SAND2021-15518PE

Sandia National Laboratories

Structural, Environmental and Mechanochemical Interactions in Solid Lubricant Material Systems

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Sandia National Laboratories, Albuquerque, NM

JAST Webinar

8 December 2021

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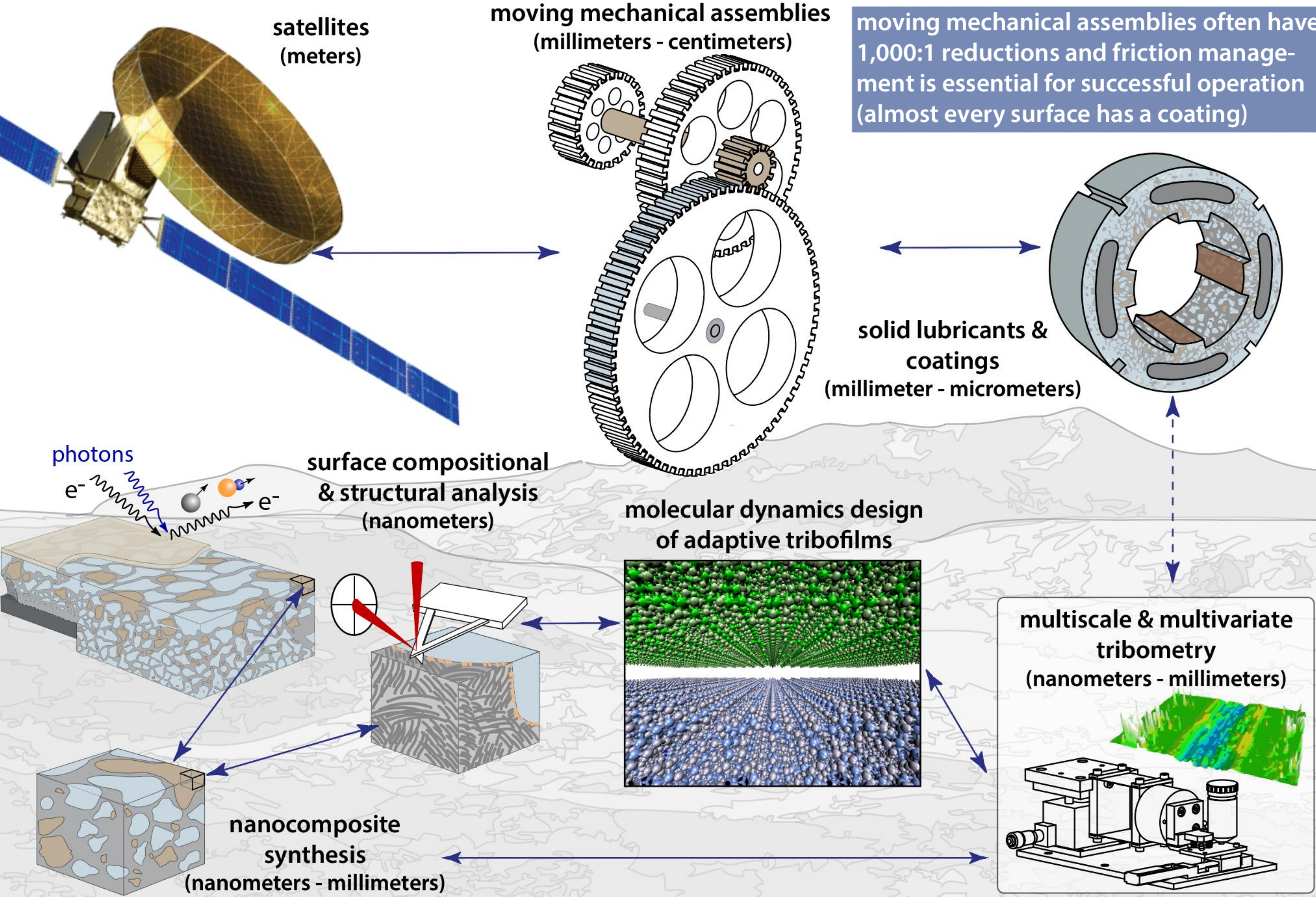
Talk Outline

1. **Brief Introduction & Motivation**

2. **Current & Ongoing Research Projects**
   - Environmental Factors Governing Run-In Behaviors and Oxidation response in MoS2
     - Aging of Pure/Composite films with Varying Structures
     - ALD MoS2 Conversion
     - Fundamental Role of Structure on Friction Response
   - Understanding Variability in Run-in for DLC Coatings
     - High Throughput Assessment of Different Film Compositions
     - Deposition Directed Surface Termination Studies
   - Self-lubricating in situ carbon films (SLIC)

Fundamental Studies & Applied Challenges



Extreme Operating Environments

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

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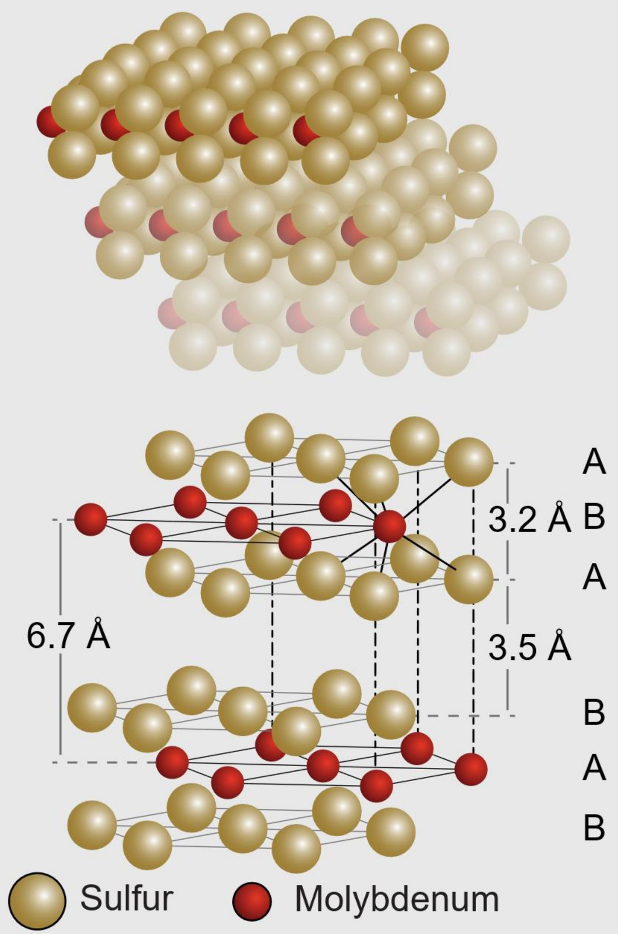
Precision Mechanisms:

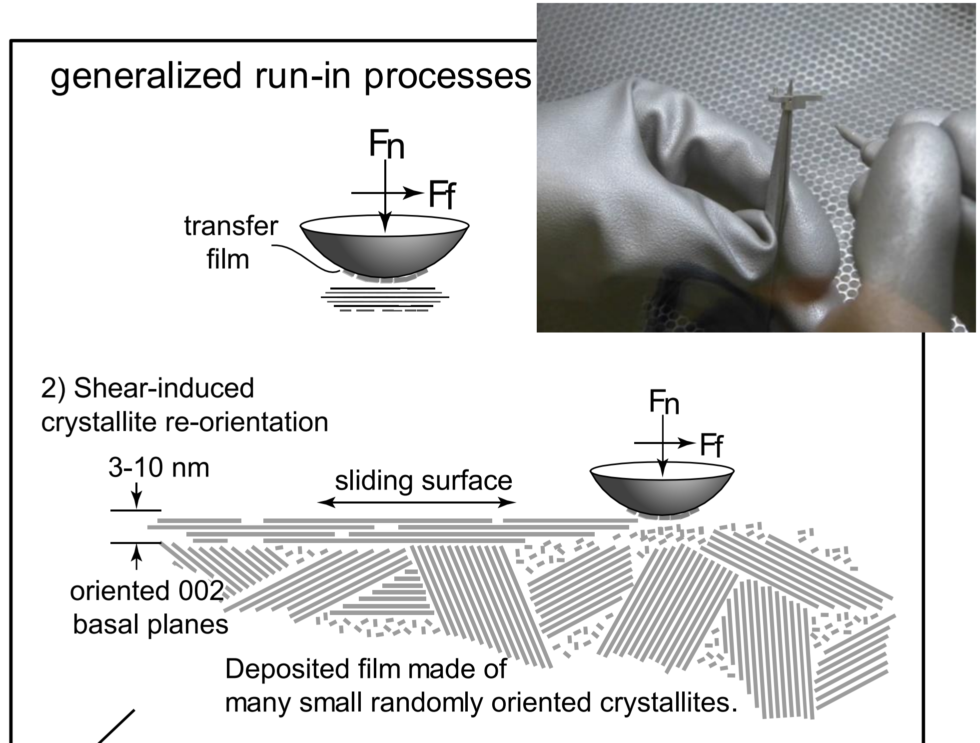
- inert gas near P_{atm}, trace O_2, H_2O, outgassing species
- store for decades; non-serviceable
- operating temperatures 200 – 350K
- large investments of time and money
- consequences (political, societal) of failure are unacceptable

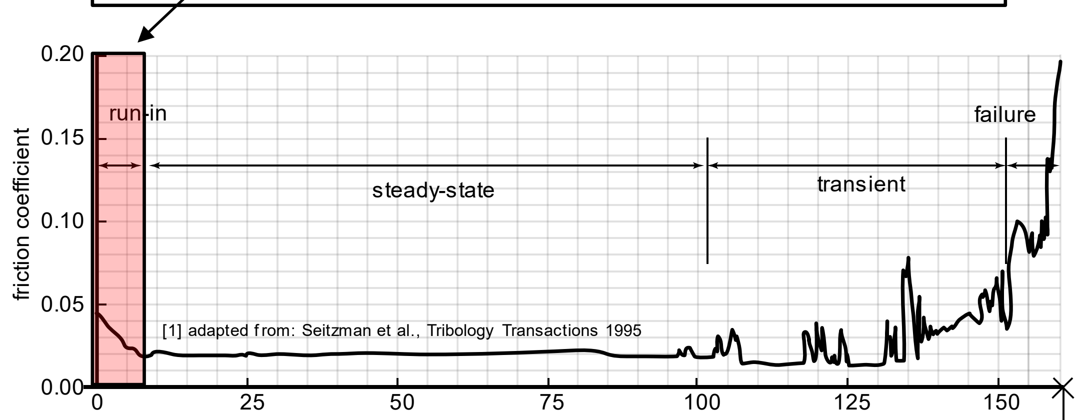
Environmental Factors Governing Run-In Behaviors and Oxidation response in MoS_2

MoS_2 – How it Works

molybdenum disulphide
μ = 0.02 - 0.06 (inert @ 1N)
μ = 0.15 - 0.25 (humid air @ 1N)

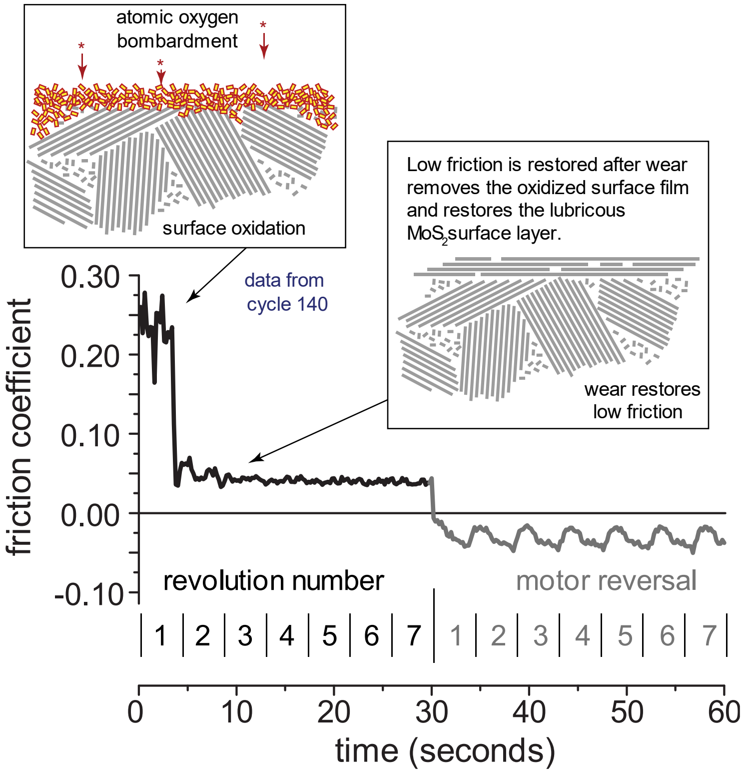






Typical friction trace for MoS2 lubricated contact. Initial run-in is followed by steady state low friction which ultimately transitions to high friction before failure [1]

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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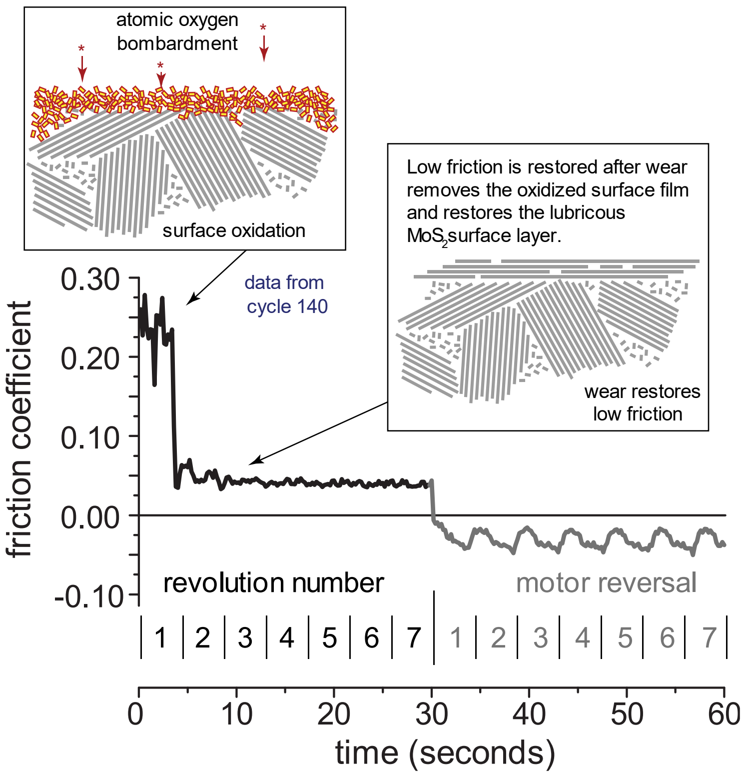

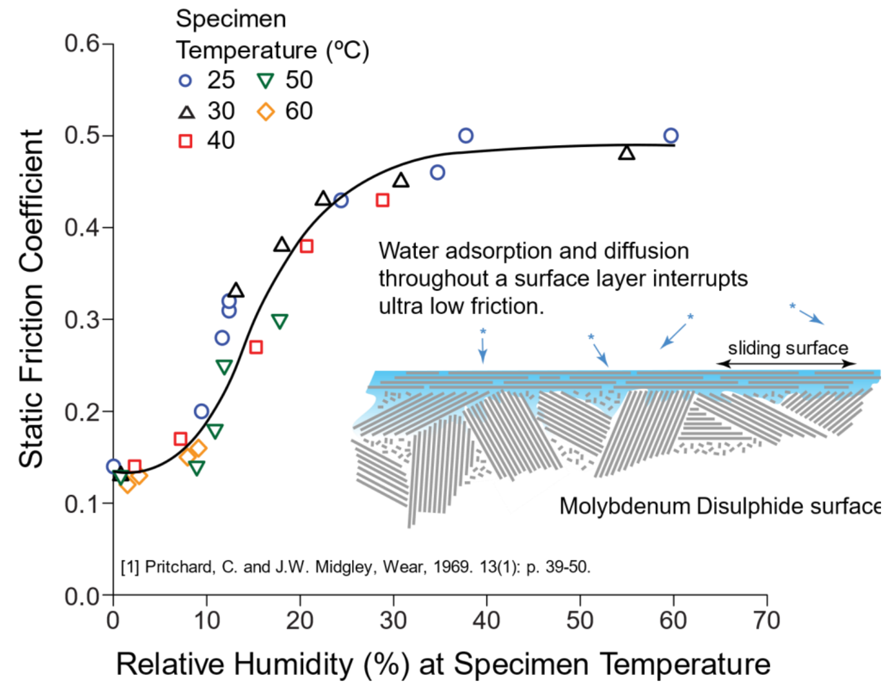

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- Water enhances static and kinetic friction behaviors via increased shear between layers

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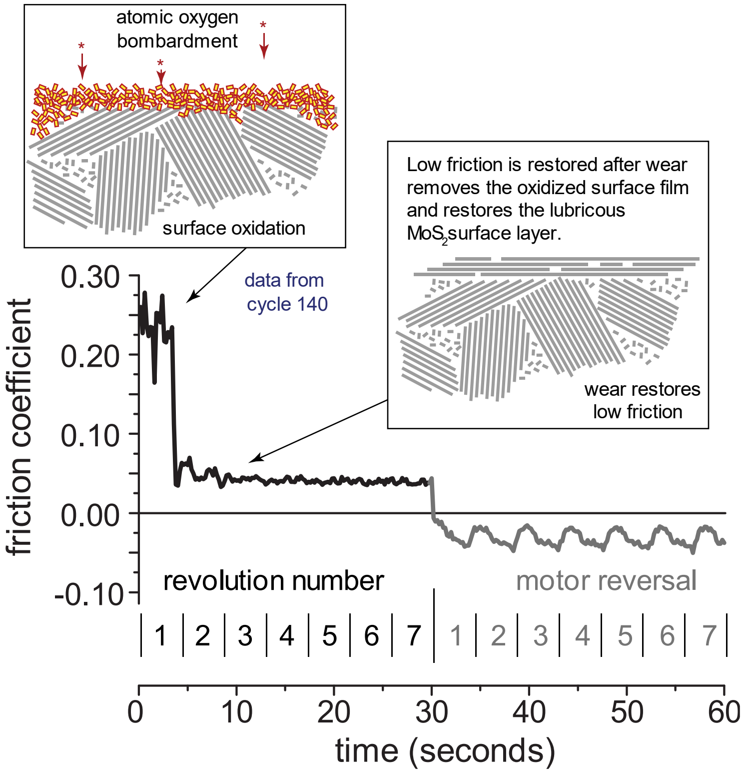

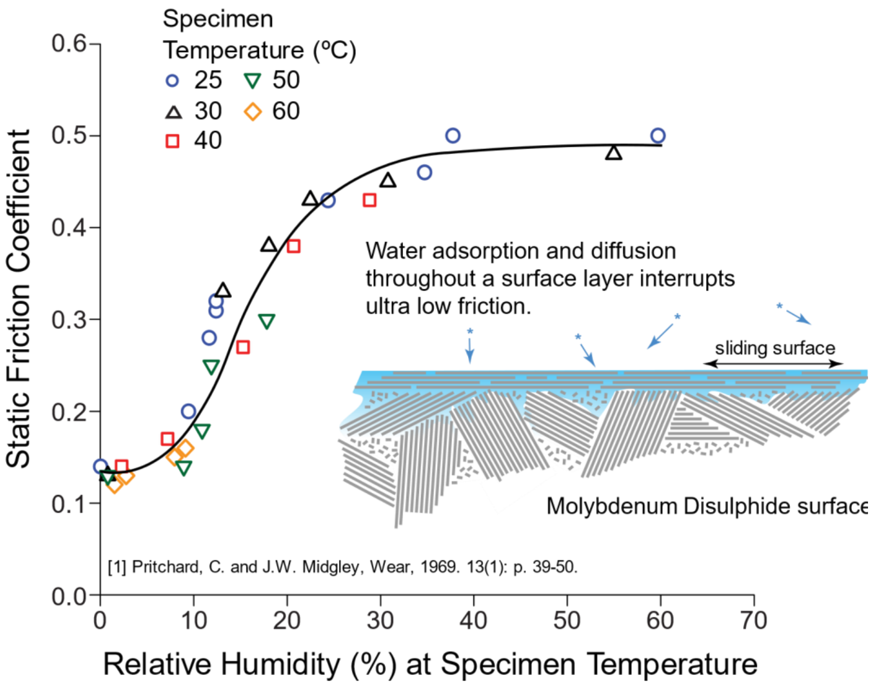

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Many components operate infrequently and for very few cycles – ***effectively living in the run-in regime***

Initial Friction Evolution (Run-In)

Drawbacks

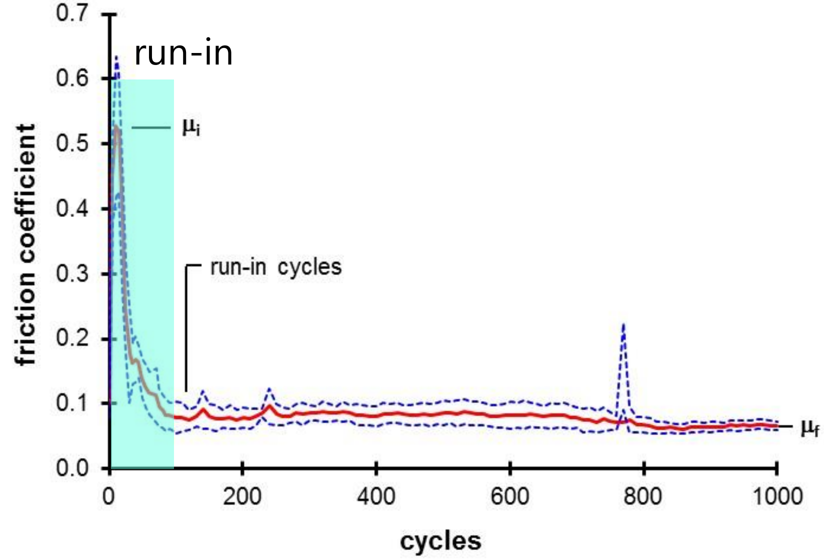
- Increased friction/wear
- Risk of seizure
- Reduced performance margin in mechanisms
- Loss of energy

Causes

- Structural (re-orientation, crystallization)
- Environmental (oxidation, adsorbates)

Solutions

- Tailor composition (compositing for densification, water-getting)
- Tailor microstructure, encourage ordering (impingement films, capping layers, deposition inputs)



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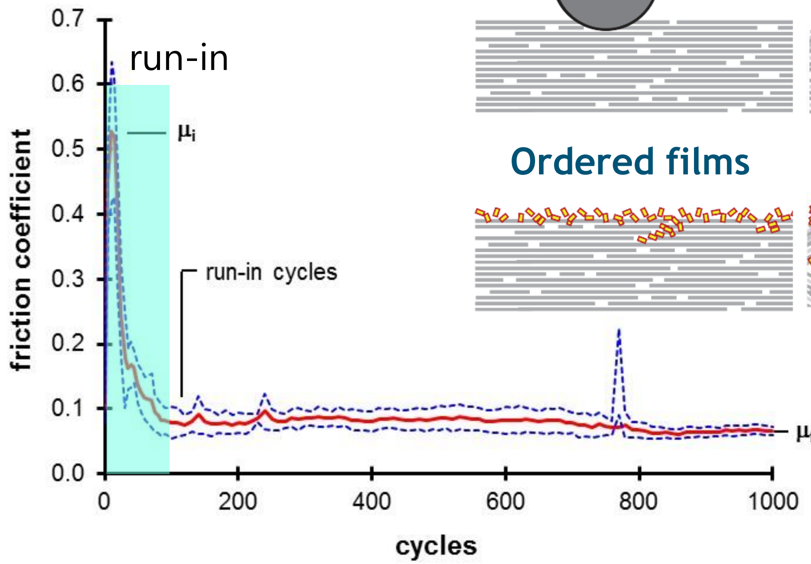

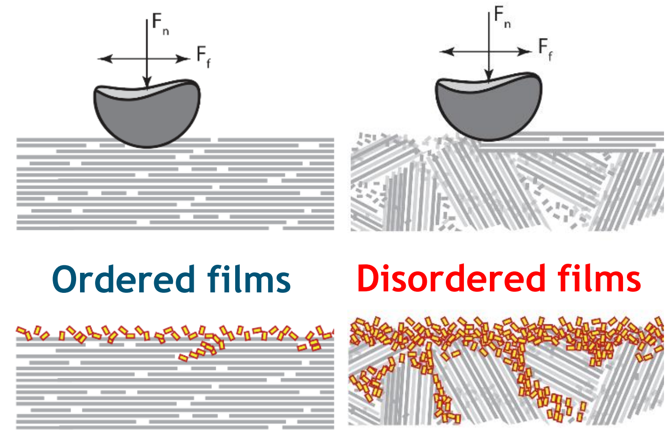

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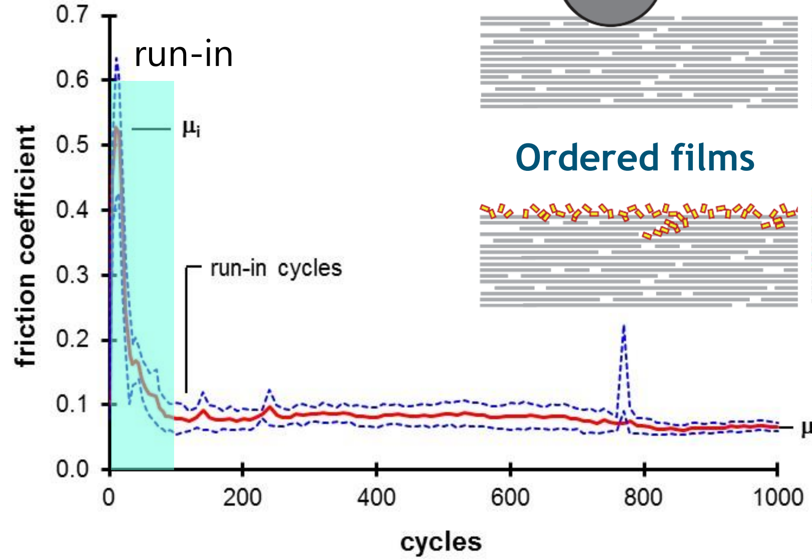
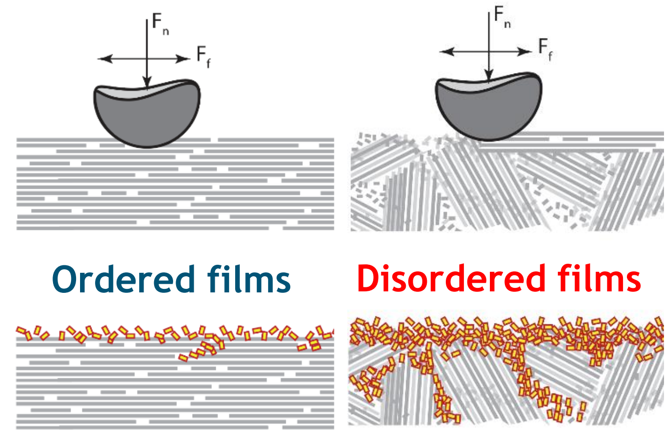
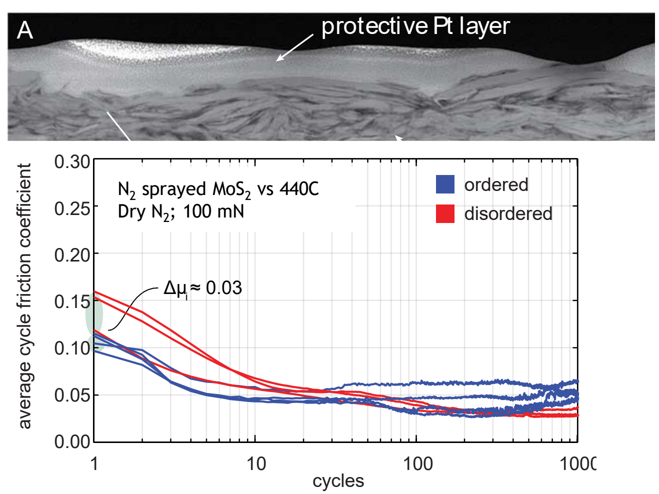
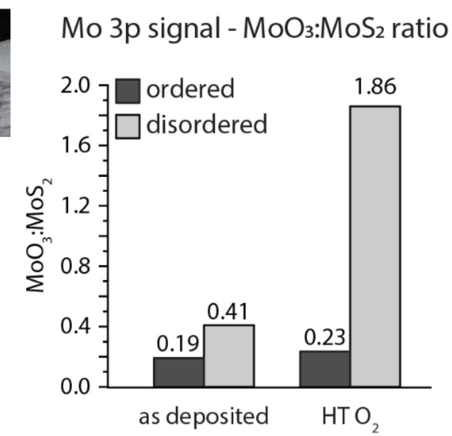






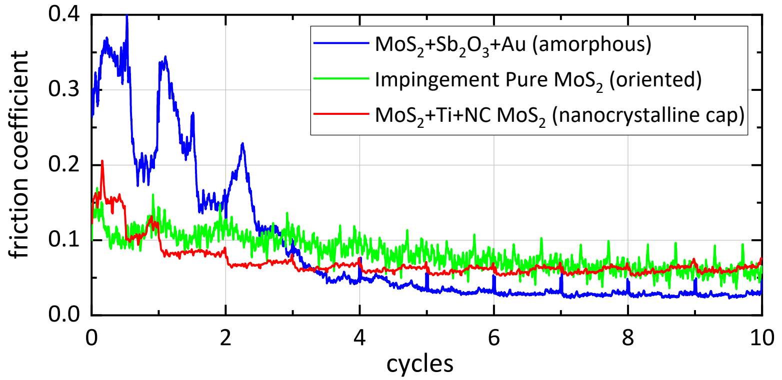


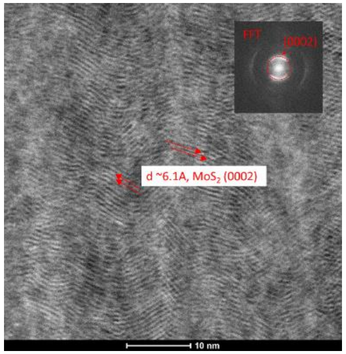
Curry, J. F. *et al. ACS Appl. Mater. Interfaces* (2017)

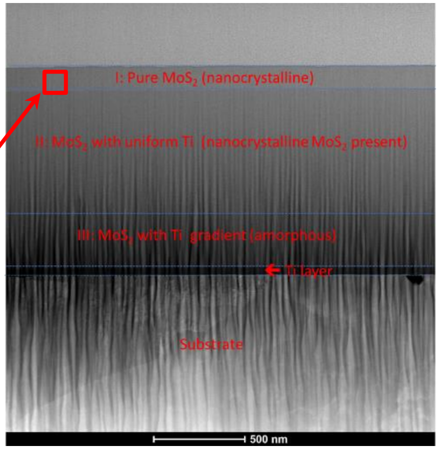
Efforts to Reduce Run-In: Structure/Composition

PVD nanocrystalline & composite films

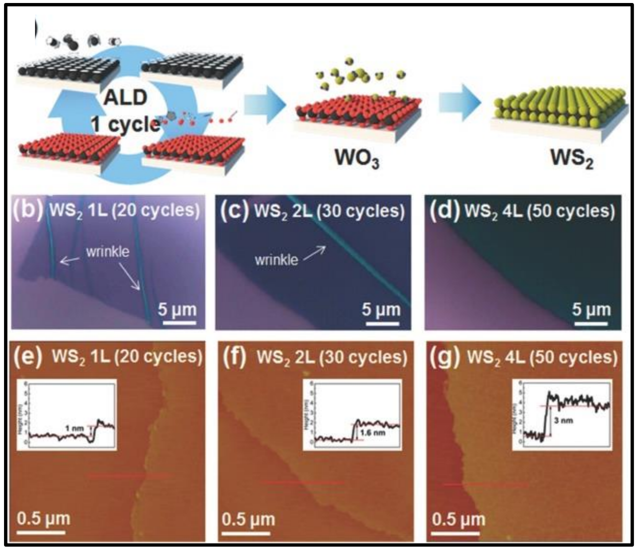
- Films with NC structures exhibit reduced run-in compared to amorphous films (composites); likely reduced reactivity as well







MoS_2+Ti+pure MoS_2 cap



MoS2 ALD at LBNL Molecular Foundry

- Developing ALD MoS2 freestanding and capped films for aging resistance
- MoOx conversion with Moly hexacarbonyl precursor and H2S conversion
- Challenges remain in optimizing synthesis to promote full conversion at low enough temperatures

Schwartzberg, A.M. and Olynick, D. "Complex Materials by Atomic Layer Deposition," *Adv. Mater.*, **27** (2015) pp. 5778-5784

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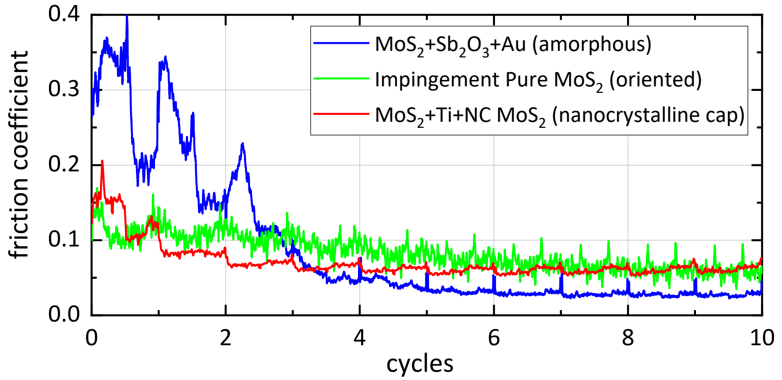

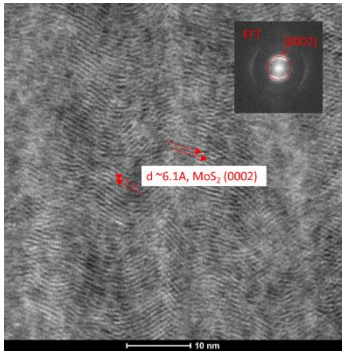

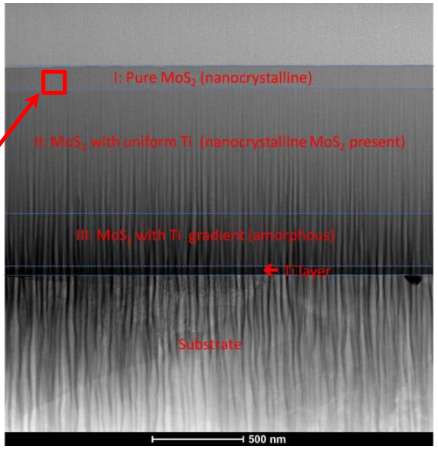

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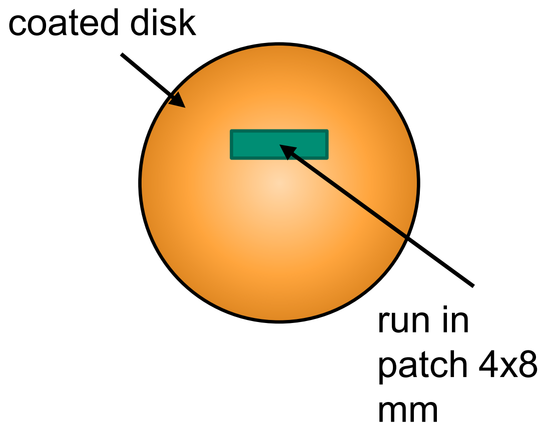
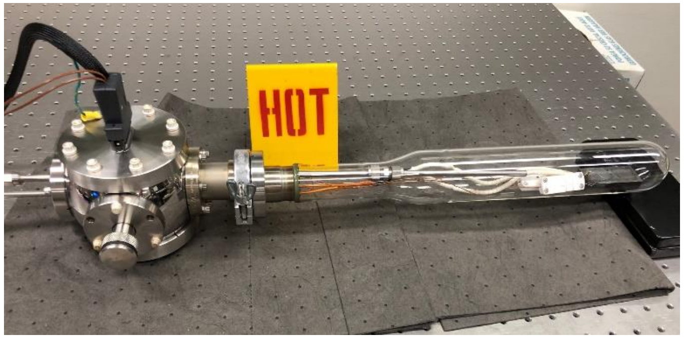
Aging Study - Experimental Methods

Run In



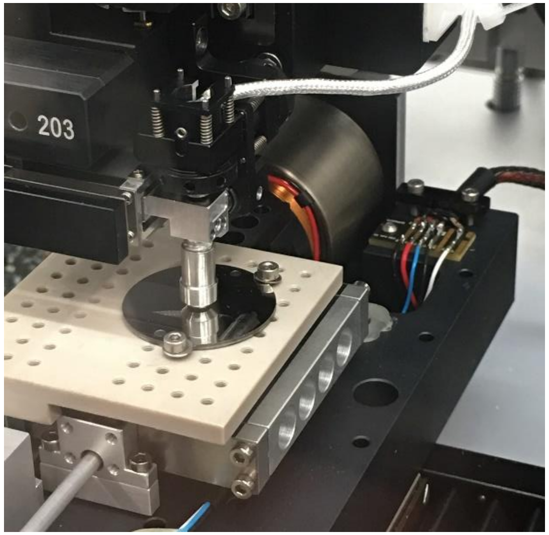
- 13-8PH or 440C stainless steel disks
- run in at 530 MPa, 50 passes, overlapping areas

Accelerated Aging



- 200ºC, dry (DP < -60ºC) air, 5 SCFH
- 12 hours

Friction Testing



- 440C ball, 3.2 mm dia.
- 1 mm/s sliding speed
- Hertz contact pressures of 275, 530 and 785 MPa

Materials Investigated:

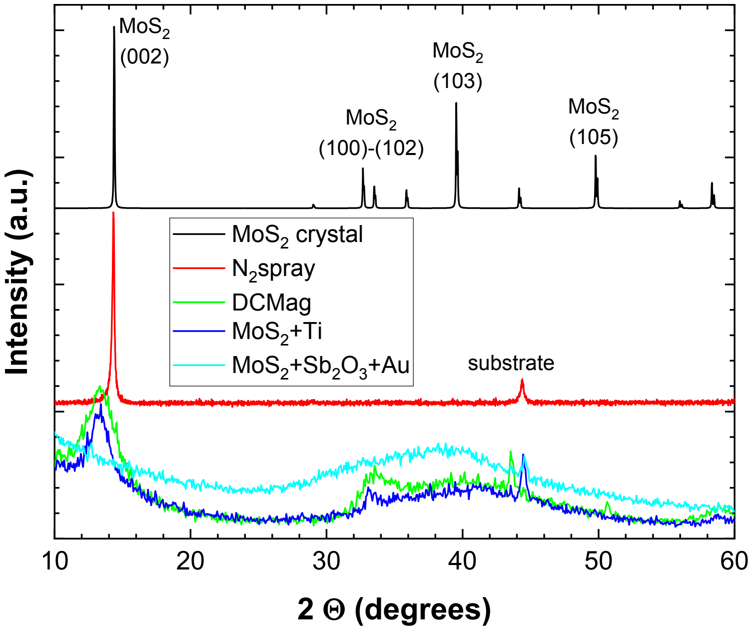
- N_2 (pure MoS_2 sprayed with N_2)
- DC (pure DC sputtered MoS_2)

} **Pure MoS_2**

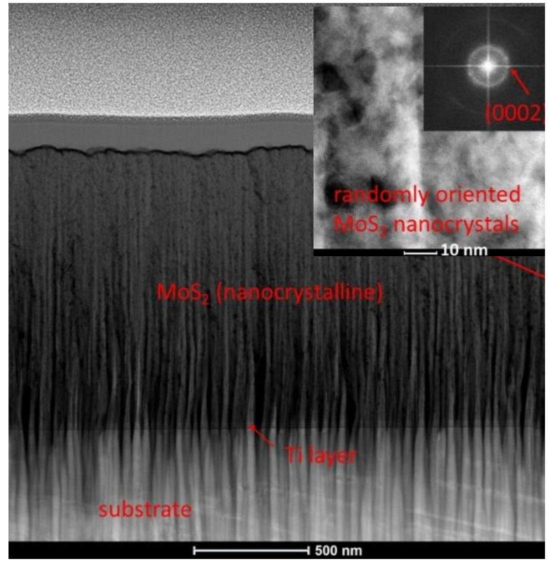
- Ti (RF sputtered MoS_2, Ti-doped)
- Sb_2O_3/Au (RF sputtered Sb_2O_3+Au-doped MoS_2)

} **Doped MoS_2**

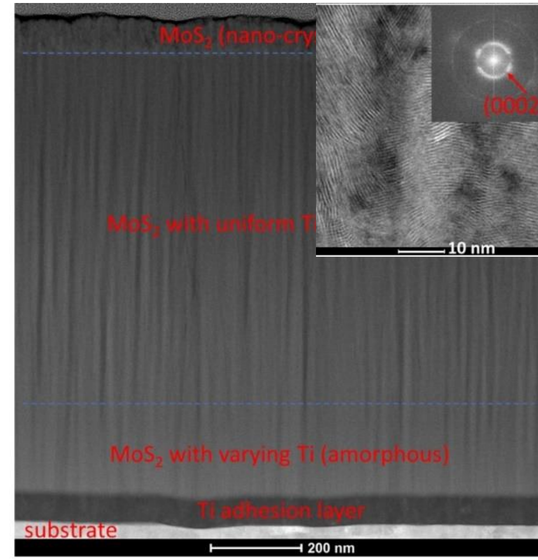
Coating Microstructures & Compositions



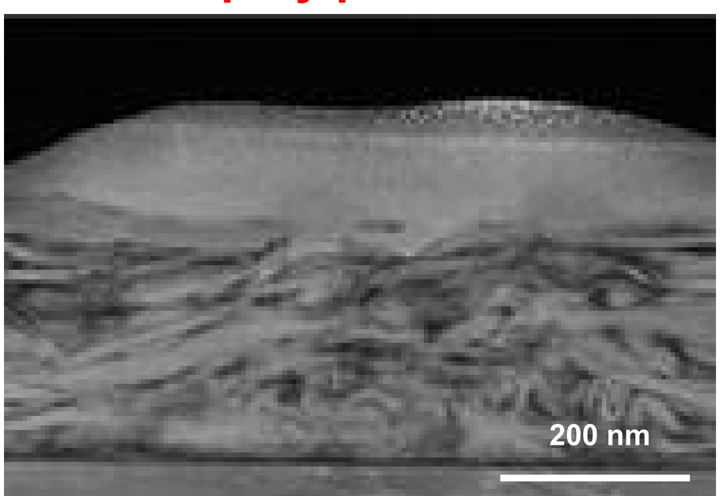
DC Mag pure MoS2



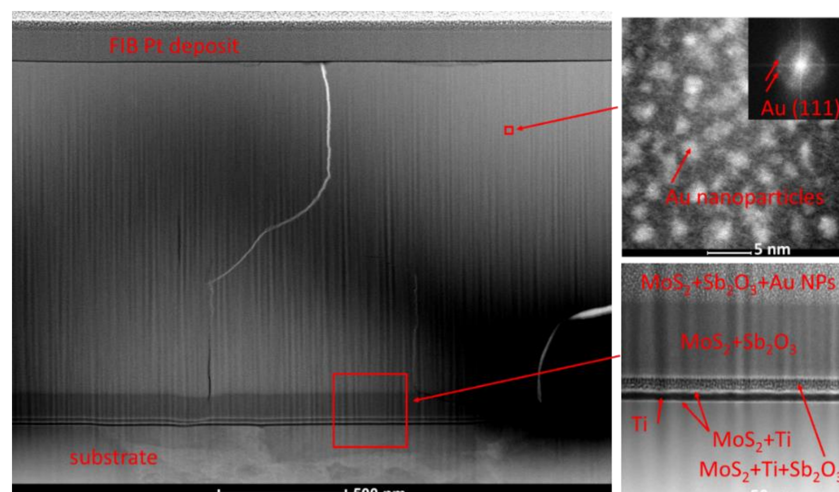
MoS2/Ti + pure MoS2



N2 spray pure MoS2



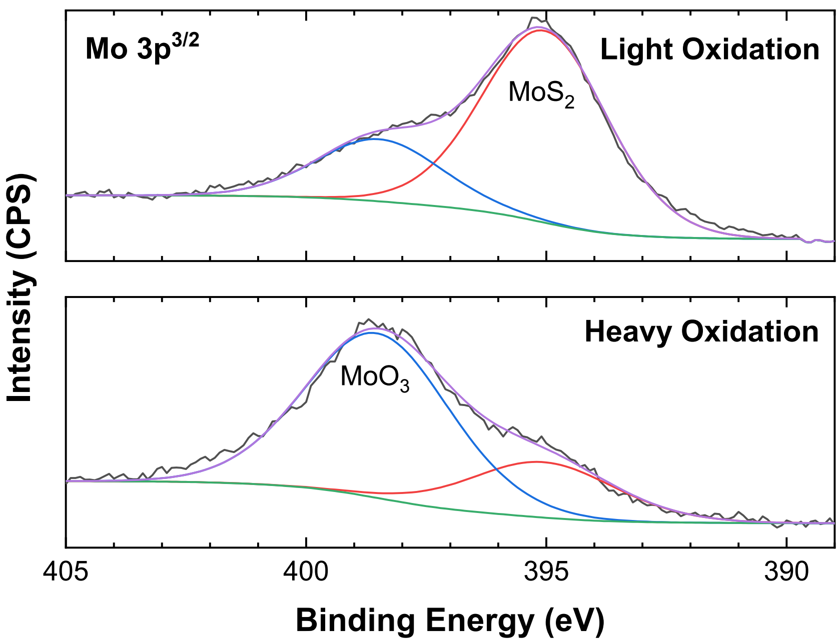
MoS2/Sb2O3/Au

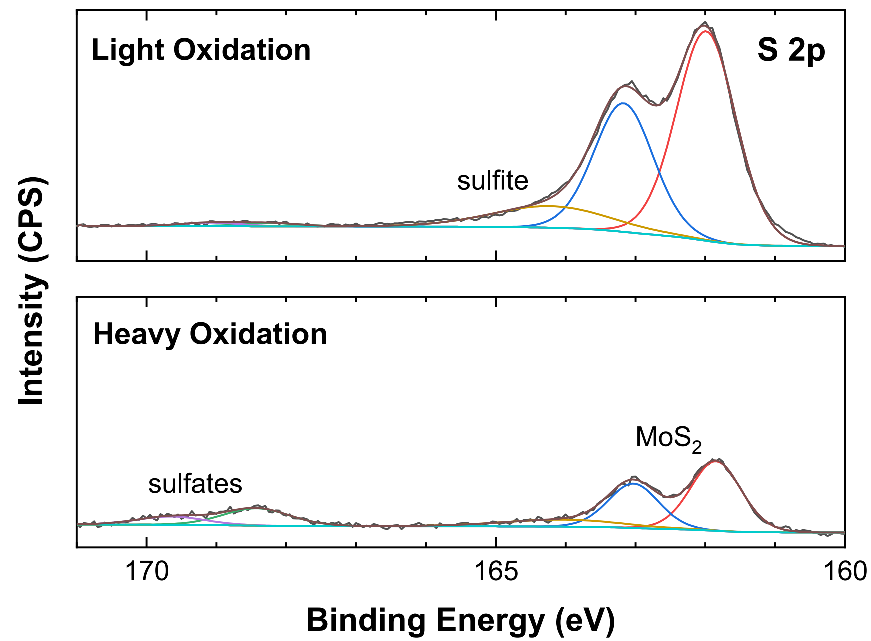


Quantifying MoS_2 Oxidation via XPS

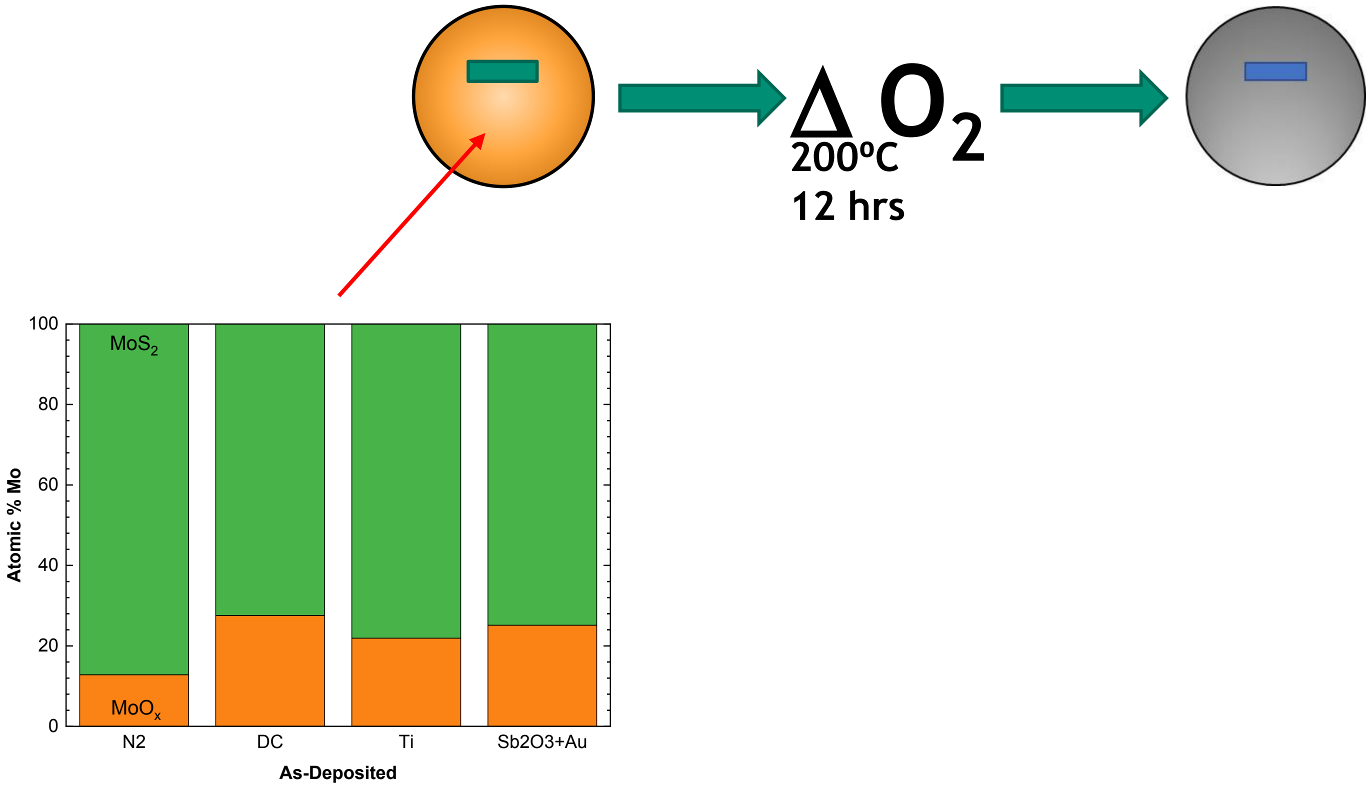
X-ray Photoelectron Spectroscopy (XPS) for surface chemical analysis

- survey scan for concentration of major elements present
- detailed scans of Mo3p, S2p spectral regions
- deconvolution of detailed scans to determine amount of Mo, S bonded to one another compared to oxidized species (MoO_3, sulfates, sulfites, etc.)
- surface sensitive – analyzing the top few nanometers





XPS Results – Pure



- Sprayed films natively contain less oxide than what is taken up during sputtering

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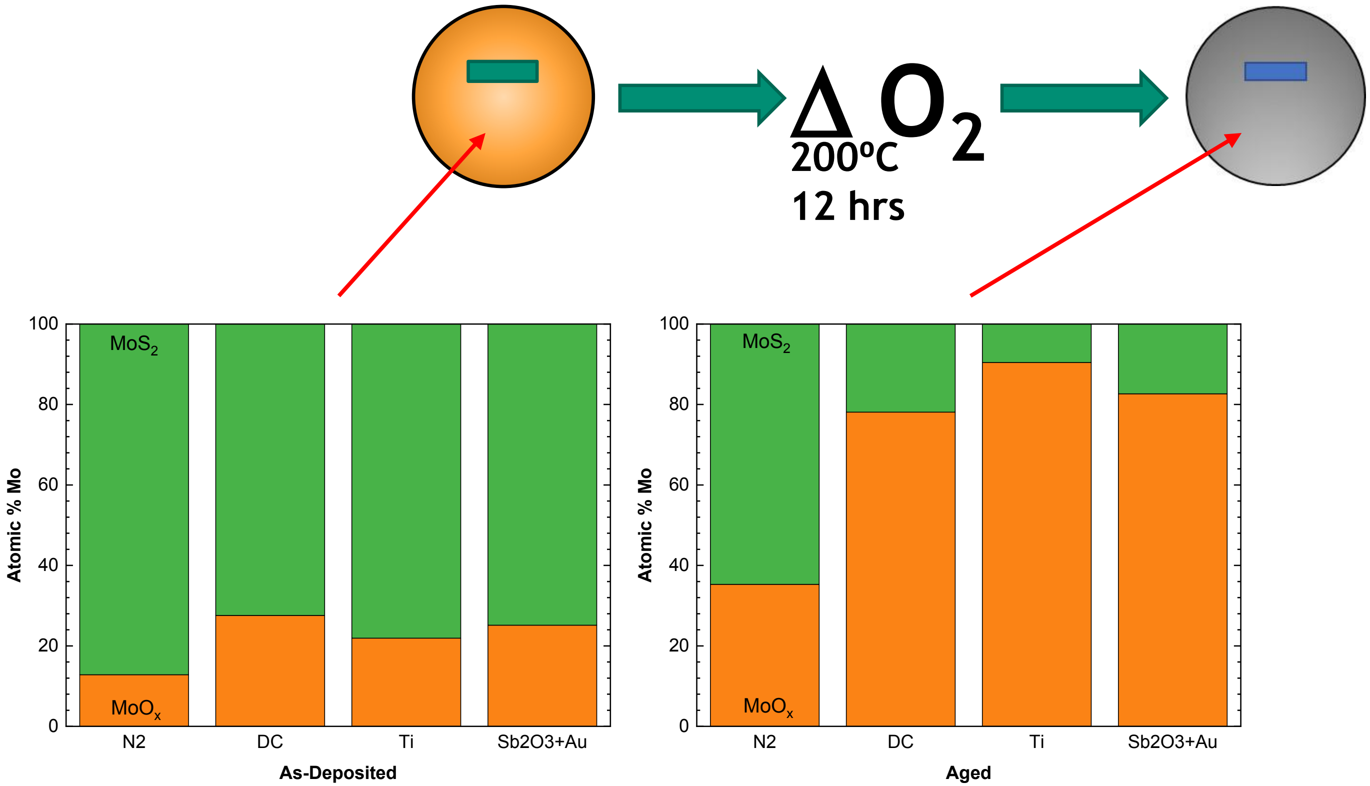

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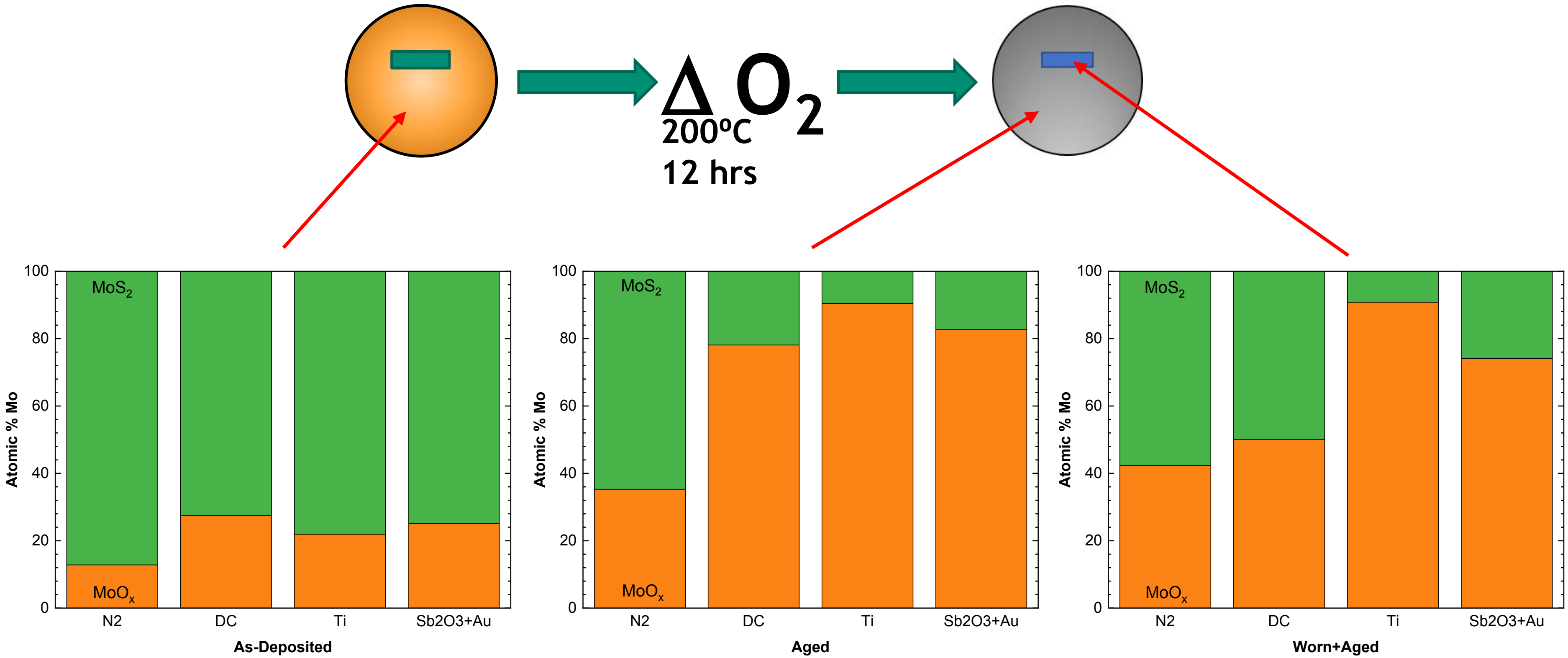
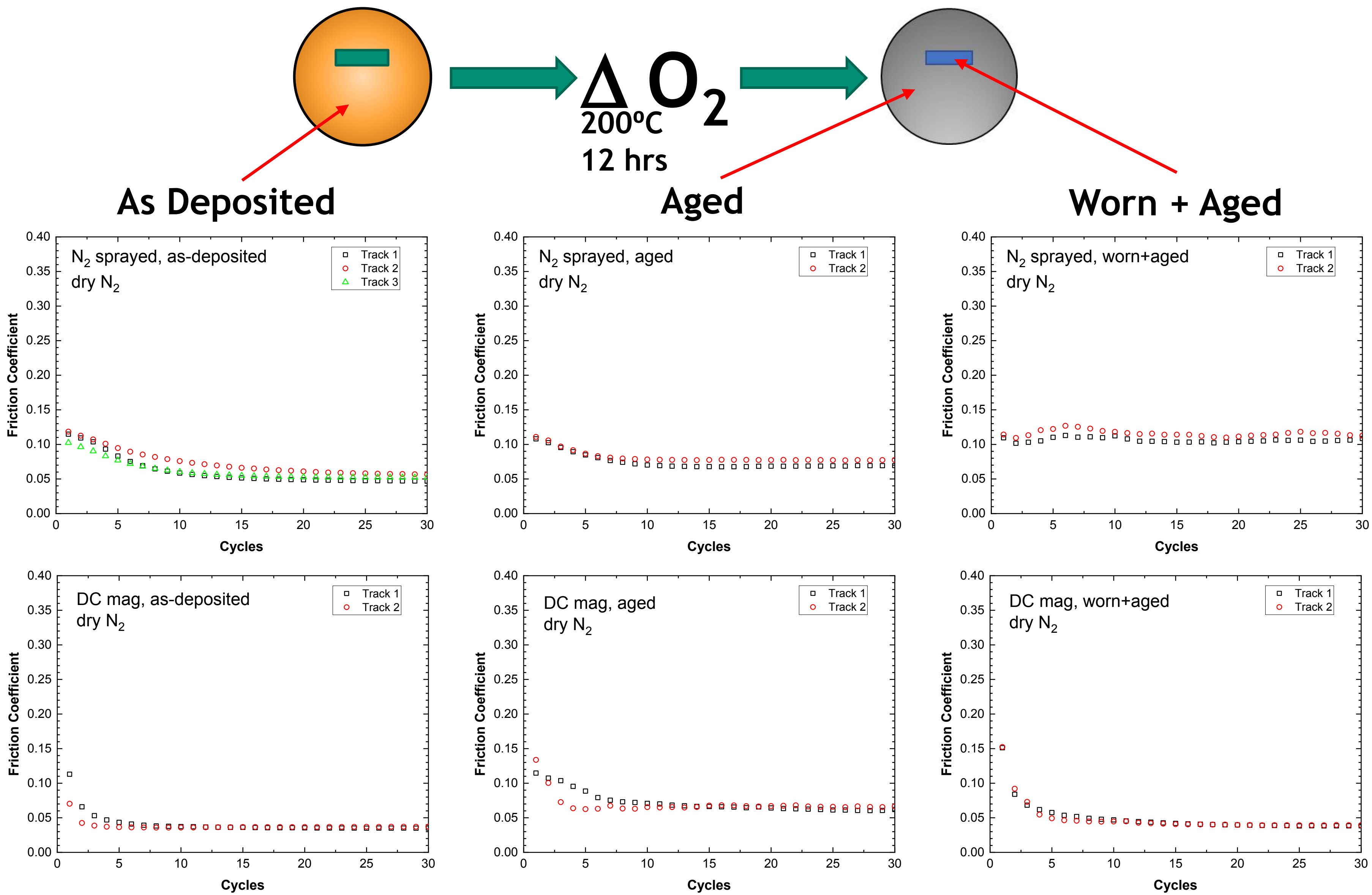
- Sprayed films in unworn state also less susceptible to oxidation

XPS Results – Pure

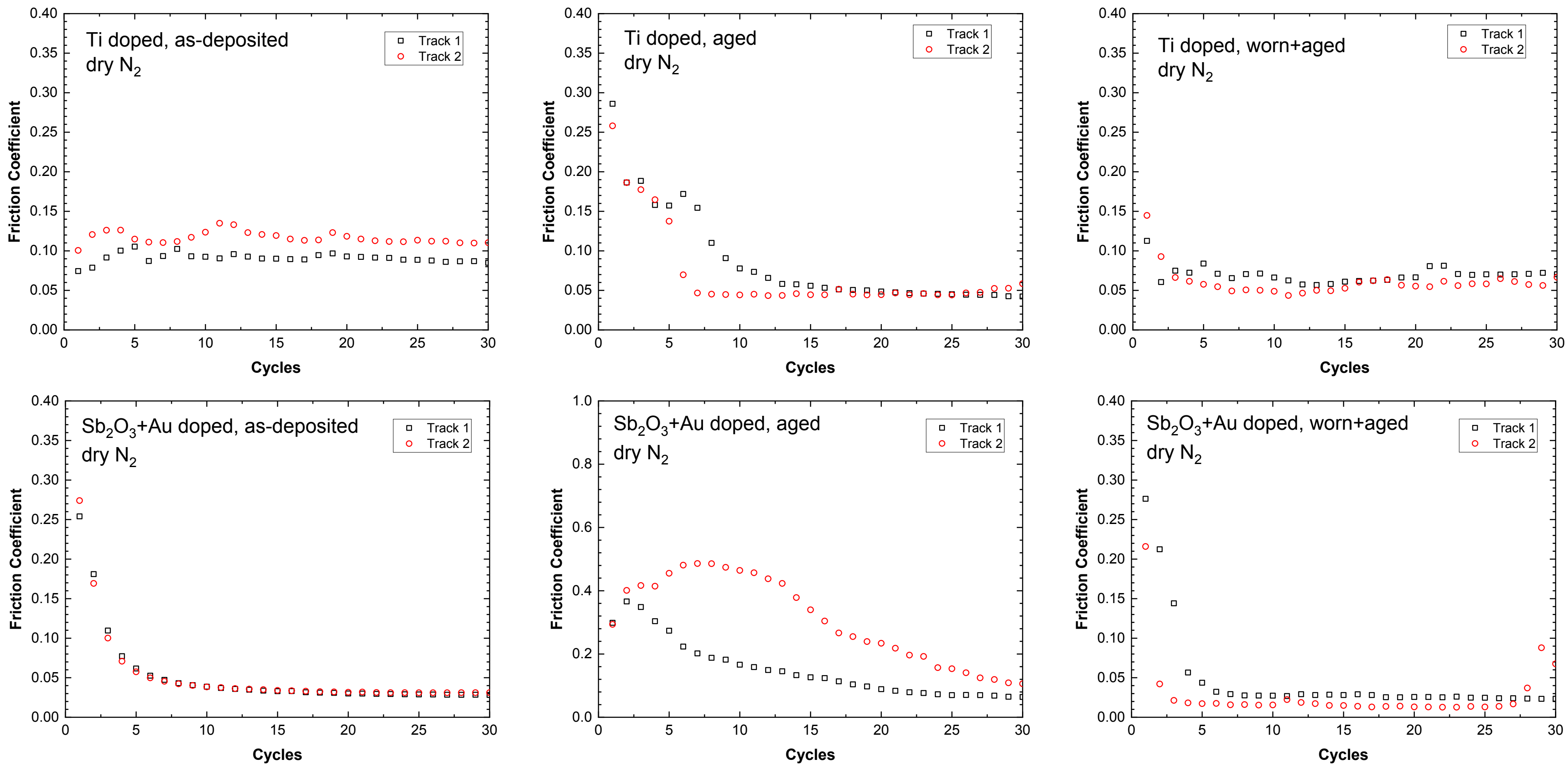


- Sprayed films natively contain less oxide than what is taken up during sputtering
- Sprayed films in unworn state also less susceptible to oxidation
- Worn area for DC mag films behaves like sprayed films, preventing oxidation
- Composite films run-in do not appear to buy significant protections to oxidation as compared to pure counterparts

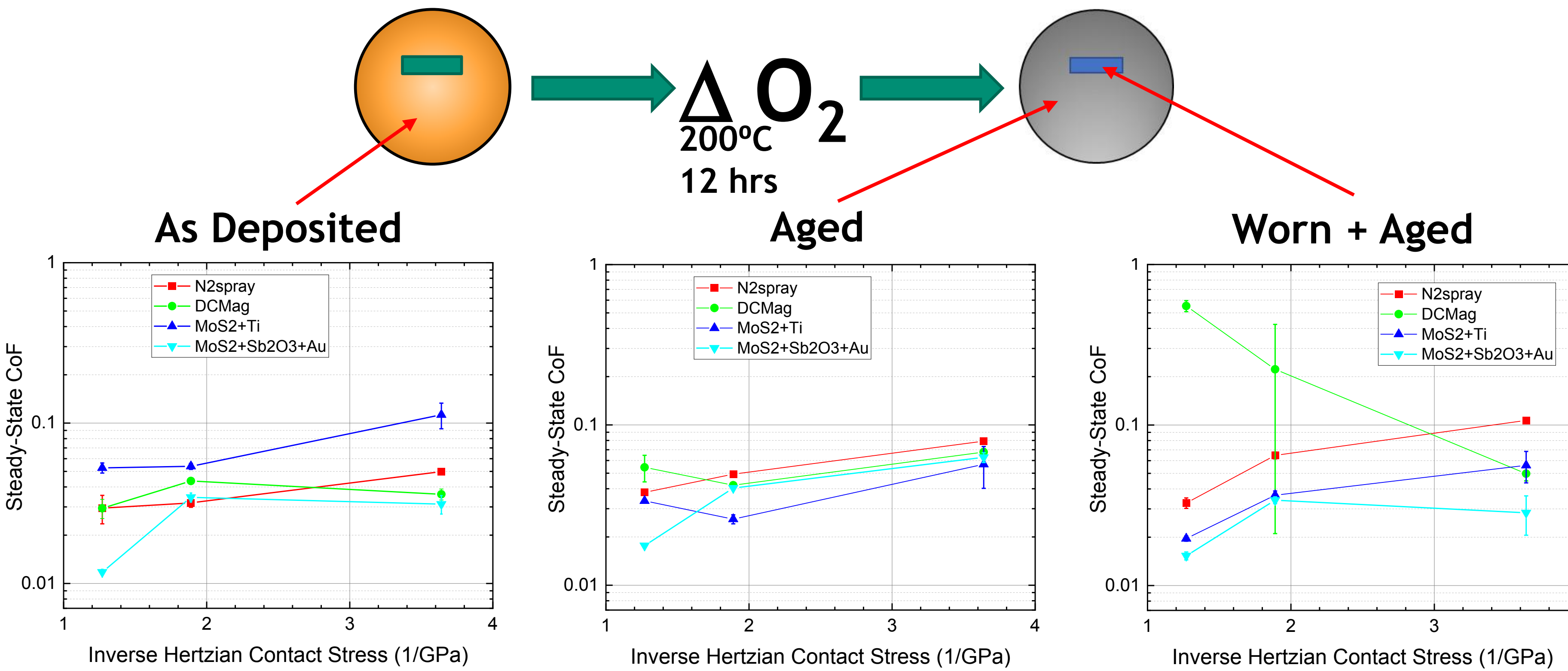
Friction Results – Pure MoS2 Films



Friction Results – Composite MoS2 Films



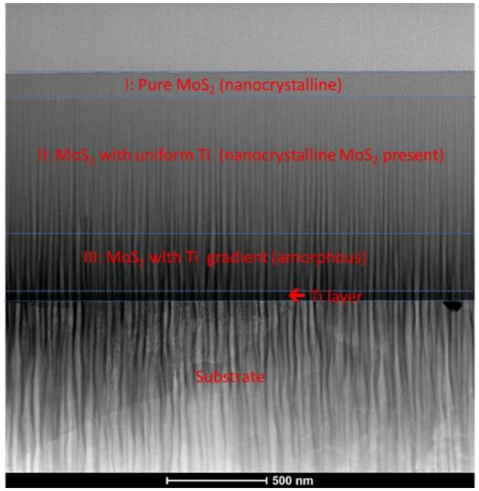
Steady State

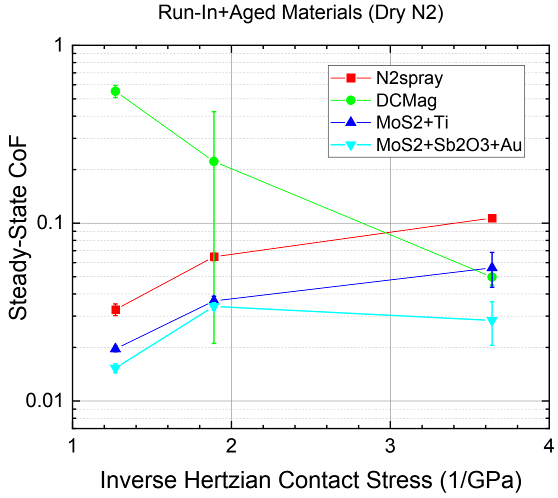


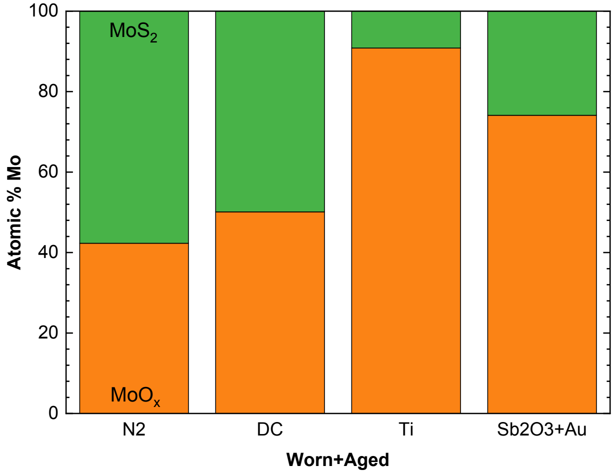
- MoS2-Sb2O3-Au generally lowest friction as deposited and behaved decently after aging
- MoS2-Ti films improved the most after aging
- Sprayed films & DC mag (both pure MoS2) relatively consistent but generally increasing after aging with DC mag exhibiting failures at higher loads

MoS2 Aging Series Takeaways

- Generally, doped films maintained lower steady state friction coefficients, and improved after aging/run-in prior to aging compared to undoped films
- Undoped films generally exhibit lowest initial friction behaviors compared to doped
- Undoped films also exhibit best oxidation resistance, likely due to lower reactivity of more crystalline materials at film surface (akin to run-in)
- Results suggest it is possible to sputtered MoS2 films with structure that can resist oxidation & minimize initial friction via surface modification







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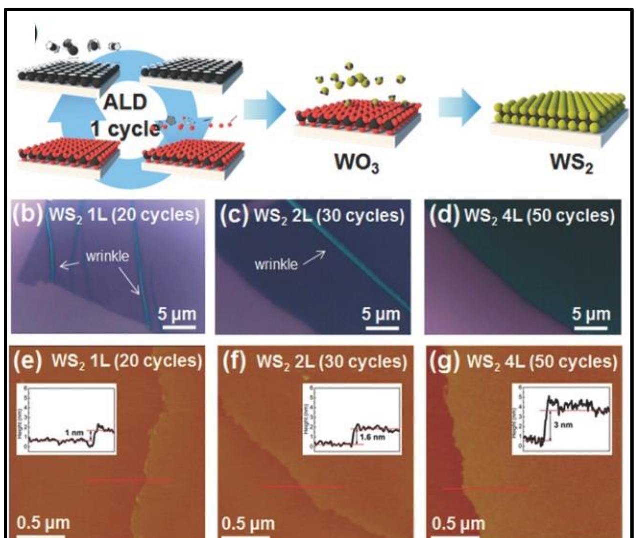

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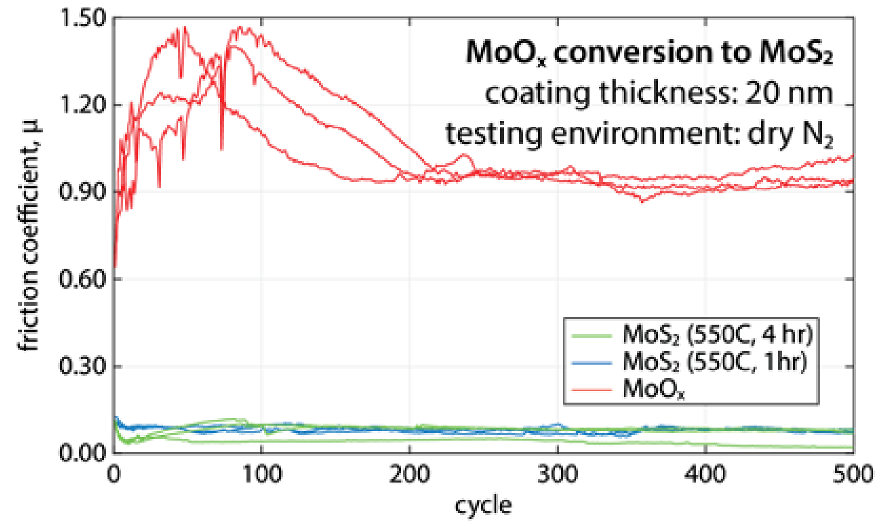
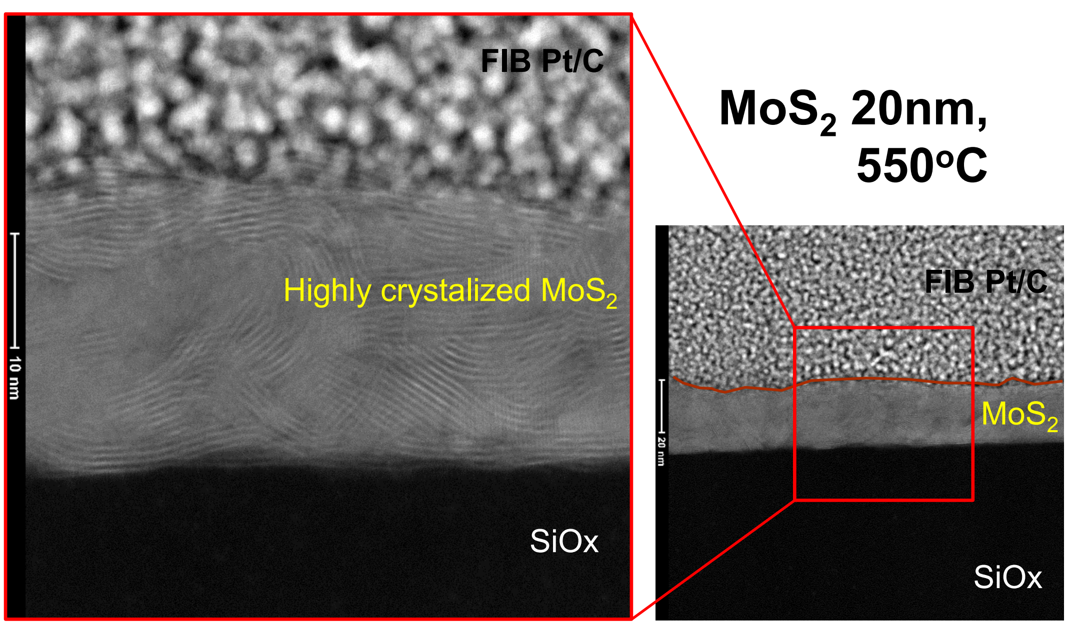
ALD MoS2 Growth & Conversion

Deposition/Conversion conditions

- Moly-hexacarbonyl precursor (AO plasma with water?), ???C for ???hr producing 20nm thick MoOx on SiOx wafer
- Initial conversion conditions: 550C for 1-4 hrs, flowing H2S/Ar mixture gas



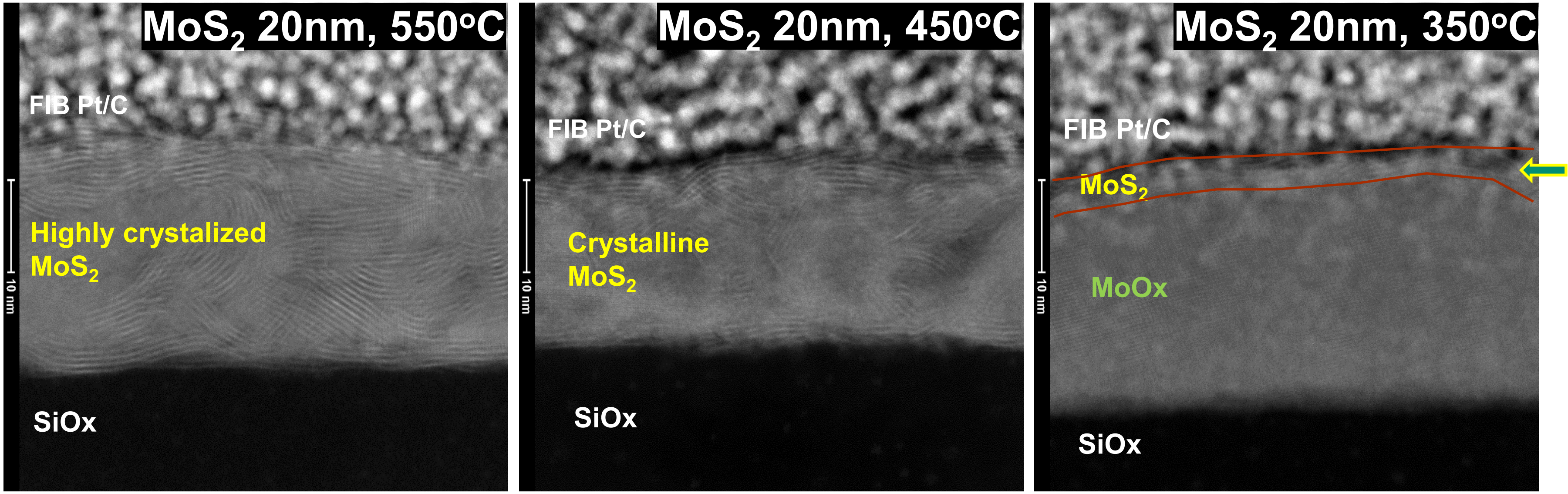


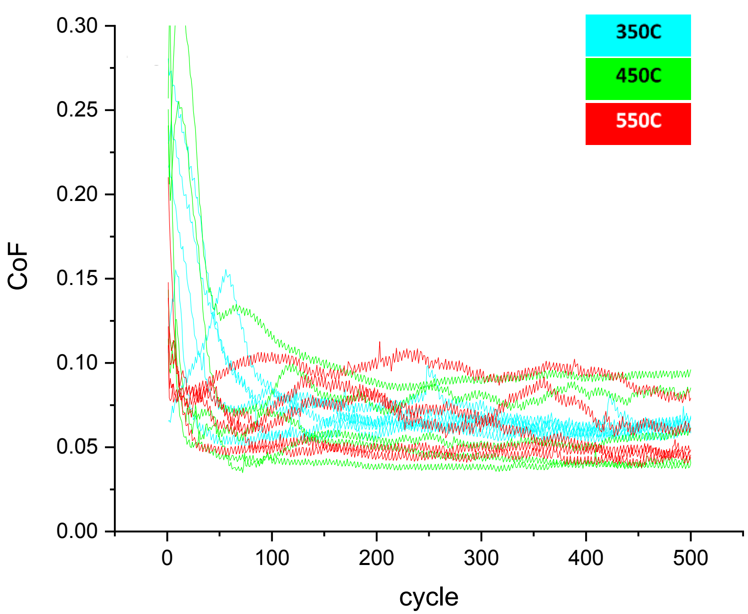
MoS_2 20nm, 550°C

TEM/friction observations

- Pre/post friction data shows clear change in friction behavior from oxide to sulfide
- TEM confirm presence of fully converted MoS2 at 550C conversion temperatures

Conversion at lower temperatures?



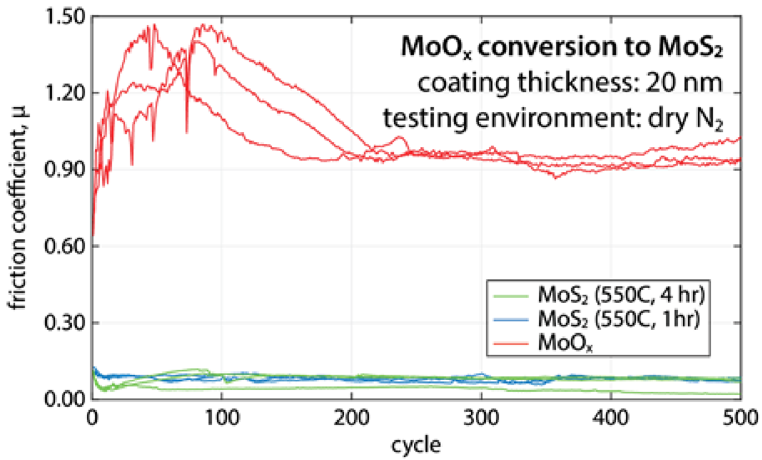


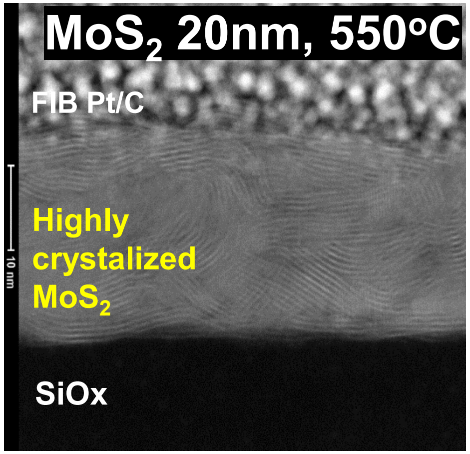
Observations

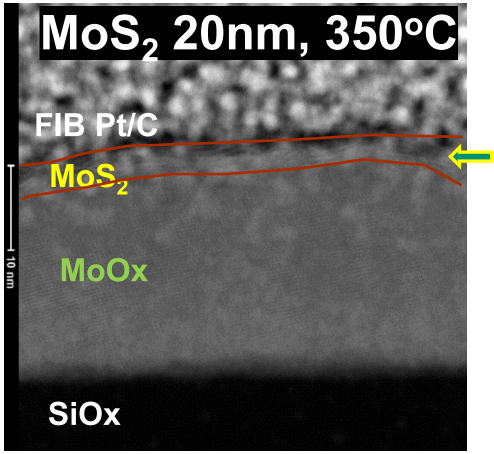
- Friction behavior at all conversion temps indicates presence of MoS2
- TEM reveals complete conversion at >=450 C
- For 350C conversion, only top few nm have converted to MoS2 (still providing ~500 cycles of low friction)

MoS2 ALD Takeaways

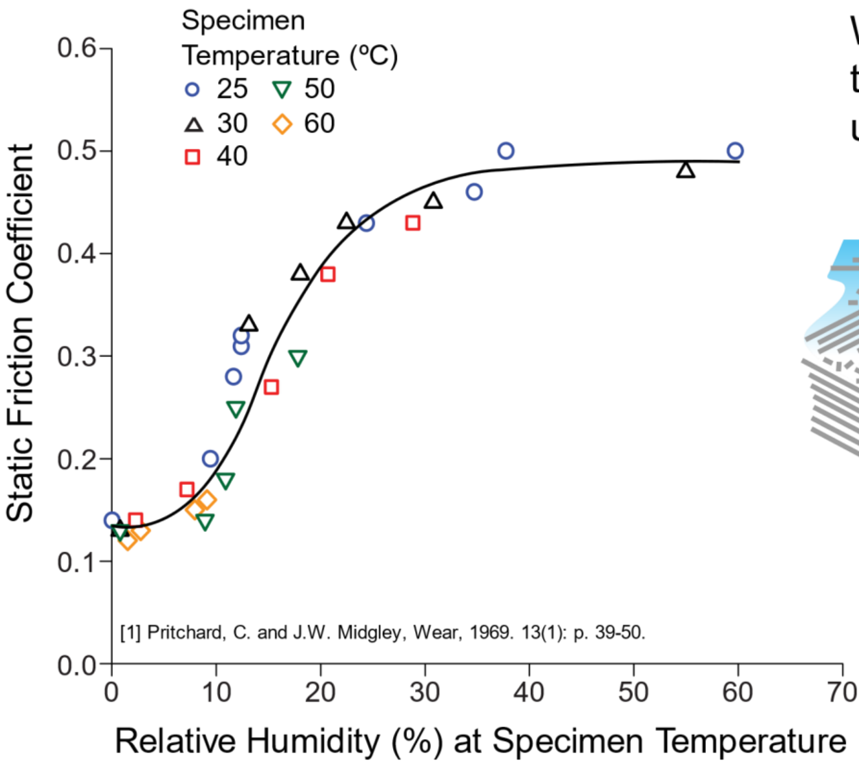
- MoOx growth and subsequent conversion is a relatively simple ALD process that can produce thin films with excellent friction behavior
- Complete conversion of 20nm oxide observed at 450C and above after 1 hr
- Incomplete conversion at 350C suggests kinetics not adequate, yet thin layer of MoS2 still provides lubrication
- Additional work required to understand if conversion is possible at lower temperatures to enable deposition on work parts (i.e. steels)



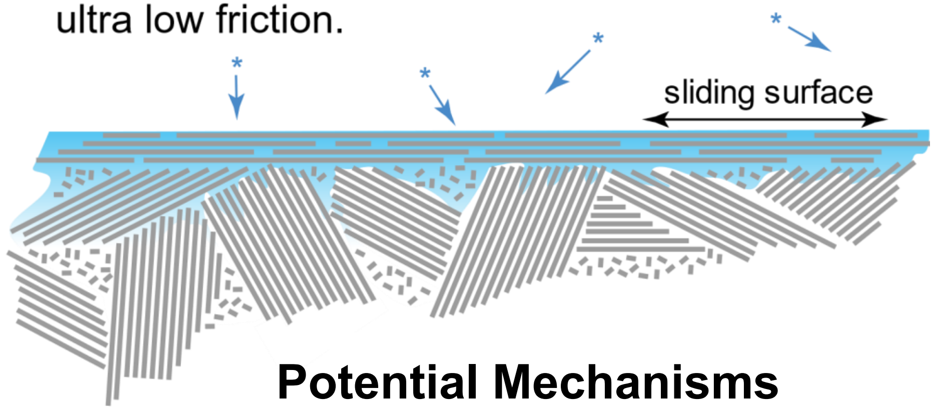




Fundamental Interactions with Water



Water adsorption and diffusion throughout a surface layer interrupts ultra low friction.



Potential Mechanisms

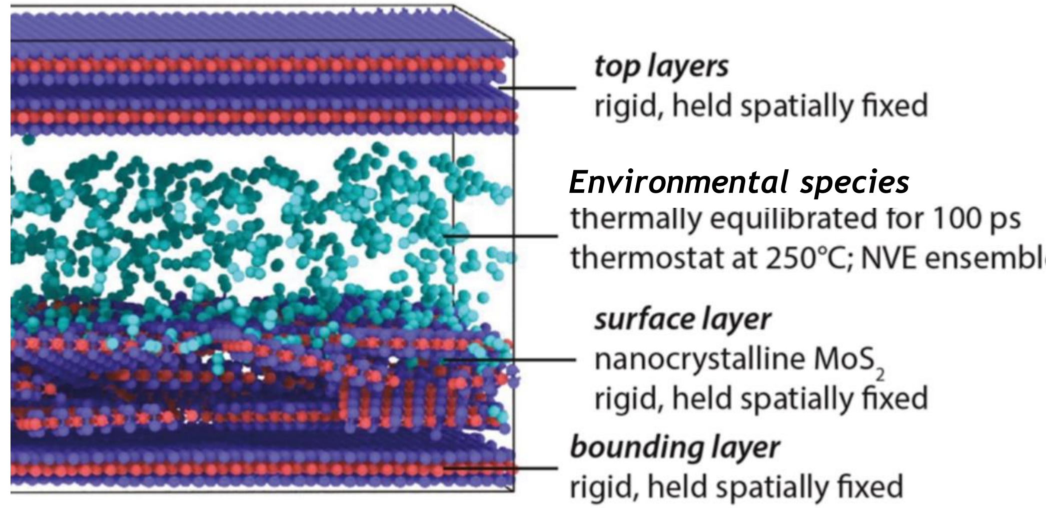
- Adsorption (polar bonding, capillary forces, edge interactions, etc…)
- Oxidation (H2O vs O2, high temp, etc…)

Little is known about how water fundamentally interacts and influences friction behavior in MoS2

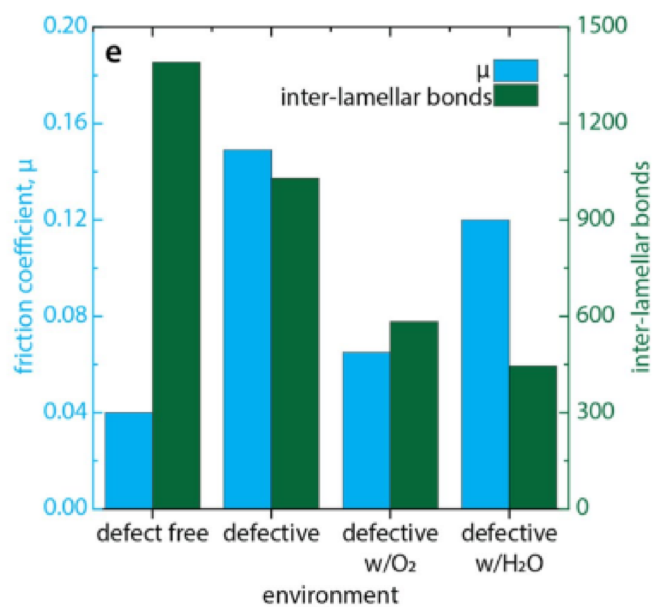
Structurally Driven Environmental Degradation of MoS2

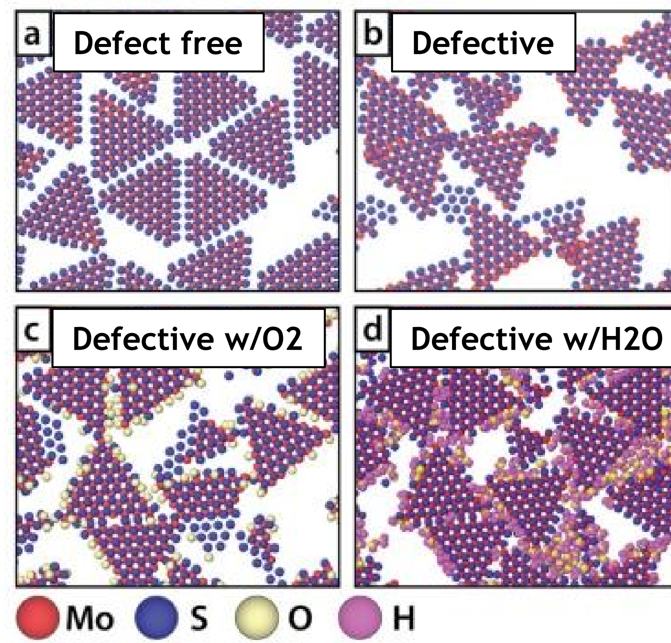
MD simulations show us a few things:

1. Sliding on pristine MoS_2 orders lamella and increases lamella size – low friction
2. O_2 and H_2O passivate edge sites preventing coalescence lamella



<u>Takeaway</u>
MD suggests changes to surface structure through environmental interactions dictate friction

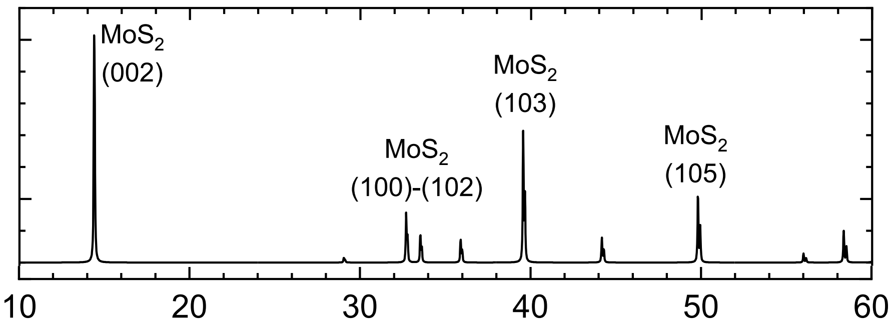


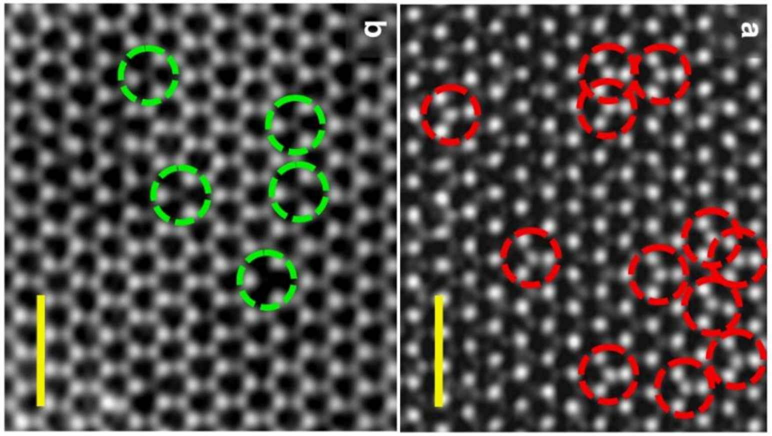


Curry, J. F. *et al. Tribol. Lett.* (2021)

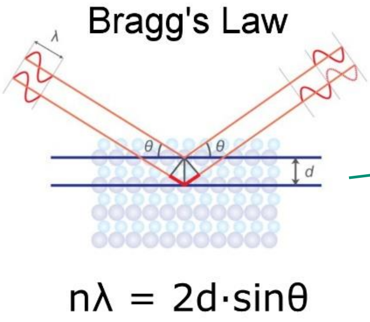
Diagnosing Surface Structure is Difficult

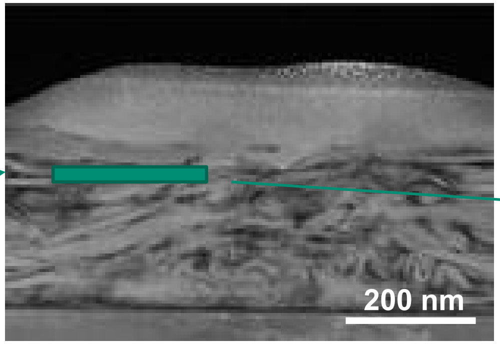
X-Ray Diffraction





STEM-ADF (Hong et al. Nat. Comm. 2015)



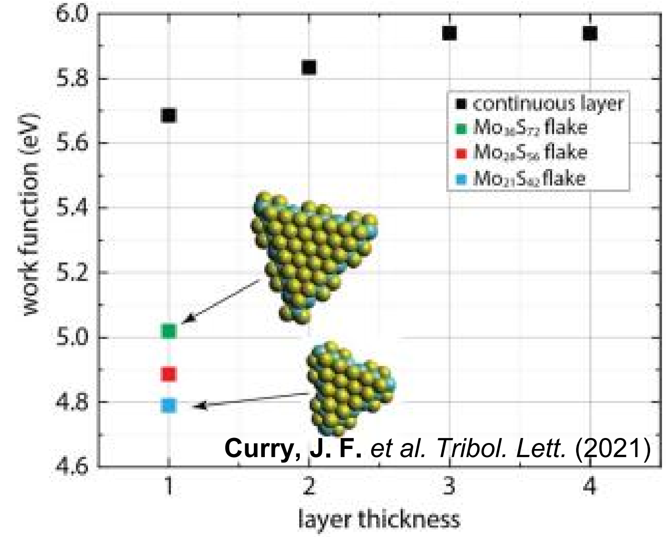


XRD can easily prove crystallite thickness, not width and depth

Existing methods for characterizing surface structure and defects (i.e. XRD, TEM, STM, etc) still make it difficult to assess near surface crystallite distribution and defect density.

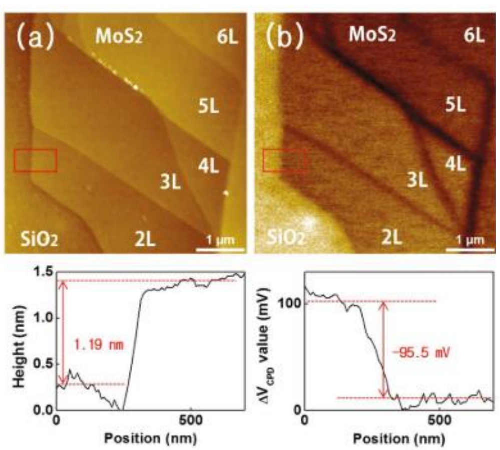
Using work function to observe changes in microstructure

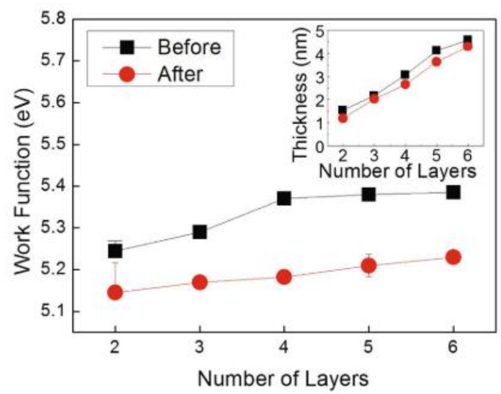
- Work function is a property of the surface
- Can be measured from KPFM, PEEM in combination with UPS
- Scales with number of layers
- Scales with the size of MoS2 lamella



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

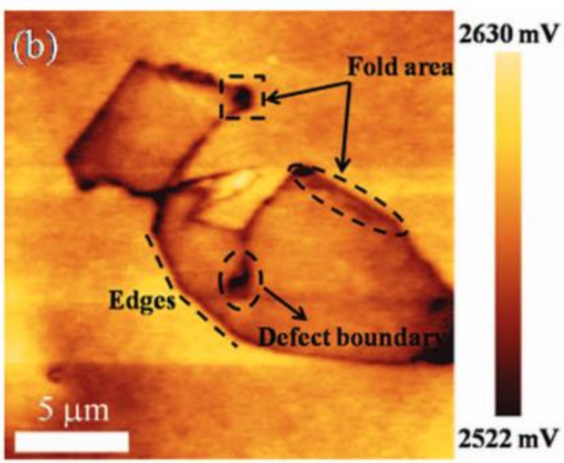
Role of thickness & adsorbates





Choi et al. *J. Korean Phys. Soc.* (2014)

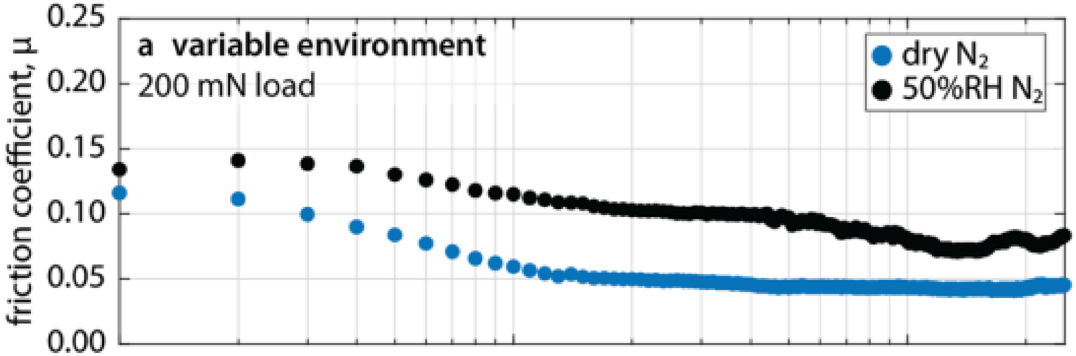
Role of grain boundaries & edges

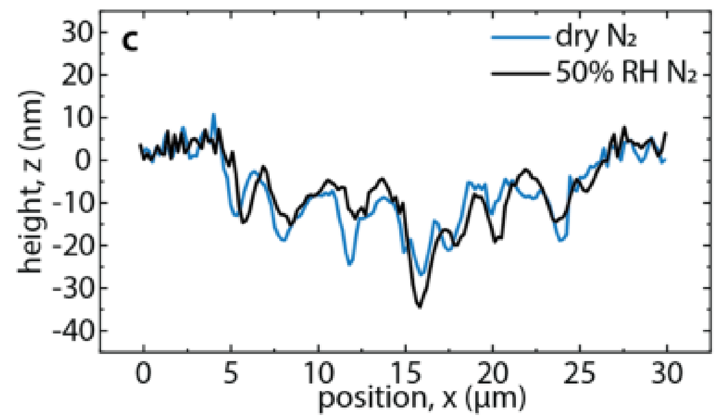


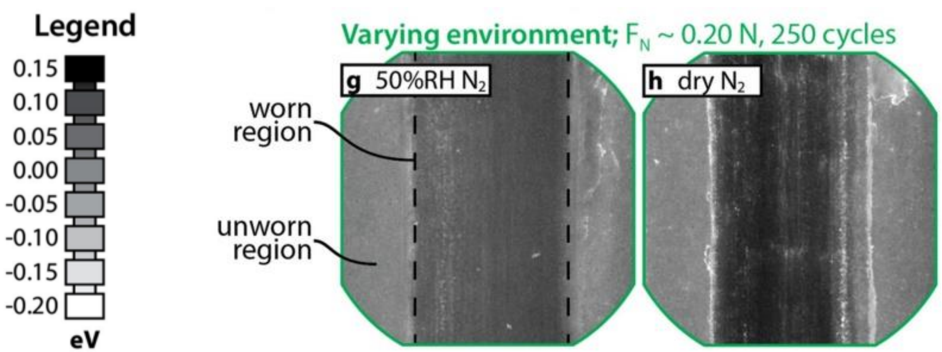
Hao et al. *AIP Adv.* (2013)

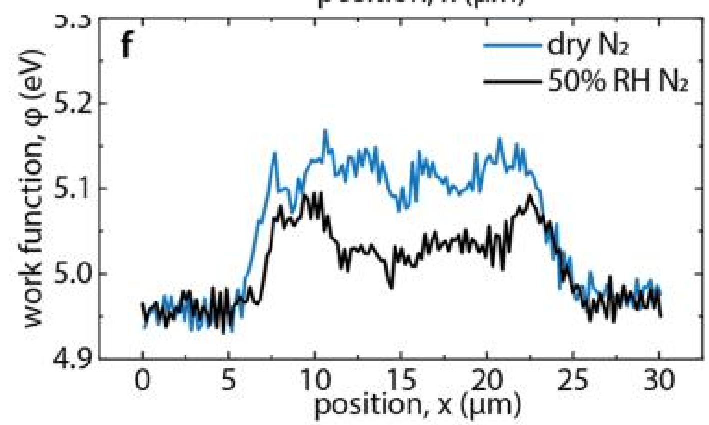
Takeaway: Work-Function can be used to assess changes in surface microstructure

Friction response driven by changes in structure







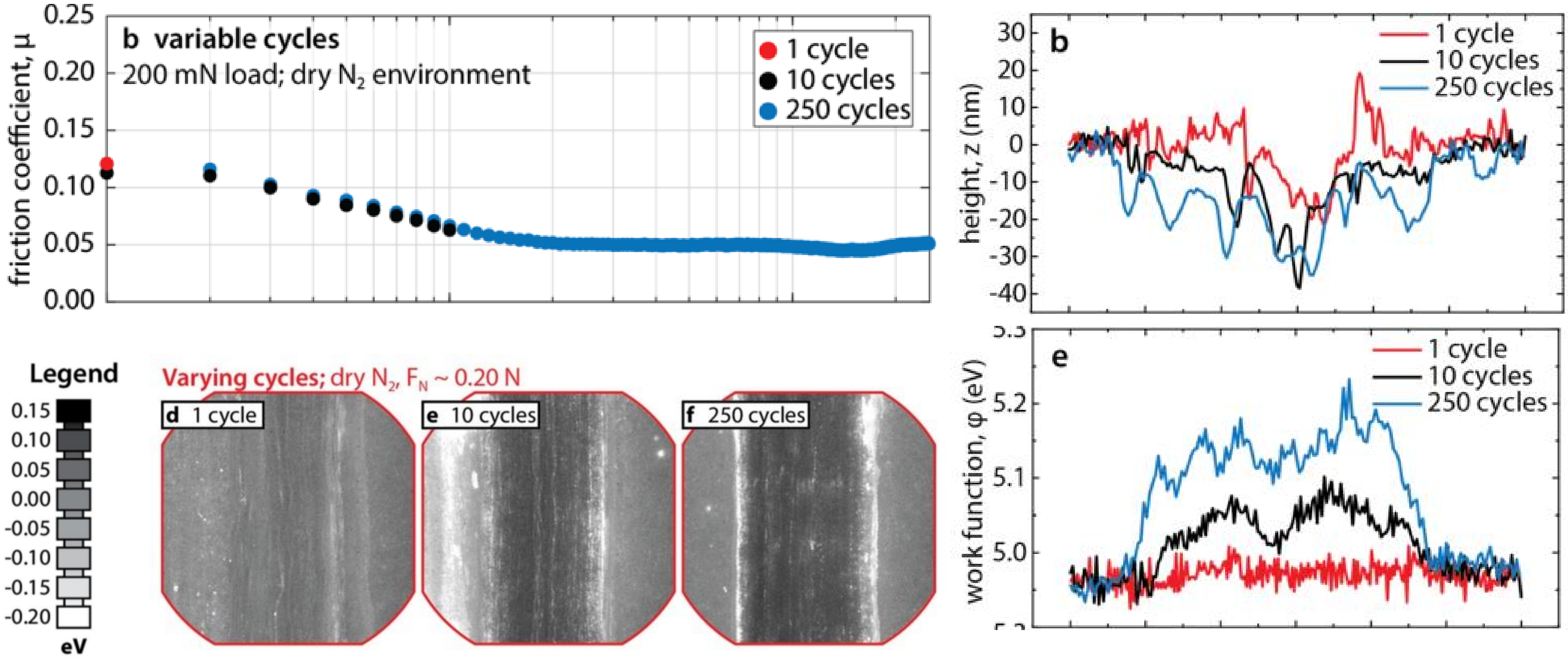


KPFM and PEEM indicate that shear in presence of H_2O still increases work function compared to bulk, but less than sliding without H_2O

Takeaway: H_2O decreases work function by inhibiting formation of large defect free lamella

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

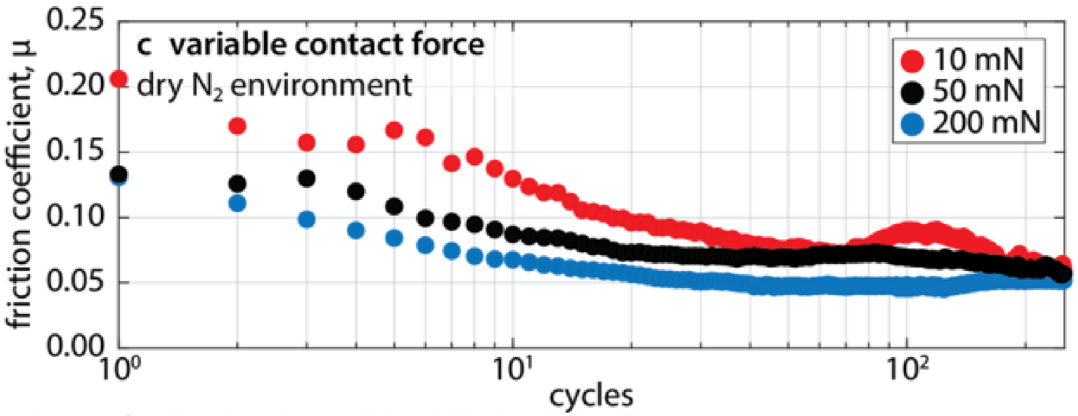
Friction response driven by changes in structure

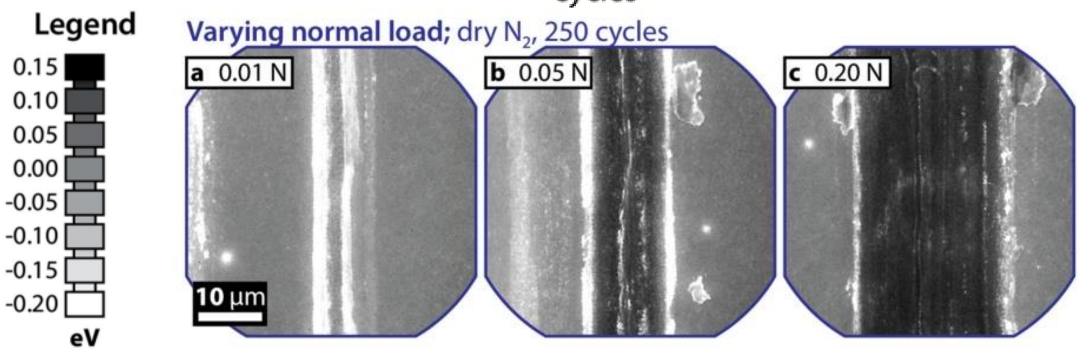


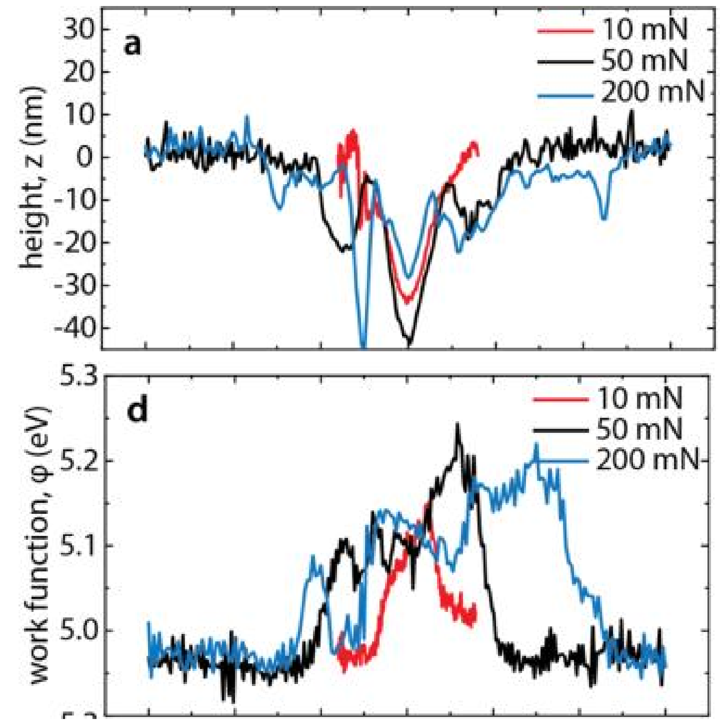
Takeaway #2: The transition to low friction "run-in" is a result of shear combining and reorienting lamella

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

Friction response driven by changes in structure





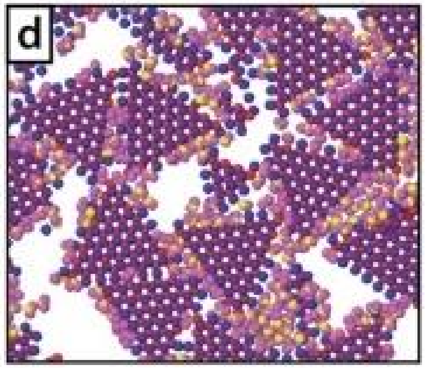


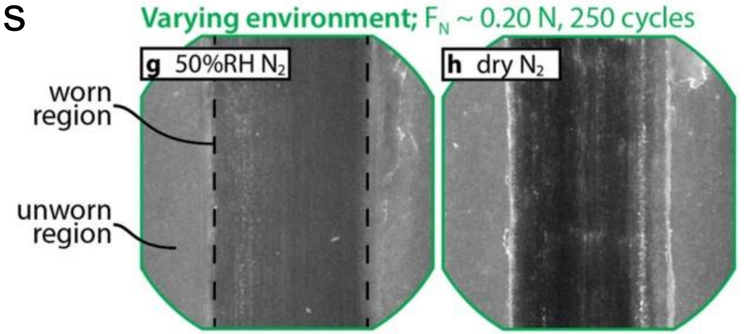
Takeaway #1: Increasing contact pressure forms larger lamella

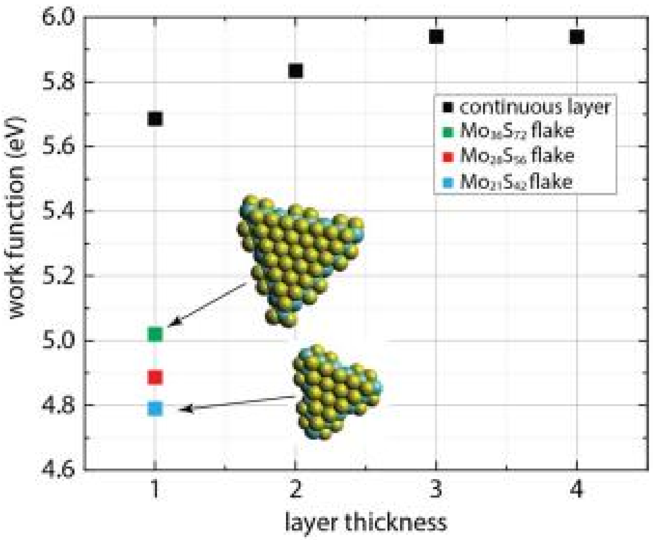
Curry, J. F. *et al. Tribol. Lett.* (2021)

Work Function on MoS2 Take-Aways

- 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
- Work function can be used to probe changes in structure at the surface at macroscale
- DFT and literature show that higher work functions are related to larger crystallites and higher layer counts (thickness) of MoS2
- Lower work functions were observed in wear scars associated with higher friction conditions (humidity, low load/cycle count) likely due to less ordered structures



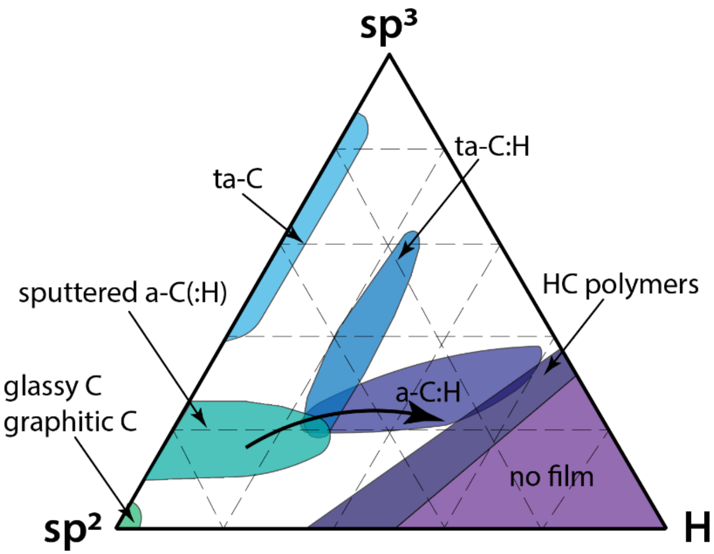




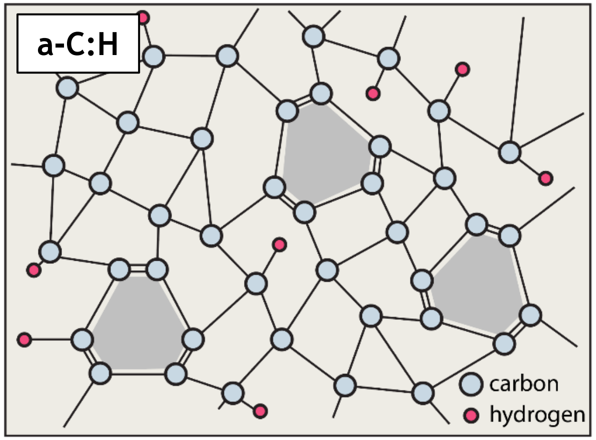
Curry, J. F. *et al. Tribol. Lett.* (2021)

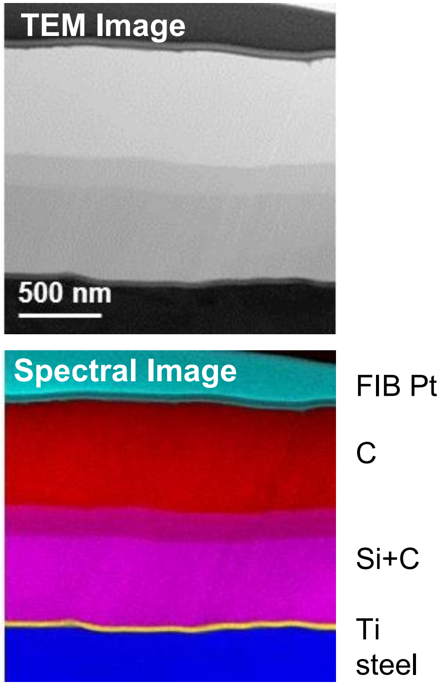
Understanding Variability in Run-in for DLC Coatings

Diamond-Like Carbon (DLC) Coatings



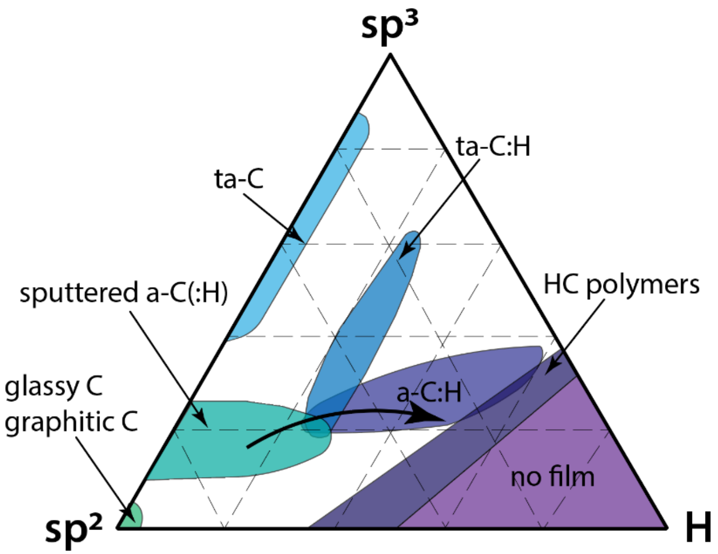
A.C. Ferrari, *PRB.* 2000





- Amorphous network of sp2/sp3/H or other dopant

Diamond-Like Carbon (DLC) Coatings



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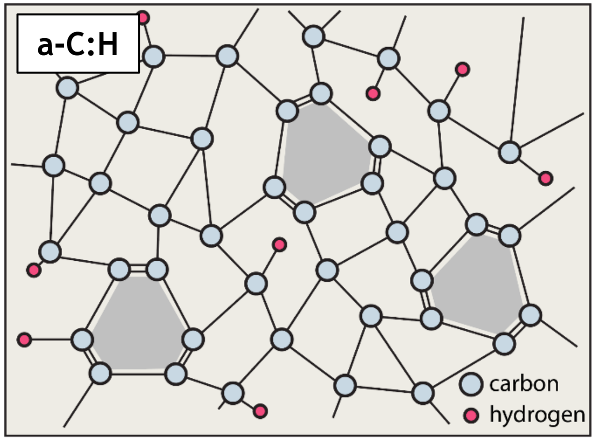
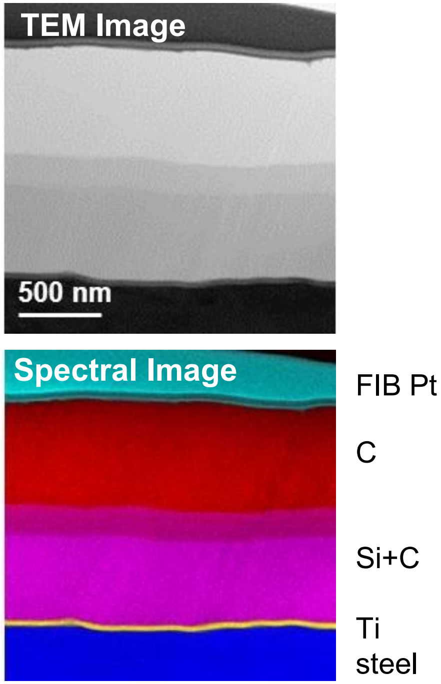
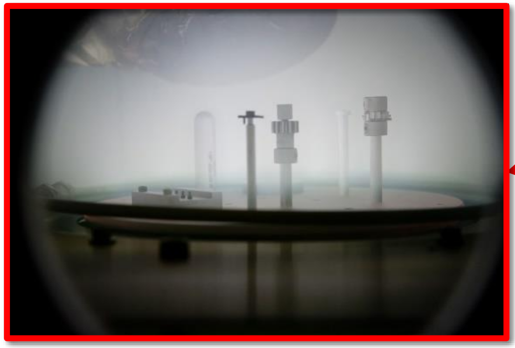




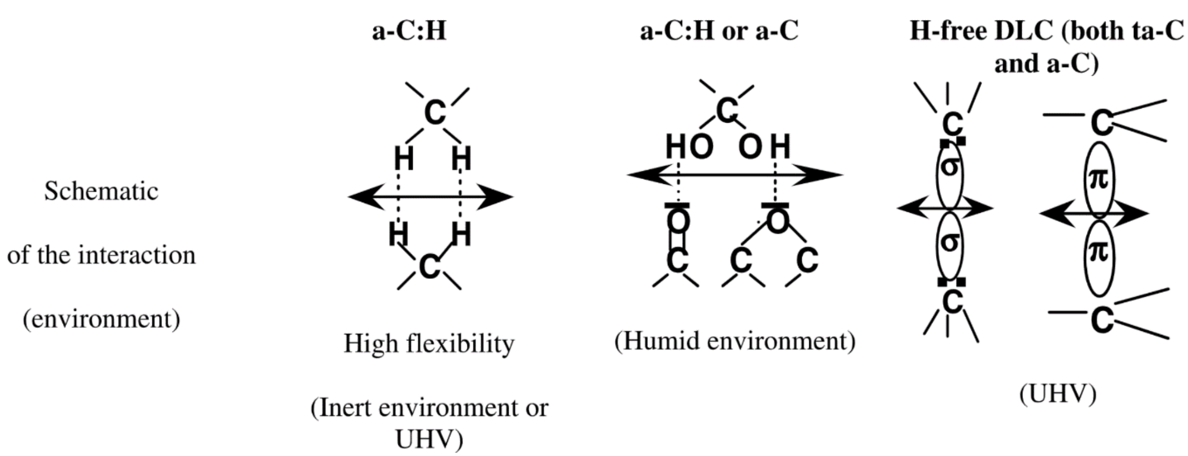


Images courtesy of Mike York, NSC

- Amorphous network of sp2/sp3/H or other dopant
- PECVD process decomposes precursor hydrocarbon gases to deposit carbon films
- Result in complex film compositions with varying tribological behavior…

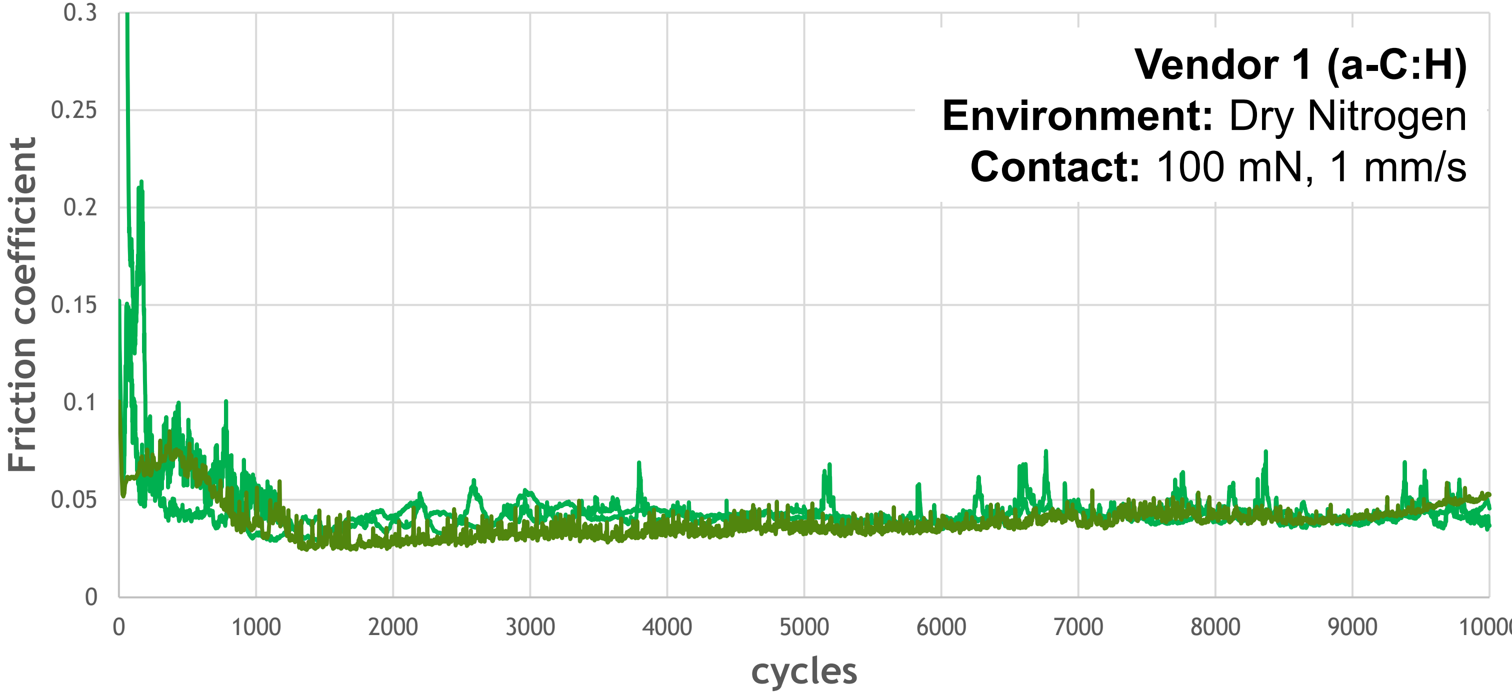
Friction Behaviors in DLCs

solid lubricant	deposition methods	μ_{ss}	F_n	environment
graphite (sp^2 bonding)	evaporation, pyrolysis of HC polymers	0.2 - 0.5	0.5 N - 1 N	dry N_2/UHV
		0.1 - 0.2	0.5 N - 1 N	humid air
DLC (mixed sp^2/sp^3 bonding)	rf and dc sputtering, ion beam, CVD	0.6 - 0.7 a-C	10 N	dry N_2/UHV
		0.001 - 0.05 a-C:H	10 N	dry N_2/UHV
		0.1 - 0.2 a-C	10 N	humid air
		0.2 - 0.3 a-C:H	10 N	humid air

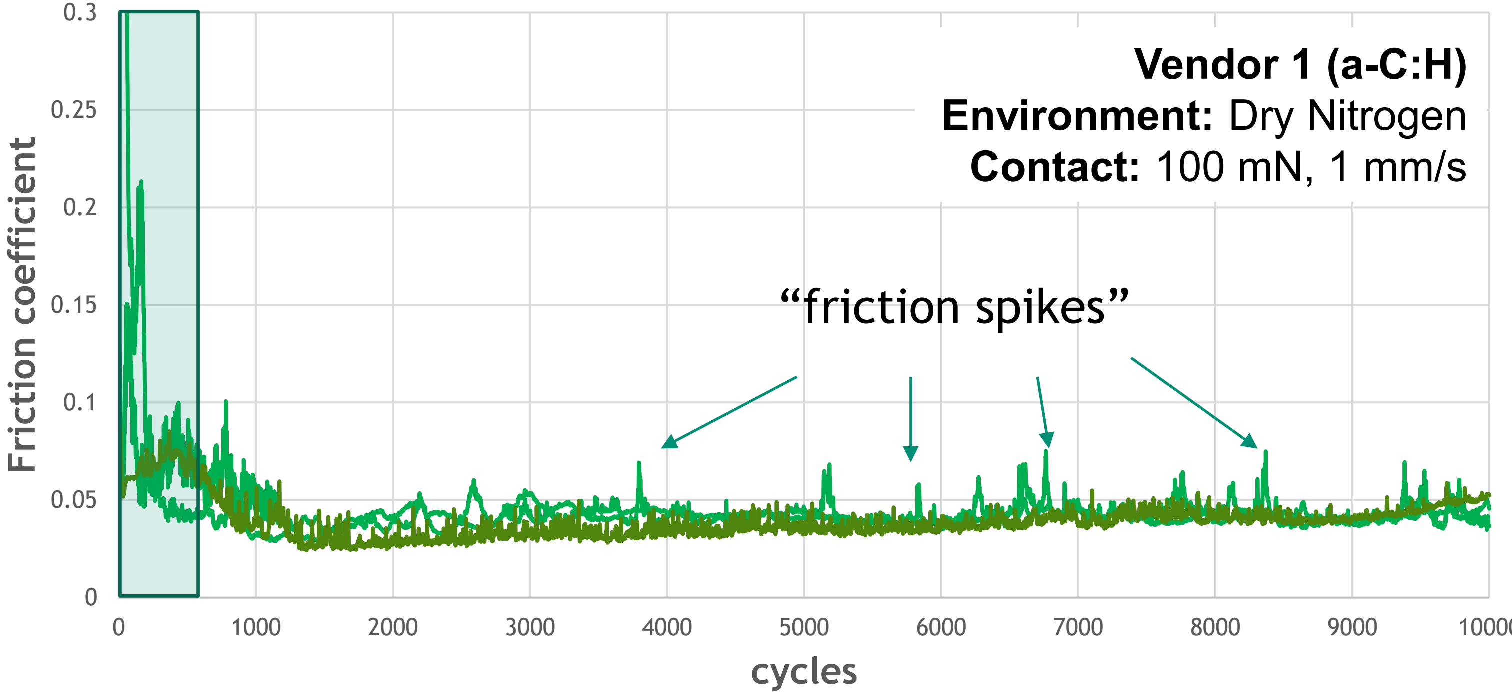


(Scharf, 2013, J. Matl. Sci.)
(Robertson, 2002, Matl. Sci. & Eng.)
(Erdemir, 2006, J. Phys. D: Appl. Phys.)

Typical DLC Friction Trace

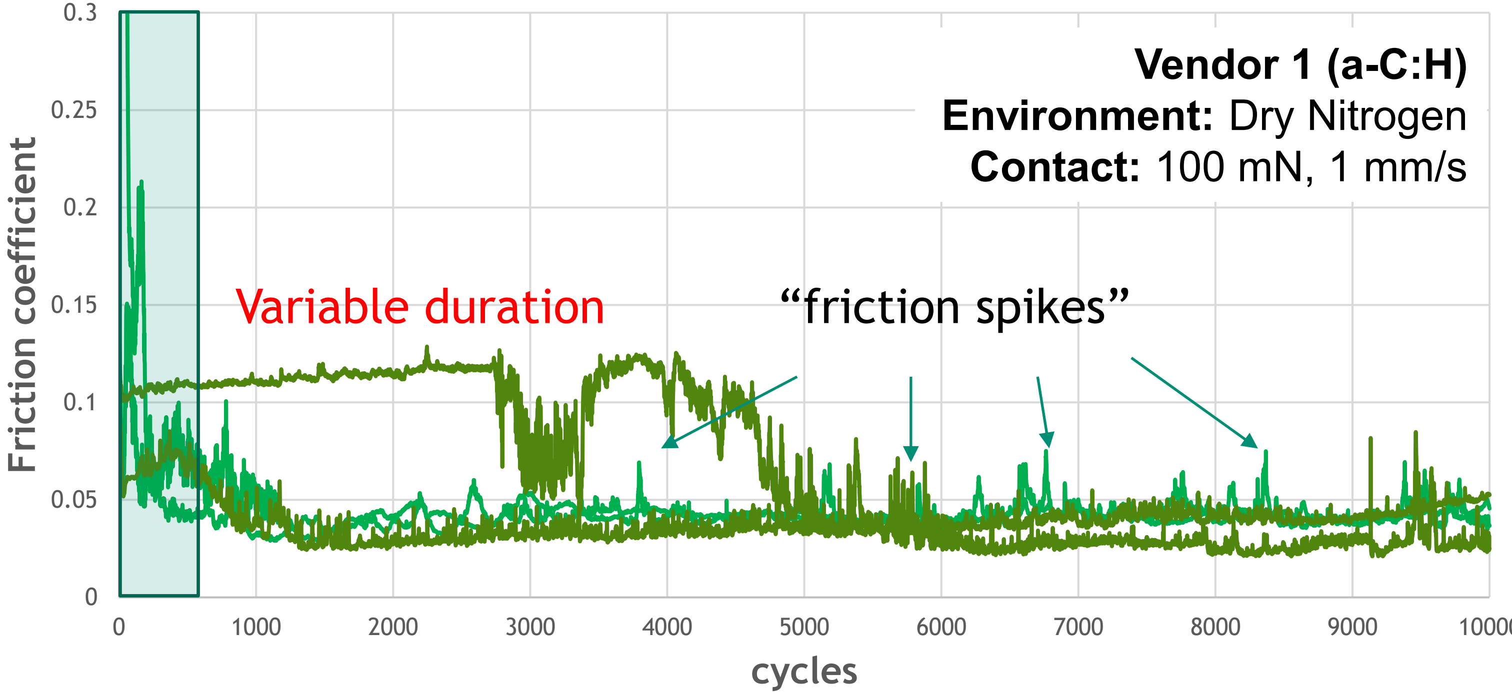


Typical DLC Friction Trace



- Coatings exhibit low steady state friction and occasionally "spikey" steady state behavior
- Magnitude of initial friction may vary between μ=0.10-0.30…

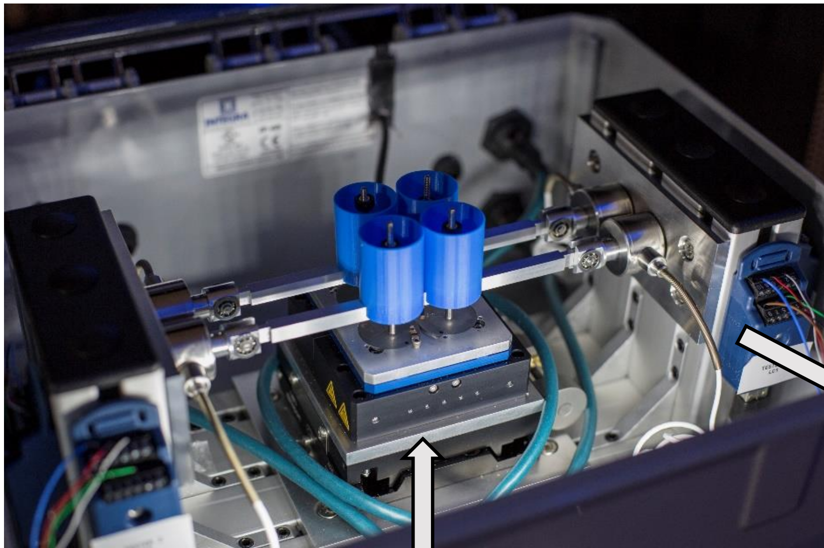
Typical DLC Friction Trace

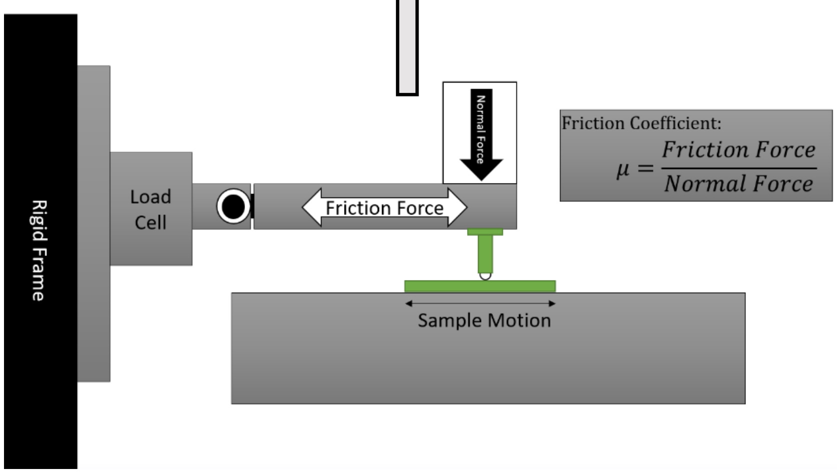


- Coatings exhibit low steady state friction and occasionally "spikey" steady state behavior
- Magnitude of initial friction may vary between μ=0.10-0.30… run-in duration varies extensively as well

Variability in run-in (like MoS2) can be a detriment to reliable operation

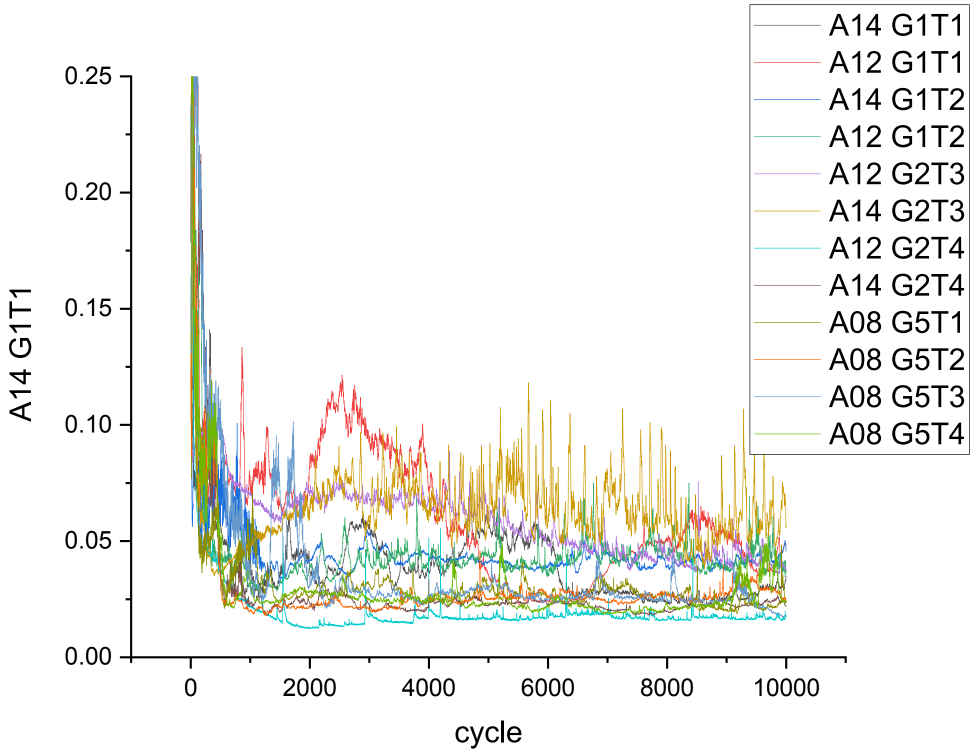
Brute Force – High Throughput Friction Testing

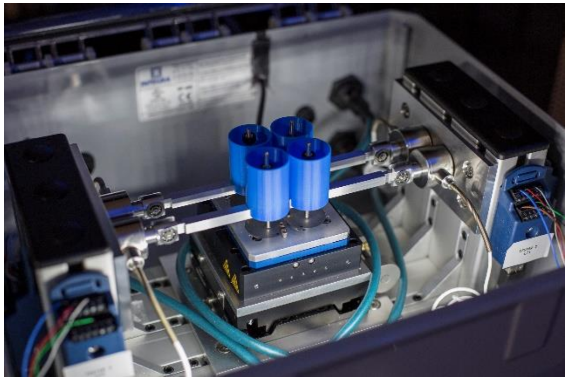




16X simultaneous testing capacity, multiple environments

HTT Case Study - Composition

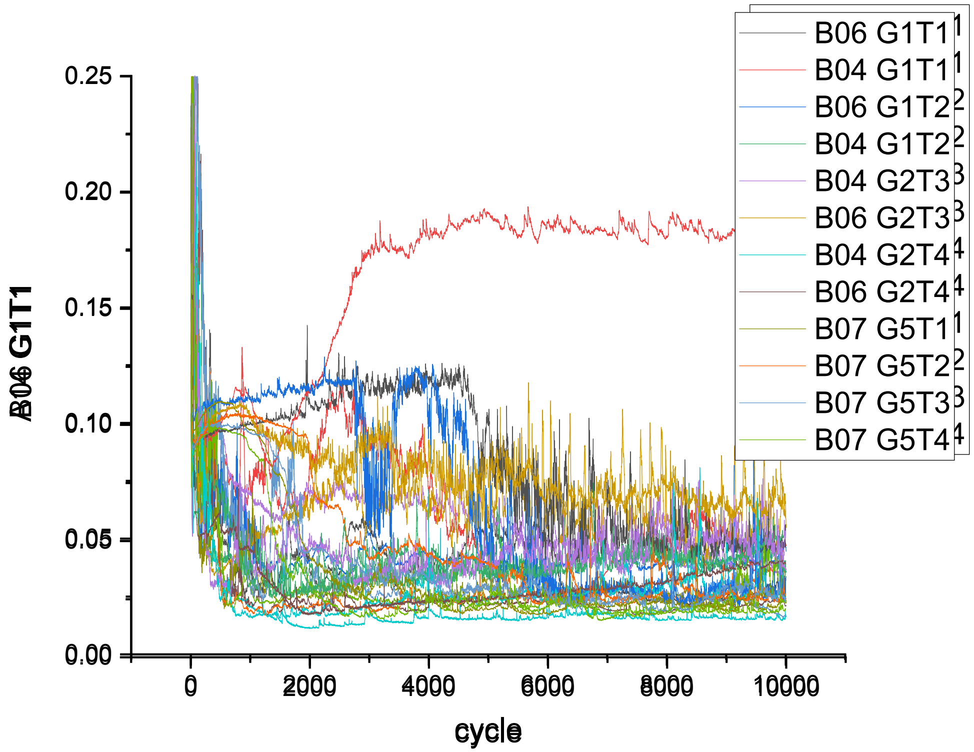


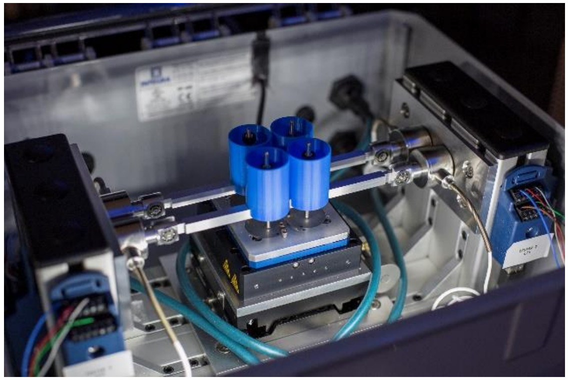


Test Specifics

- 5 DLC coatings; 4 samples each; 3-4 tests per sample @ 10,000 cycles
- 500 MPa (130 mN) max hertz contact pressure; 2mm stroke, 1 mm/s

HTT Case Study - Composition





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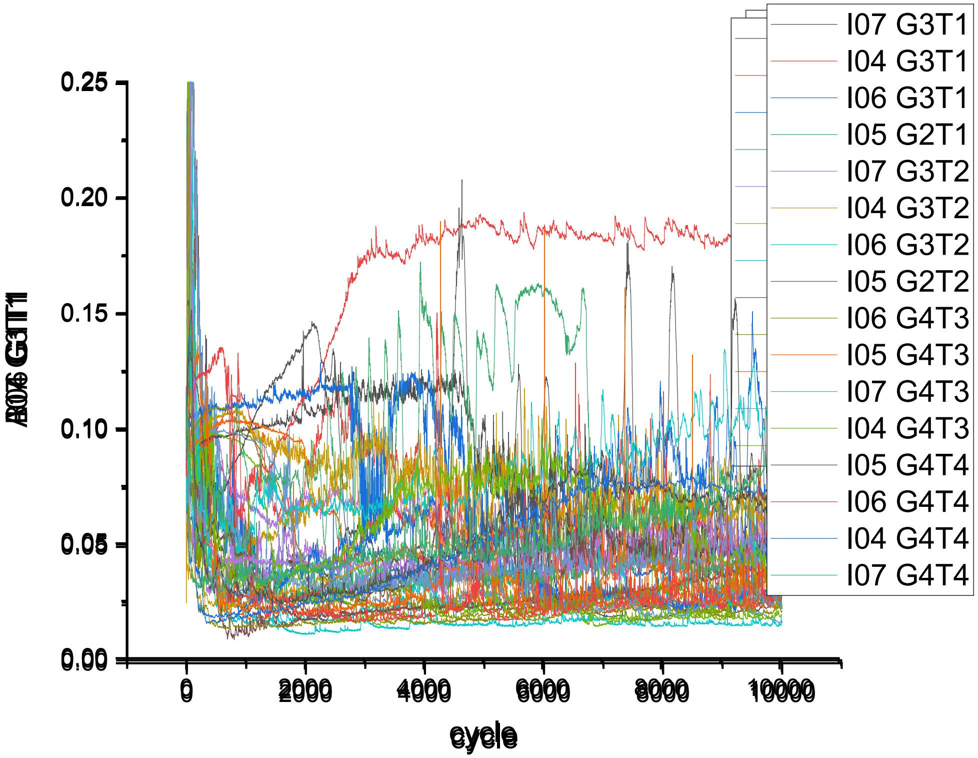

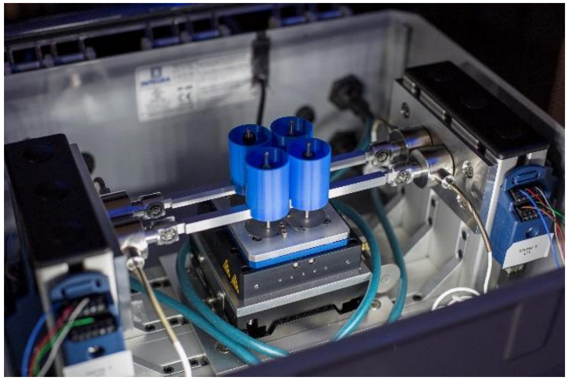

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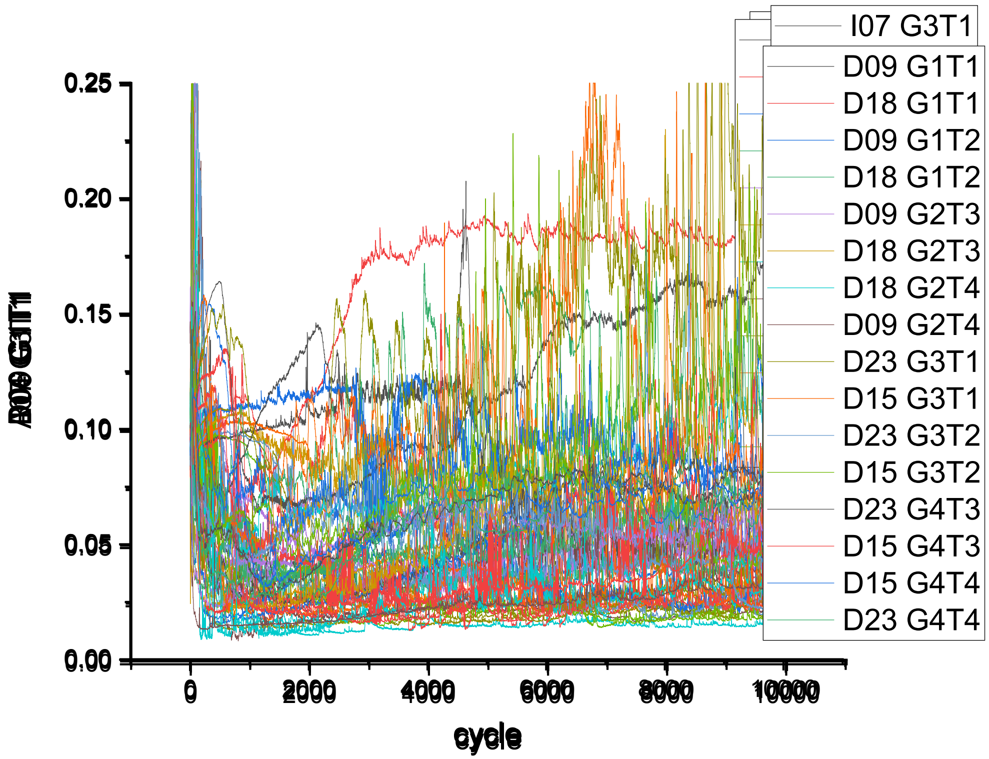

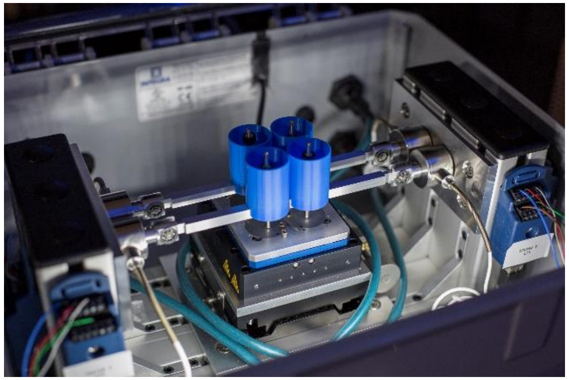

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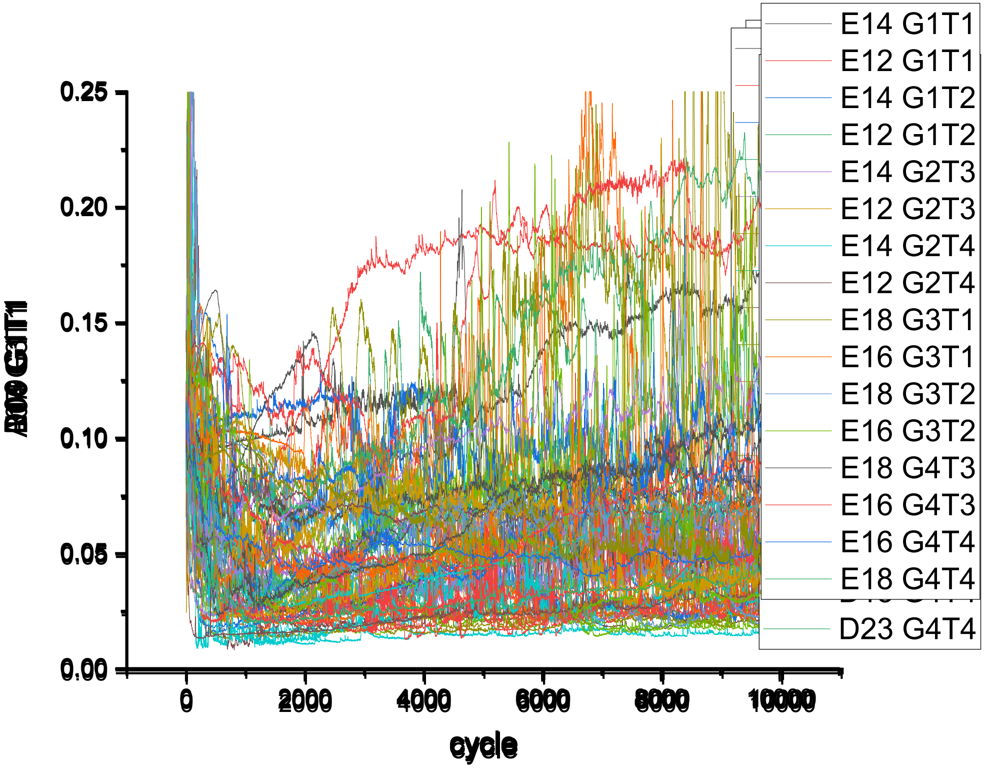
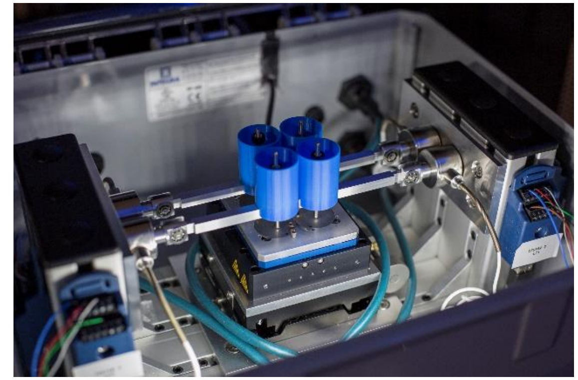
HTT Case Study - Composition



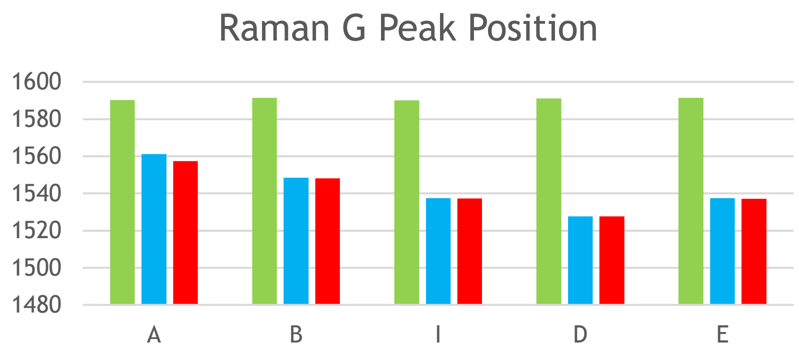


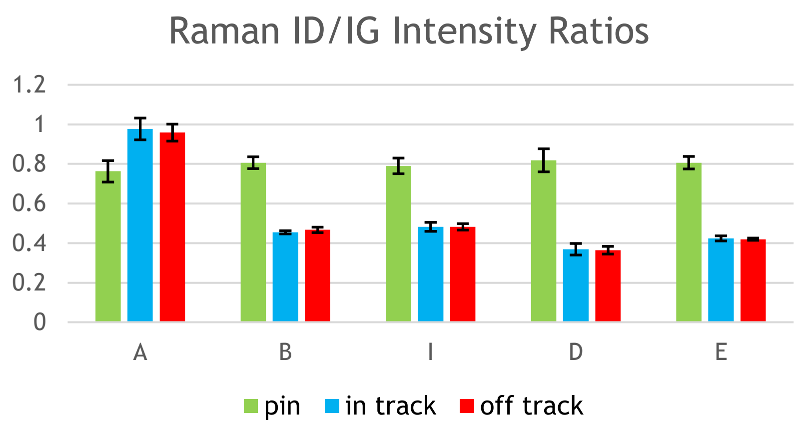
Test Specifics

- 5 DLC coatings; 4 samples each; 3-4 tests per sample @ 10,000 cycles
- 500 MPa (130 mN) max hertz contact pressure; 1mm stroke, 1 mm/s

~75 experiments running 10K cycles each, taking under 1 week to finish

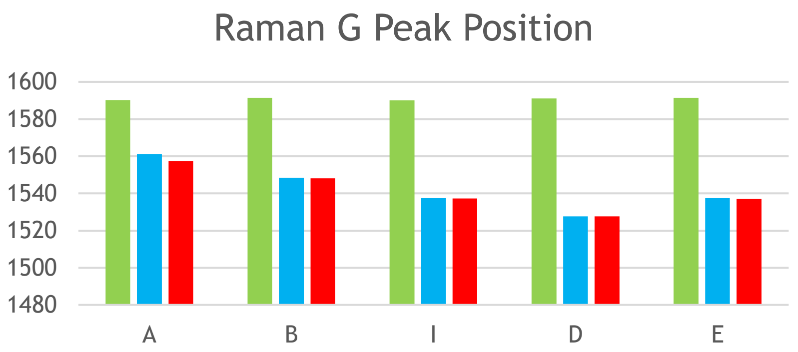
HTT Case Study - Results

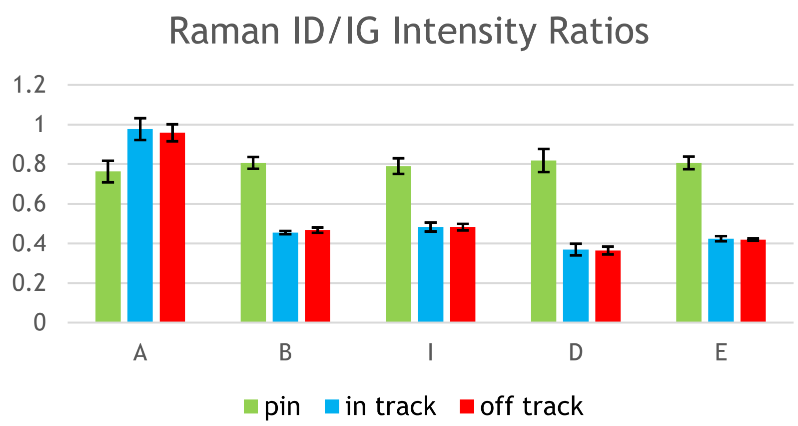


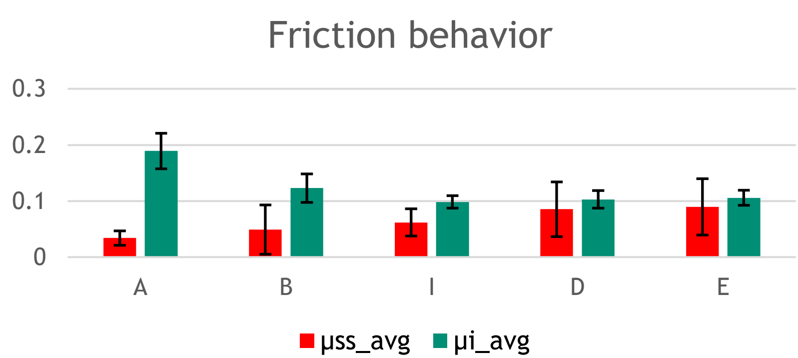


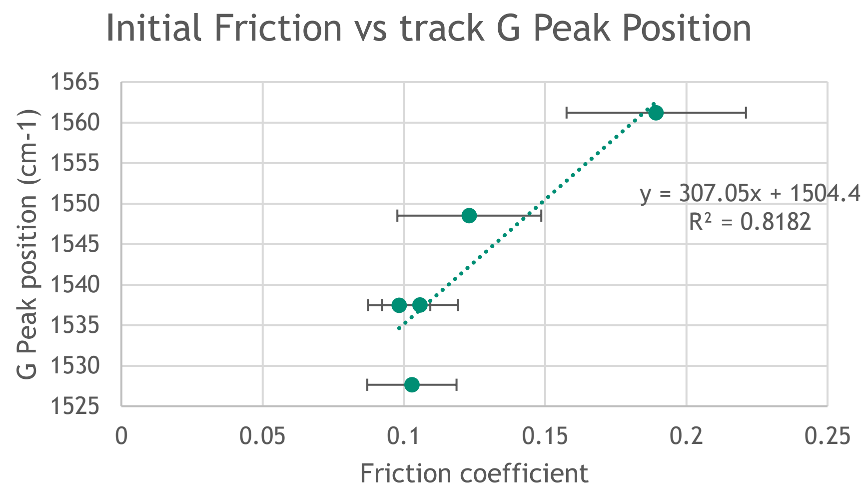
- In/out track Raman similar, exhibit differences unlike pin surface (unique from original film)

HTT Case Study - Results



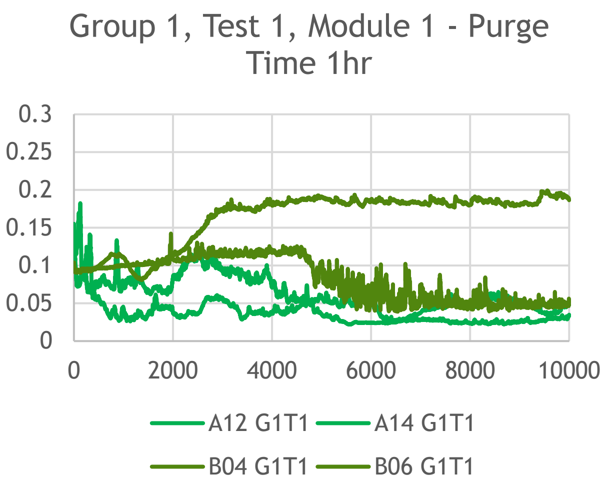


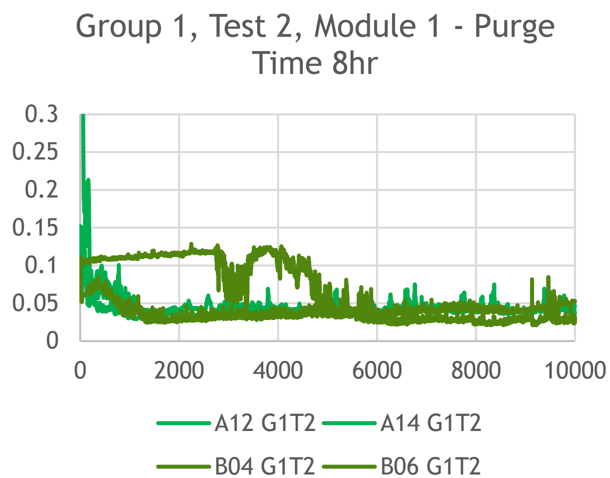


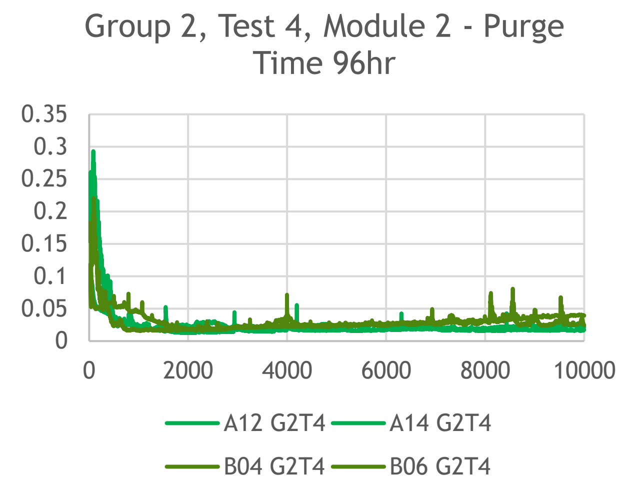


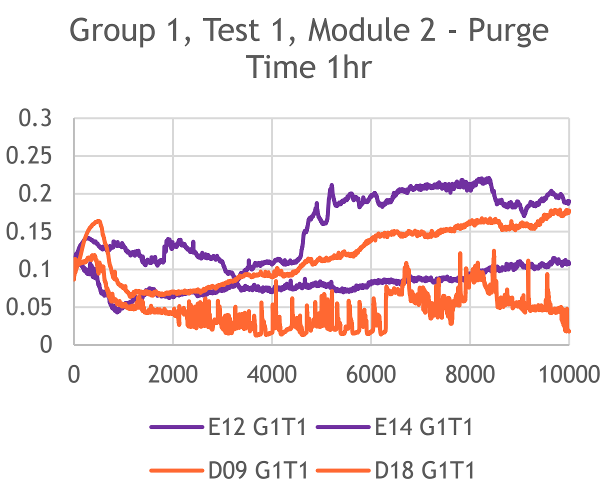
- In/out track Raman similar, exhibit differences unlike pin surface (unique from original film)
- Steady state and initial friction are inverse; correlate well with G peak pos & ID/IG of track

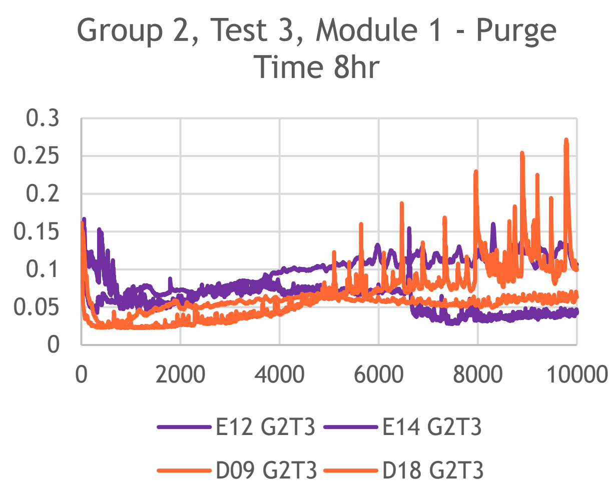
Effects of Purge Time on Variability

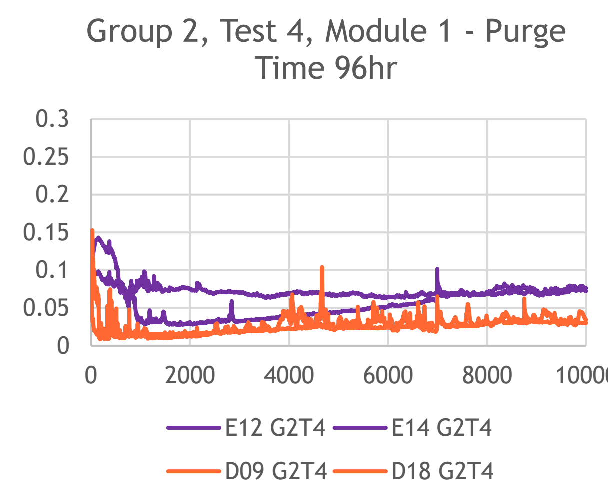








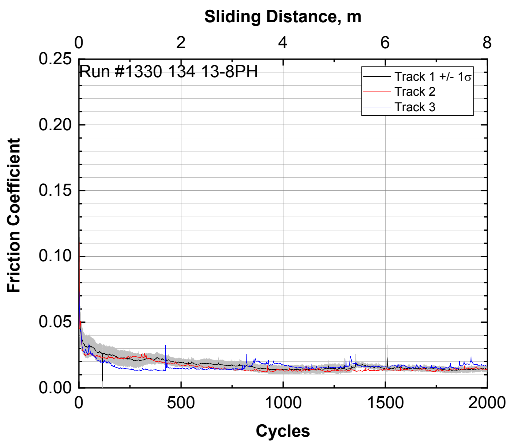




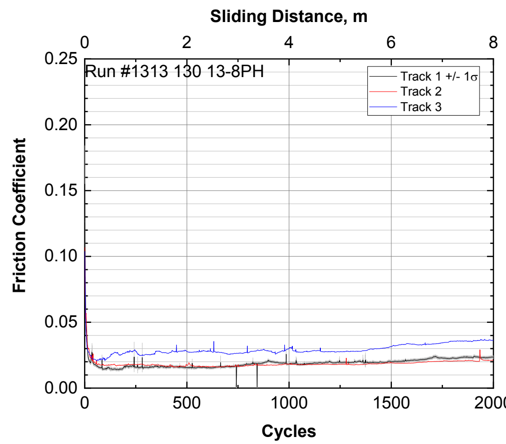
*** All labels are friction coefficient vs cycle**
**** 1 hour purges target <20 ppm O_2/H_2O**

Role of Surface Termination

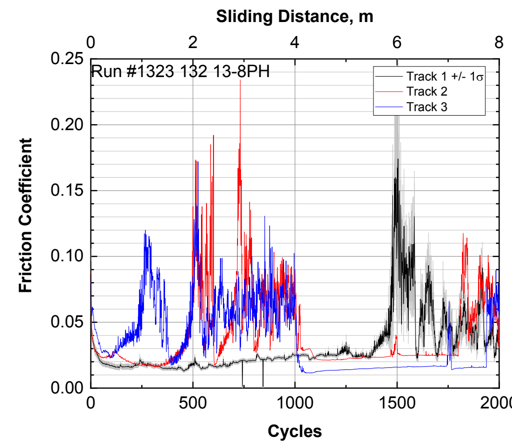
Baseline
(Run 5)



Ramp Down C6H12, Ramp Up Ar
(Run 2)



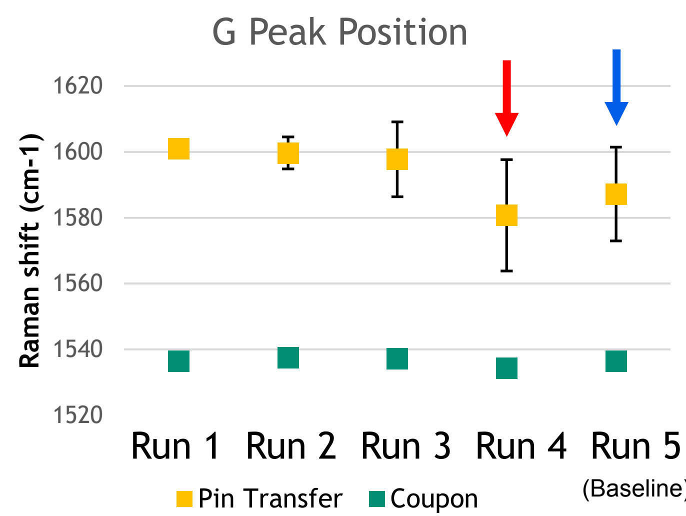
Ramp RF Power Down
(Run 4)

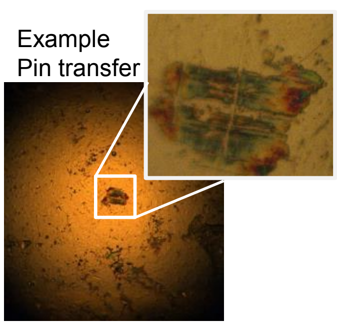


- All deposition runs same as baseline process, except for how the process ended
  - baseline involves shutting down gas precursors and RF power simultaneously
- The friction behavior was clearly modified by changing the shut-down process
  - there may be a decrease in run-in for some processes compared to baseline – more data (high throughput testing) will be used to evaluate significance

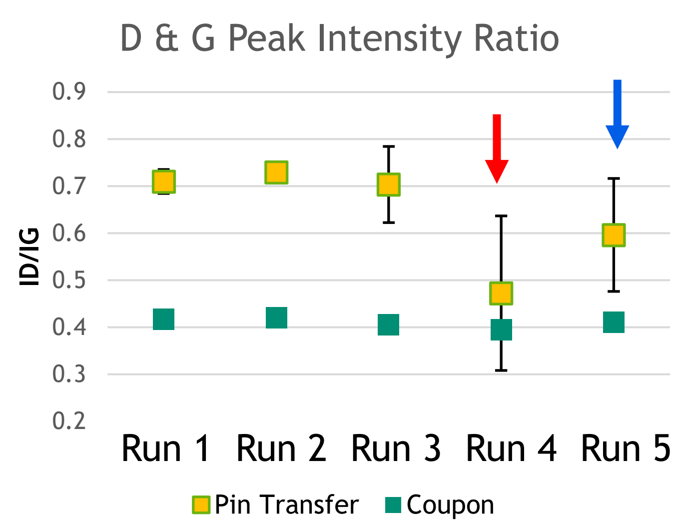
Surface Termination - Raman

- As expected, spectra from coupons remained similar
- Spectra taken on pin transfer exhibited differences from baseline (Run 5) in all cases:
  - RF power ramp down (Run 4) – lower ID/IG and G Peak position; also exhibited highly erratic friction behavior
  - Runs 1,2,3 – higher ID/IG and G Peak position; exhibited similar friction behavior to baseline
- Results suggest that changes in friction behavior are linked to process changes that alter interfacial chemistry





Run ID	Process
1	Ar ion etch
2	Ramp Down C_6H_{12}, Ramp Up Ar
3	Ramp Down C_6H_{12}
4	Ramp RF Power Down
5	Baseline Process

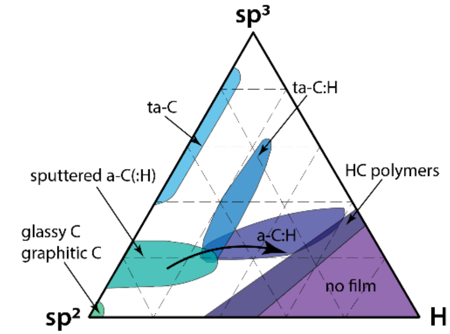


Key Take-Aways

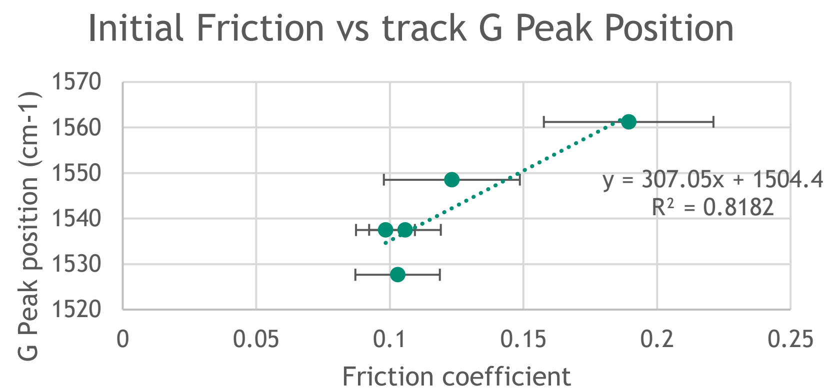
- Confirmed link between composition (via Raman/HFTF) and friction behavior for different vendors

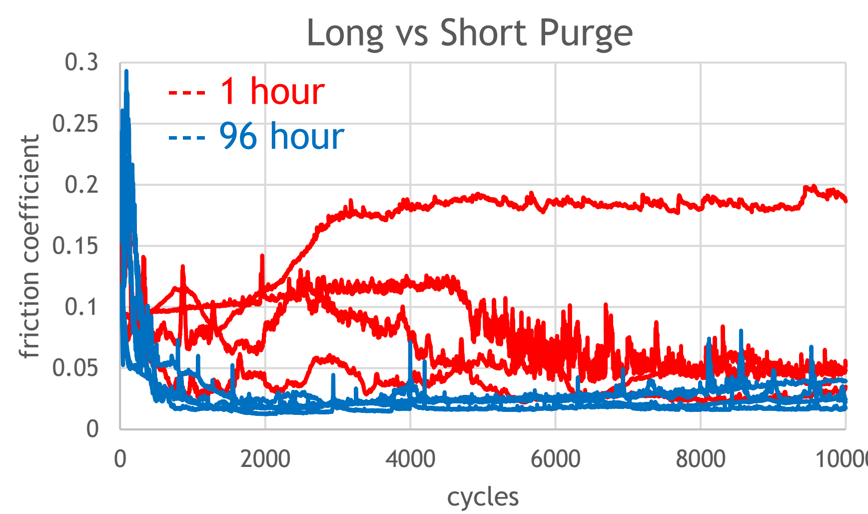
- Surface termination simple route to change friction behavior

- Can utilize these relationships to design better coatings gear to run-in



A.C. Ferrari, *PRB.* 2000





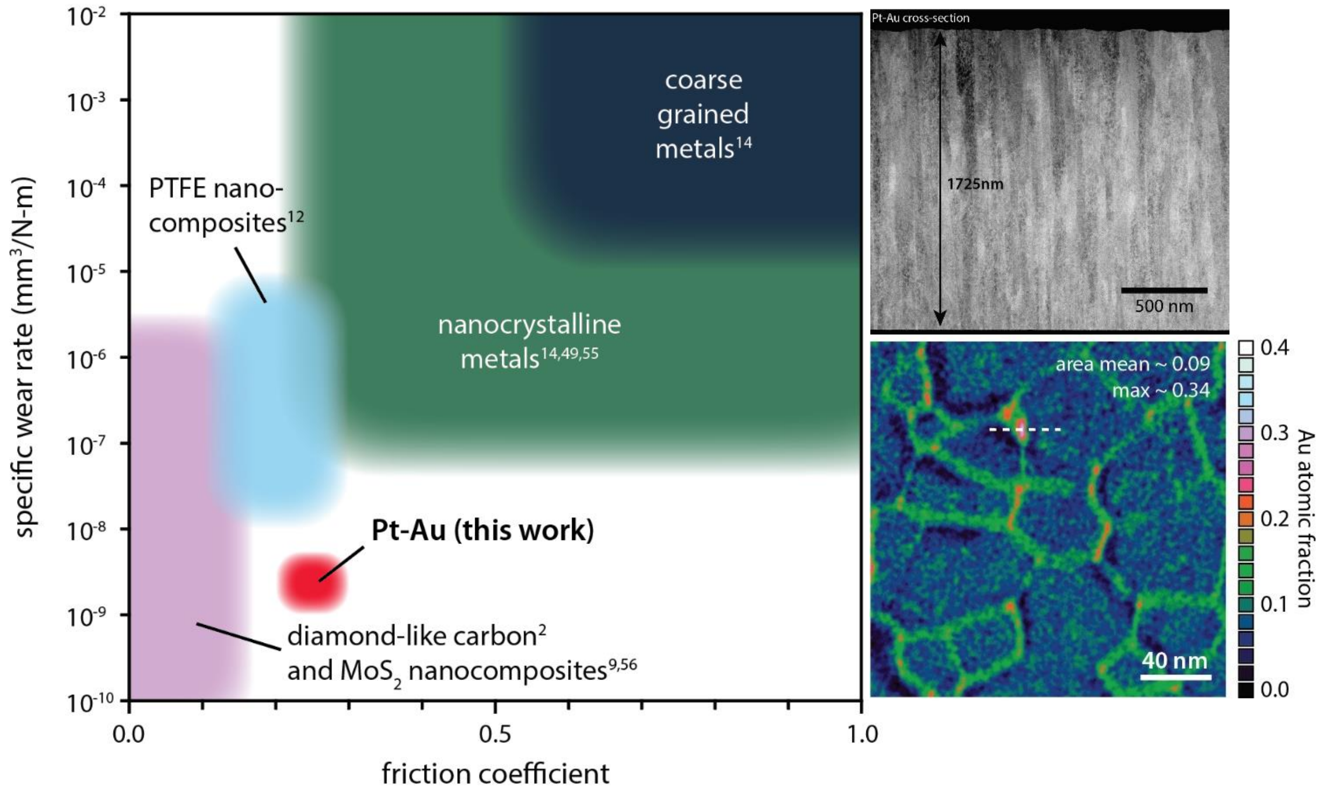
Ongoing / Future Work

- Additional In situ testing (Raman/NEXAFS) for cycle resolved changes at surface

- What factors during purge change (ambient RGA)

Self-lubricating in situ carbon films **(SLIC)**

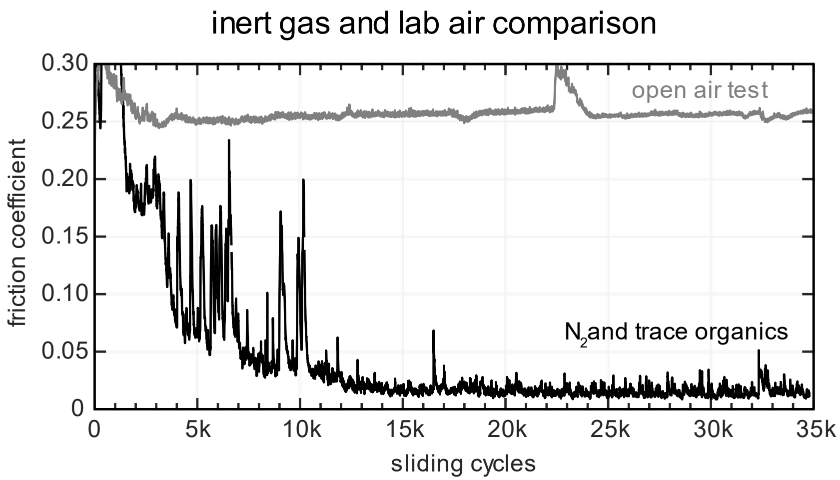
Discovery: Ultra-Low Wear Pt-Au



- Byproduct of LDRD on development of stable, ultra-nanocrystalline alloys
- Most tests run in air – needed to check performance in inert environments…

Curry et al, Adv. Mater. 2018

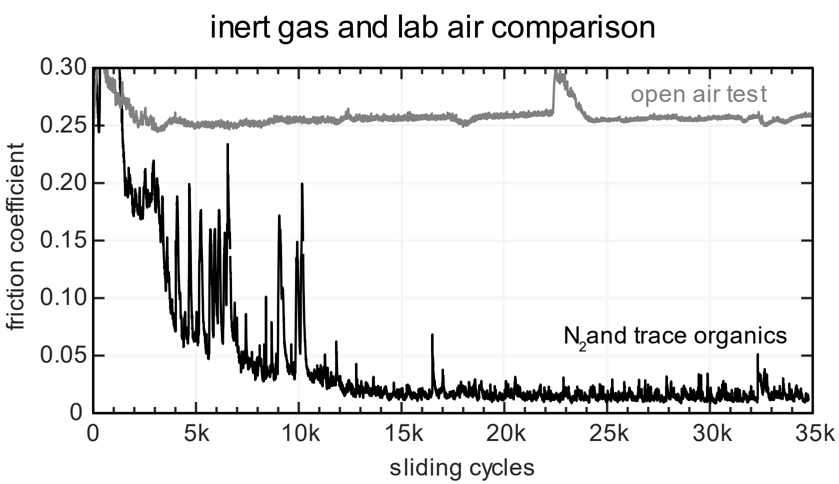
Unexpected Tribocatalysis

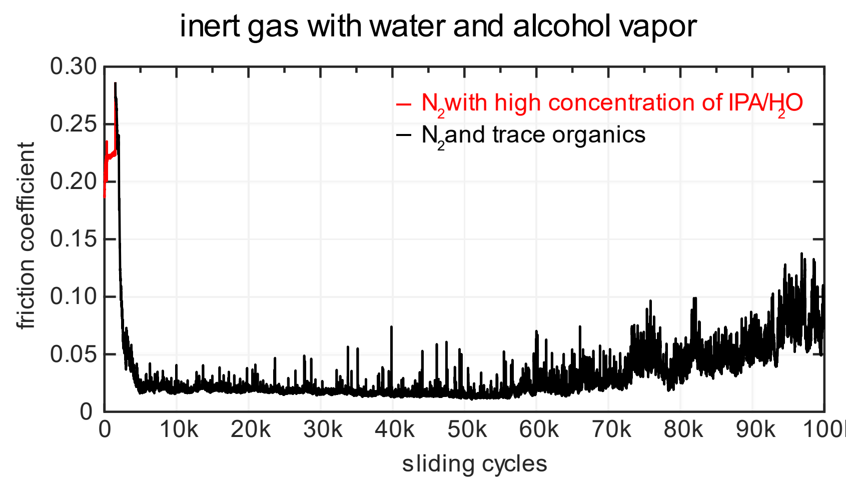


- Testing in inert environments lowers friction?

Argibay et al, Carbon 2018

Unexpected Tribocatalysis

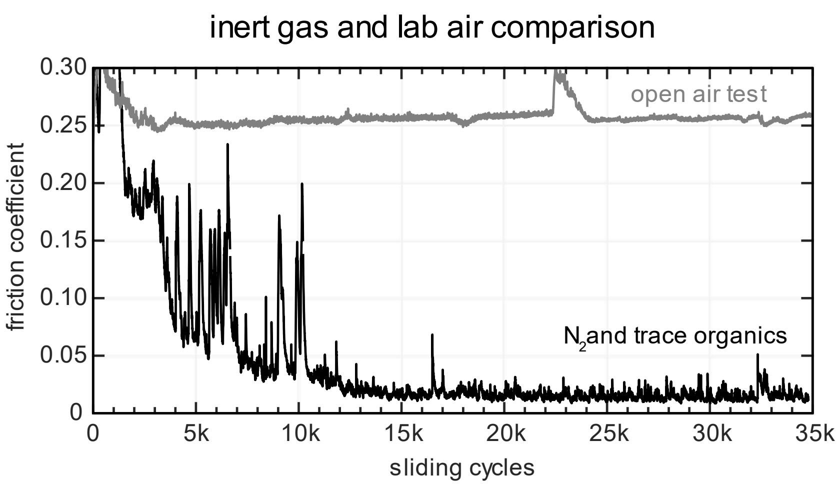


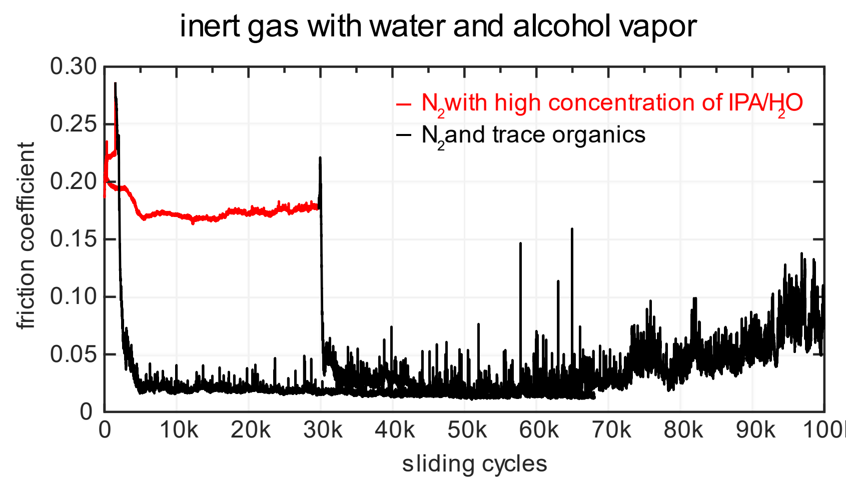


- Testing in inert environments lowers friction?
- Priming the enclosure with hydrated IPA accelerates drop…

Argibay et al, Carbon 2018

Unexpected Tribocatalysis

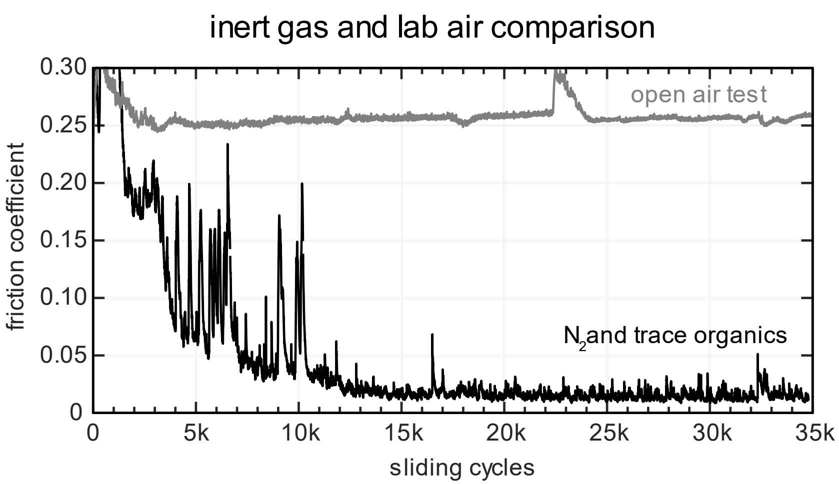


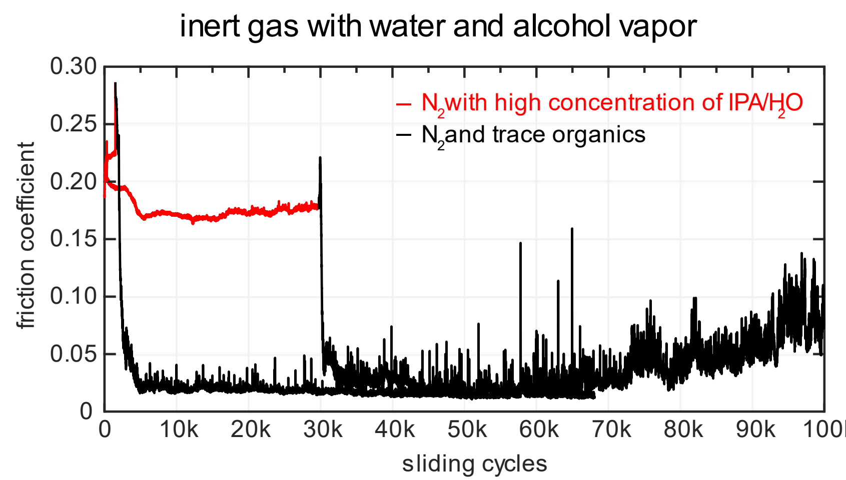


- Testing in inert environments lowers friction?
- Priming the enclosure with hydrated IPA accelerates drop… and prolongs it

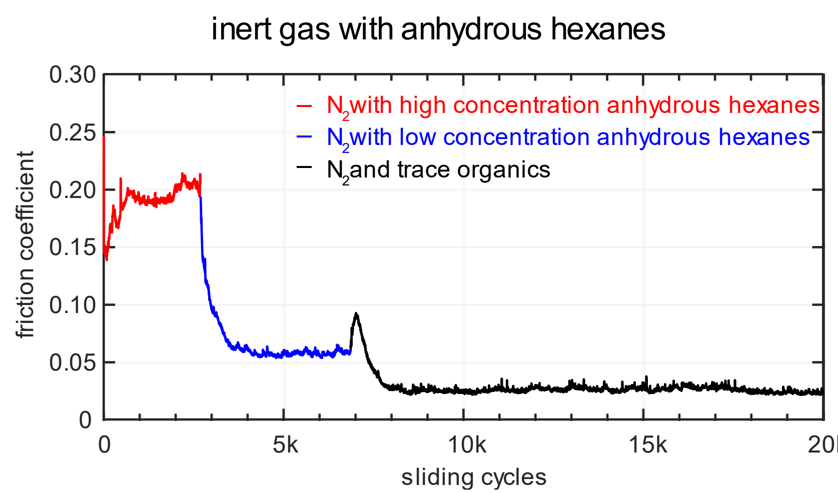
Argibay et al, Carbon 2018

Unexpected Tribocatalysis



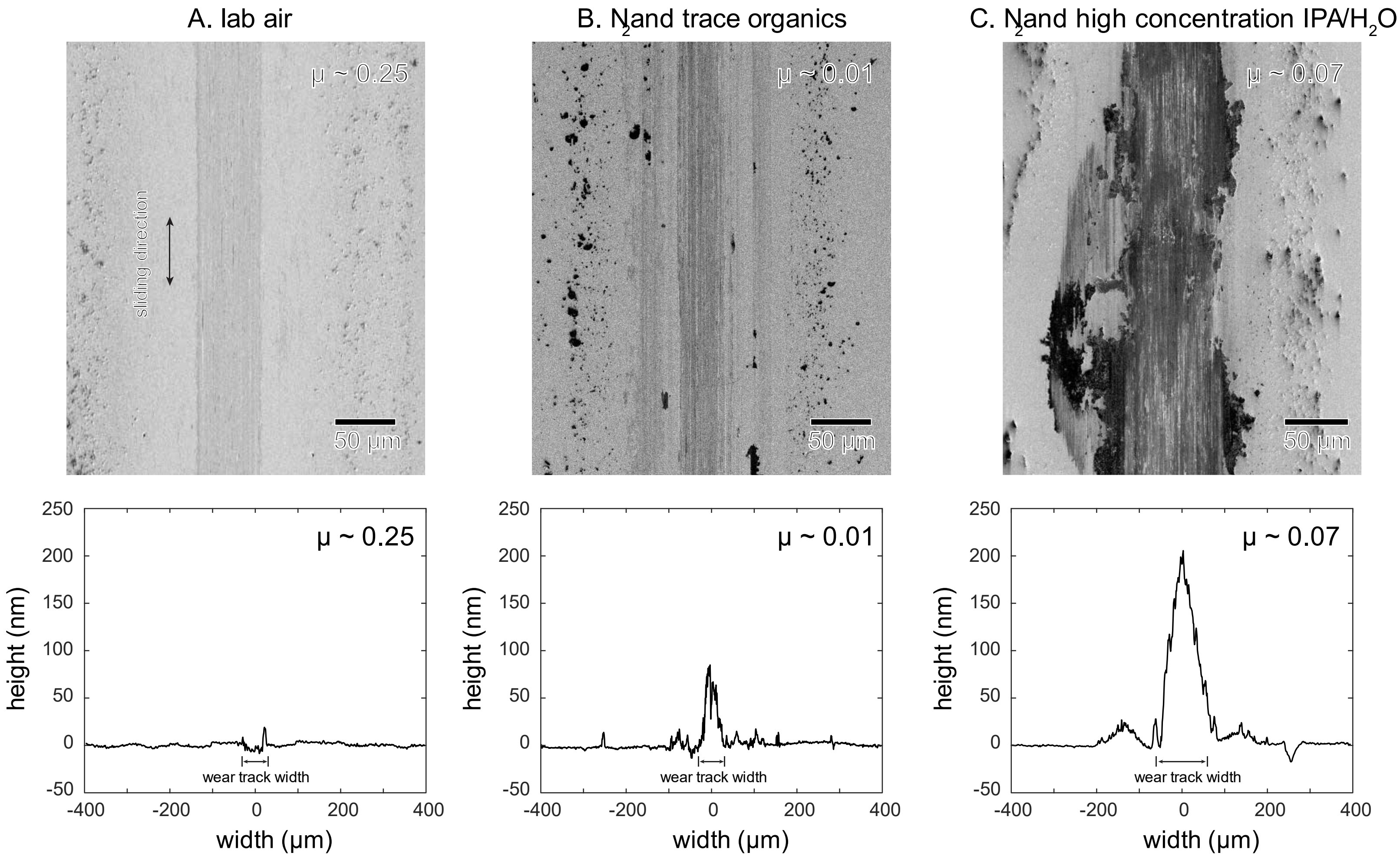


- Testing in inert environments lowers friction?
- Priming the enclosure with hydrated IPA accelerates drop… and prolongs it
- Any amount of anhydrous hexanes increased friction, with higher/lower friction at higher/lower concentrations
- Unclear what role water/oxygen play



Argibay et al, Carbon 2018

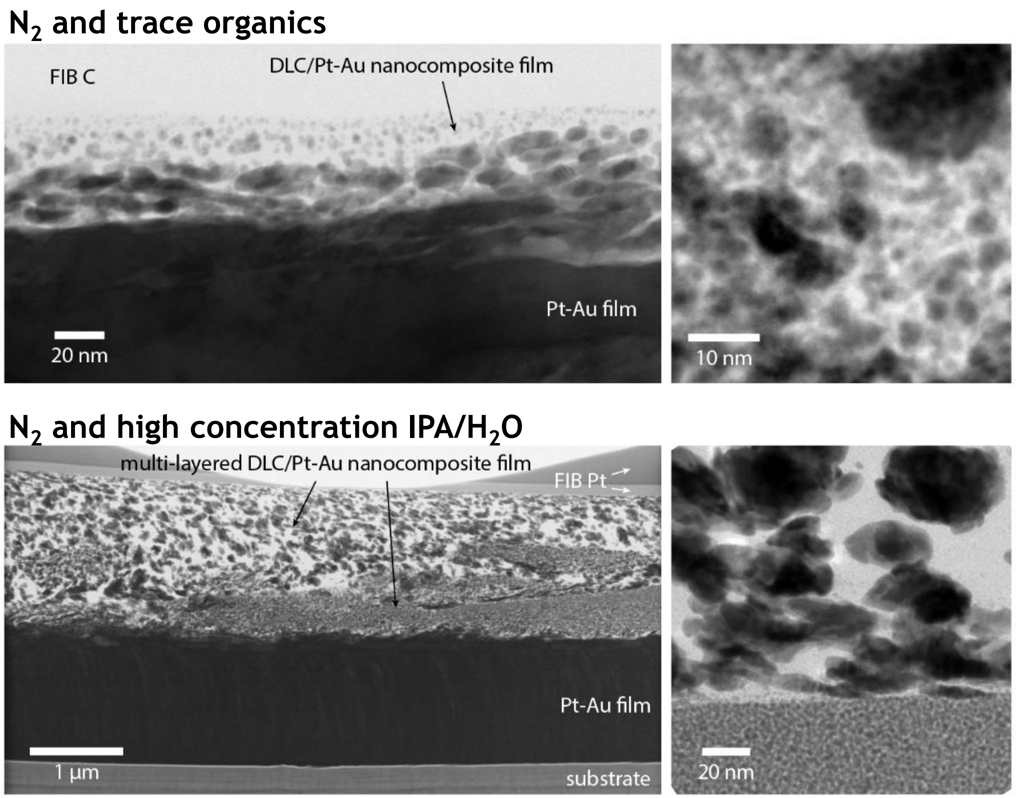
Accumulation is Key

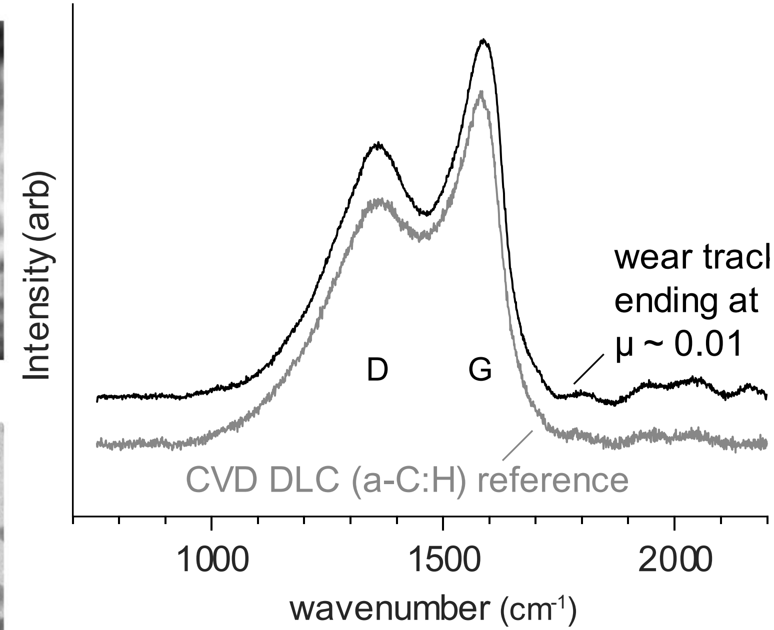


- Concentration also affects film growth in wear scar
- Highest concentrations produces thick films unable to reach low friction state

Argibay et al, Carbon 2018

A Tribo-polymeric Nanocomposite





- Films are actually composite of Pt-Au nanoparticles and DLC-like carbon, confirmed by TEM & Raman
- High concentrations exhibit phases of larger, less mixed/layered particles, possibly limiting mixing & Pt interaction at surface

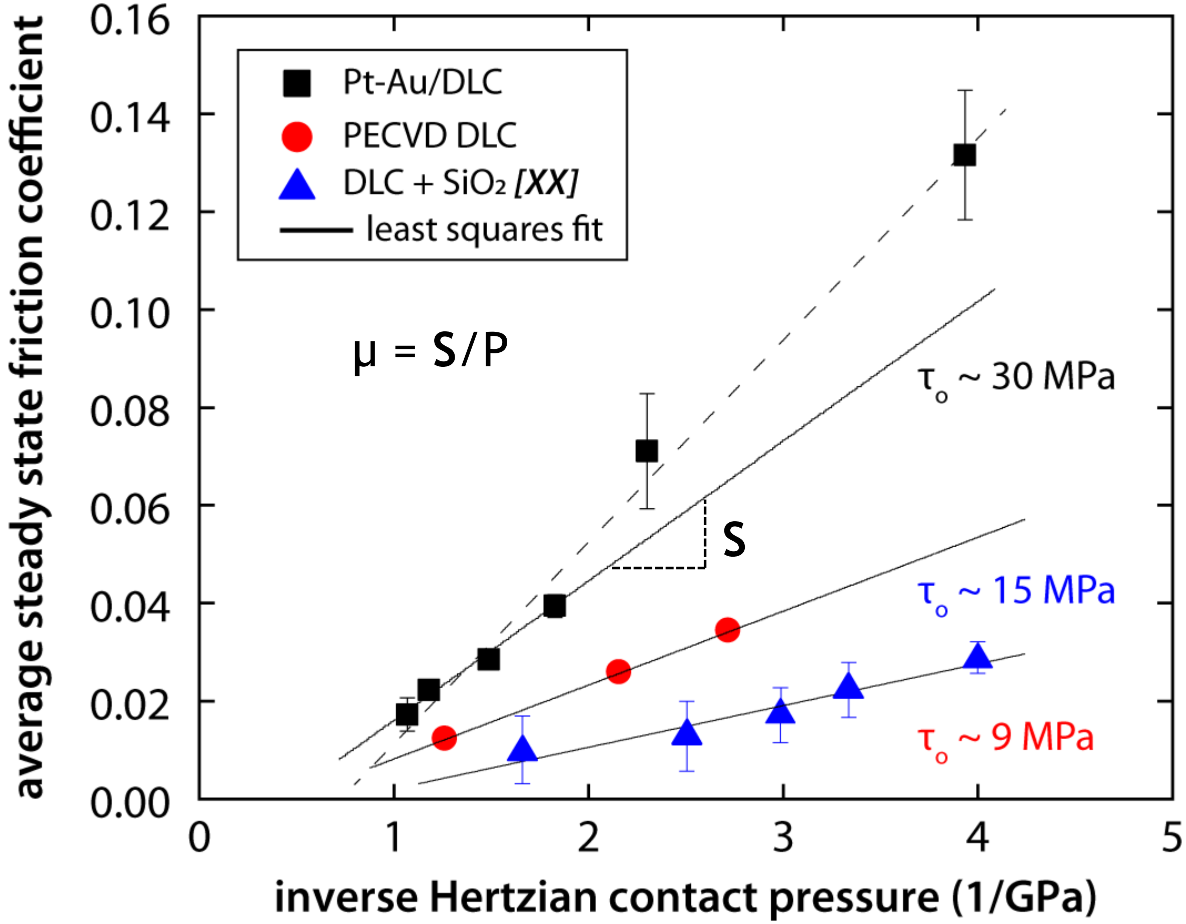
Argibay et al, Carbon 2018

Qualifying Shear Strength

- Can derive shear strengths from Hertzian contact model

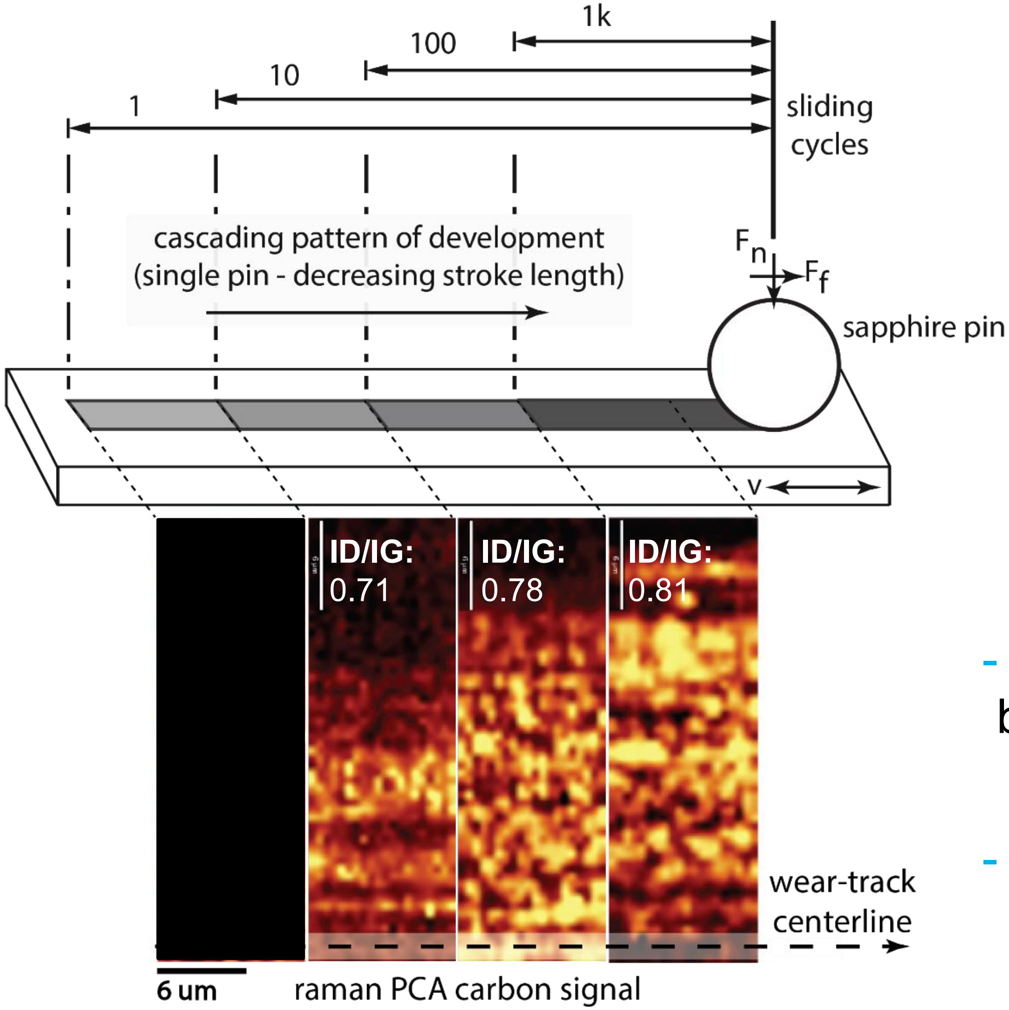
- Shear strength comparable to commercially available

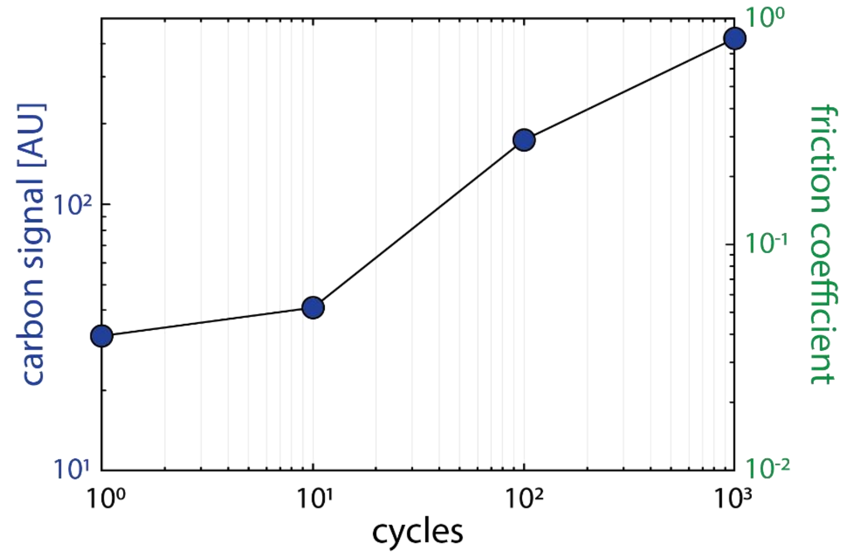
- Discrepancies may be due to composite nature of film and lower hydrogenation (20 vs 40%)



(Scharf, 2013, J. Matl. Sci.)

Time Dependent Formation

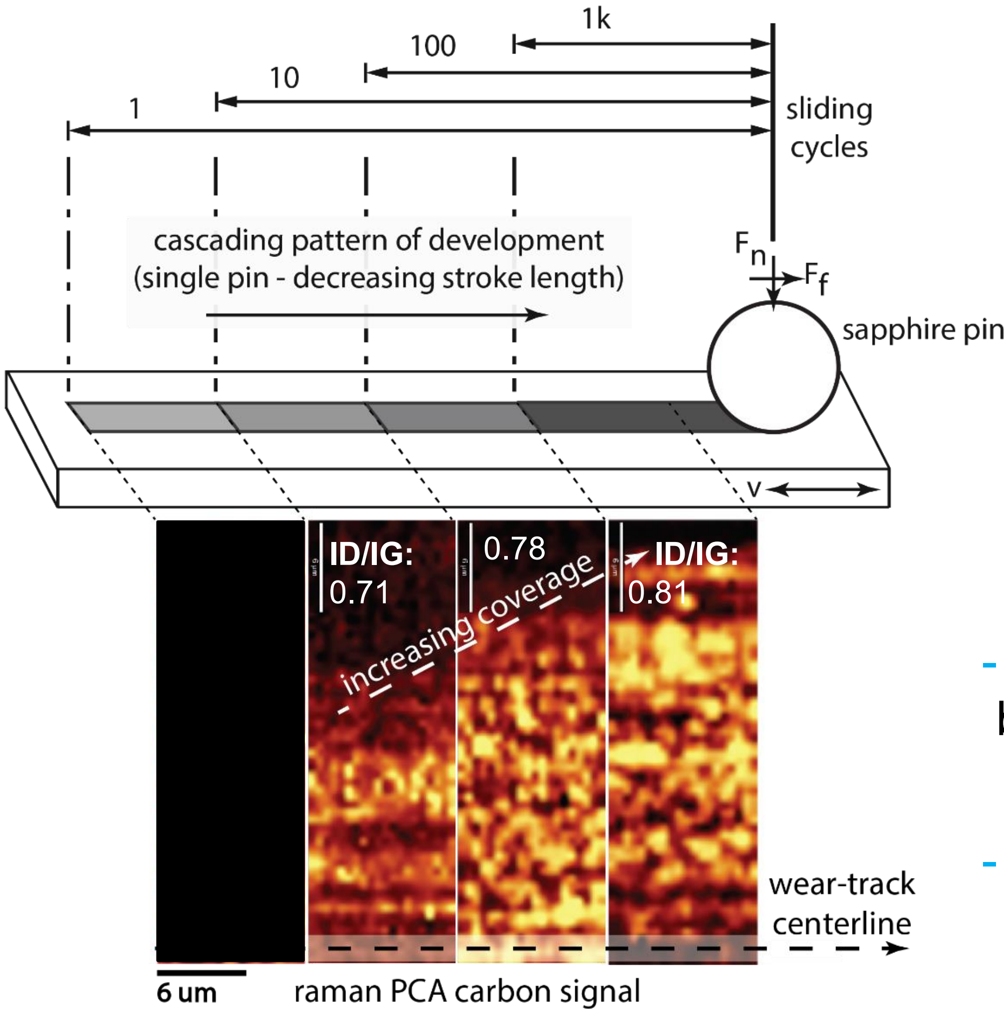


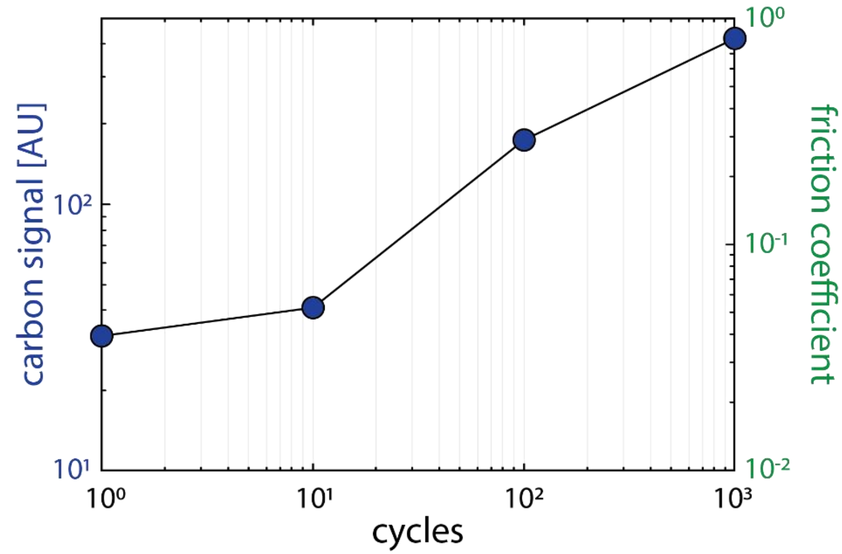


- Stripe tests elucidate time dependent behaviors
- Increasing cycle count leads to:
  - Stronger carbon signals in wear track

Jones, M. R. *et al.*, *JOM* (2021)

Time Dependent Formation

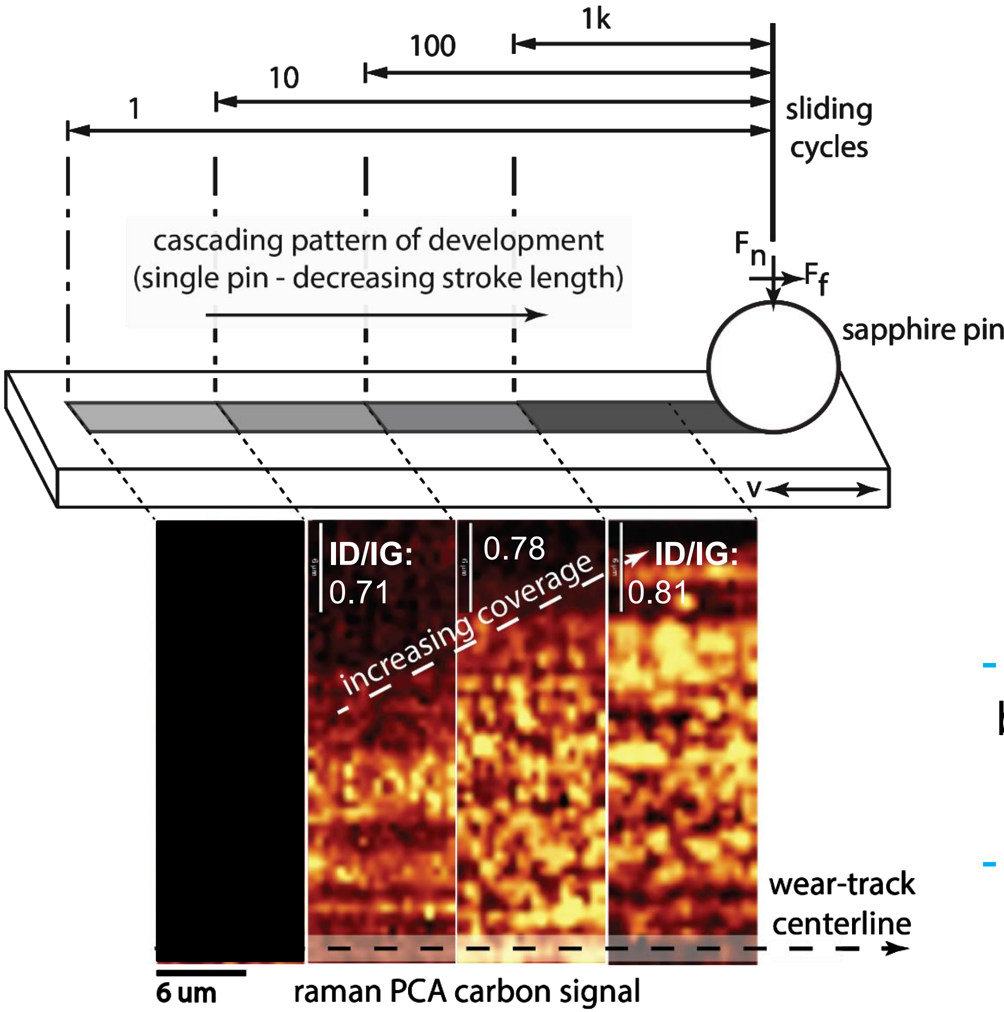


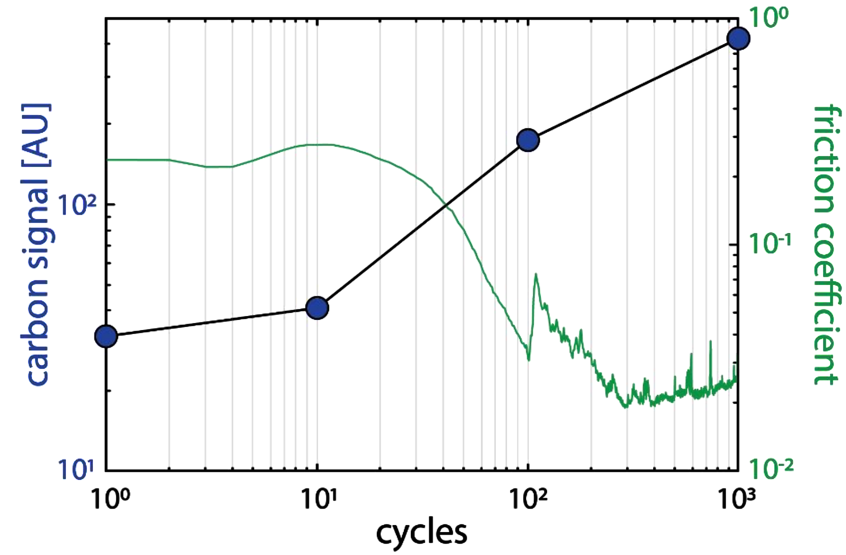


- Stripe tests elucidate time dependent behaviors
- Increasing cycle count leads to:
  - Stronger carbon signals in wear track
  - Higher coverage in wear scar

Jones, M. R. *et al.*, *JOM* (2021)

Time Dependent Formation



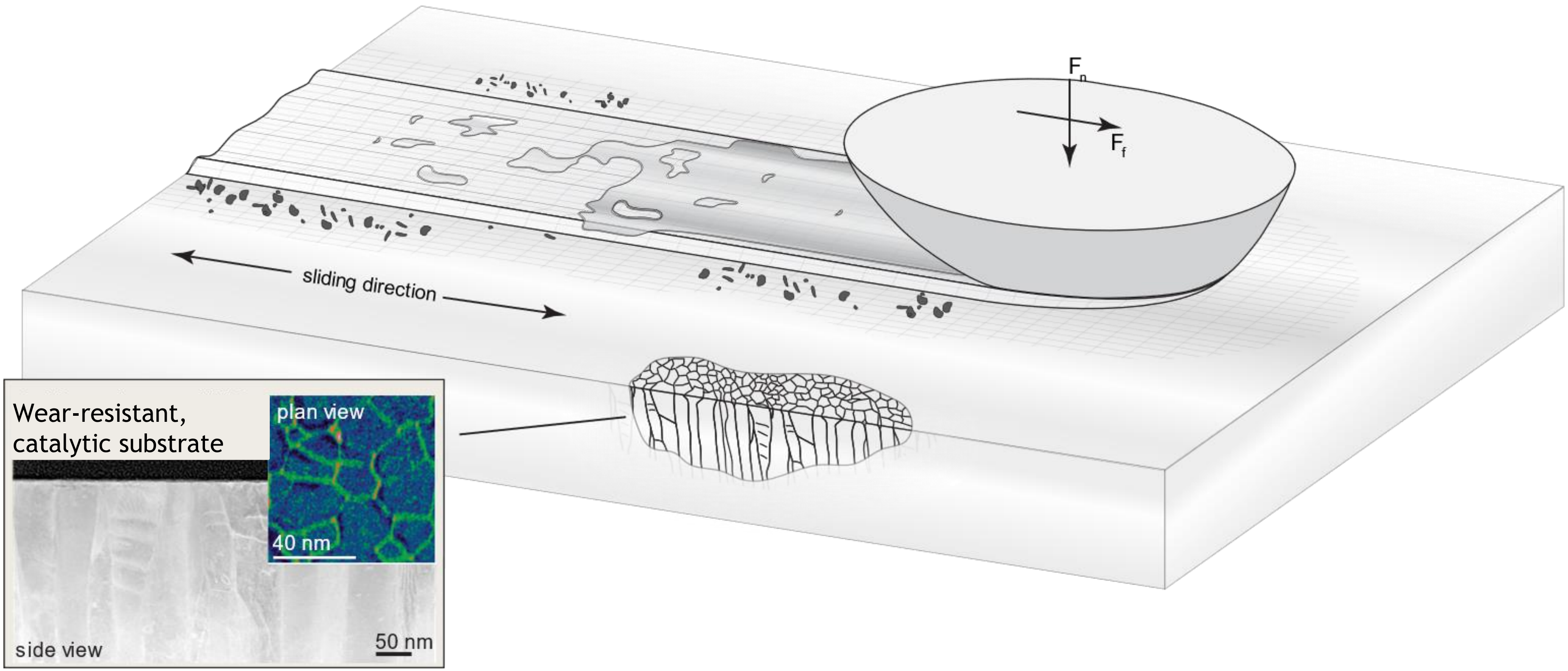


- Stripe tests elucidate time dependent behaviors

- Increasing cycle count leads to:
  - Stronger carbon signals in wear track
  - Higher coverage in wear scar
  - Decreasing friction coefficient

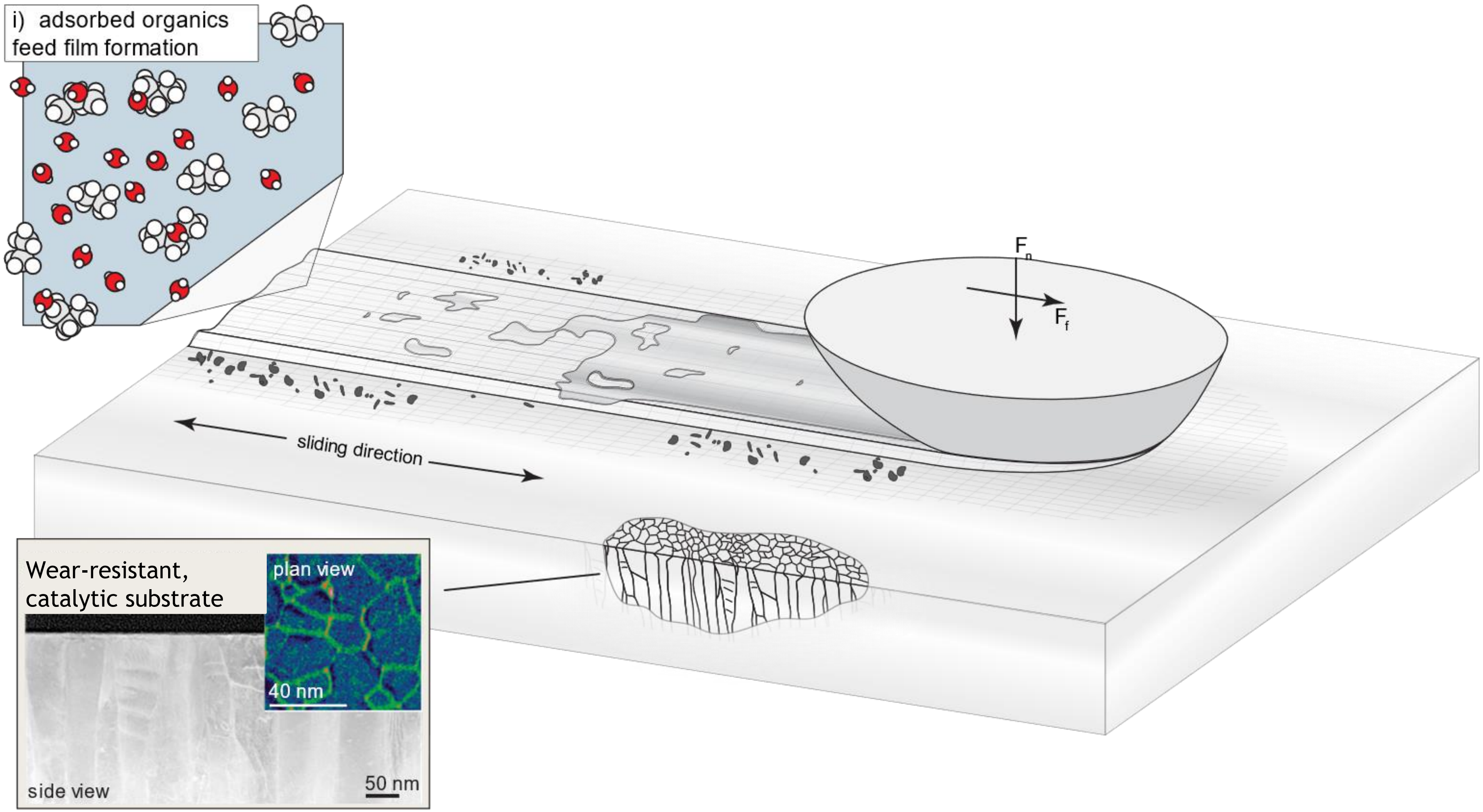
Jones, M. R. *et al.*, *JOM* (2021)

Mechanisms of Formation



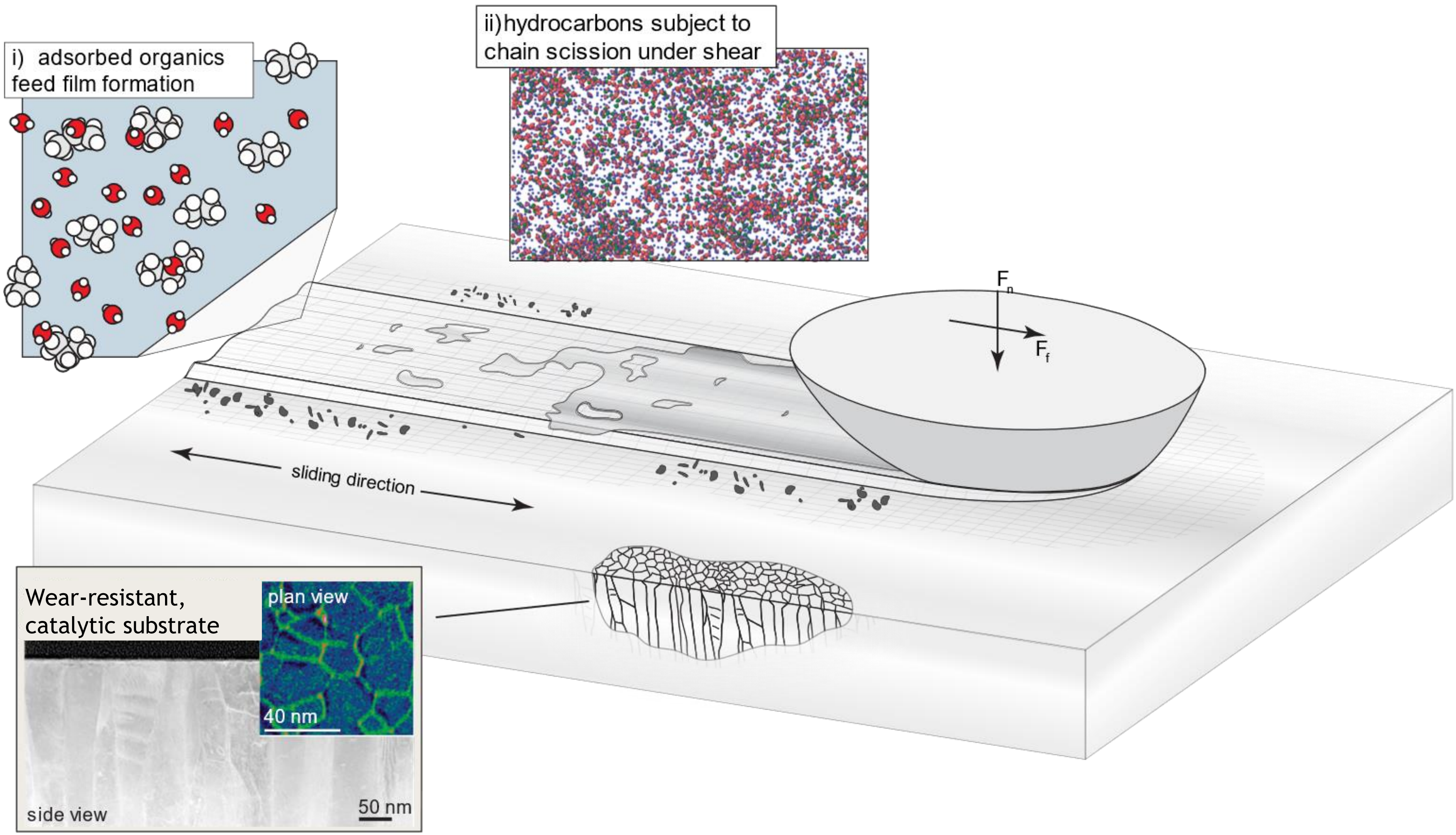
Argibay et al, Carbon 2018

Mechanisms of Formation



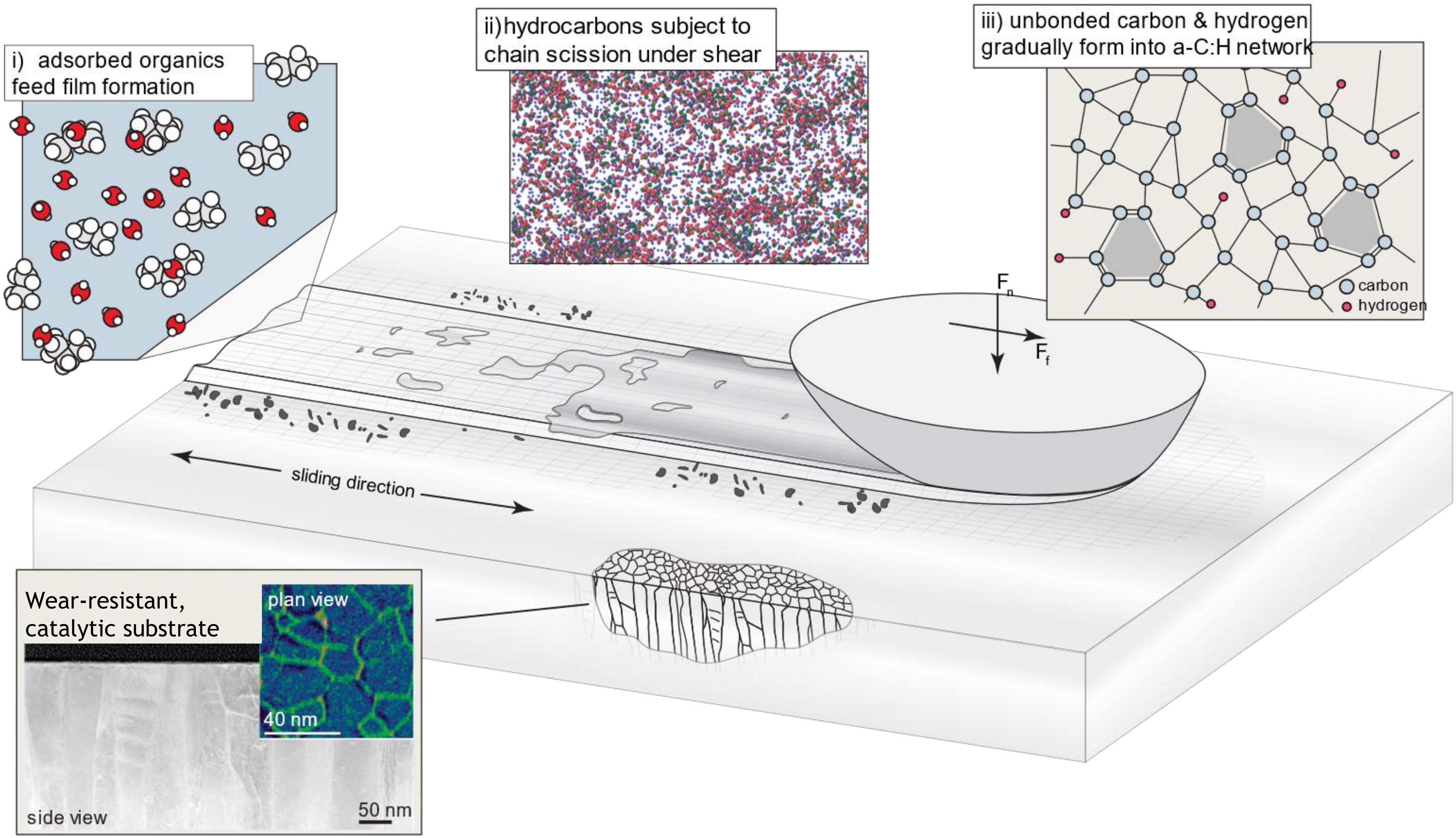
Argibay et al, Carbon 2018

Mechanisms of Formation



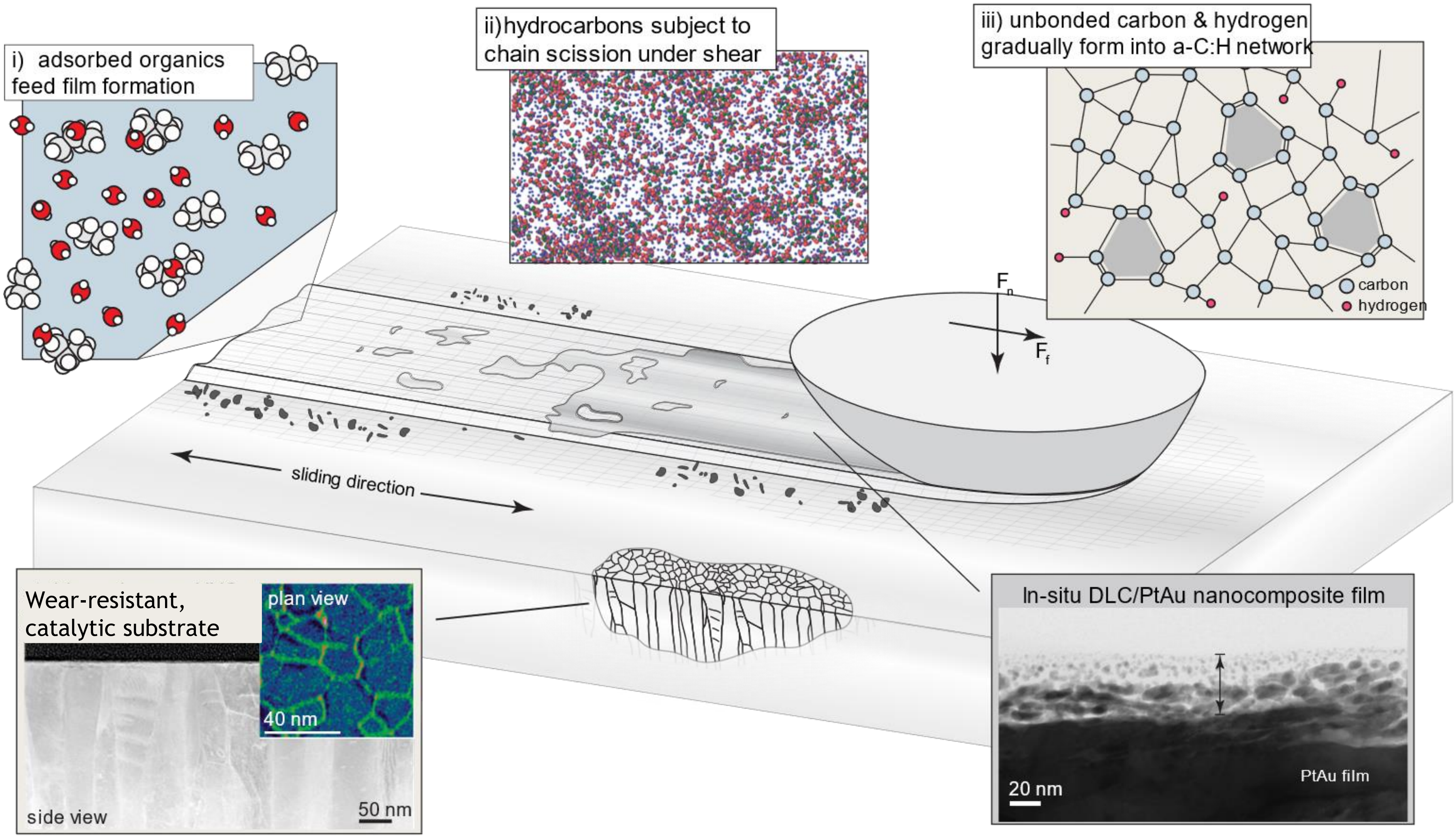
Argibay et al, Carbon 2018

Mechanisms of Formation



Argibay et al, Carbon 2018

Mechanisms of Formation



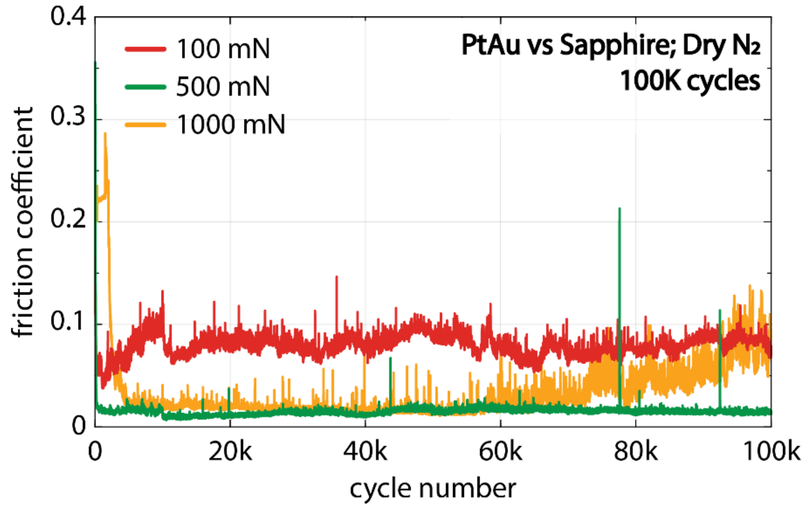
Argibay et al, Carbon 2018

Key Take-Aways

Outcomes

- Trace concentrations of hydrocarbons enable formation of SLIC, a DLC-like tribofilm

- SLIC films exhibit stress/cycle dependent formation (& temperature in ethanol!)



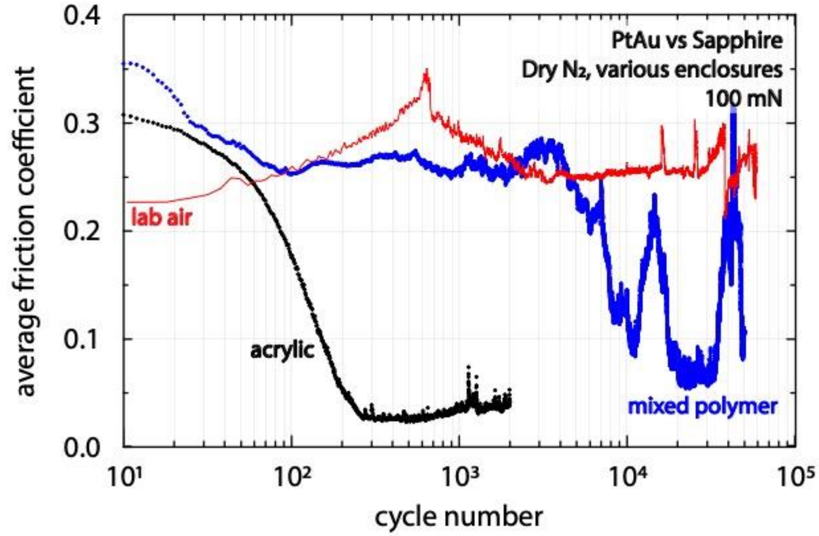
Ongoing / Future Work

- Understanding of underlying mechanisms behind competing stress, time & wear

- What do the resulting microstructures give us in terms of friction, wear or conductivity?

- What is the ideal composition or concentration of species in the environment for formation?

- Can we utilize as deposition method?



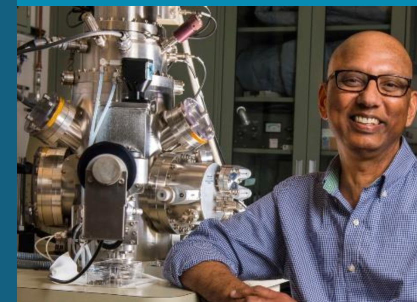
Acknowledgements

Sandia

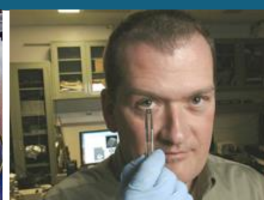
- **Michael Dugger** MoS_2 Aging studies
- **Michael Chandross, Scott Bobbitt** MoS_2 MD/DFT
- **Frank DelRio** AFM/KPFM on MoS_2
- **Brendan Nation, JW Johnson, Morgan Jones, Brian Wisler** Experimental Support
- **Taisuke Ohta** XPEEM defect characterization on MoS_2
- **Prof. Thomas Beechem** DLC characterization, in-situ Raman capability development

- **Florida State University Prof Brandon Krick, Tomas Babuska** in vacuo oxidation studies of MoS_2
- **UT Austin Prof Filippo Mangolini, Robert Chrostowski** NEXAFS/APPES experiments on MoS_2/DLC

Sandia National Laboratories



Thank You



Questions?

U.S. DEPARTMENT OF ENERGY NNSA

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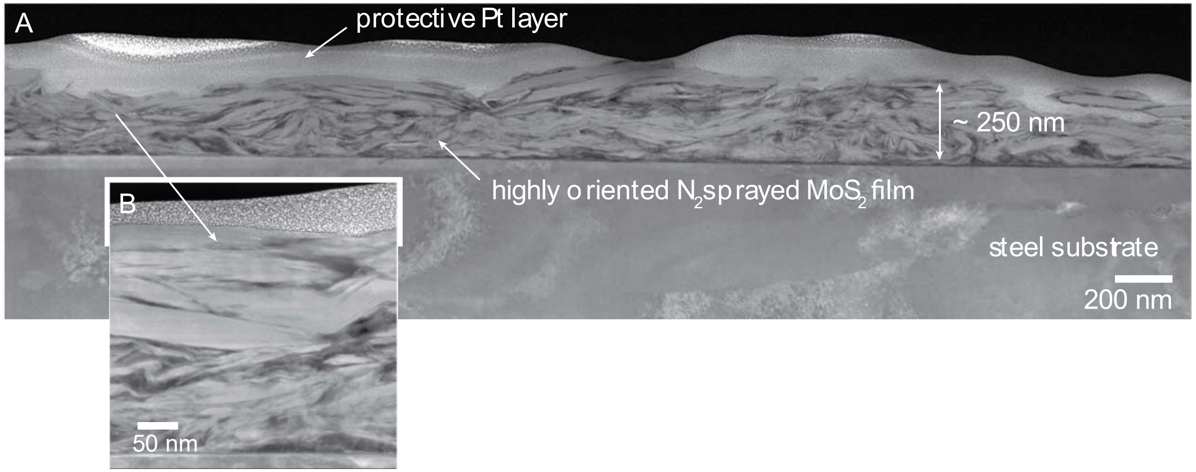
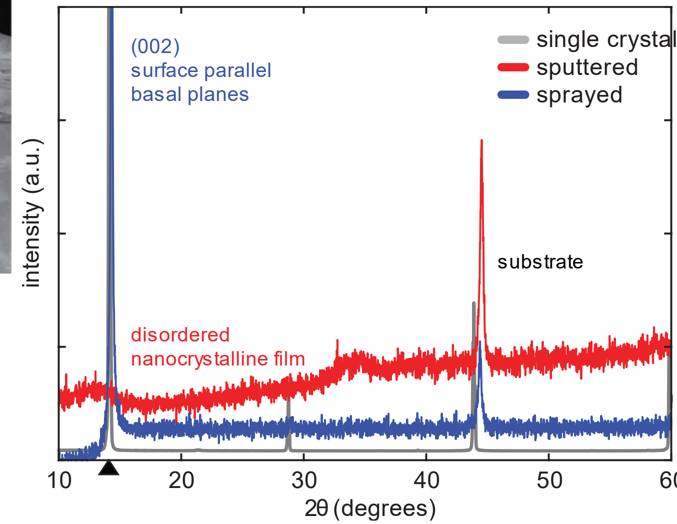
APPENDIX

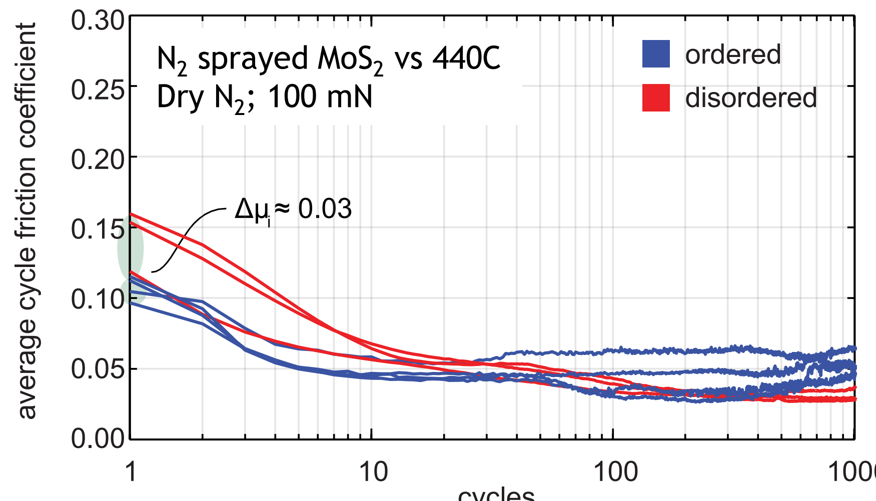
Solution? Start Simple



highly ordered (N2 sprayed) [1]

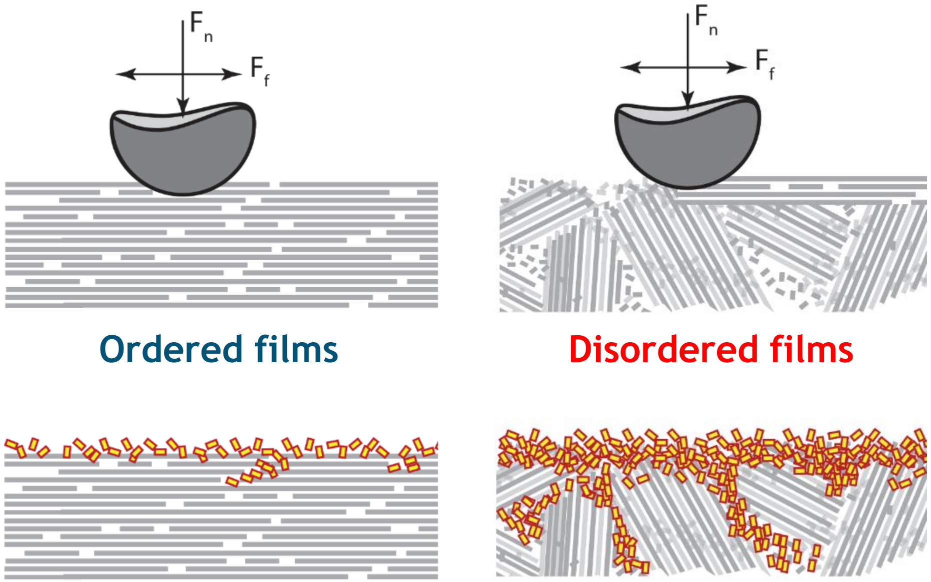


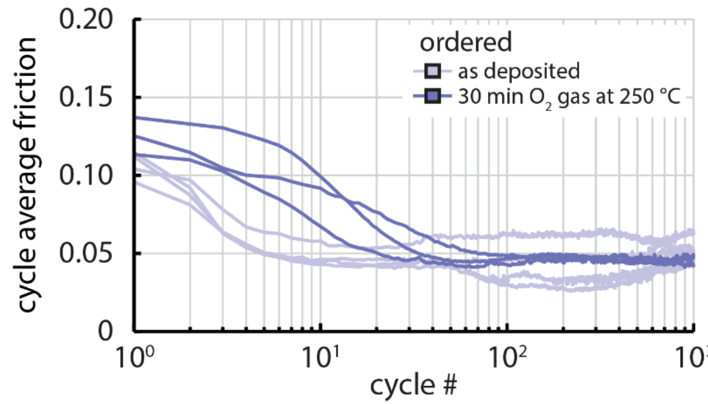
- To make things simple, we focused on ordered, impinged films
  - Blast N_2/MoS_2 onto surface to get films close to basally oriented as deposited
  - Exhibit lower initial friction coefficients
  - Ordering may help prevent degradation

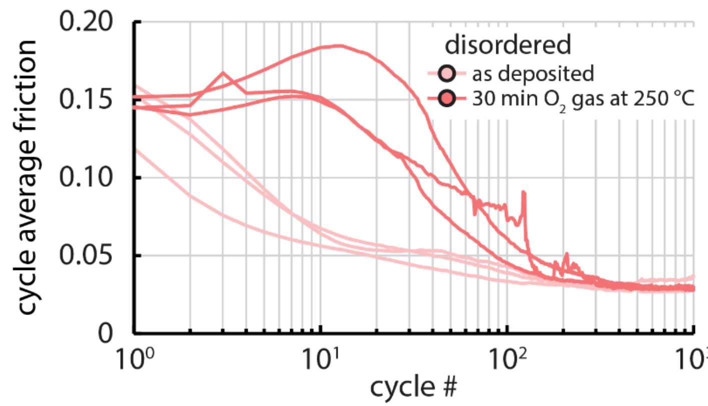


Curry et al, Tribology Letters 2016

Accelerated Aging Study







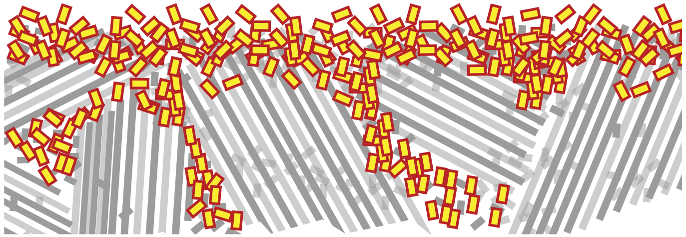
- Less defects/edges may also limit oxidation to surface

- Exposures of 30 min O_2 @ 250°C (also AO) show ordered films exhibit less oxidation & surface limited, reducing effects on run-in

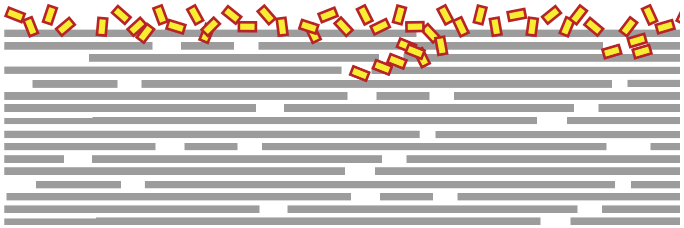
First demonstration of microstructure's role in limiting oxidation; **run-in (ordered) surfaces help prevent aging related issues**

Curry et al., ACS AMI 2017

Run-In Factor: Oxidation



disordered structure



highly-ordered structure

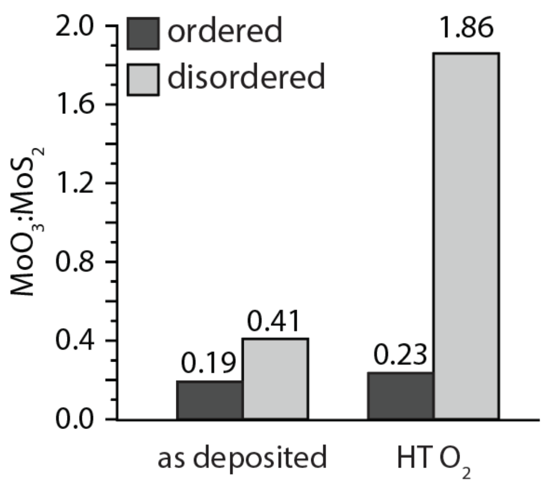
- Oxidation resistance should benefit in the same way that run-in does from ordered surfaces

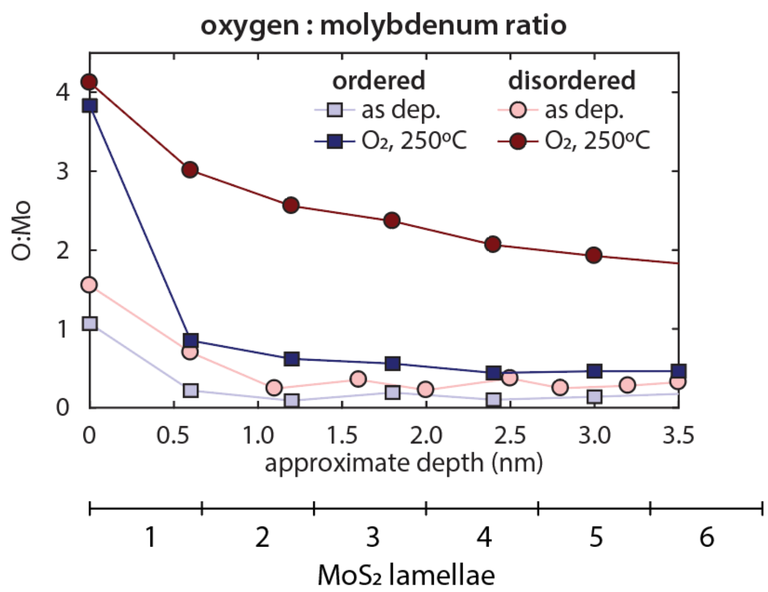
- Higher degree of basal orientation and less available edge site (large crystals) should reduce oxidation

- 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





- 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

Oxidation vs Microstructure – XPS & LEIS Study

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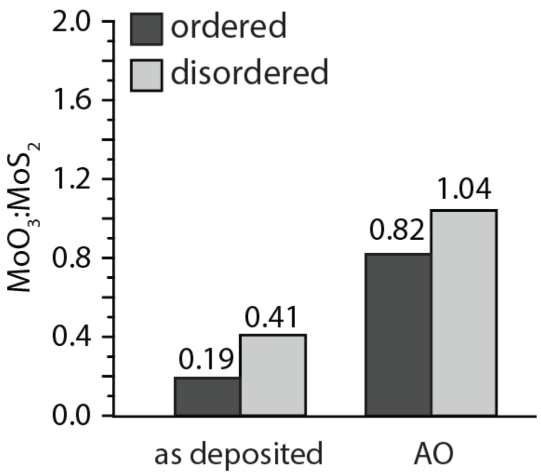

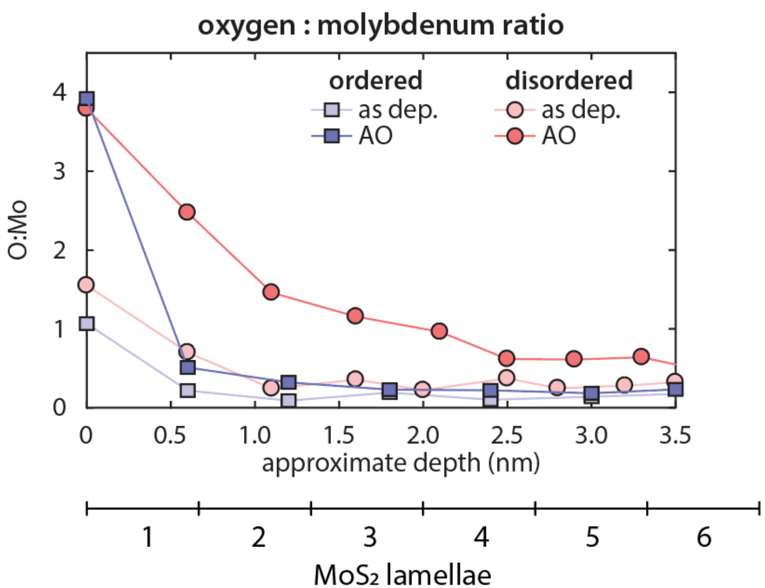

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Oxygen Gas (30 min @ 250°C)

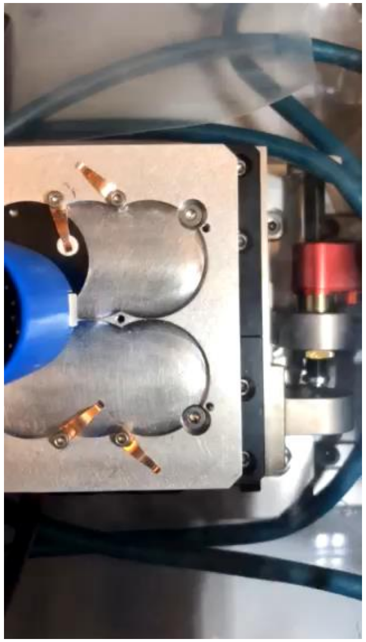
- XPS indicates minimally more oxide for ordered films while disordered films have more
- LEIS shows this is mostly surface limited for ordered films and through the surface for disordered

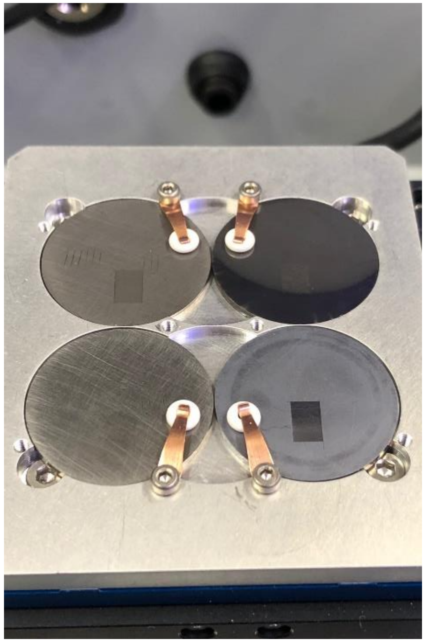
Atomic Oxygen (30 min @ RT)

- 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

raster

- Perform XPS inside versus outside rubbed area
- Return to run-in area after aging for additional friction testing

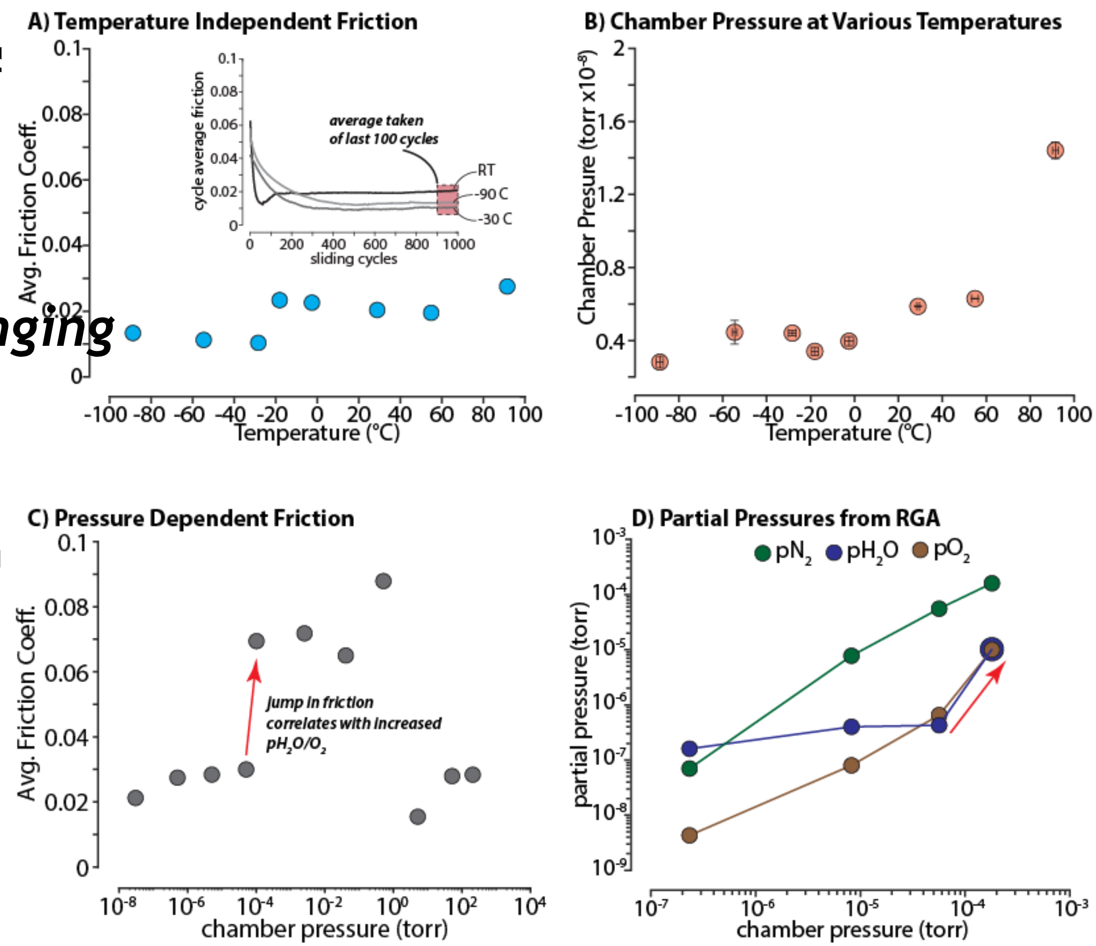




Link #4: Structurally driven temperature dependent friction of MoS_2 - friction

Constant Pressure, Changing Temperature

- In UHV, at constant pressure, the friction behavior of MoS2 is INDEPENDENT of temperature
- Coating shows no measurable wear at any temperature



Constant Temperature, Changing Pressure

- In UHV, at constant temperature, the friction behavior of MoS2 changes with pressures
- A high friction regime at moderate vacuum ranges
- Friction drops again at higher pressures
- The increase in friction correlates with an increase in the pH_2O and pO_2

Question: How is structure driven by temperature and pressure?

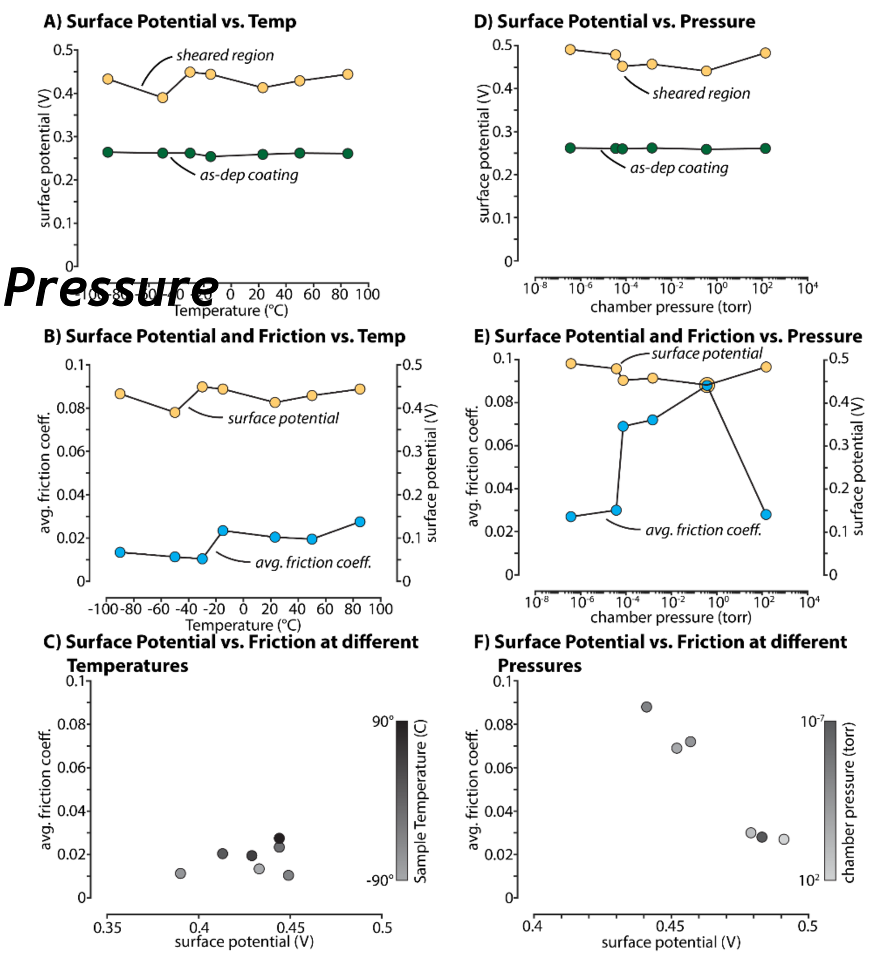
Link #4: Structurally driven temperature dependent friction of MoS_2 - KPFM

Constant Pressure, Changing Temperature

- Sheared regions at all temperatures have higher surface potential then coating
- No apparent trend between friction and surface potential

Constant Temperature, Changing Pressure

- A decrease in surface potential = increase in friction coefficient
- Increase in surface potential = decrease in friction coefficient
- Strong relationship between friction and surface potential



Takeaway: Structure does not evolve with temperature, only pressure