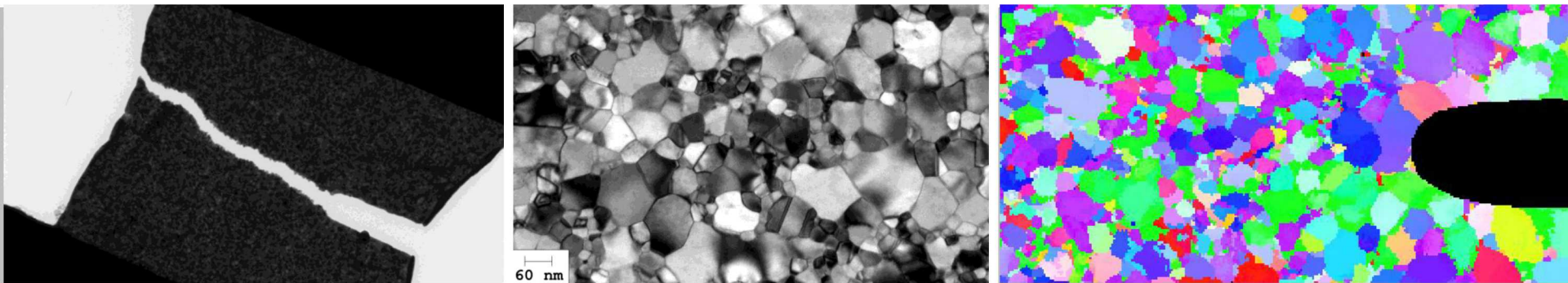


*Exceptional service in the national interest*



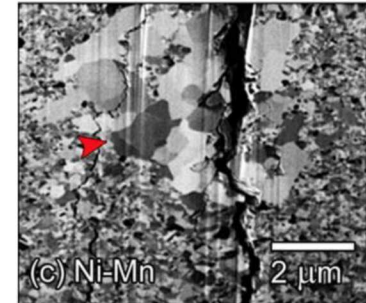
# Exploring Nanocrystalline Deformation Mechanisms Active During In-situ TEM High Cycle Fatigue

Christopher M. Barr, Daniel Bufford, William Mook,  
Brad Boyce, and Khalid Hattar

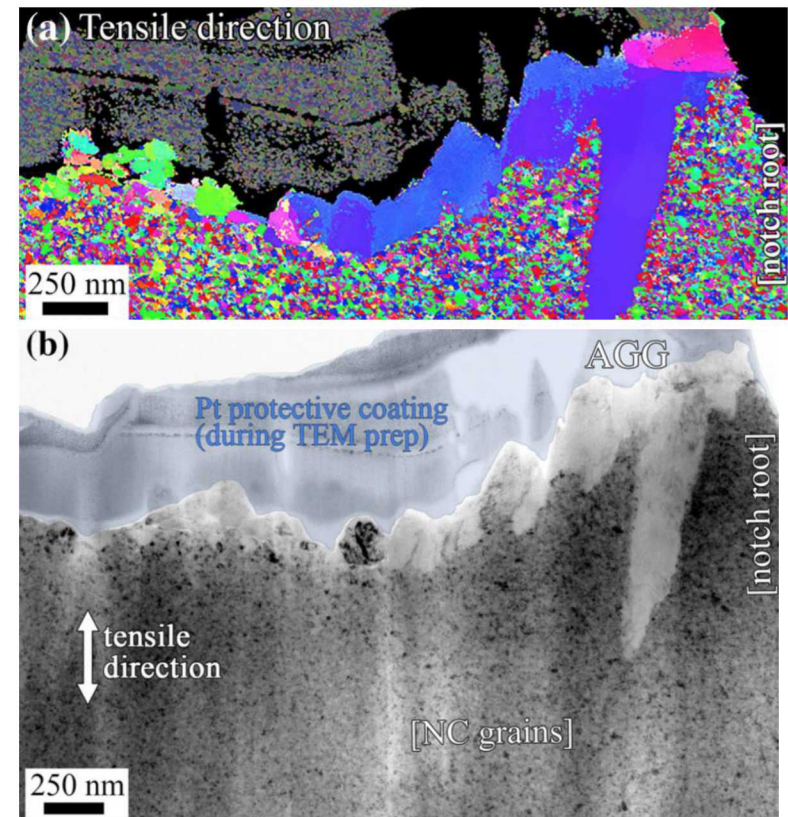
Sandia National Laboratories

# Nanocrystalline Alloys: Fatigue and Fracture

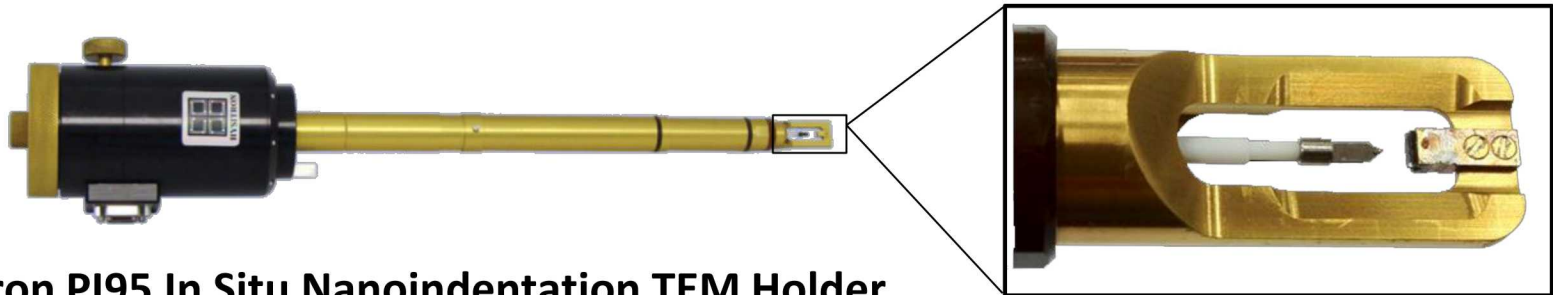
- Improved fatigue resistance compared to coarse grain counterparts
- Progressive microstructural changes with cyclic loading, often below yield stress
- Fatigue in nanocrystalline metals
  - Grain boundary migration and grain growth
  - Crack initiation
- What are the underlying mechanisms associated with these phenomena?
  - *In situ* TEM deformation techniques provide the spatial resolution needed to investigate these questions
  - Ideally coupled with bulk scale testing



Boyce and Padilla, Met Trans A, 11 (2011)

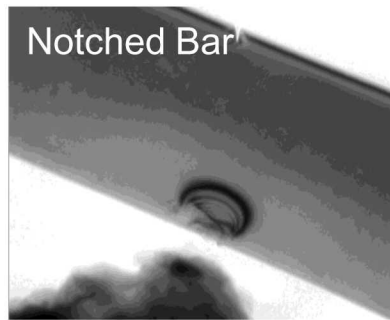
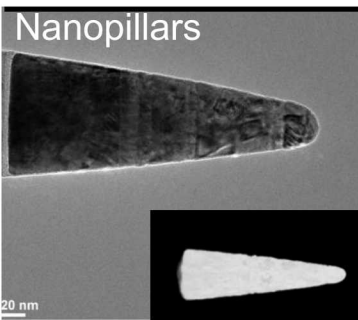
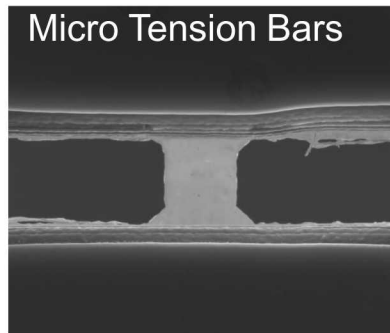
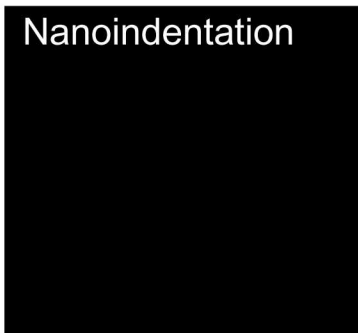


# *In situ* Quantitative Mechanical Testing



## Hysitron PI95 In Situ Nanoindentation TEM Holder

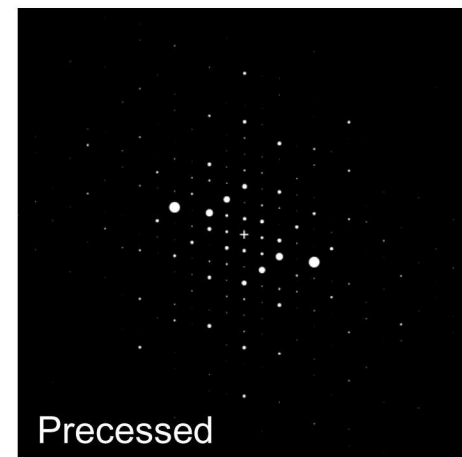
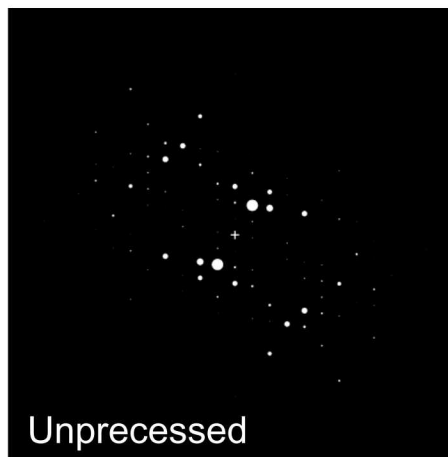
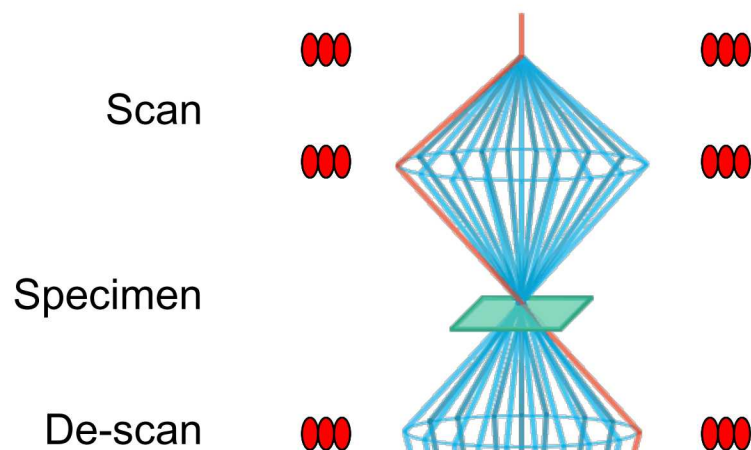
- Sub nanometer displacement resolution
- Quantitative force information with  $\mu\text{N}$  resolution
- Concurrent real-time imaging



- A variety of sample geometries
- Load functions examined at I<sup>3</sup>TEM:
  - 1) Indentation
  - 2) Tension
  - 3) Fatigue
  - 4) Creep (irradiation and thermal)
  - 5) Compression
  - 6) Future: Nanowear



# Precession Electron Diffraction (PED) Microscopy



## Advantages

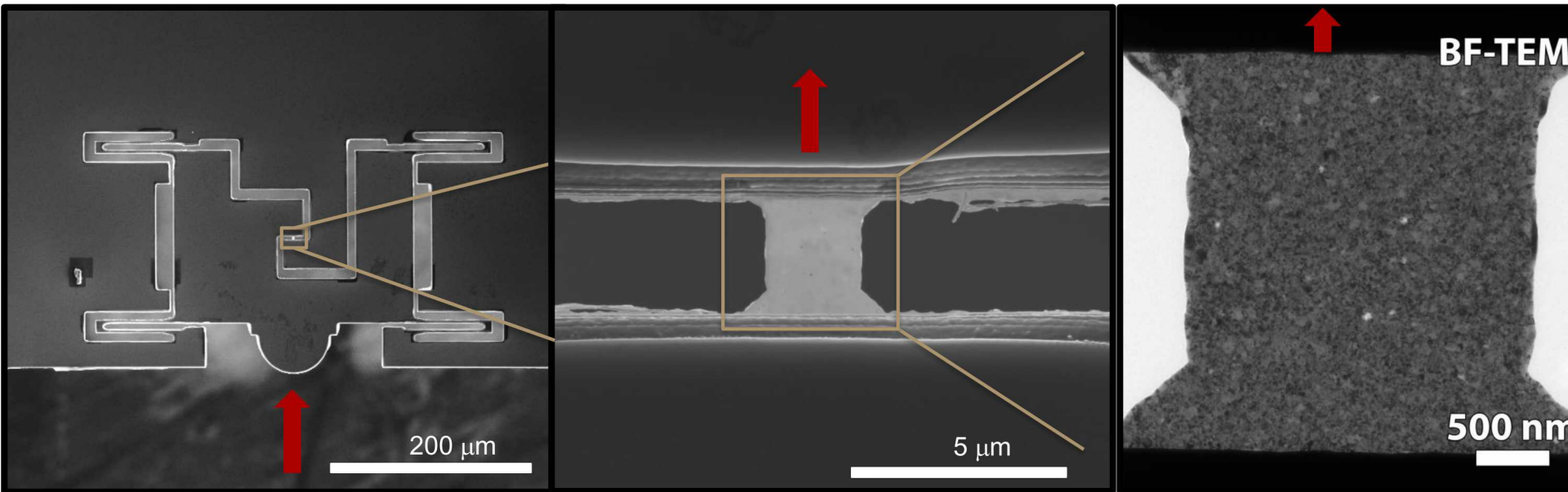
- < 8 nm spatial resolution (LaB6)
- < 2 nm spatial resolution (FEG)
- Near kinematical electron diffraction
- Symmetry ambiguities are resolved
- Fast and automated acquisition



**NanoMEGAS**  
Advanced Tools for electron diffraction

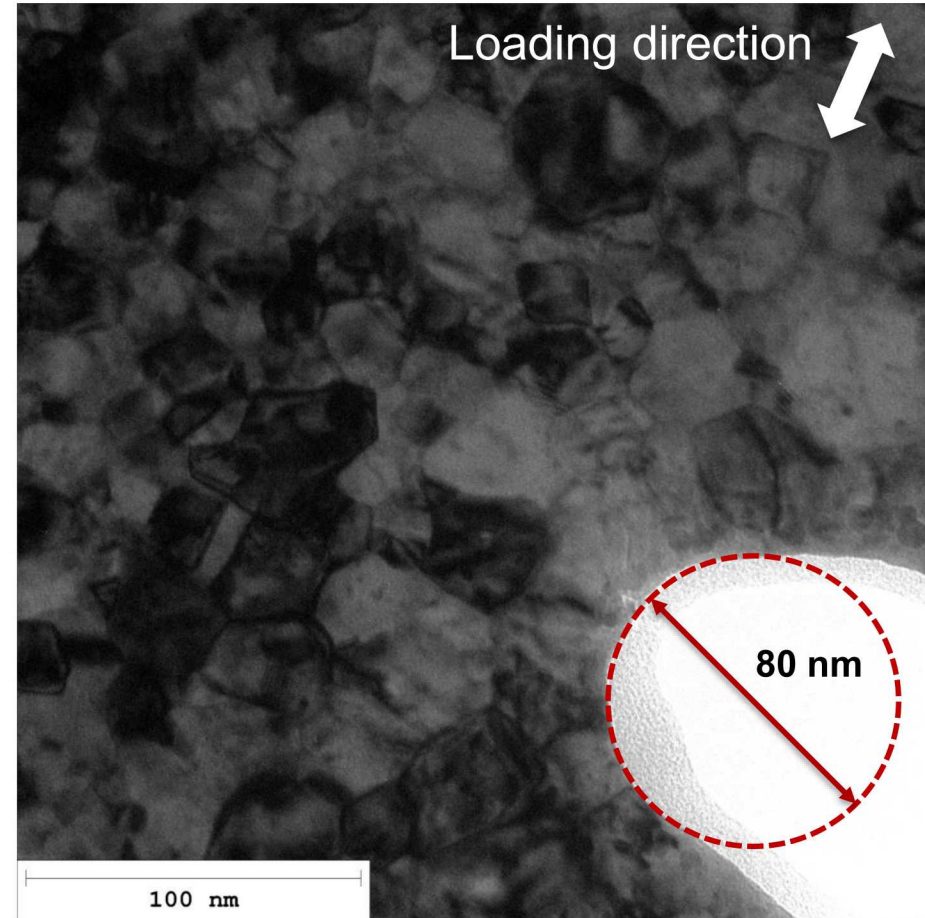
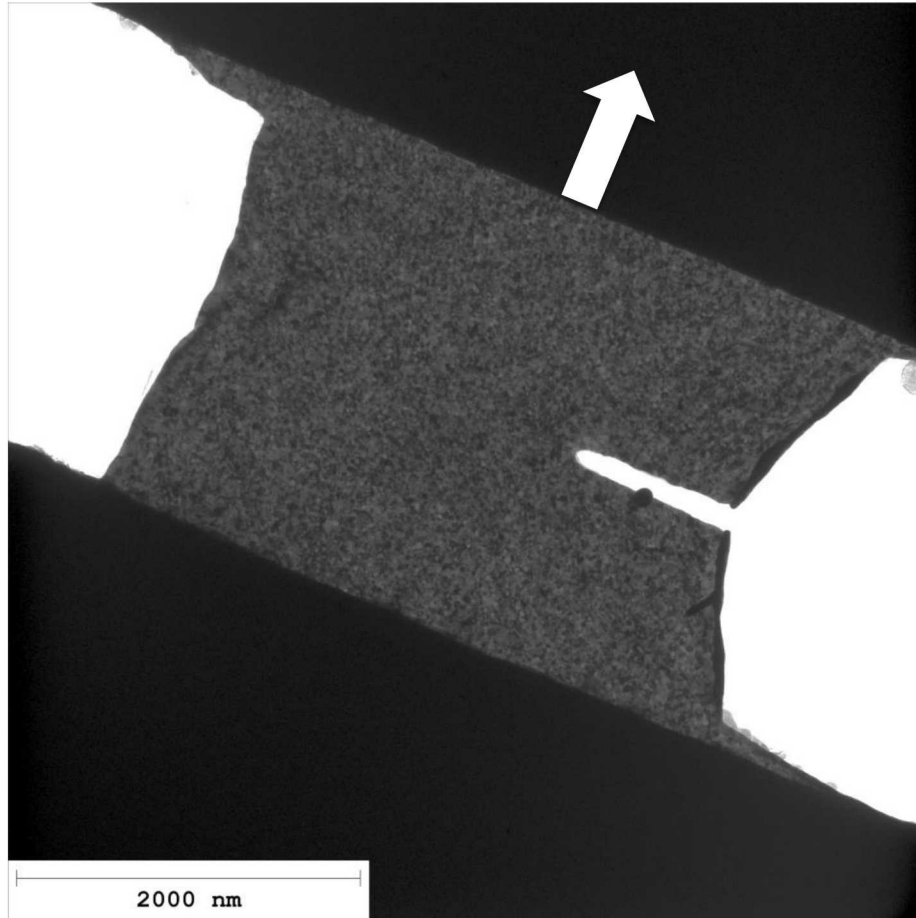
# Tension Specimen Fabrication

- Hysitron “Push-to-Pull” devices
  - Microfabricated Si test frame
  - Pt film (40nm) floated onto device, then FIB milled. Final FIB cut: minimize I-beam imaging → minimize Ga
  - Notched test → improved “chance” of observing crack initiation and propagation



- Nearly pure tension, uniform cross sectional area, stable load frame
- Sensitive to shape of edges, issues with magnetic materials
- $F_{\text{applied}} = F_{\text{measured}} - F_{\text{spring}}$

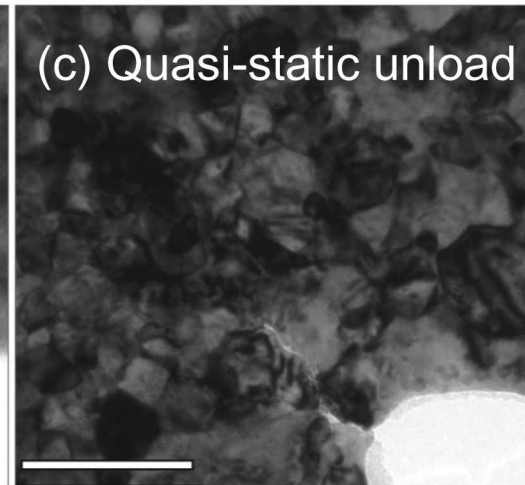
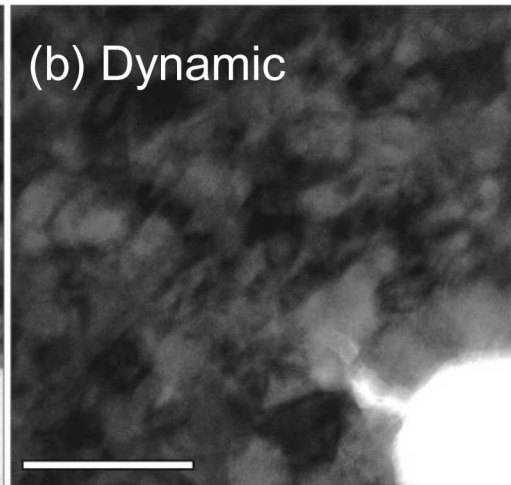
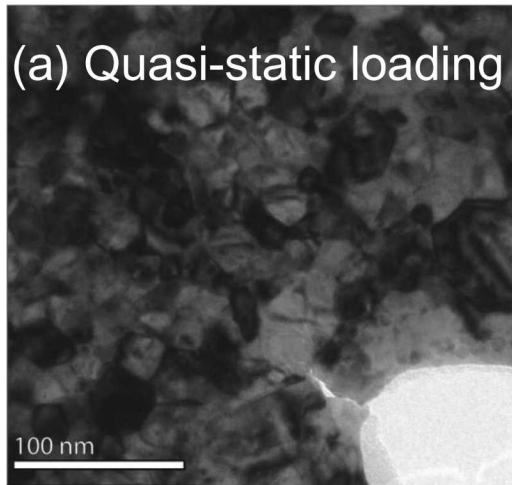
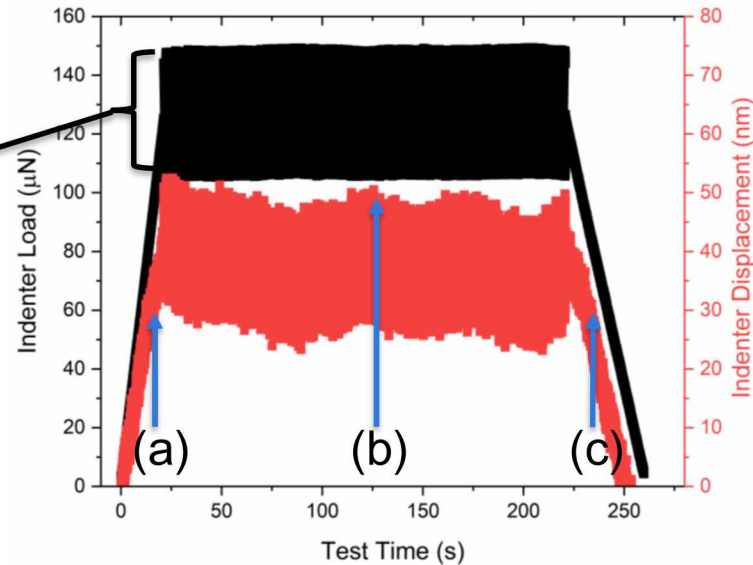
# Notched NC Pt: In-situ Cyclic Loading/Fatigue



- Notch length = 950 nm, Gauge width = 3.3  $\mu\text{m}$
- Notch created by FIB “line”

# Methodology: Cyclic Loading in TEM Protocol

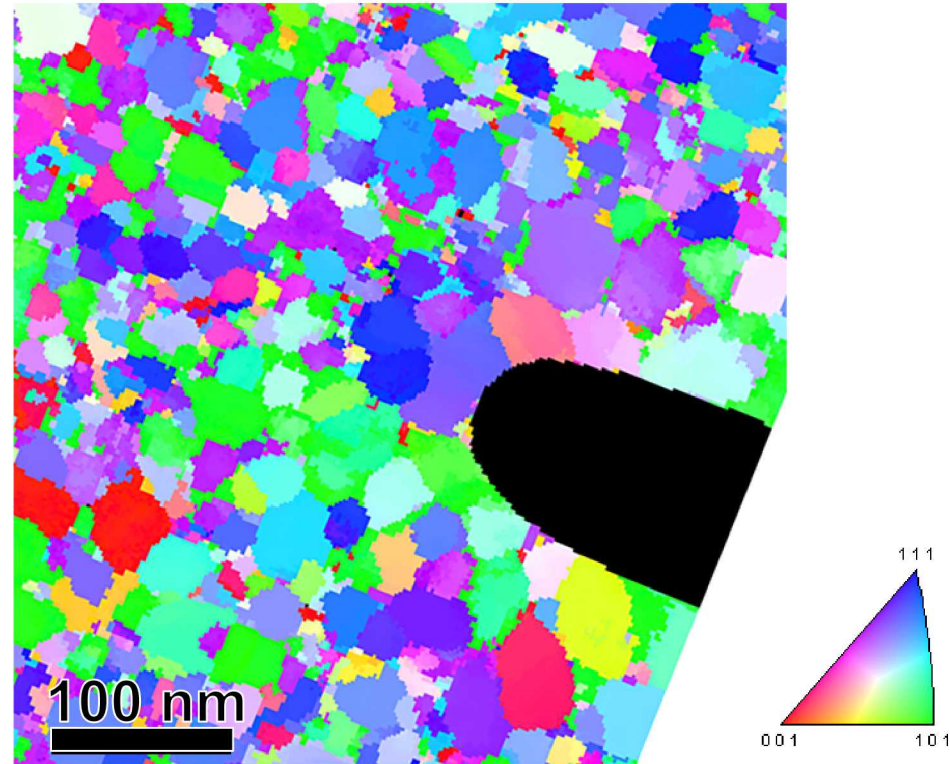
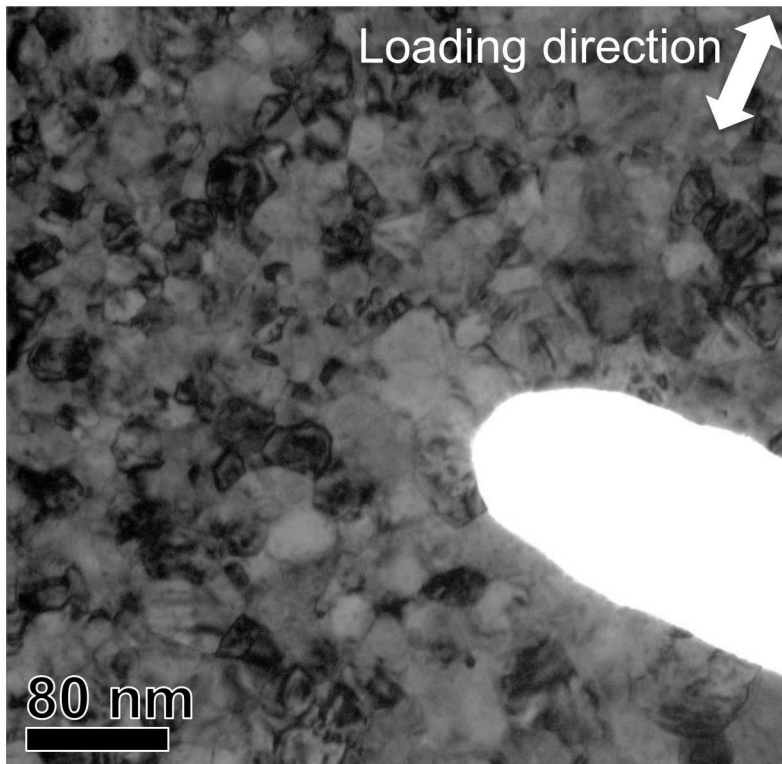
Mean load ( $P_{\text{mean}}$ ) = 135  $\mu\text{N}$   
Amplitude load ( $P_{\text{amp}}$ ) = 35  $\mu\text{N}$



- Motion blur  $\rightarrow$  loading frequency exceeded the frame rate (15 frames/s  $\rightarrow$  **13 cycles per frame**)



# Advantages of PED coupled In-situ Experiments Sandia National Laboratories

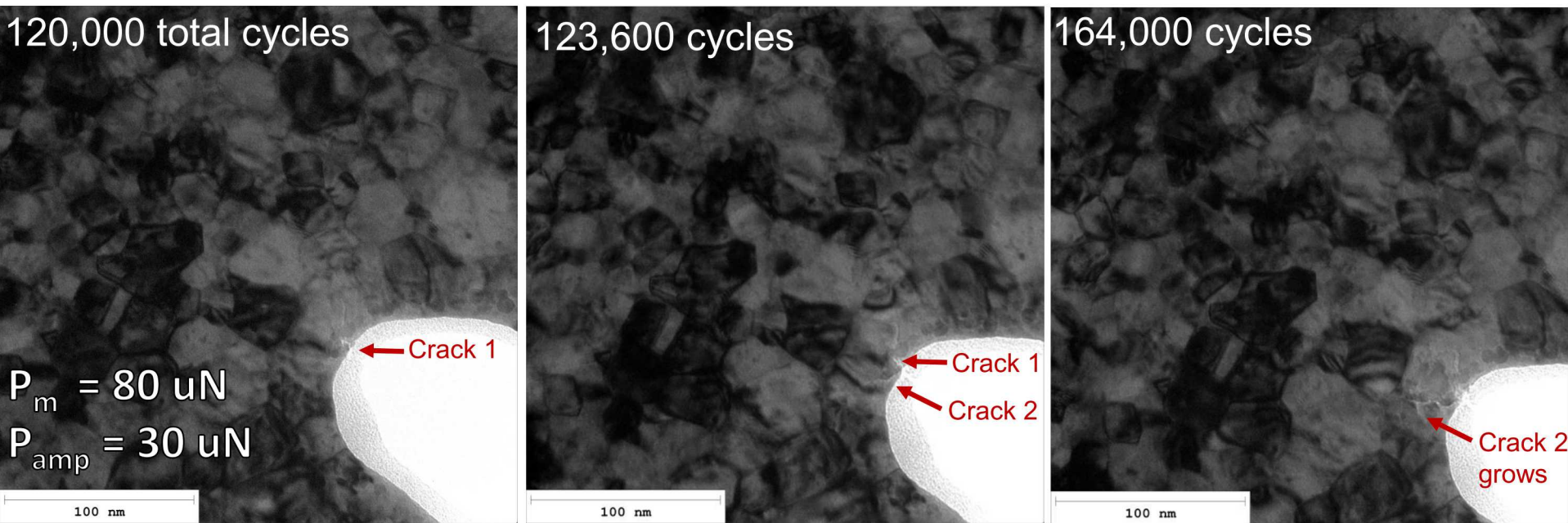


- Ability to couple grain orientation and grain boundary misorientation with crack propagation
  - Feasible to track relative grain rotation or variation in GB misorientation under loading

**PED orientation maps pre-, intermediate, and post- in-situ mechanical test can assist in deconvoluting possible NC stress assisted grain growth**

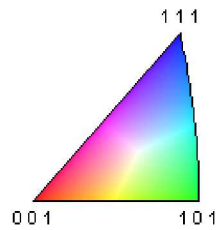
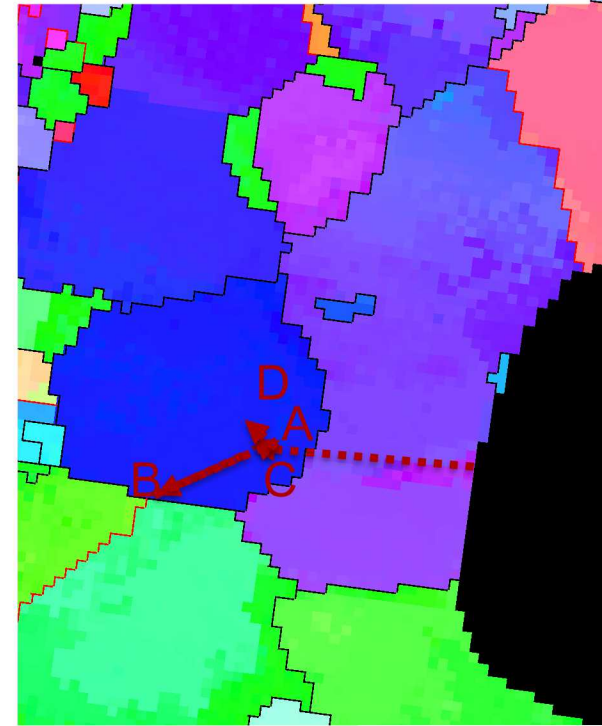
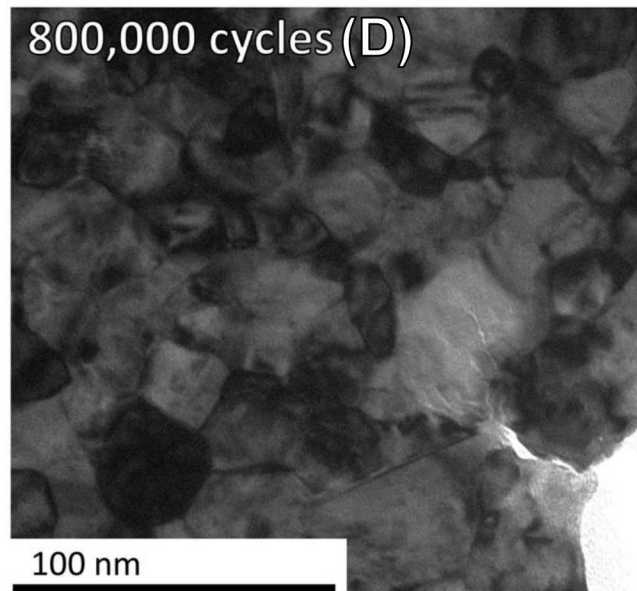
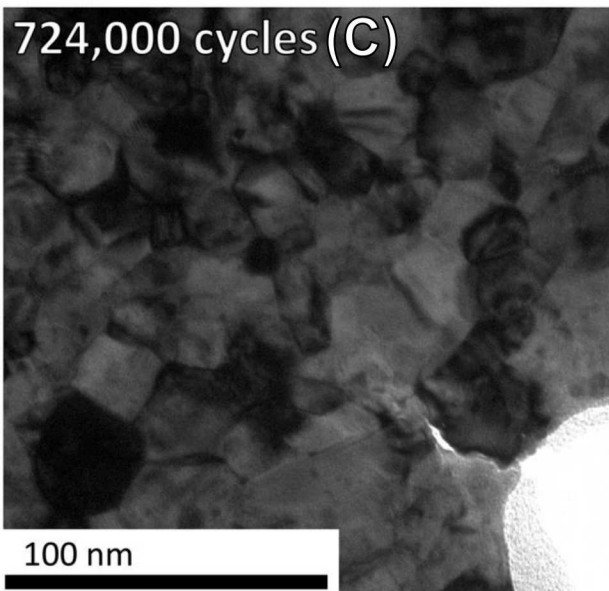
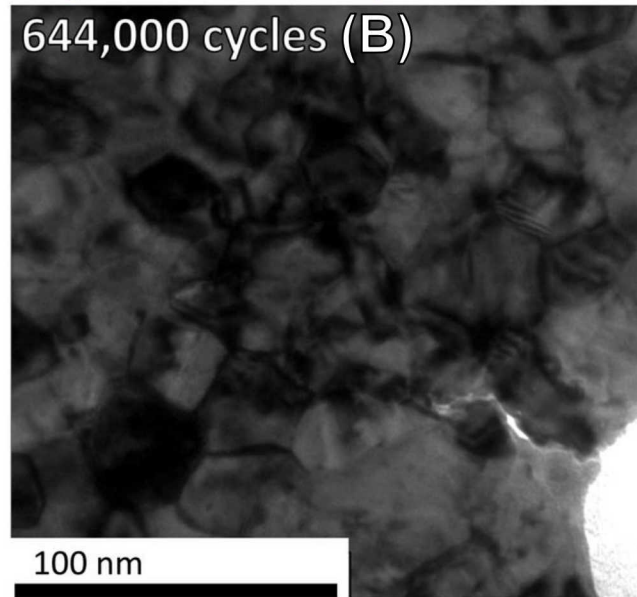
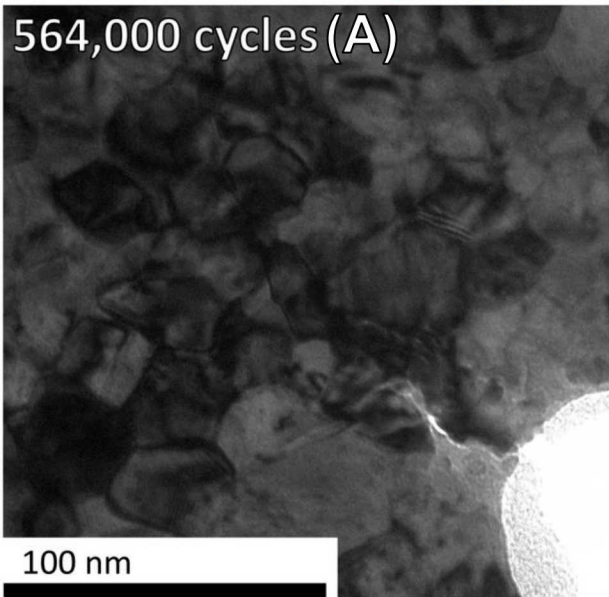


# Crack Initiation at Notch

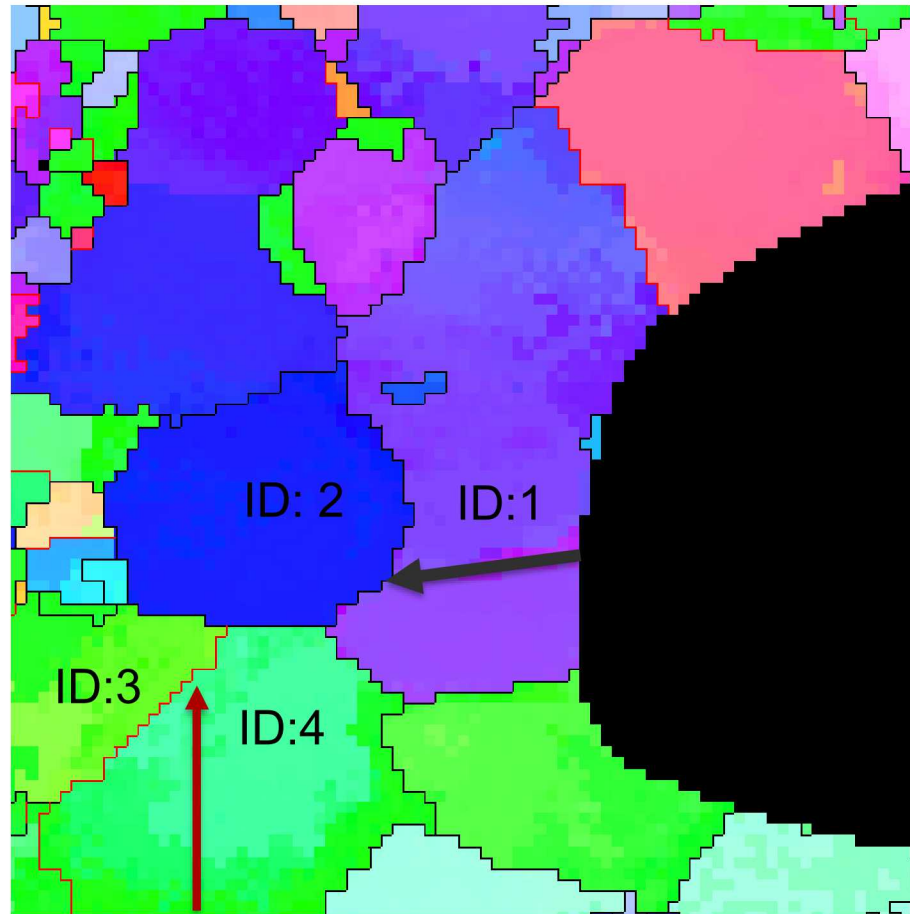


- Crack initiation and initial propagation at notch tip
- Second crack initiates at  $\sim 90^\circ$  to first crack, both  $45^\circ$  to notch tip normal
- Intra-granular crack (crack #2) propagates until reaching initial grain boundary and is subsequently arrested

# Crack Propagation, Closure, and Re-Direction



# Cyclic Loading: GB Misorientation Changes Sandia National Laboratories



$\Sigma 3$  of interest ("GB-1")

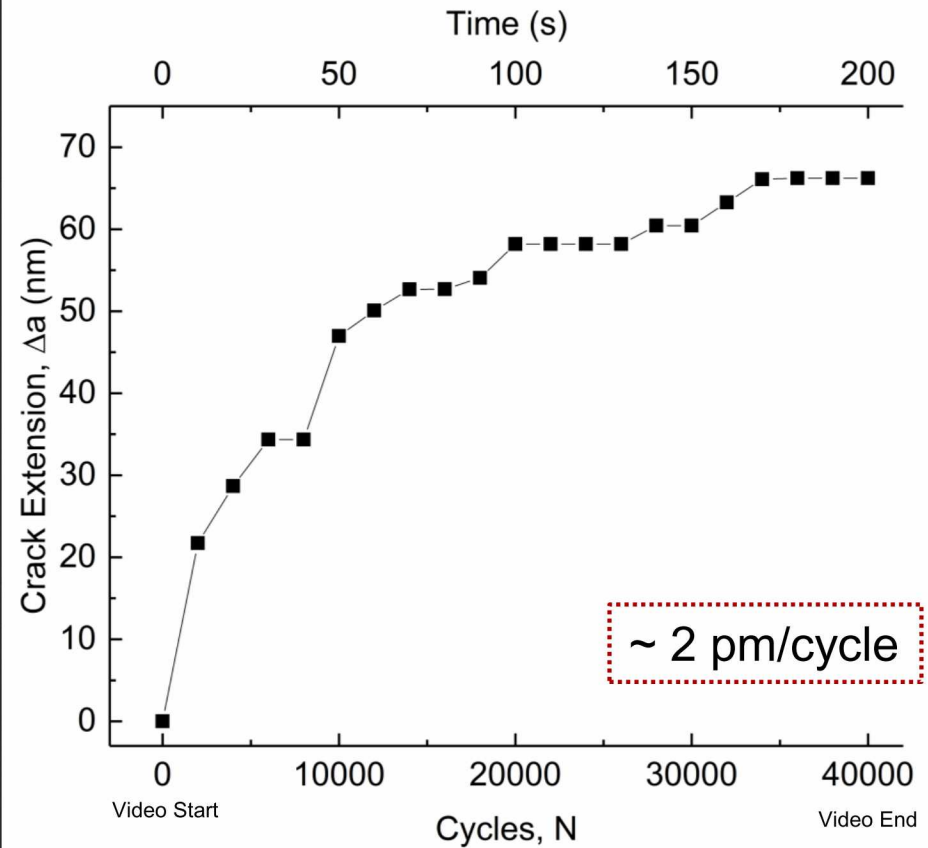
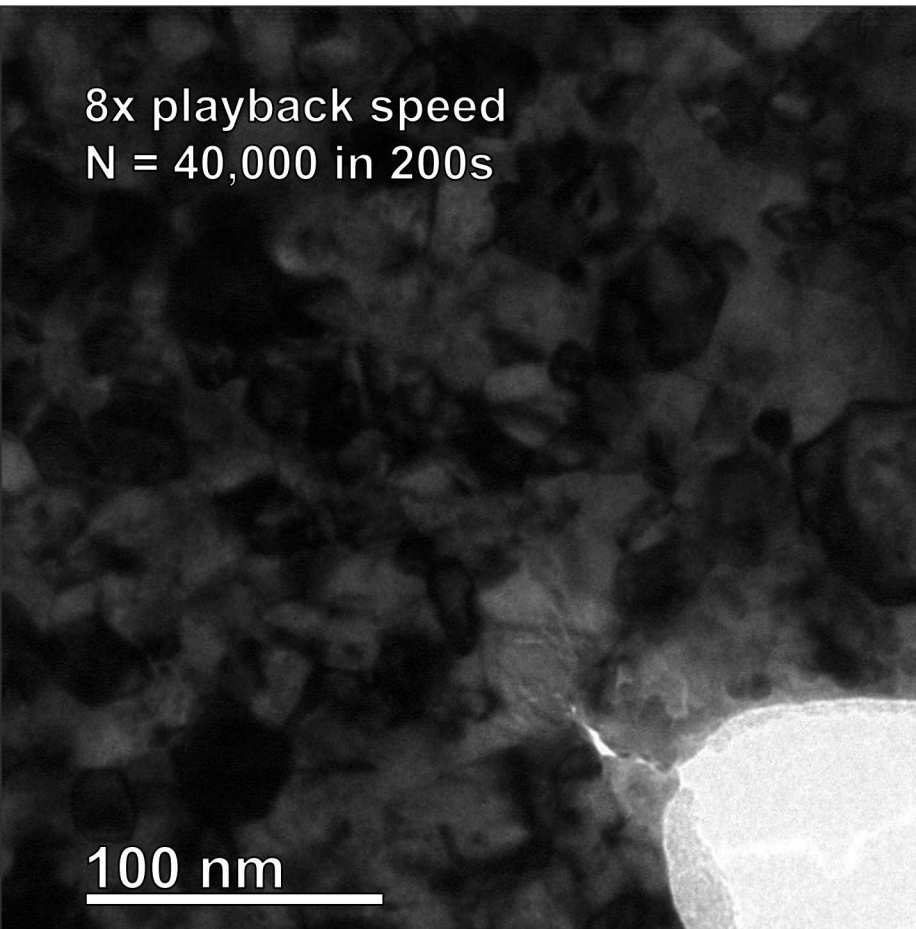
PED map at 124,000 total cycles

- Deformation and stress intensity of cyclic loaded crack tip induced modification (change in macroscopic misorientation) of the coherent  $\Sigma 3$
- Decrease in  $\Sigma 3$  coherency from nearly ideal twin misorientation to greater than  $4^\circ$  deviation likely associated:
  - Grain rotation and dislocation-GB impingement

GB-1 (Twin Boundary $\Sigma 3$ )	Misorientation	Deviation from Ideal $\Sigma 3$
Prior to Cyclic Loading	$59.9^\circ$ [1 1 1]	$0.8^\circ$
Crack Impinges GB at Grains 1-2	$56.9^\circ$ [7 7 6]	$4.9^\circ$

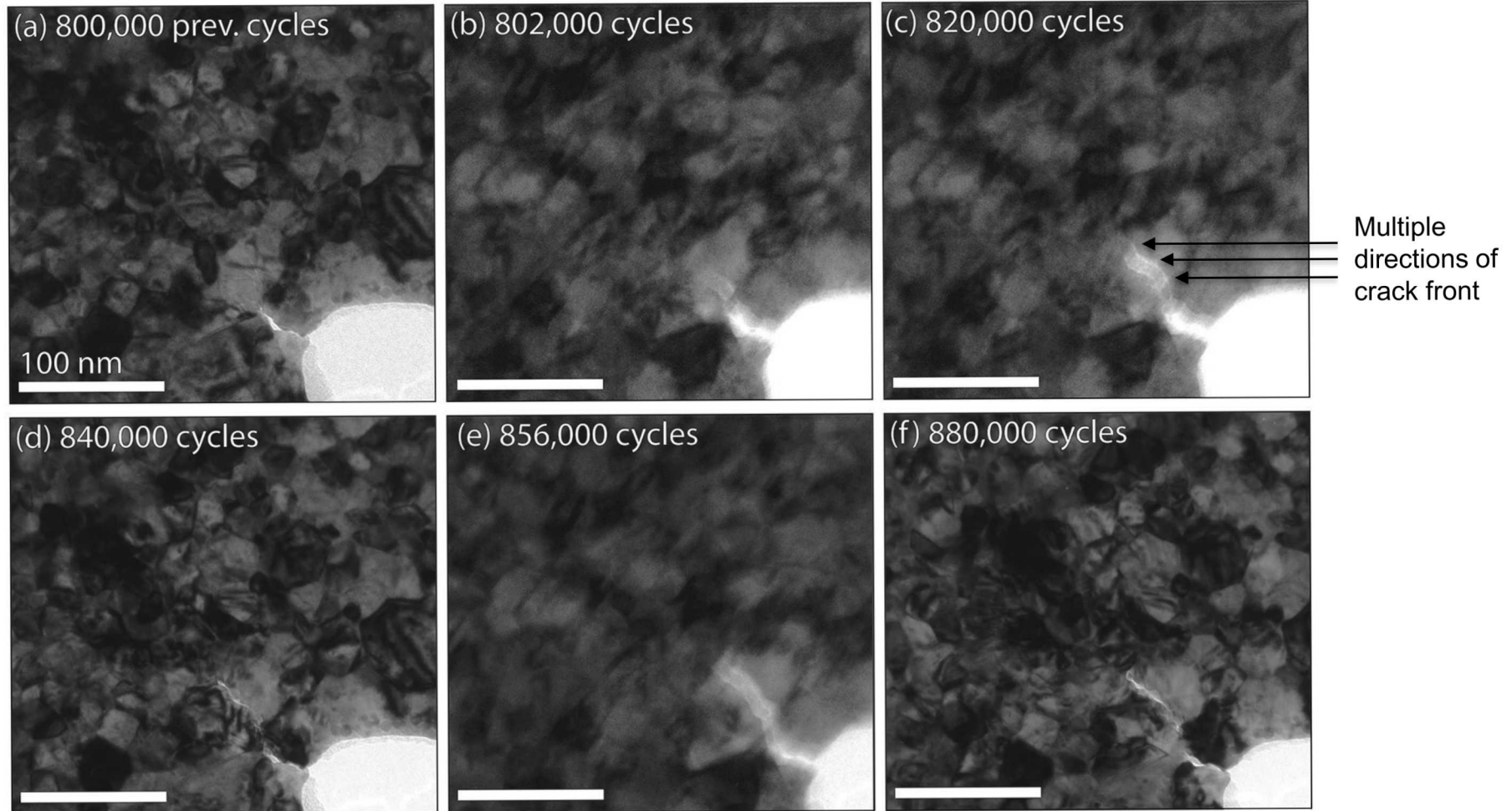


# Cyclic loading: transgranular crack propagation Sandia National Laboratories



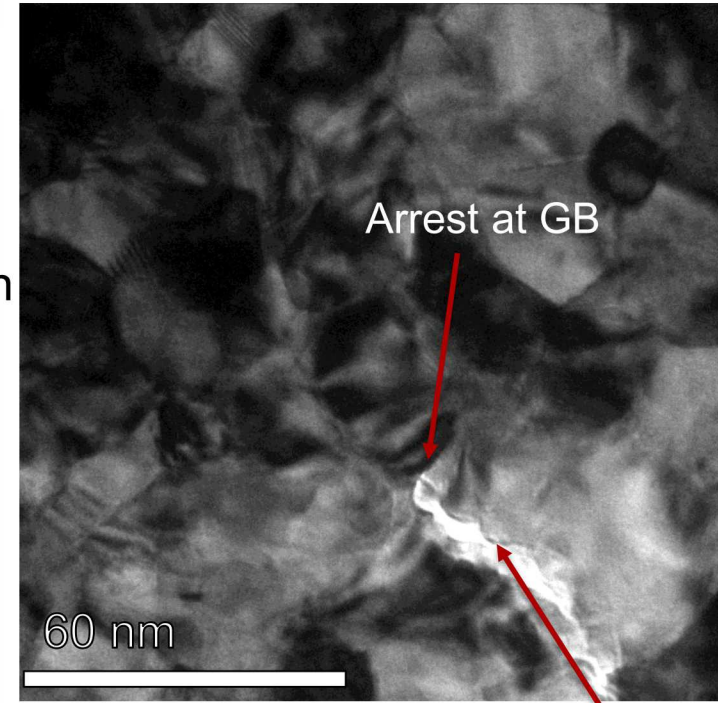
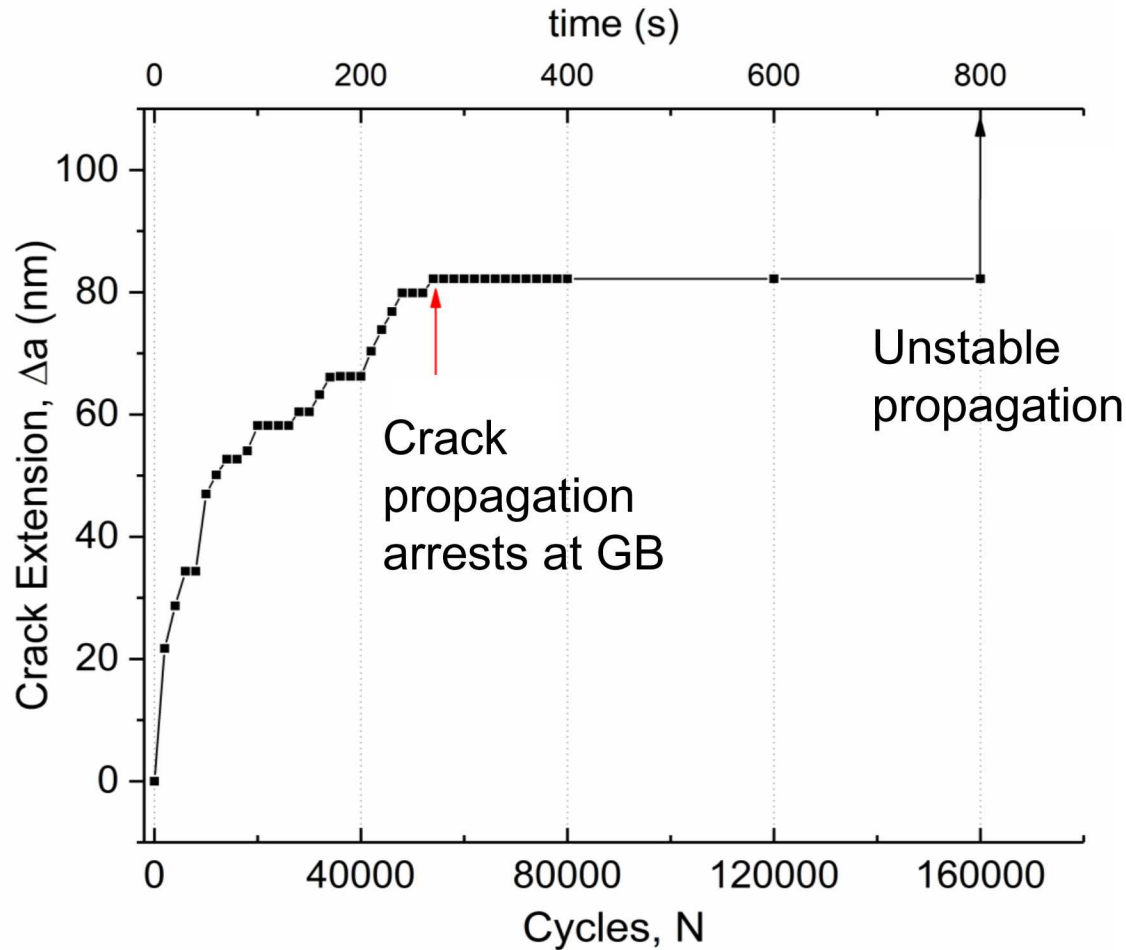
- Mean load: 135  $\mu\text{N}$ ; Amplitude load: 35  $\mu\text{N}$
- 200 Hz, 200s test (15 fps 1k x 1k camera)
- $da/dN = 1.7 \times 10^{-12} \text{ m/cycle}$
- Non-linear crack extension rate
- Crack propagation path changes “direction”

# Fatigue crack propagation and arrest



- Rapid propagation – transgranular between  $N = 800K$  to  $\sim 854K$
- After 854K cycles, cyclic loading crack impinges grain boundary – no further propagation
- Transgranular crack propagation non-linear

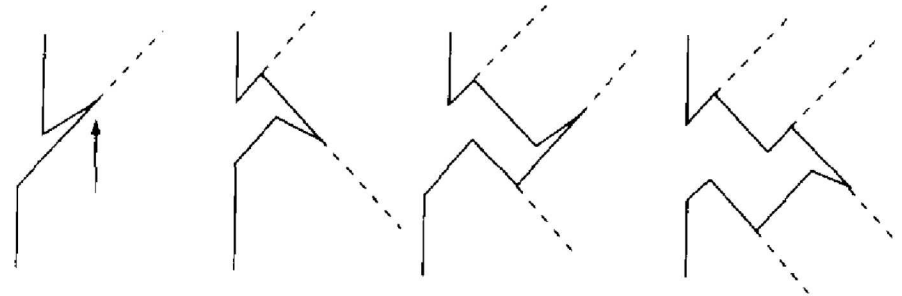
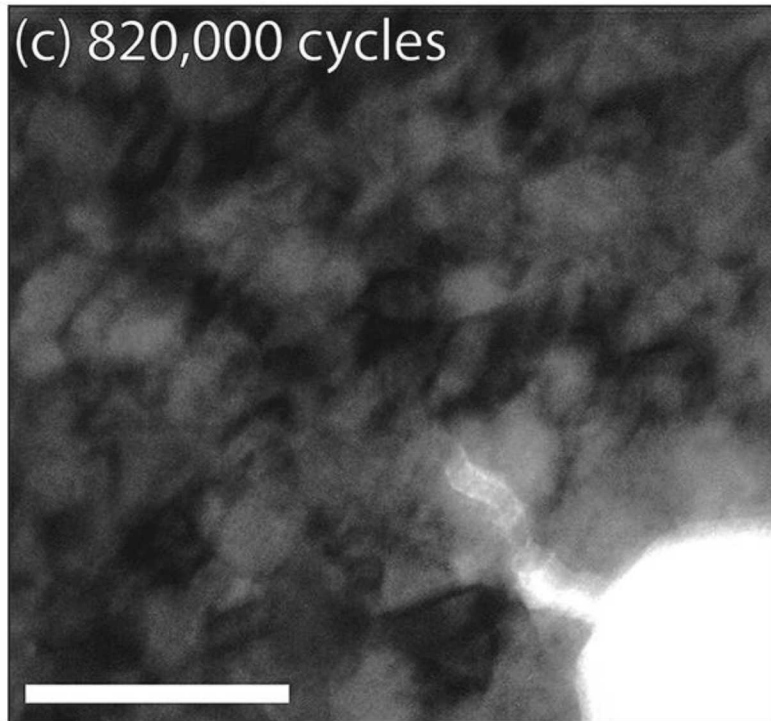
# Fatigue crack propagation and arrest



- Rapid propagation – intragranular between  $N = 800K$  to  $\sim 854K$
- After 854K cycles, cyclic loading crack impinges grain boundary – no further propagation for addition  $\sim 106K$  cycles
- Transgranular crack propagation non-linear: “serpentine fashion”



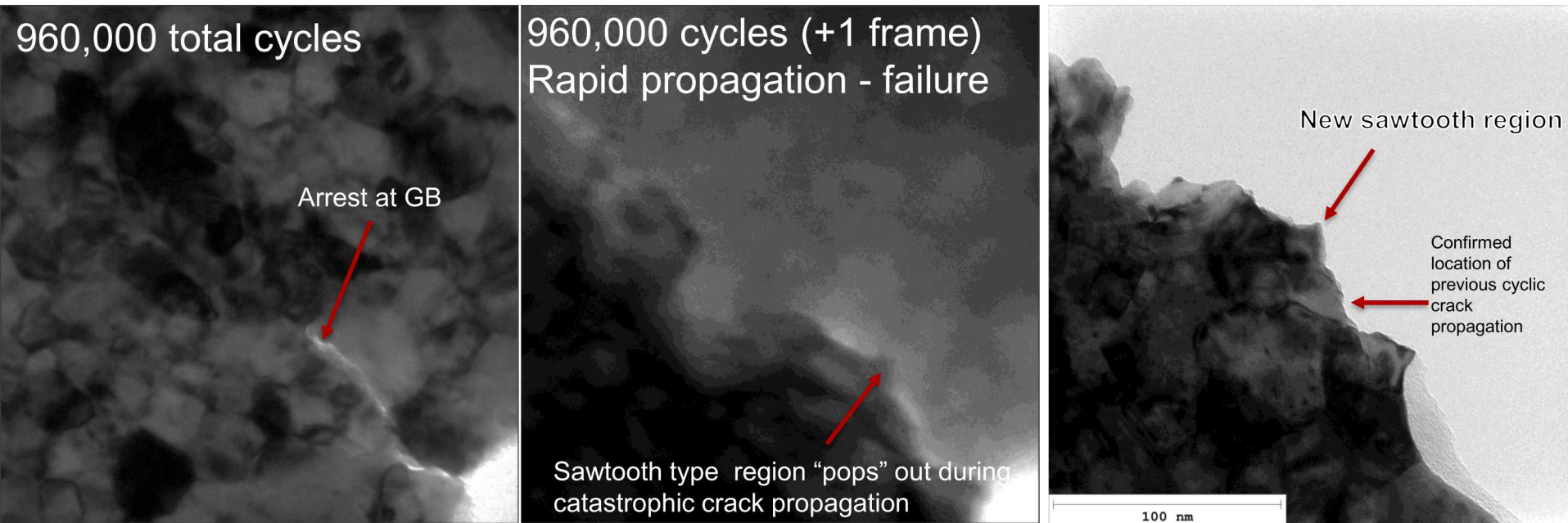
# Transgranular crack propagation: non-linear



Ohr, Mat Sci & Eng72  
(1985) 1-35

- Classic in-situ tensile straining work by Ohr and colleagues observe zig-zag crack propagation → associated with emission of dislocations on alternating slip planes
- Non-uniform “fatigue” crack propagation observed here transgranular has similar alternating crack propagation fronts – possible mechanisms still under review
- PED data provides opportunity to associate with crystal orientation (and subsequent slip plane normals)

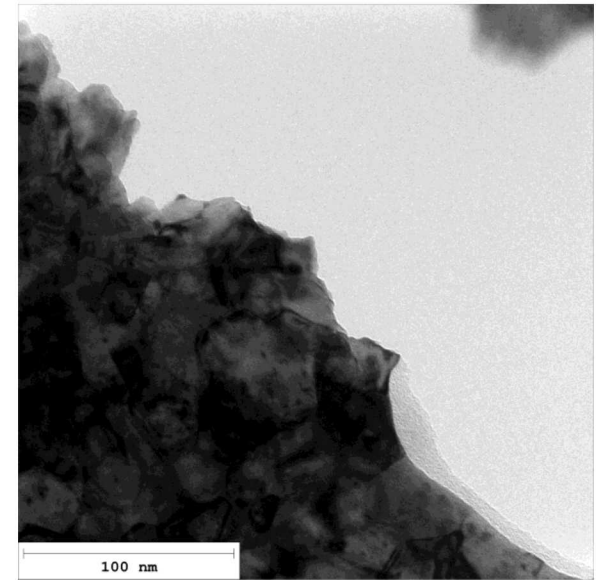
# Unstable propagation and failure



- Unstable propagation of crack occurred just under 1M total cycles
- Sample failed on initial ramp ( $\sim 100$   $\mu\text{N}$ ) to monotonic mean load (135  $\mu\text{N}$ ) at 960,000 cycles
- Fracture surface consistent with location of previous fatigue crack propagation
- Indications of “sawtooth” intra-grain plasticity during this rapid failure mechanism

# Discussion and Conclusions:

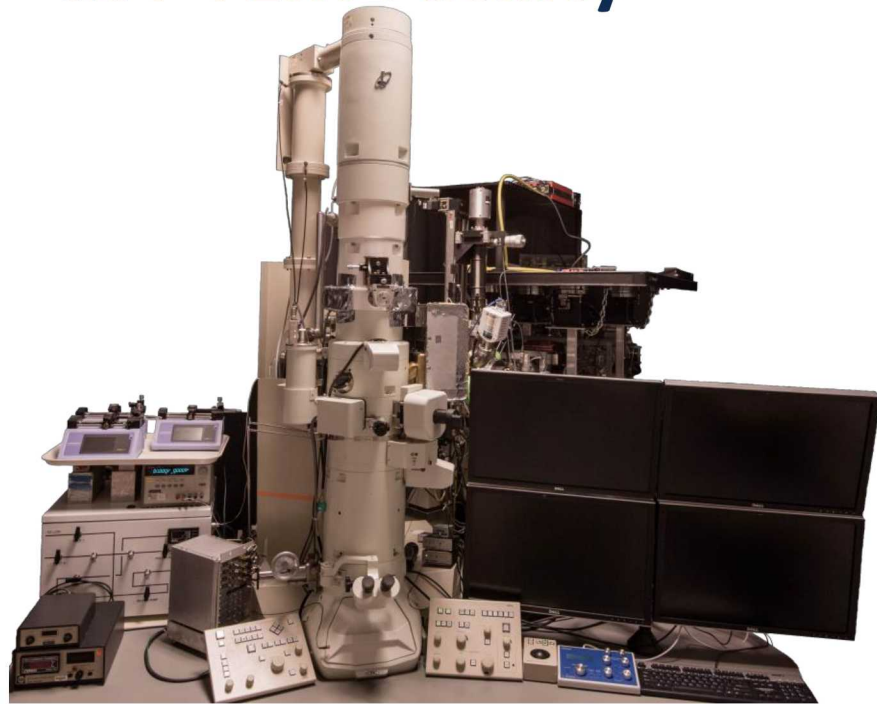
- PI-95 in tension-tension “fatigue” mode (nanoDMA) provide wealth of new in-situ TEM mechanical testing potential
  - Ideally coupled with ACOM TEM or other methods to enhance analysis
- Dual crack tip initiation observed at notch
- **Cyclic load induced crack propagation is effectively arrested at GBs** – indications of effectiveness of NC materials under fatigue
- **~ 2 pm/cycle crack growth rate (dA/dN)!**
  - **Non-uniform, non-straight crack propagation – “zig-zag” motion**
- **Localized deformation:** Coupled ACOM-TEM indicates grain rotation, dislocation based plasticity active → **clear change in  $\Sigma 3$  coherency in front of crack tip**
- **Failure associated with classical in-situ TEM mechanical “saw-tooth” plasticity**



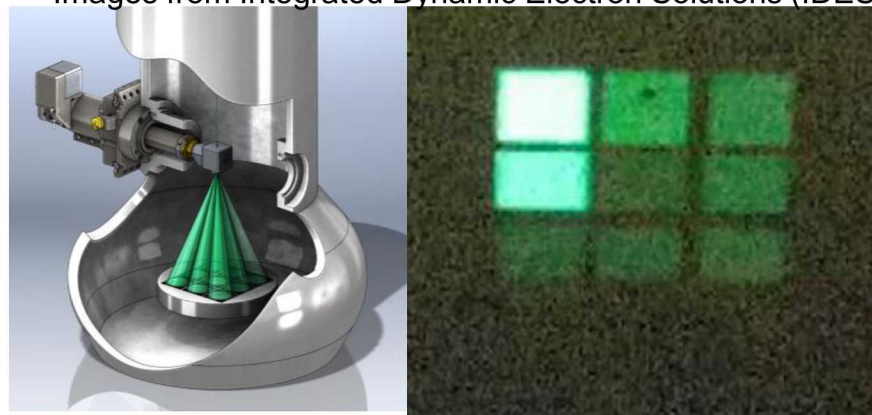
Saw-tooth classically explained by local plastic flow – (e.g. Wilsdorf and Kumar et al. Acta 2003)



# Future In-Situ Mechanical Testing Directions at I<sup>3</sup>TEM Facility



Images from Integrated Dynamic Electron Solutions (IDES):

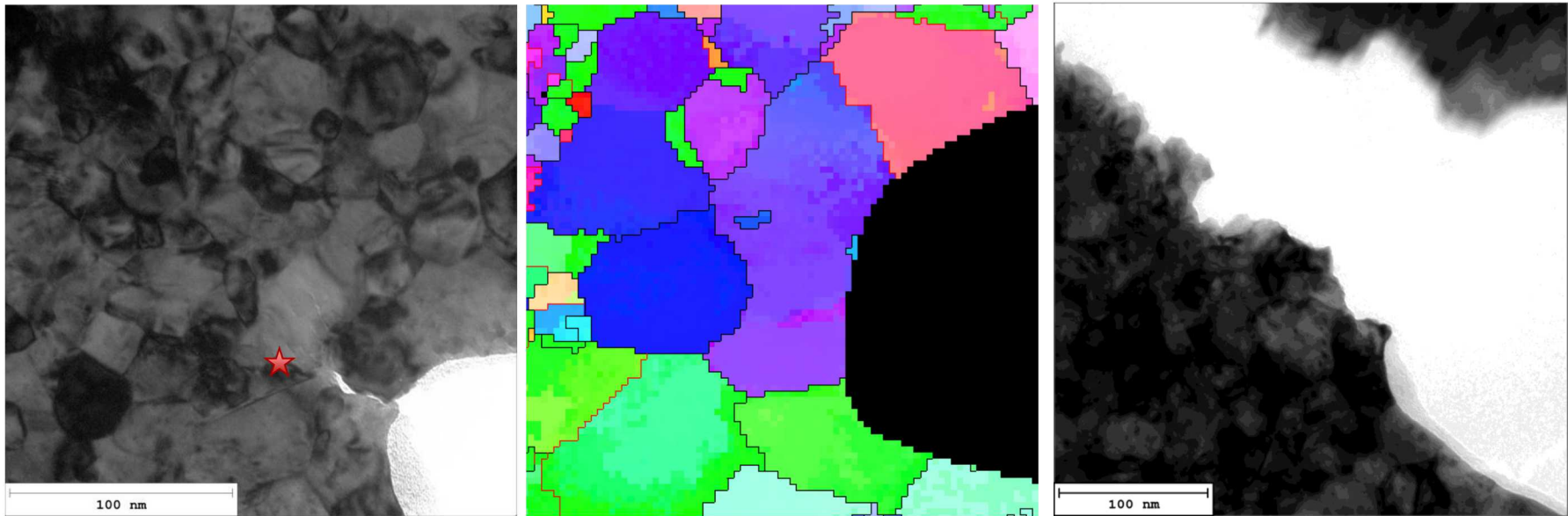


- High cycle fatigue – more detailed quantification of grain growth
  - Other model alloys, multi-layers, alloying effects (e.g. Pt-10Au vs. Pt)
- Creep, radiation-induced creep
- TEM notched three point bend
- Under development capabilities:
  - DTEM and/or movie mode with crack propagation for improved temporal resolution under high Hz cyclic loading

Combining the precision of Hysitron's Pico-indenter with harsh environments capable in Sandia's In-situ Ion Irradiation TEM a wealth of previously impossible experiments are now feasible.

# Summary:

**On-going efforts to combining PED/ACOM with quantitative mechanical testing provides new correlations between structure-property relationships with unprecedented orientation and mechanical property information.**



Work performed by C.M.B, D.C.B., B.B, K.H. was fully supported by the Division of Materials Science and Engineering, Office of Basic Energy Sciences, U.S. Department of Energy. FIB sample preparation was performed, in part, at the Center for Integrated Nanotechnologies, an Office of Science User Facility operated for the U.S. Department of Energy (DOE) Office of Science.

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