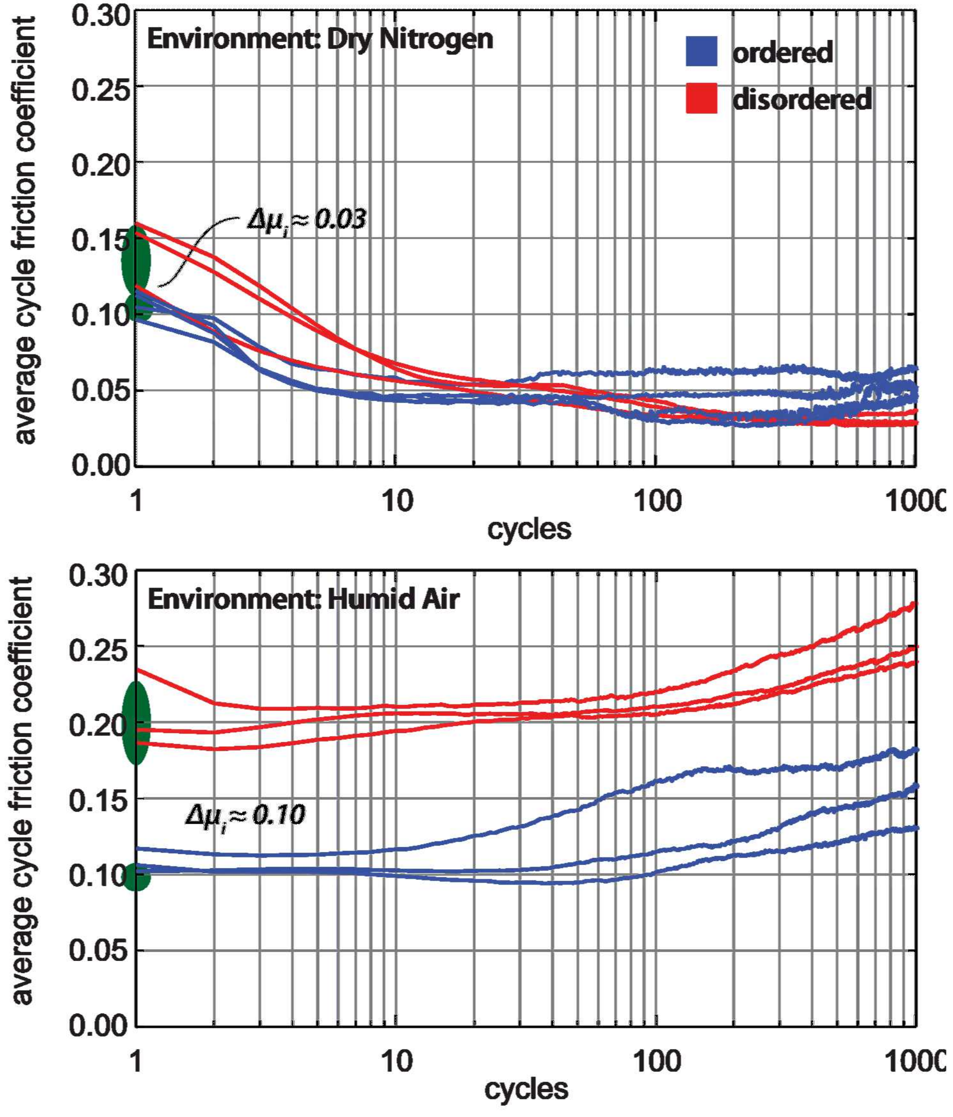


Run-In Governed by Microstructure

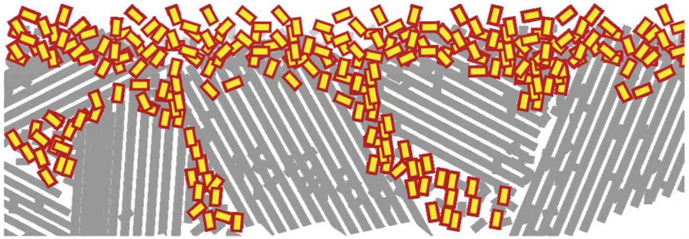
- Sprayed (ordered) films exhibit consistently lower initial friction than sputtered (disordered) films

- Ordered films unaffected by present of water initially

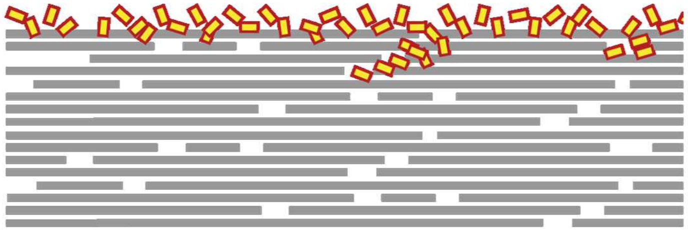
- Where long-range, ordered films exist – they persist. It is hypothesized that water poisons this ability.



Run-In Factor: Oxidation



disordered structure



highly-ordered structure

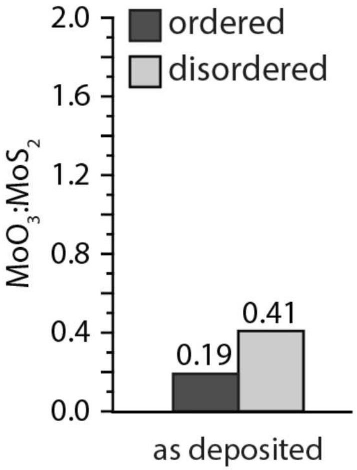
- Oxidation resistance should benefit in the same way that run-in does from ordered surfaces

- Higher degree of basal orientation and less available edge site (large crystals) should reduce oxidation

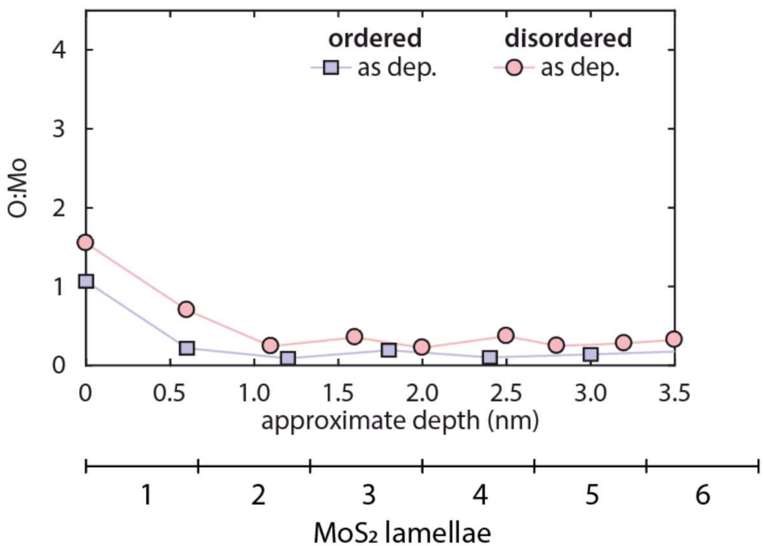
- Ordered structure also provides more tortuous path into the bulk for further interactions

Oxidation vs Microstructure – XPS & LEIS Study

Mo 3p signal - $MoO_3:MoS_2$ ratio



oxygen : molybdenum ratio



- Look at amount of Mo as sulfide or oxide after exposures to O2 @ 250C and Atomic Oxygen (30 min)

Oxidation vs Microstructure – XPS & LEIS Study

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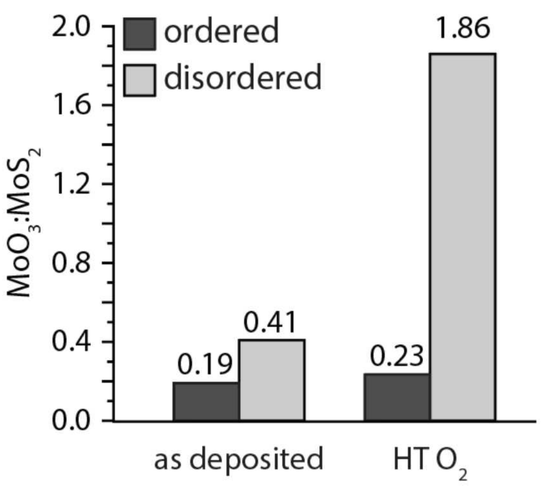

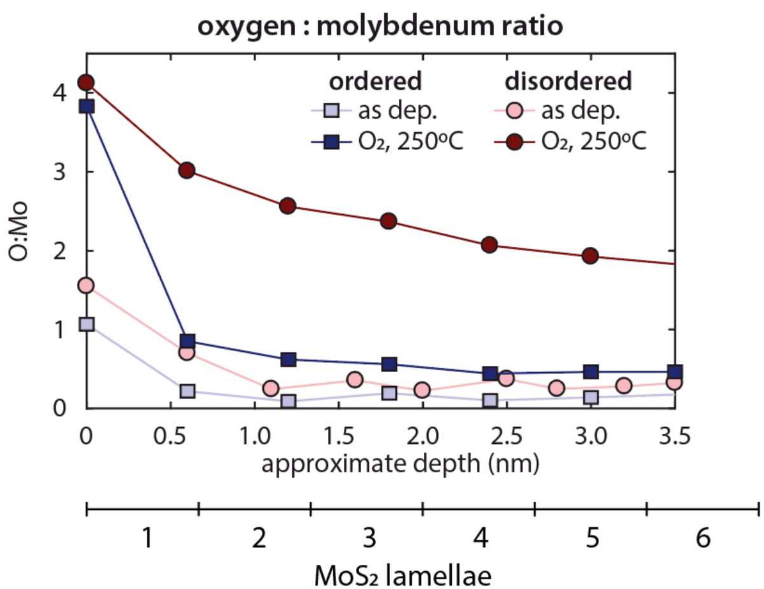

- Look at amount of Mo as sulfide or oxide after exposures to O2 @ 250C and Atomic Oxygen (30 min)

Oxygen Gas (30 min @ 250°C)

- XPS indicates minimally more oxide for ordered films while disordered films have more

- LEIS shows this is mostly surface limited for ordered films and through the surface for disordered

Oxidation vs Microstructure – XPS & LEIS Study

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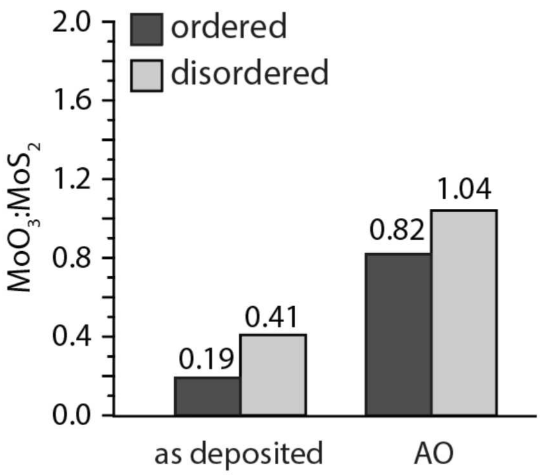
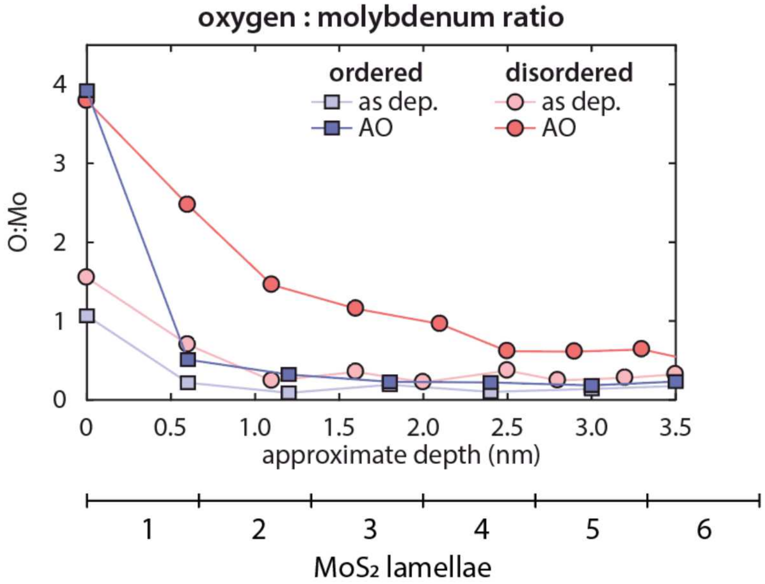




- Look at amount of Mo as sulfide or oxide after exposures to O2 @ 250C and Atomic Oxygen (30 min)

<u>Oxygen Gas (30 min @ 250°C)</u>

- XPS indicates minimally more oxide for ordered films while disordered films have more

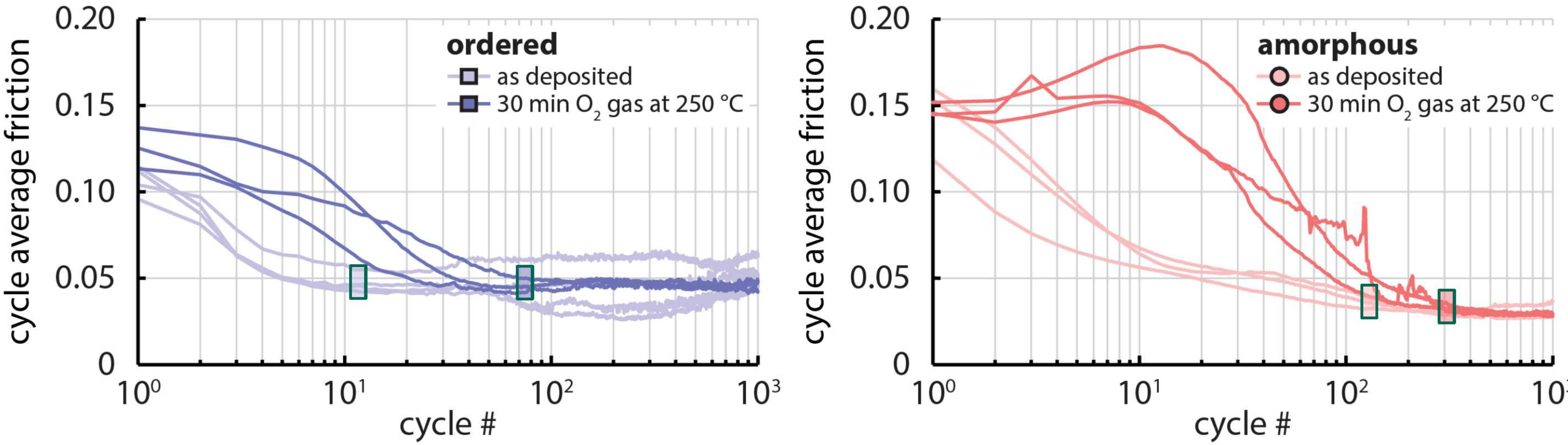
- LEIS shows this is mostly surface limited for ordered films and through the surface for disordered

<u>Atomic Oxygen (30 min @ RT)</u>

- AO exposures show similar increases in oxidation via XPS

- Again LEIS shows oxygen only at surface for ordered films and not much below the surface for disordered

Friction comparison for HT O2 aged coupons

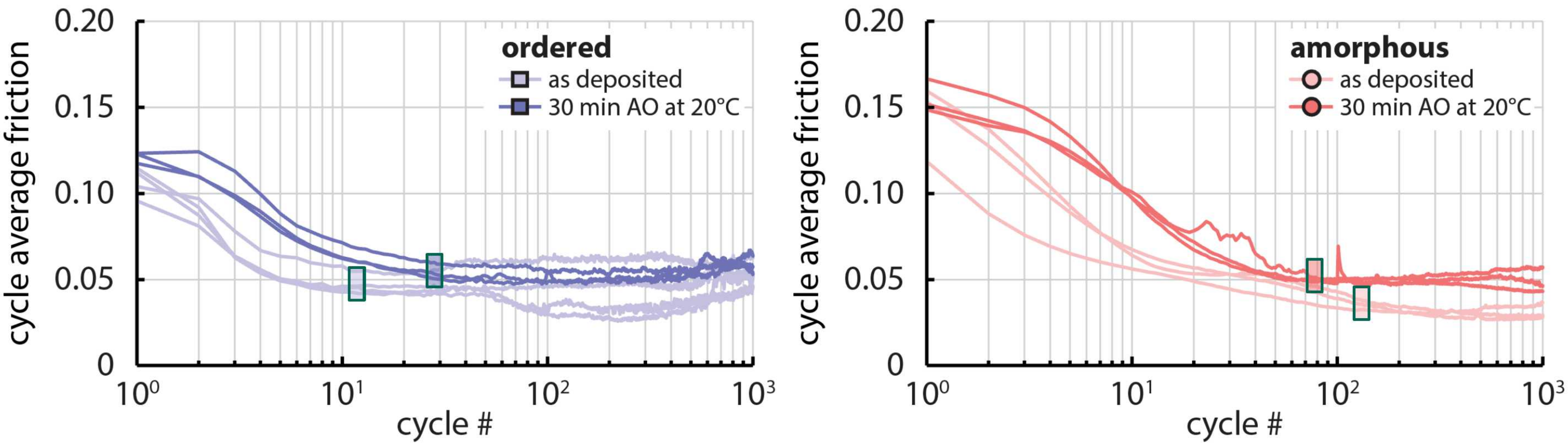


- Exposing Films to oxygen gas at 250˚C for 30 min revealed differences in run-in behavior

- Both films were effected, with disordered films experience much longer high friction run-in phases

- Believe prolonged run-in for disordered films is due to oxygen diffused into the subsurface

Friction comparison for AO aged coupons



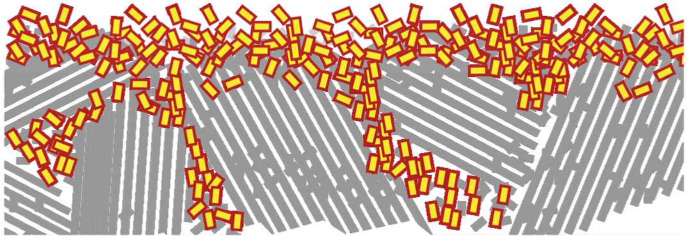
- Exposing Films to atomic oxygen at RT for 30 min revealed minimal differences in run-in behavior

- Attribute the slight increases to thin layer of oxide formed on the surface (confirmed by LEIS depth profile)

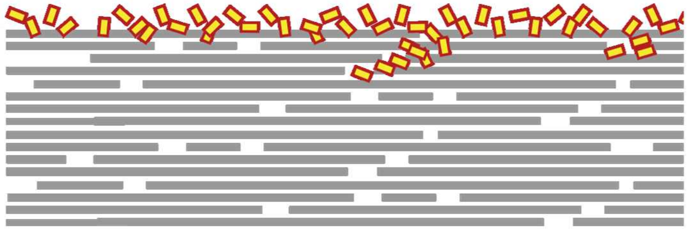
- Recent (and past) examples in literature suggest ozone and atomic oxygen form passivating surface layer preventing further interaction with oxygen (Sen et al., J. Appl. Phys, 2014)

Oxidation vs Microstructure: Mechanisms

High density of edge sites at surface /subsurface promote oxidation with O_2



disordered structure



highly-ordered structure

Oxidation vs Microstructure: Mechanisms

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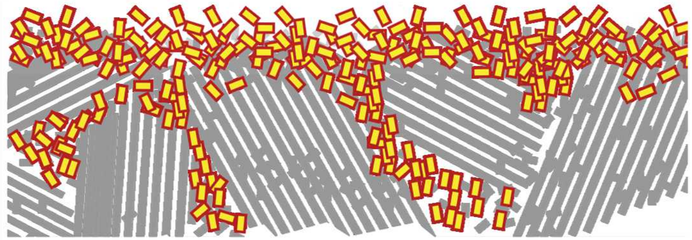
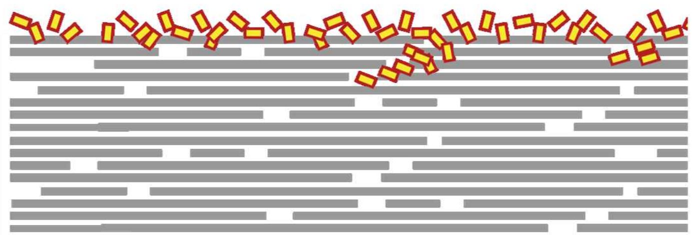


disordered structure

Ordering limits oxidation to outer surface… (O_2 & AO)



highly-ordered structure

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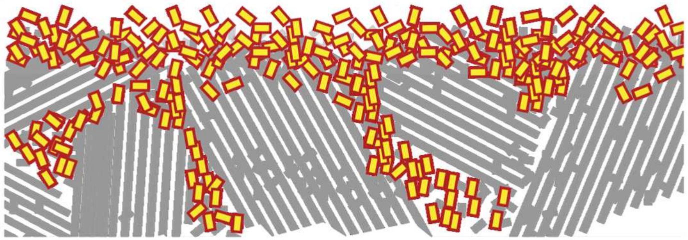

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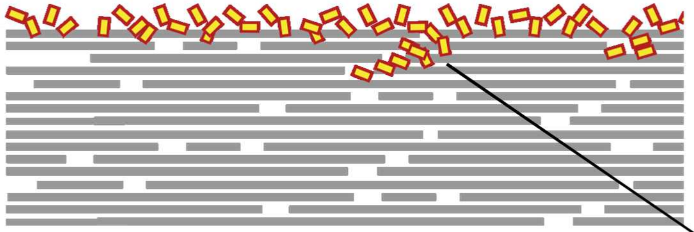

highly-ordered structure

O_2 only interacts with edges/boundaries… doesn't dissociate on surface

Oxidation vs Microstructure: Mechanisms

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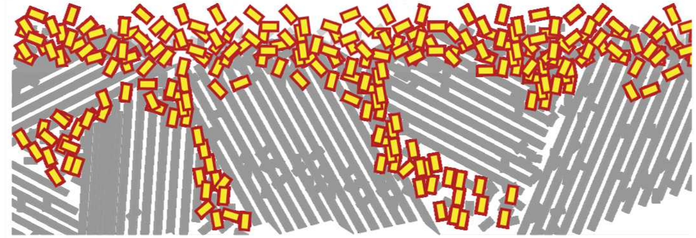

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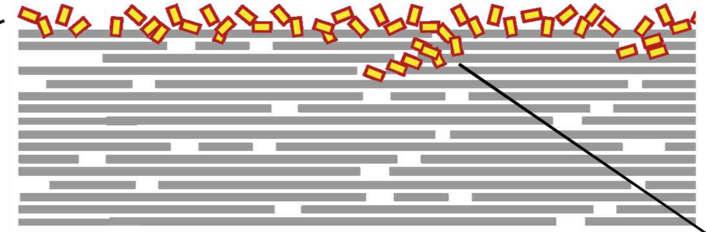


highly-ordered structure

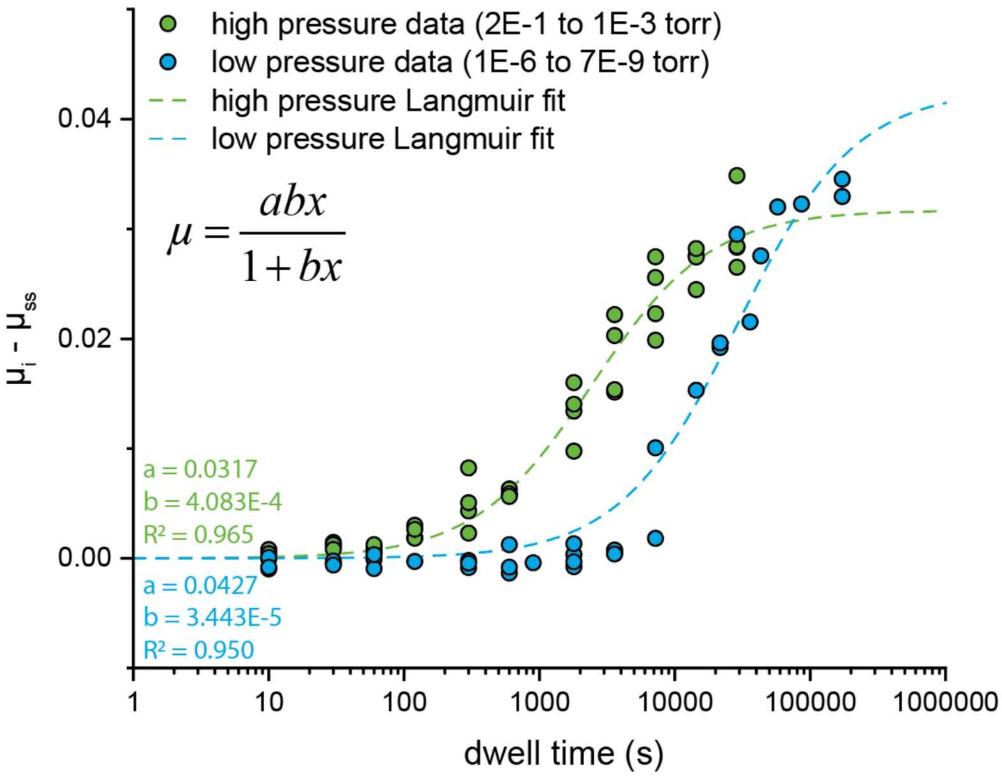
O_2 only interacts with edges/boundaries… doesn't dissociate on surface

Oxidation studies on ordered and disordered films reveal the importance of large, basally oriented phases in preventing oxidation

Langmuir Equation – Adsorption Approximation

- If adsorption/desorption are at play, Langmuir might help explain results

- Adsorbate behaves as ideal gas at isothermal conditions; describing the fraction of occupancy of an adsorbent at possible adsorption sites



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Treating pressure as time?

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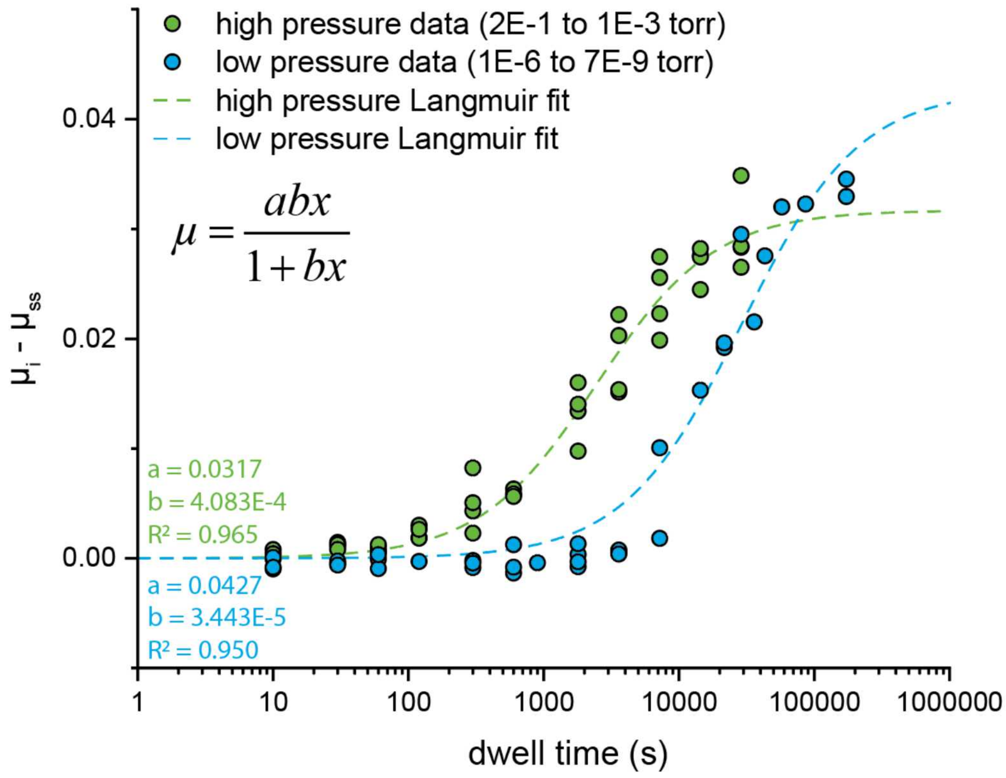

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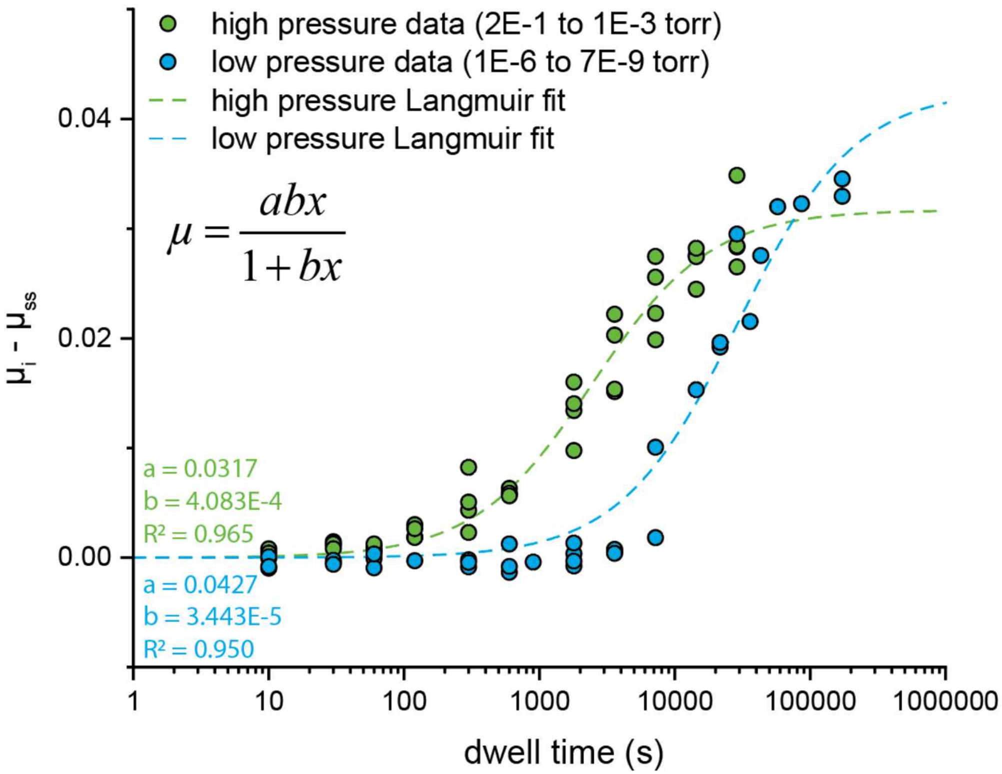
Treating pressure as time?

- Adsorbate behaves as ideal gas at isothermal conditions; describing the fraction of occupancy of an adsorbent at possible adsorption sites

- Fits the high pressure curves better, not capturing the roll-off at long dwell times for low pressures

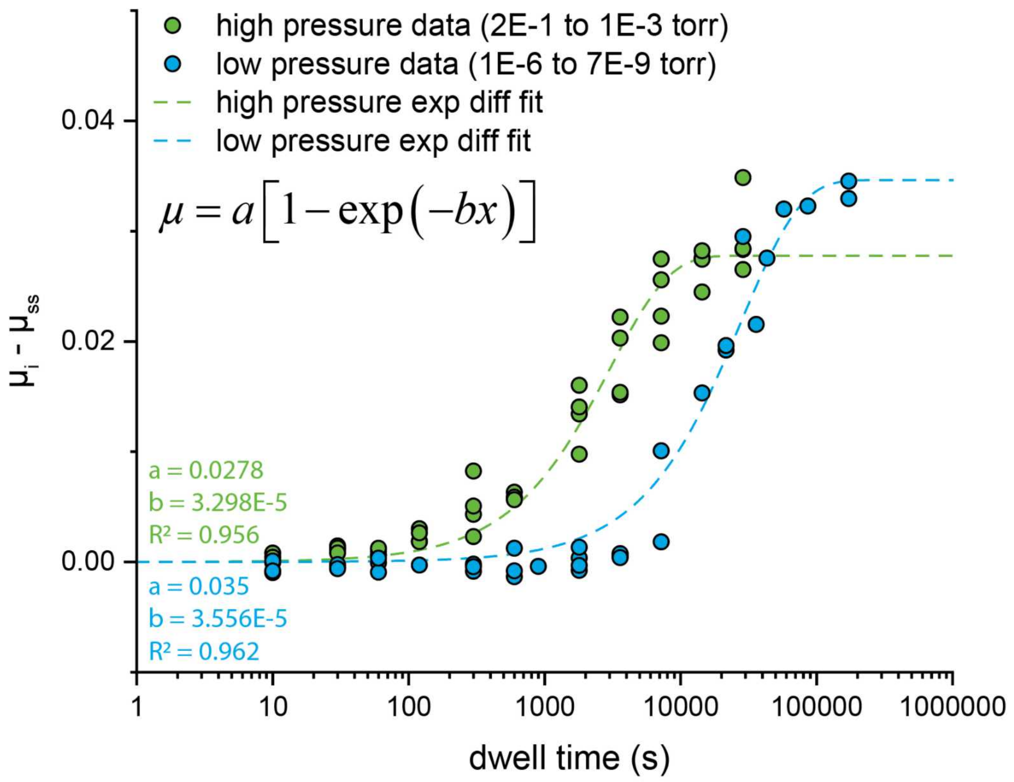
- Certain assumptions may not apply
  - Absence of corrugation
  - All sites being equivalent (basal vs edge)
  - Adsorbate interactions (water vs hydrocarbons)



Exponential Growth – Diffusion Approximation

- As Matsunaga suggested – behavior might be best described by diffusion processes

- Borrowed models from surface concentration via grain boundary diffusion…



G. E. P. Box and H. L. Lucas (1959) "Design of Experiments in Non-Linear Situations" *Biometrika* Vol. 46 No. 1 pp. 77-80.

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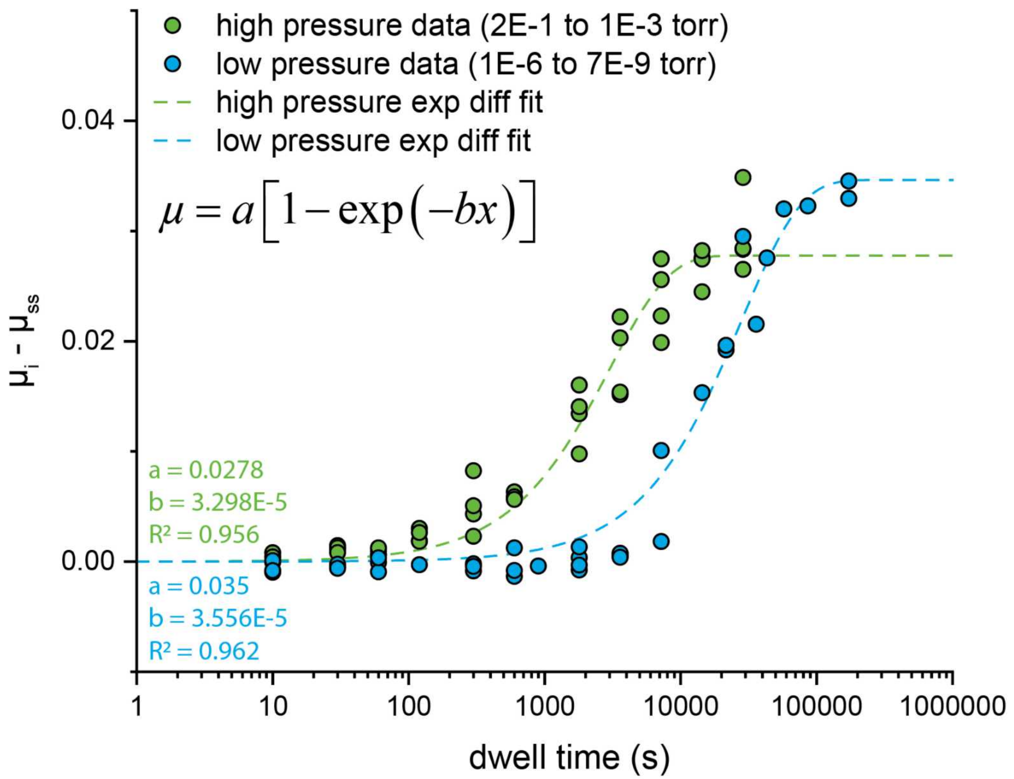

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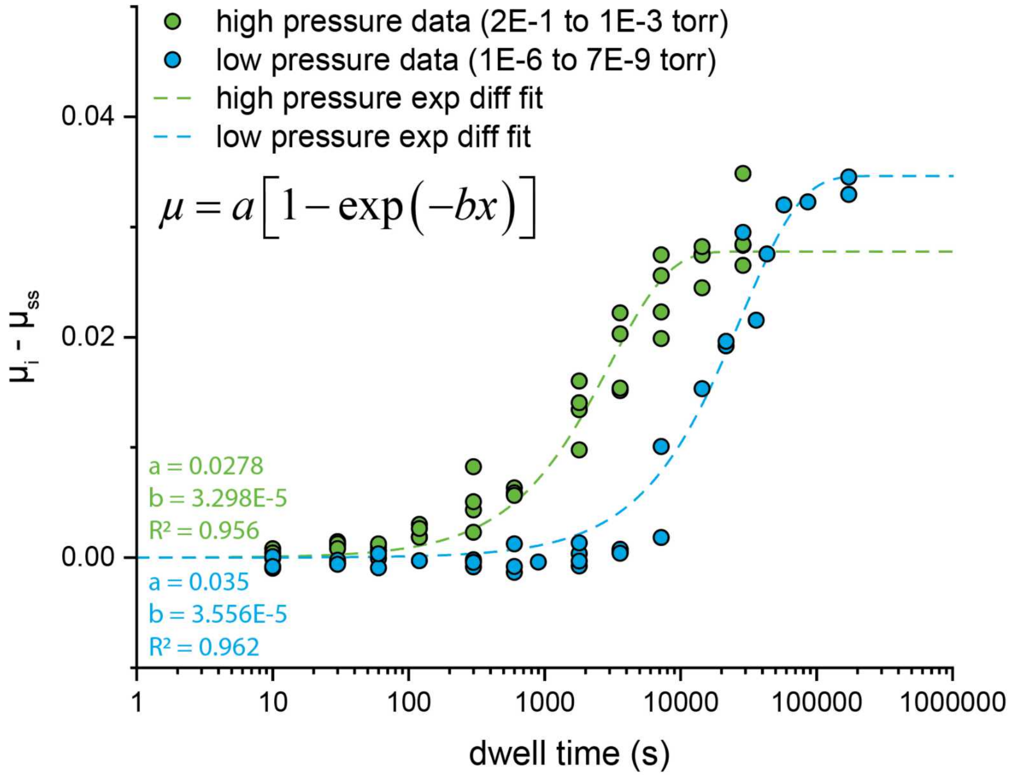
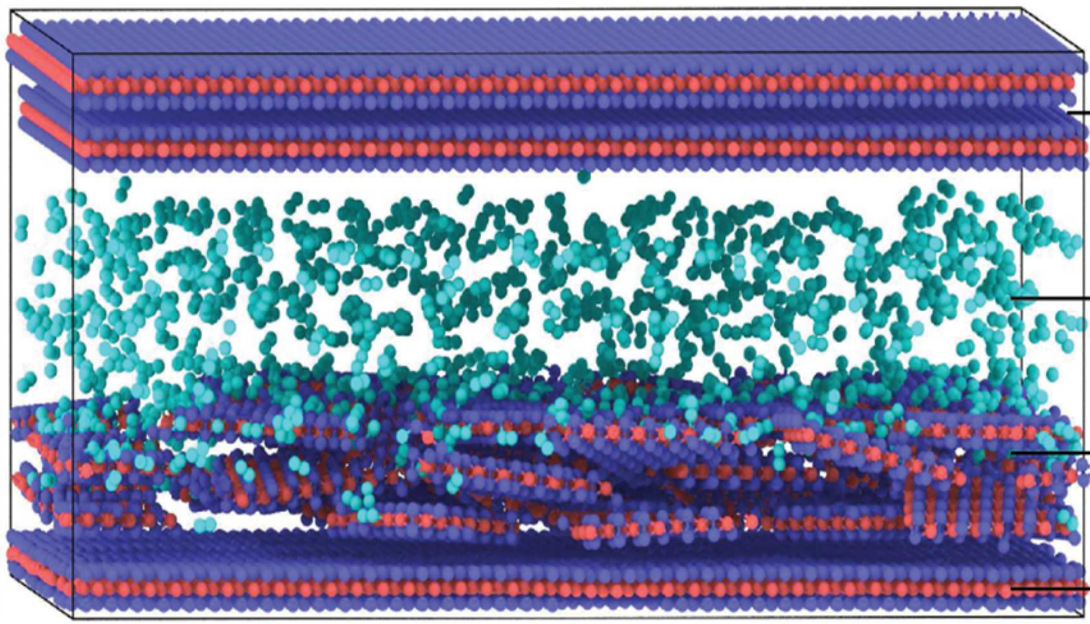


“All models all wrong – but some are useful” – G. Box

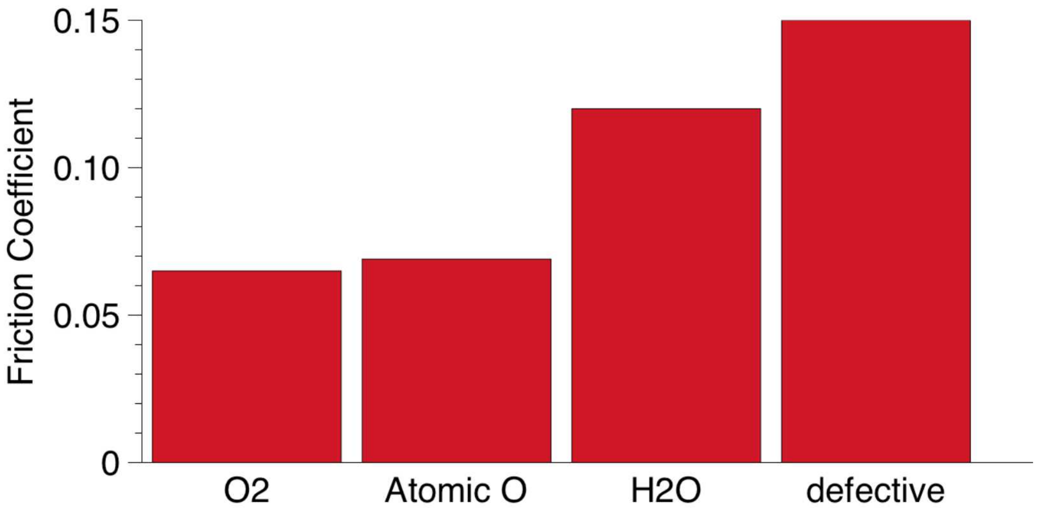
G. E. P. Box and H. L. Lucas (1959) “Design of Experiments in Non-Linear Situations” *Biometrika* Vol. 46 No. 1 pp. 77-80.

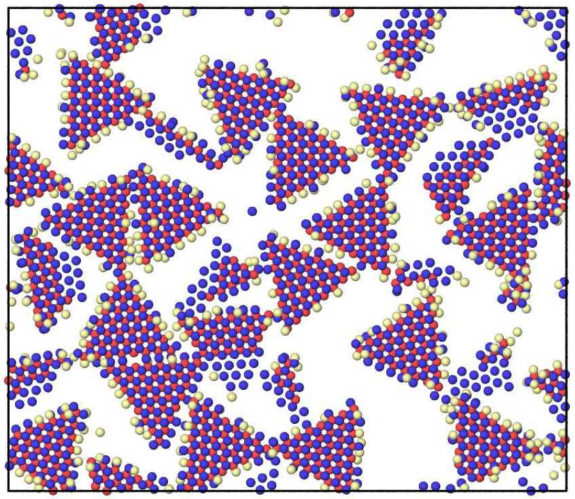
Molecular Dynamics Approach



- Take systems that have "run-in" (i.e. reached steady-state shearing)
- Remove top layers
- Apply O2, AO or H_2O at 100 atm
- Replace top layers

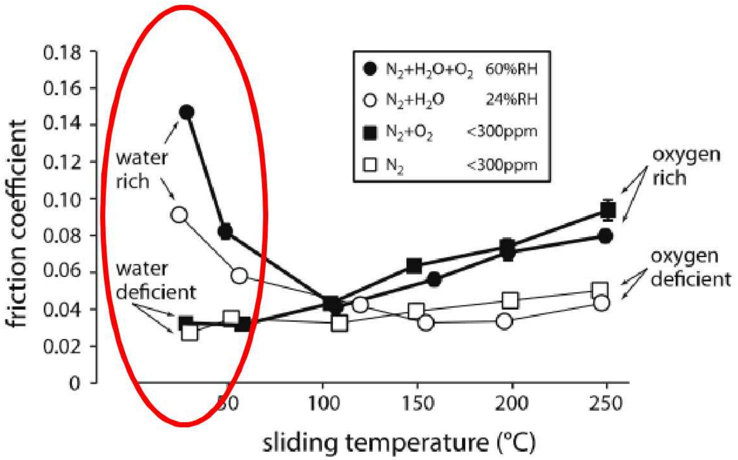
How do oxygen and water interact?



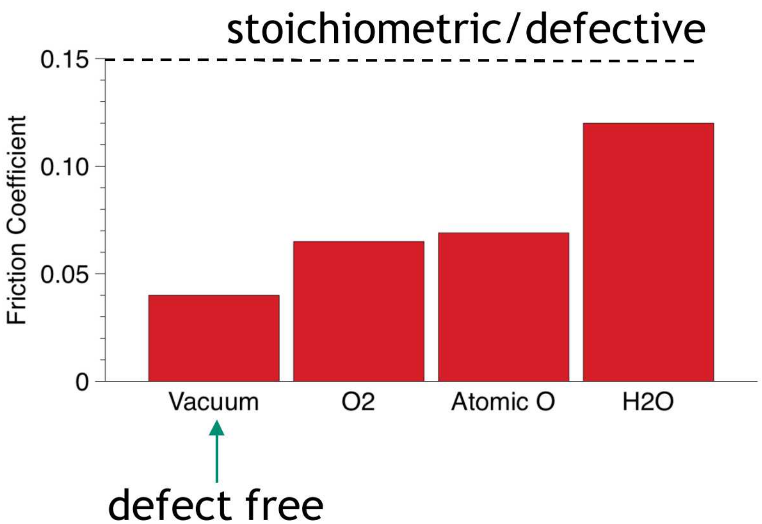


- Friction goes down?
- This is unfair…
  - Water and oxygen passivate defect sites
  - Need to do this in the pure system, too
  - Look at non-stoichiometric (i.e. defect-free) nanoplatelets

Friction in Environments

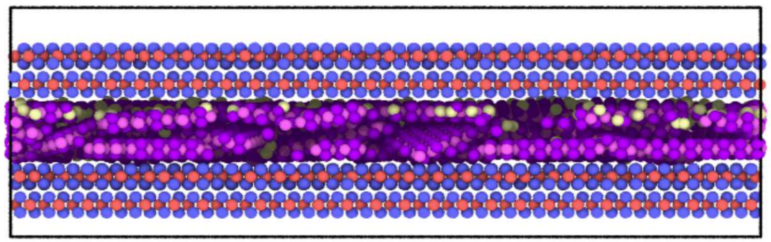


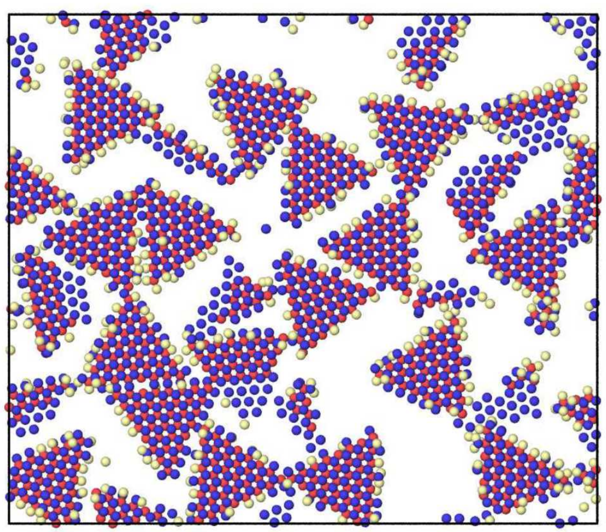
Khare and Burris, Tribo. Lett. 2013



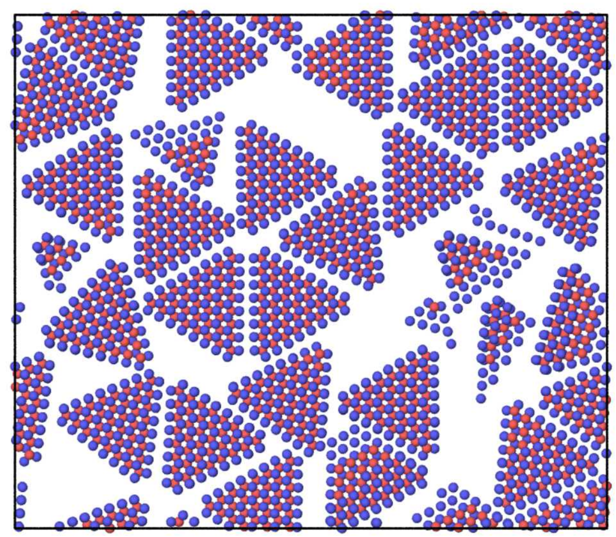
•Changes with added oxygen or water match experimental results

Effects of Oxygen on Inter-platelet bonding





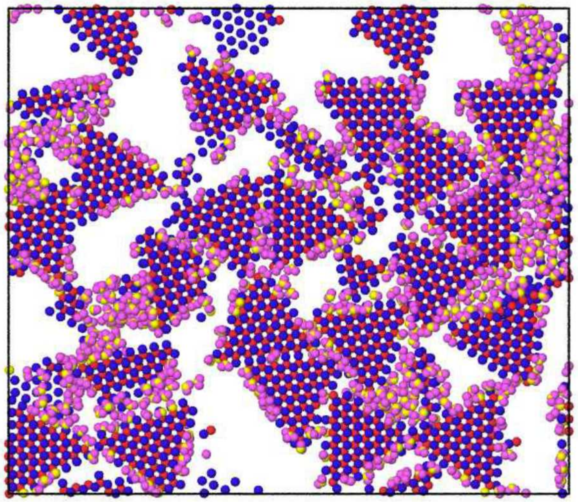
oxygen passivated



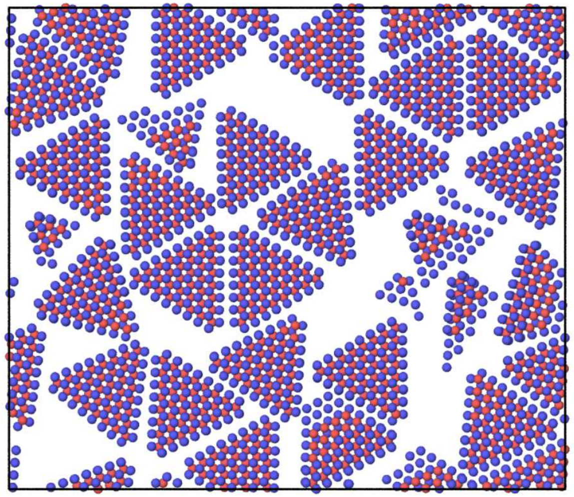
defect free

- Oxygen bonds to defect sites & prevents formation of larger sheets
- Molecular oxygen looks very similar

Effects of water on Inter-platelet bonding



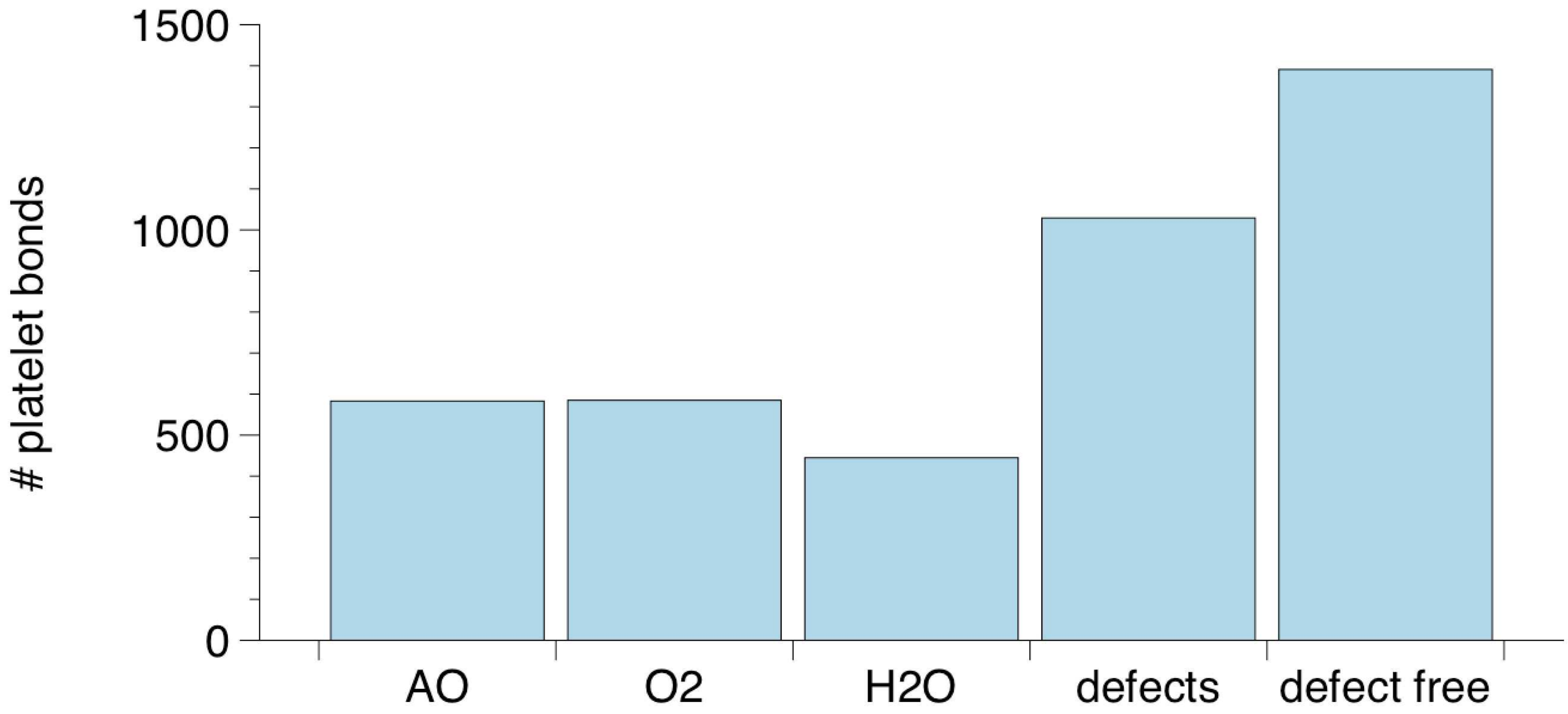
water passivated



defect free

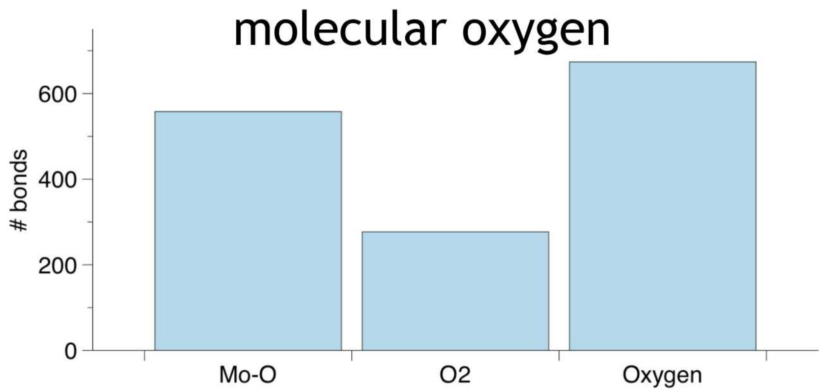
- Water also bonds to defect sites & prevents formation of larger sheets
- Water aggregates with itself more than oxygen does

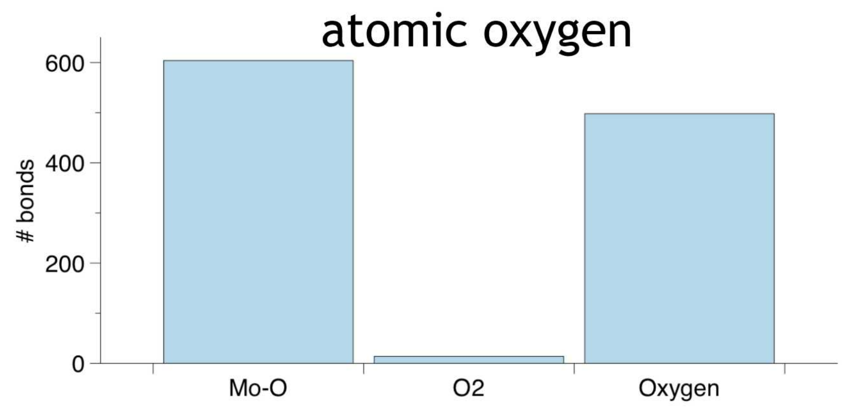
Counts of inter-platelet bonds confirm

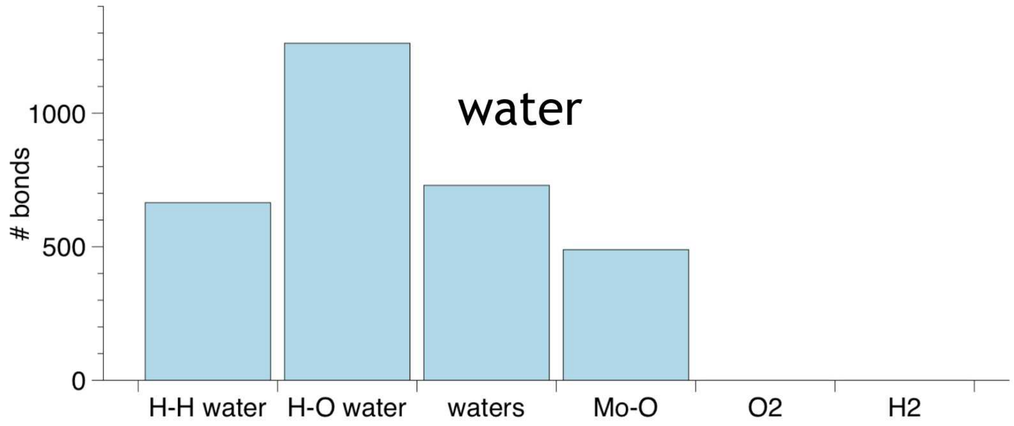


Environmental species interrupt formation of larger flakes

What can we say about chemistry?

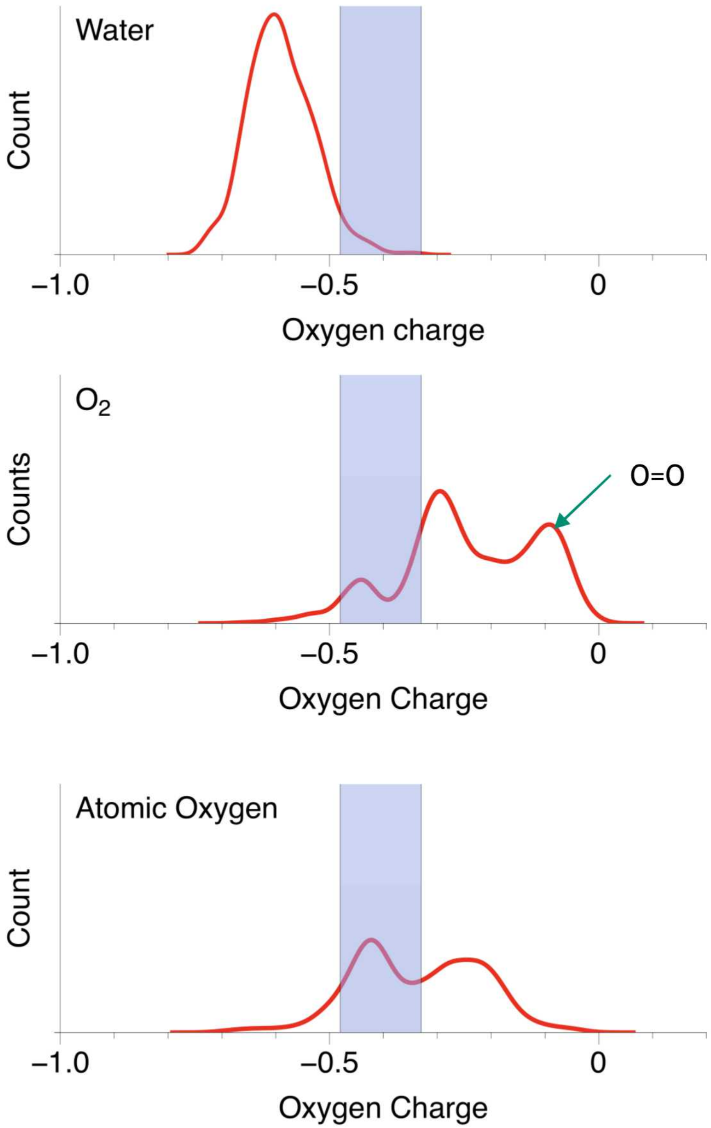


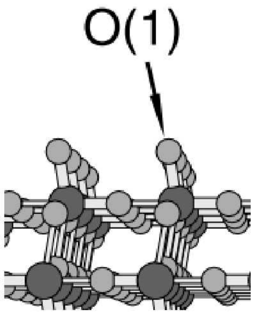




- Water does not dissociate (no O2 or H2 formed)
- Molecular O shows little dissociation (mostly in O2)
- Atomic oxygen forms little O2

Charge on Oxygens confirms chemistry





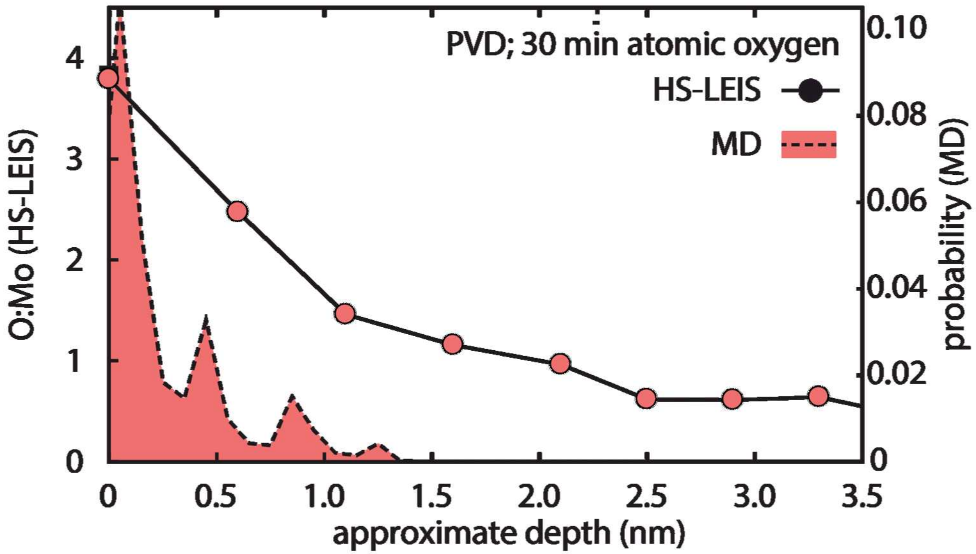
Tokarz-Sobieraj et al.
Surf. Sci. 2001

- Oxygen bonded to Mo has partial charge from -0.48 (Tokarz-Sobieraj et al. Surf. Sci. 2001) to -0.33 (Yin et al., J. Mol. Model 2001).
- Oxygen in water has partial charge from -0.6 to -0.8(Astrand, et al., J. Phys. Chem. A 1998).
- Water shows only physisorption
- Atomic oxygen shows chemisorption
- Molecular oxygen shows slight amount of chemisorption

Summary

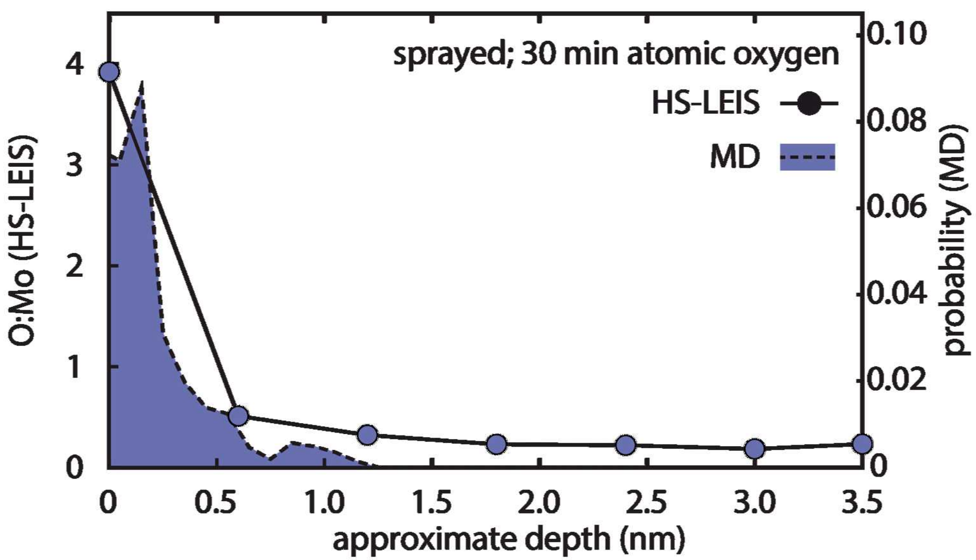
- MoS_2 shows purely elastic contact
- Shear is predominantly due to inter-lamellar interactions
- Simple model predicts temperature dependence
- Environment hinders formation of large sheets
- No chemistry with water
- Little chemistry with molecular oxygen
- Lots of chemistry with atomic oxygen

LEIS & MD Depth Profiles



sputtered (nanocrystalline/amorphous)

- oxygen at surface of coating
- oxygen slowly decays after several lamellae

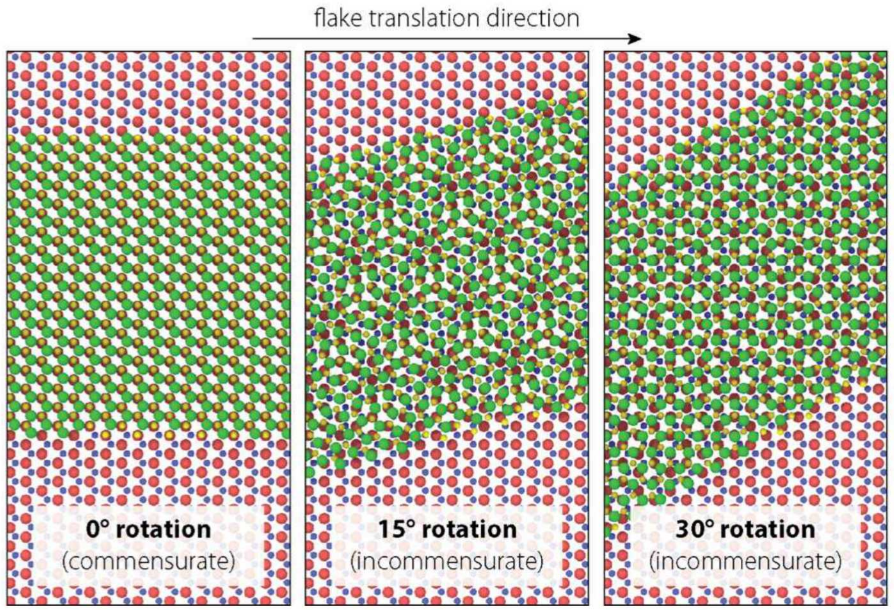


sprayed (highly ordered film)

- oxygen at surface of coating
- decays quickly after ~ lamellae

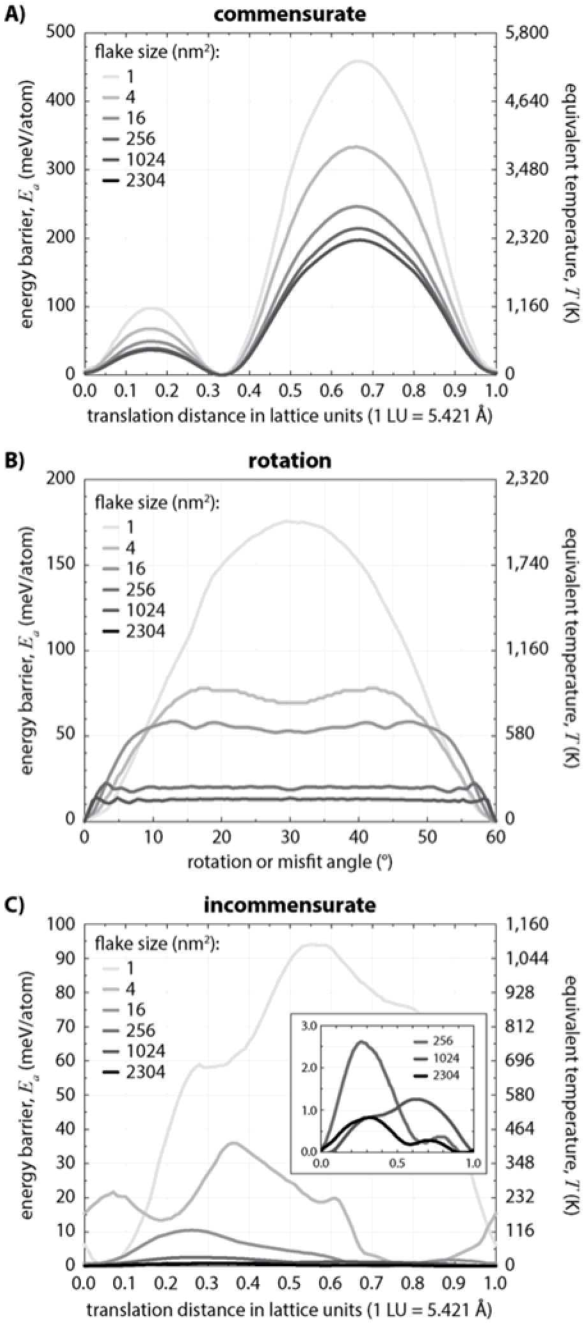
MD accurately represents oxygen depth profiles as seen in LEIS experiments

Elastic contact => Energy Barriers: Our work



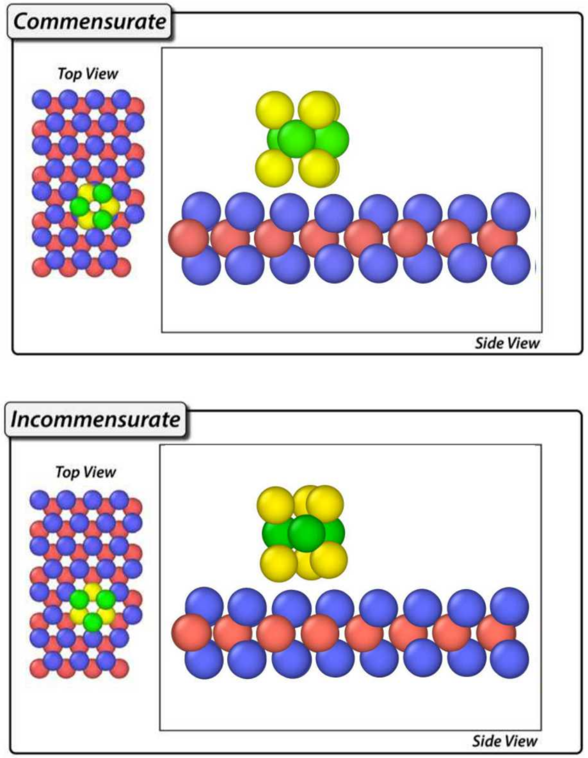
commensurate
egg shell

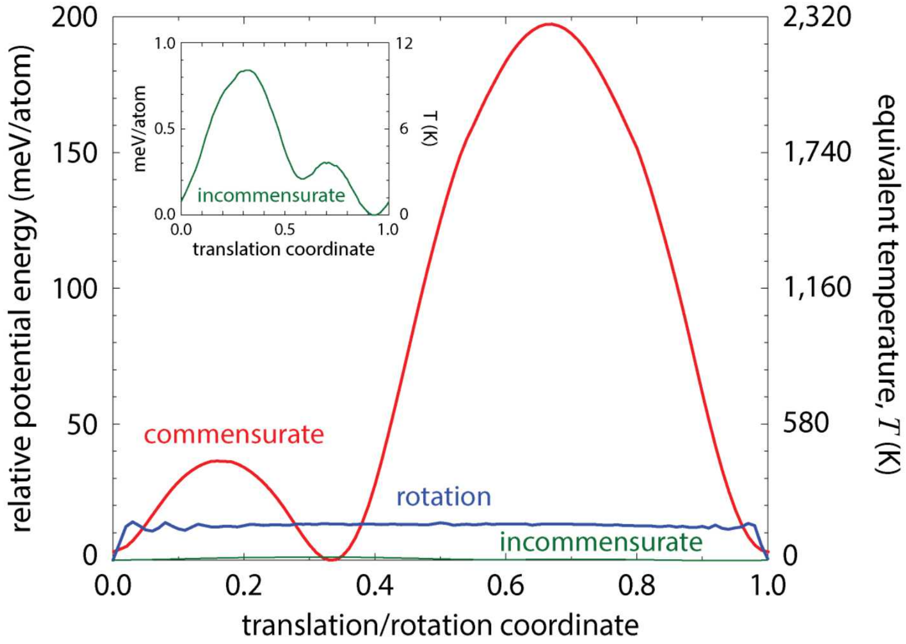
incommensurate
egg shell



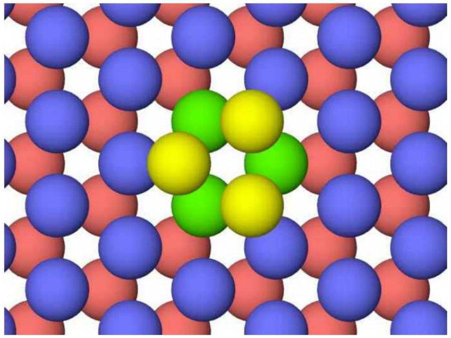
Nudged elastic band calculations for barriers

Commensurate vs. Incommensurate Sliding



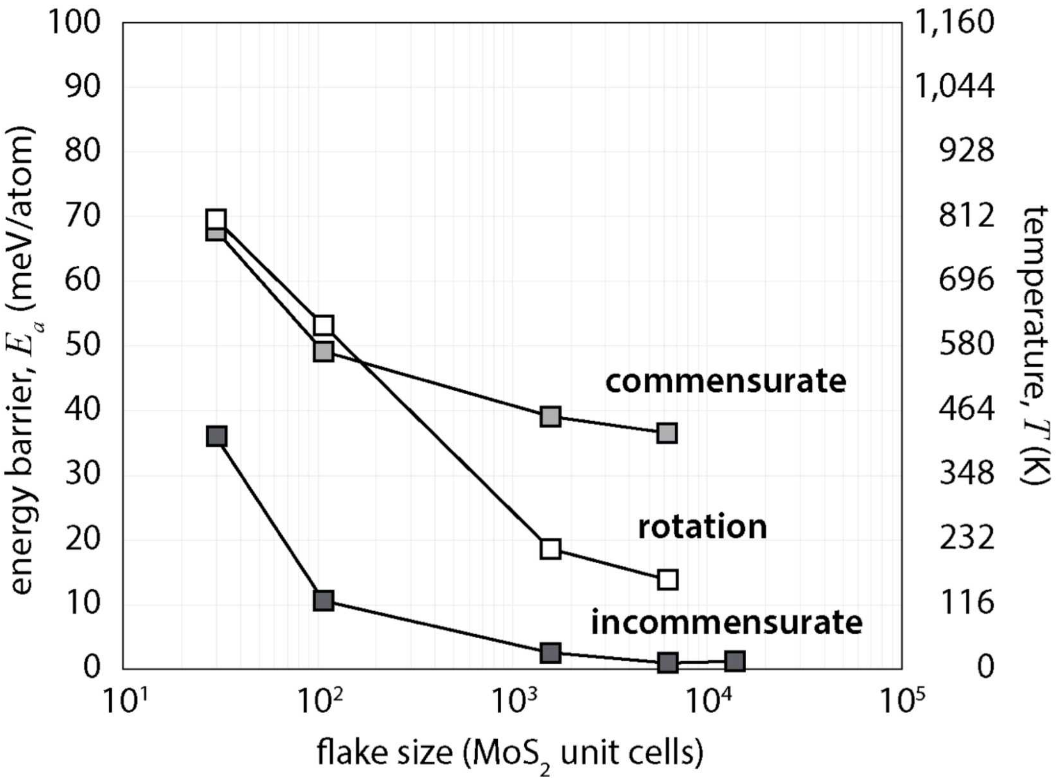


- Commensurate barrier ~= 300 K
- Incommensurate barrier ~= 10K
- Rotation barrier ~= 150K



rotation

Barriers converge with increasing flake size; make a toy model



Probability & Failure to cross barrier:

$$p_n \propto \exp\left(\frac{-E_n}{kT}\right)$$

$$f_n = 1 - p_n$$

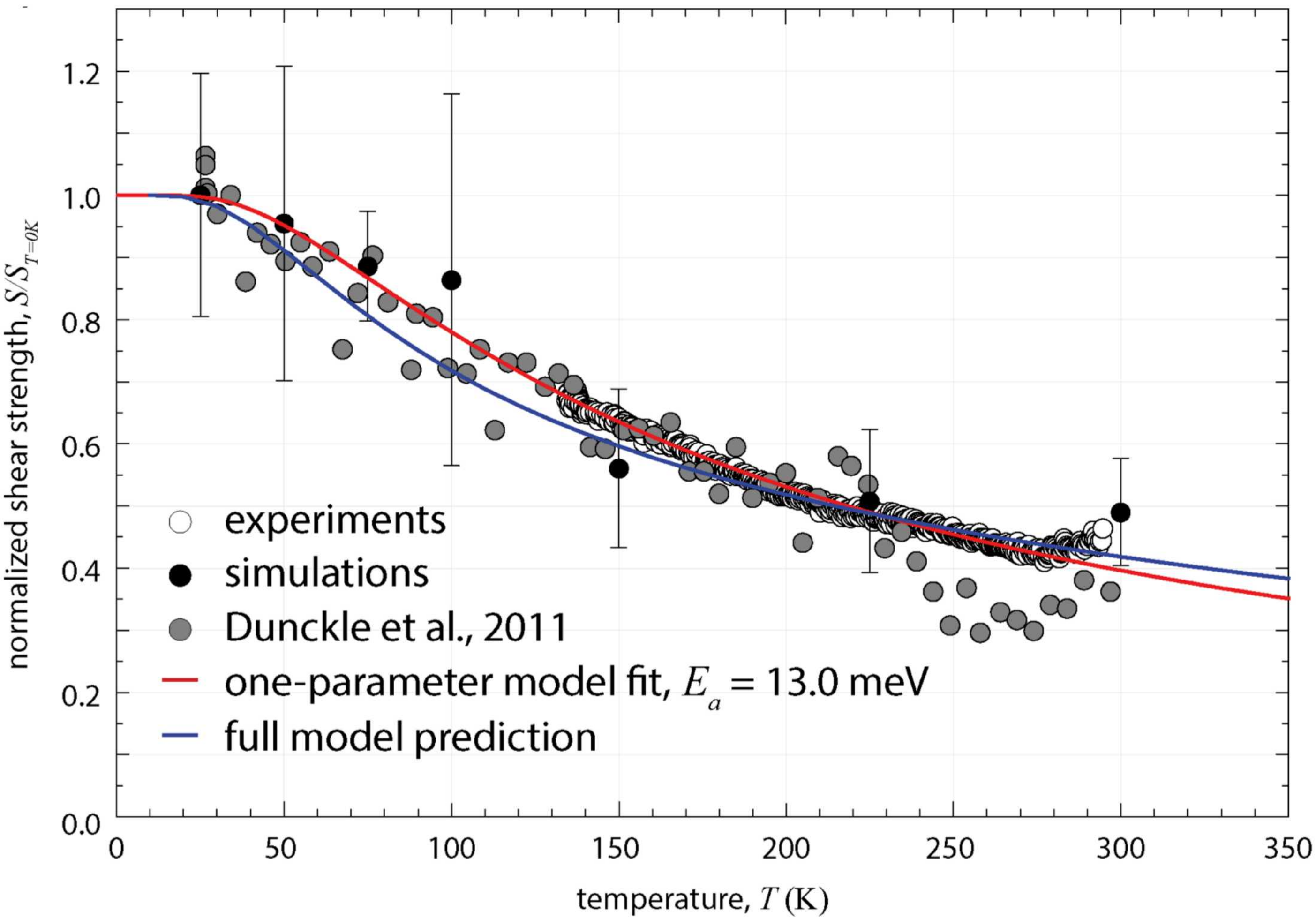
Total sliding probability & friction:

$$p_{slide} = p_r p_i + f_r p_c$$

$$f_{slide} = 1 - p_{slide} = 1 - \left(p_r p_i + f_r p_c\right)$$

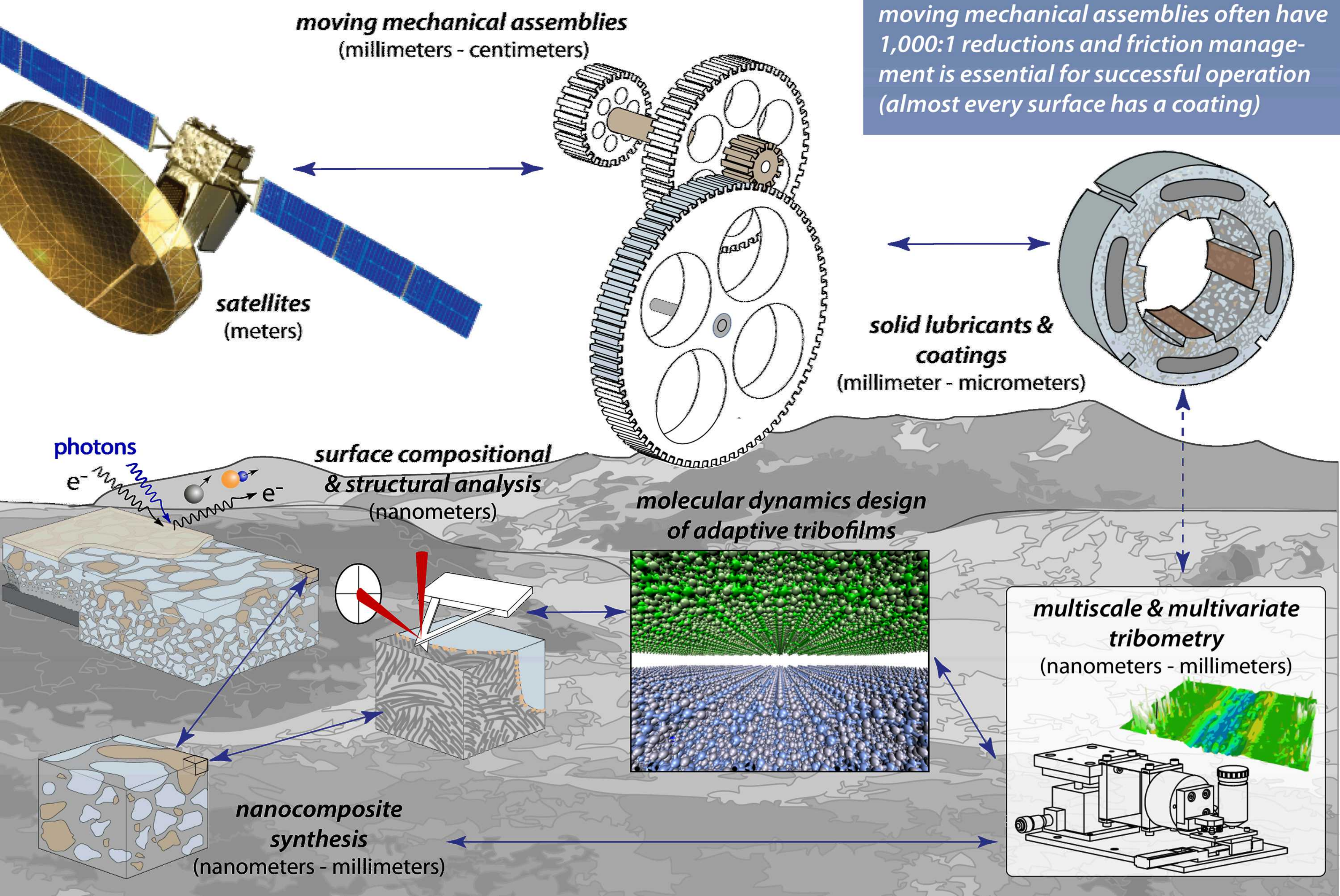
Results of toy model

$$f_{slide} = C_0\left[1 - \exp\left(-\frac{E_i + E_r}{kT}\right)\right]$$



Fundamental Studies & Applied Challenges

moving mechanical assemblies often have 1,000:1 reductions and friction management is essential for successful operation (almost every surface has a coating)



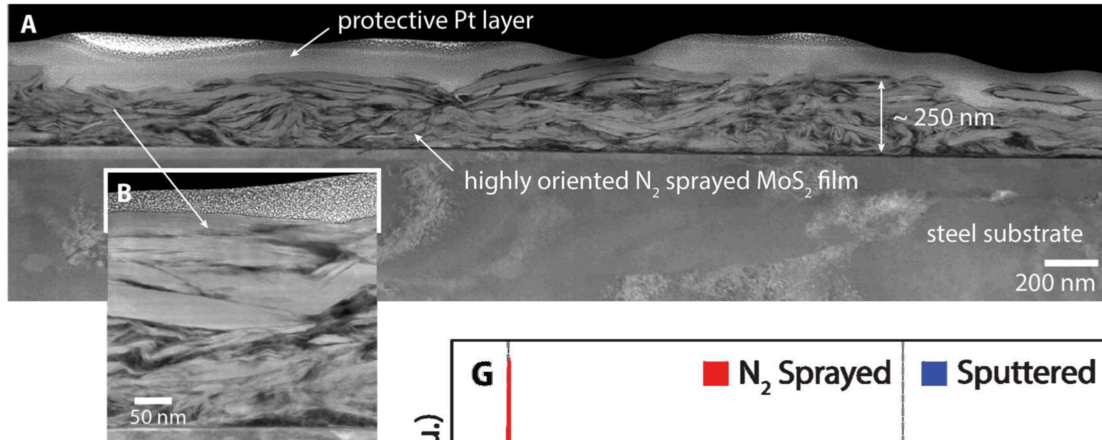
Effects of Microstructure

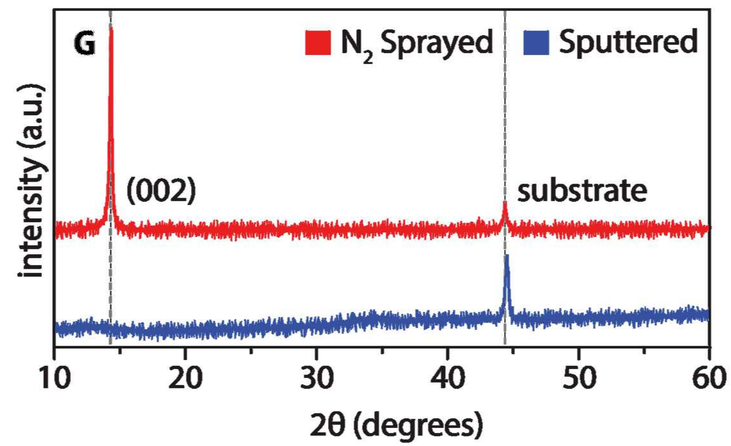
Nitrogen Spray Deposited MoS_2

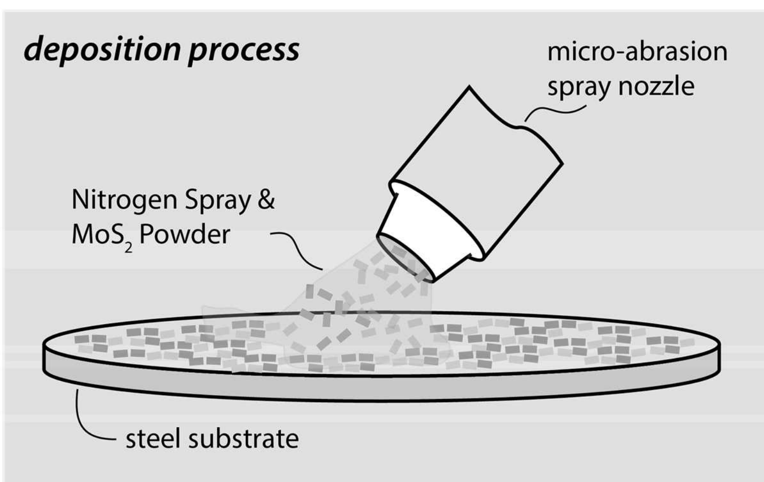
- Deliver MoS_2 powder to surface in dry N_2 gas

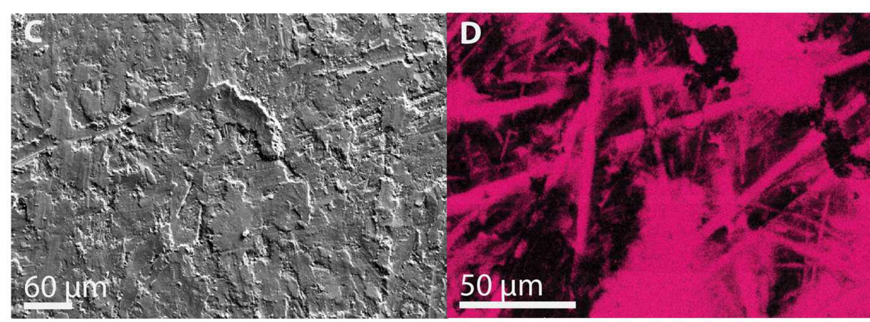
- High kinetic energy imparted shears MoS_2 onto surface to produce a higher orientation of basal planes.

- Similar to burnishing, large continuous crystallites will form, reducing presence of surface defects

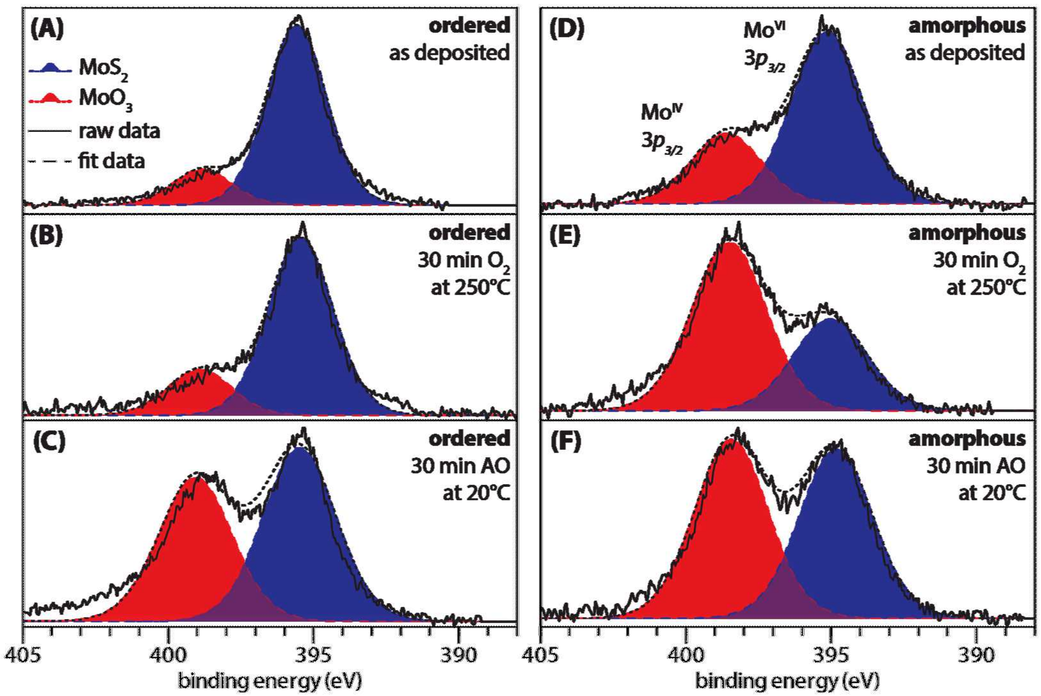


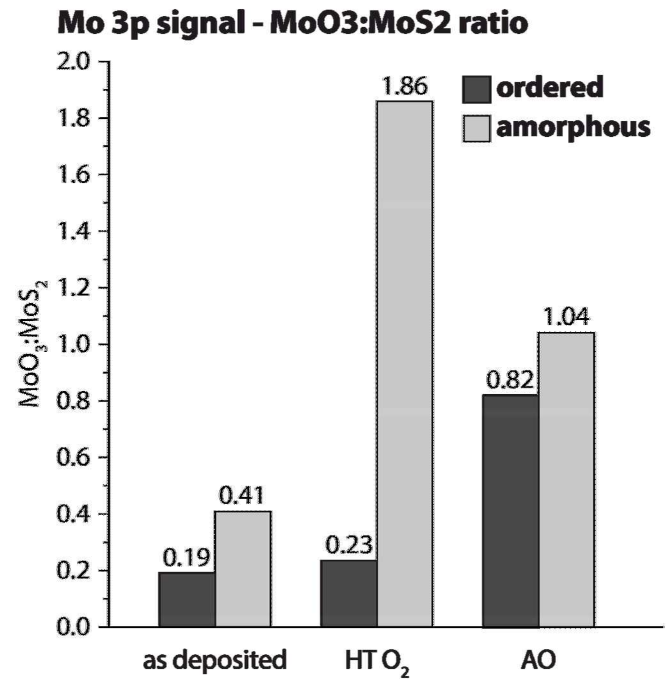






XPS Mo 3p Spectra for Aged MoS_2





Analysis

(1) Higher MoO_3 concentration in as dep. PVD samples with higher ratio MoO_3:MoS_2 **(0.41)** than sprayed **(0.19)**

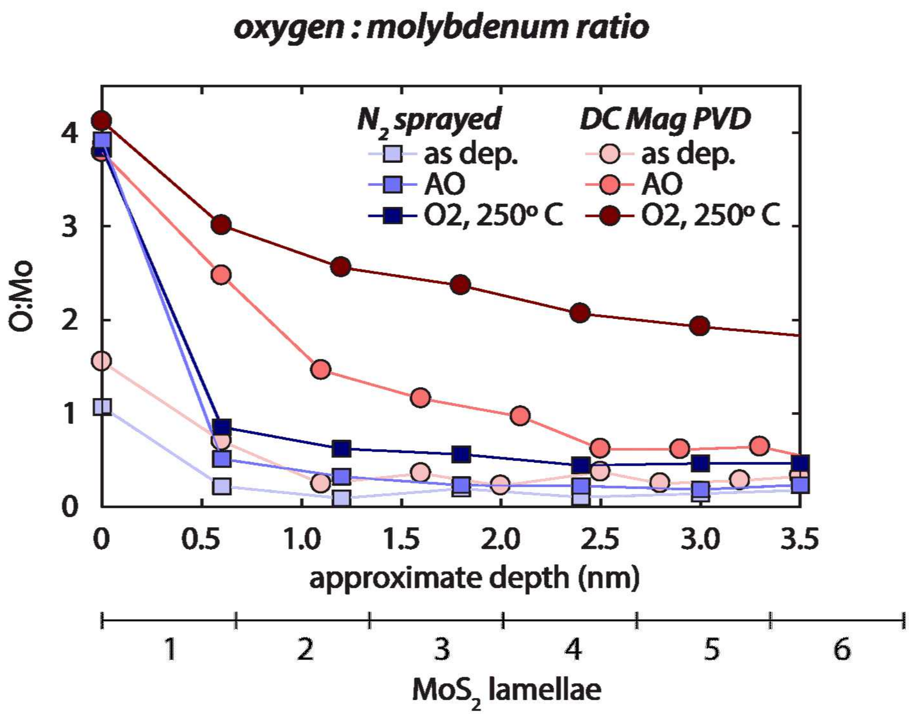
(2) Minor increase of MoO_3:MoS_2 for ordered coatings after HT O2 **(0.23)**

(3) Significant increase in MoO_3:MoS_2 for amorphous films **(1.86)** - likely due to greater oxygen diffusion

(4) High MoO3 concentration for AO in ordered **(0.82)** and amorphous coatings **(1.04)**, yet limited to first layer for sprayed as shown in HS-LEIS

(5) XPS results agree well and complement work done in HS-LEIS

HS-LEIS: Atomic Ratios vs Depth



Analysis

(1) Oxygen content of the surface is the same for both coating types regardless of exposure

(2) Oxygen persists through the depth of sputtered coatings exposed to either O_2 or AO

(3) Oxygen is limited to the first atomic layer for sprayed coatings to either O_2 or AO

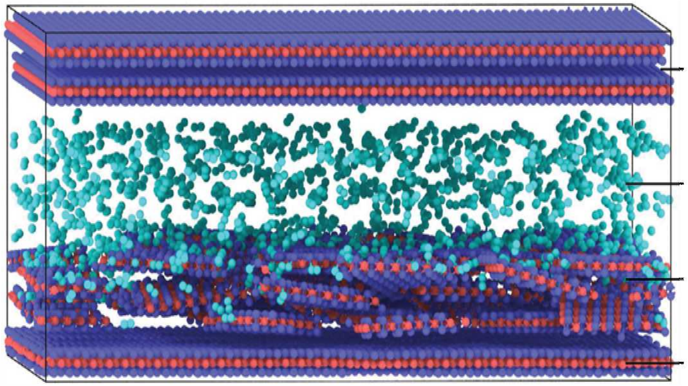
Role of Water in Modifying Friction - Chemistry

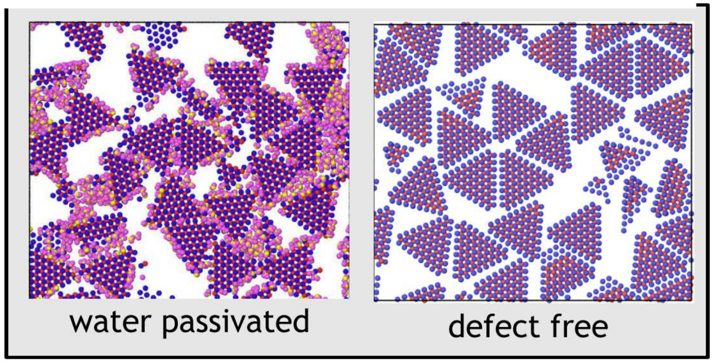
- How does water actually modify friction?

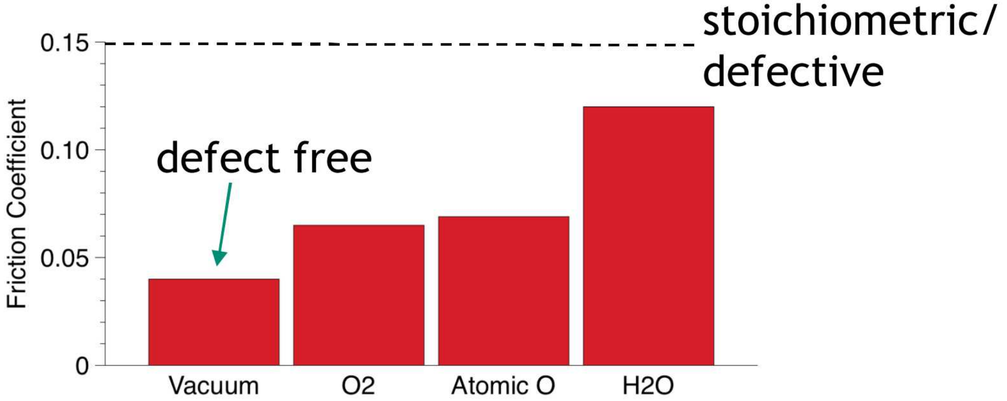
- MD Simulations show water bonds to defect sites and aggregates, preventing formation of larger sheets

- More edge sites likely enhance adsorption as well, with more energetic and a higher number and potential sites

- Water can also increase drag between lamellae via polar bonding with water molecules [1]







[1] Holinksi & Gansheimer, ASLE Transactions 1971

Role of Water in Modifying Friction – Flake Kinetics

- Increased friction may be explained by flake rotation mechanisms

- Relies upon model recently developed establishing a link between the probability of flake rotation as a function of temperature

- In this case, adsorption sites effectively pinning flake rotation, which then behaves in the same fashion as we described earlier…