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Sandia National Laboratories

Retarding Field Energy Analyzer Based Analysis of DC Sputtered High Performance MoS2 Tribological Coatings

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Introduction

- Sputter deposited MoS_2 coatings have a long history as dry lubricants for aerospace applications because of their ultra-low friction and high performance in vacuum.[1]
- Notably, the James Webb Space Telescope (JWST) utilizes a MoS_2 nanocomposite in it's Near Infrared Camera (NIRCam).[2]
- Under specific conditions, basally oriented (002) MoS_2 crystals can create high out-of-plane growth rates, resulting in branching and highly porous microstructures[3,4] (Fig. 1).
- These porous structures significantly reduce oxidation resistance[5] and wear life[6], jeopardizing component reliability.

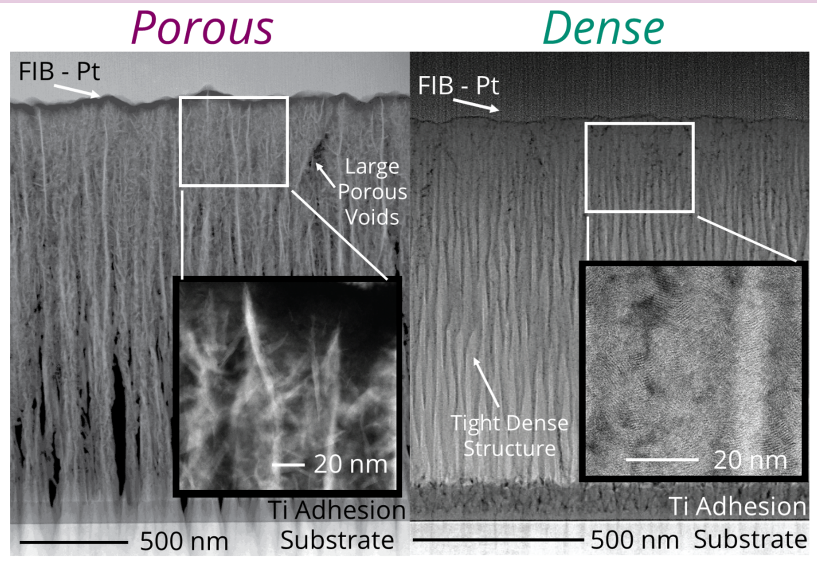


Figure 1: TEM images of porous and dense MoS_2 films with Ti adhesion layers deposited at different conditions.

- Previous work has shown MoS2 films deposited with seemingly identical conditions, produce a wide range of porosities.[6]
- Here we demonstrate the use of a Retarding Field Energy Analyzer (RFEA) to characterize the plasma during deposition to better understand and predict drivers for these microstructural changes.

Experimental Setup

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

- The sensor is positioned directly under the target – in line with the deposition flux

Equipment Configuration

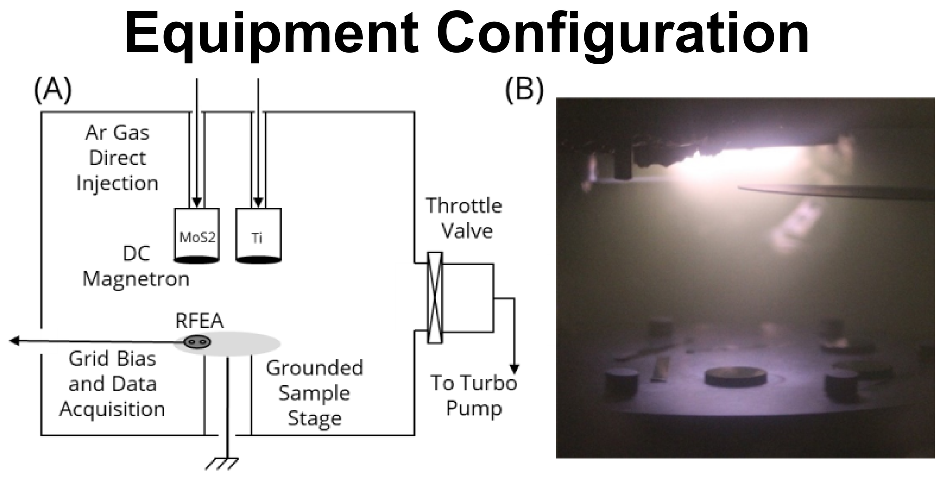
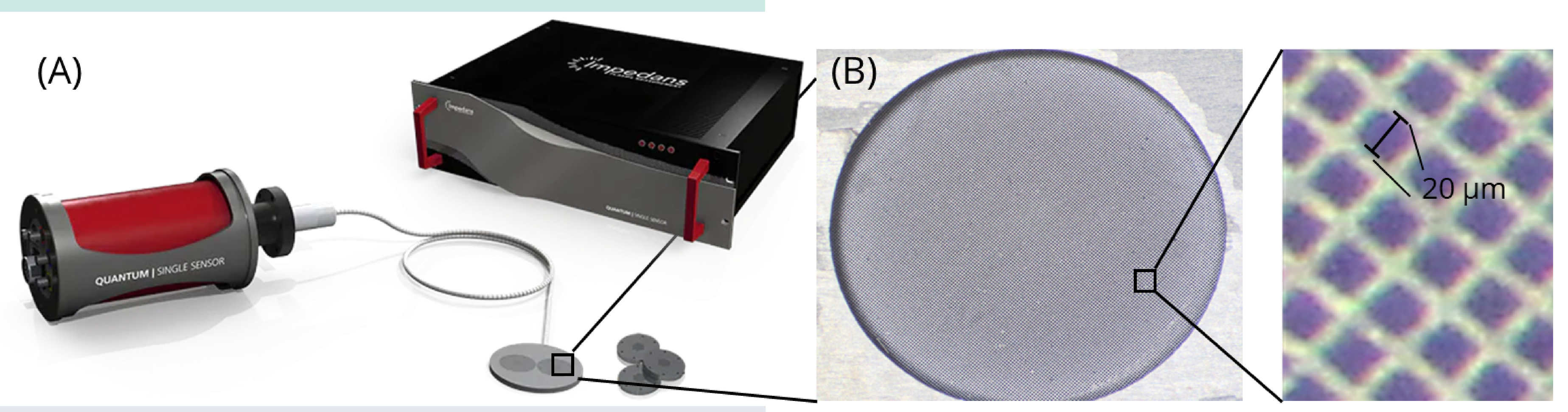


Figure 2: (A) Diagram of the Ti and MoS_2 sputter deposition system and (B) an image of the MoS_2 sputter process.



What Is an RFEA?

- Used industrially for "fingerprinting" – a method of transferring proven PVD 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

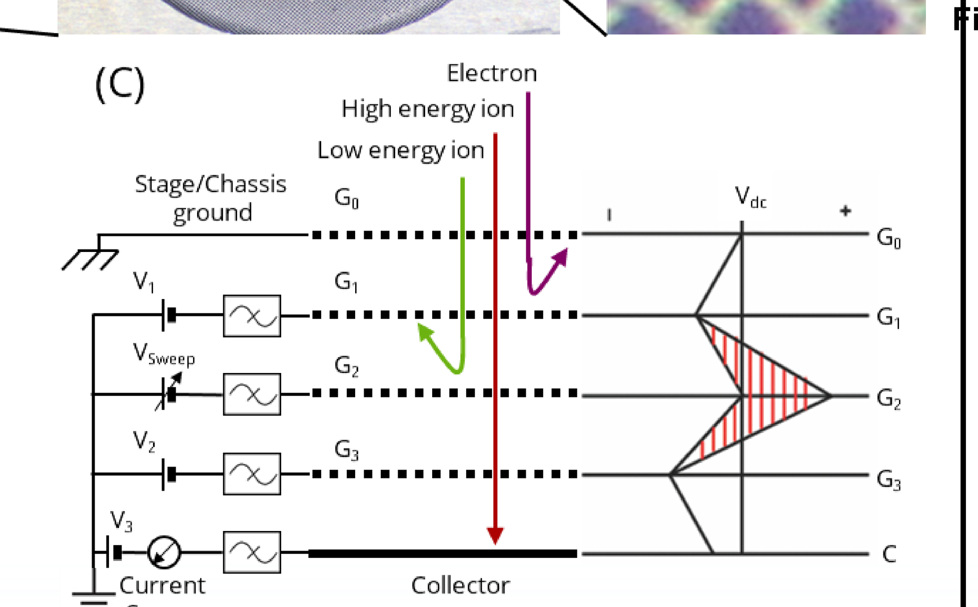


Figure 3: (A) Impedans Quantum diagnostic tool (B) image of the sensor puck with inset of G_0, (C) Diagram of grid layout, electronics and relative voltages.

Anatomy of An RFEA (Fig. 3C)

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

Results and Discussion

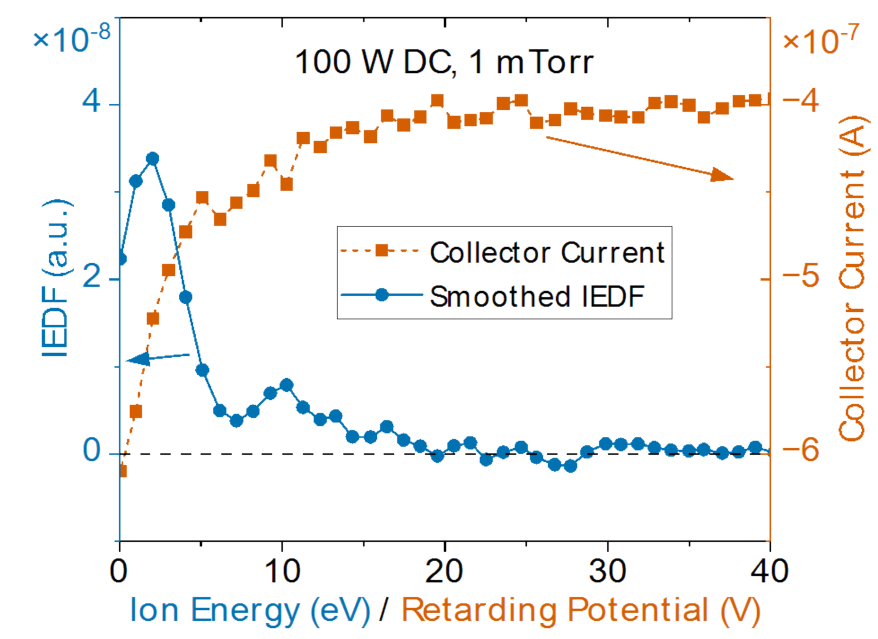


Figure 4: Collector current and ion energy distribution function (IEDF) for a 100 W DC sputter deposition at 1 mTorr. IEDF has been smoothed via Savitzky-Golay for clarity.

I-V Curves

- The "retarding potential" represents the potential 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.

Analysis

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

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

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.

Ion Energy Distribution

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

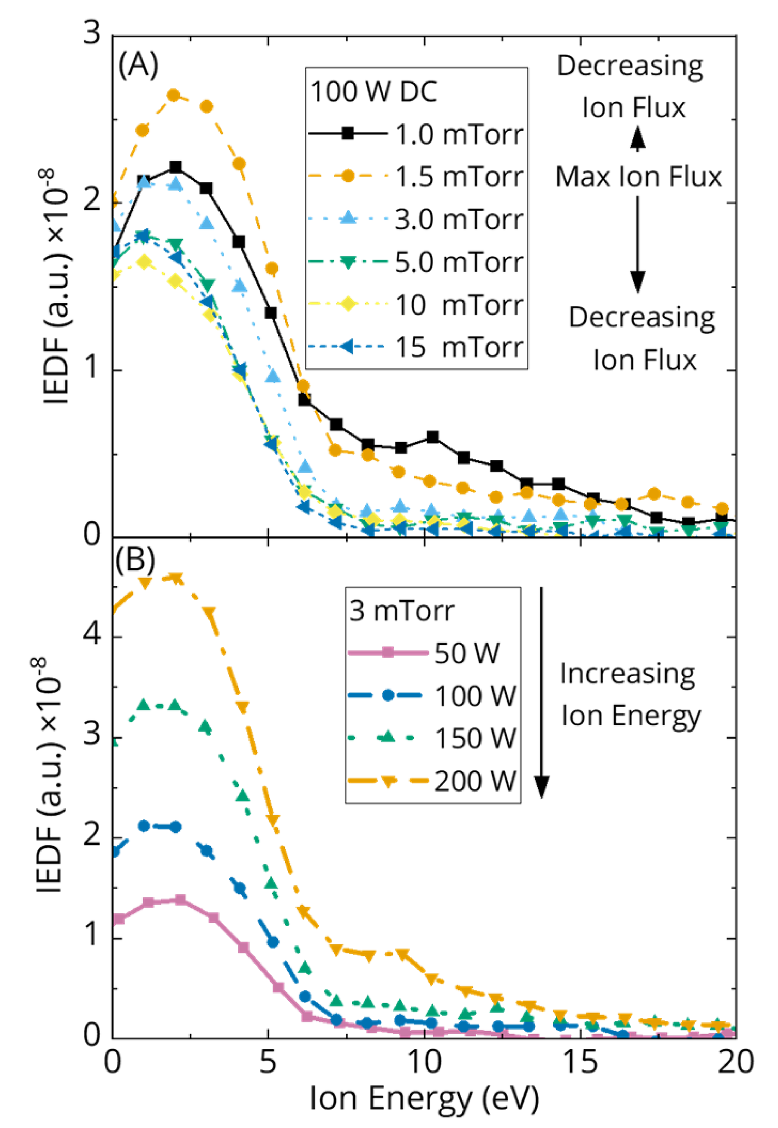


Figure 5: IEDF for (A) sputter pressure and (B) DC sputter power as a function of ion energy.

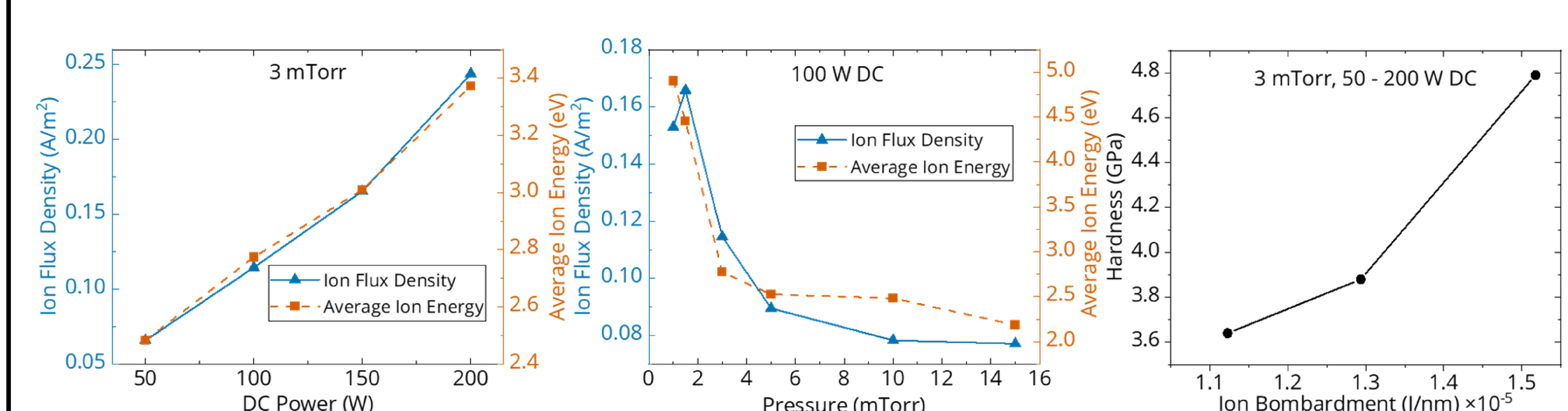


Figure 6: Ion flux density and average ion energy as a function of (A) DC sputter power and (B) sputter pressure, (C) thin film density and nano indentation hardness as a function of ion bombardment.

Average Ion Energy and Flux Density

- 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.

Connection To Materials Properties

- Figure 6C demonstrates the effect increased ion bombardment has on the hardness of deposited films. This is well established for ion beam assisted depositions[9] but has yet to be fully explored for sputter deposition.

Conclusions

- This study successfully employed in-situ characterization of MoS_2 deposition plasma using an RFEA.
- The results provide evidence that supports well established heuristics and simulation[8,10].
- When compared to film material properties, the hardness of studied films appear to correlate with ion bombardment – more materials characterization must be completed before this relationship can be fully explored.
- Future work will also focus on characterization of high energy plasma that employ substrate bias (i.e. PDC, HiPIMS etc.)

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