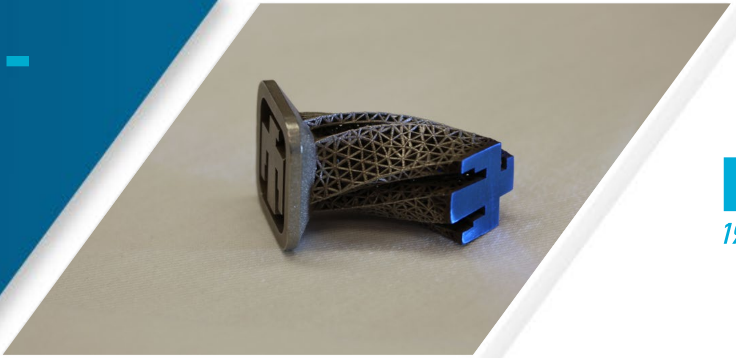
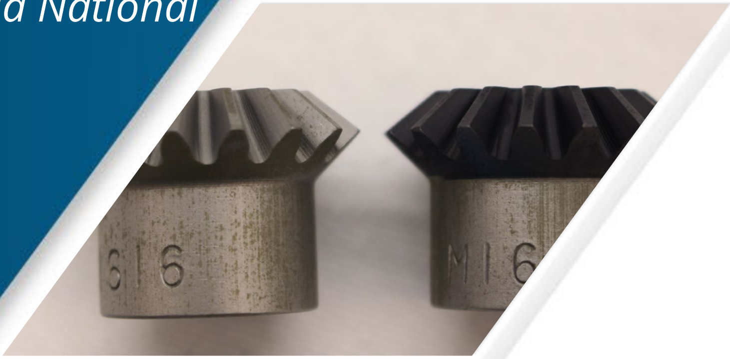
SAND2024-14937C

Sandia National Laboratories

Exceptional service in the national interest

A Sputter Flux Diagnostic Based Approach to Enabling Low Run-In for Sputter Deposited MoS_2 Solid Lubricants





Material, Physical and Chemical Sciences Center, Sandia National Laboratories

Steven R. Larson, Alex Mings, Tomas F. Babuska, Michael T. Dugger, Ping Lu, Jon Vogel, John F. Curry

AVS 70th International Symposium

Tampa, FL

1949-2024

SANDIA NATIONAL LABORATORIES

U.S. DEPARTMENT OF ENERGY

NNSA National Nuclear Security Administration

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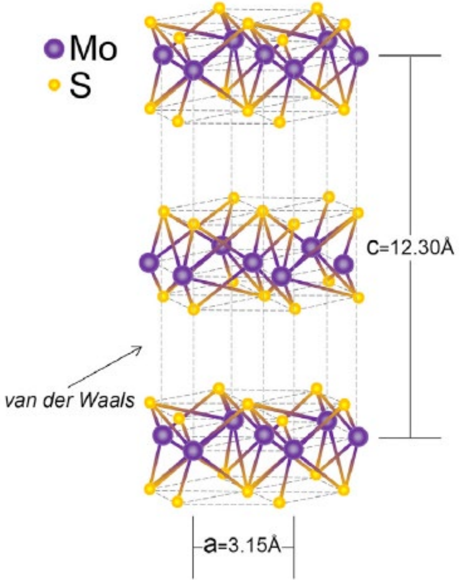
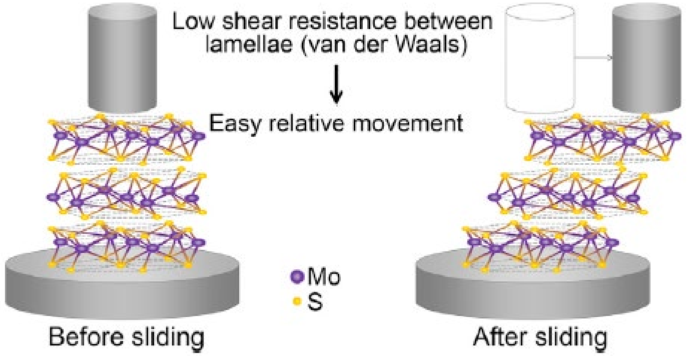
SAND-########

MOS$_2$ FILMS FOR EXTREME ENVIRONMENT LUBRICATION

https://shop.sdp-si.com/

view along basal planes



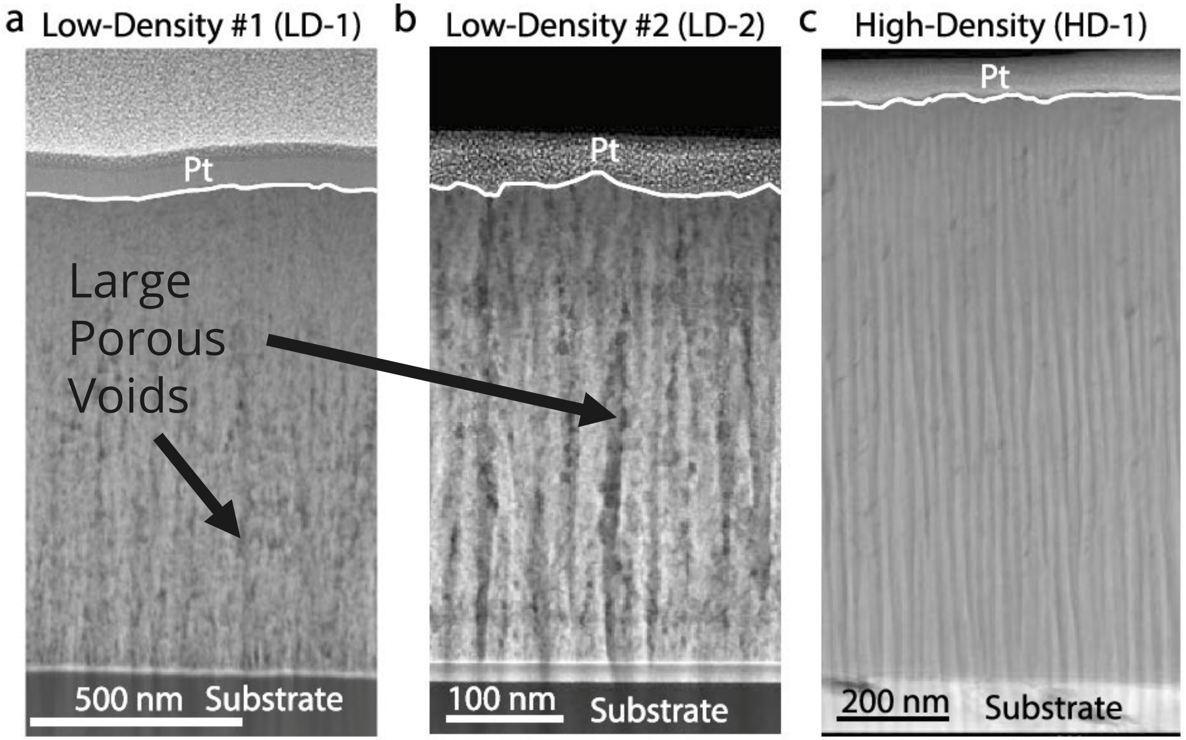


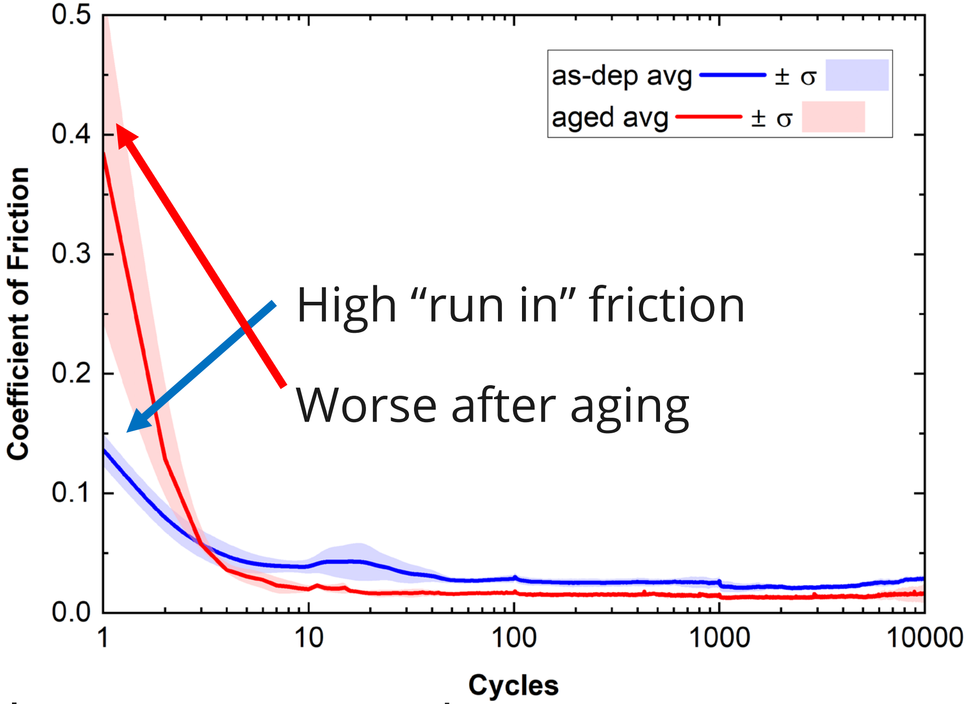
doi.org/10.1016/j.triboint.2017.12.033

Aerospace / precision mechanisms share similar concerns

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

PROBLEMS WITH CURRENT COATINGS



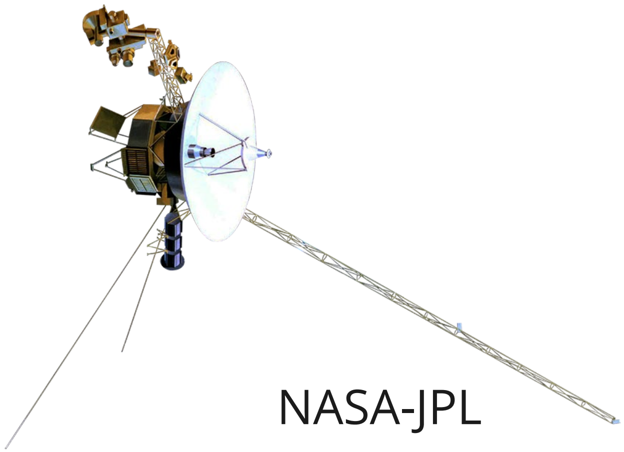


- Commercial coating are inconsistent!
  - doi.org/10.1007/s11249-022-01642-y
  - Same companies 6 months apart

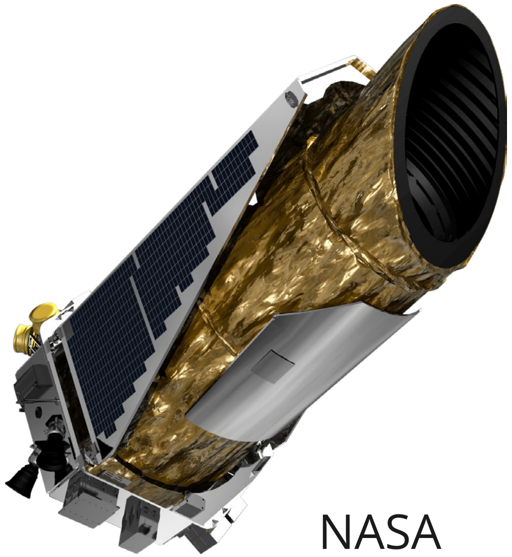
- Films get worse with age
  - Accentuated with low cycle applications
- Lubrication is important
  - Voyager 2, Keplar, Galileo all partially failed due to lubrication

REAL PROBLEMS FOR TRIBOLOGY (AND NASA)

- <u>Voyager 2:</u> Science platform seized due to mitigation of lubricant out of motor gear shaft, delayed use for 16 months, all future experiments ran at 0.083 deg/s instead of 1deg/s(1)
- <u>Keplar:</u> 2/4 reaction wheels seized due to uneven lubrication of mechanical bearings causing galling, prolonged mission delays, had to use radiation pressure to compensate(2)
- <u>Galileo:</u> sticking of 3/18 antenna ribs in stowed position due to high friction between pins and sockets, over 100 personal did simulations and testing to report the MoS2 bonding failed(3)



NASA-JPL



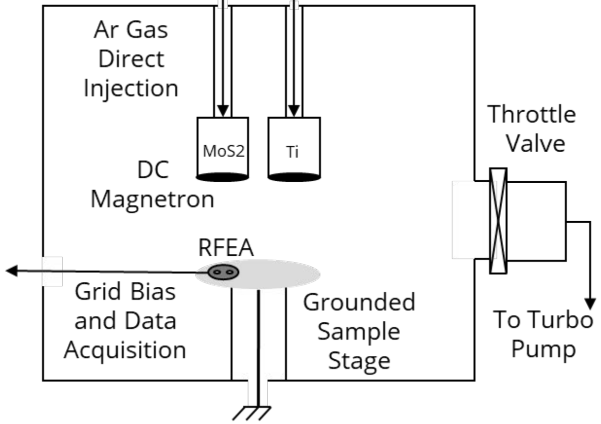
NASA

[1] Physics Today 43, 7, 40 (1990); doi: 10.1063/1.881251
[2] Kepler Mission Manager Update: Kepler Returns to Science Mode. (2015, April 15). Retrieved June 10, 2018, from https://www.nasa.gov/mission_pages/kepler/news/keplerm-20132901.html
[3] Miyoshi, K. (1999). *Aerospace Mechanisms and Tribology Technology: Case Studies*.

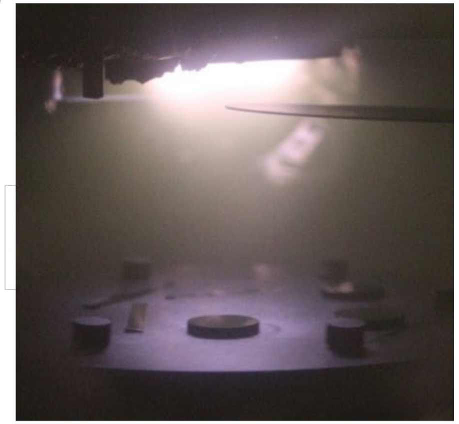
SNL MoS_2 Thin Film Synthesis Capability

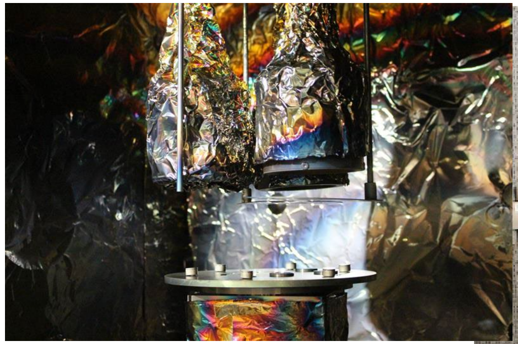
- **Rapid turnaround for testing hypotheses**
- Filled a gap in capabilities for in-house research and production of PVD solid lubricants
  - Develop process-structure-property understanding to speed up responses to mission needs
  - **Collaborate with production** to solve production issues
  - **Increase efficiency vs external vendor**
  - **Built for ~1/4 the price of a commercial tool**
- ~**150 deposition runs per year**
  - Supporting research and many development projects
- **Built for flexibility**
- Multiple RF, DC, Pulsed DC and HIPIMS power supplies with HIPIMS bias capable stage with liquid cooling

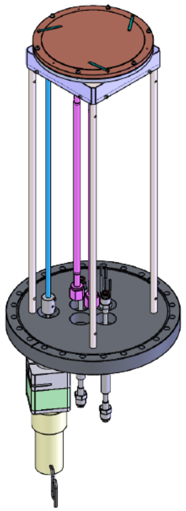
(A)



(B)

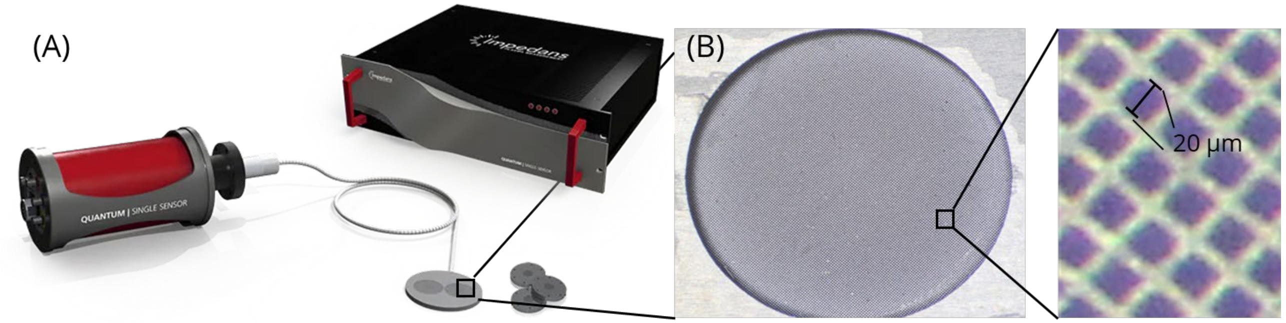


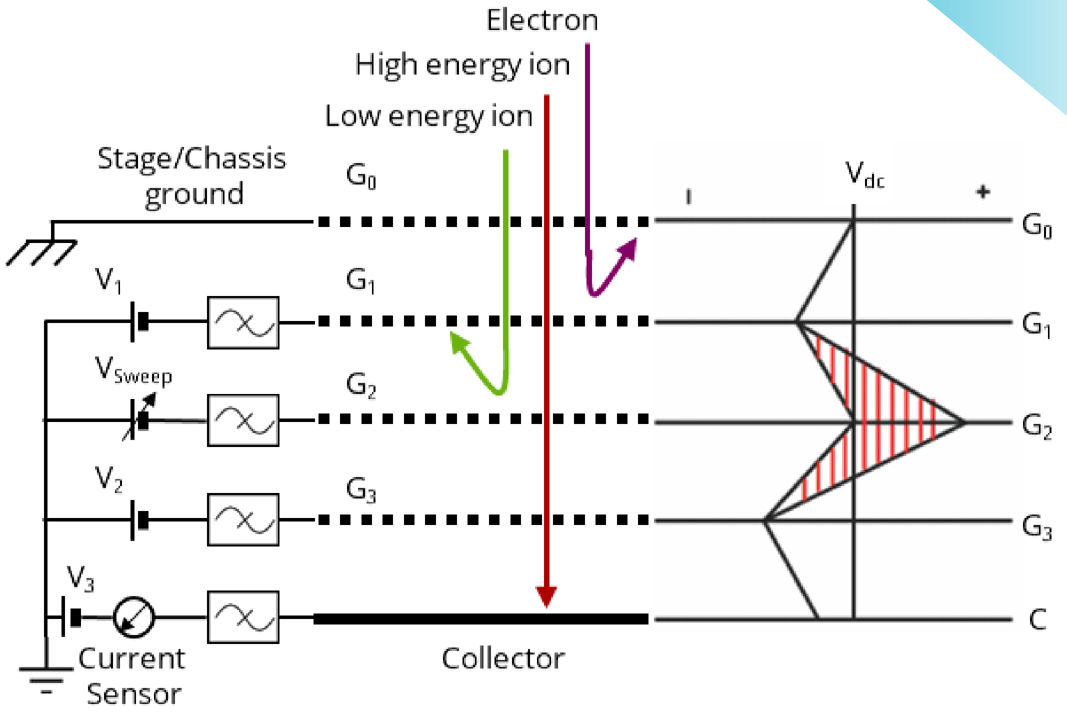




- 3 in. sputter cathodes
- 2x RF, 1x DC, 1x P-DC, 2x HIPIMS

DEPOSITION FINGERPRINTING - RETARDING FIEND ENERGY ANALYZER





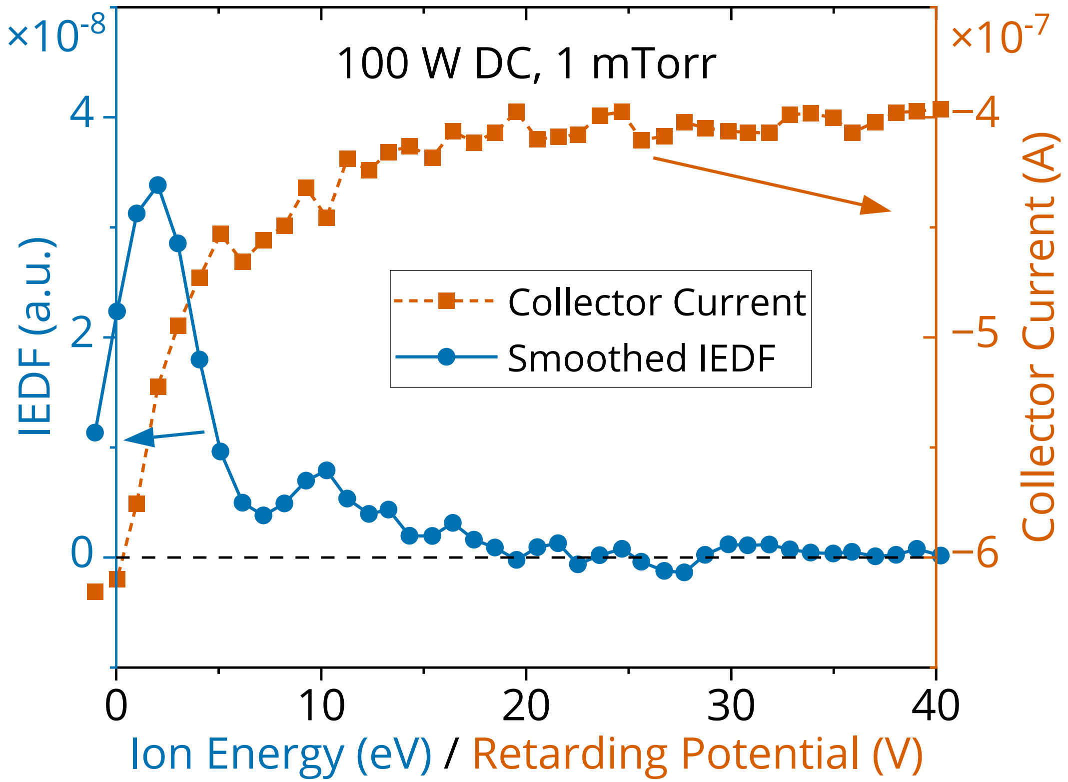
<u>What Is an RFEA?</u>

- Used industrially for “fingerprinting” – a method of transferring proven processes across different equipment setups
- Measures ion flux, the ion energy distribution, deposition rate and, deposition material ionization
- Isolates the effects of varying deposition conditions on plasma behavior
- Allows for direct correlation between plasma characteristics and film material properties

<u>How does this thing work?</u>

- <u>Grid 0</u> (G_0) - holes less than Debye length to prevent plasma formation. Held at stage bias (ground)
- <u>Grid 1</u> (G_1) - Electron repulsion grid (-60 V)
- <u>Grid 2</u> (G_2) - Discriminator grid, sweeps to control ion flux based on energy
- <u>Grid 3</u> (G_3) - Secondary electron suppression grid (-70 V)
- <u>Collector</u> (C) - QCM and collector (-60 V)

WHAT ARE WE MEASURING



I-V Curves

- The "retarding potential" represents the voltage on Grid 2 (ion repulsion) and the current is measured by the collector.
- A drop in current represents ion repulsion at that potential.
- The total drop in current is proportional to the total ion flux.

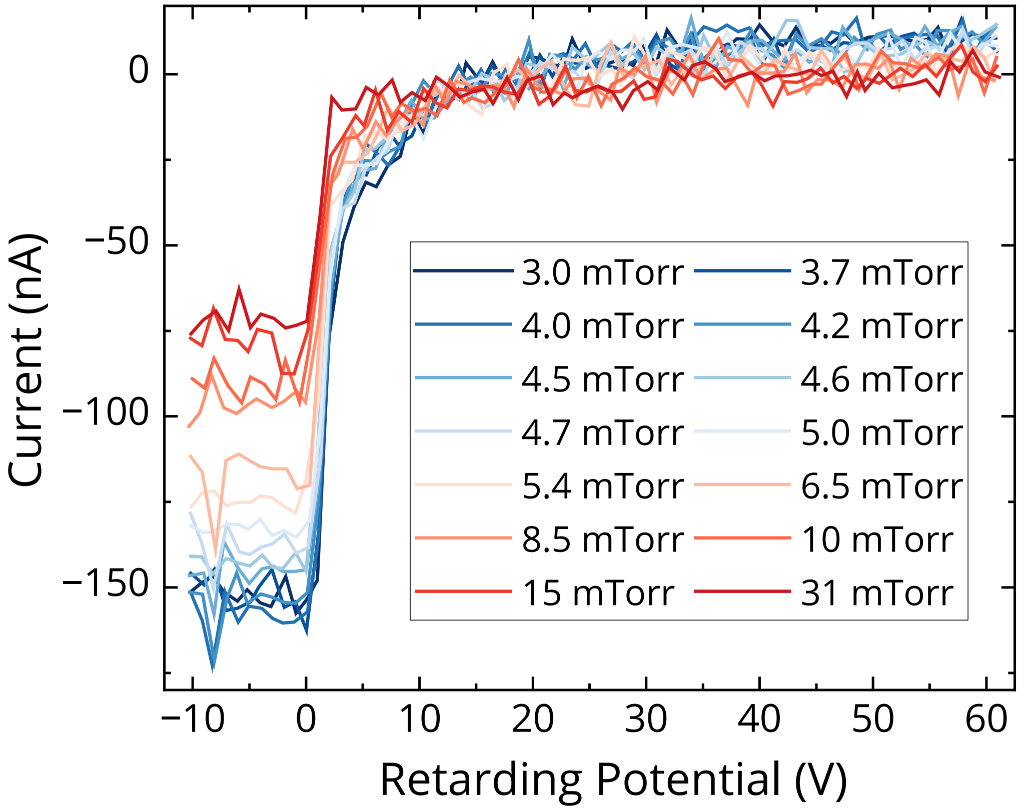
$$IEDF = \frac{dI}{dV} = f(E)$$

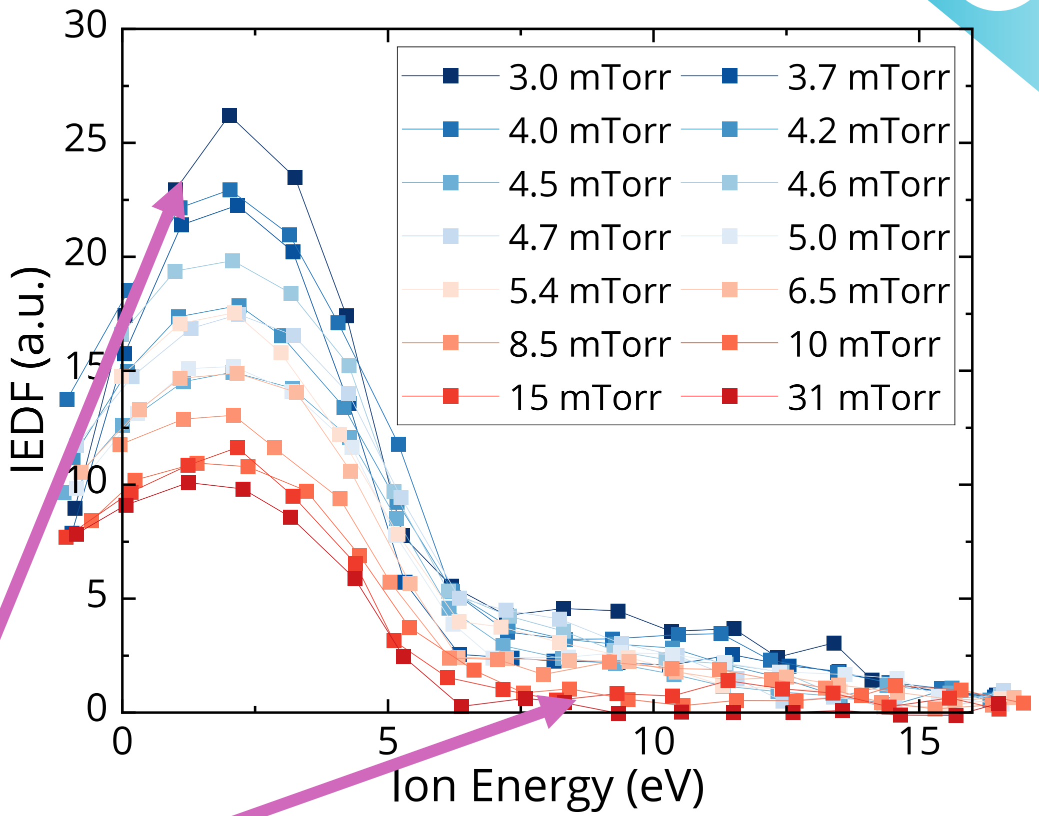
Where I is ion current, V is retarding potential and E is ion energy

- Savitzky-Golay smoothing is employed to minimize distortion in the ion energy distribution function (IEDF).[7]
- The IEDF represents the probability an ion has a specific kinetic energy.

7 Caldarelli, A. *et al.* Data processing techniques for ion and electron-energy distribution functions. *Physics of Plasmas* **30** (2023).

PRESSURE SERIES DATA





- Secondary peak at pressures lower than 5mtorr ~10 eV
  - Ar ions + the start of MoS2 ionization
- Peak Ion density ~4 mtorr

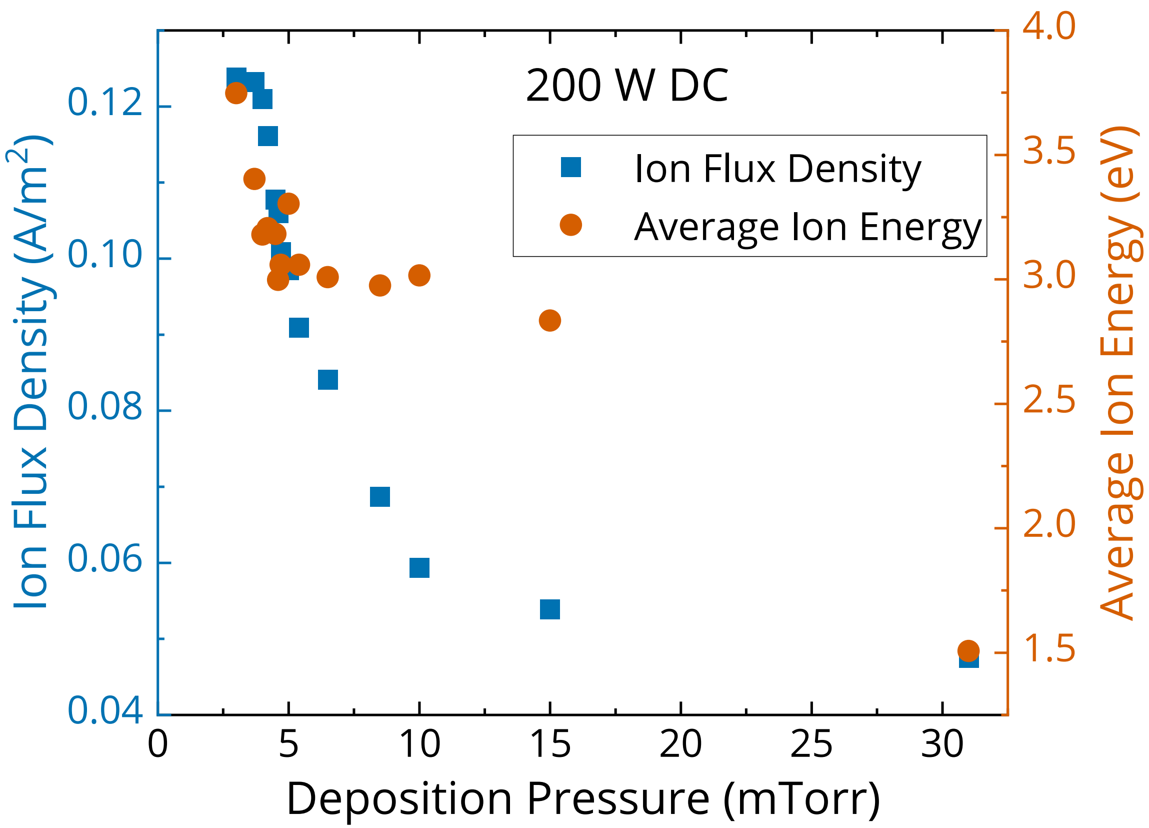
Deposition Conditions

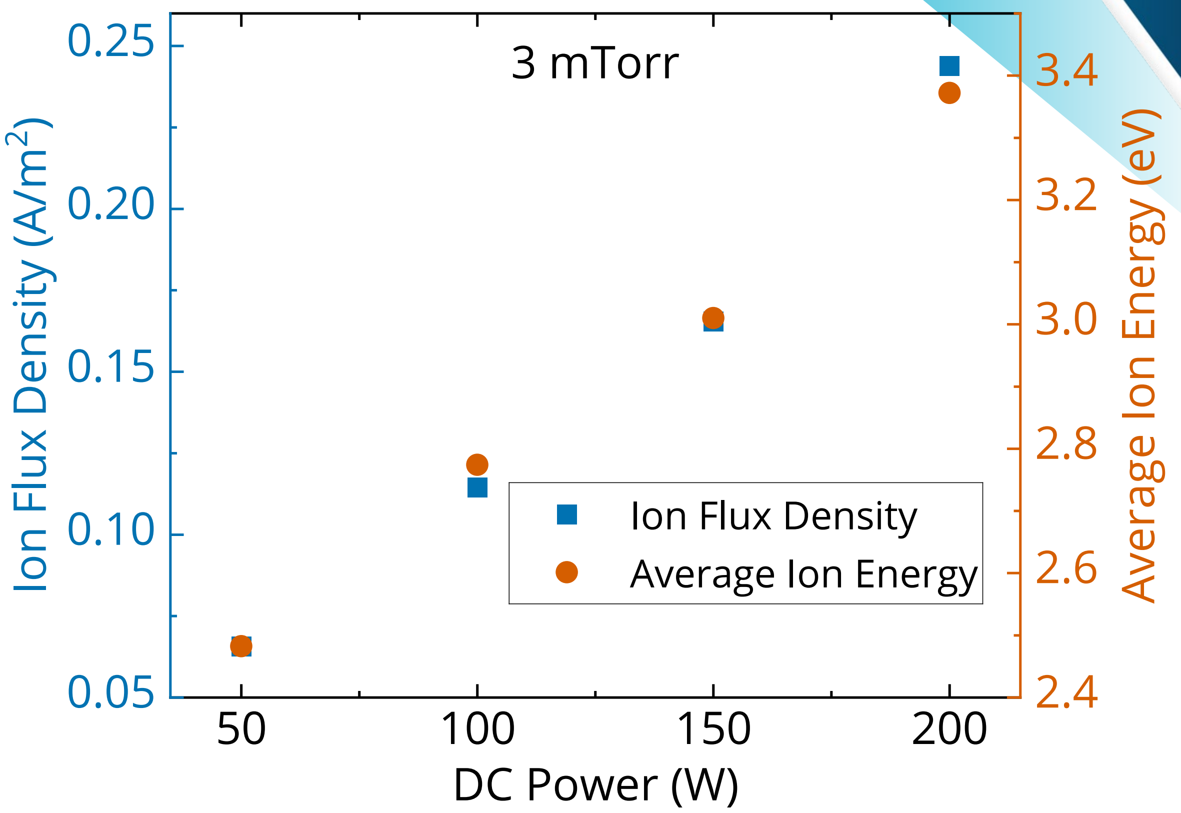
- Base pressure: <1×10^{-6} Torr
- Target to substrate distance: 4 in.
- Target diameter: 3 in.
- Ti adhesion layer thickness: 100 nm
- MoS_2 thickness: 1 μm

Sensor Conditions

- Positioned directly under the target – in line with the deposition flux

ION FLUX DENSITY AND AVERAGE ION ENERGY





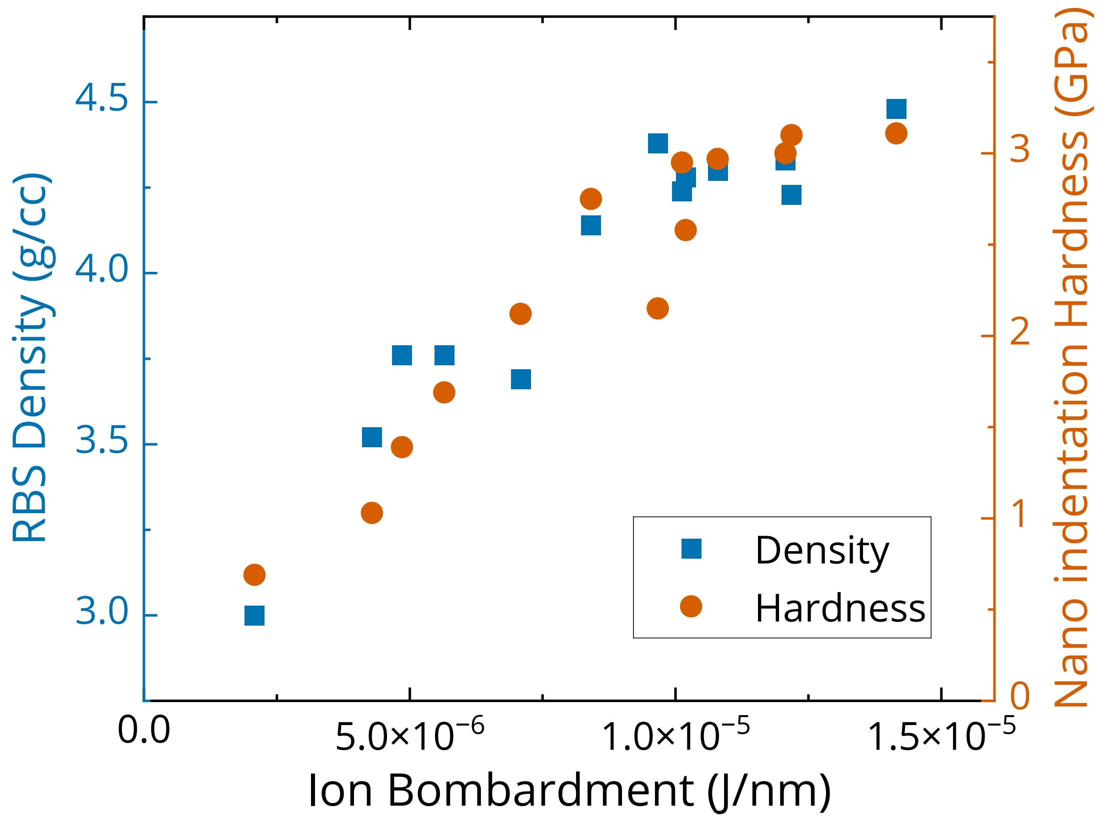
$$E_{AVG} = \frac{\int_{E_{min}}^{E_{max}} Ef(E)dE}{\int_{E_{min}}^{E_{max}} f(E)\,dE} \qquad J_{ion} = \frac{\int_{E_{min}}^{E_{max}} f(E)dE}{AT}$$

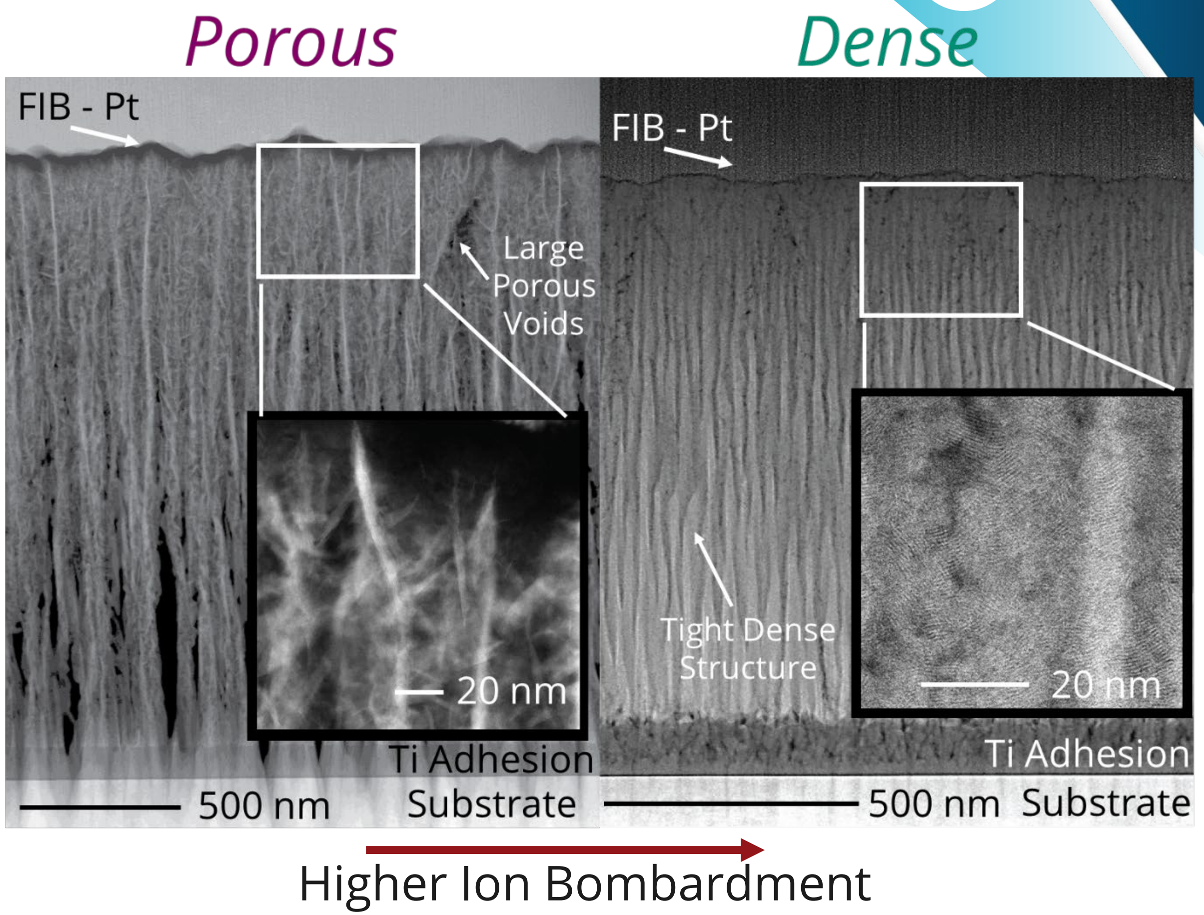
Where I is ion current, V is retarding potential and E is ion energy, A is the area of the aperture and T is the transmission of each of the 4 grids

- Both ion flux density and average ion energy increase with increased sputter power –resulting from increased accelerating voltage and amperage.
- Lower deposition pressure dramatically increases average ion energy – resulting from an increased mean free path.
- Lower pressure / higher power conditions produce higher energy ions – a well studied phenomenon.[8]

8 Garofano, V., Montpetit, F., Glad, X., Gangwar, R. K. & Stafford, L. Experiments and kinetic modeling of the ion energy distribution function at the substrate surface during magnetron sputtering of silver targets in radio frequency argon plasmas. *Journal of Vacuum Science & Technology A* **37** (2019).

RELATING TO MATERIAL AND TRIBOLOGICAL PROPERTIES

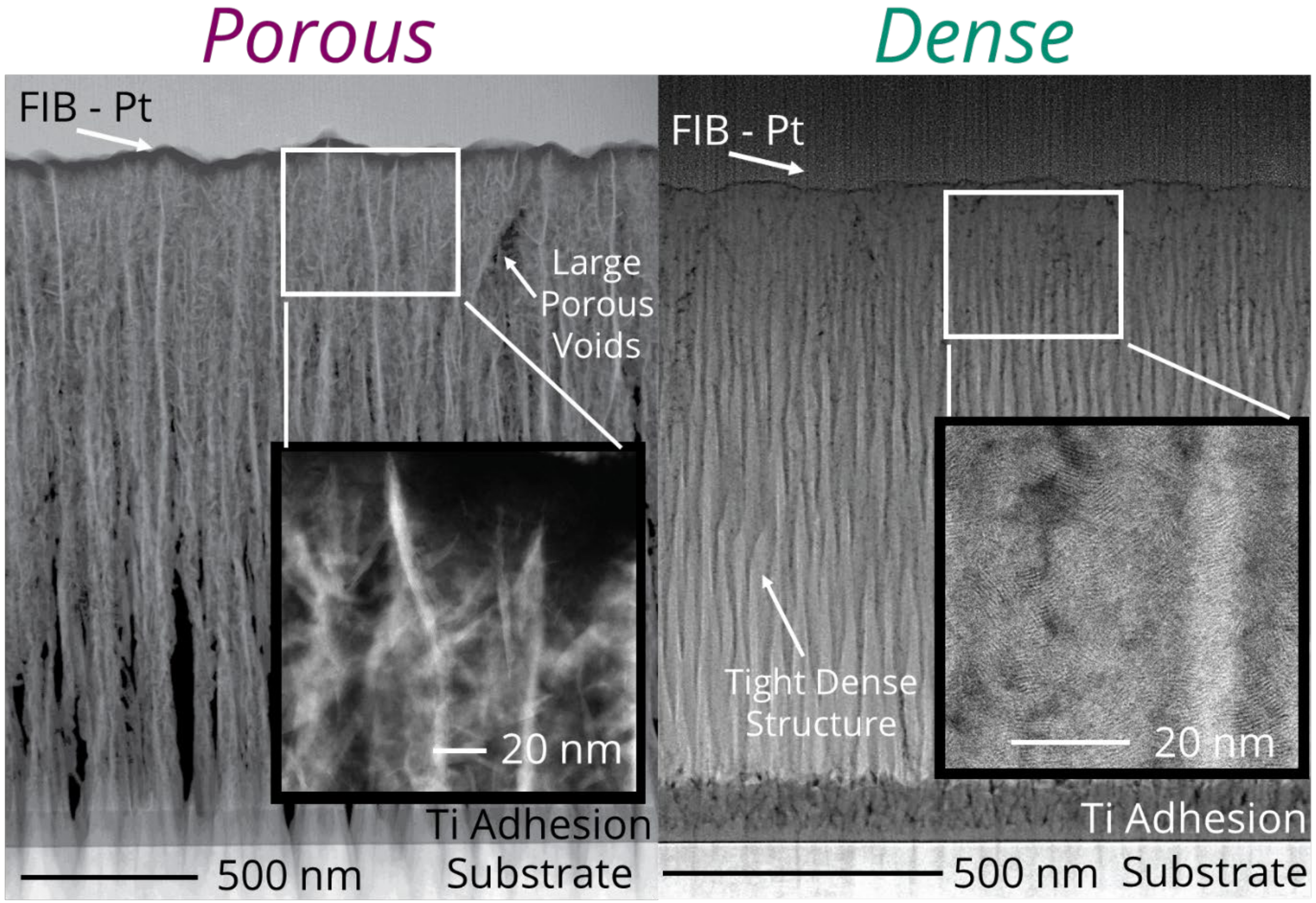


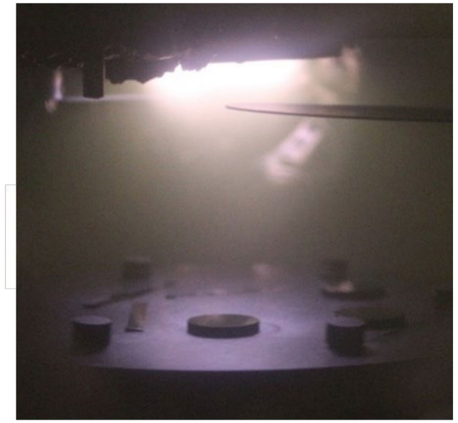


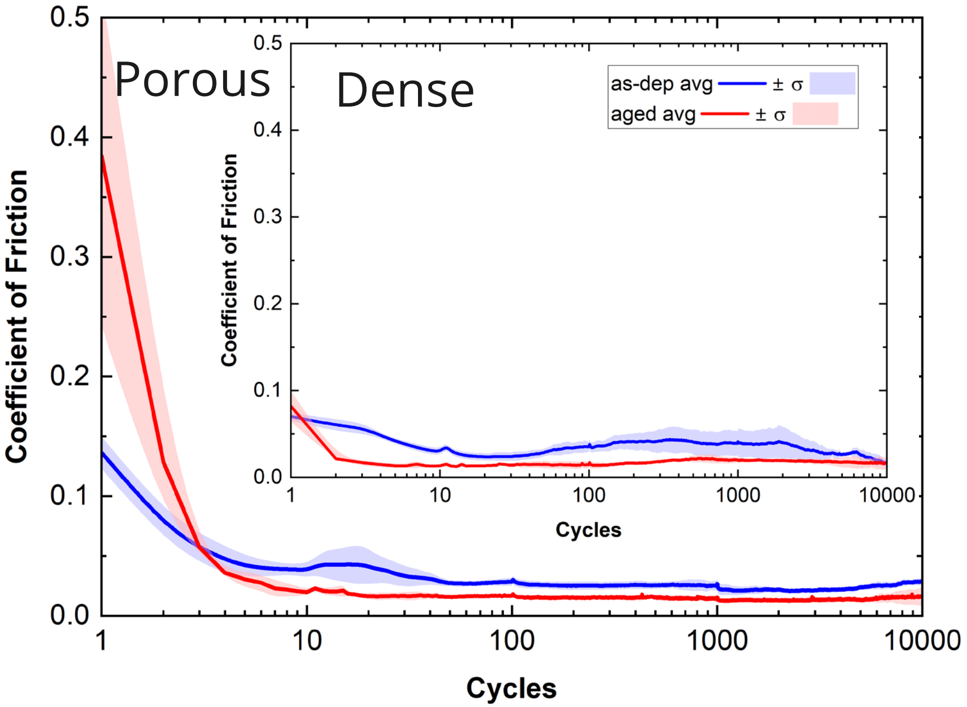
- Increased ion bombardment increases hardness of the film
  - This is well established for more controlled ion beam assisted depositions[9]
- This can be directly seen in TEM of the deposited films
  - Low bombardment films as significantly more porous

(9) Smidt, F. Use of ion beam assisted deposition to modify the microstructure and properties of thin films. *International Materials Reviews* **35**, 61-128 (1990).

QUESTIONS

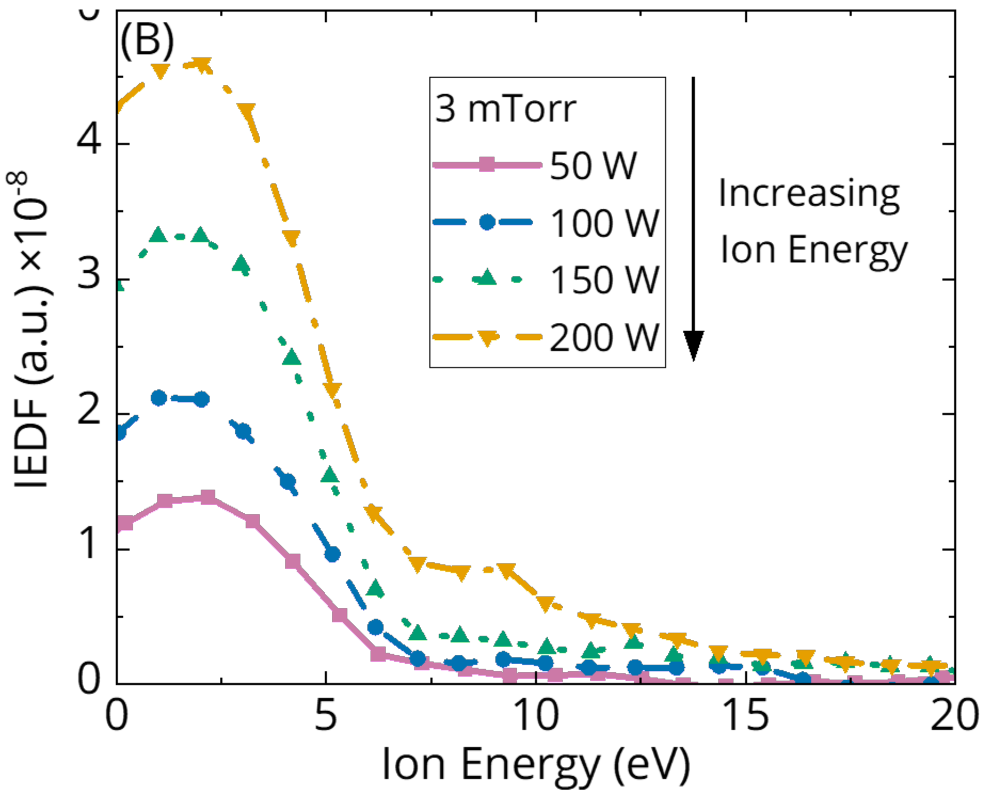

Porous
Dense
FIB - Pt
Large
Porous
Voids
20 nm
Ti Adhesion
500 nm
Substrate
FIB - Pt
Tight Dense
Structure
20 nm
Ti Adhesion
500 nm
Substrate

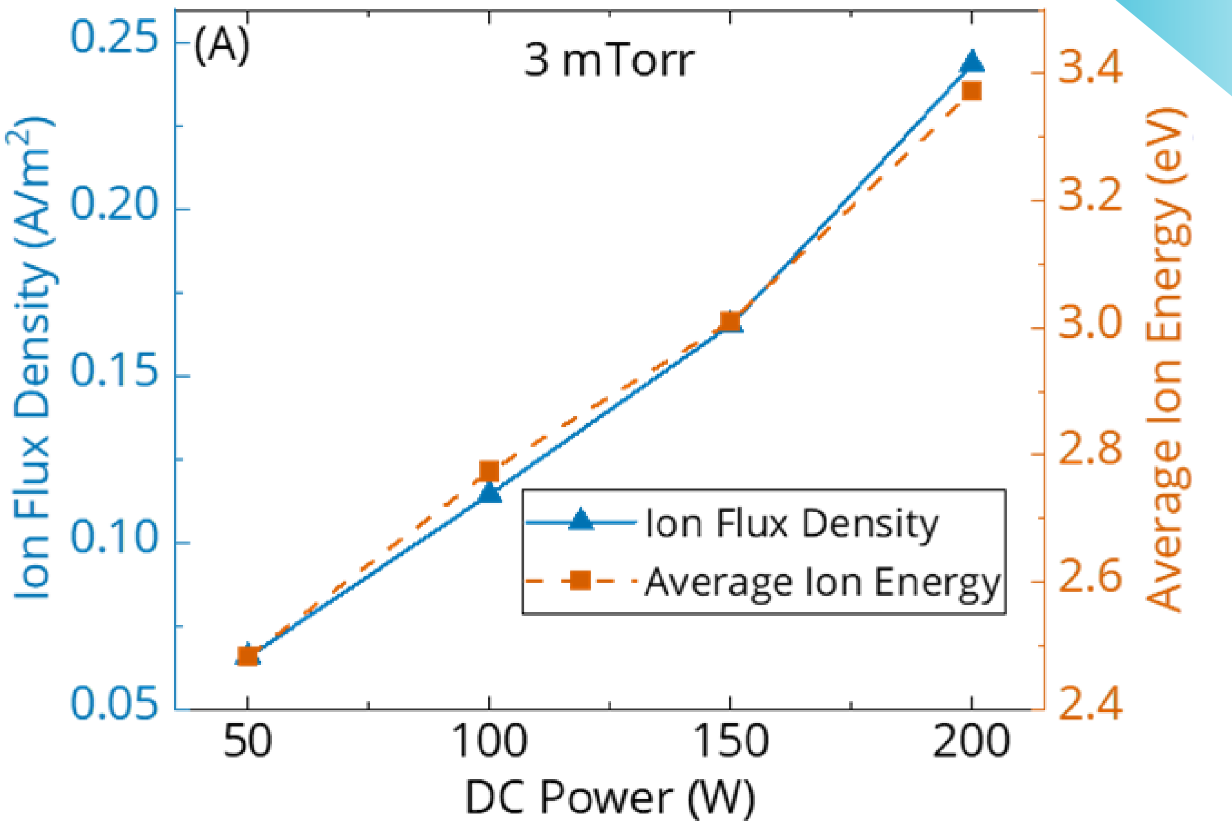



Porous
Dense
as-dep avg ± σ
aged avg ± σ
Coefficient of Friction
Cycles
Coefficient of Friction
Cycles
0.5
0.4
0.3
0.2
0.1
0.0
1
10
100
1000
10000

BACK UP SLIDES

POWER SERIES DATA





- Lower pressure / higher power conditions produce higher energy ions – a well studied phenomenon.[8]

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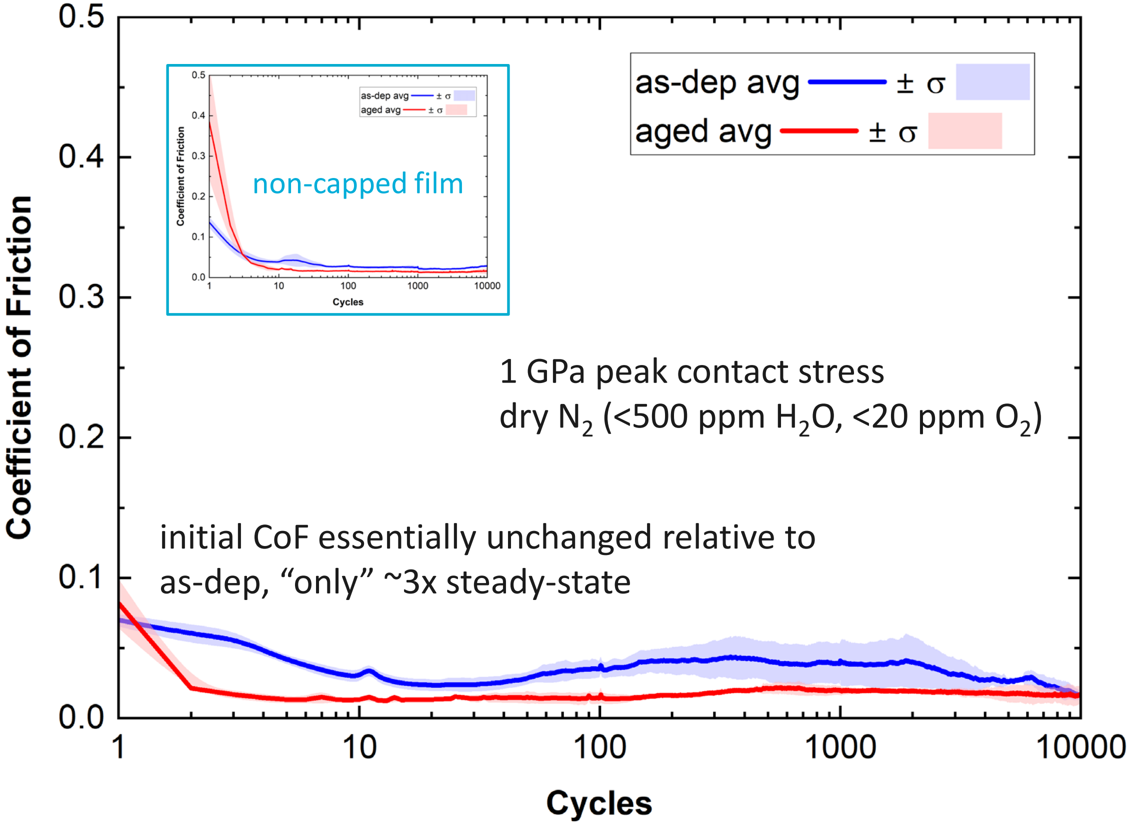
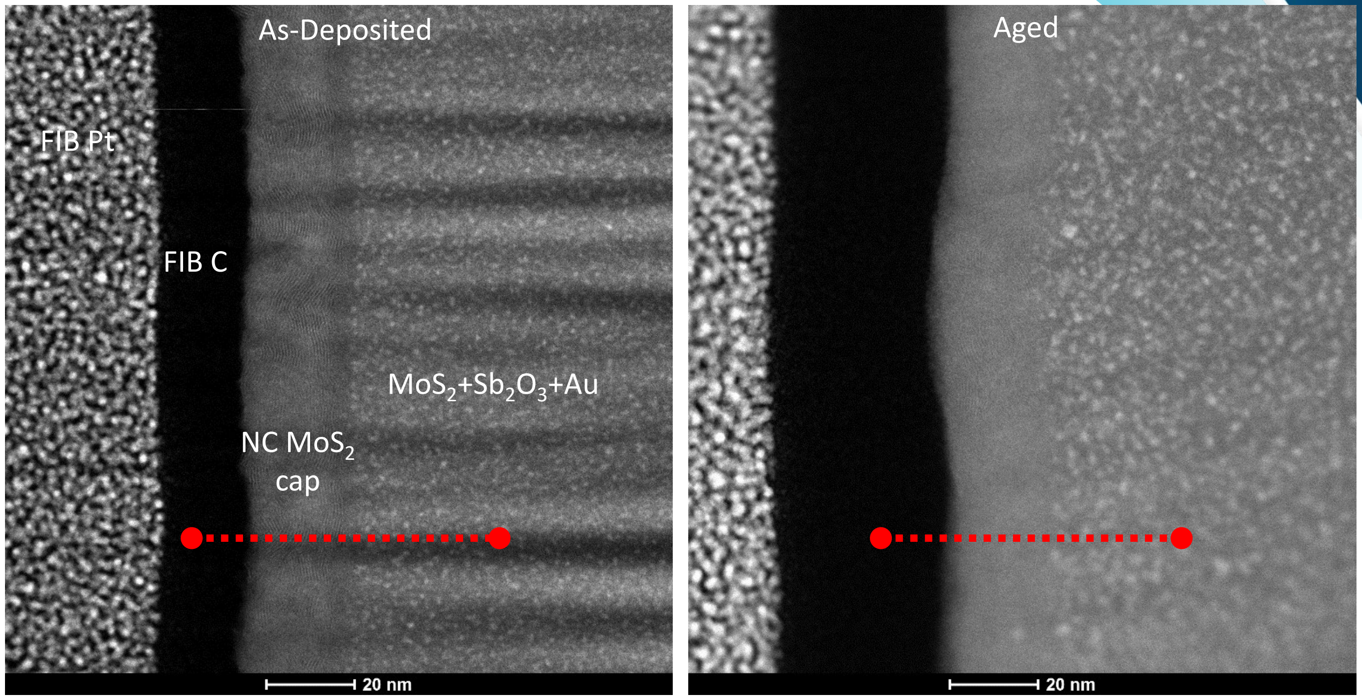
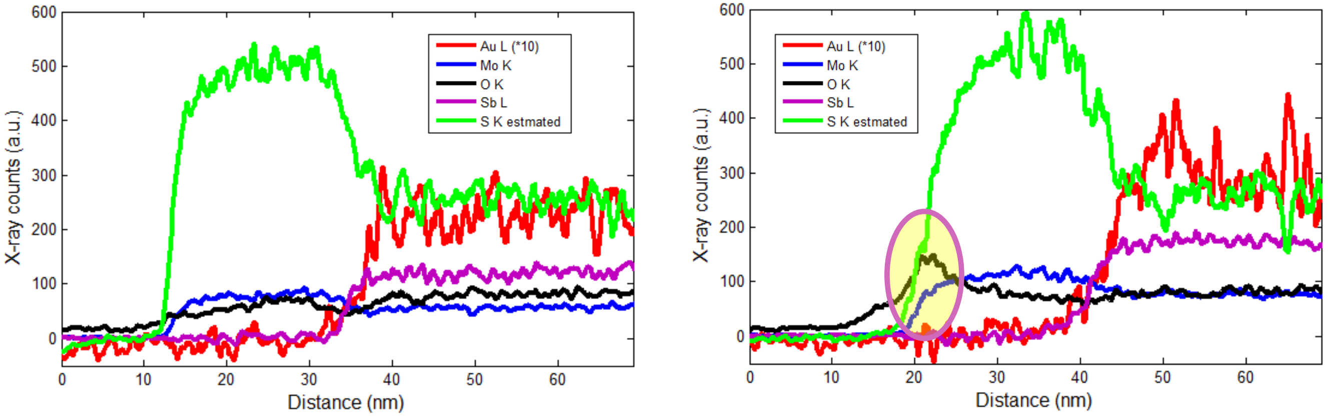
TRIBOLOGICAL BEHAVIOR OF MULTILAYER PVD MoS_2 PRE- AND POST-AGING

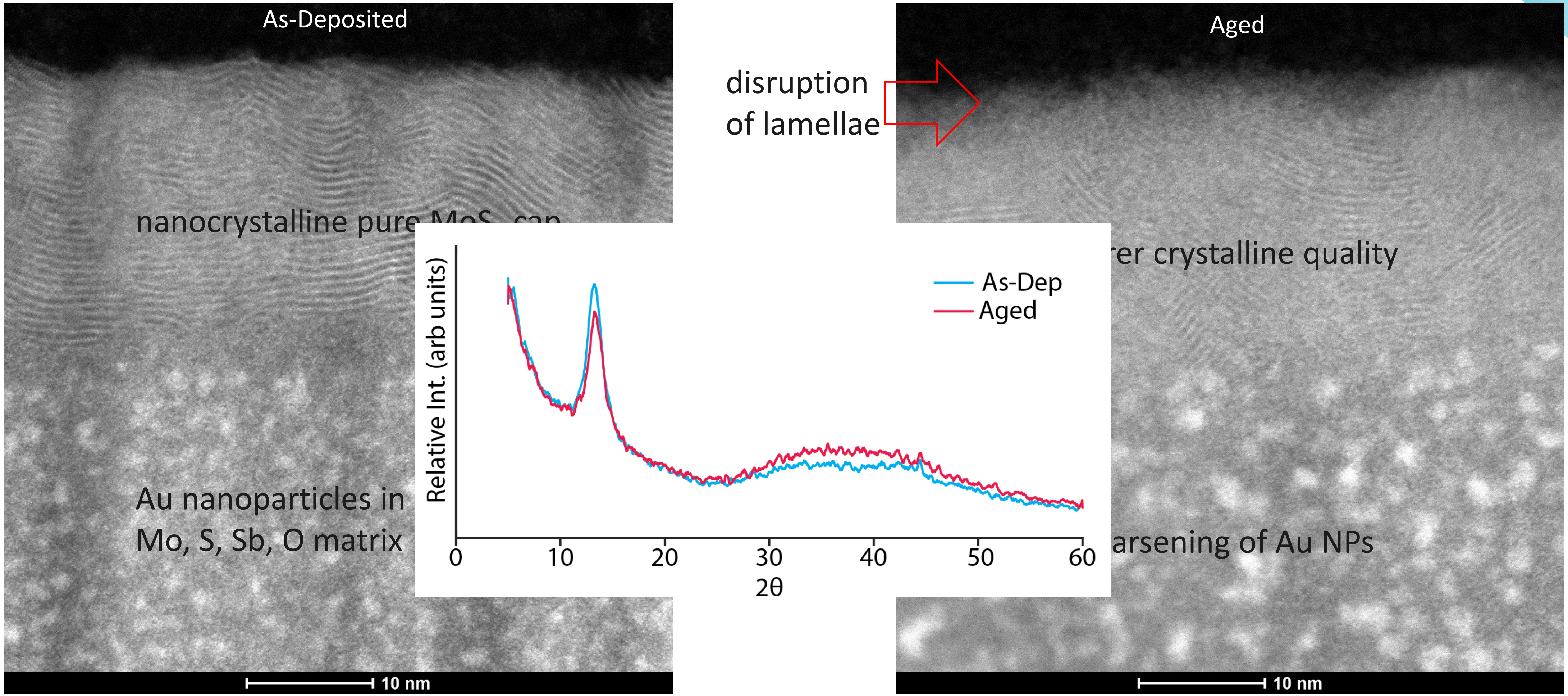






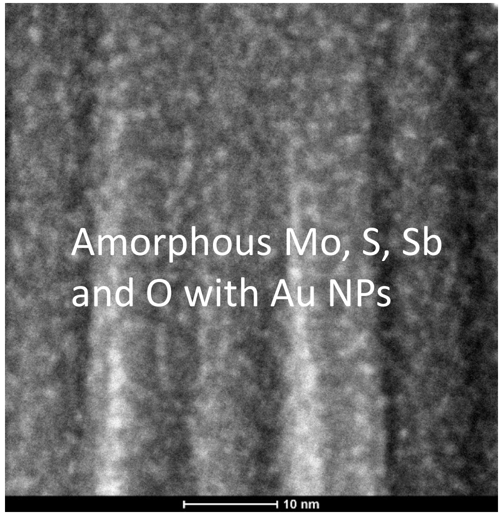
- Starting friction is virtually unchanged in the aged film relative to the as-deposited film
- Oxygen is incorporated into the top ~5 nm of the aged film

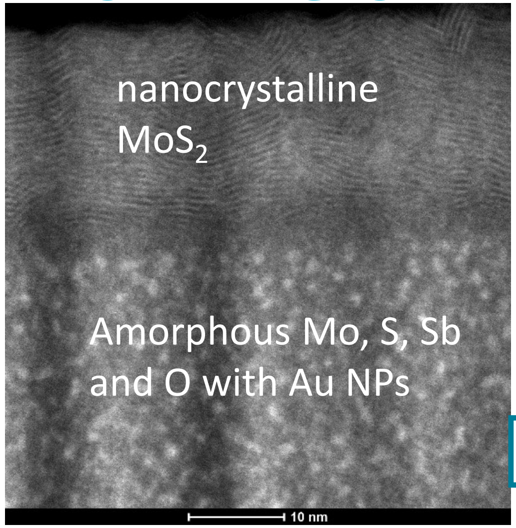
DETAILED STRUCTURE OF MULTILAYER MOS_2 NEAR SURFACE, PRE- AND POST-AGING

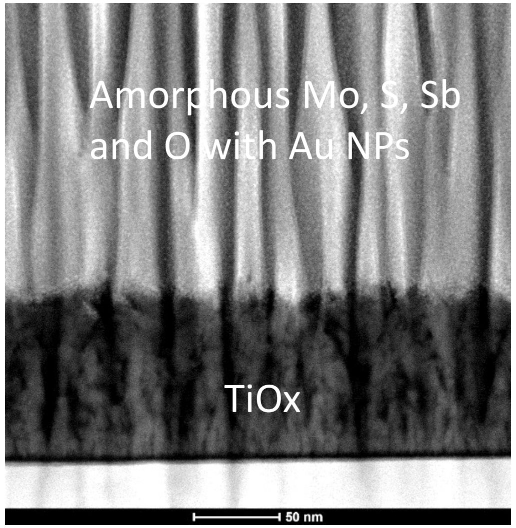


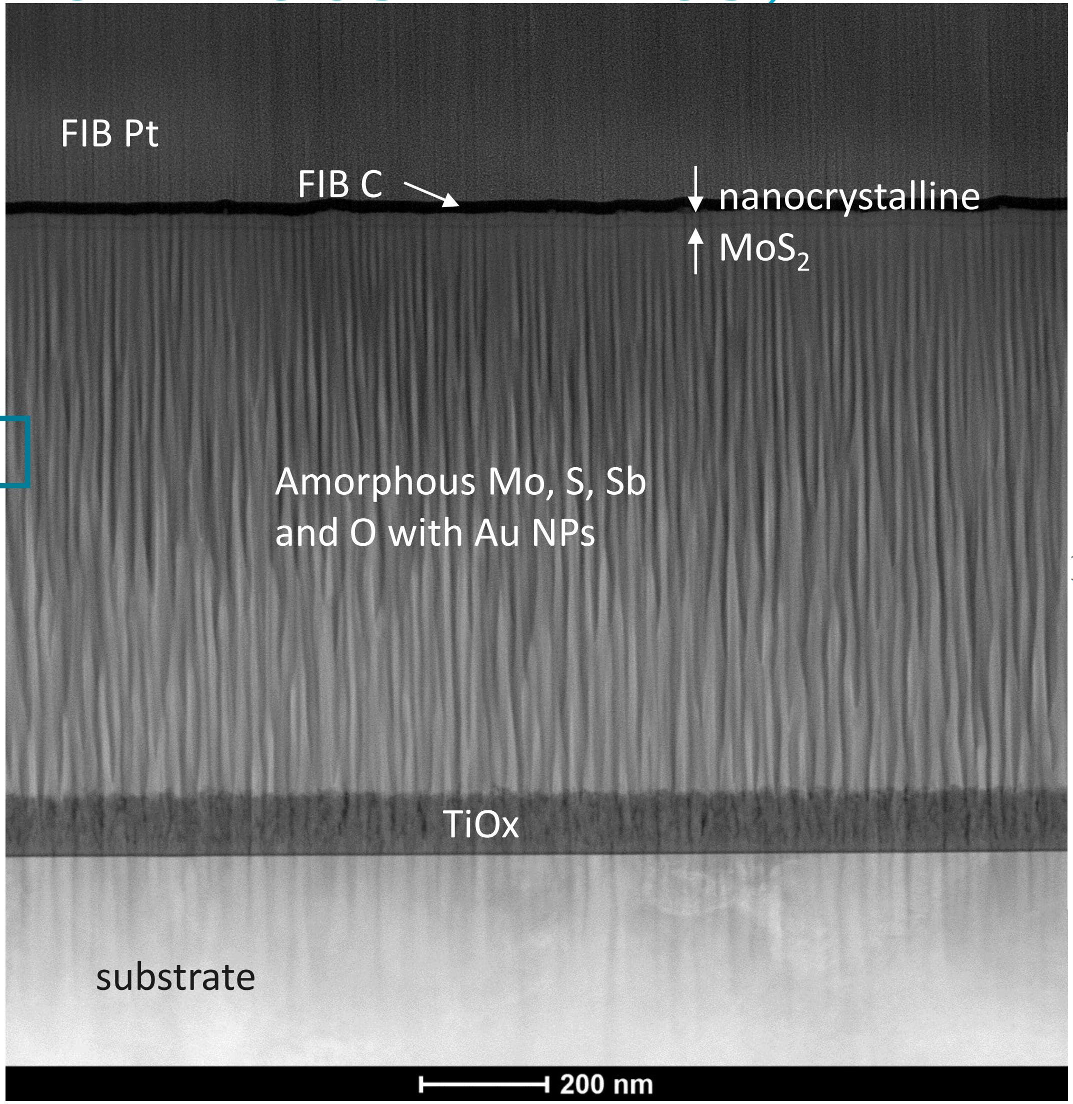
- Accelerated aging causes disruption of the crystalline lamellae at the surface, poorer crystallinity in the nanocrystalline cap, and coarsening of Au nanoparticles in the bulk

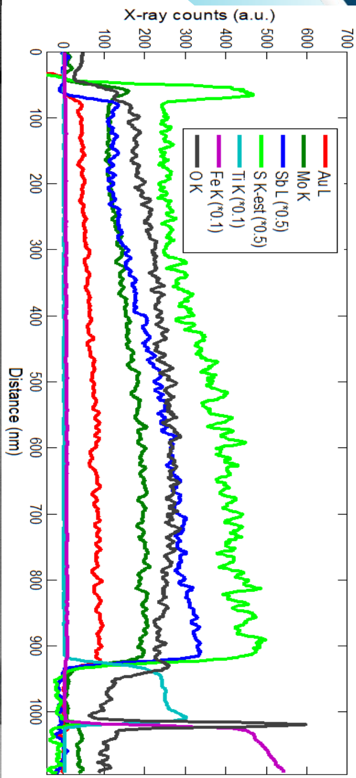
REALIZATION OF EBC ON AMORPHOUS PVD MOS_2









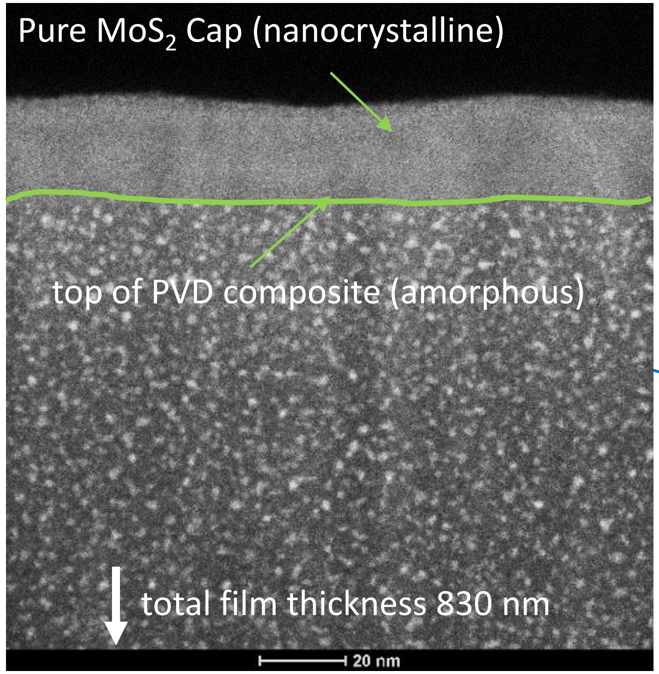


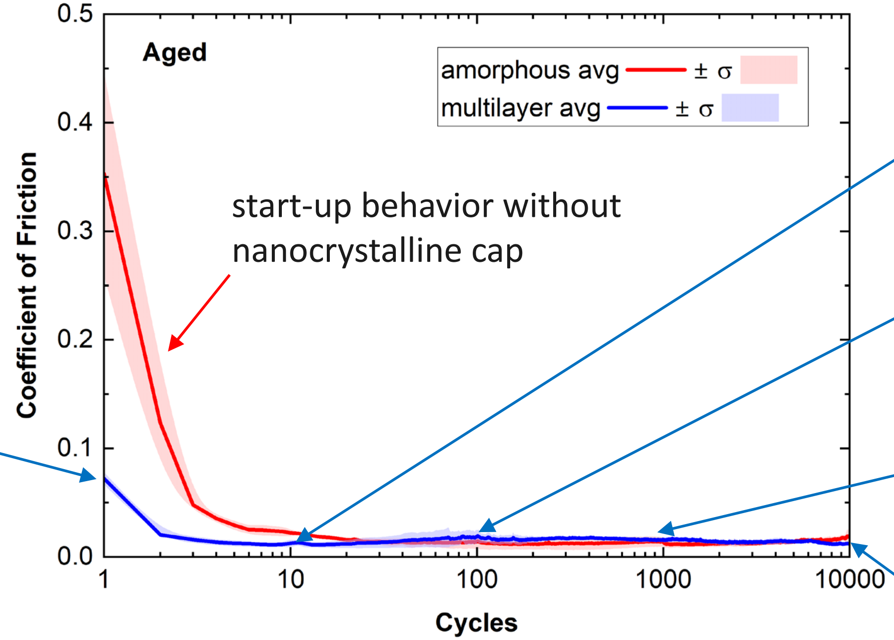
concentration gradient through bulk of film is an artifact of section thickness

- A dense nanocrystalline pure MoS_2 layer was deposited on top of a dense, amorphous Sb_2O_3 and Au-doped layer

Low Friction Persists When the Nanocrystalline Cap is Worn Through

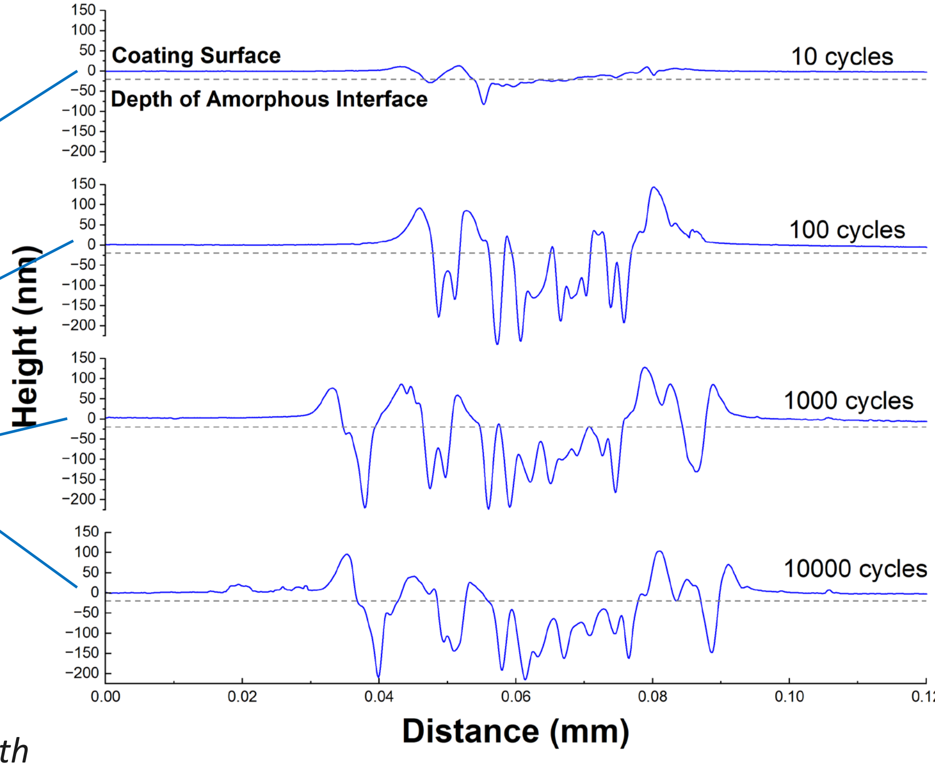
Multilayer Film





Hypothesis: The nanocrystalline cap provides low starting friction and environmental stability, while stress-induced transformation below the sliding interface enables a smooth transition to low-wear bulk behavior

Wear Track Profiles



- No increase in friction is observed when the nanocrystalline layer is worn through over most of the track width at ~100 cycles
  - capping of the amorphous coating allows combination of the low start-up friction of nanocrystalline pure MoS_2 with the wear resistance of amorphous coatings