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# Gradient Microstructures in Single Crystals Induced by Sliding Contact (Invited)

S. V. Prasad\*, J. R. Michael, P. G. Kotula, C. C Battaile

Material, Physical, & Chemical Sciences Center

Sandia National Laboratories, Albuquerque, NM 87185

[svprasa@sandia.gov](mailto:svprasa@sandia.gov)

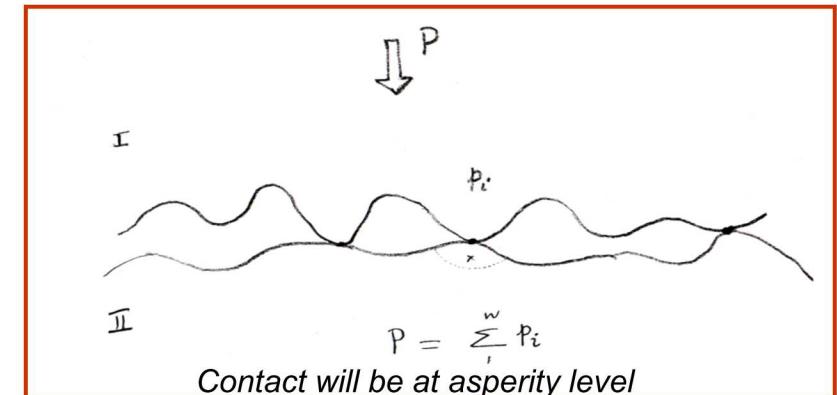
B. S. Majumdar

New Mexico Institute of Mining and Technology, Socorro, NM 87801

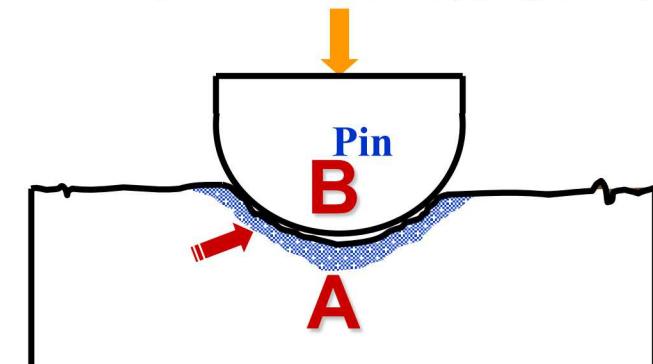
## Historical Background

- Sir George Beilby (1900-1921):
  - Top layer of the polished surface (*Glassy*) is different in structure from the underlying metal.
    - Sir George Beilby, *Aggregation and flow of solids; Being the records of an experimental study of the micro-structure and physical properties of solids in various states of aggregation*, Macmillan & co, London, 1921.
  - Controversy on the “Beilby” layer persisted for a few decades, mainly due to limitations with XRD to characterize the near surface layers, until the advent of electron diffraction, G. P. Thompson, *Proc. Roy. Soc. A*128 (1930) 649.
- TEM and ED studied in the late 1930’s on Au films revealed diffuse rings, which confirmed the existence of very fine crystal grains in the polished layer.
  - W. Cochrane, *Proc. Roy. Soc. A*160 (1938) 228.
- This concept has found widespread applications, ranging from honing in internal combustion engines to metal forming operations, including ECAP.

Engineering surfaces are not flat



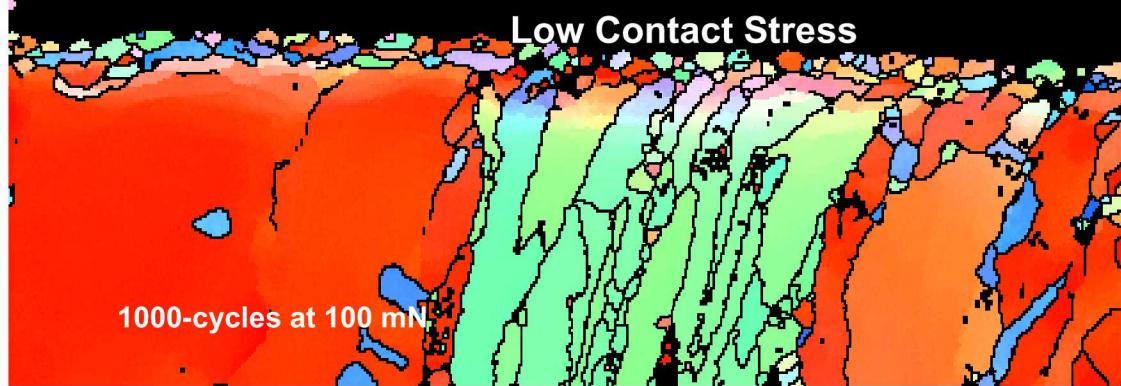
Archard, J. F. *J. Appl. Phys.* **24** (1953) 981  
Holm, R. *Electrical Contacts* (Springer, 1946)



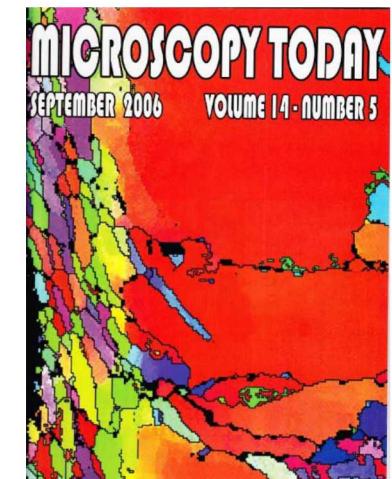
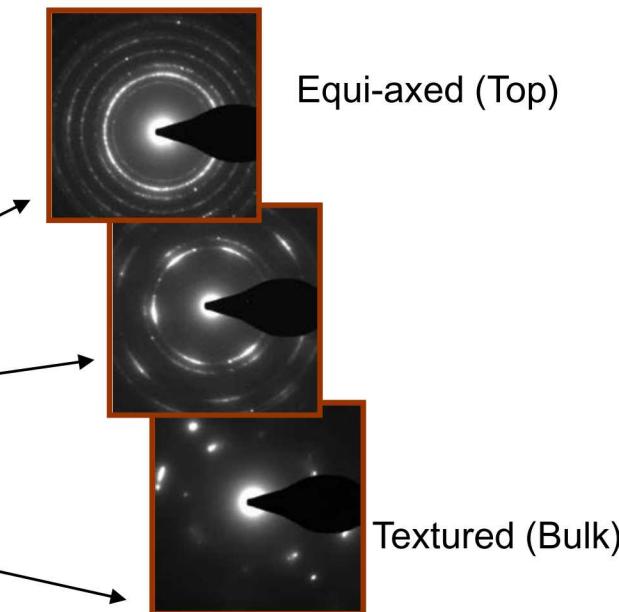
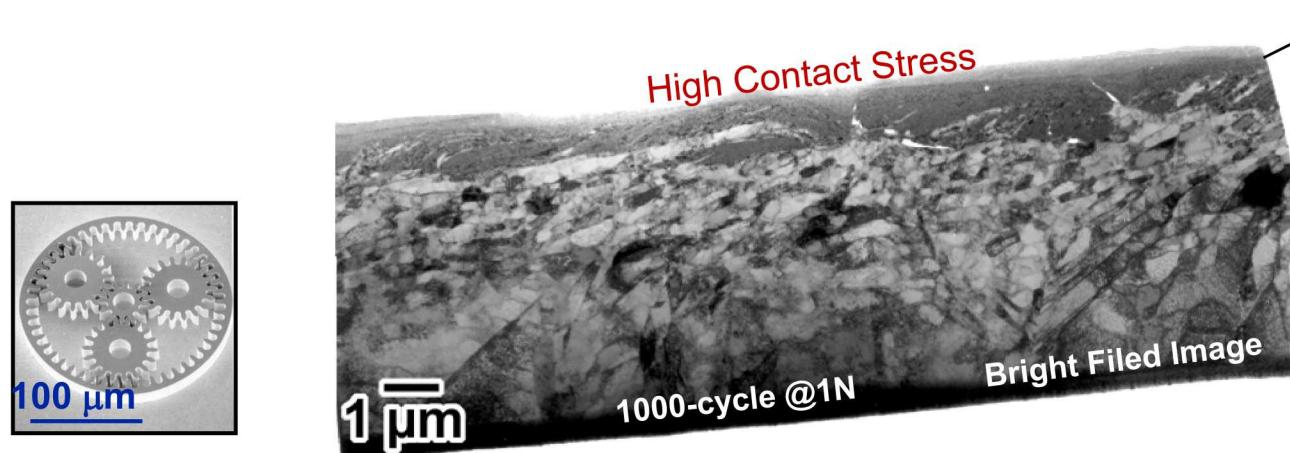
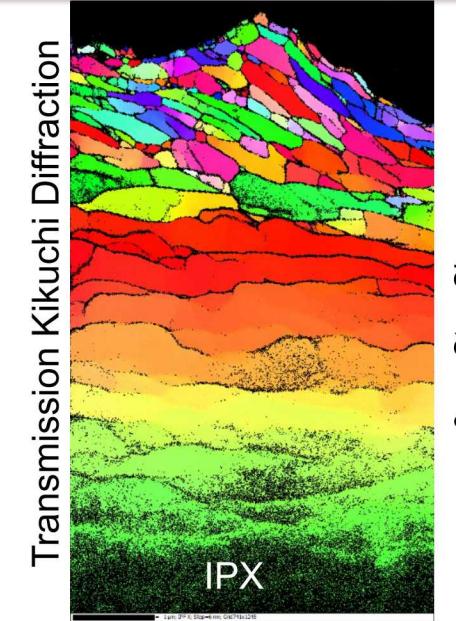
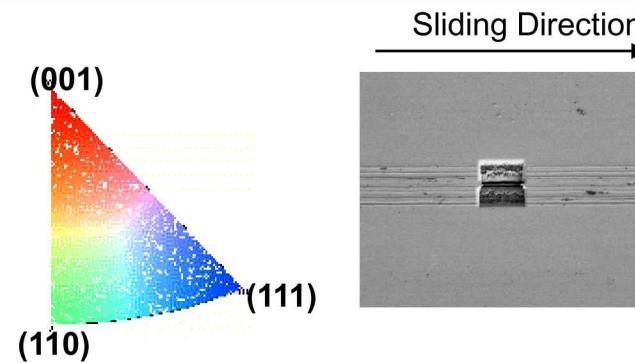
- Plastic deformation
- Diffusion
- Fracture initiation
- Tribocorrosion and Environmental Reactions

# Modern Tools (FIB, Orientation Imaging, TEM) can provide a wealth of Information

*Cross-sections of wear surfaces on Eletroformed Nickel*



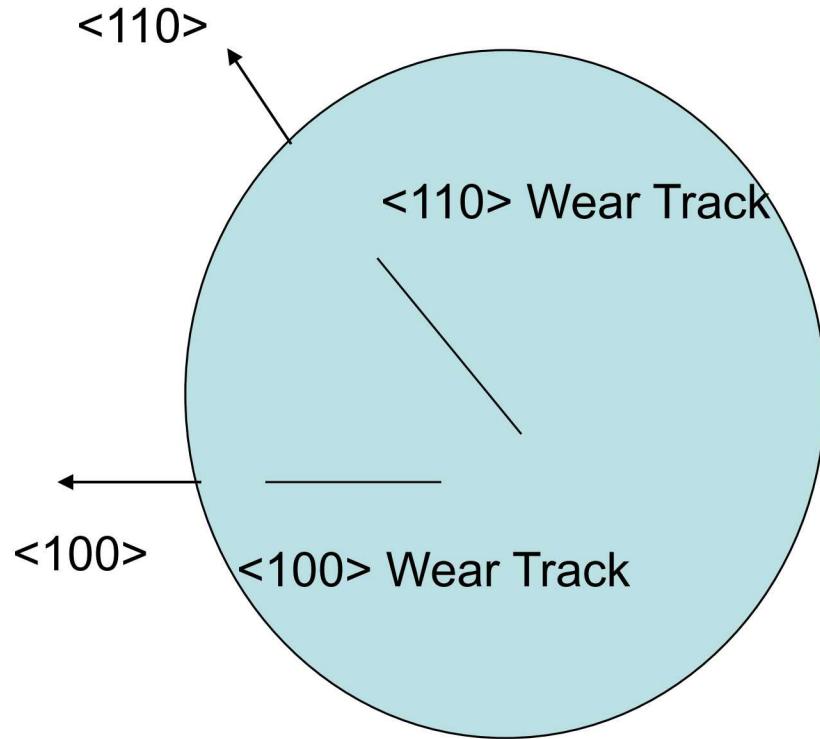
- Bending of columns in the direction of sliding
- More deformation in (110) columns, compared with (001)
- Top surface layer with equiaxed grains (recrystallized)



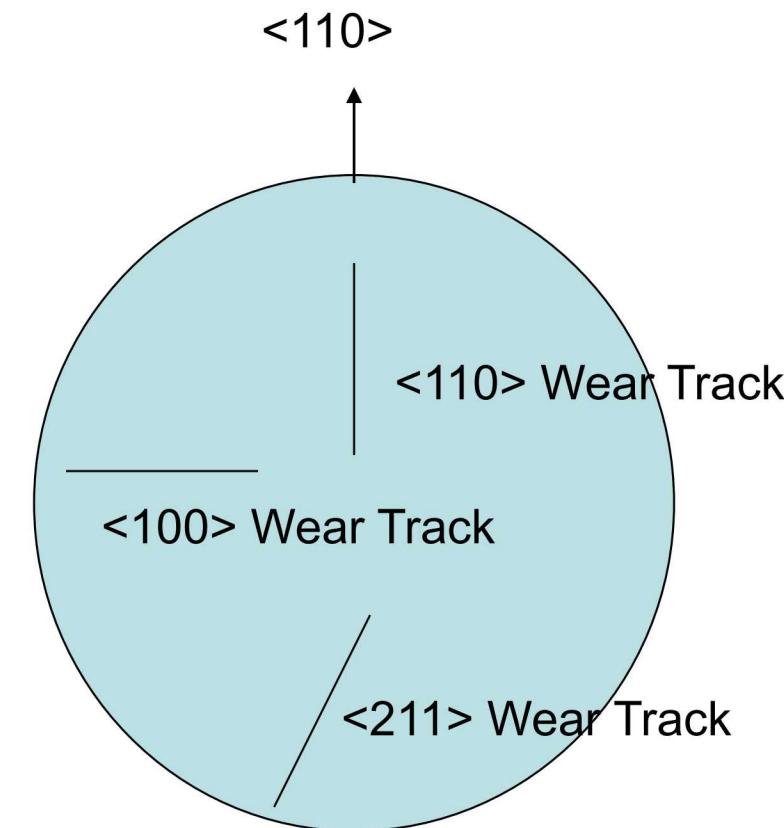
An understanding of the seminal work, dating back to Sir George Beilby and others in England, coupled with our recent work at the turn of this century, led us to investigate friction-induced deformation on single crystals.

- EBSD and FIB to identify the crystallographic directions (Ni Crystal)
- Rotary friction module to make friction measurements on specific crystal faces along different crystallographic directions
- Analysis of friction induced structures by EBSD and TEM
- Application of this work to the tribology of nanocrystalline thin films

# Crystallographic configurations for friction testing on single crystal Ni



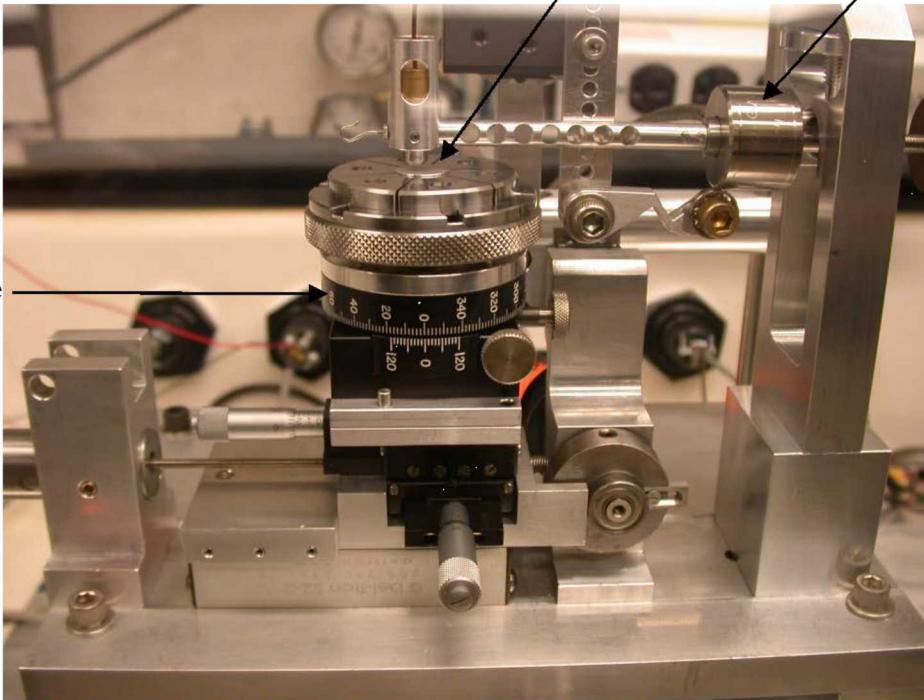
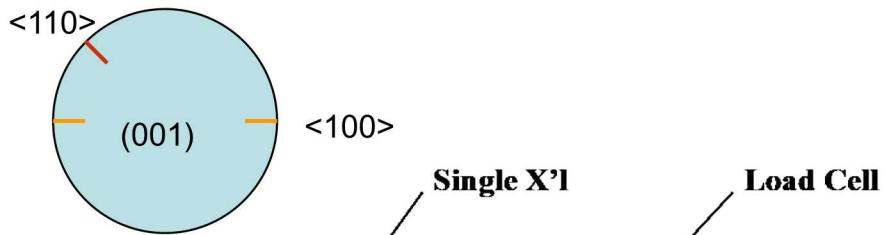
(001) Crystal Face



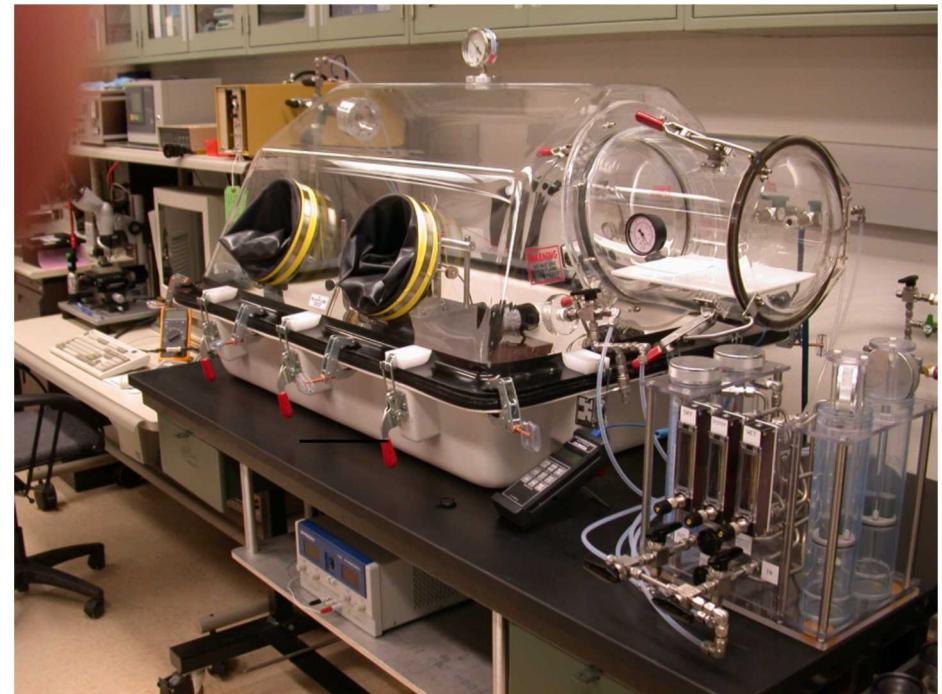
(011) Crystal Face

Identified the crystallographic directions with EBSD and marked with FIB

# Rotary Friction Test Module Designed and Fabricated

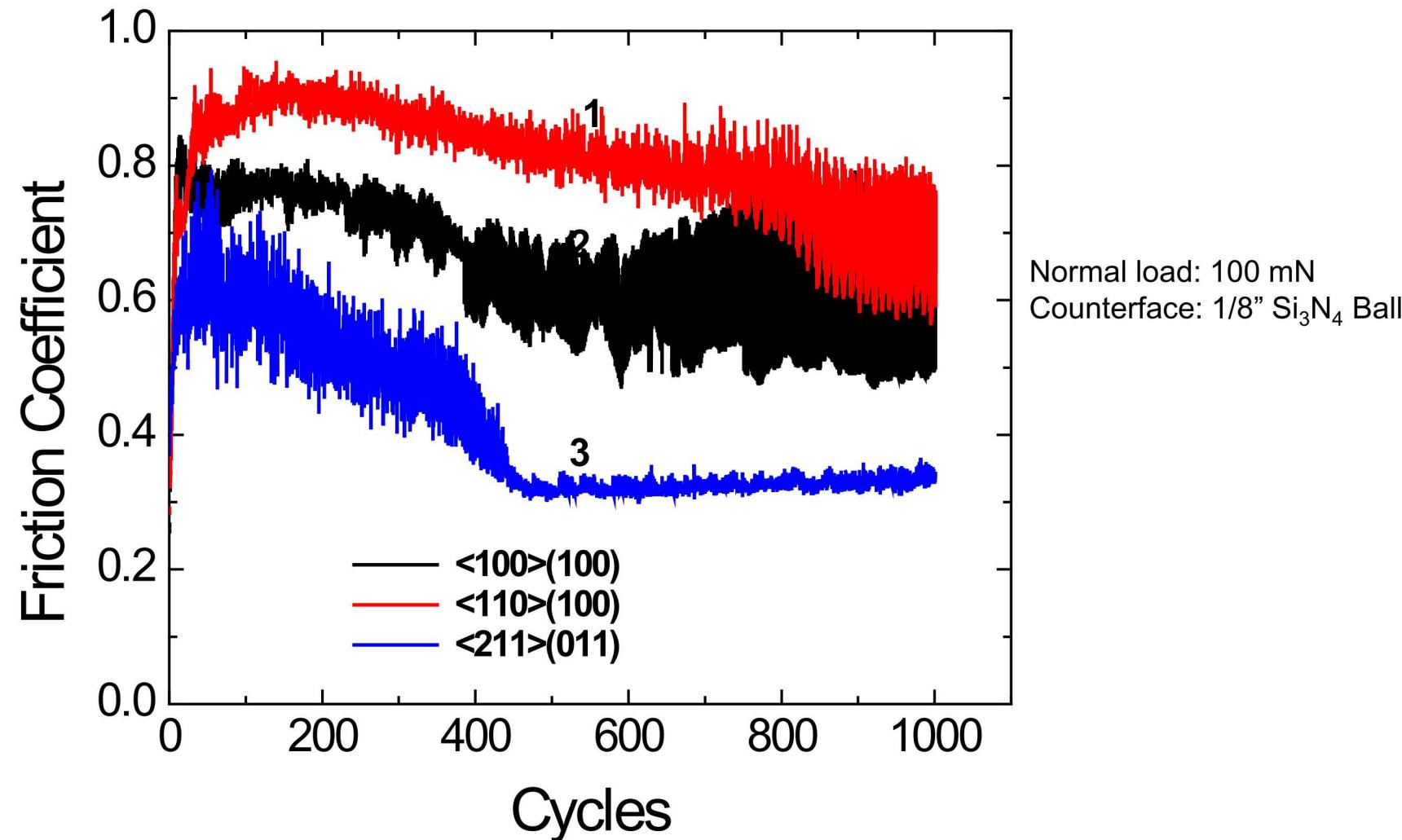


Rotary Stage for Single Crystal Alignment

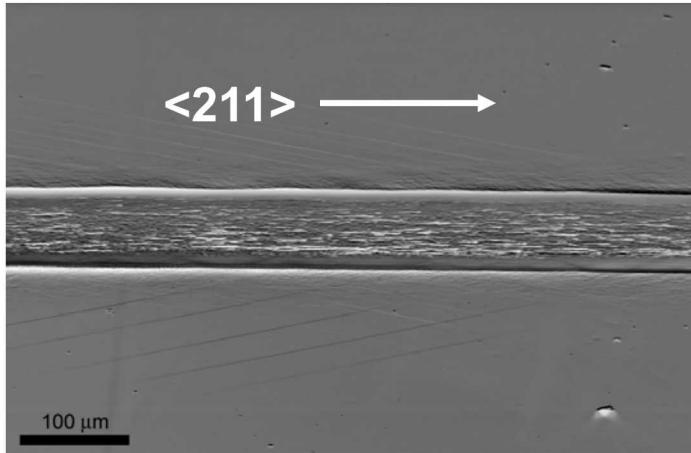


Environmental chamber

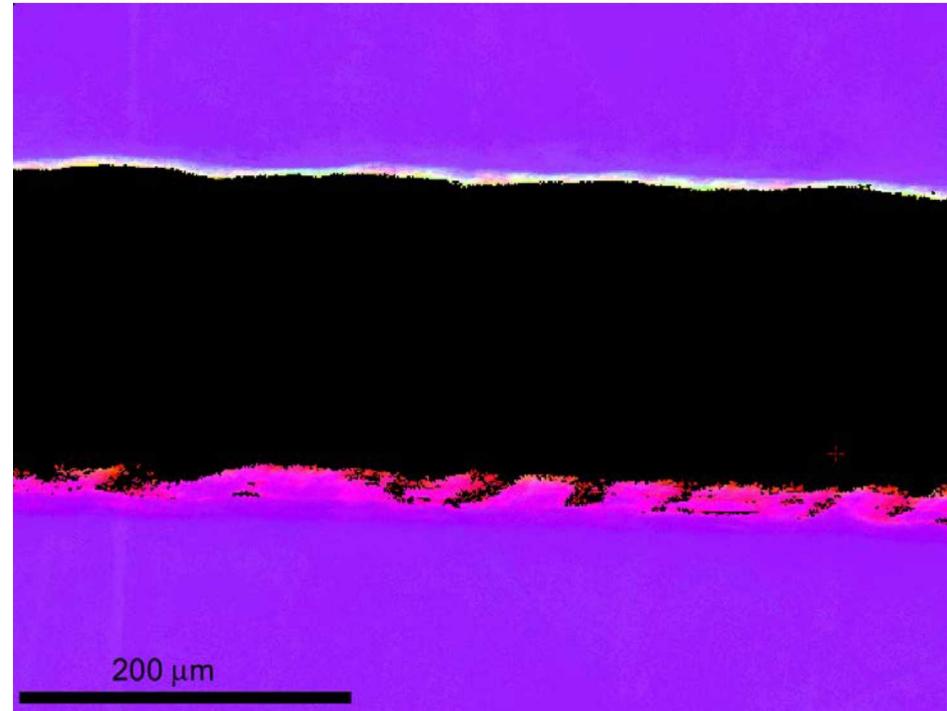
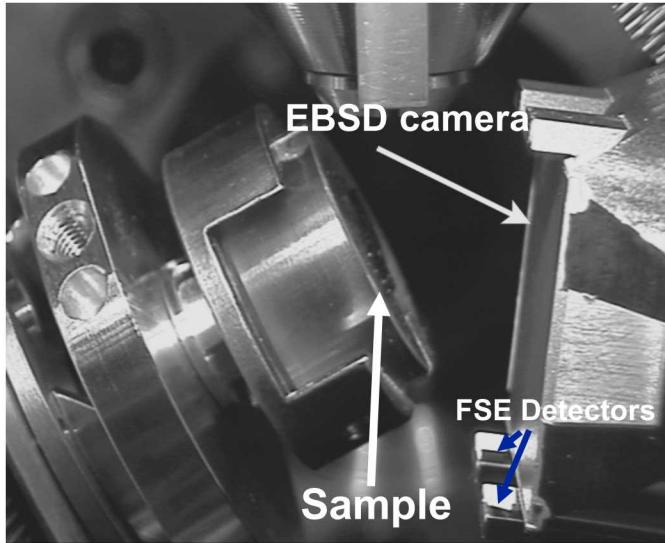
# Friction behavior is dependent on crystallographic orientations



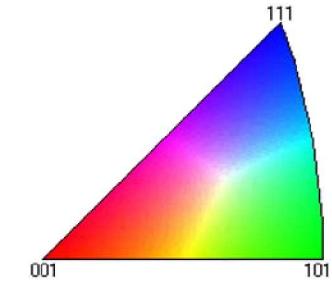
# SEM imaging (FSE) and EBSD of wear scars in plan-view provided partial information



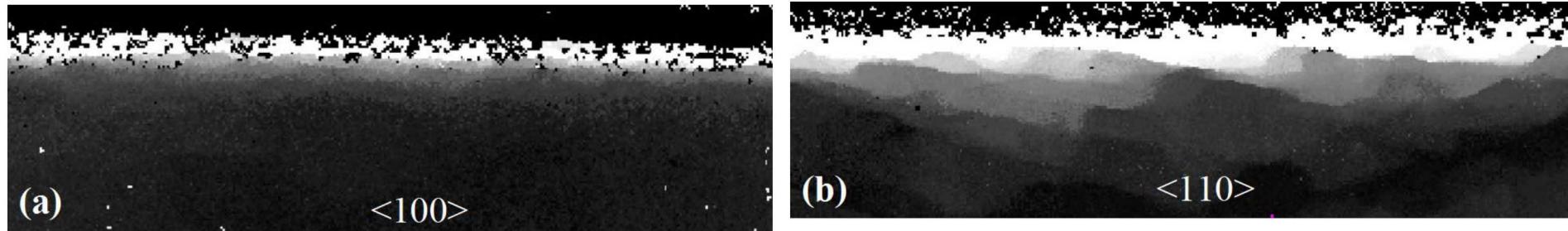
SEM image of forward scattered electrons (FSE). Note visibility of  $\{111\}$  slip traces. (Image not corrected for 70° tilt.)



EBSD IPF map with respect to the sliding direction. Note dark region due to high plastic deformation in wear scar

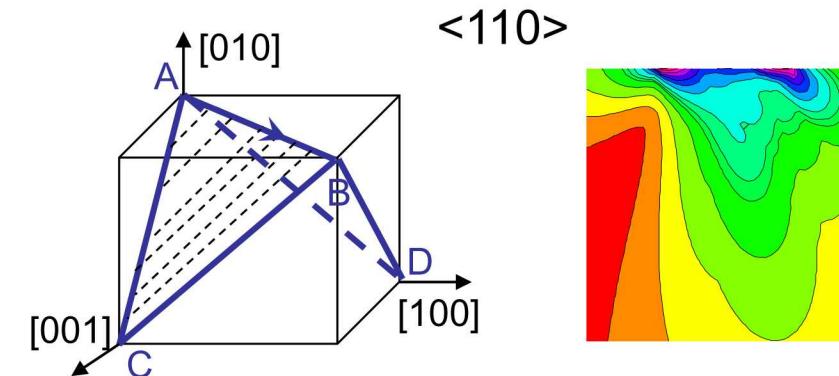
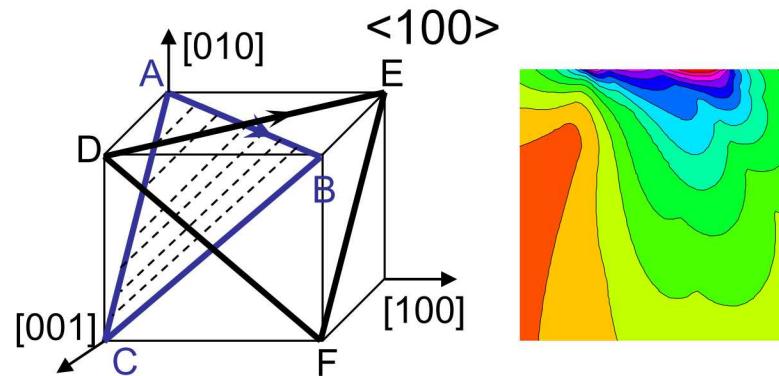


# Note the similarities in crystallographic orientation changes and critical resolved stress profiles (bottom)

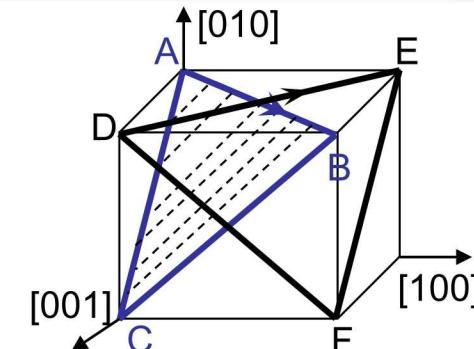
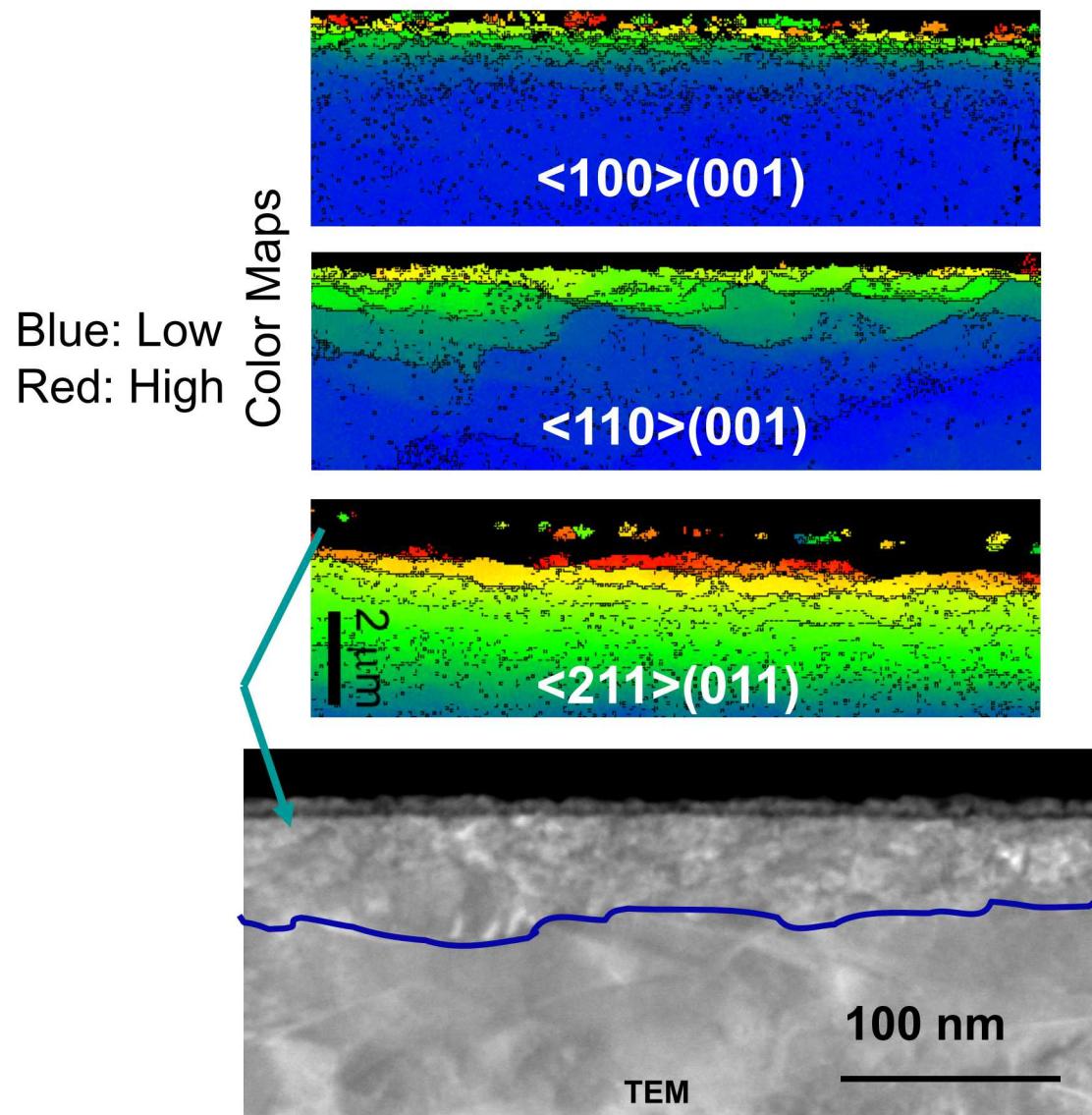


Maps showing the orientation changes relative to undeformed regions on (100) crystal surface. Brighter color represents larger orientation change. The magnitude of orientation change was about 6° total in for the friction track in the <100> direction and about 13 ° for the track in <110> direction.

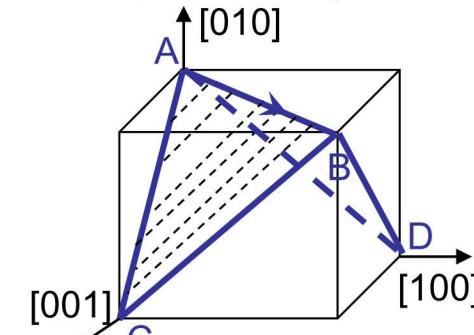
Slip system orientations show intersecting slip systems for <100> wear (ABC plane in AB direction, and DEF in DE), but not for <110> (ABC in AB and ABD in AB), suggesting more hardening for <100>. Color maps of resolved shear stress from analyses of plastic deformation show the strong asymmetry induced by sliding (as opposed to static) contact.



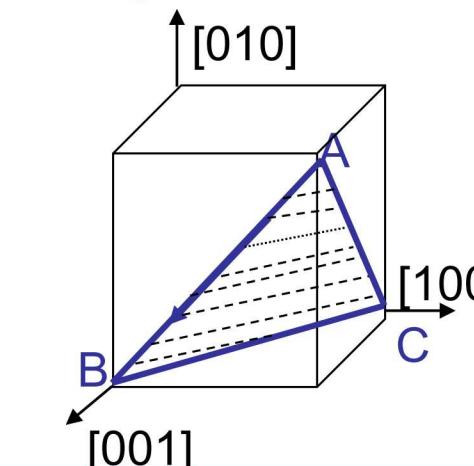
# Depth of deformation is related to crystallography: Misorientation wrt undeformed single crystal



**Strong dislocation interactions- high work hardening**

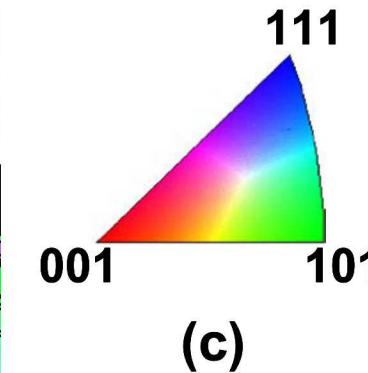
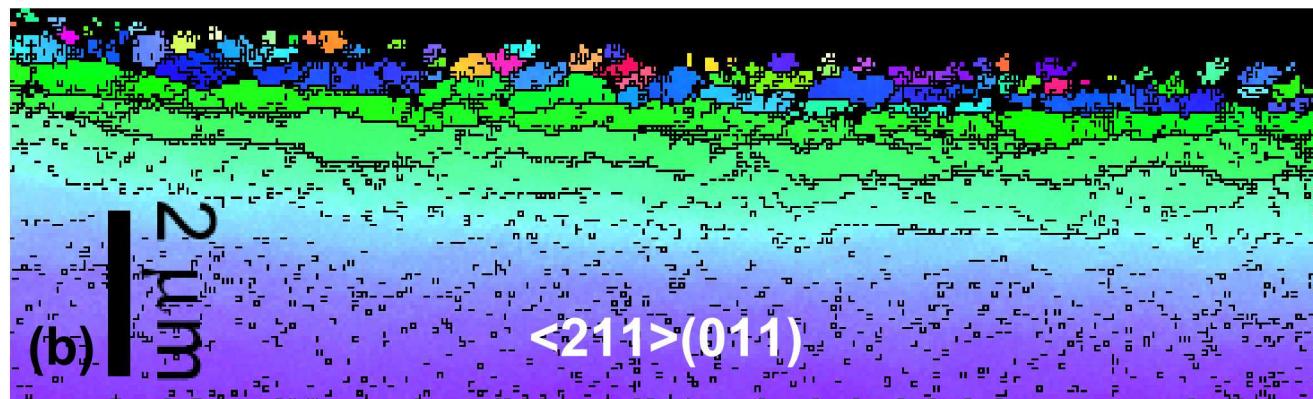
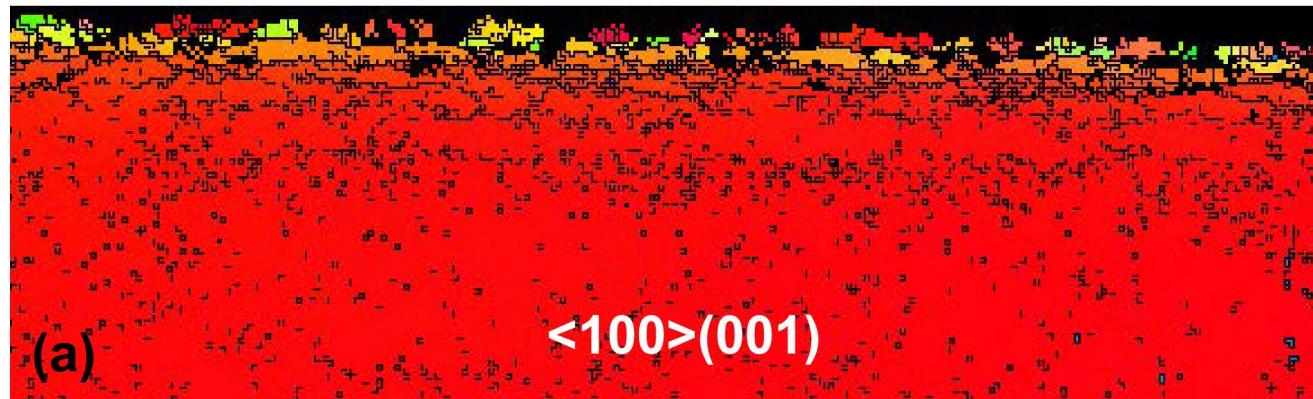


**Weak dislocation interactions- low work hardening**



**Very weak dislocation interactions- low work hardening – rapid recrystallization**

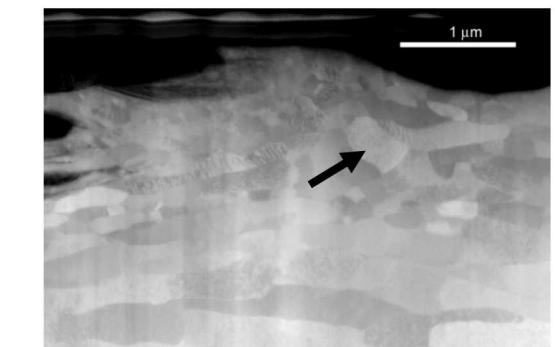
# EBSD: Crystallographic maps



Sliding Direction

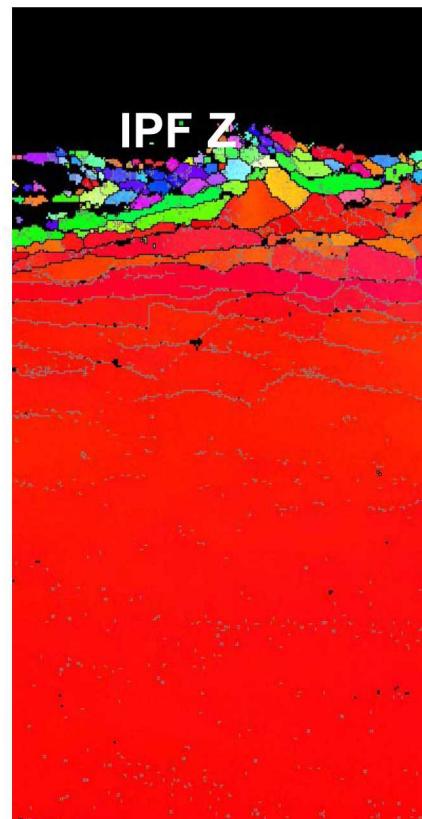
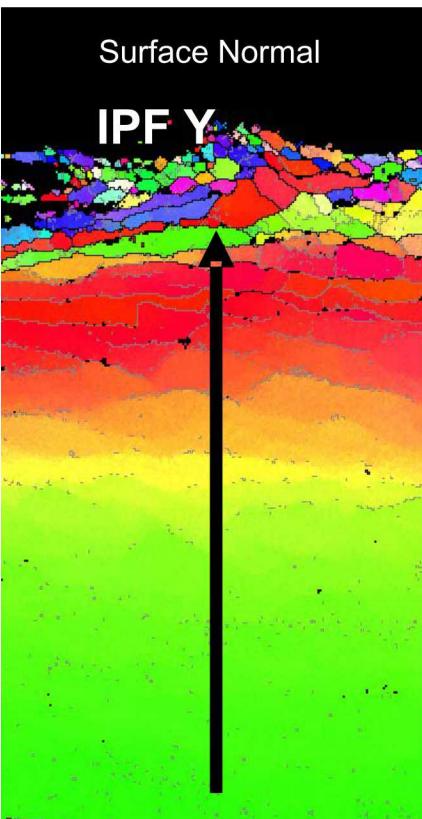
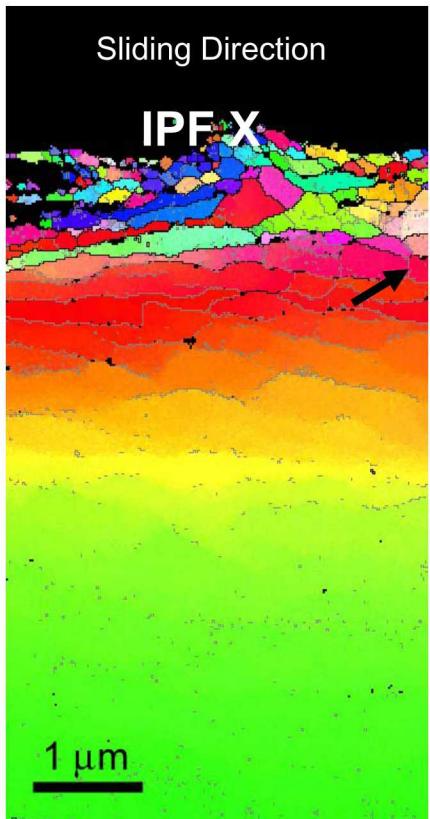
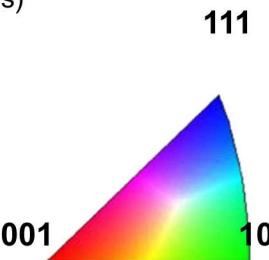
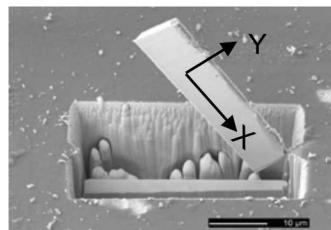


# Application of EBSD to monitor the evolution of subsurface microstructures induced by sliding contact (Higher Contact Stresses)

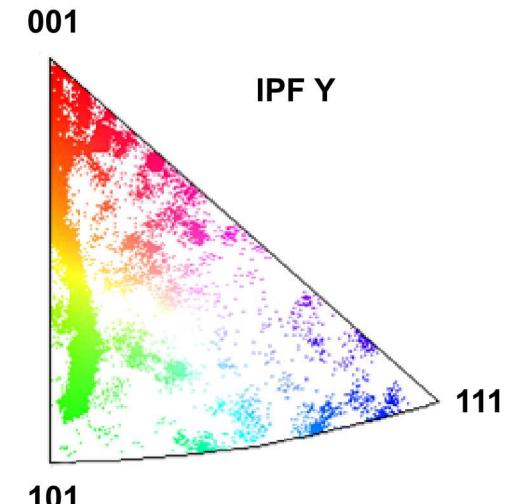
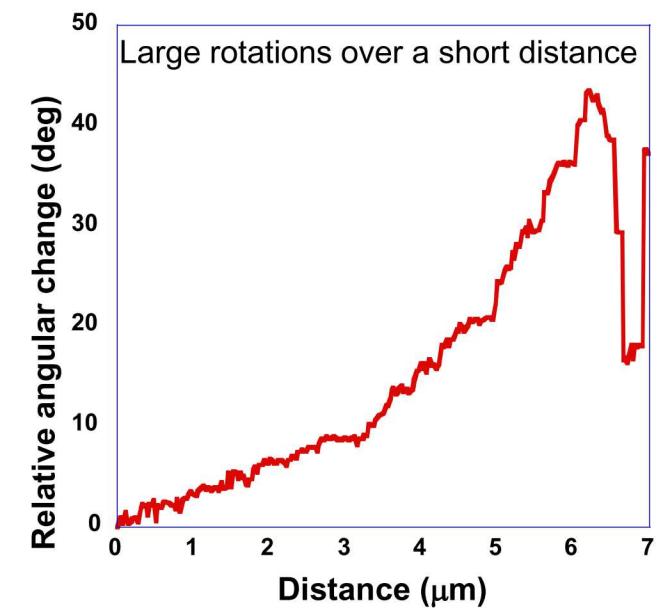


$<110>$  on  $(011)$

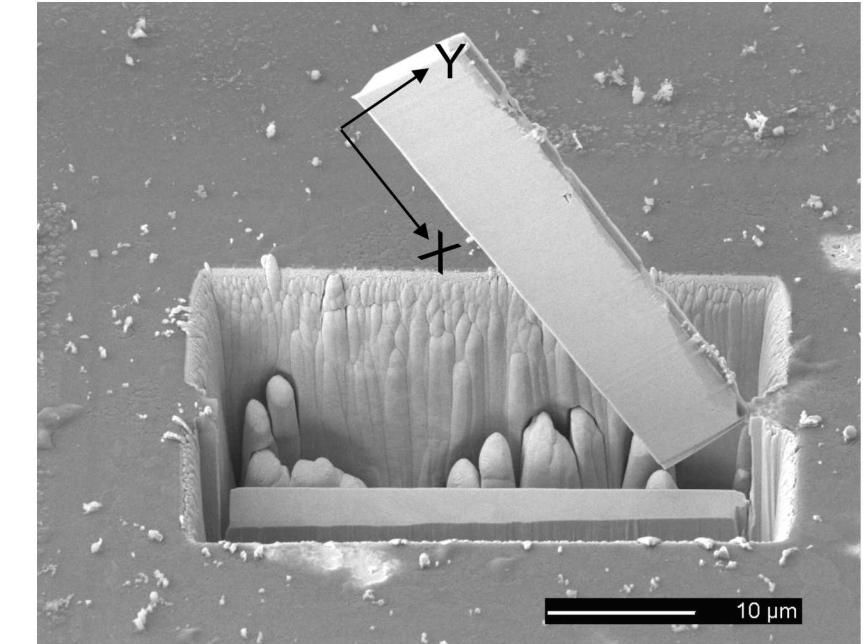
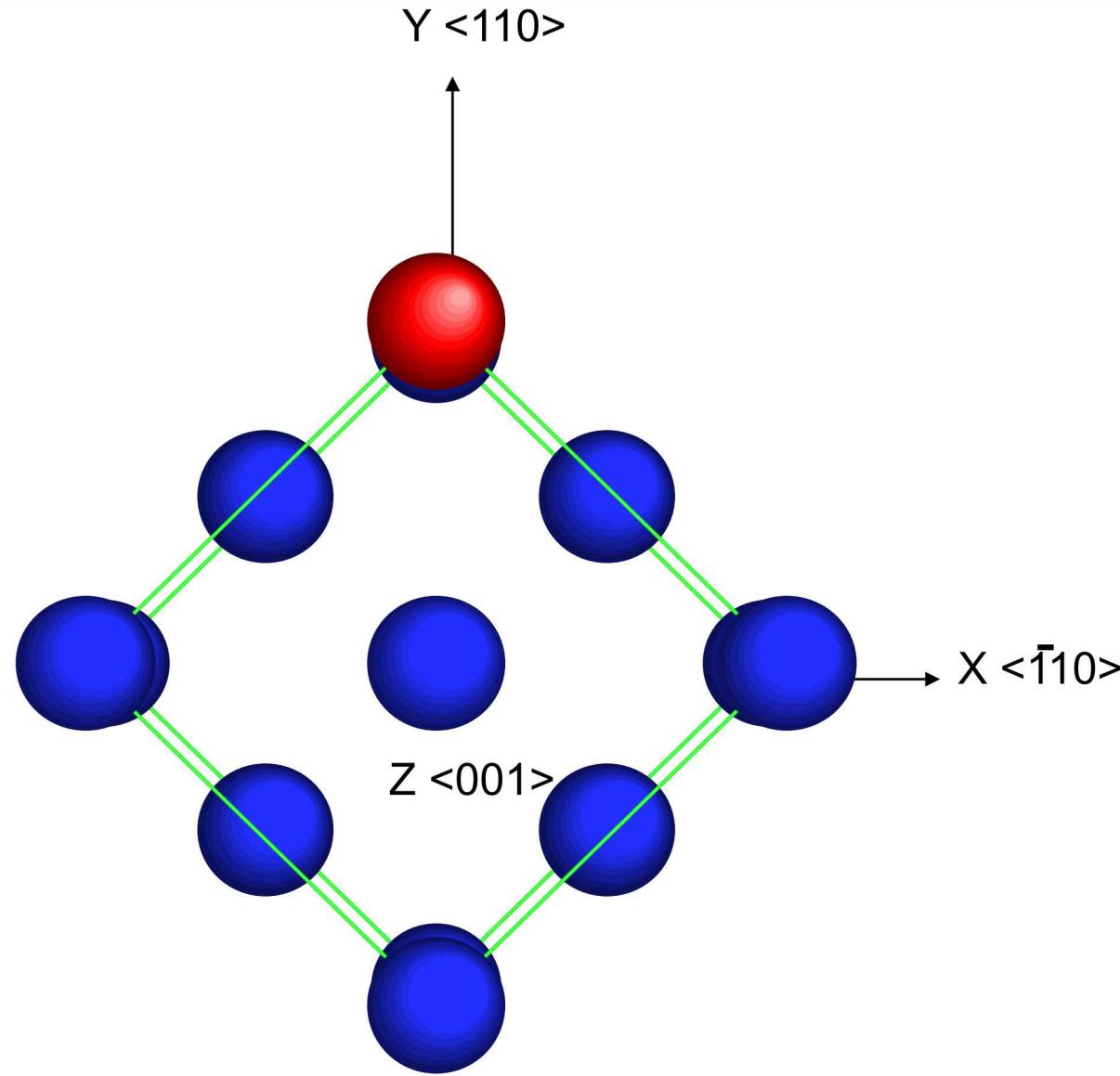
(100 grams for 2000 Cycles)



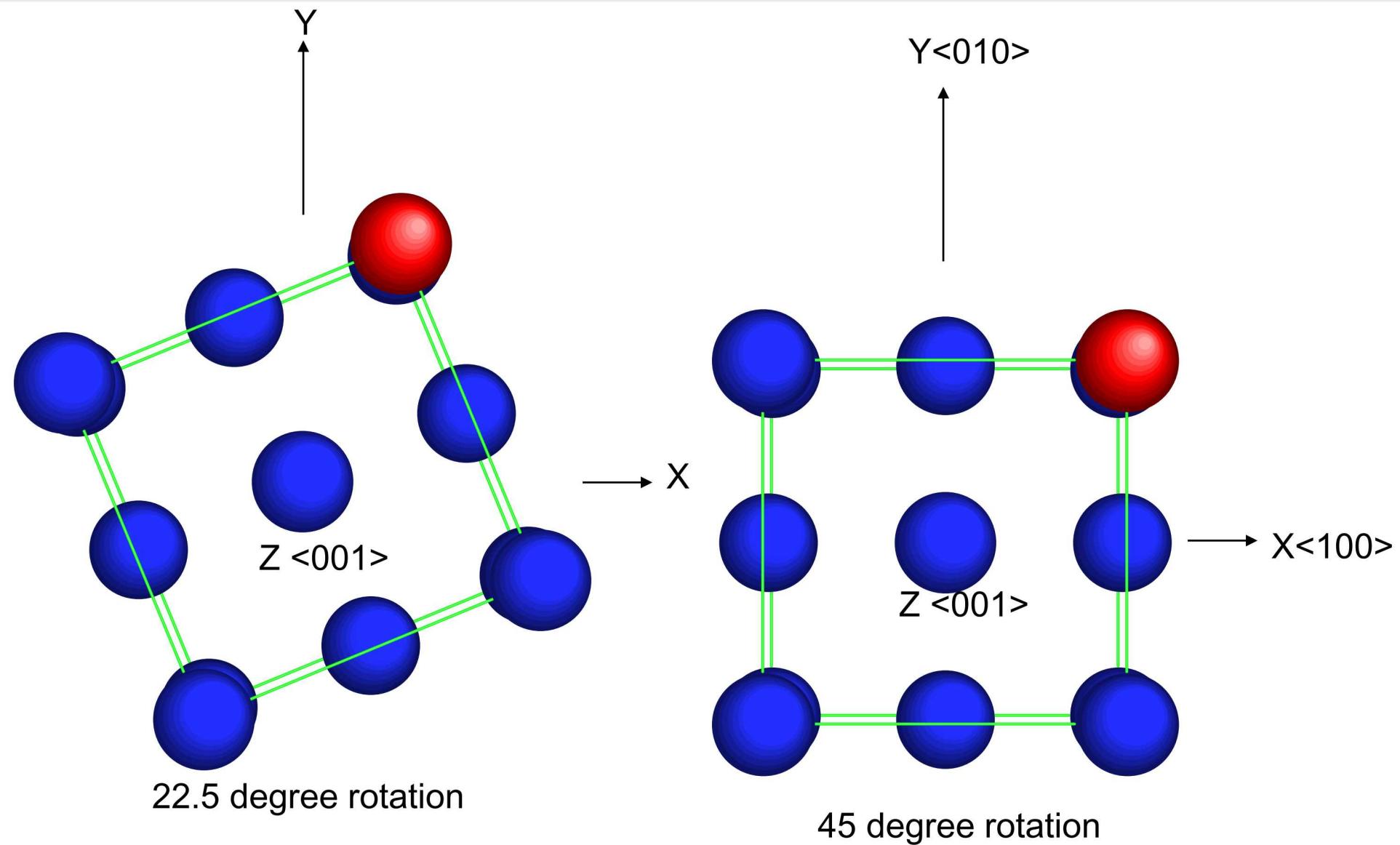
200 x 385 x 25 nm steps



# Schematic illustration of grain rotation. 1

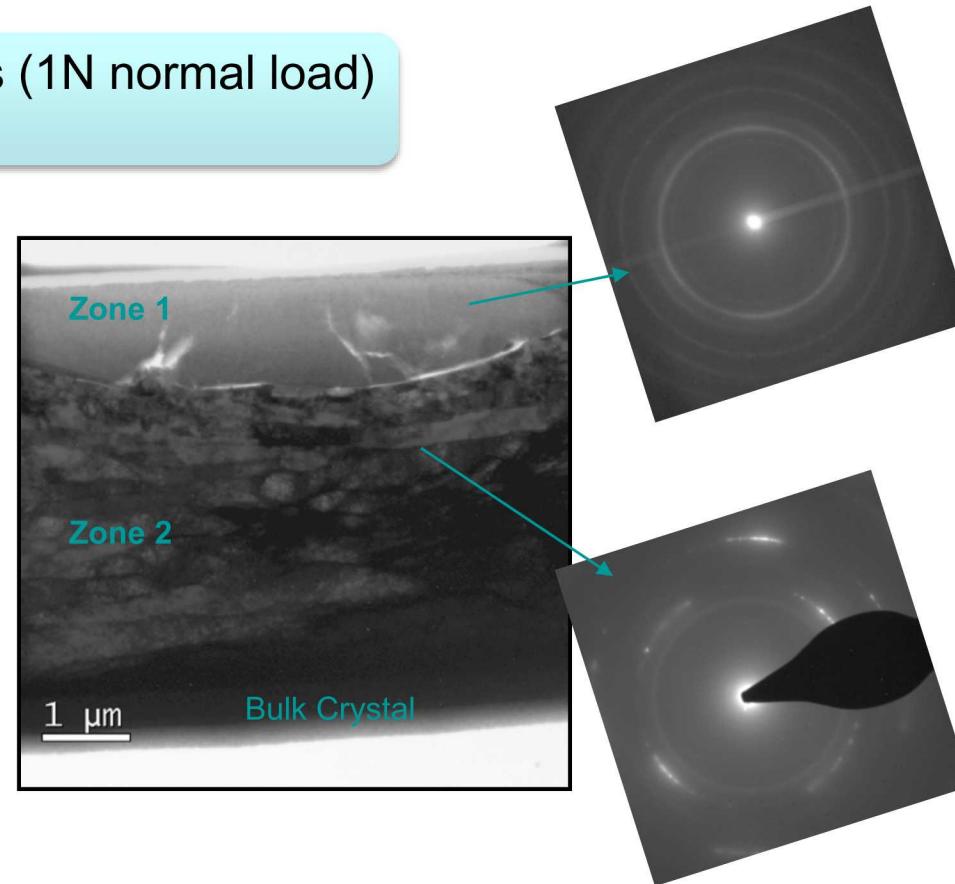
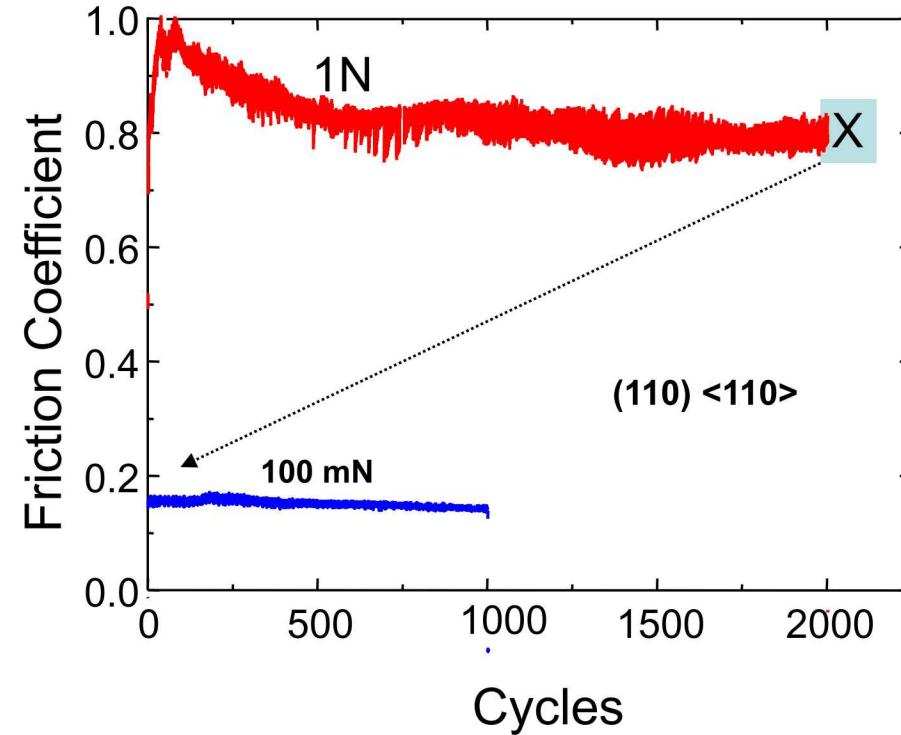


# Schematic illustration of grain rotation. 2

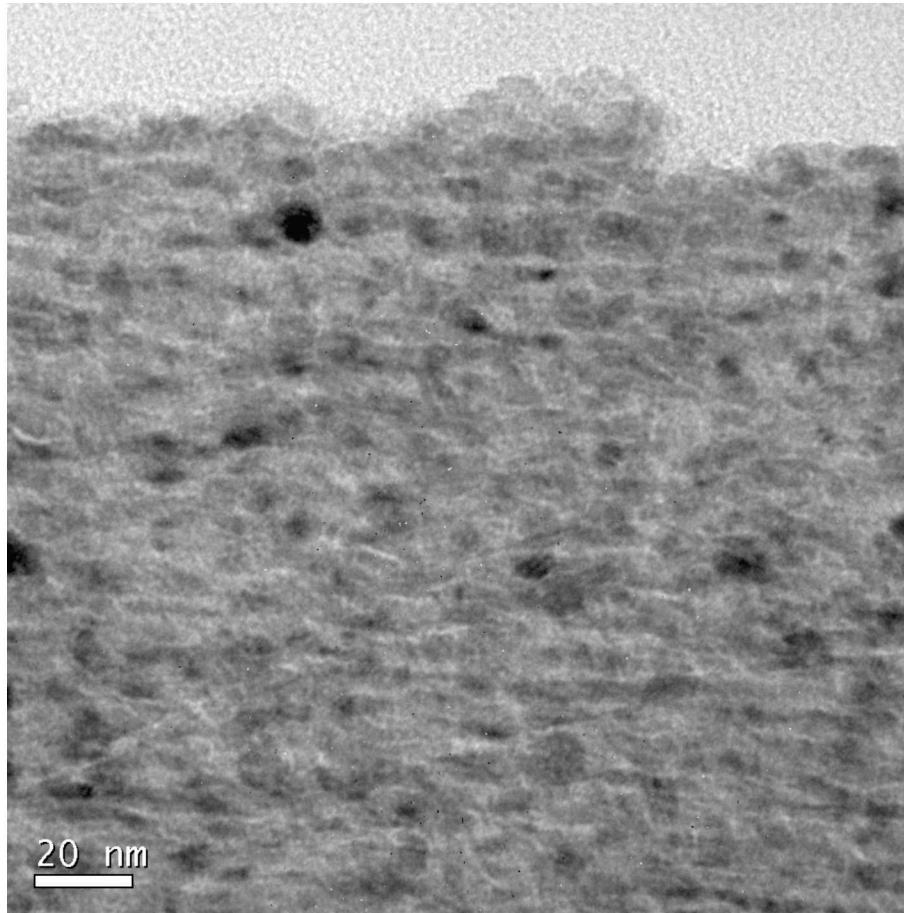


# Higher normal loads (1N) produced unique substructures with interesting friction behavior

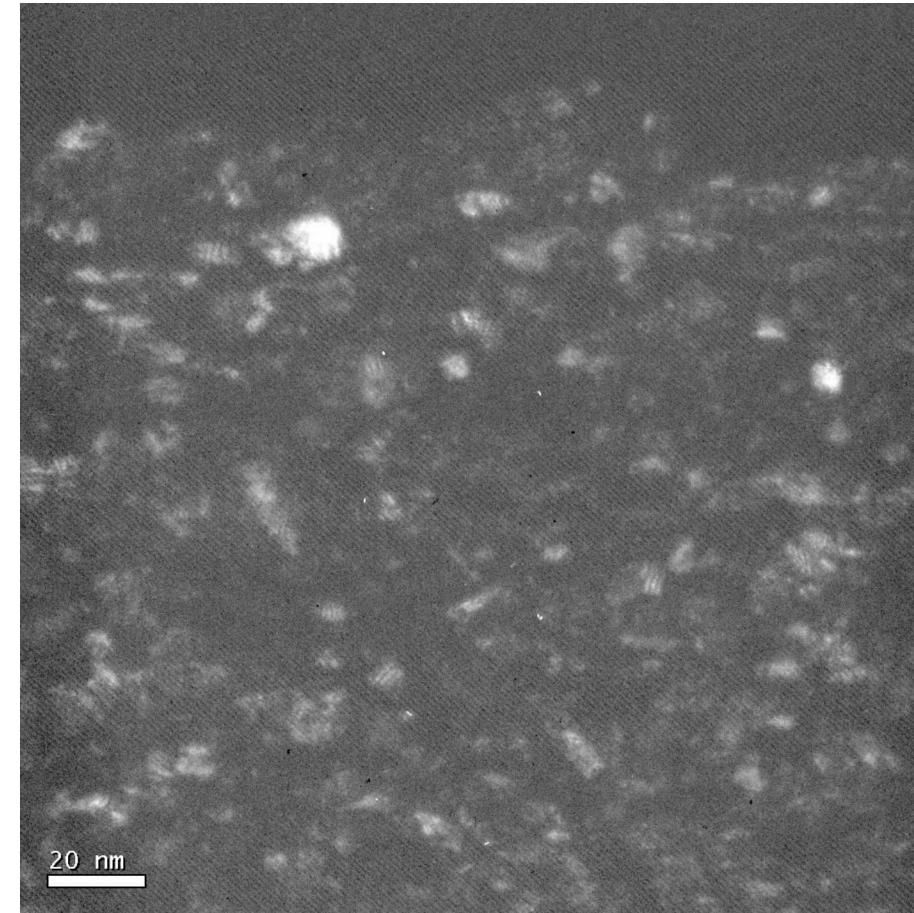
Create the unique structures at higher contact stress (1N normal load) and make the friction measurement at 100 mN load



# High resolution images of Zone 1

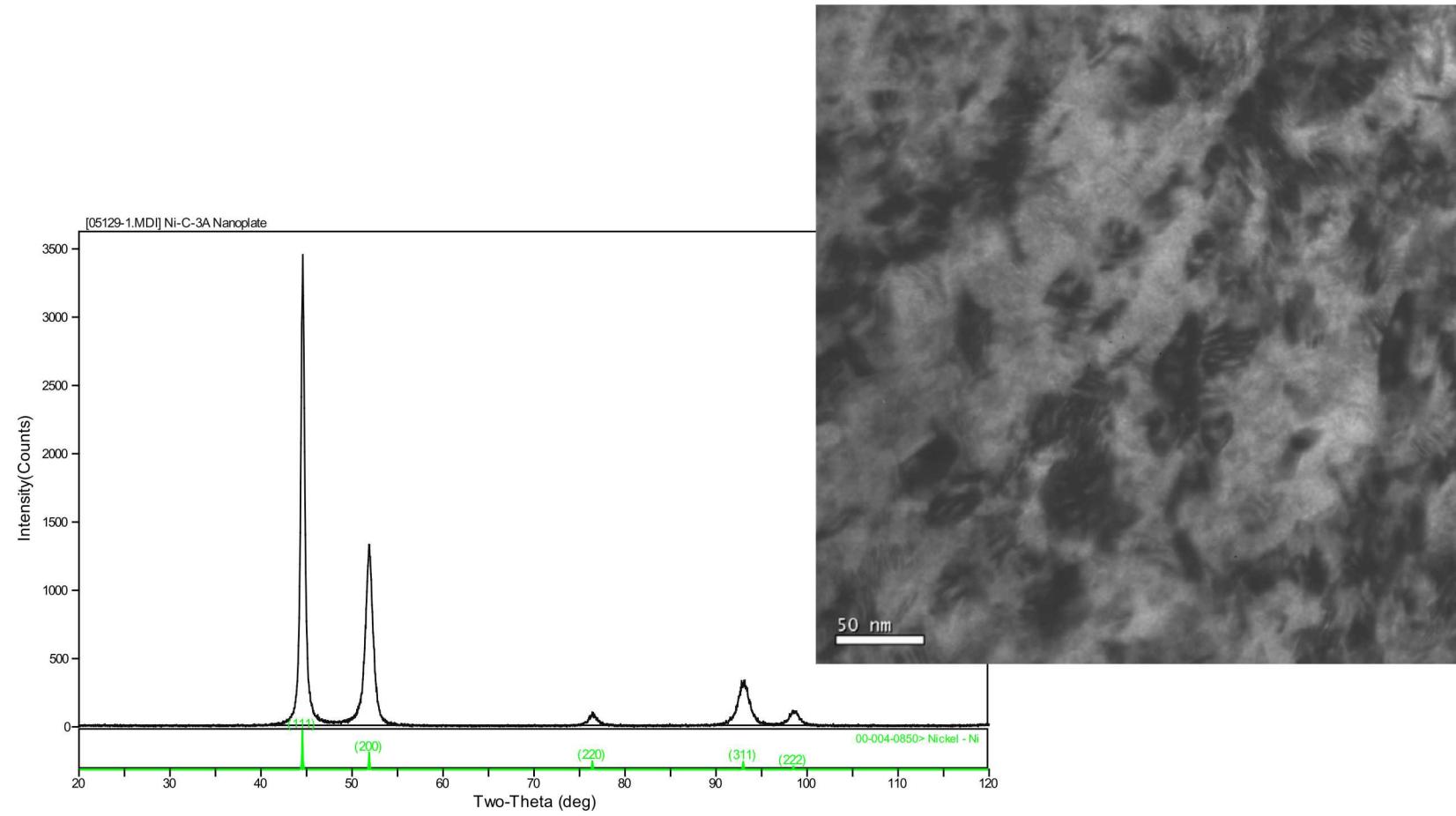


Bright-field TEM image

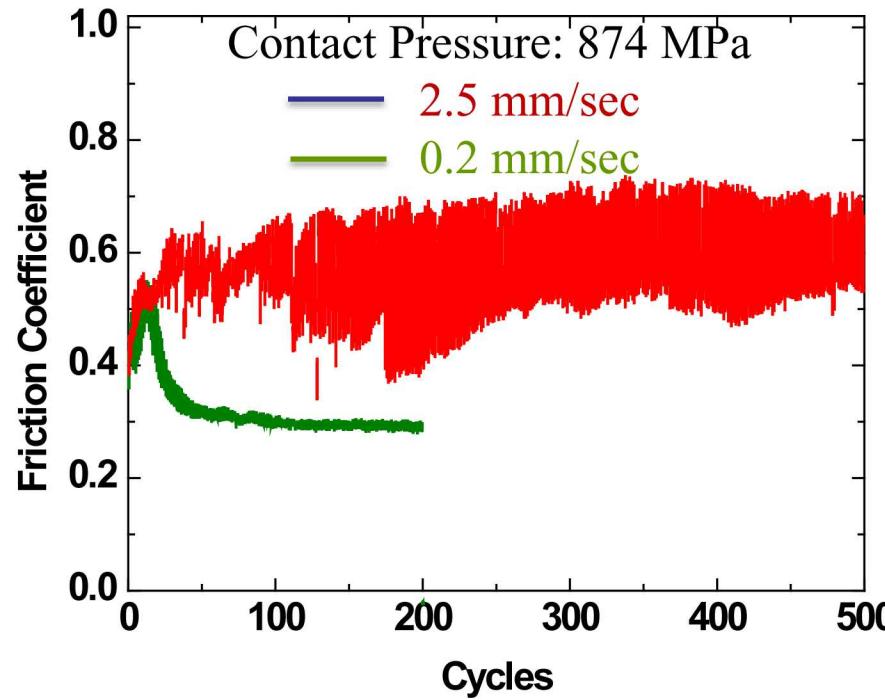


Dark-field TEM image

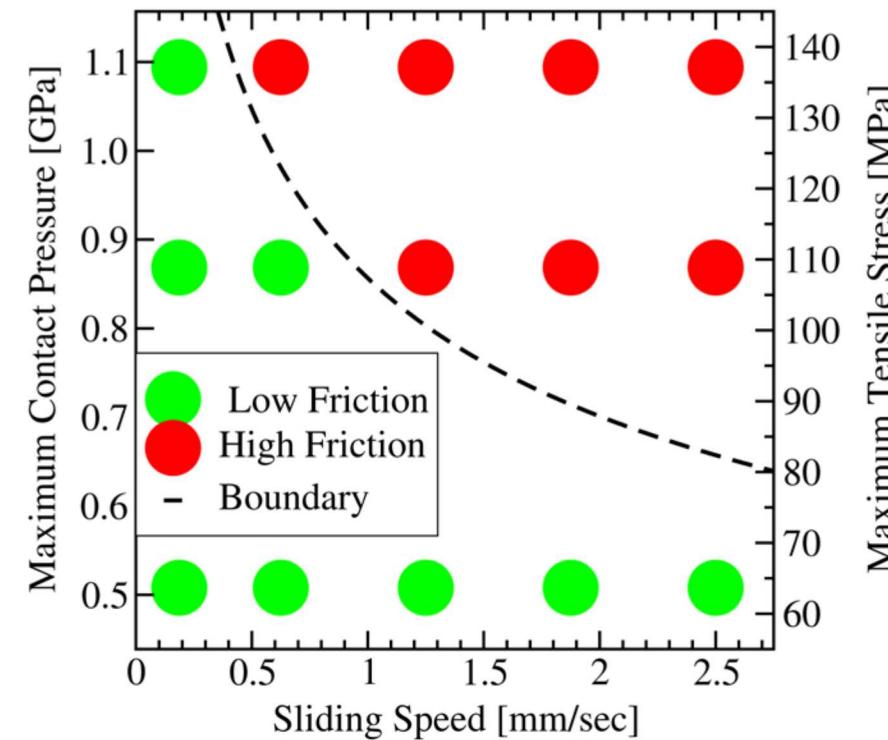
# Application to Nanocrystalline Thin Films



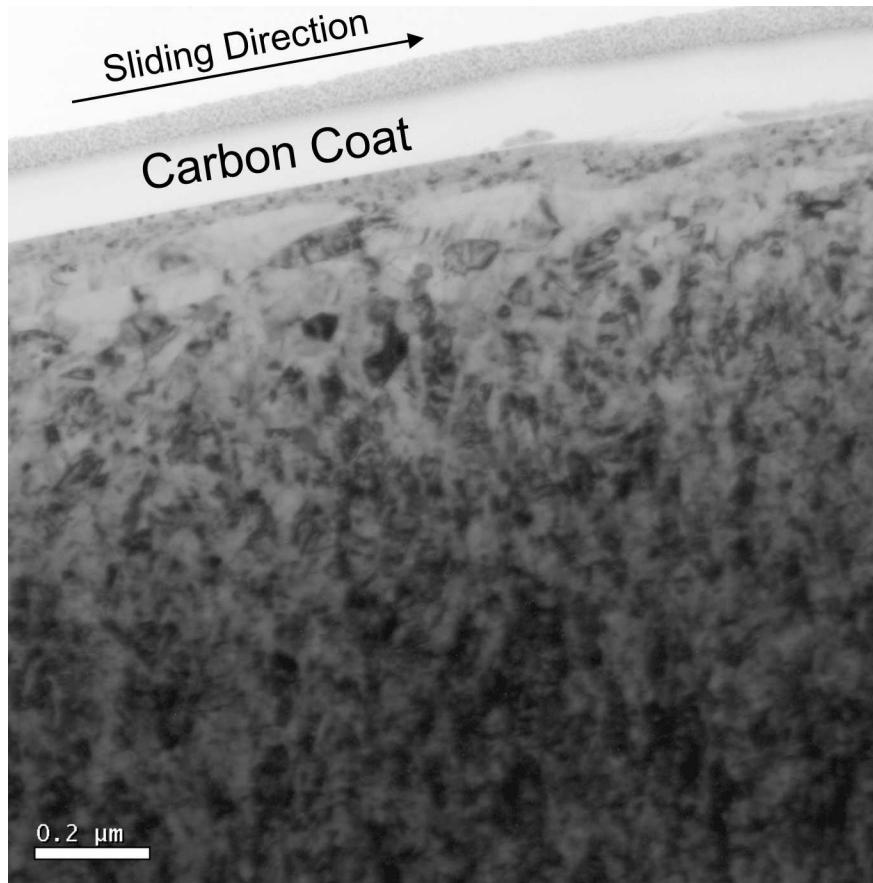
# Nanocrystalline Ni films exhibited two different friction behaviors depending on the sliding speed and contact stress



Friction Transitions show a clear dependence on stress and sliding speed

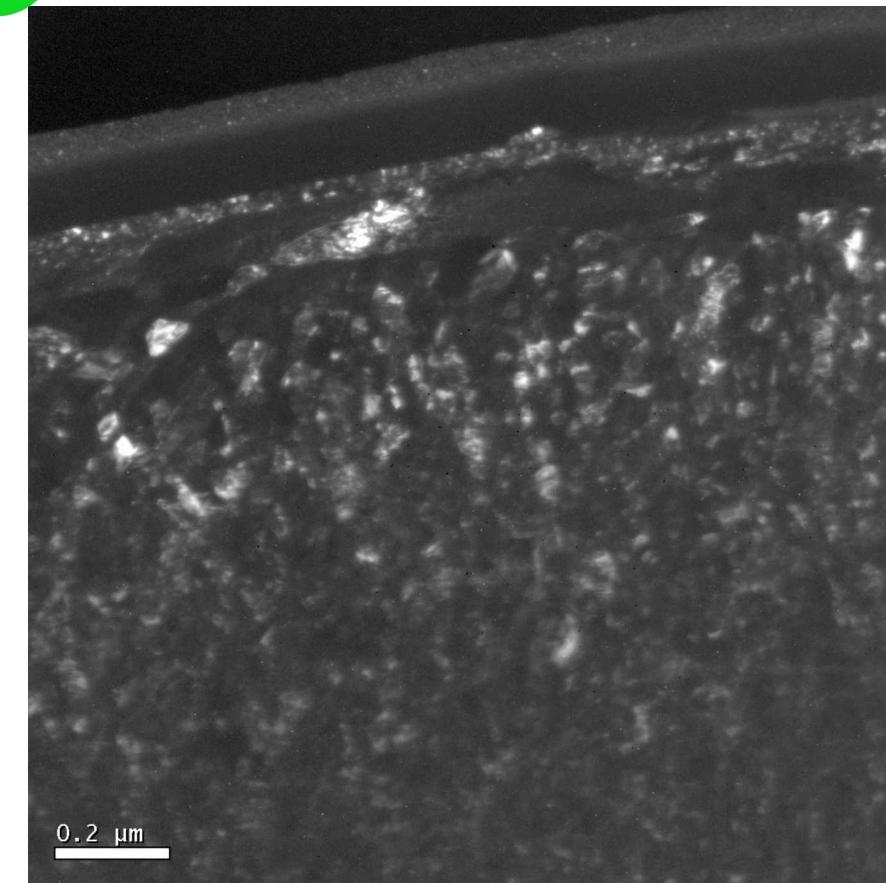


# Transmission Electron Microscopy of Subsurfaces: Low Friction Case



BF-TEM

Sample preparation: FIB microscopy with low KeV cleaning  
Low magnification micrographs



DF-TEM

Ni-3C-A1 #1 051202D Track 19, 10g, 20rpm, 600cycles

# XSTEM image showing three distinct regions: (Ultrananocrystalline (1), recrystallized zone (2) and the bulk

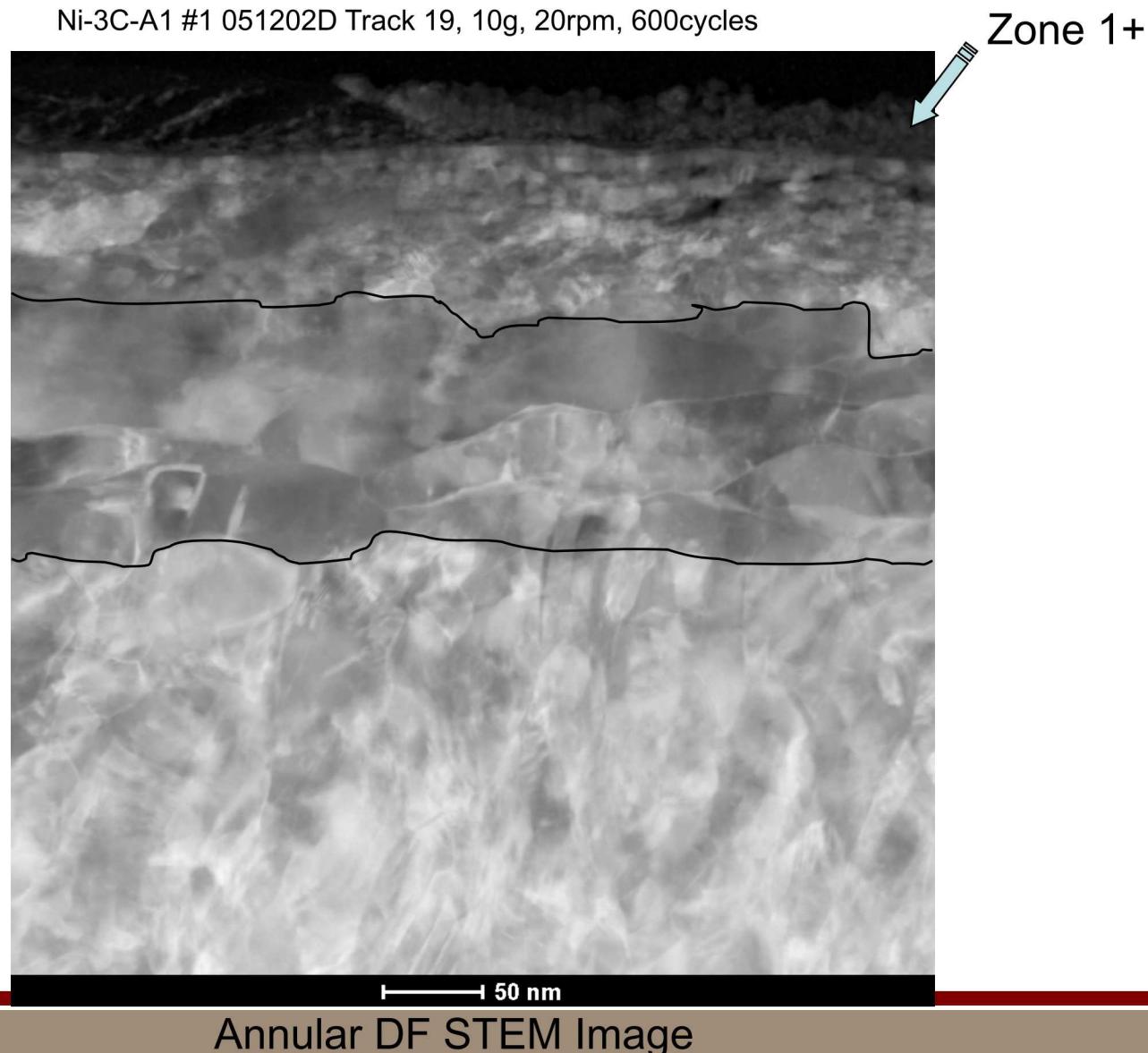


**Zone 1**  
Ultra Nanocrystalline

**Zone 2**  
Grain Growth +  
Texture

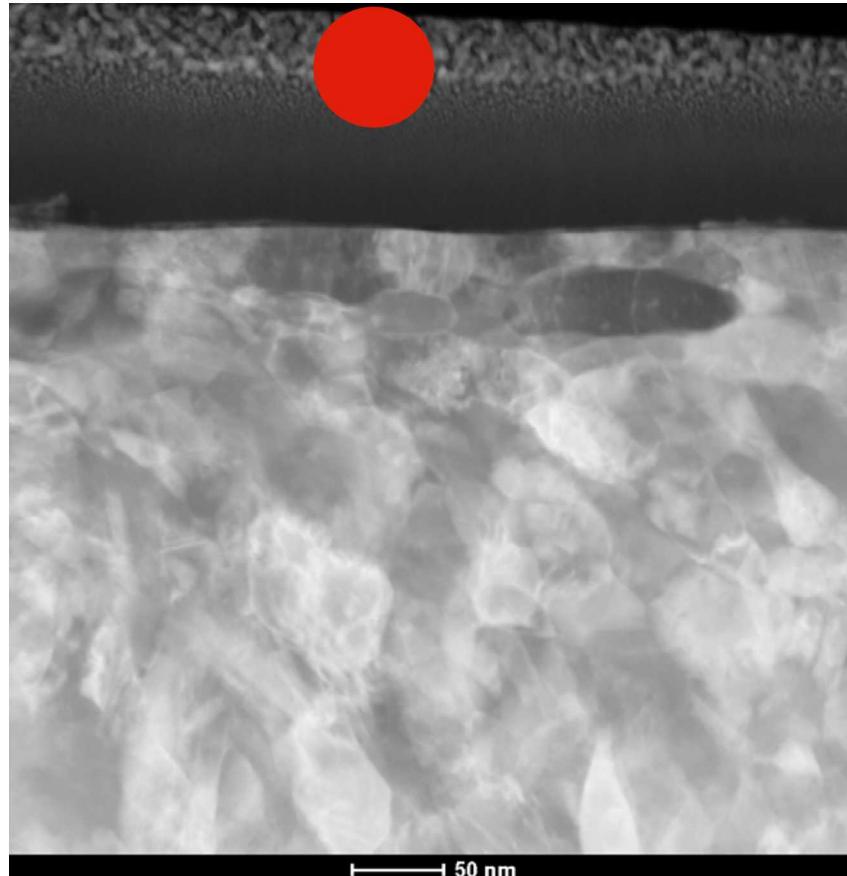
**Zone 3**  
Bulk

Annular DF Image



# Comparison of Subsurfaces: High Friction (Red) and Low friction (Green)

Ni-C-3A #1 051201G Track 14, 100g, **20rpm**, 600cycles



No Zone 1

Annular DF STEM Images

Ni-C-3A #1 051129B Track 3, 100g, **3rpm**, 200cycles



Zone 1 Present

- Friction-induced deformation is related to crystallography.
  - Advanced microscopy techniques are key towards understanding the friction-induced microstructures.
- Grain boundary sliding appears to be a viable deformation accommodating mechanism, when ultrafine nanocrystalline layers with 2-10 nm size were present. This is a viable route to reduce the COF and mitigate stick-slip behavior.
- However, friction behavior of nanocrystalline materials appears to sensitive to contact stress and sliding speed.

## Acknowledgements



Rotary friction module was designed and fabricated by Rand Garfield  
FIB samples were prepared by Michael Rye

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