

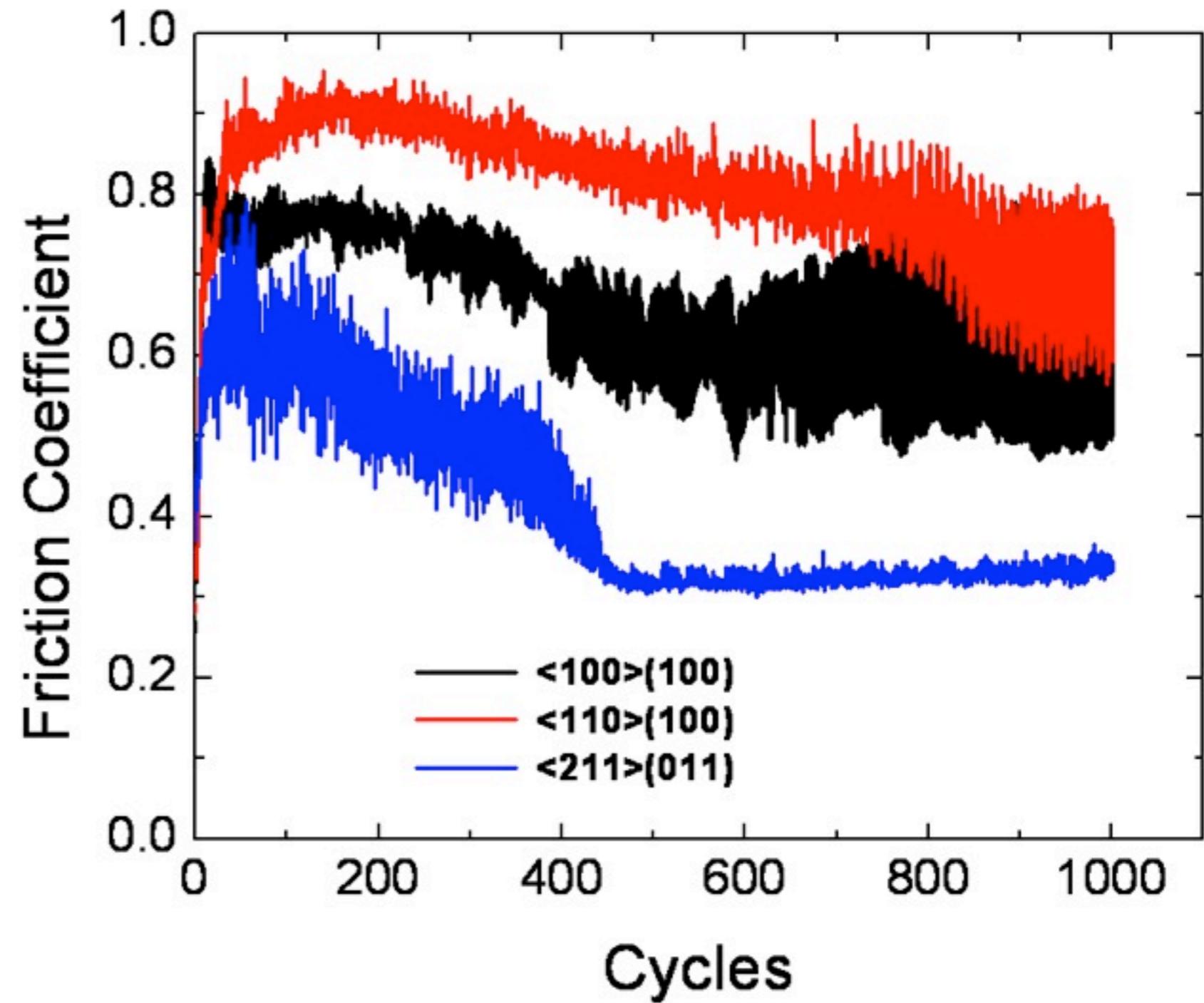
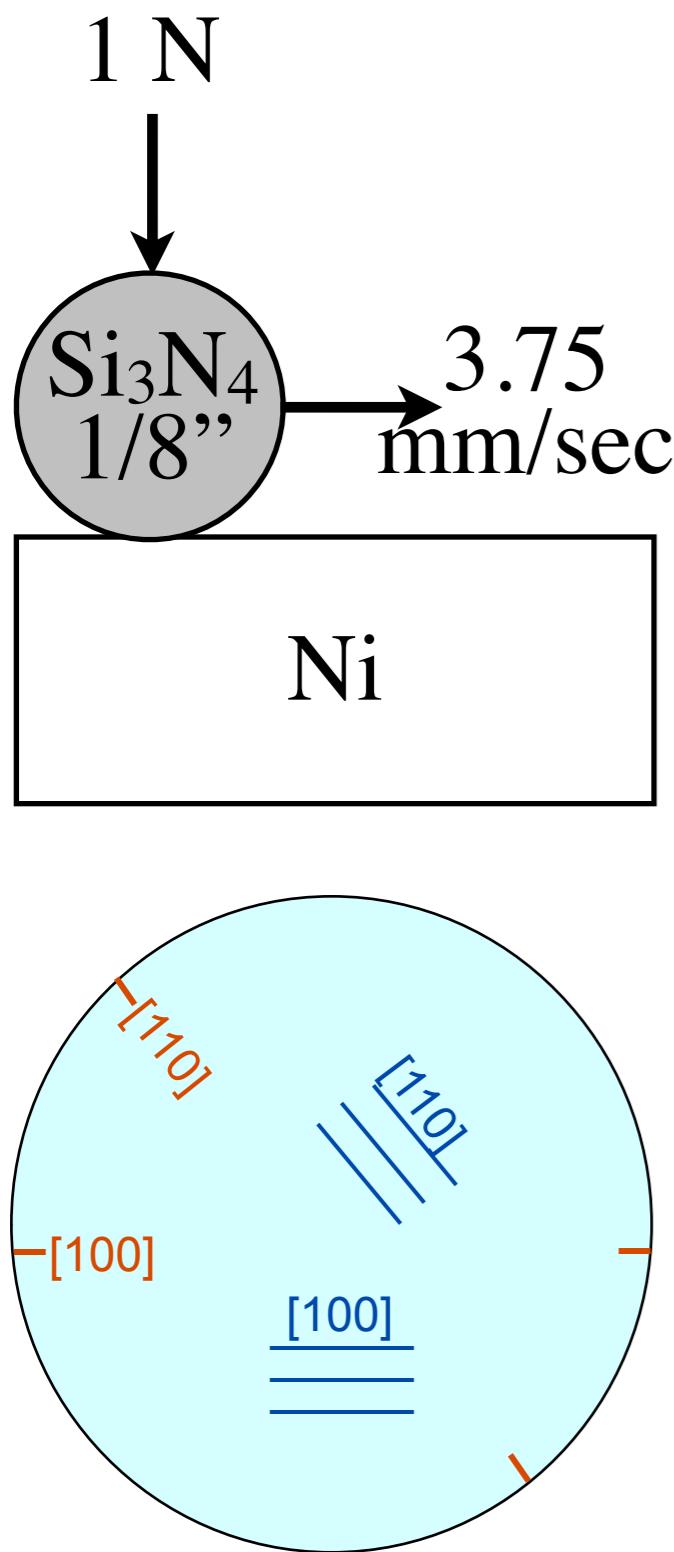


# Sub-Micro-Pillar Compression and Nanoindentation of Nanocrystalline Nickel Tribofilms

Corbett Battaile,  
Somuri Prasad, Joe Michael, Brad Boyce, Blythe Clark  
Sandia National Laboratories  
Albuquerque NM

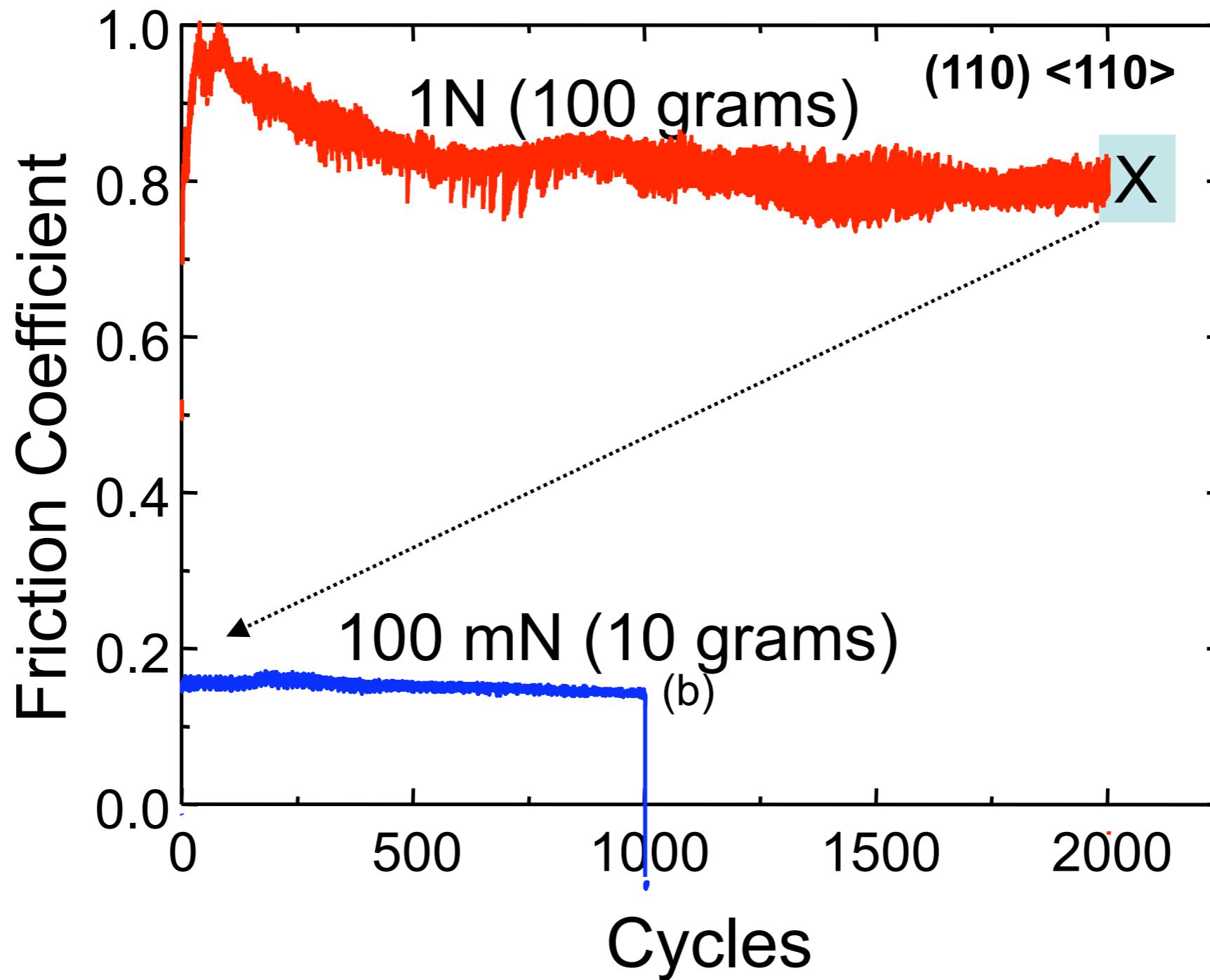
ICMCTF 2011, San Diego CA

# Prior Experiments: Friction Coefficient

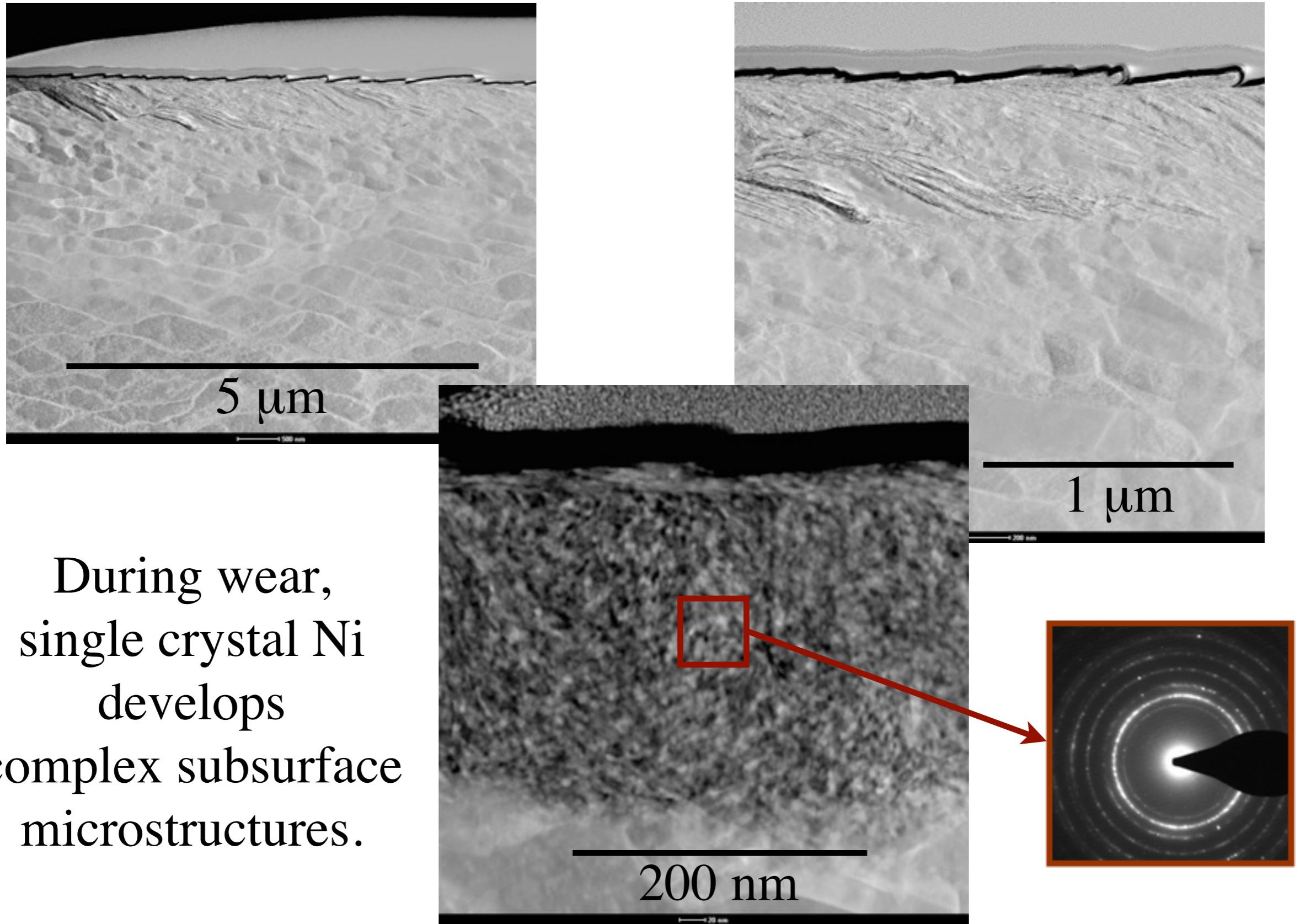


# Prior Experiments: Friction Coefficient

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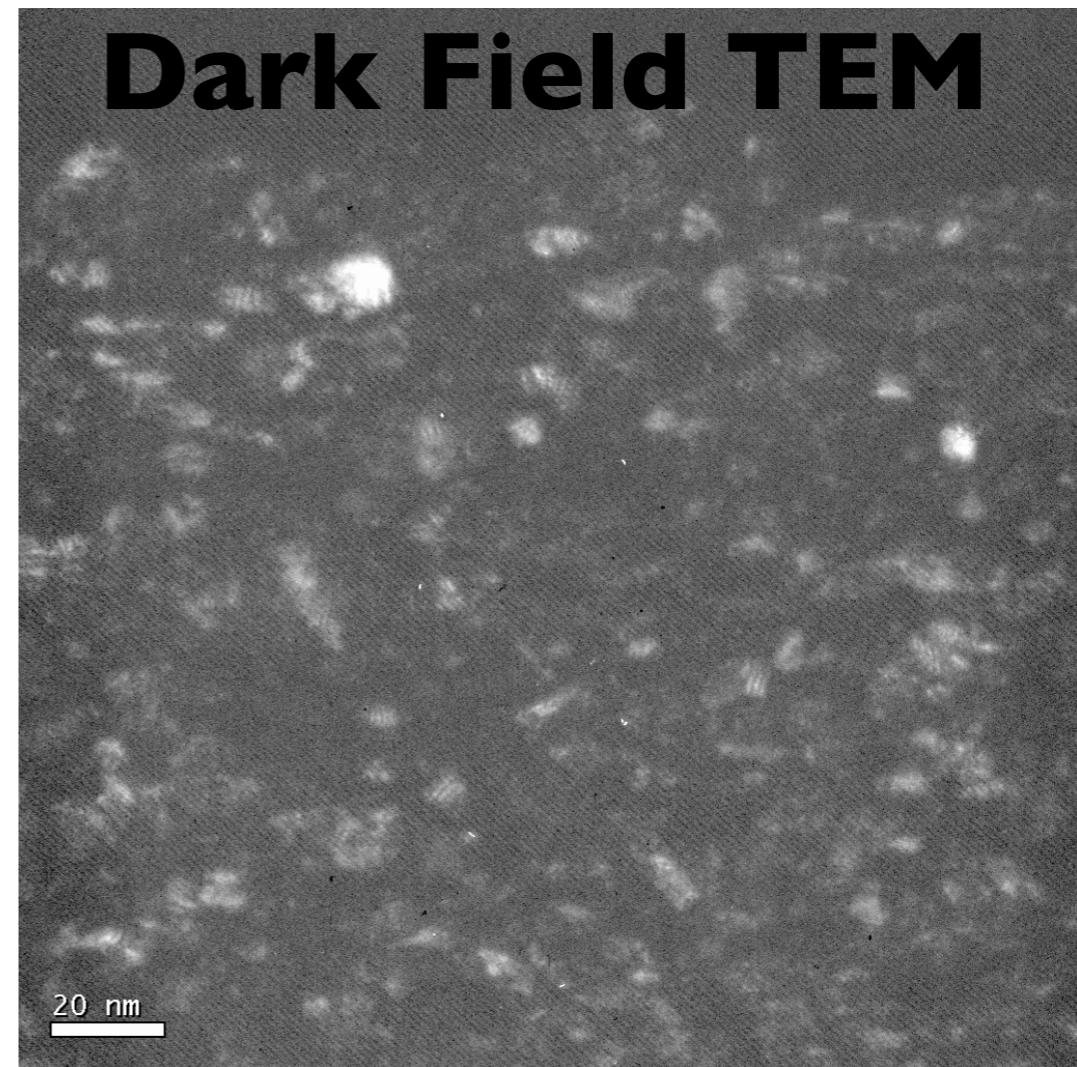
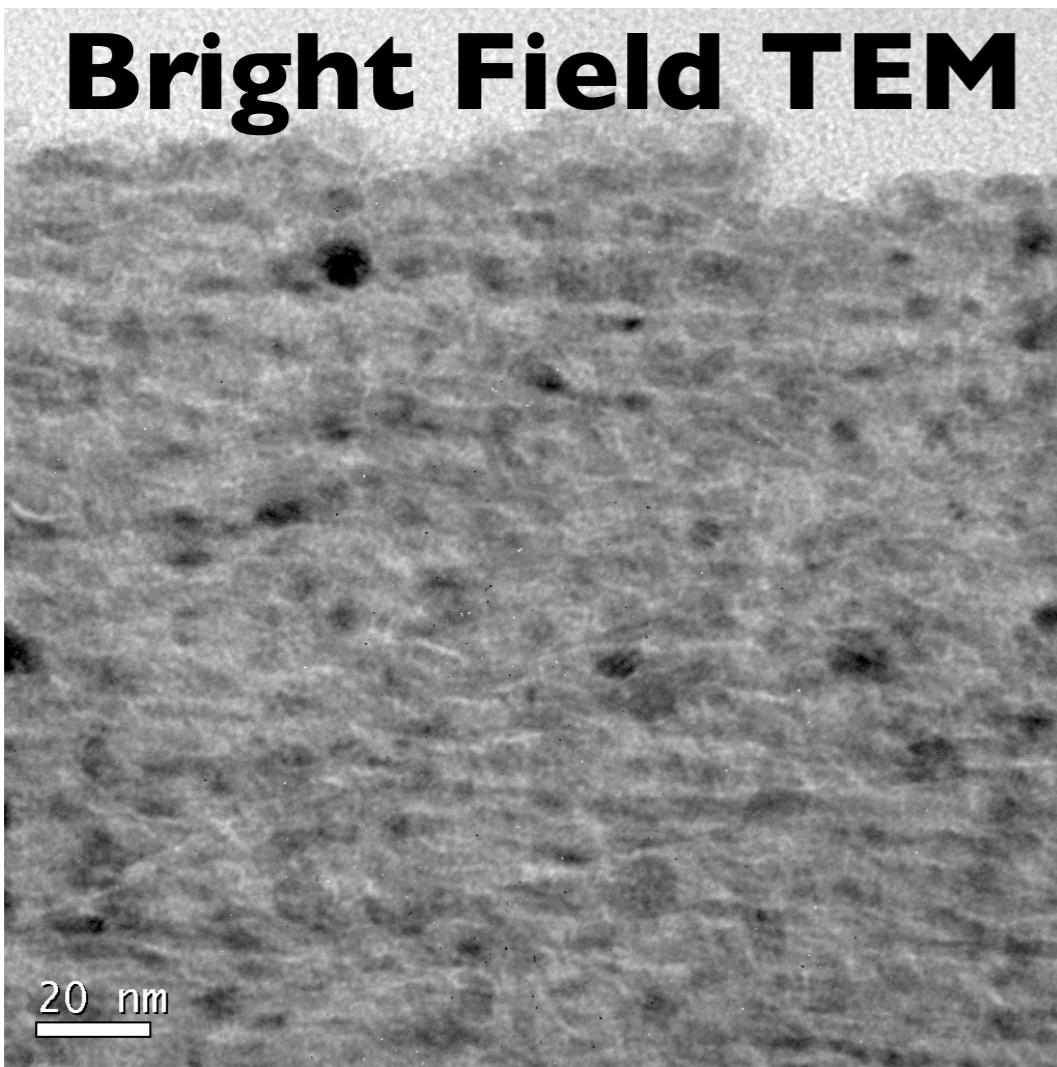
# Prior Experiments: TEM



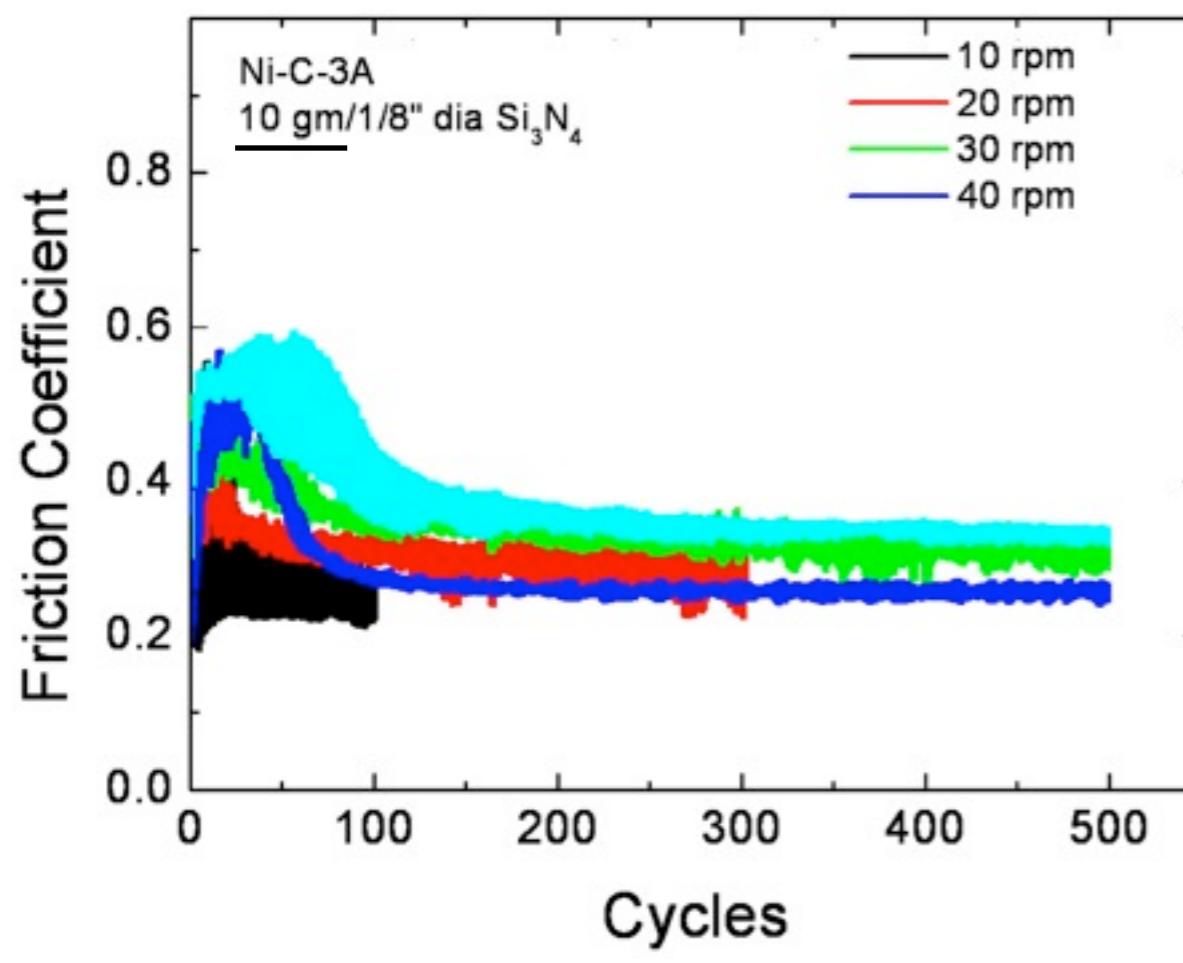
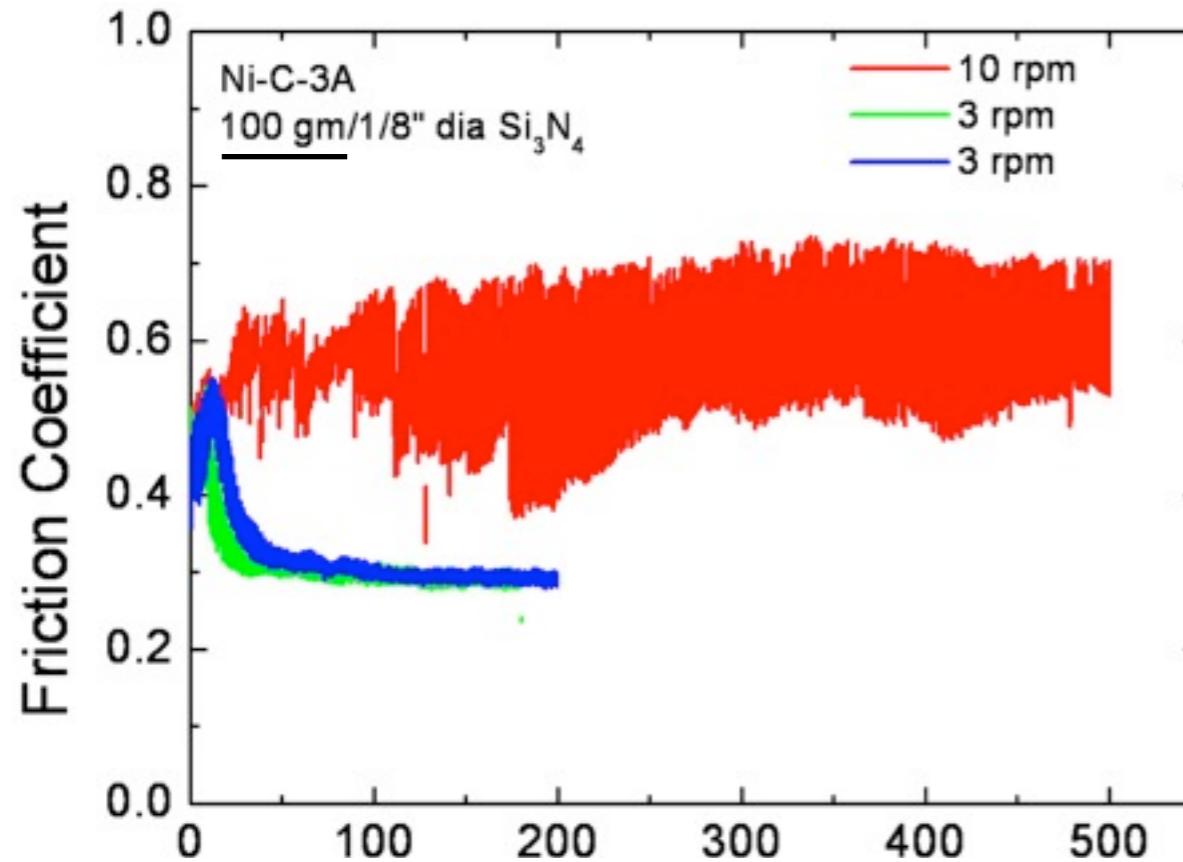
# Prior Experiments: TEM

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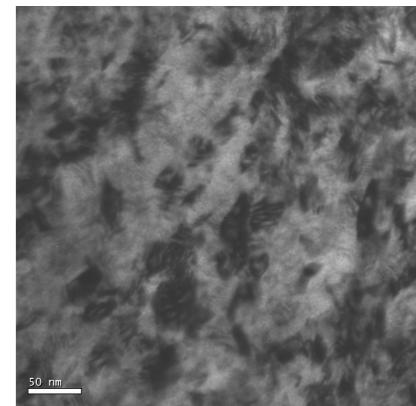
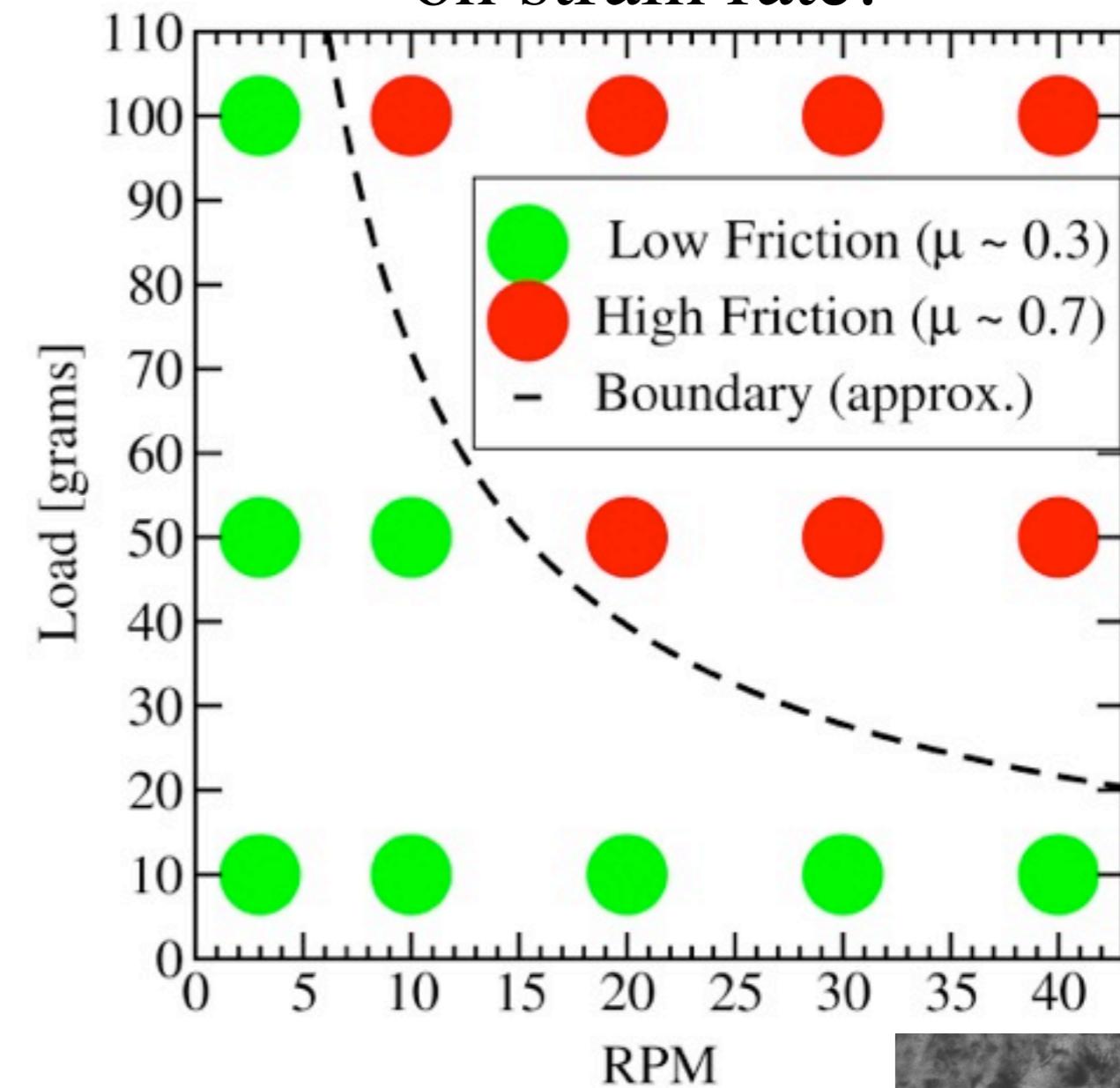
Higher magnification TEM images show  
very small (< 10 nm) grains.



# Prior Experiments: Strain Rate Dependence

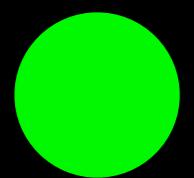


Transition to low friction shows a clear dependence on strain rate.



# Prior Experiments: More TEM

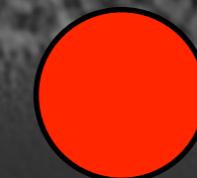
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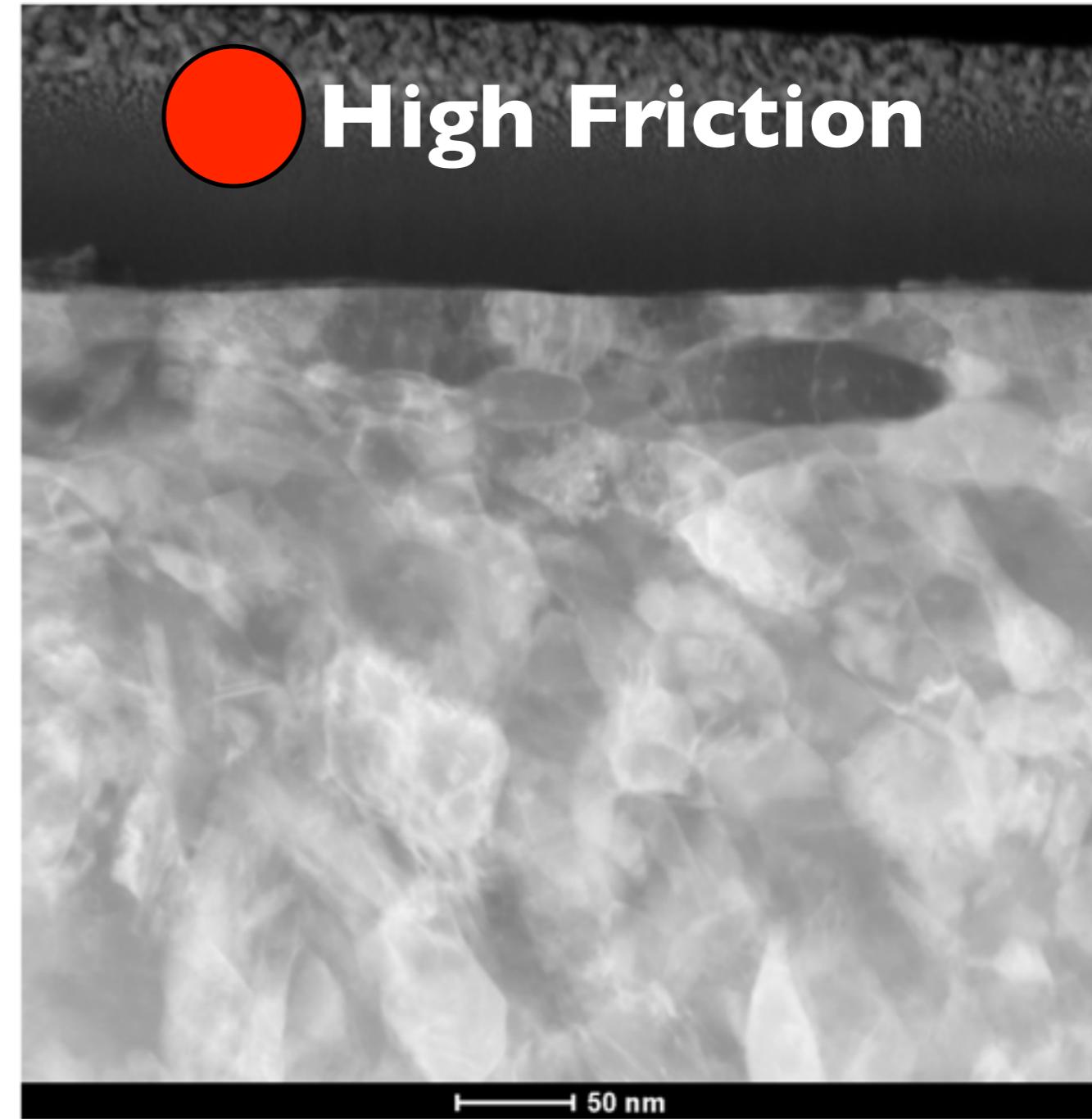
**Low Friction**



100 g, 3 RPM



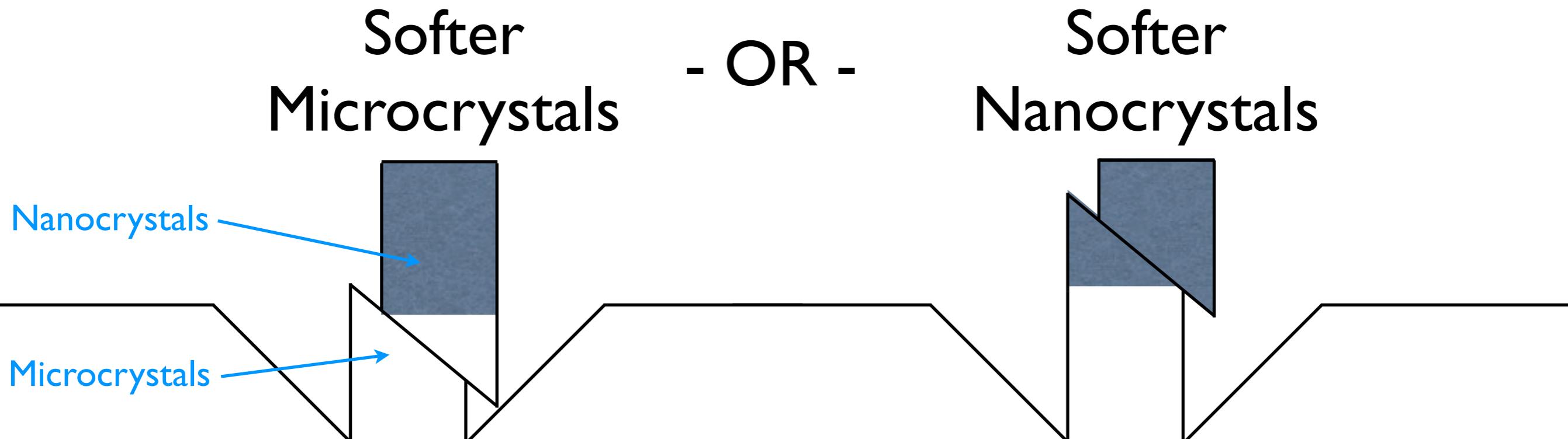
**High Friction**



100 g, 20 RPM

# Direct Characterization of Mechanical Properties?

- Use pillar compression and nanoindentation to directly determine the mechanical properties of the surface material.



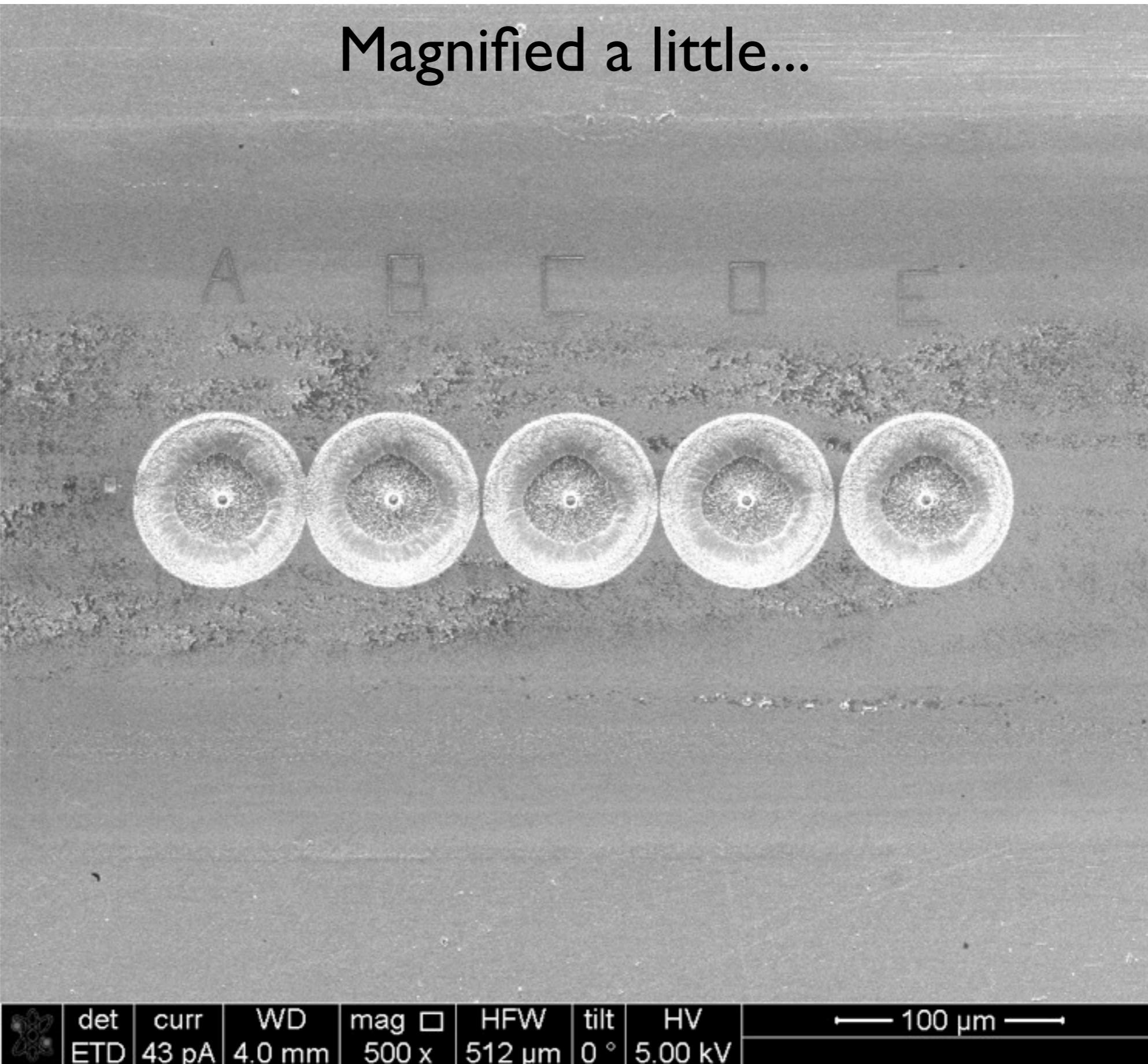
# FIB Milling of Sub-Micro-Pillars in Wear Track

FIB craters in a wear track on single-crystal nickel. Each crater contains a pillar atop a pedestal.

	det	curr	WD	mag	□	HFW	tilt	HV	400 µm
ETD	43 pA	4.0 mm	200 x		1.28 mm	0 °	5.00 kV		

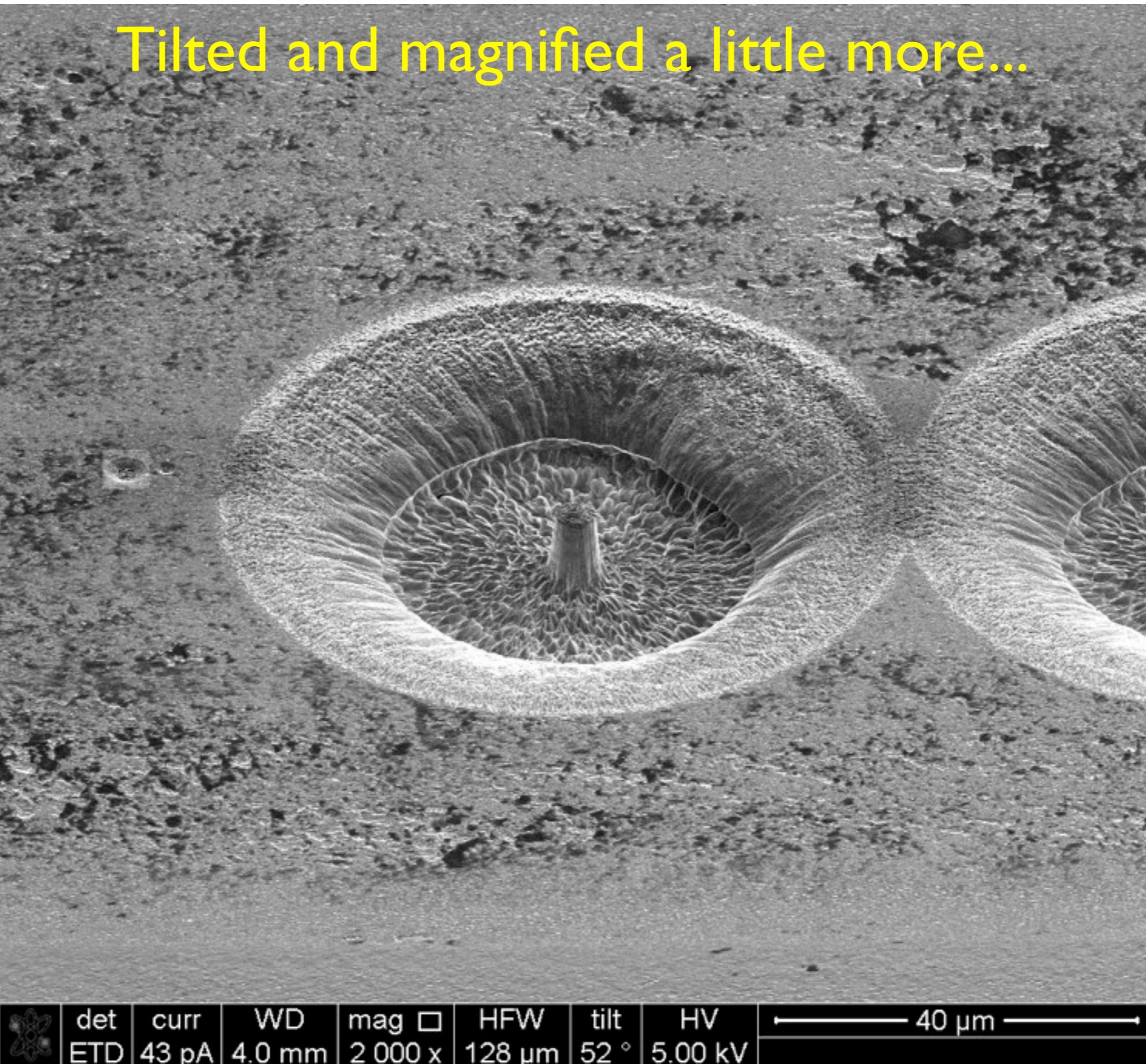
# FIB Milling of Sub-Micro-Pillars in Wear Track

Magnified a little...



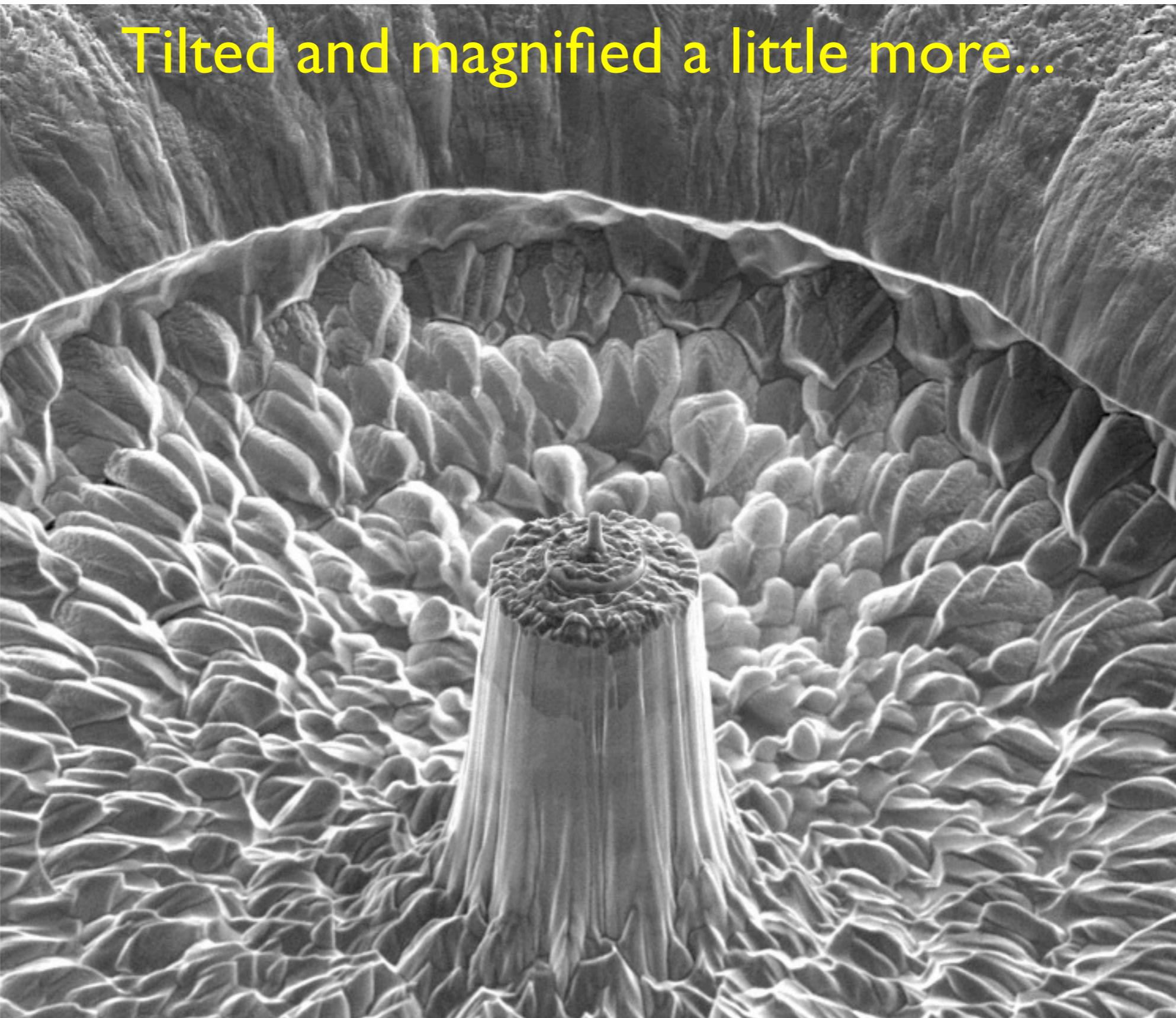
# FIB Milling of Sub-Micro-Pillars in Wear Track

Tilted and magnified a little more...



# FIB Milling of Sub-Micro-Pillars in Wear Track

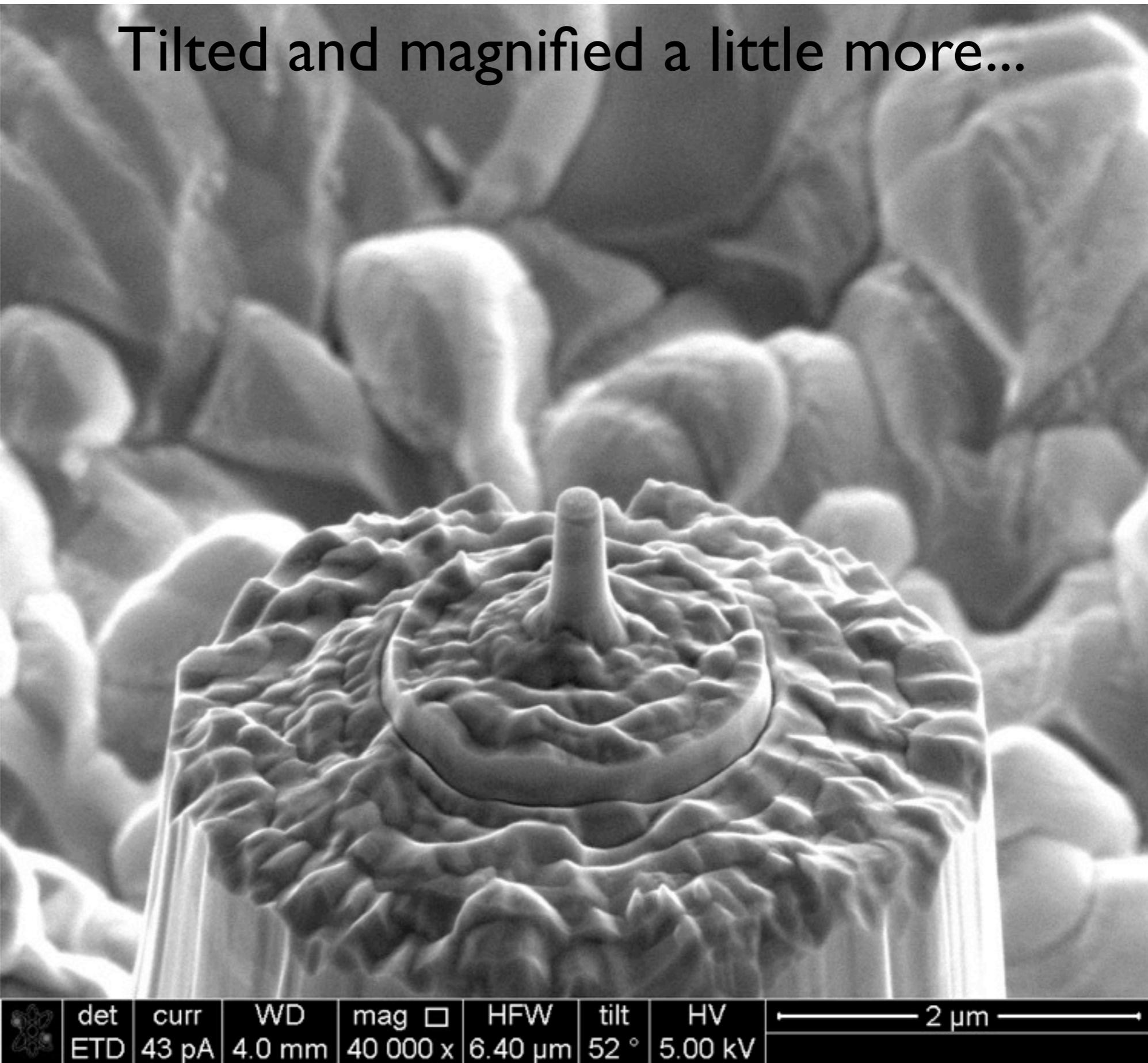
Tilted and magnified a little more...



	det	curr	WD	mag	□	HFW	tilt	HV	5 μm
ETD	43 pA	4.0 mm	10 000 x		25.6 μm	52 °	5.00 kV		

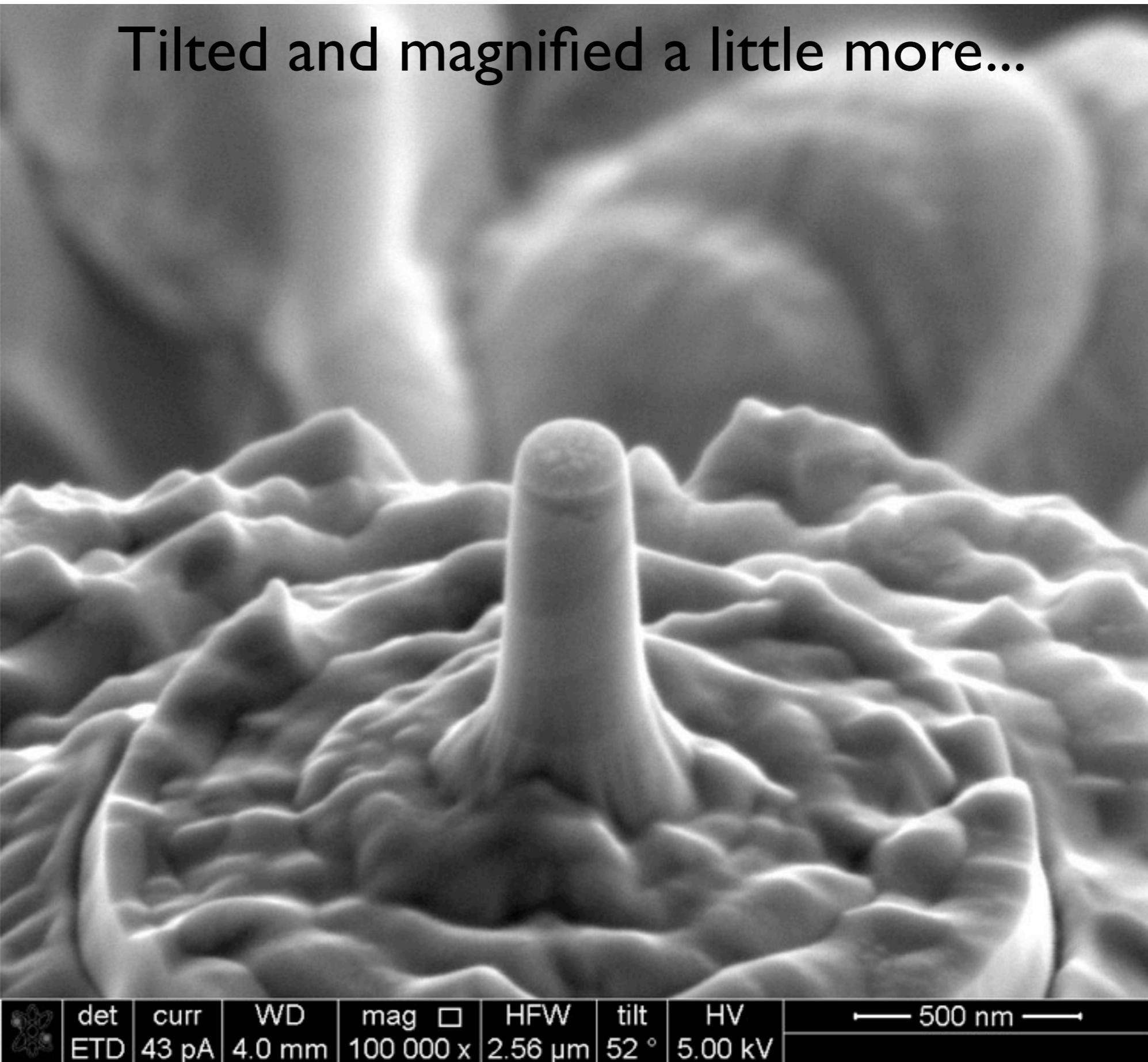
# FIB Milling of Sub-Micro-Pillars in Wear Track

Tilted and magnified a little more...



# FIB Milling of Sub-Micro-Pillars in Wear Track

Tilted and magnified a little more...



det

curr

WD

mag

□

HFW

tilt

HV

— 500 nm —

ETD

43 pA

4.0 mm

100 000 x

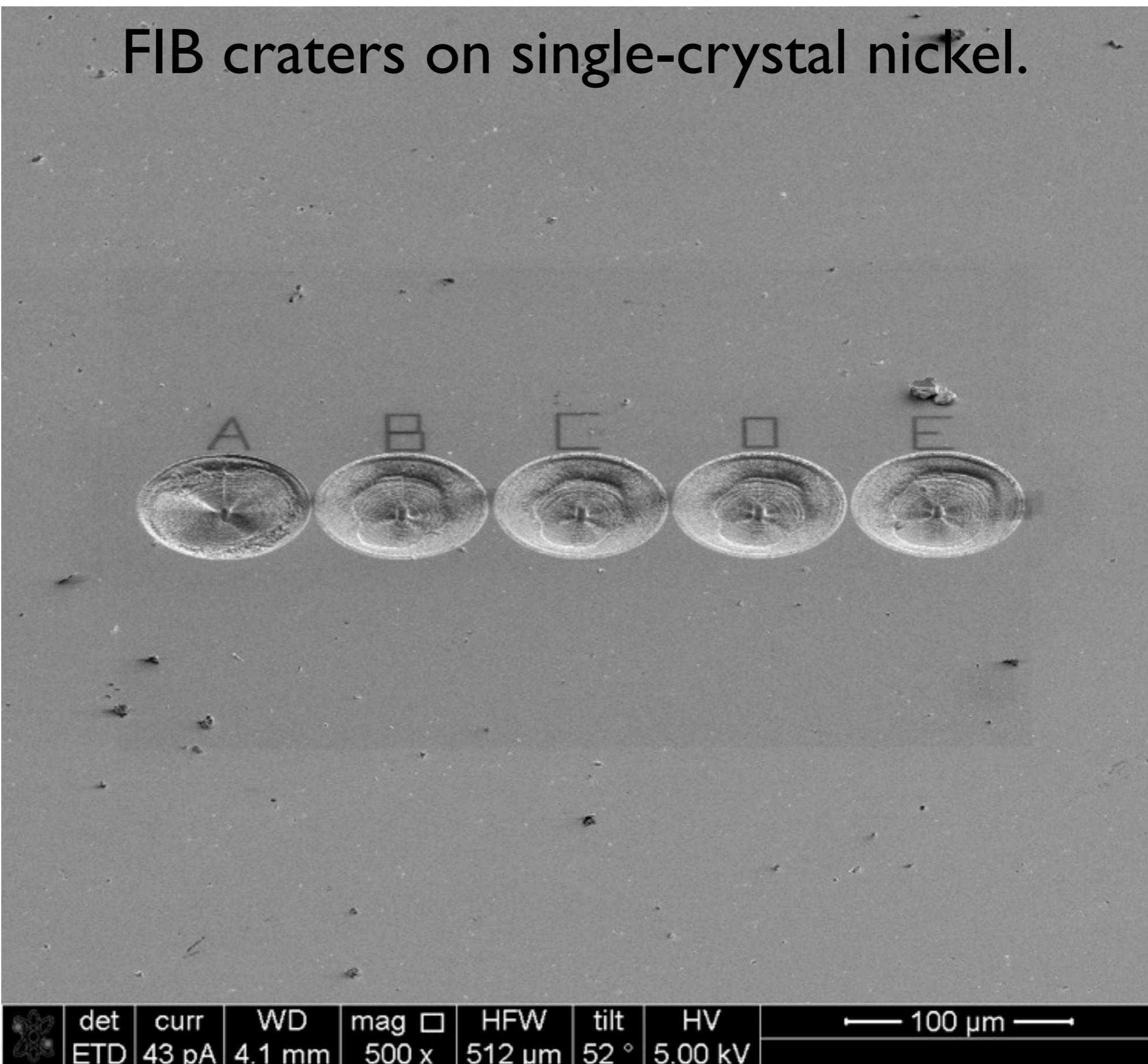
2.56 μm

52 °

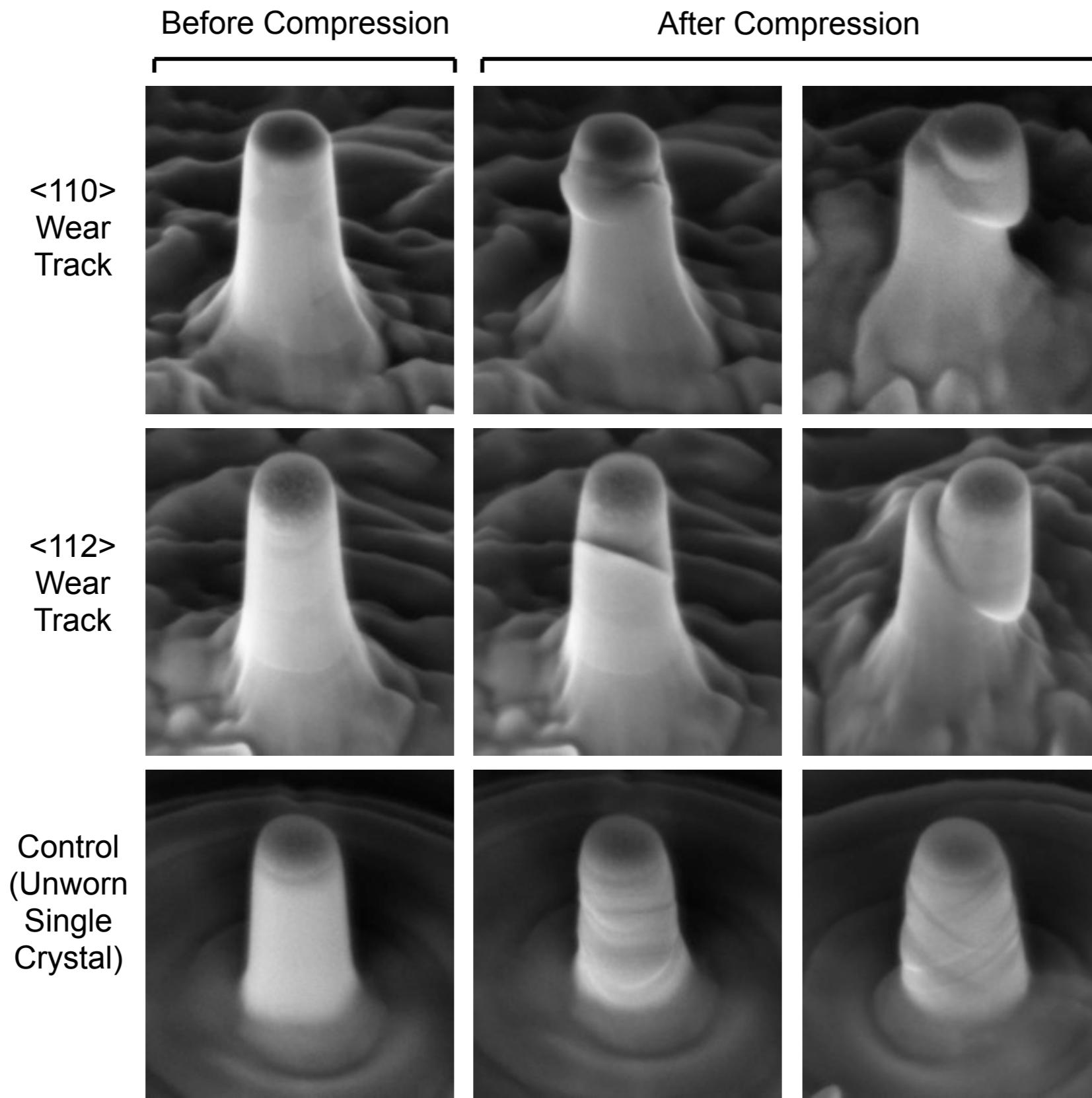
5.00 kV

# FIB Milling of Sub-Micro-Pillars in Single Xtal

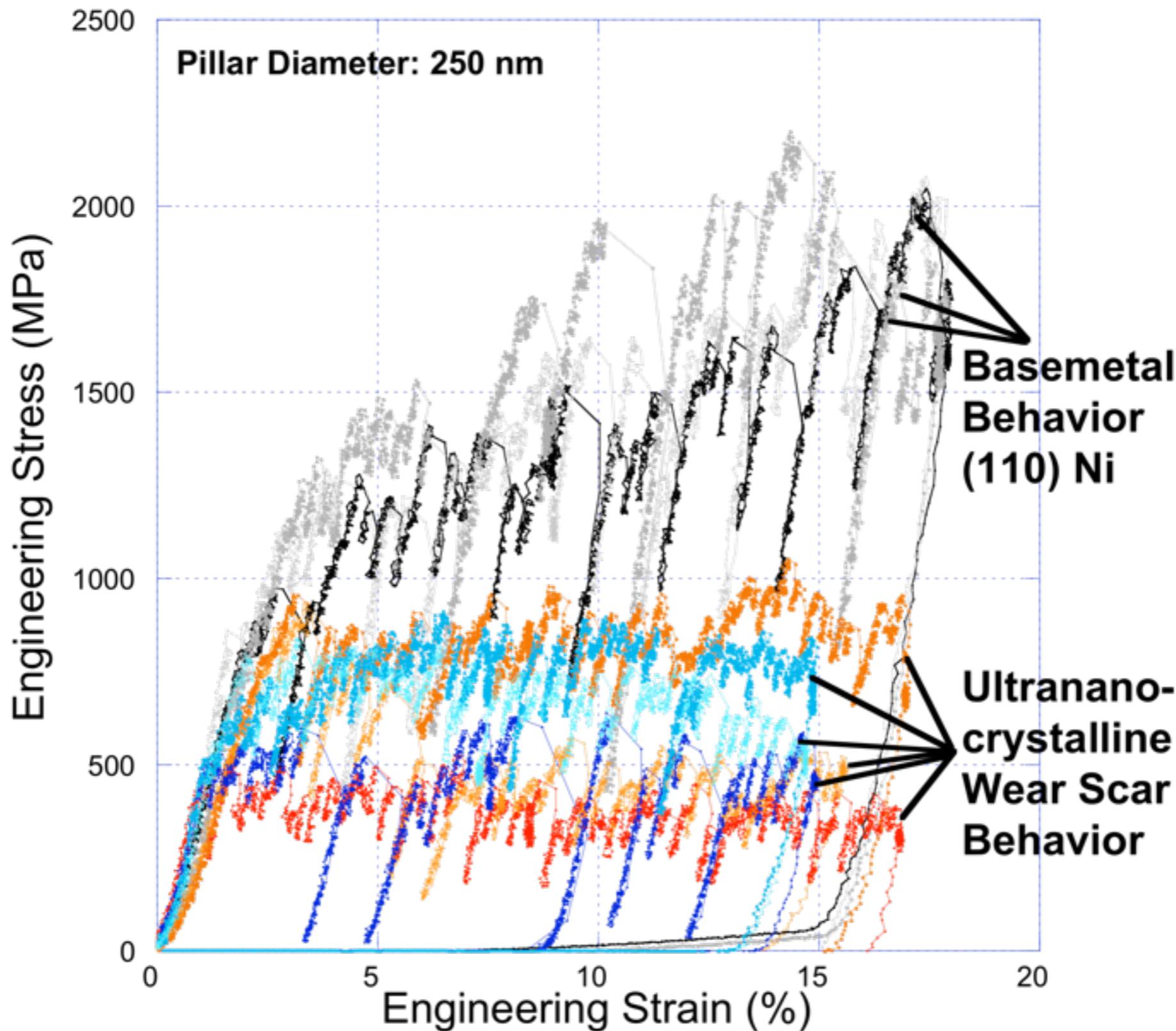
FIB craters on single-crystal nickel.



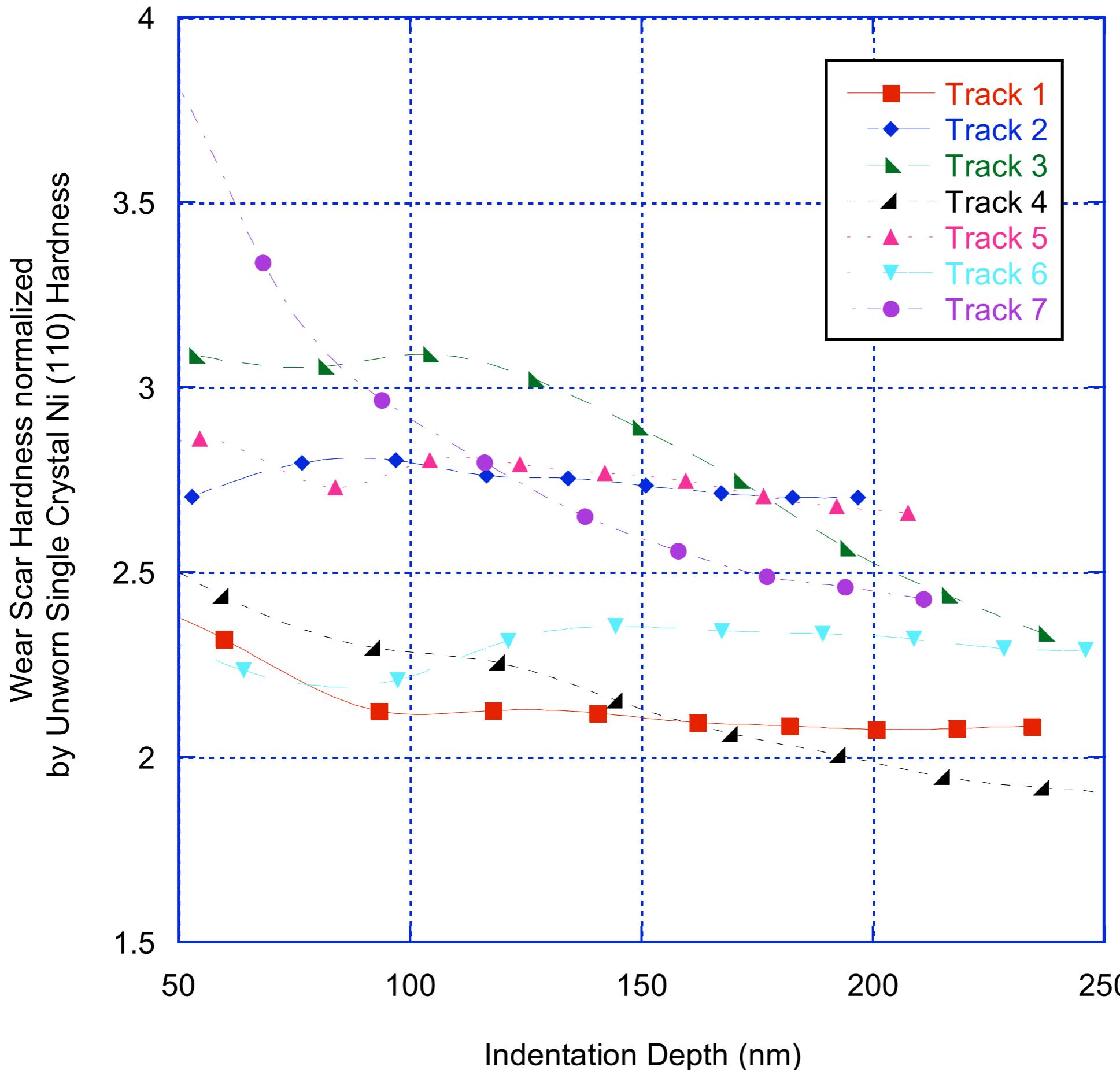
# Pillar Compression Testing



# Pillar Compression Testing

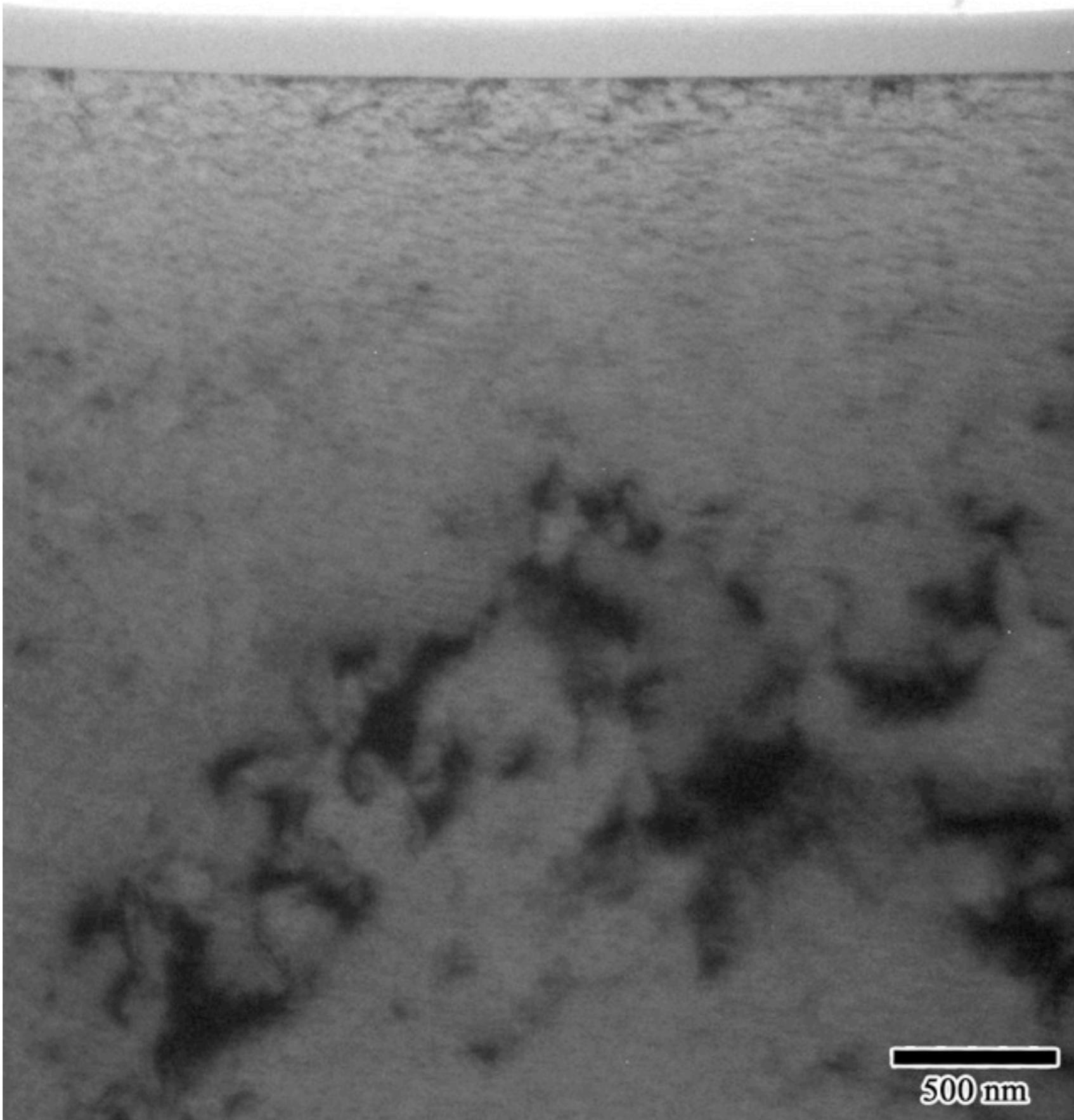


# Nanoindentation Testing

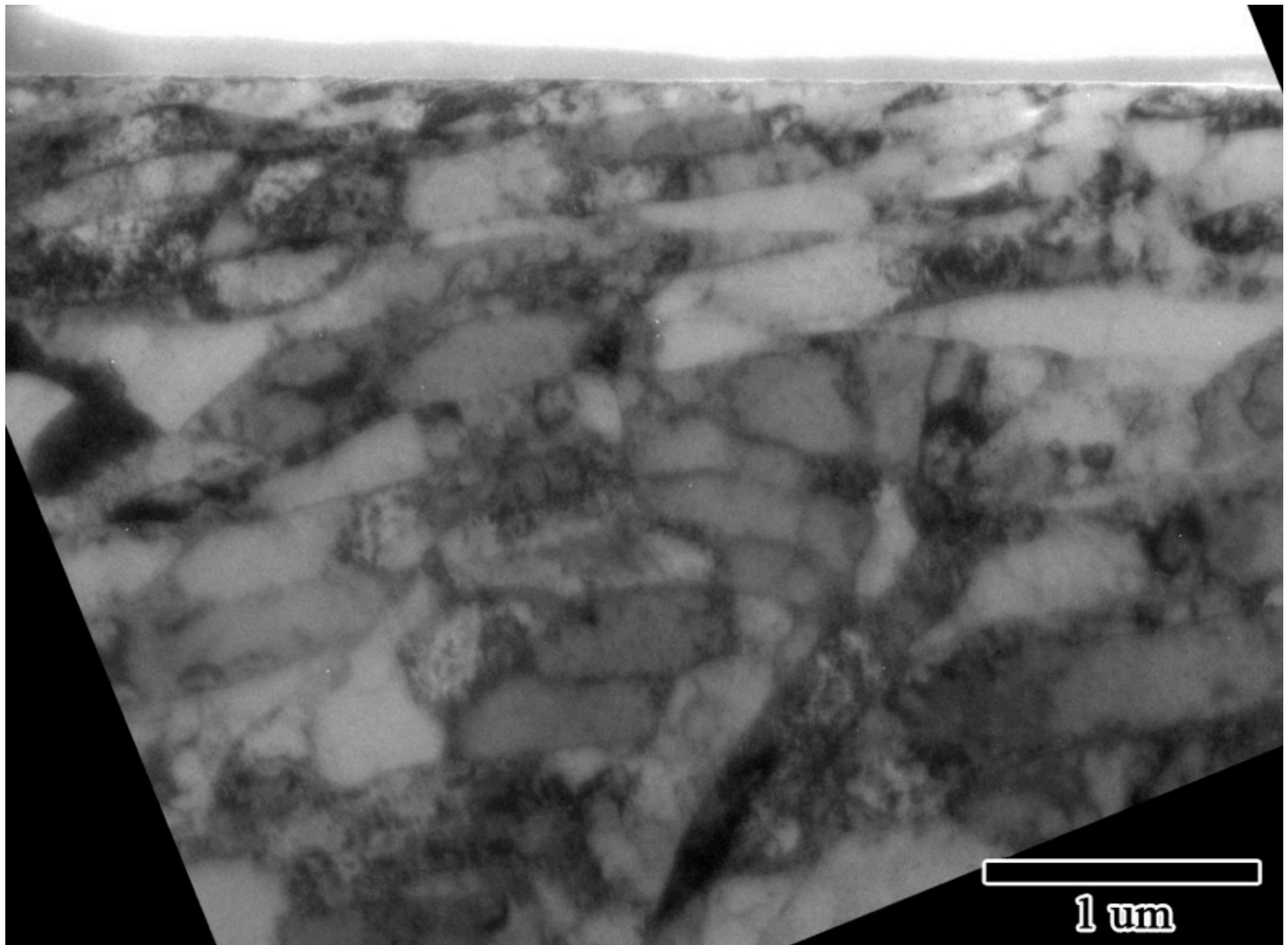


# TEM of Single Crystal

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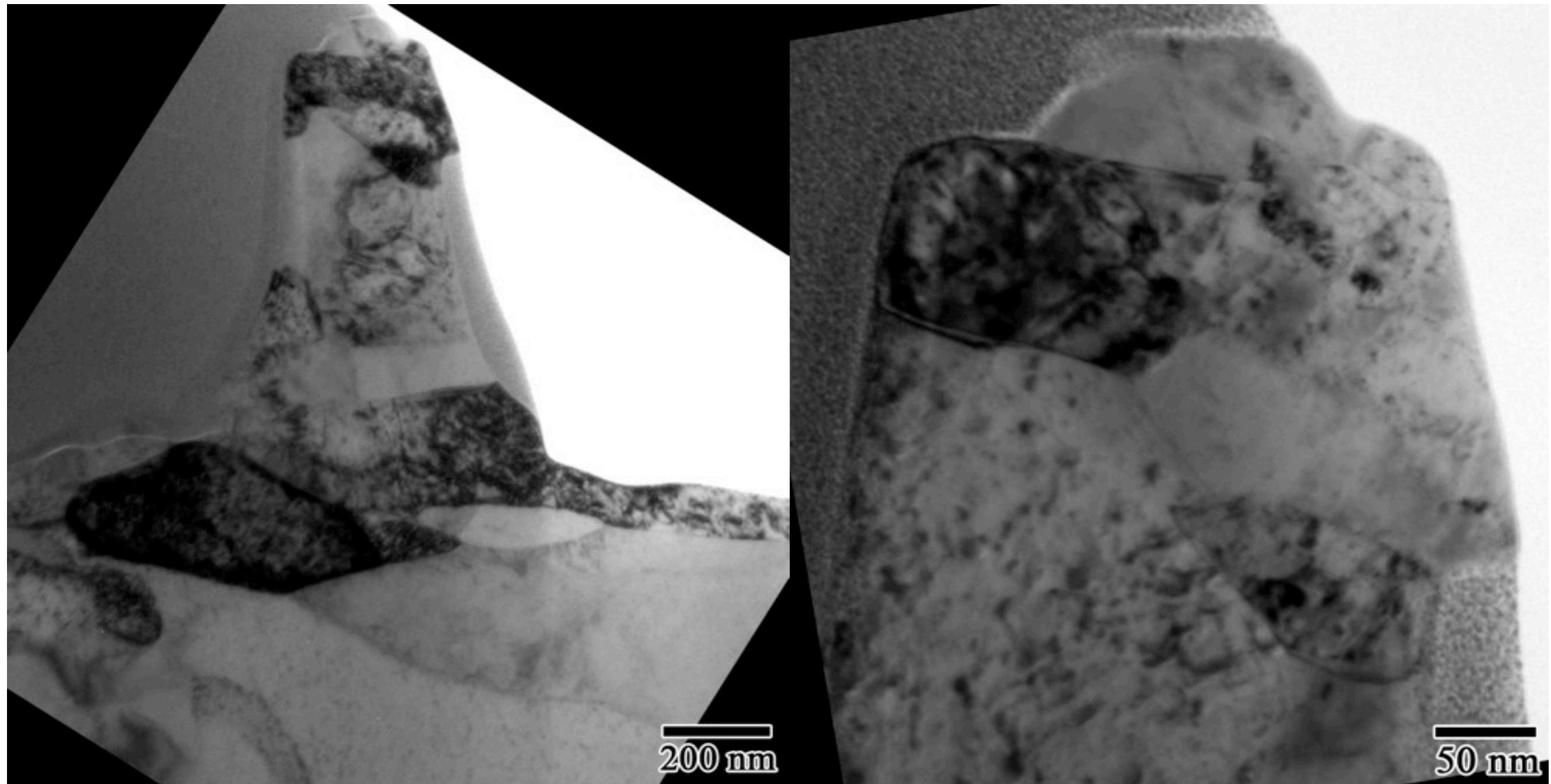


# TEM of Wear Track #1



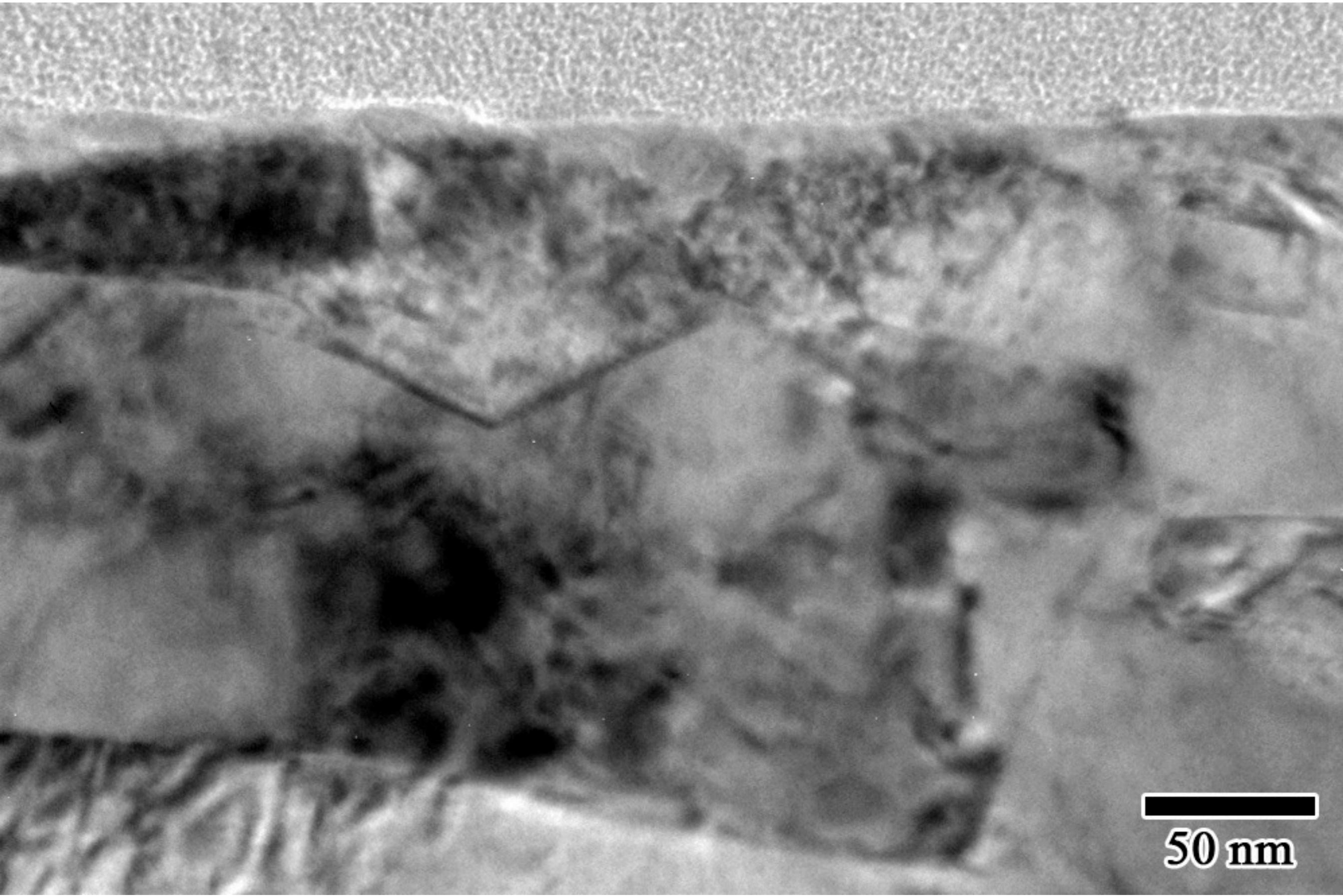
# TEM of Wear Track #1 Pillar B

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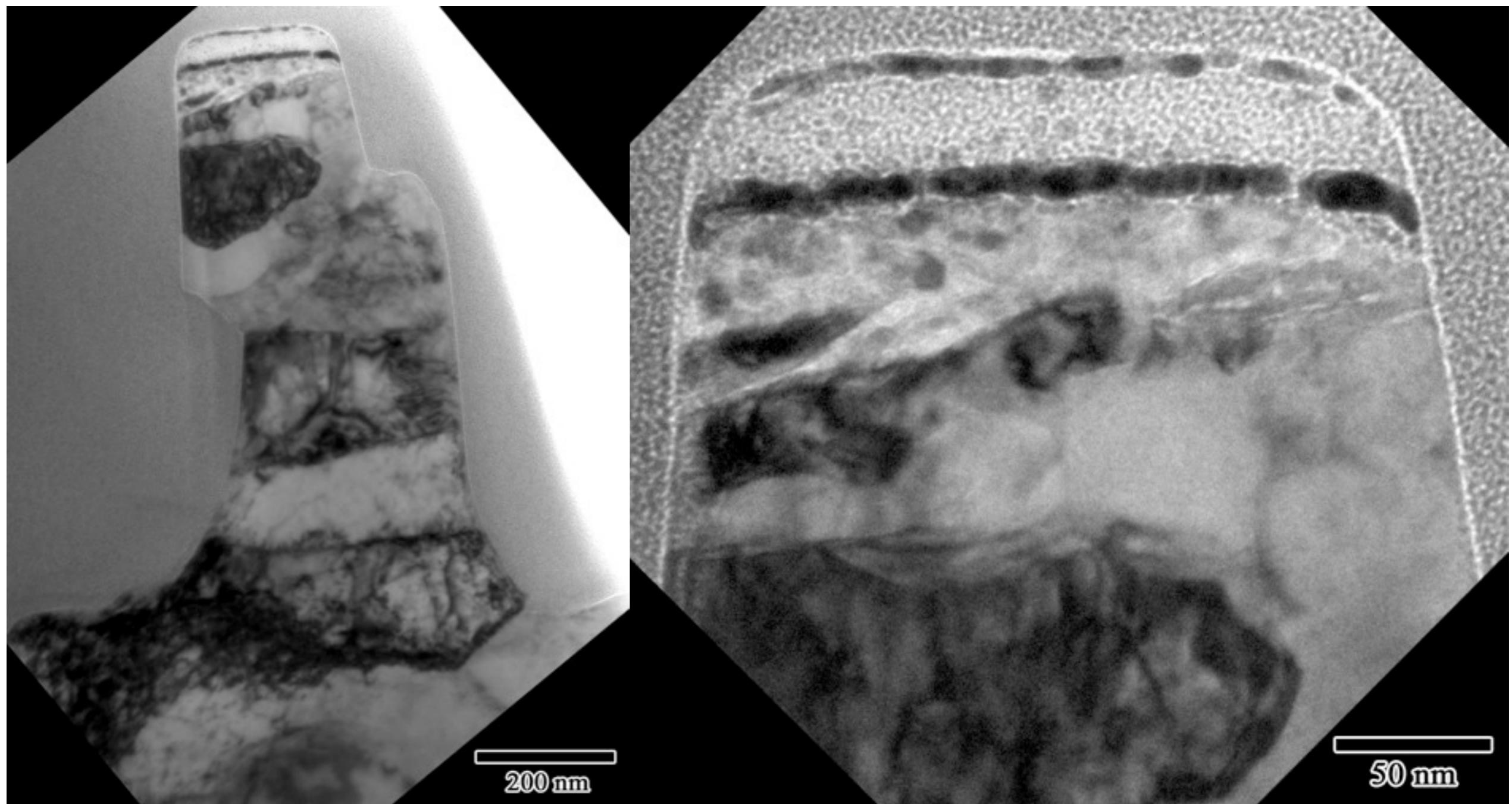


# TEM of Wear Track #5

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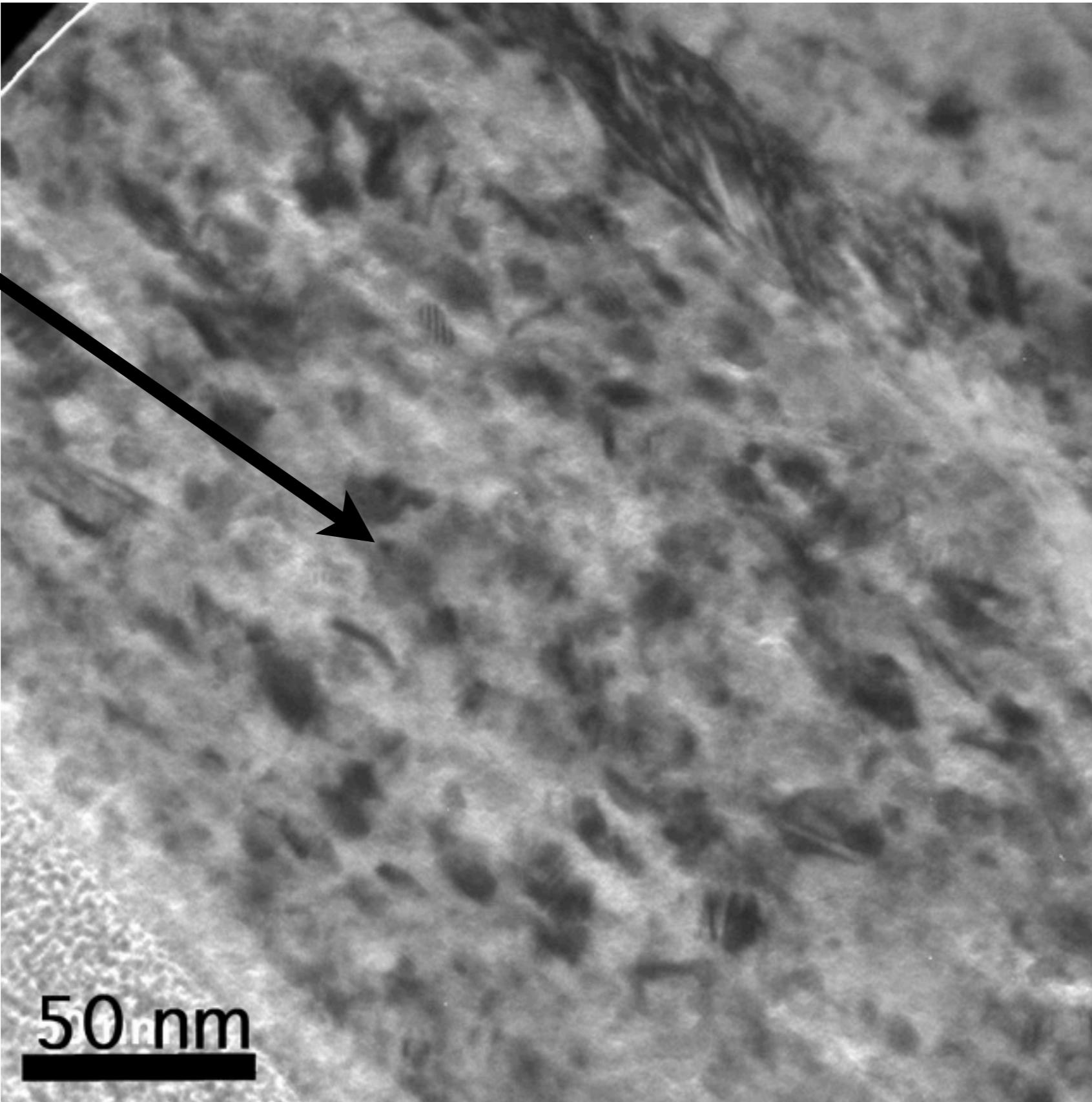
# TEM of Wear Track #5 Pillar D



# TEM of Wear Track #7 (untested)

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UNC  
layer!



# Summary and Conclusions

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- Prior experiments on single-crystal nickel suggest **crystallography-** and **rate-dependent friction** and wear behavior related to **subsurface nanostructure formation**.
- Focused ion beam milling was used to create sub-micro-pillars inside wear tracks and on the parent, single-crystal nickel surface.
- Pillar **compression tests** indicate that the pillars are significantly **softer** than the parent, single crystal.
- **Nanoindentation tests** indicate that the pillars are **100% to 300% harder** than the parent, single crystal.
- Transmission electron microscopy shows that none of the tested wear tracks developed an ultra-nanocrystalline surface layer. Friction coefficients were high (0.5 - 0.8) in all cases.
- Nanoindentation shows harder wear tracks because grain boundaries inhibit dislocation motion. Pillar compression shows softer tracks because free surfaces allow dislocations to escape. Nanocrystalline pillars don't work harden because grain boundaries provide ready sources for dislocation nucleation.

# Acknowledgements and Thanks

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- Rand Garfield (tribology)
- Michael Rye (focused ion beam)
- Bonnie McKenzie (scanning electron microscopy)
- Chris Weinberger (insightful discussions)