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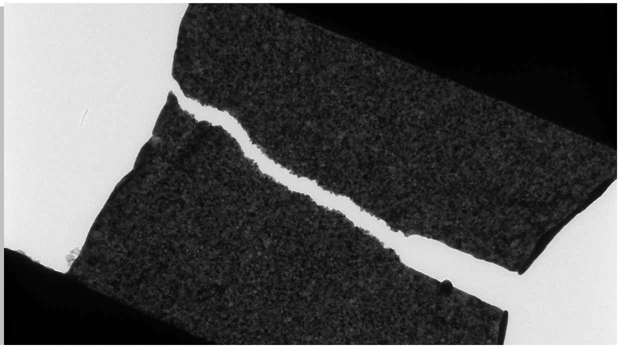
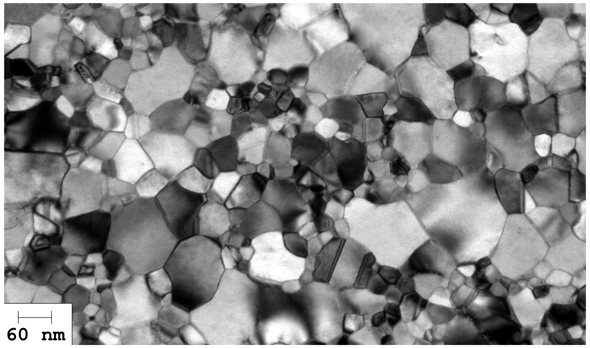

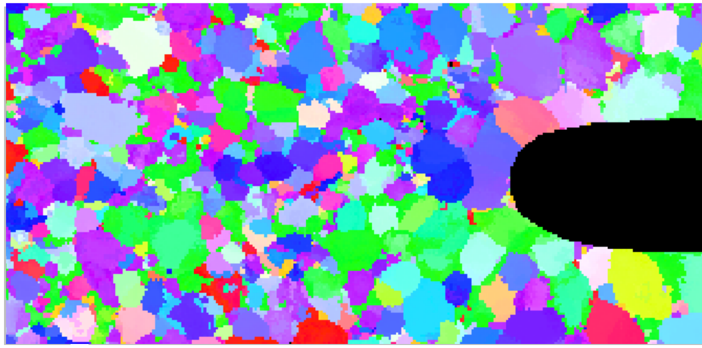

Investigation of Grain Growth in Nanocrystalline Alloys through Coupled In-situ TEM Fatigue and Crystallographic Orientation Mapping

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

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U.S. DEPARTMENT OF ENERGY

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Nanocrystalline Alloys: Fatigue and Fracture

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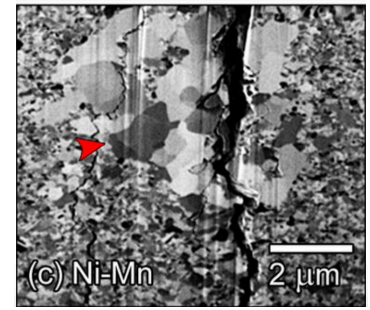
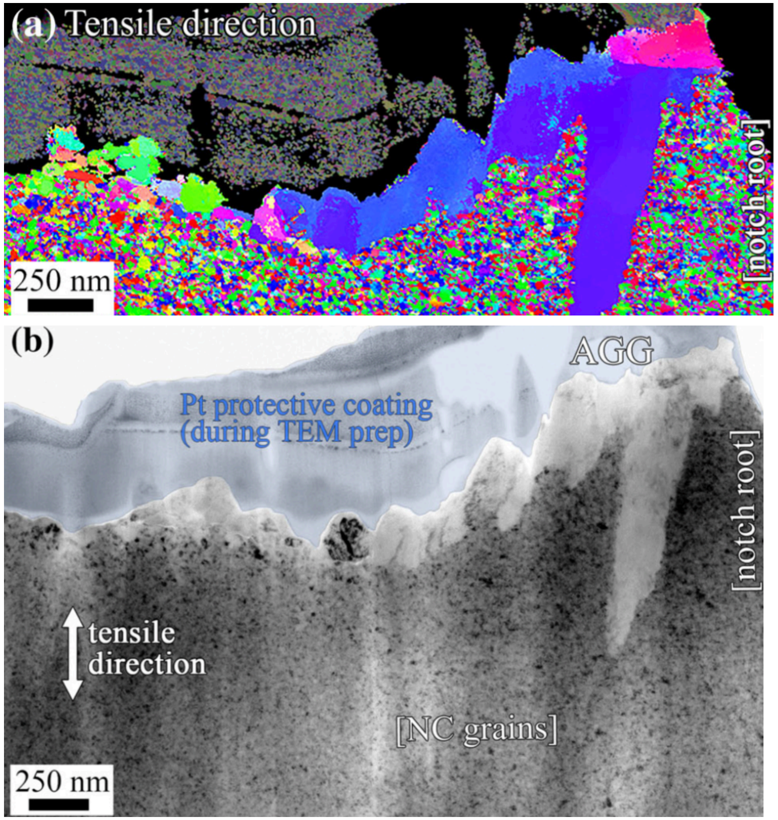
- **Improved fatigue resistance compared to coarse grain counterparts**
- Progressive microstructural changes with cyclic loading, often below yield stress
- Fatigue in nanocrystalline metals
  - Grain boundary migration and grain growth
  - Crack initiation
- **What are the underlying mechanisms associated 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)



T.A. Furnish et al., J. Mat Sci 52:46-59 (2017)

In situ Quantitative Mechanical Testing

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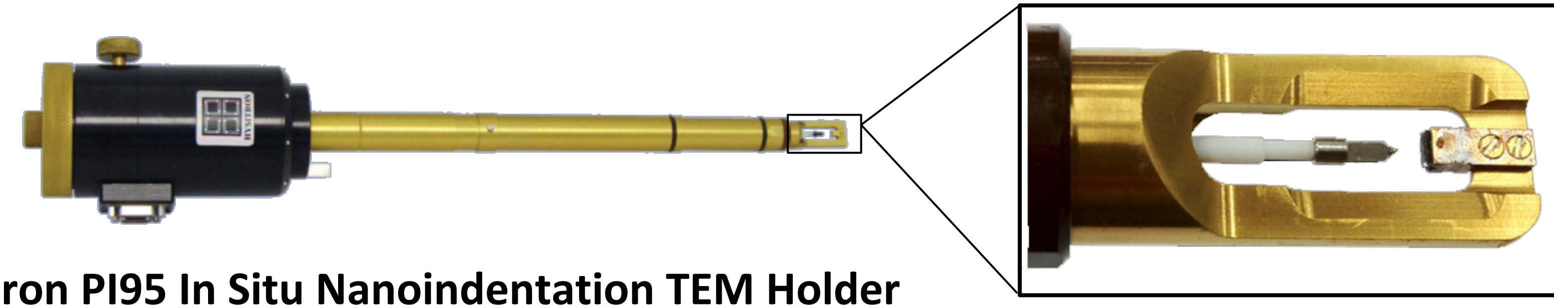
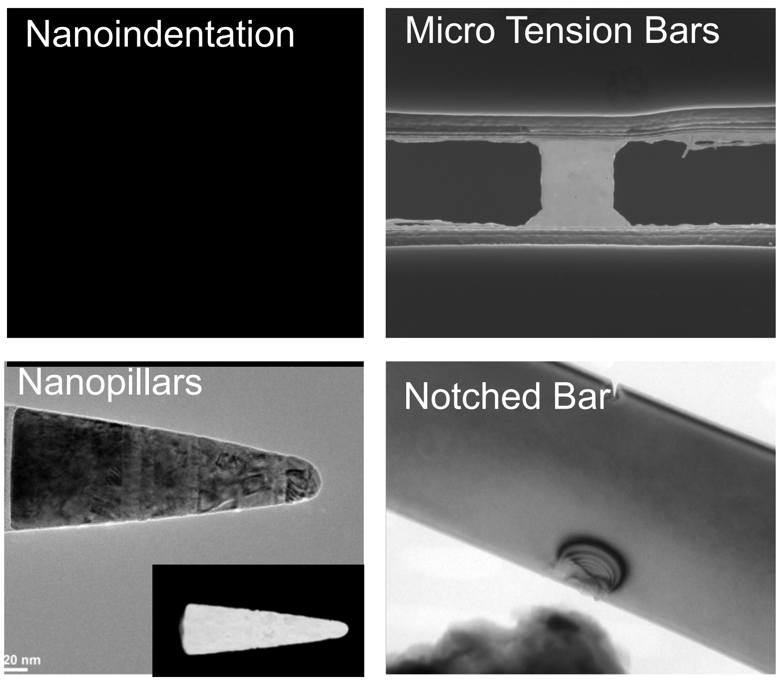


Hysitron PI95 In Situ Nanoindentation TEM Holder

- **Sub nanometer displacement resolution**
- **Quantitative force information with µN resolution**
- **Concurrent real-time imaging**



- A variety of sample geometries
- Load functions examined at I³TEM:
  1) Indentation
  2) Tension
  3) Fatigue
  4) Creep (irradiation and thermal)
  5) Compression
  6) Future: Nanowear

Precession Electron Diffraction (PED) Microscopy

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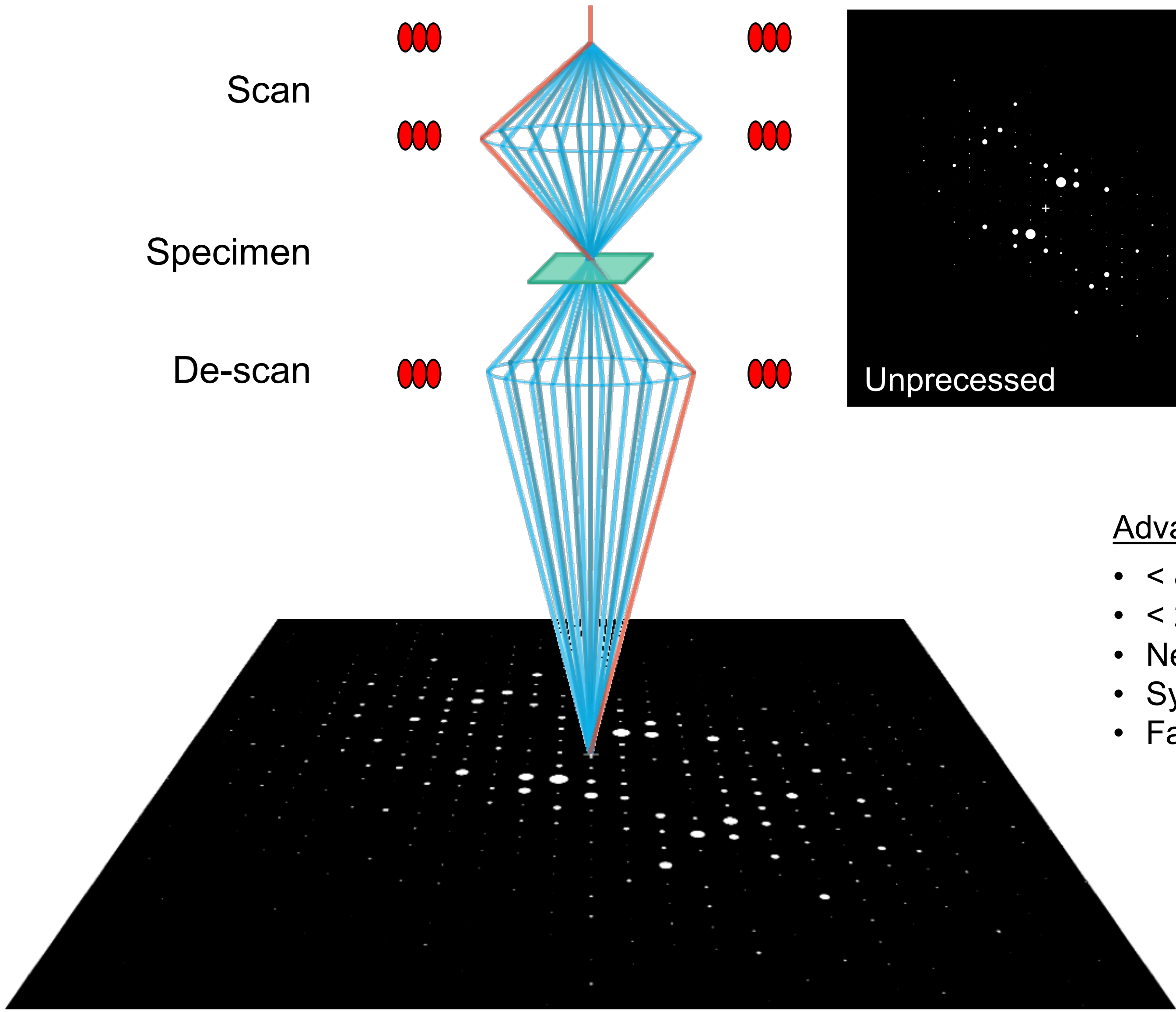
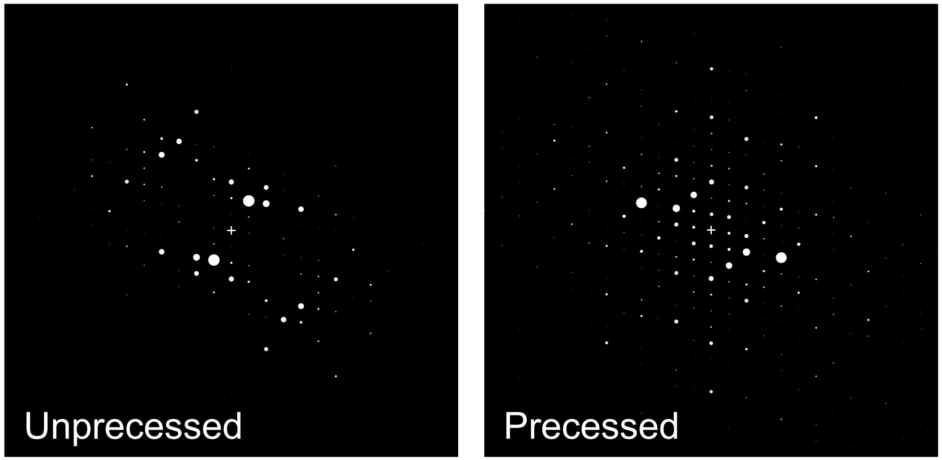




Advantages

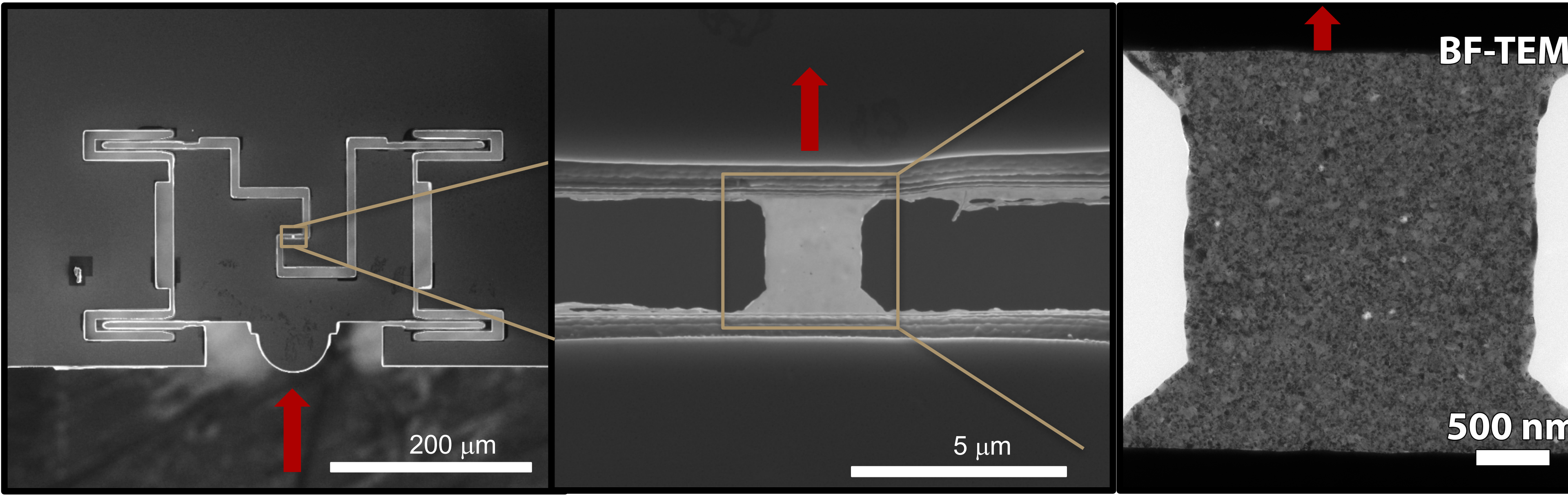
- < 8 nm spatial resolution (LaB6)
- < 2 nm spatial resolution (FEG)
- Near kinematical electron diffraction
- Symmetry ambiguities are resolved
- Fast and automated acquisition

App Five   NanoMEGAS Advanced Tools for electron diffraction

Chris Own, PhD Dissertation, Northwestern University, 2004   Slide courtesy: K. J. Ganesh, K. Hattar

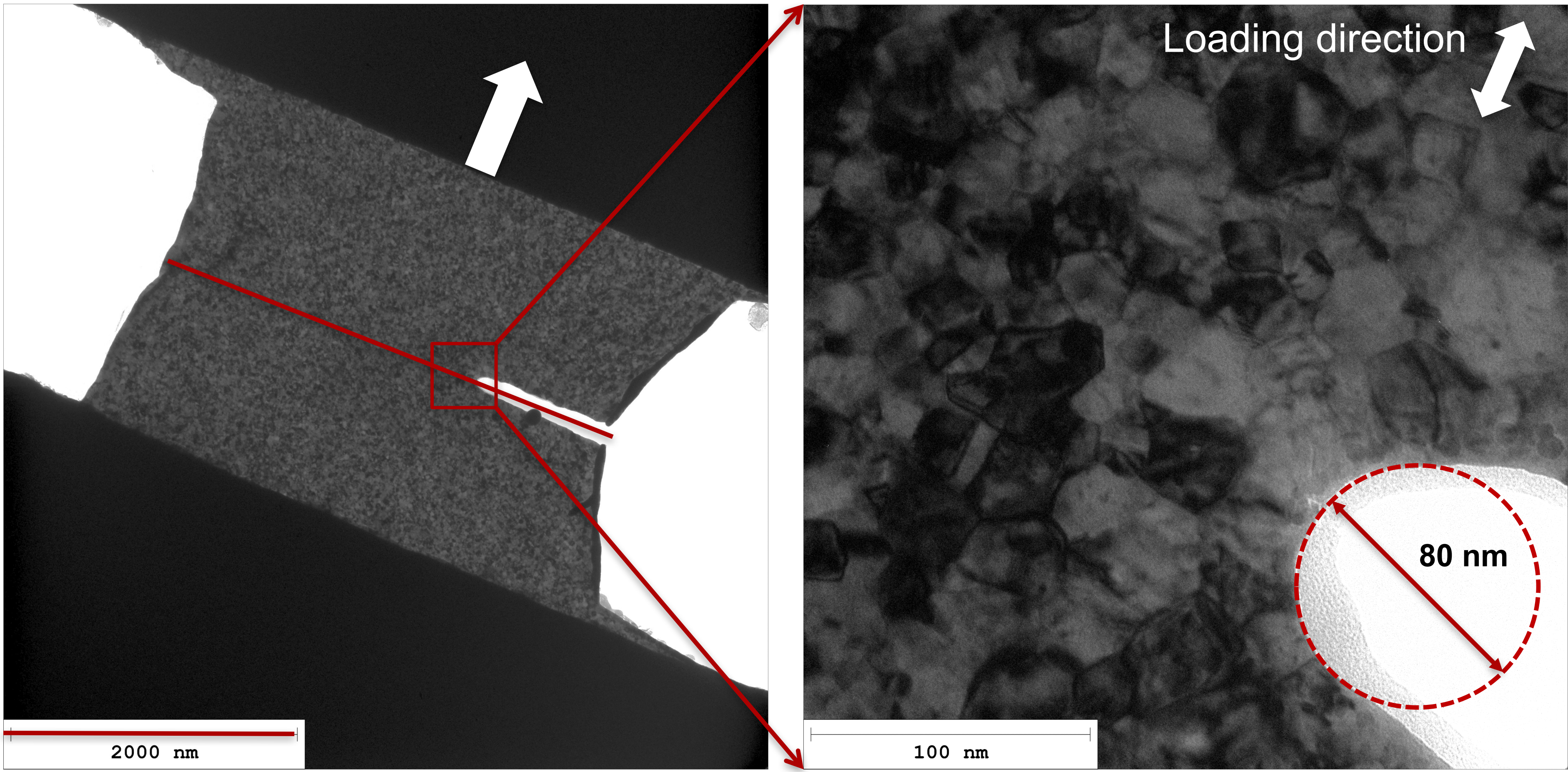
Tension Specimen Fabrication

- 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
- $F_{applied} = F_{measured} - F_{spring}$

Notched NC Pt: In-situ Cyclic Loading/Fatigue



- Notch length = 950 nm, Gauge width = 3.3 um
- Notch created by FIB "line"

Methodology: Cyclic Loading in TEM Protocol

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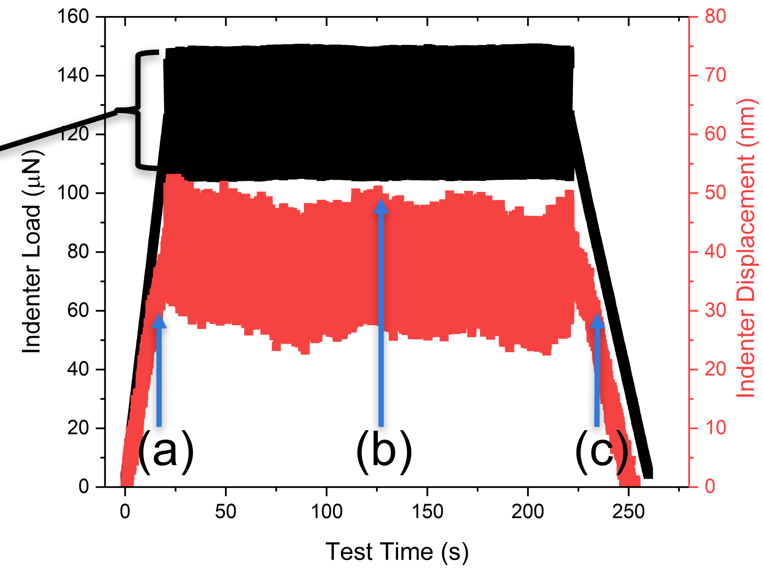
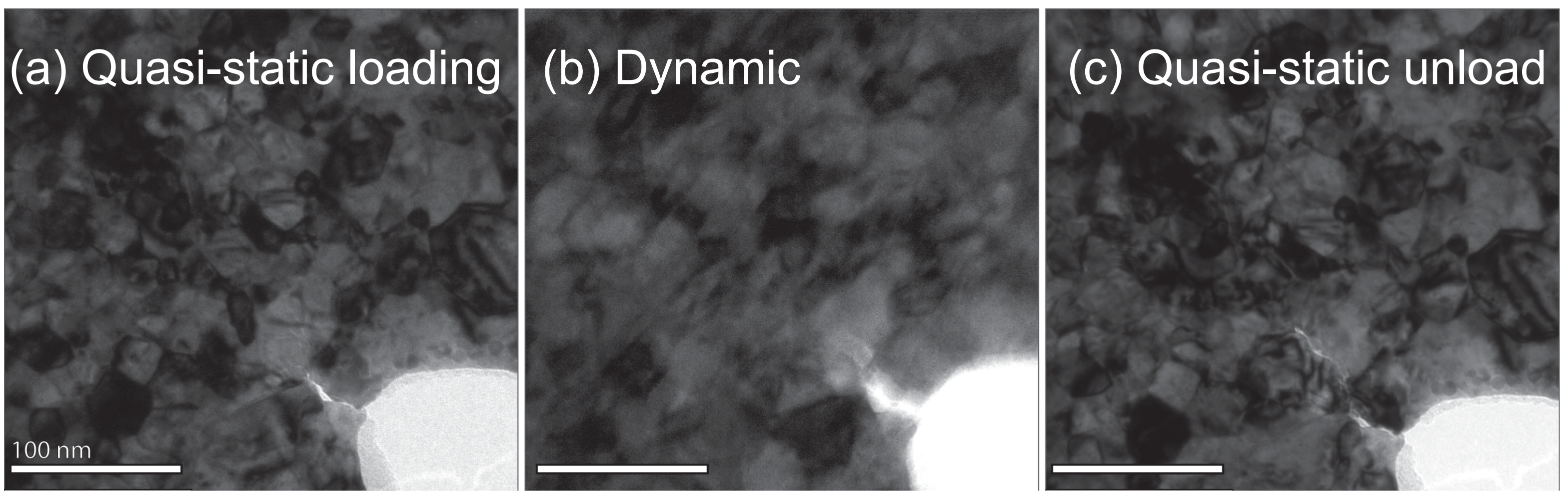
Mean load (P_{mean}) = 135 uN

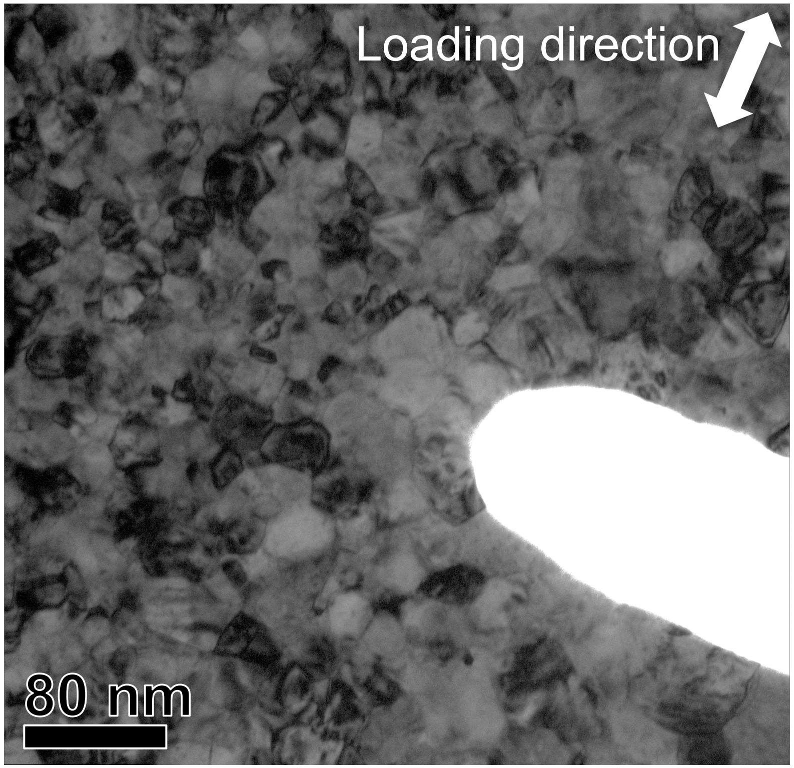
Amplitude load (P_{amp}) = 35 uN

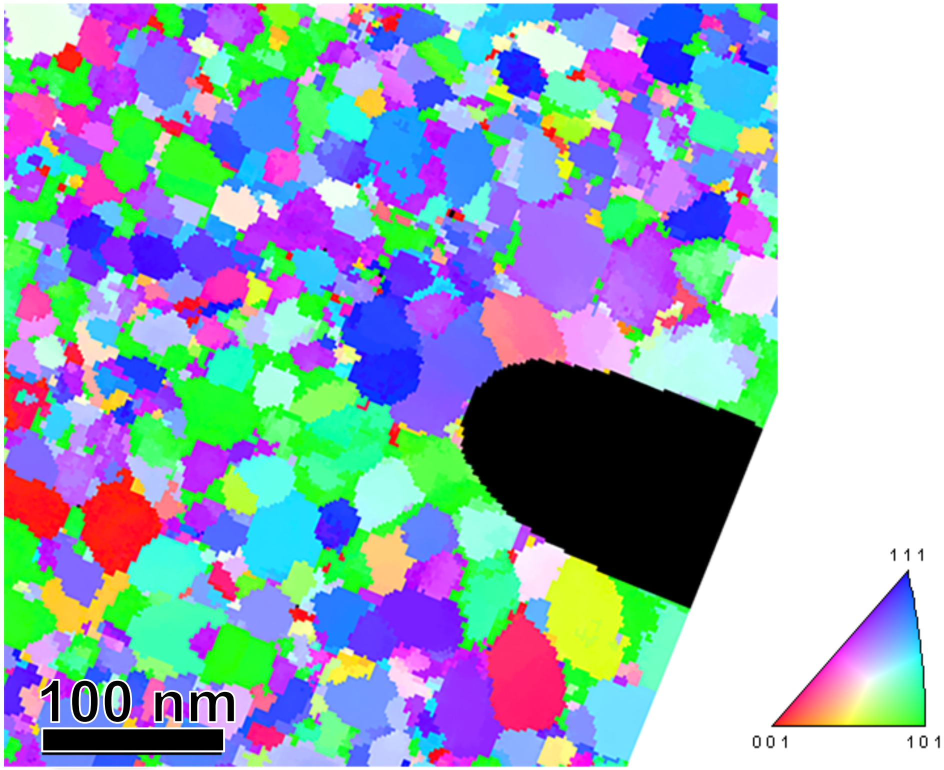




- Motion blur → loading frequency exceeded the frame rate (15 frames/s →**13 cycles per frame**)

Advantages of PED coupled In-situ Experiments

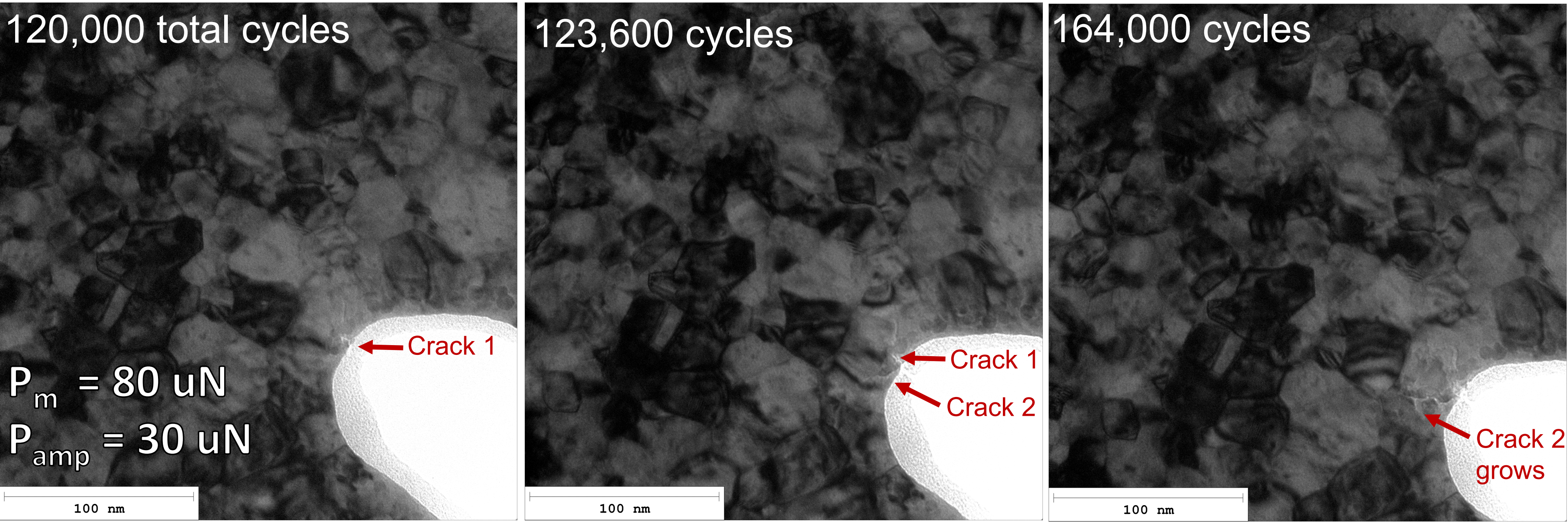




- Ability to couple grain orientation and grain boundary misorientation with crack propagation
  - Feasible to track relative grain rotation or variation in GB misorientation under loading

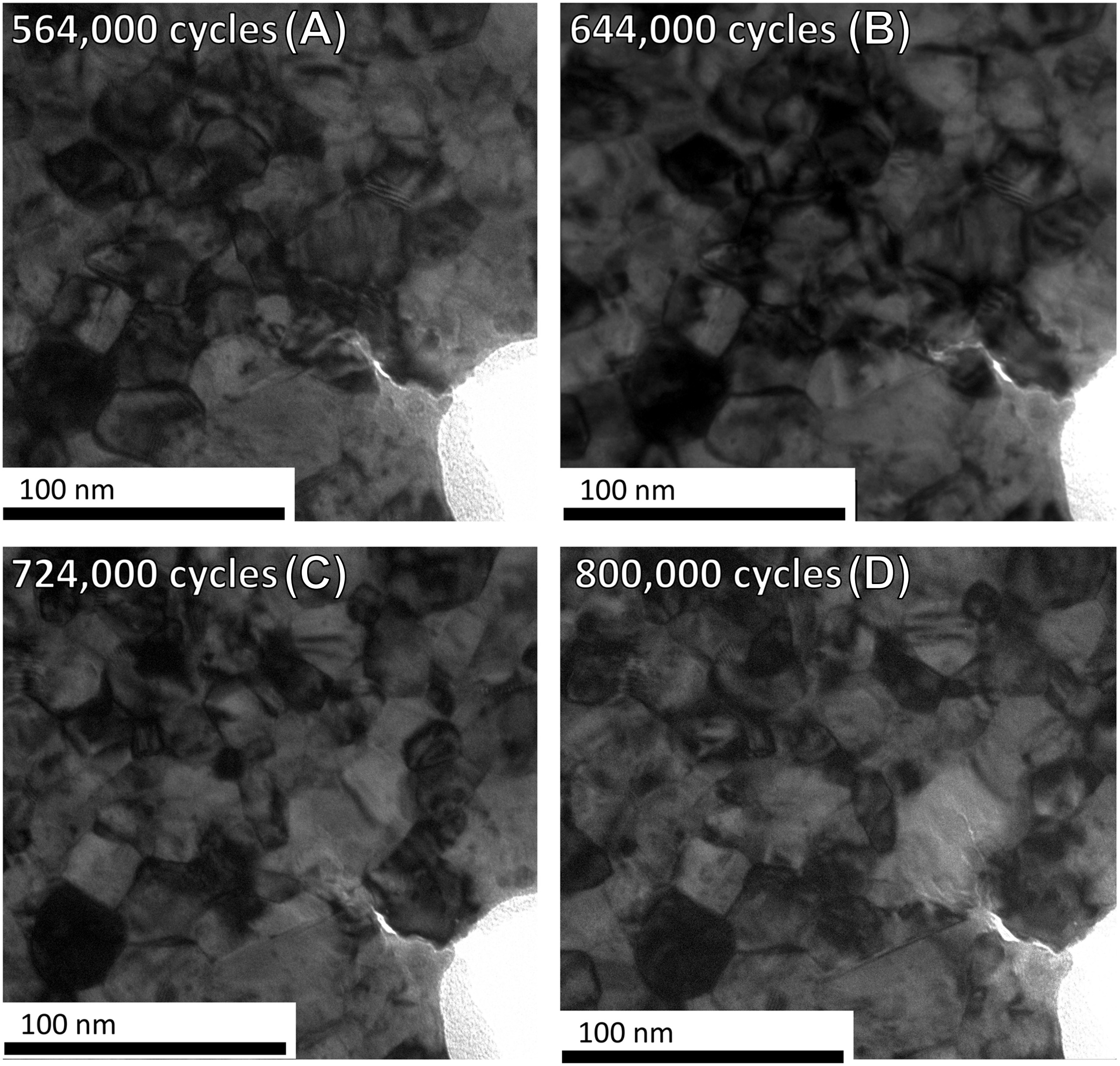
PED orientation maps pre-, intermediate, and post- in-situ mechanical test can assist in deconvoluting possible NC stress assisted grain growth

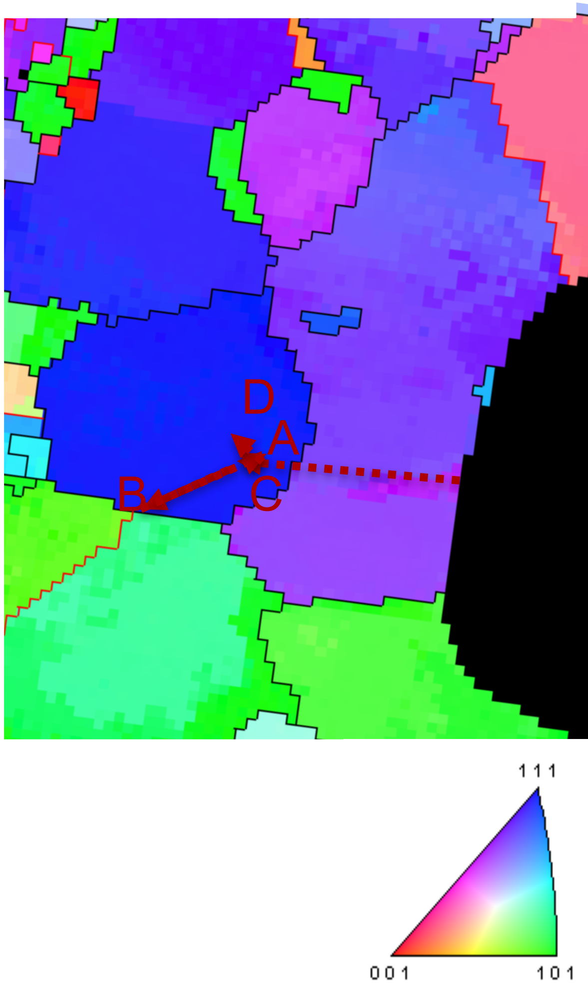
Crack Initiation at Notch



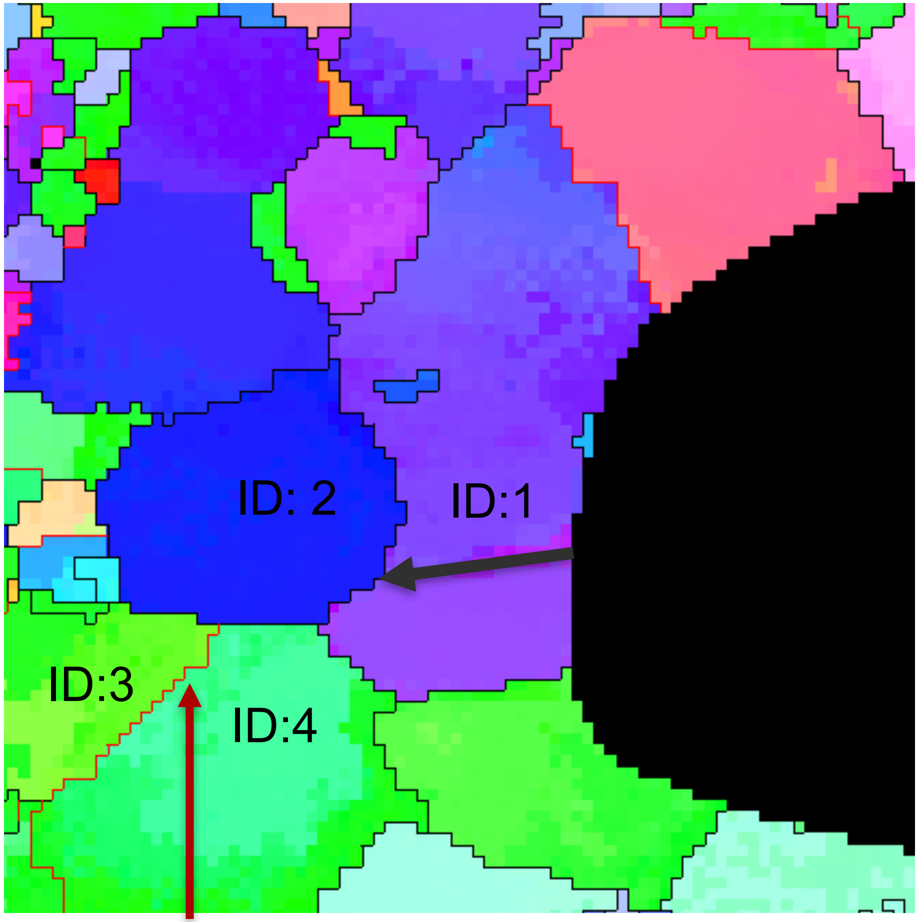
- Crack initiation and initial propagation at notch tip
- Second crack initiates at ~ 90° to first crack, both 45° to notch tip normal
- Intra-granular crack (crack #2) propagates until reaching initial grain boundary and is subsequently arrested

Crack Propagation, Closure, and Re-Direction





Cyclic Loading: GB Misorientation Changes



Σ3 of interest ("GB-1")

PED map at 124,000 total cycles

GB-1 (Twin Boundary Σ3)	Misorientation	Deviation from Ideal Σ3
Prior to Cyclic Loading	59.9° [1 1 1]	0.8 °
Crack Impinges GB at Grains 1-2	56.9 [7 7 6]	4.9 °

- Deformation and stress intensity of cyclic loaded crack tip induced modification (change in macroscopic misorientation) of the coherent Σ3
- Decrease in Σ3 coherency from nearly ideal twin misorientation to greater than 4° deviation likely associated:
  - Grain rotation and dislocation-GB impingement

Cyclic loading: transgranular crack propagation

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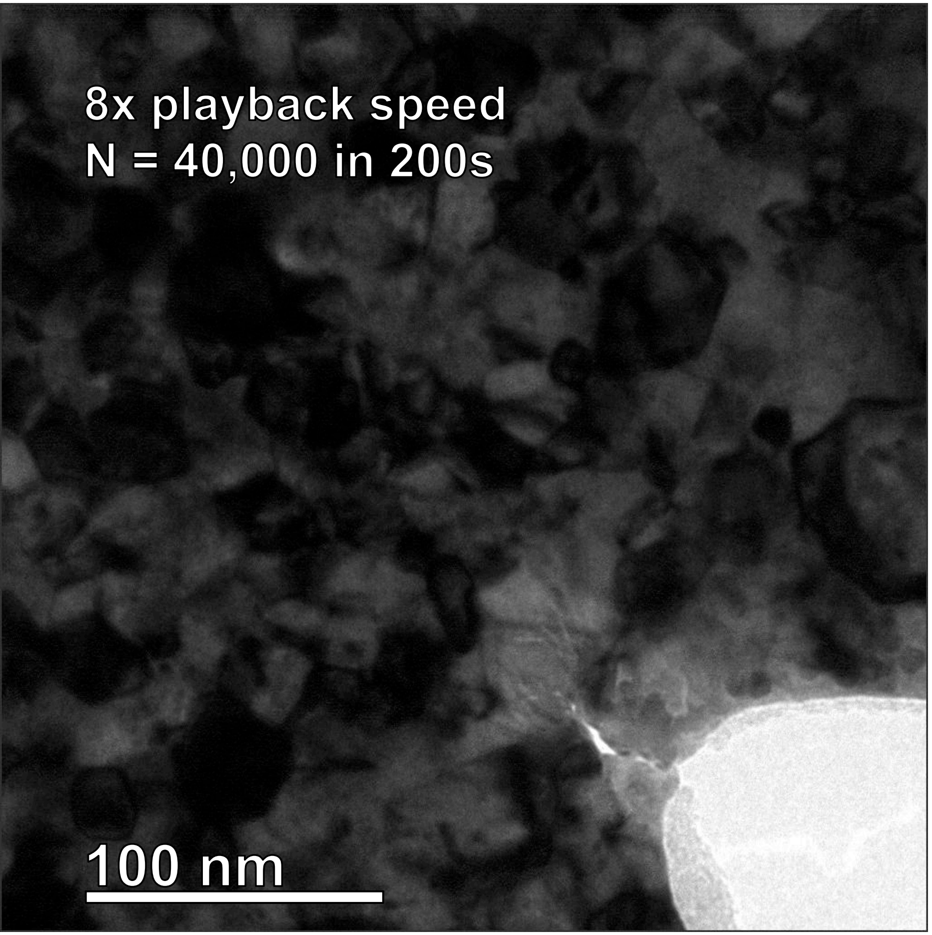
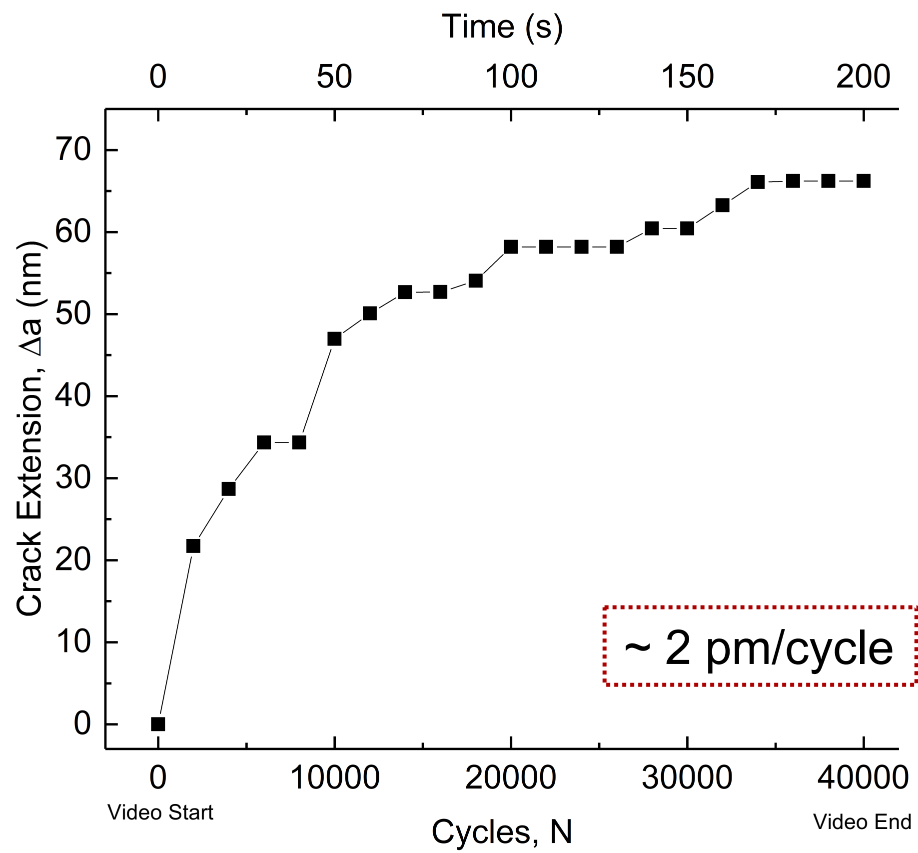
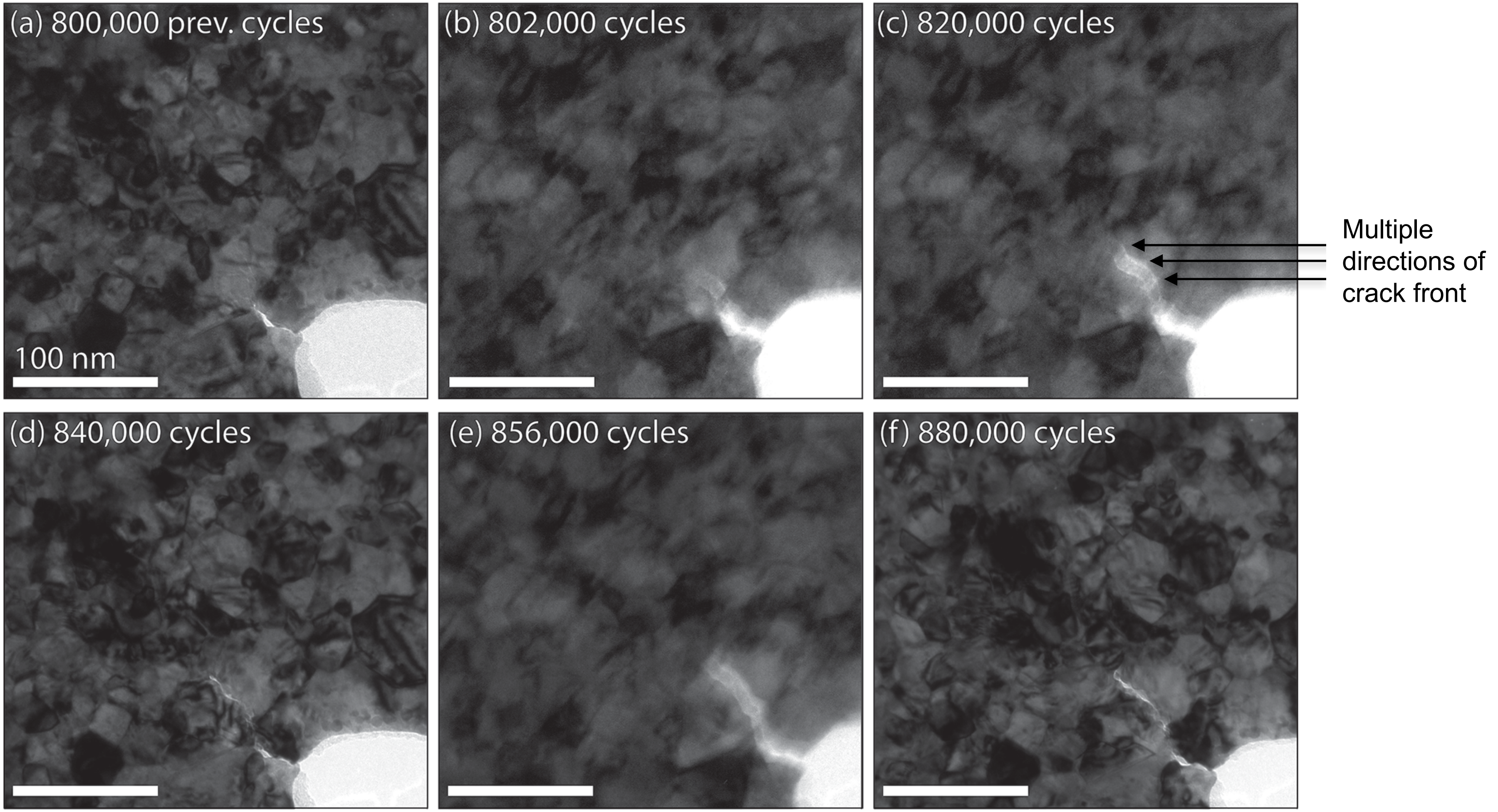




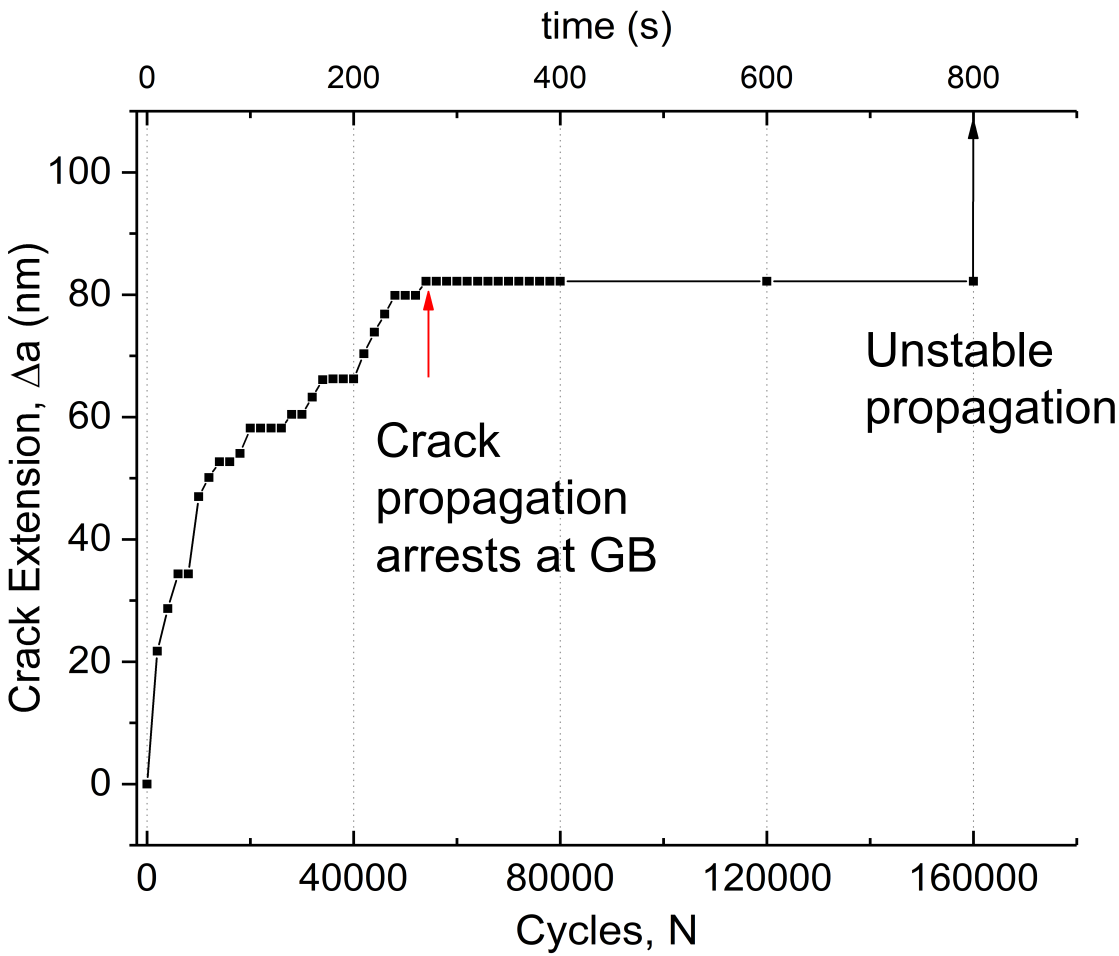
- Mean load: 135 uN; Amplitude load: 35 uN
- 200 Hz, 200s test (15 fps 1k x 1k camera)
- da/dN = 1.7×10^{12} m/cycle
- Non-linear crack extension rate
- Crack propagation path changes "direction"

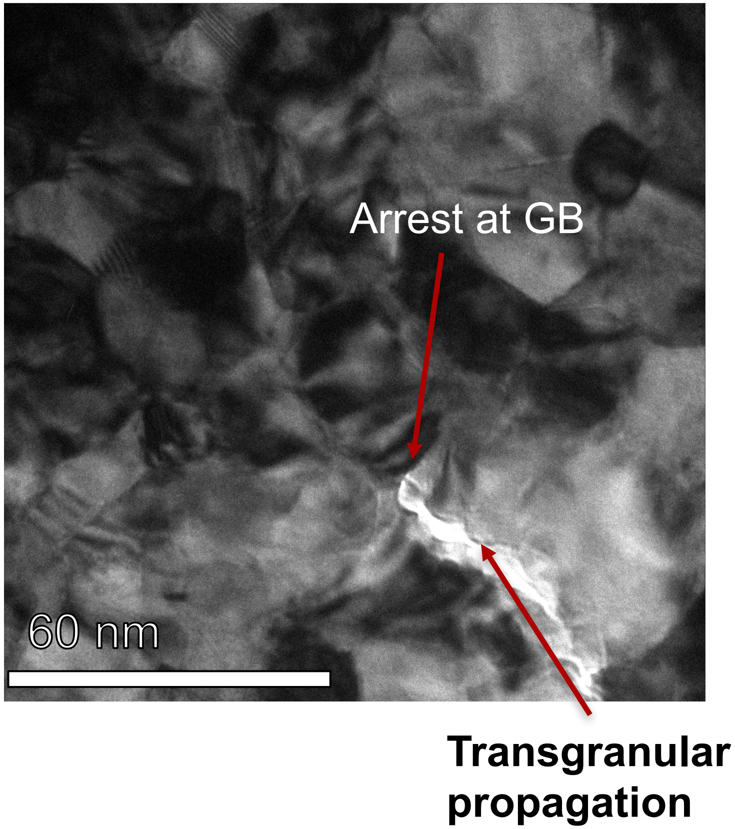
Fatigue crack propagation and arrest



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

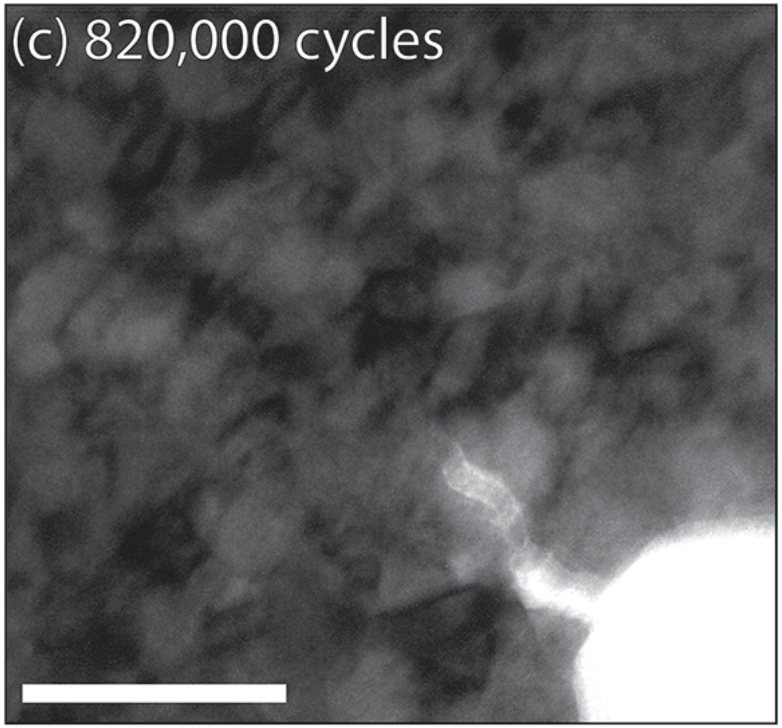
Fatigue crack propagation and arrest

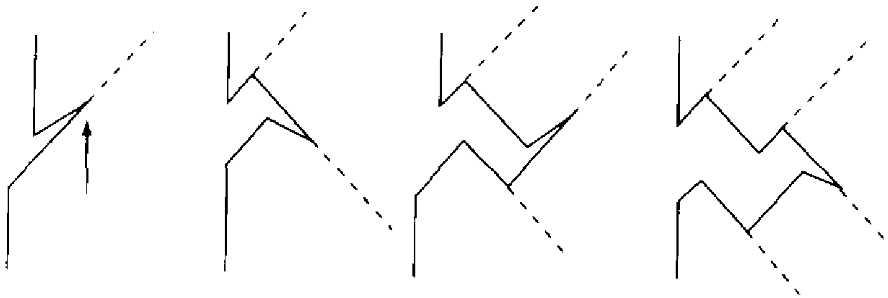




- Rapid propagation – intragranular between N = 800K to ~ 854K
- After 854K cycles, cyclic loading crack impinges grain boundary – no further propagation for addition ~106K cycles
- Transgranular crack propagation non-linear: “serpentine fashion”

Transgranular crack propagation: non-linear



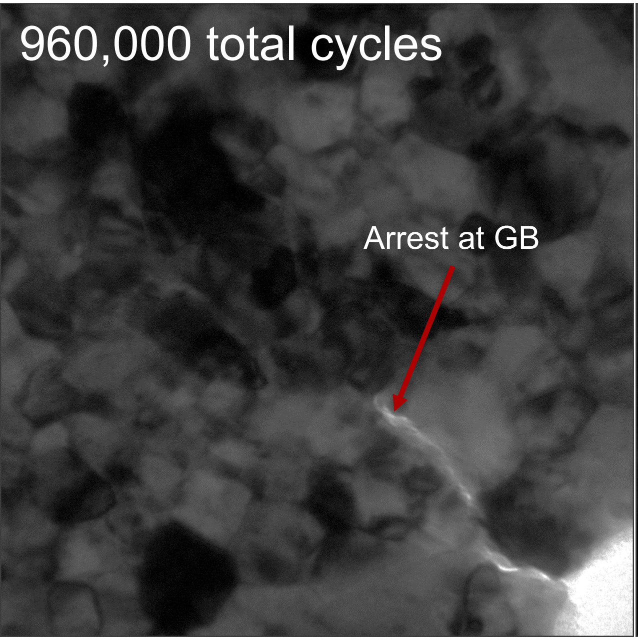


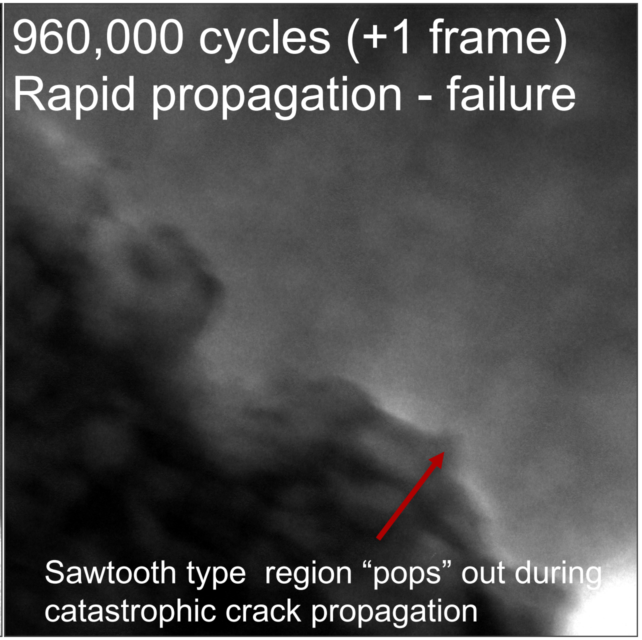
Ohr, Mat Sci & Eng72 (1985) 1-35

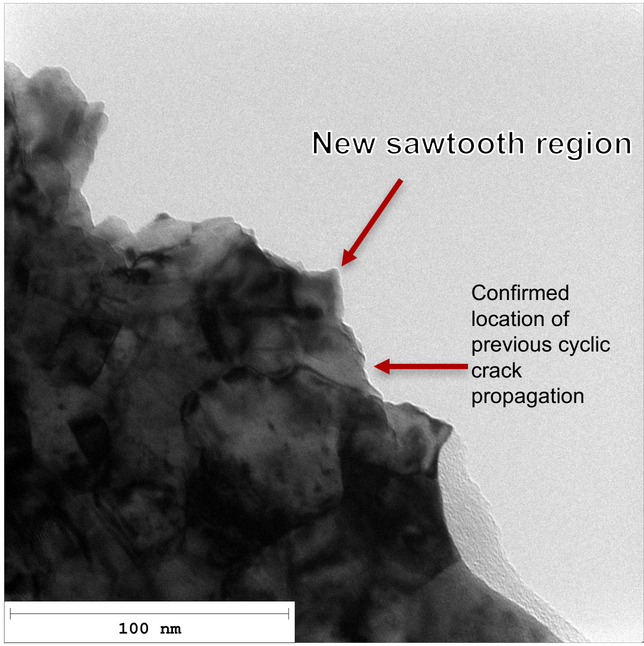
- Classic in-situ tensile straining work by Ohr and colleagues observe zig-zag crack propagation → associated with emission of dislocations on alternating slip planes

- 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



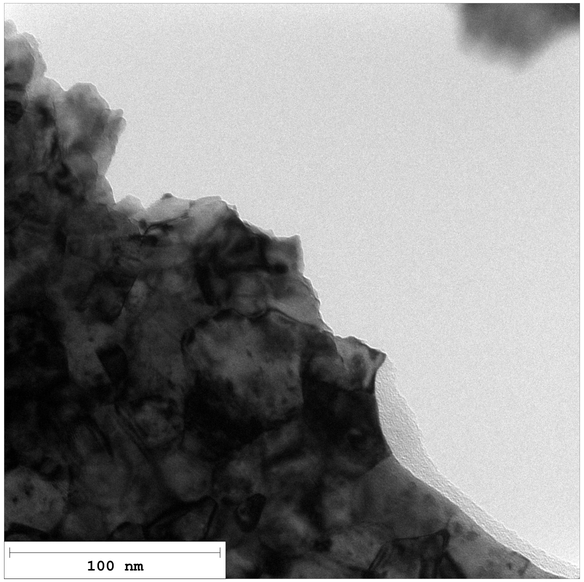




- Unstable propagation of crack occurred just under 1M total cycles
- Sample failed on initial ramp (~100 uN) to monotonic mean load (135 uN) at 960,000 cycles
- Facture surface consistent with location of previous fatigue crack propagation
- Indications of “sawtooth” intra-grain plasticity during this rapid failure mechanism

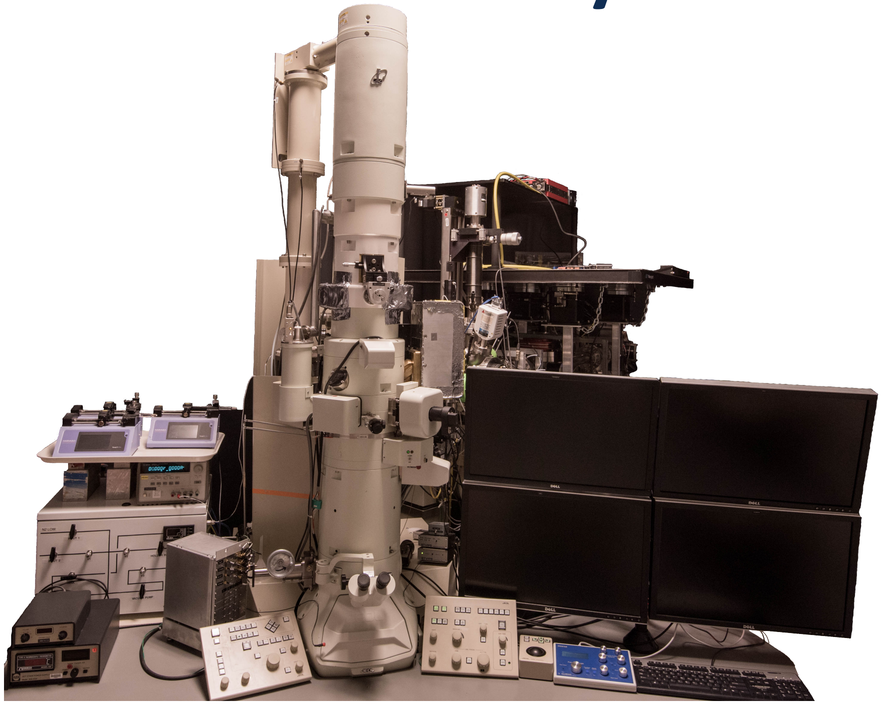
Discussion and Conclusions:

- 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 Σ3 coherency in front of crack tip**
- **Failure associated with classical in-situ TEM mechanical “saw-tooth” plasticity**

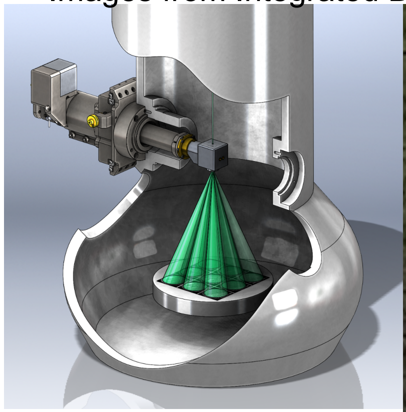


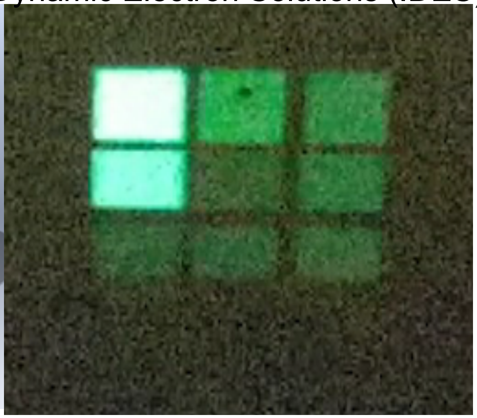
Saw-tooth classically explained by local plastic flow – (e.g. Wilsdorf and Kumar et al. Acta 2003)

Future In-Situ Mechanical Testing Directions at I^3TEM Facility



Images from Integrated Dynamic Electron Solutions (IDES):



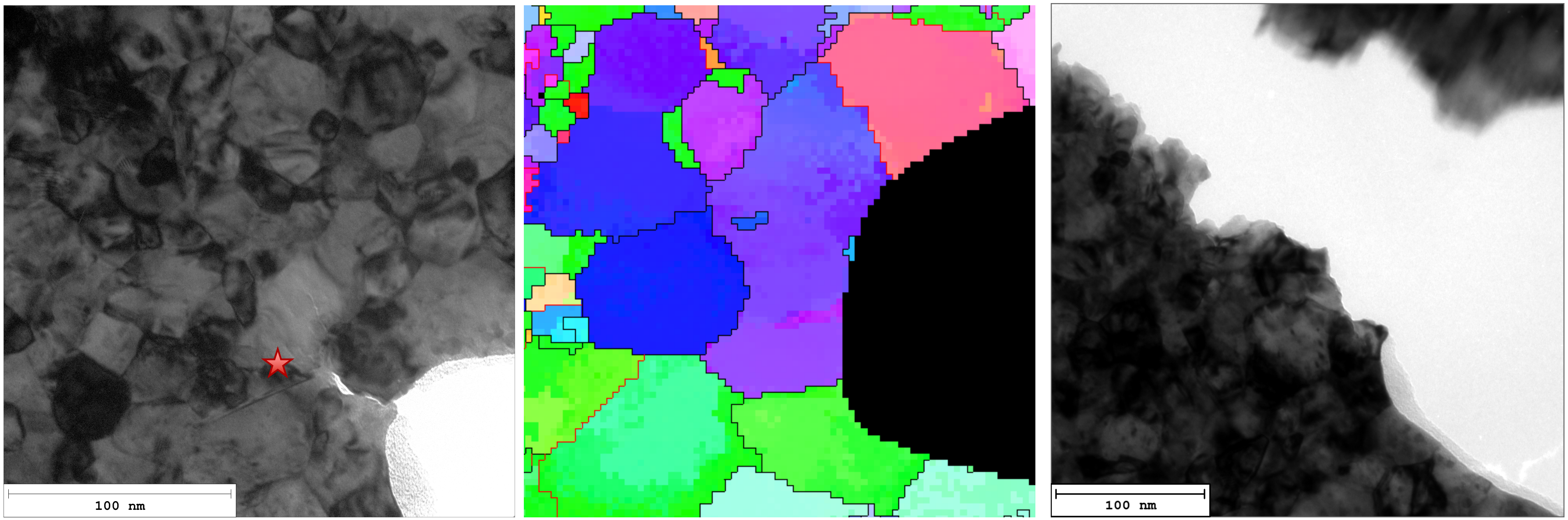


- High cycle fatigue – more detailed quantification of grain growth
  - Other model alloys, multi-layers, alloying effects (e.g. Pt-10Au vs. Pt)
- Creep, radiation-induced creep
- TEM notched three point bend
- Under development capabilities:
  - DTEM and/or movie mode with crack propagation for improved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