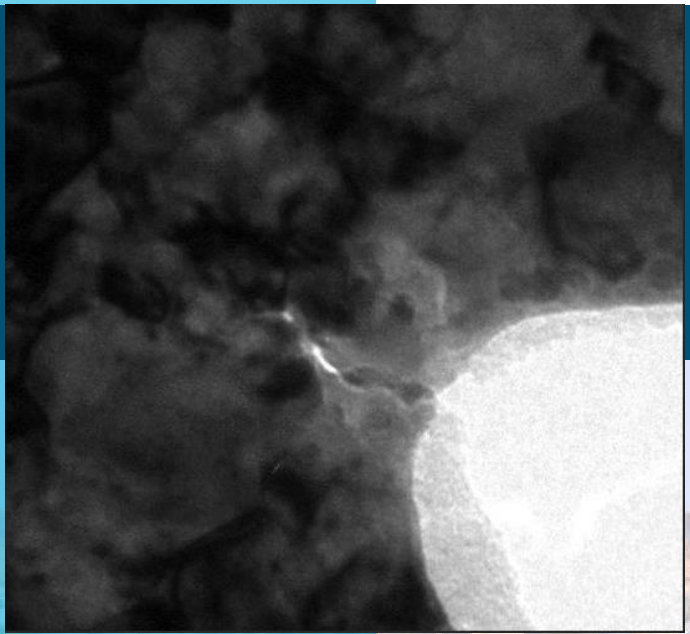
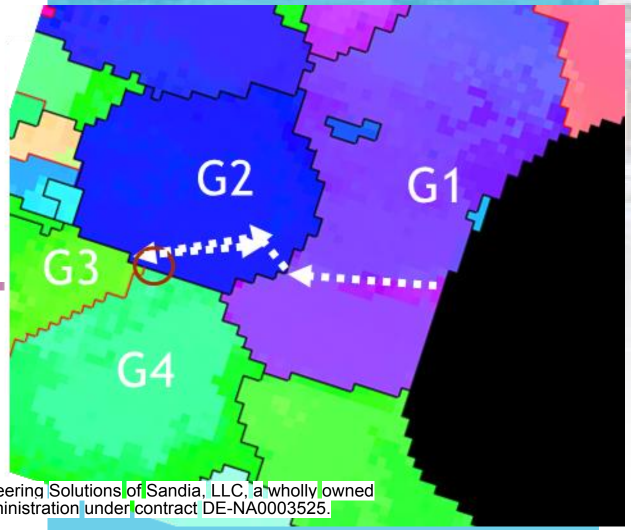
SAND2022-2483C

Exploring Nanoscale Fatigue through Coupled In-situ Microscopy and Modeling



Khalid Hattar[1], Christopher Barr[1], Ta Duong[2], Zac Milne[1], Daniel Bufford[1], Abhilash Molkeri[2], Nathan Heckman[1], David Adams[1], Ankit Srivastava[2], Michael Demkowicz[2], and Brad Boyce[1]

[1]Sandia National Laboratories
[2]Texas A&M University



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Utilization In-situ TEM to Explore Fatigue in Nanocrystalline Metals

Typically improved fatigue endurance limit but undesirable higher crack growth rates compared to coarse grain counterparts

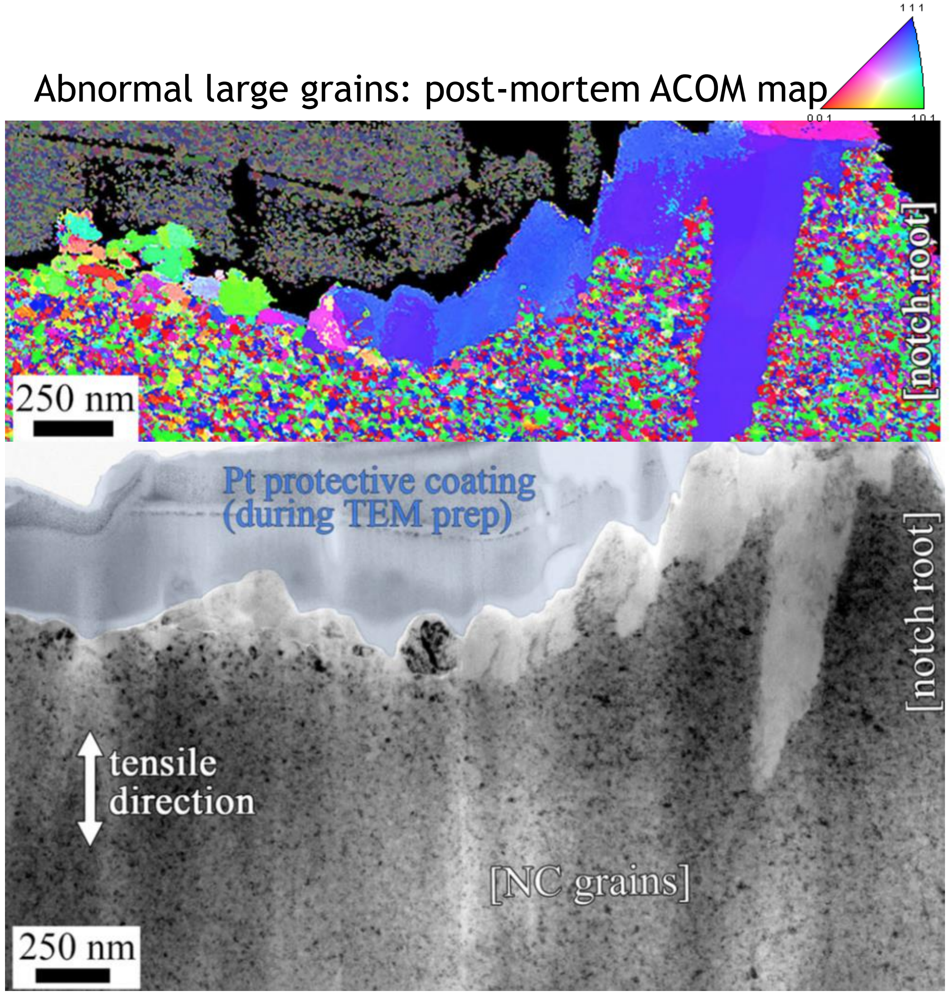
Fatigue behavior in nanocrystalline metals associated with:

- Localized grain growth and GB migration
- Grain rotation and sub-grain formation in abnormally large grains

What are the underlying mechanisms associated with these GB dependent phenomena?

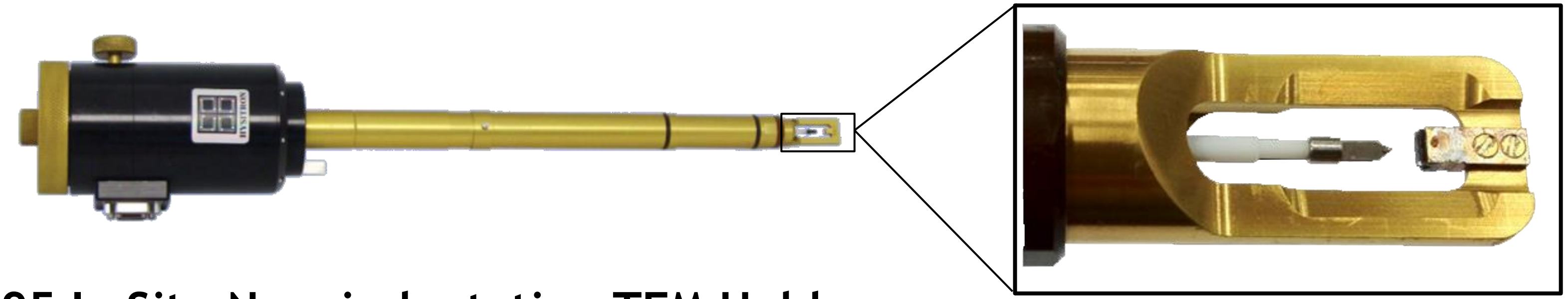
- **In situ TEM cyclic loading techniques provide the spatial resolution needed to investigate these questions**

Abnormal large grains: post-mortem ACOM map



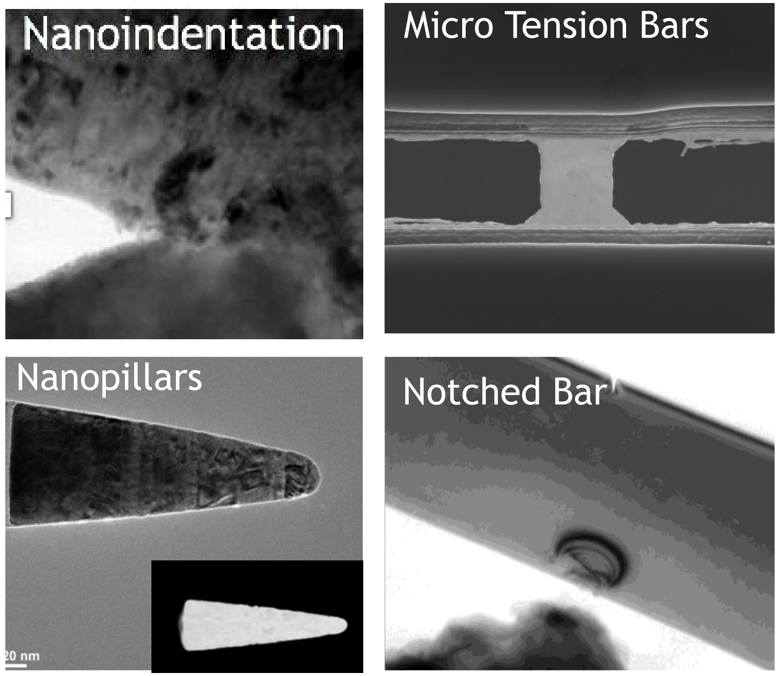
T.A. Furnish et al., J. Mat Sci 52:46-59 (2017)

In-situ Quantitative Mechanical Testing



Bruker Hysitron PI-95 In Situ Nanoindentation TEM Holder

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



- A variety of sample geometries provides
- Test conditions of interest to our BES team:
  1) Tension
  2) Compression
  3) Fatigue*
  4) Creep

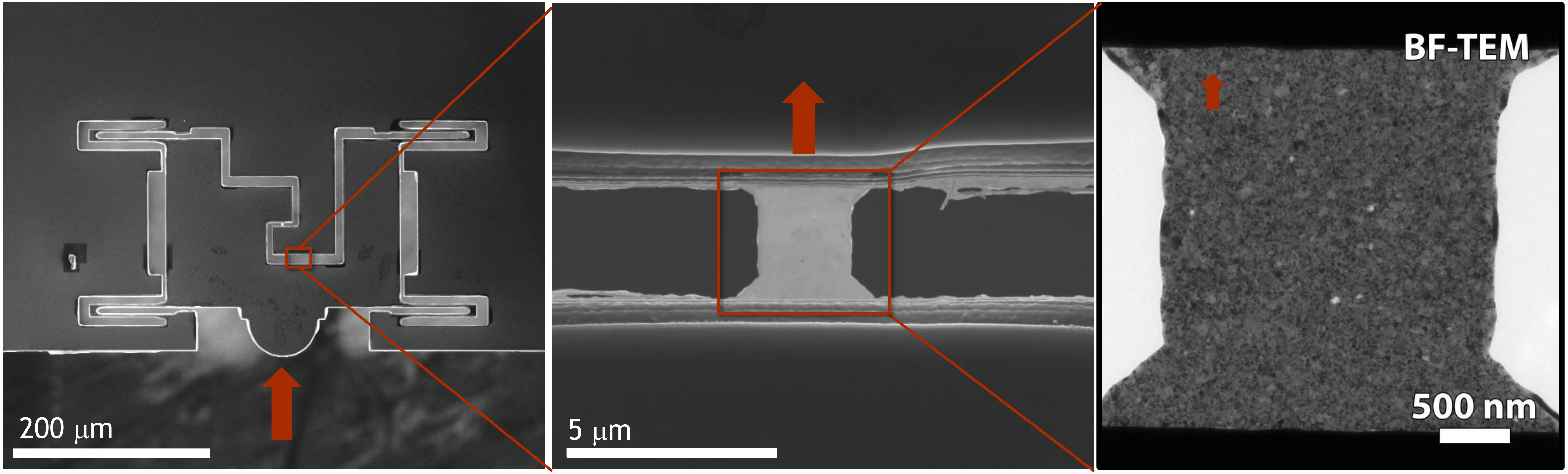
Can be combined with other external stimuli at I³TEM (IR laser, heavy and light ion irradiation, and heating)

Specimen Fabrication

- Bruker 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

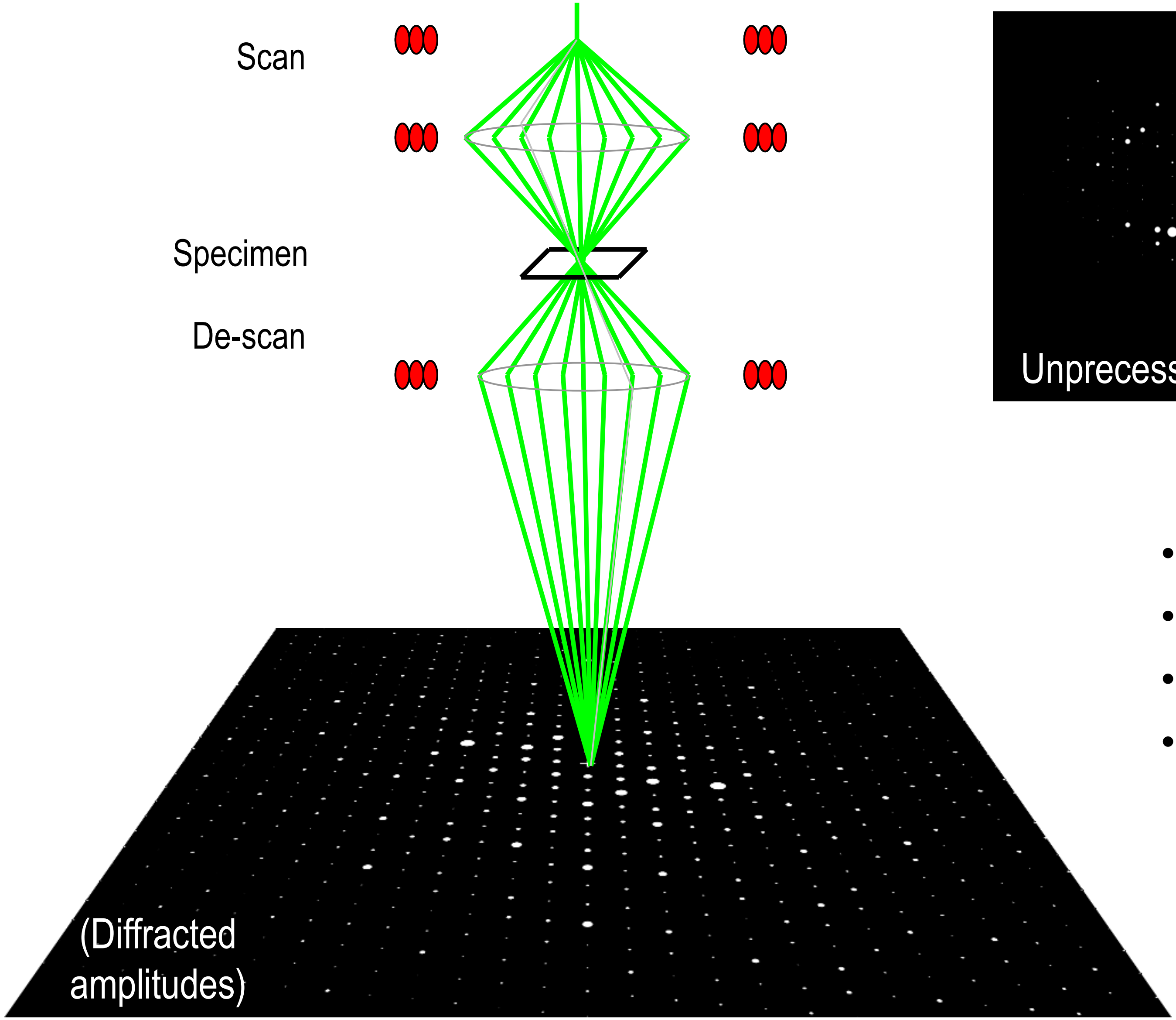
Nearly pure tension, uniform cross sectional area, stable load frame

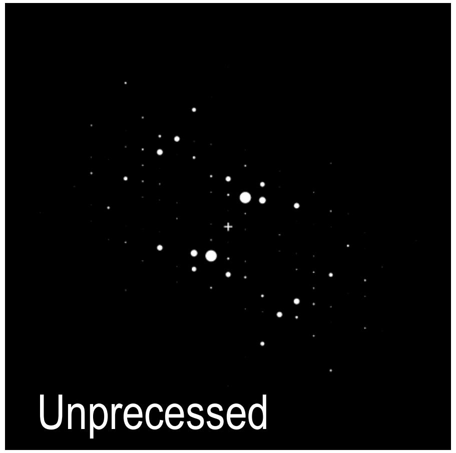
$$F_{applied} = F_{measured} - F_{spring}$$

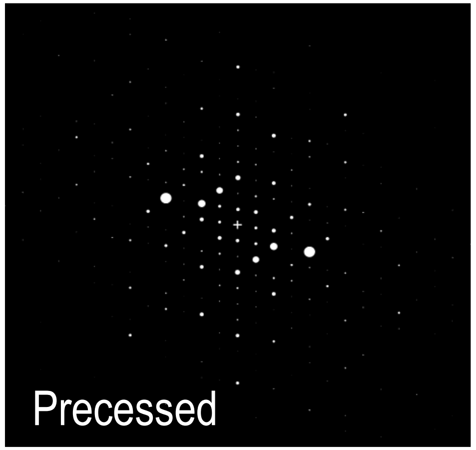


Bufford et al. Nano Letters (2016)

Precession Electron Diffraction Microscopy –Automated Crystal Orientation Mapping





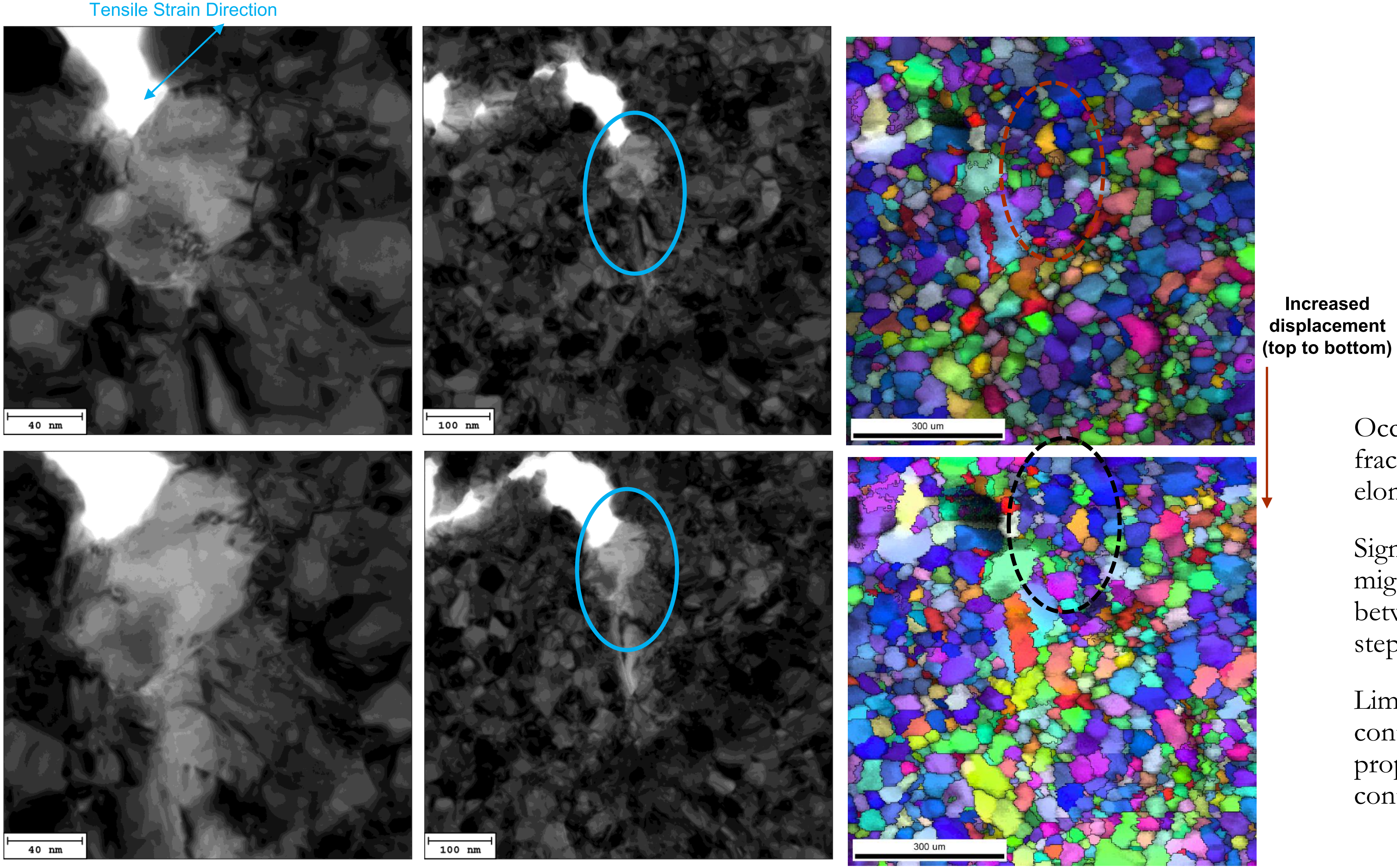


Advantages:

- < 10 nm spatial resolution
- Near kinematical electron diffraction
- Symmetry ambiguities are resolved
- Fast and automated acquisition
    - ≈200 grains in 15 min.
    - Angular resolution ≈1°

AppFive NanoMEGAS Advanced Tools for electron diffraction

In situ Qualitative Mechanical Testing and ACOM

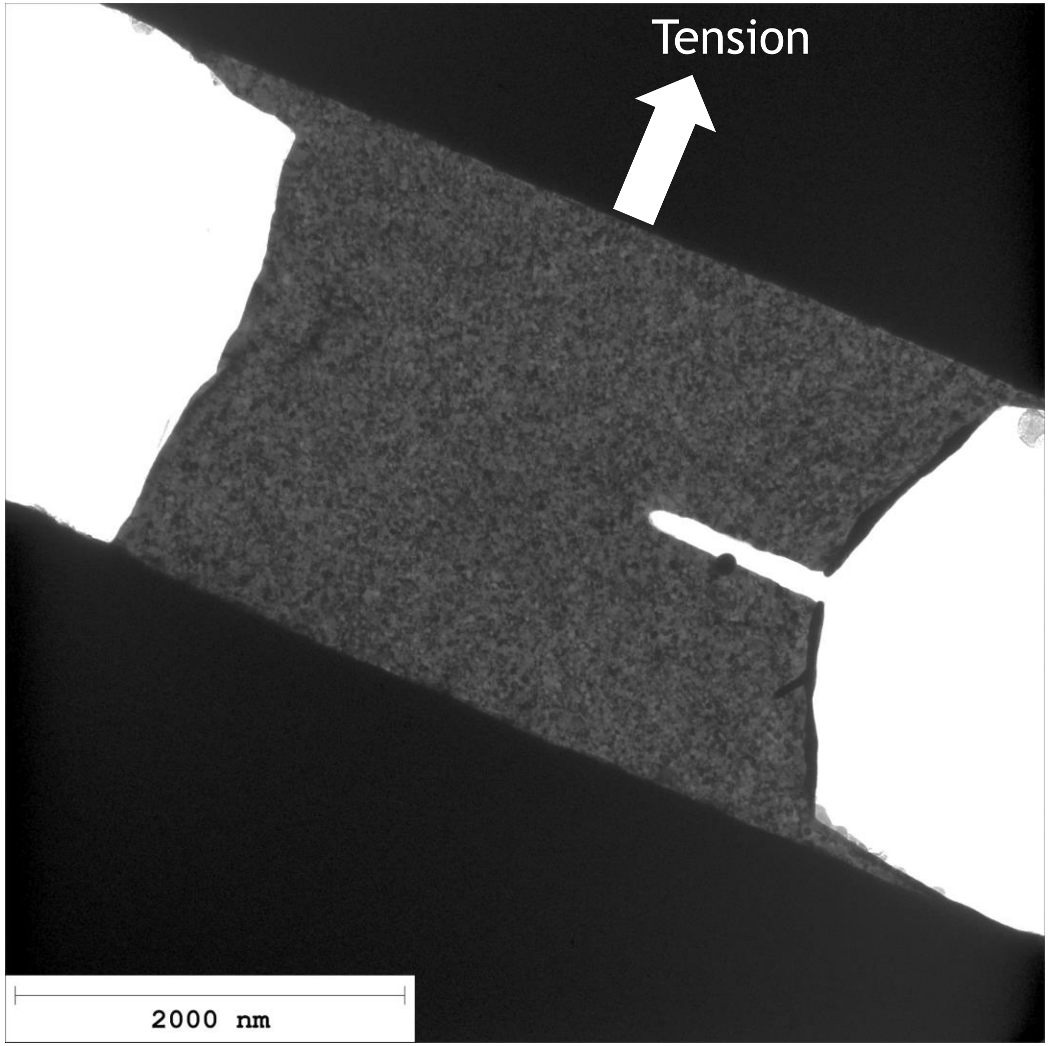


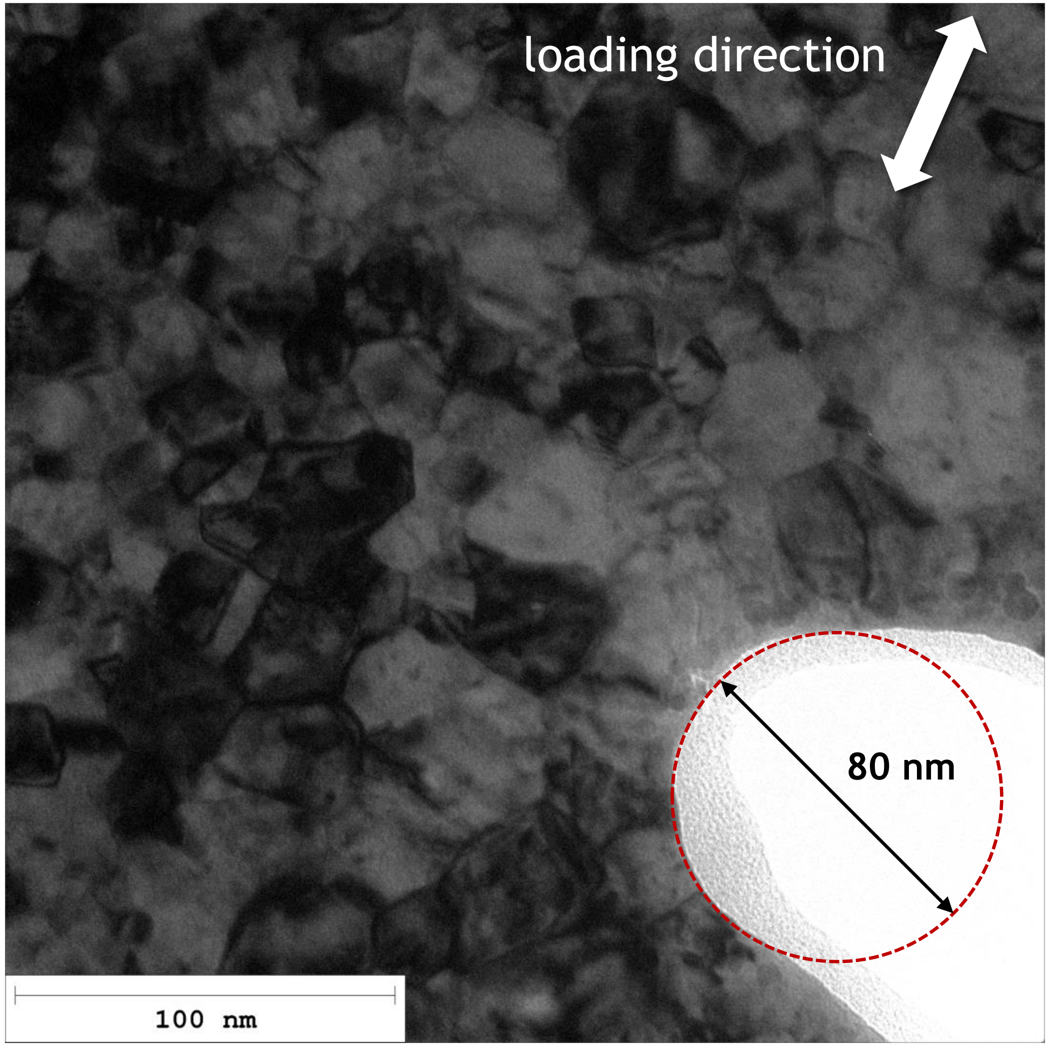
Occasional ductile fracture and grain elongation

Significant dislocation migration/activity between deformation steps

Limited displacement control and crack propagation → limits control study

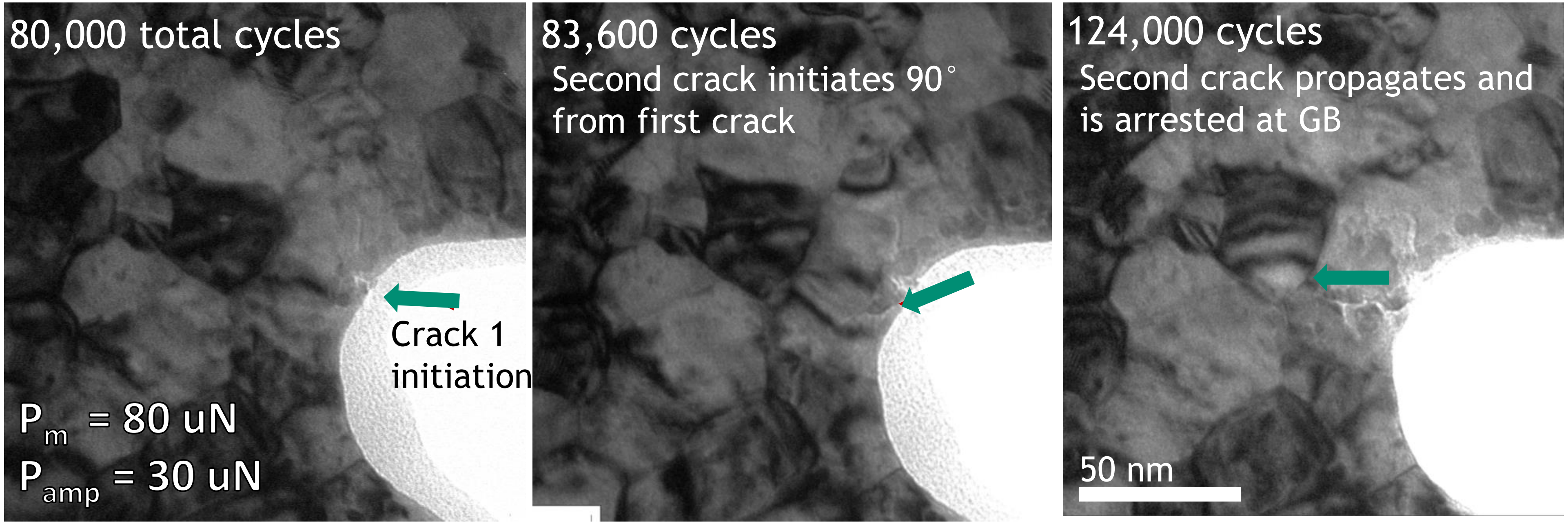
Notched Pt: In-situ Cyclic Loading





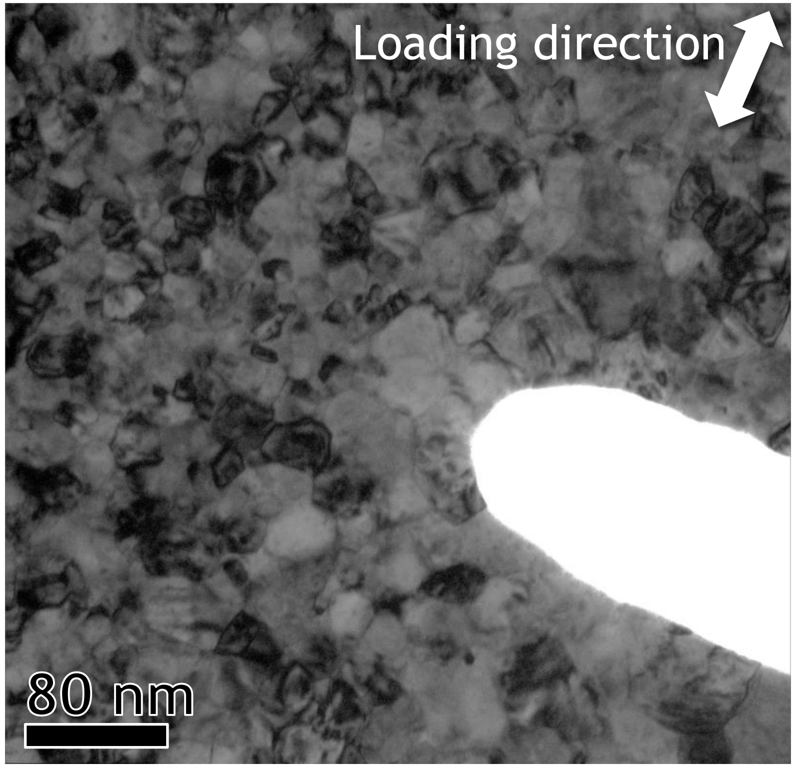
- Notch length = 950 nm, Gauge width = 3.3 um
- Notch created by FIB "line"

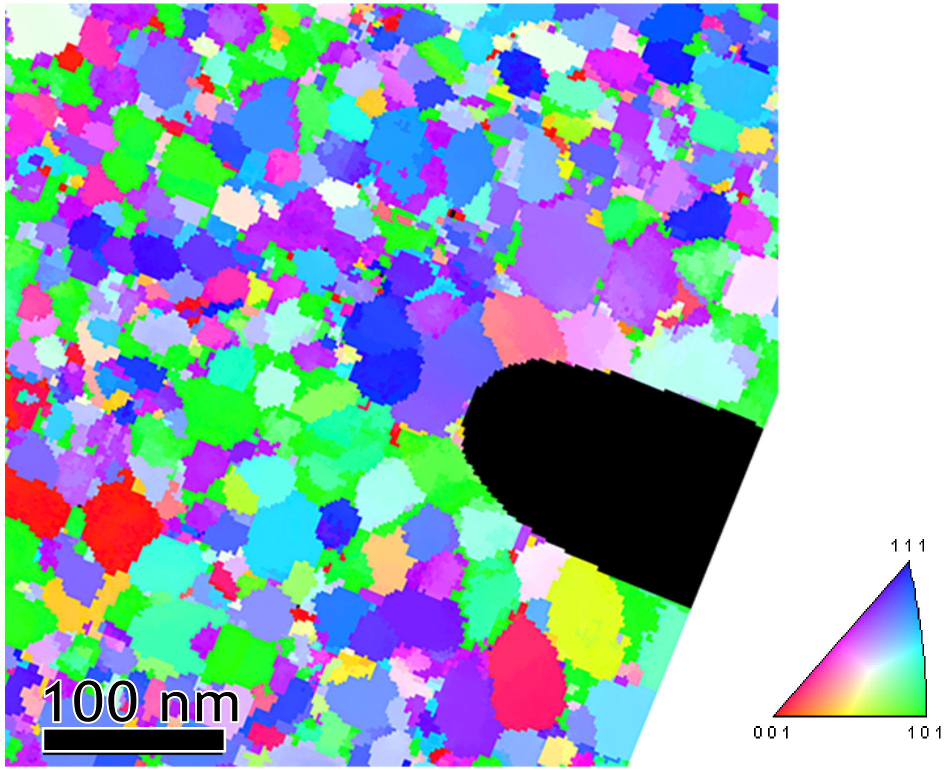
Crack Initiation at Notch



- Crack initiation
- Second crack initiates at ~ 90° to first crack, both 45° to notch tip normal
- Transgranular crack propagates until reaching initial grain boundary (25.7° [13 2 8] misorientation) and is subsequently arrested for over 200,000 additional cycles

ACOM-PED coupled In-situ TEM Fatigue

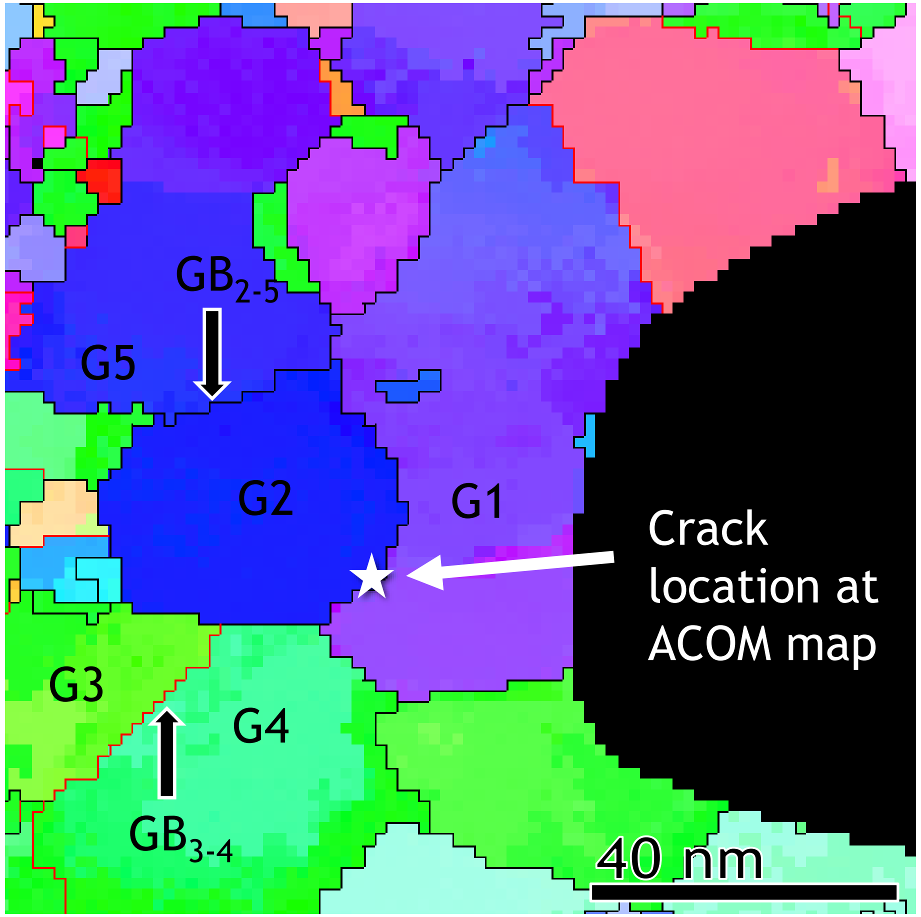




- Ability to couple grain orientation and grain boundary misorientation with crack propagation
  - Feasible to track relative grain rotation or variation in GB misorientation under loading

- **PED orientation maps pre-, intermediate-, and post- test assist in understanding NC stress assisted grain growth and defect-GB interactions**

Cyclic Loading: Local GB Misorientation Changes



* Intermediate PED map at 164,000 total cycles – crack at $GB_{1\text{-}2}$

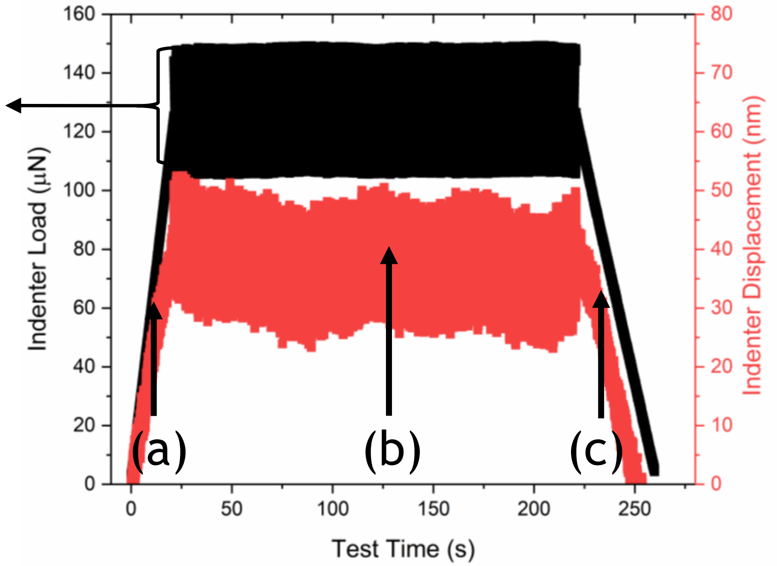
- Deformation from cyclic loading and crack transgranular crack propagation → clear change in relative GB misorientation
- **Likely active mechanisms:**
  - Grain rotation and associated dislocation-GB impingement associated with dislocation activity ahead of fatigue notch and crack path

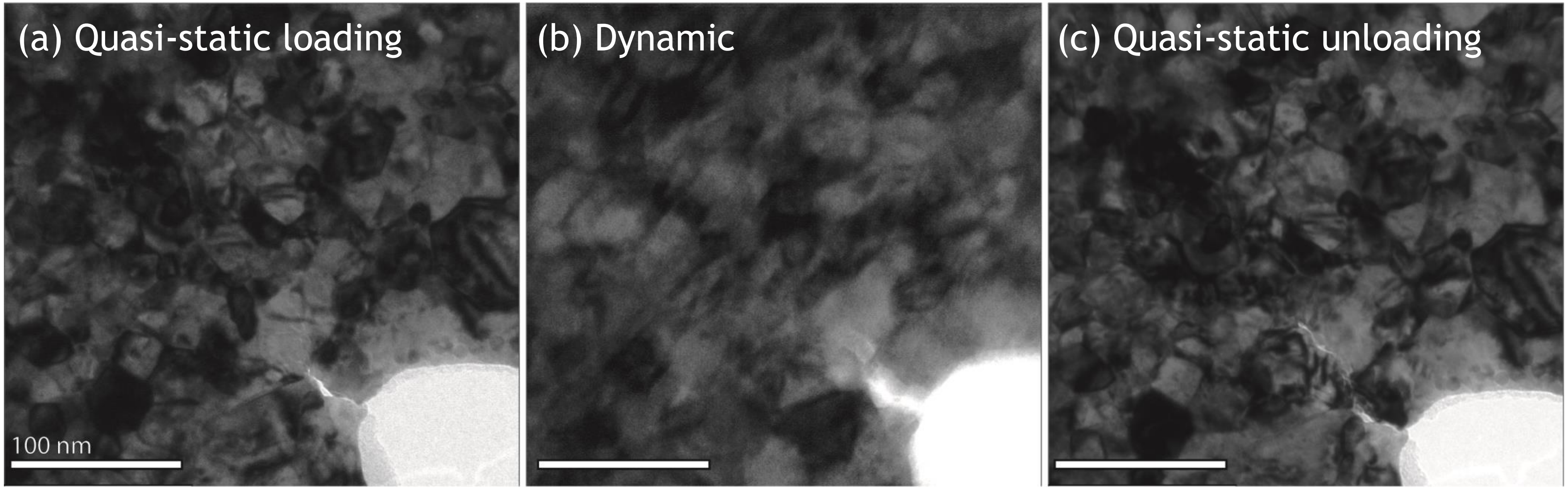
IDs:	Pre- Test ACOM	Intermediate ACOM
$GB_{3\text{-}4}$	59.3° [1 1 1]; Σ3 Dev. 0.9°	56.9° [7 7 6]; Σ3 Dev. 4.9°
$GB_{2\text{-}5}$	34.9° [12 10 11];Σ7 Dev. 4.1°	38.0° [12 11 13]; Σ7 Dev. 1.9°

Cyclic Loading in TEM Protocol

Mean load (P_{mean}) = 135 uN
Amplitude load (P_{amp}) = 35 uN

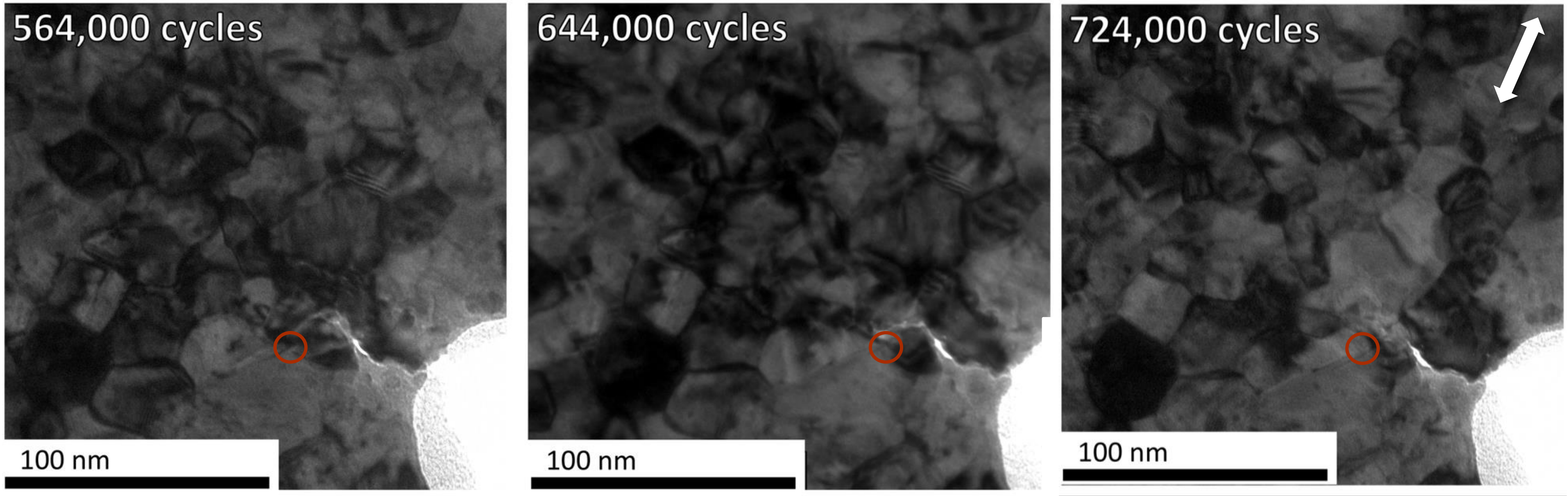
- **200 Hz Frequency**
- **40,000 cycles in 200s**





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

Crack Propagation, Deflection, Closure, and Healing

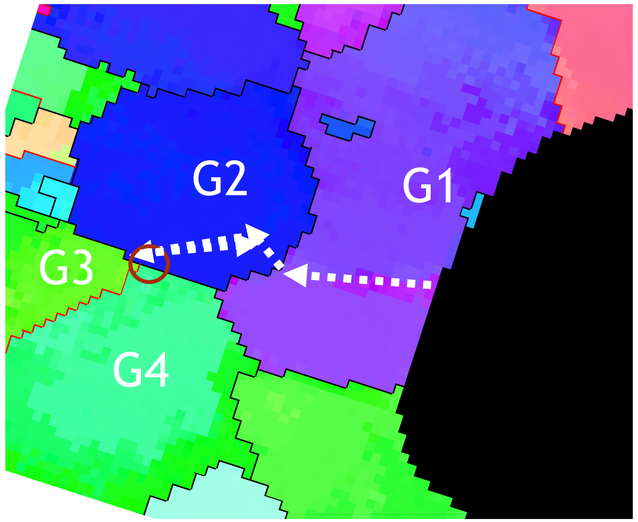


loading direction

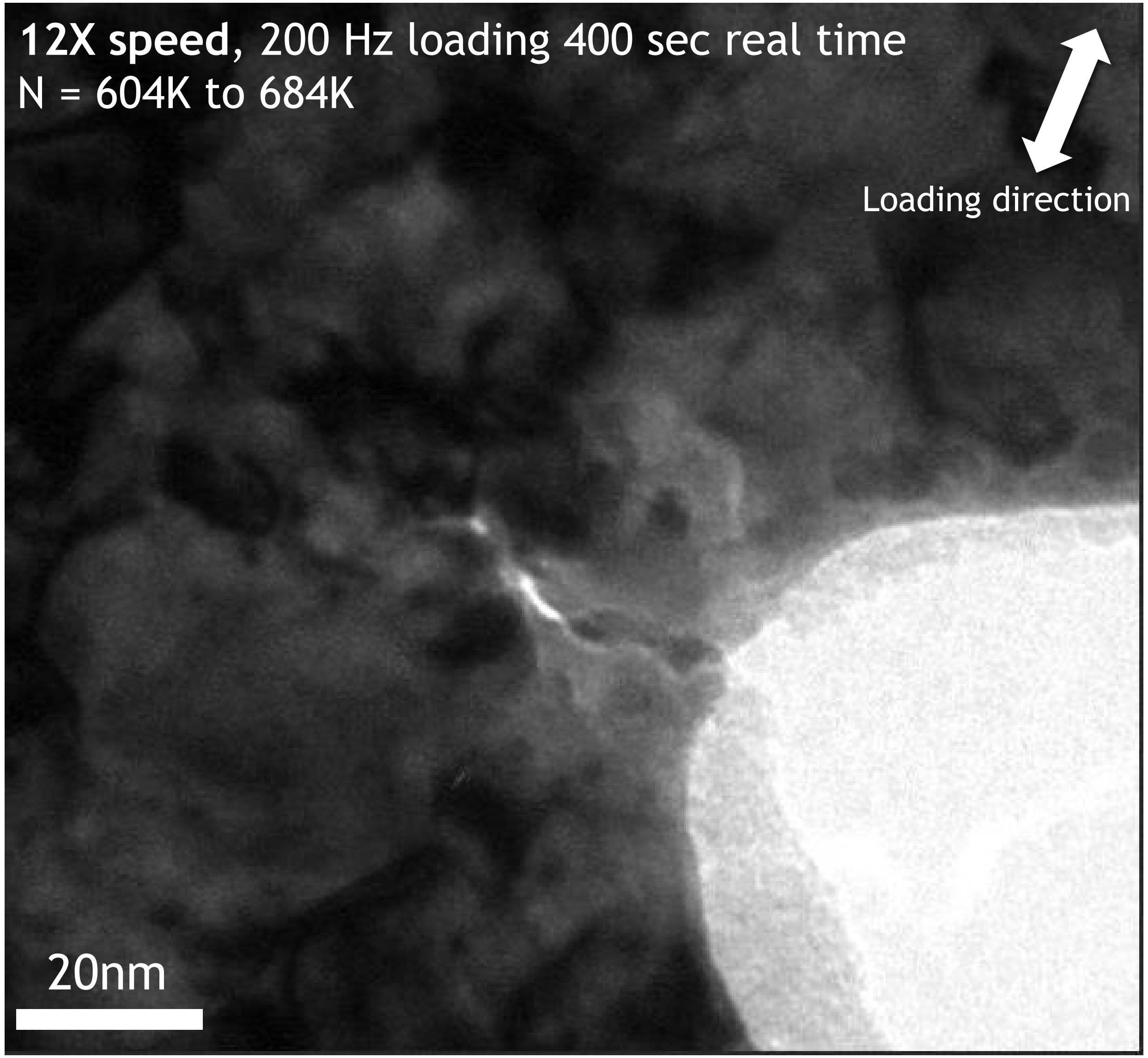
Crack follows two highest Schmid factor slip systems in G2

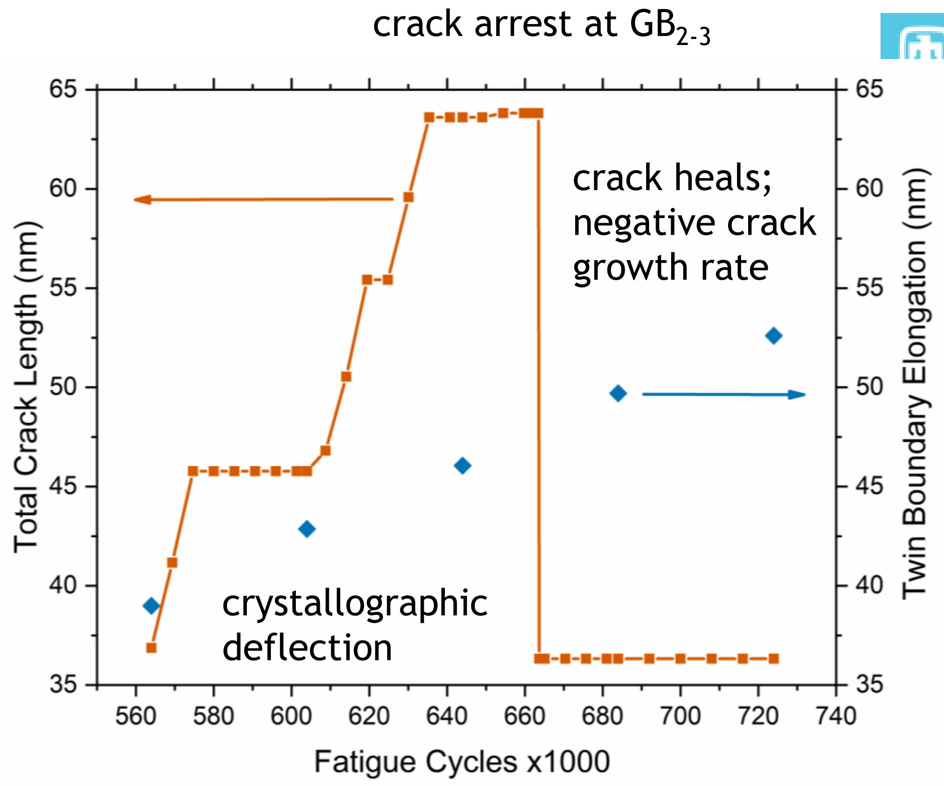
Near Mode I propagation:
(-1 1 -1)<1 1 0>, m = 0.420

Deflected away from Mode I on:
(-1 -1 1)<1 -1 0), m = 0.408



Crack Healing: In-situ TEM Fatigue





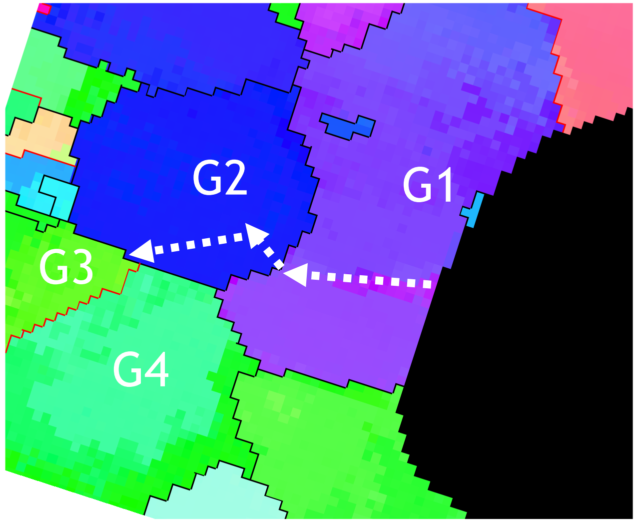
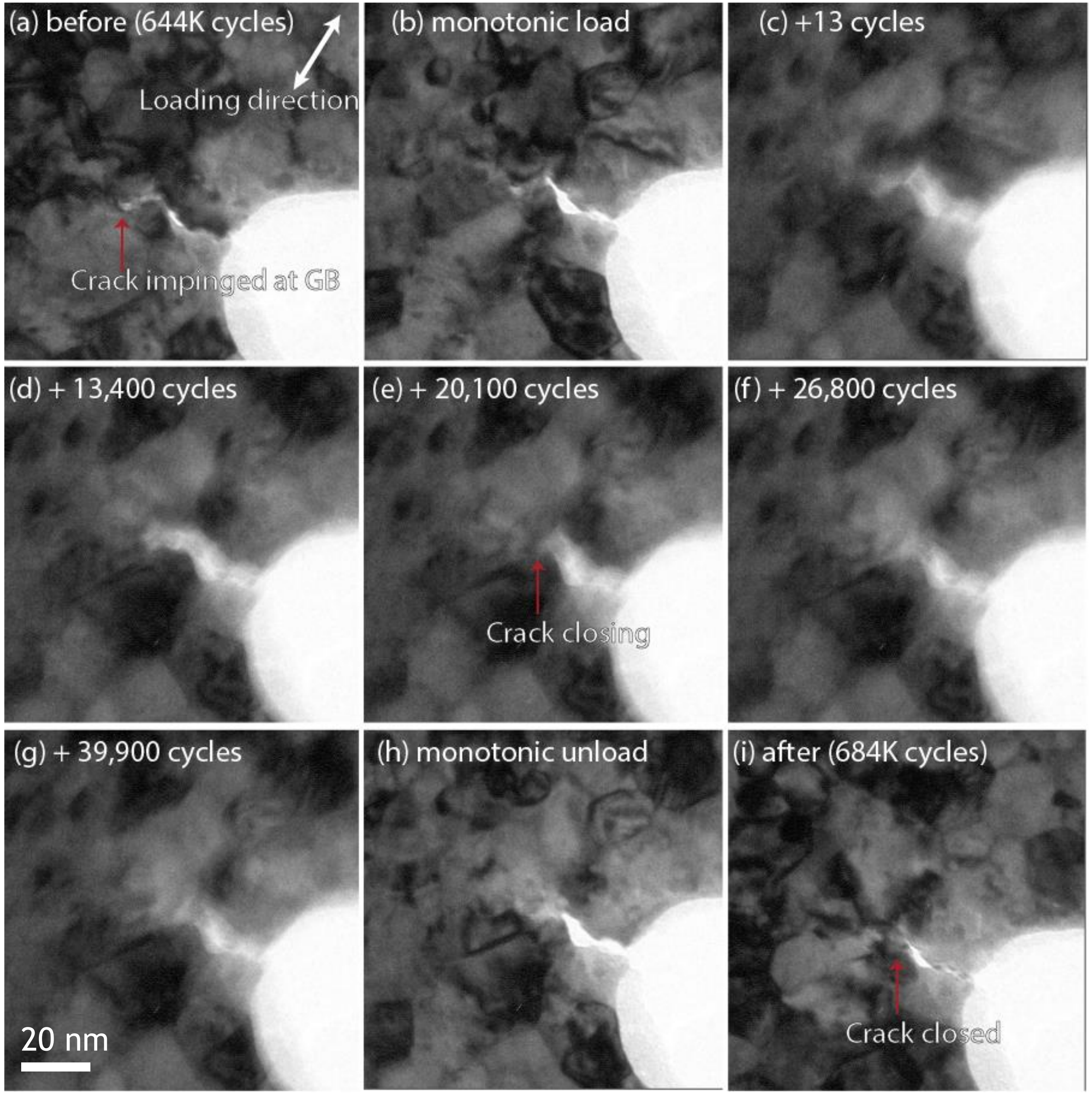


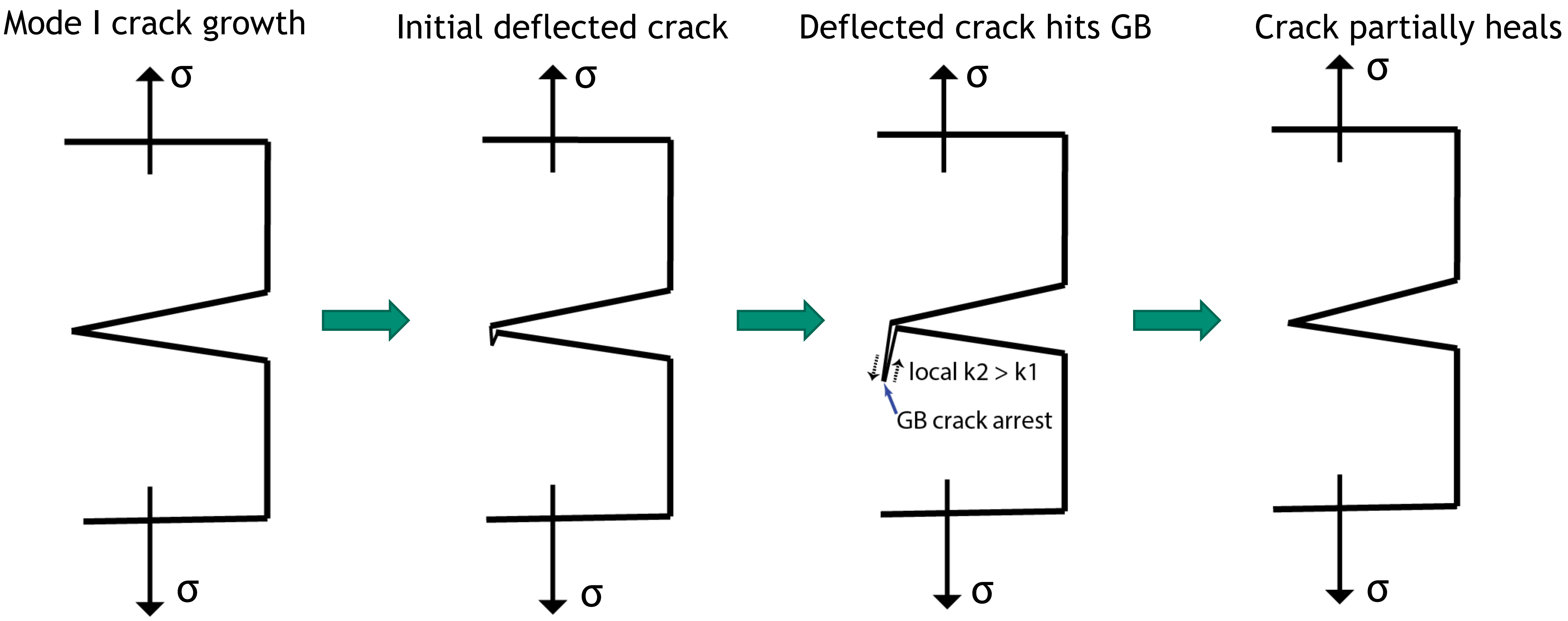
Image Sequence: Crack Healing



- Crack tip observes mixed mode loading: high and local shear during test
- Roughness-induced crack closure observed on loading and loading (side-to-side contact)
- Surface asperities on crack flank implies atomic fresh surfaces created and destroyed during cyclic loading
- Contrast changes after healing event
- Unloading – no indications of previous deflected crack
- What is the mechanism?

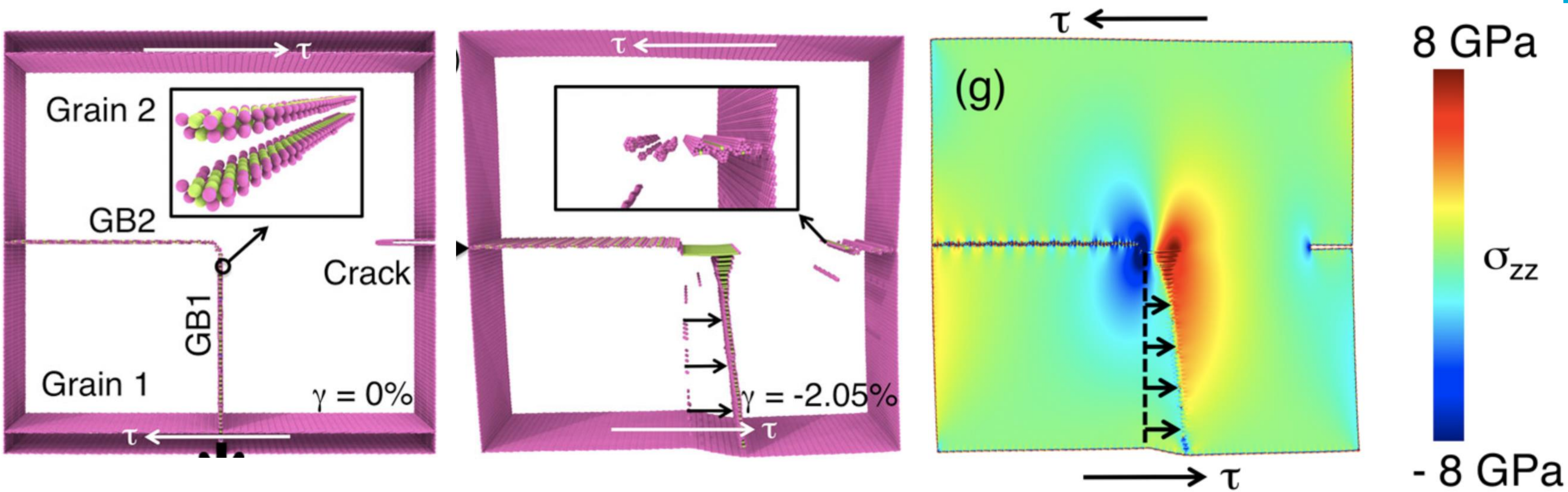
Cold Welding!
✓ Orientation the same on both sides of crack
✓ Shear and compressive component
✓ Vacuum, noble metal
✓ Atomically fresh surfaces

Simple Crack Propagation Schematic



<u>Key aspects of proposed crack healing</u>:
(1) small deflected crack tip opening
(2) roughness induced closure → crack flank contact
(3) Occurs in single grain – crack flanks share similar crystallography

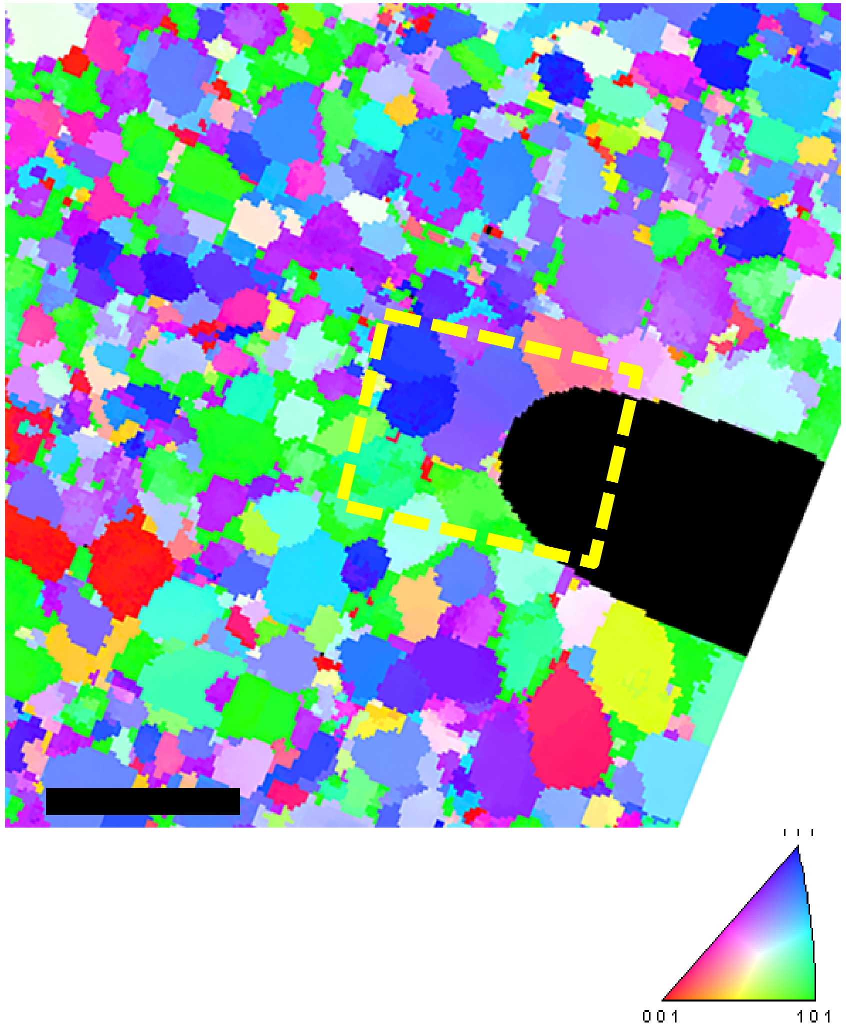
Boundary Migration Stresses Can Close Cracks

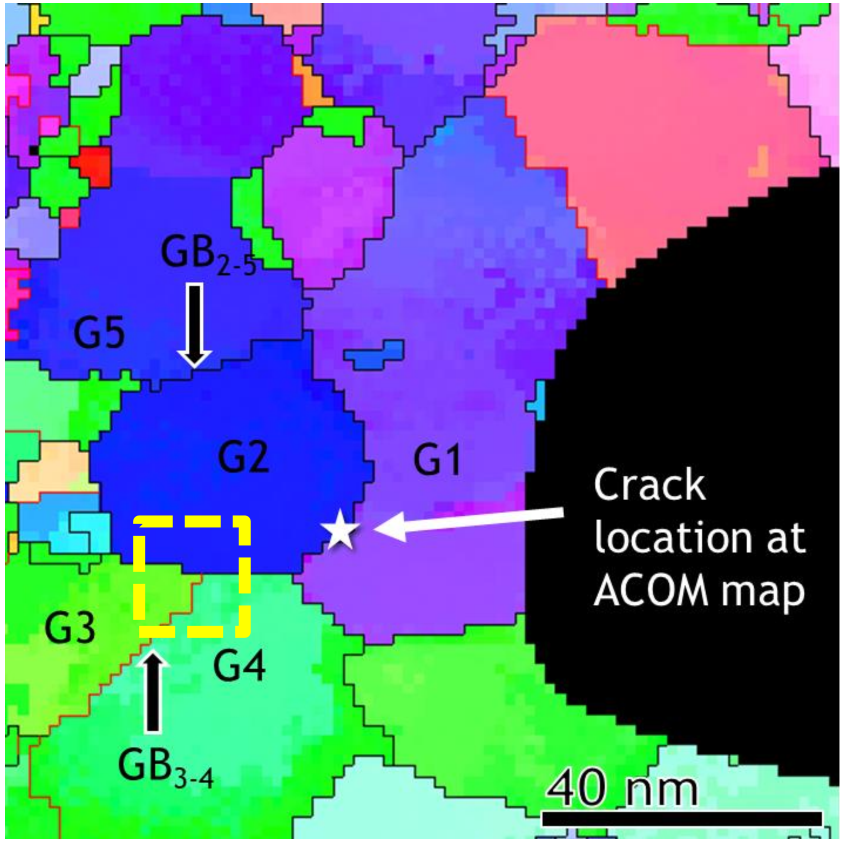


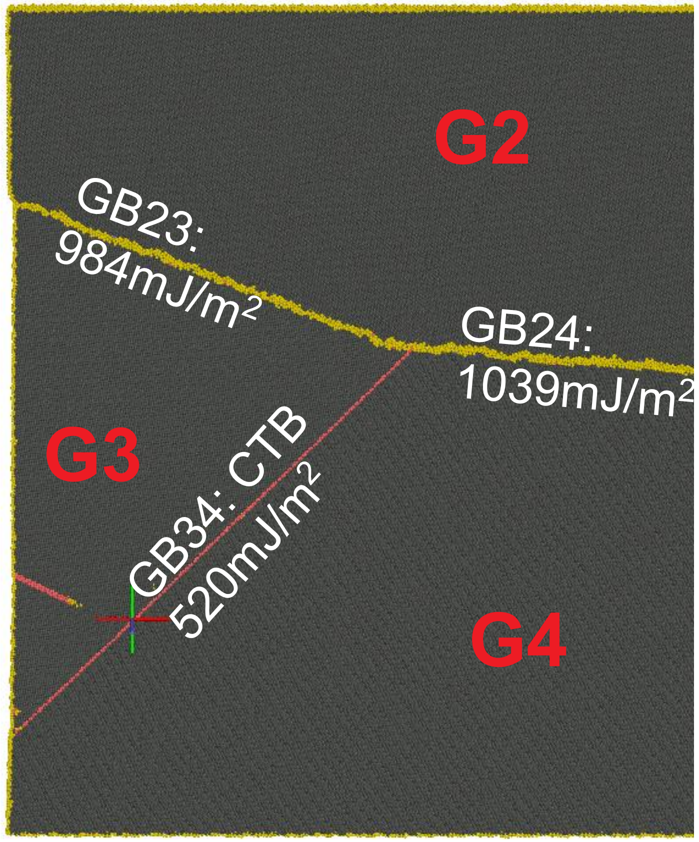
The misfit at the tip of a migrating boundary displaces the faces of nearby cracks

G. Xu and M. J. Demkowicz, PRL **111**, 145501 (2013)

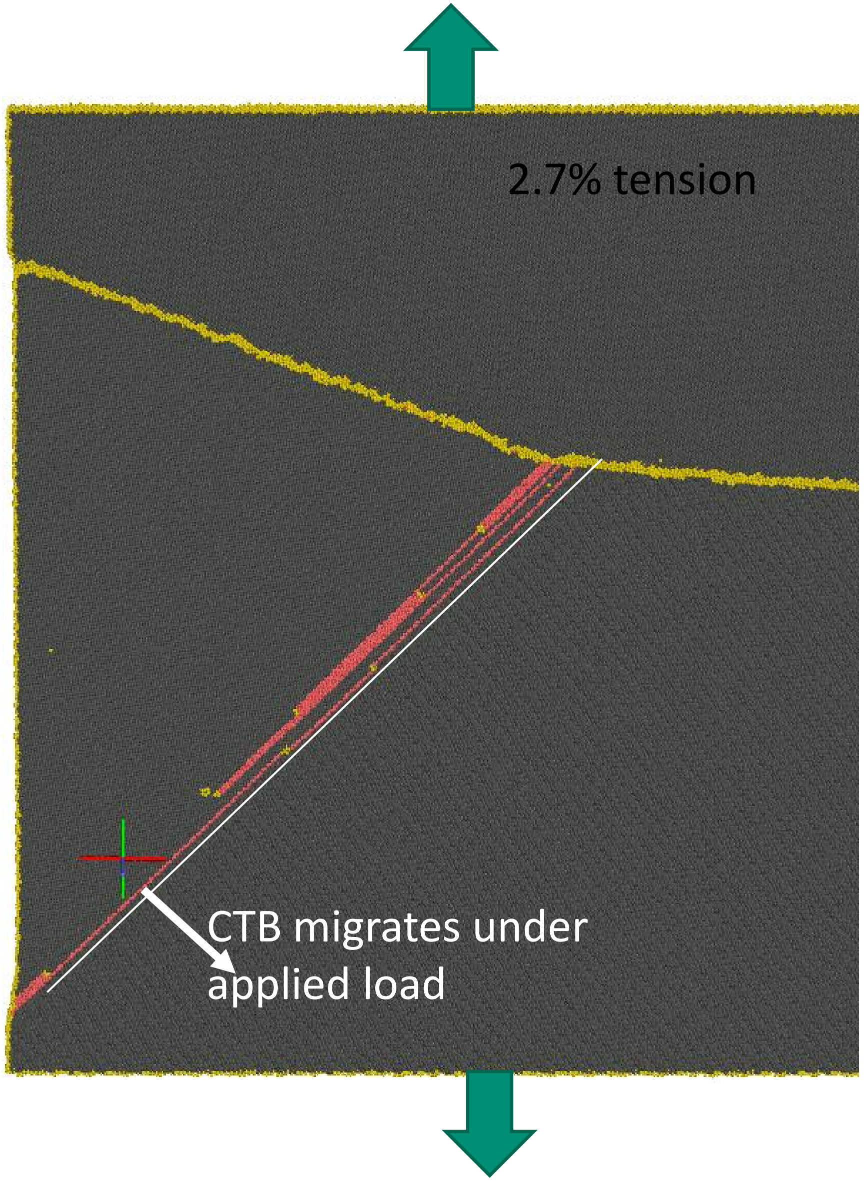
MD Simulation Based Directly on PED-ACOM Data



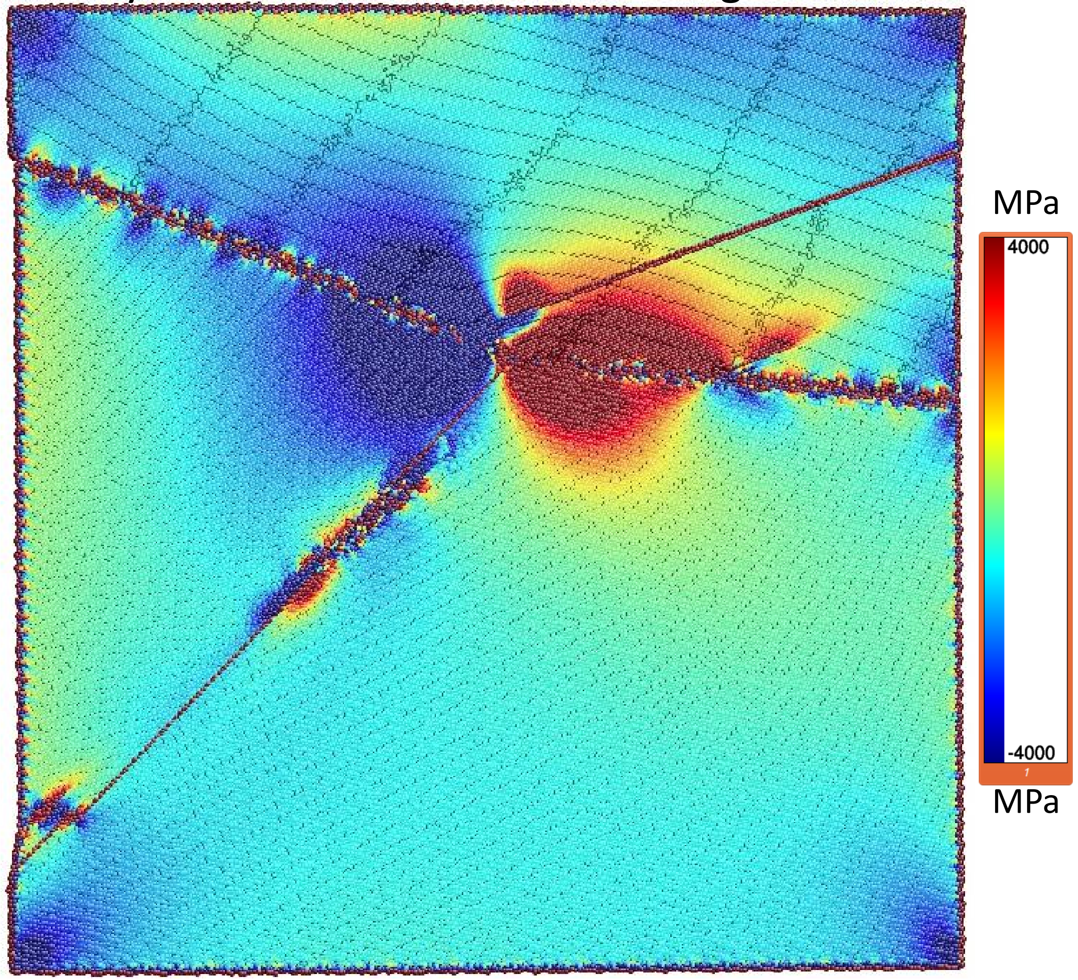




Stresses Induced by CTB Migration

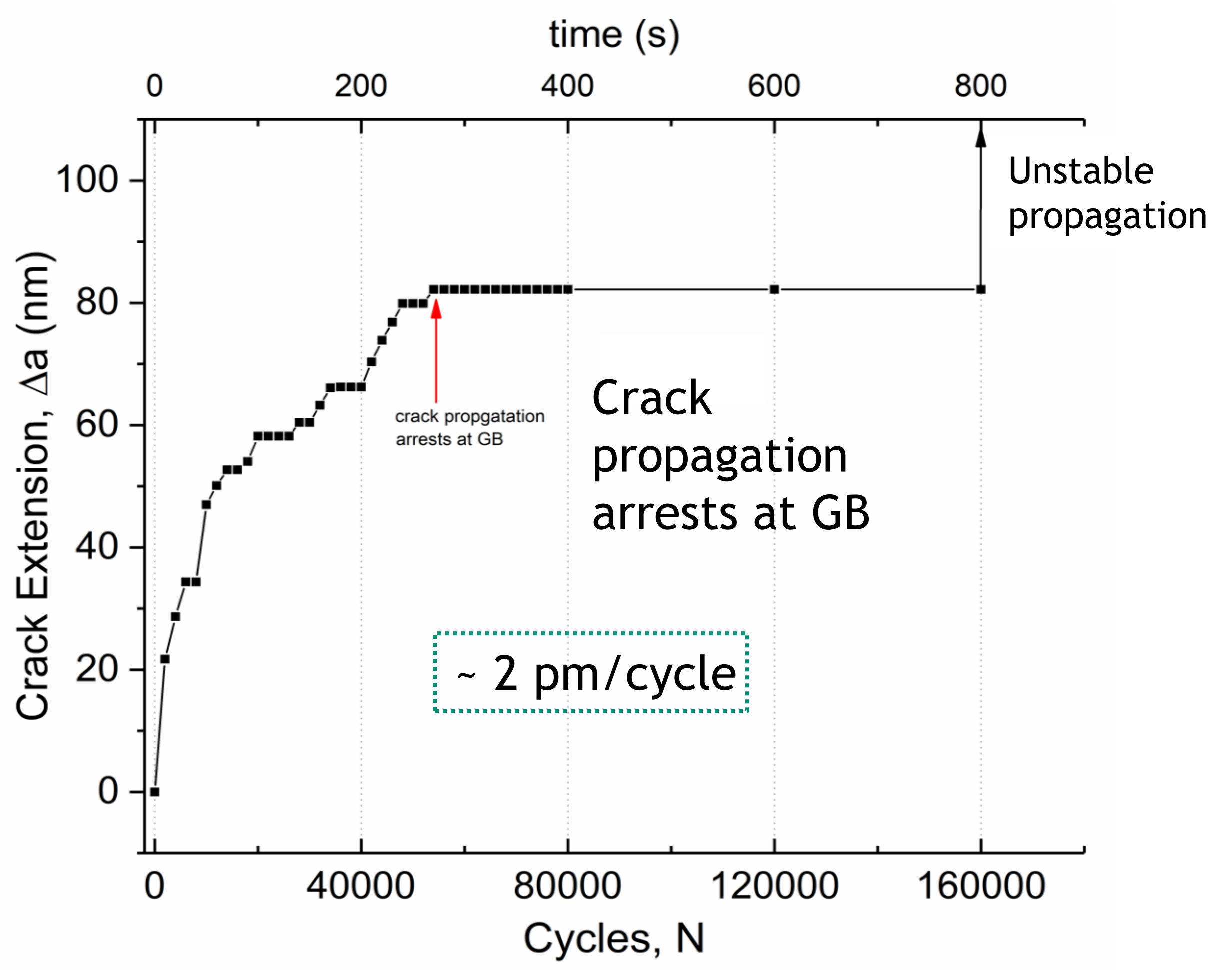


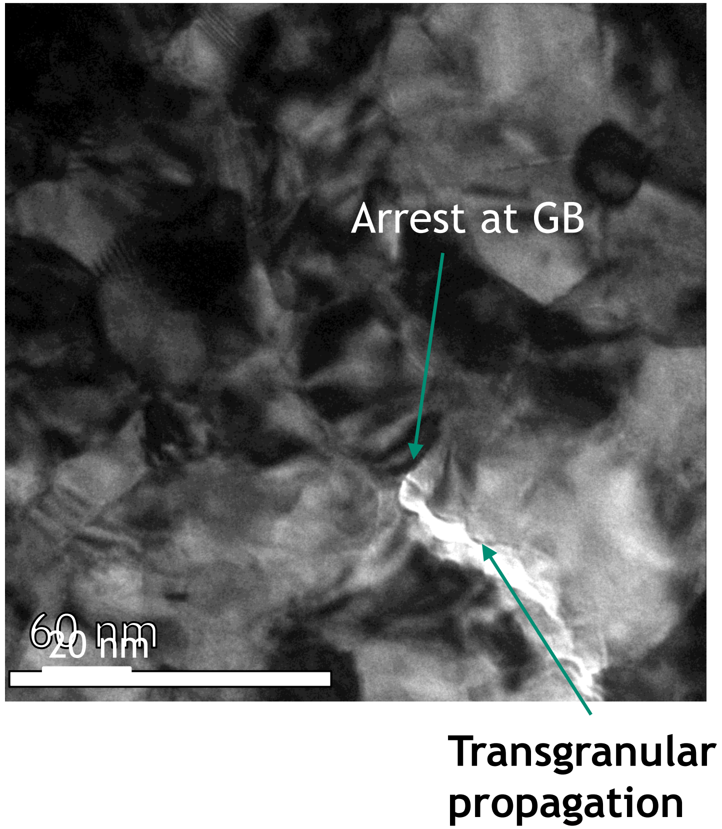
Hydrostatic stress due to CTB migration



Ongoing work will confirm the effect of these stress fields on crack face displacements

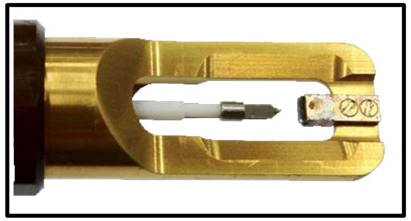
After Crack Healing: Transgranular Propagation Along Mode I





- Rapid propagation – intragranular between N = 800K to ~ 854K
- After 854K cycles, cyclic loading crack impinges grain boundary – no further propagation for addition ~106K cycles
- Mode I transgranular crack propagation

New Mechanisms Observed Through In-situ TEM Fatigue Testing



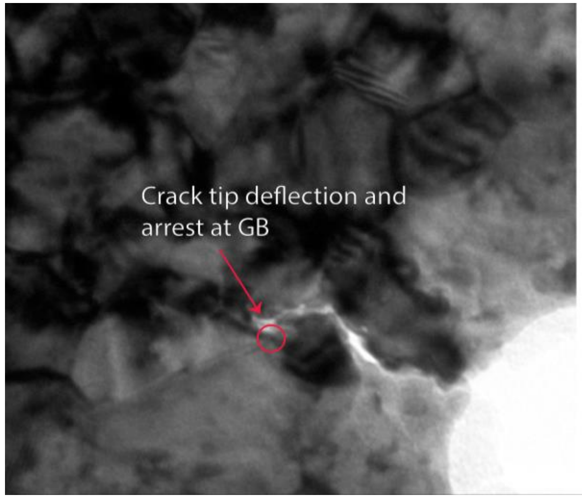
A few “first” TEM observations:

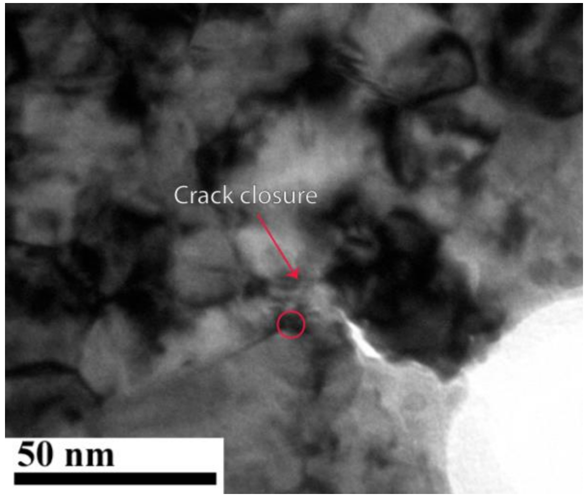
(1) Roughness induced crack closure – unlike coarse grain, deflected grain is arrested at GB

- Crystallographic crack deflection typically thought of as a toughness mechanism but is limited in NC materials due to the topological length scale (grain size)

(2) Fatigue crack healing of deflect cracks – new toughening mechanism!

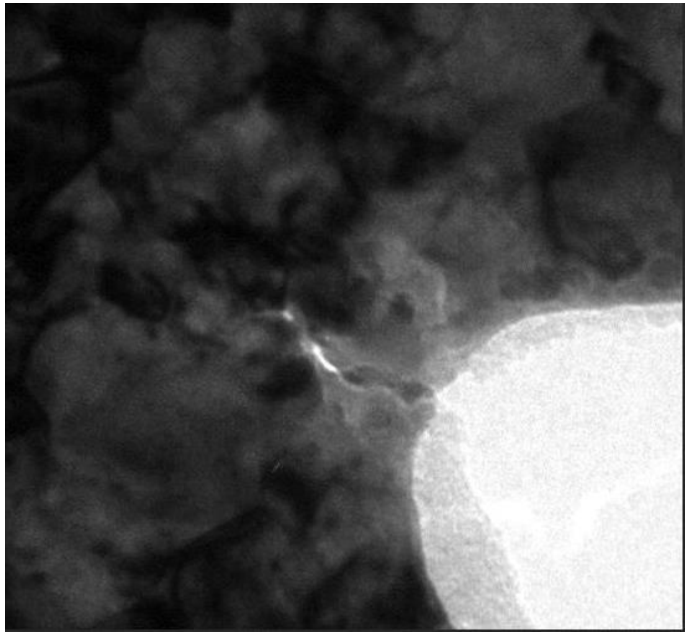
- Feasible in deflected cracks with shear and compressive components
- Vacuum and noble metal, fresh atomic surfaces
- Single grain – consistent with J. Lou orientated attachment crack healing mechanism in nanowires

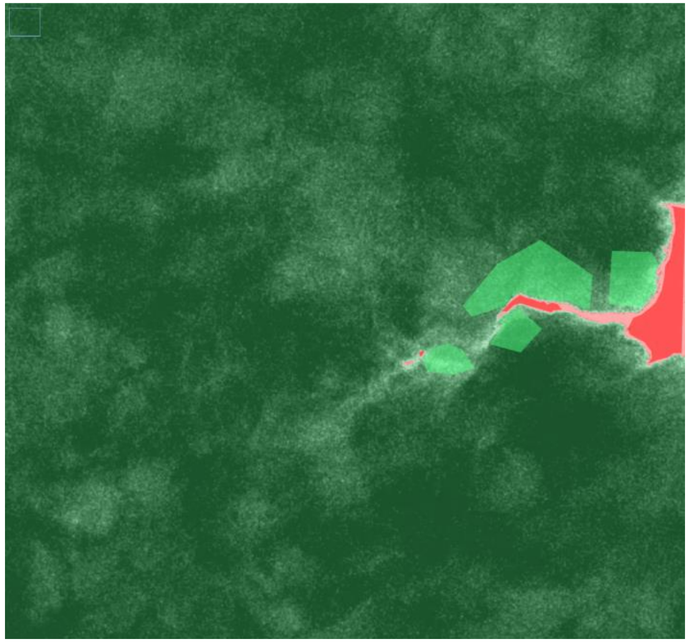


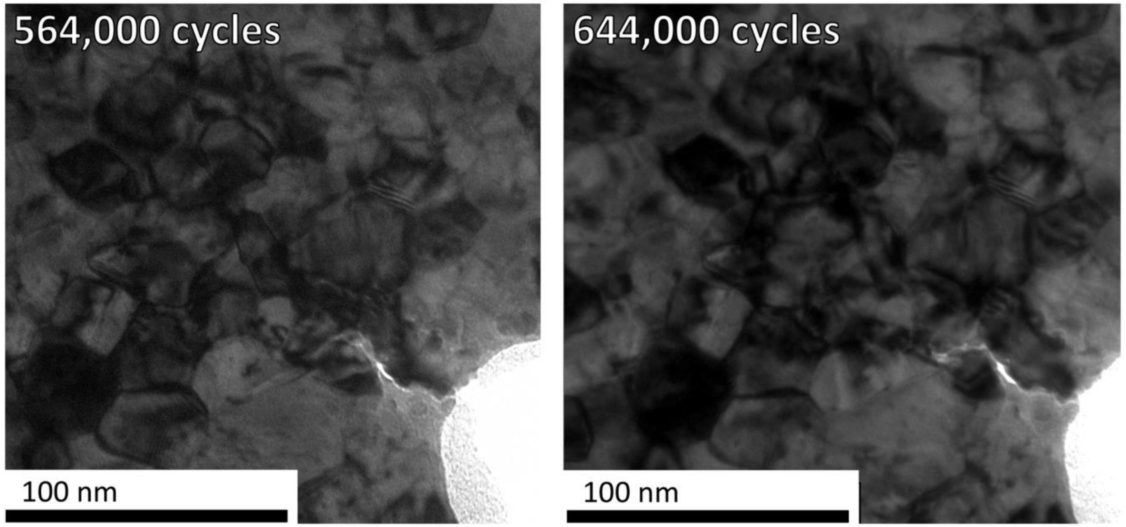


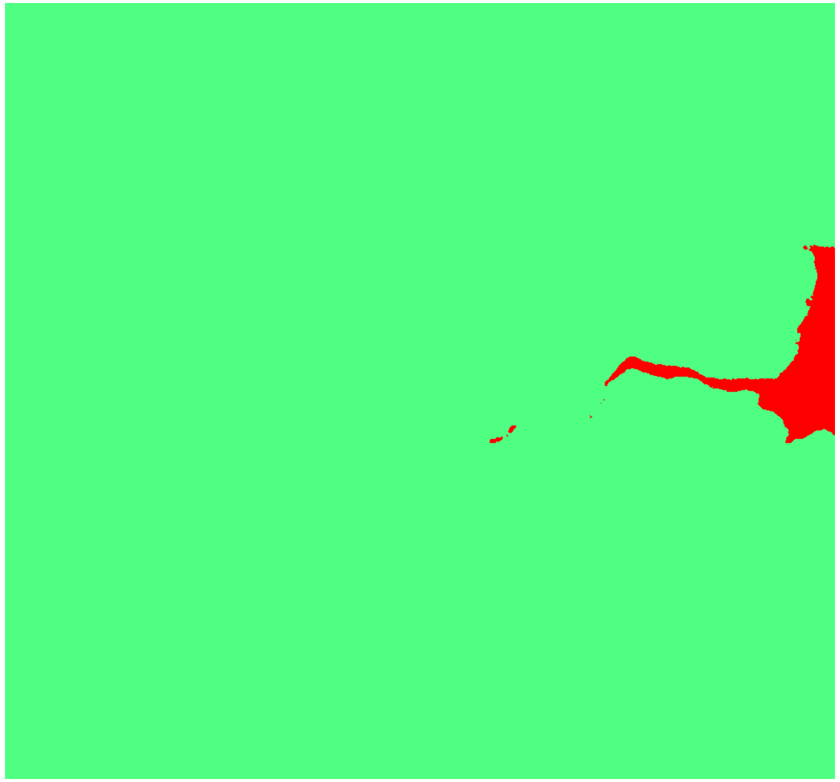
New implications for understanding nanoscale fatigue mechanism in NC metals through in-situ TEM

Can We gain More Insight through Artificial Intelligence (AI)/ machine Learning (ML)

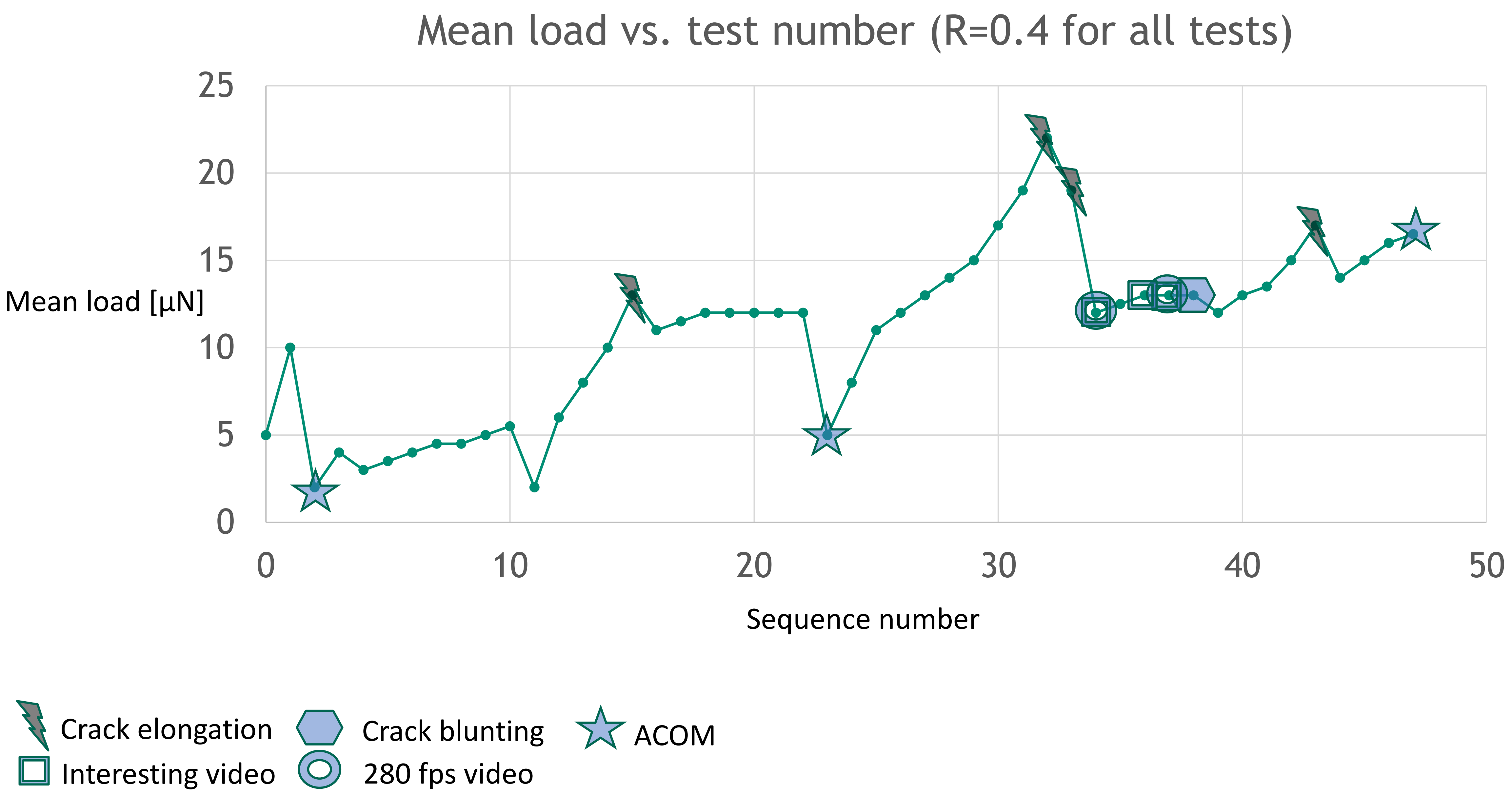




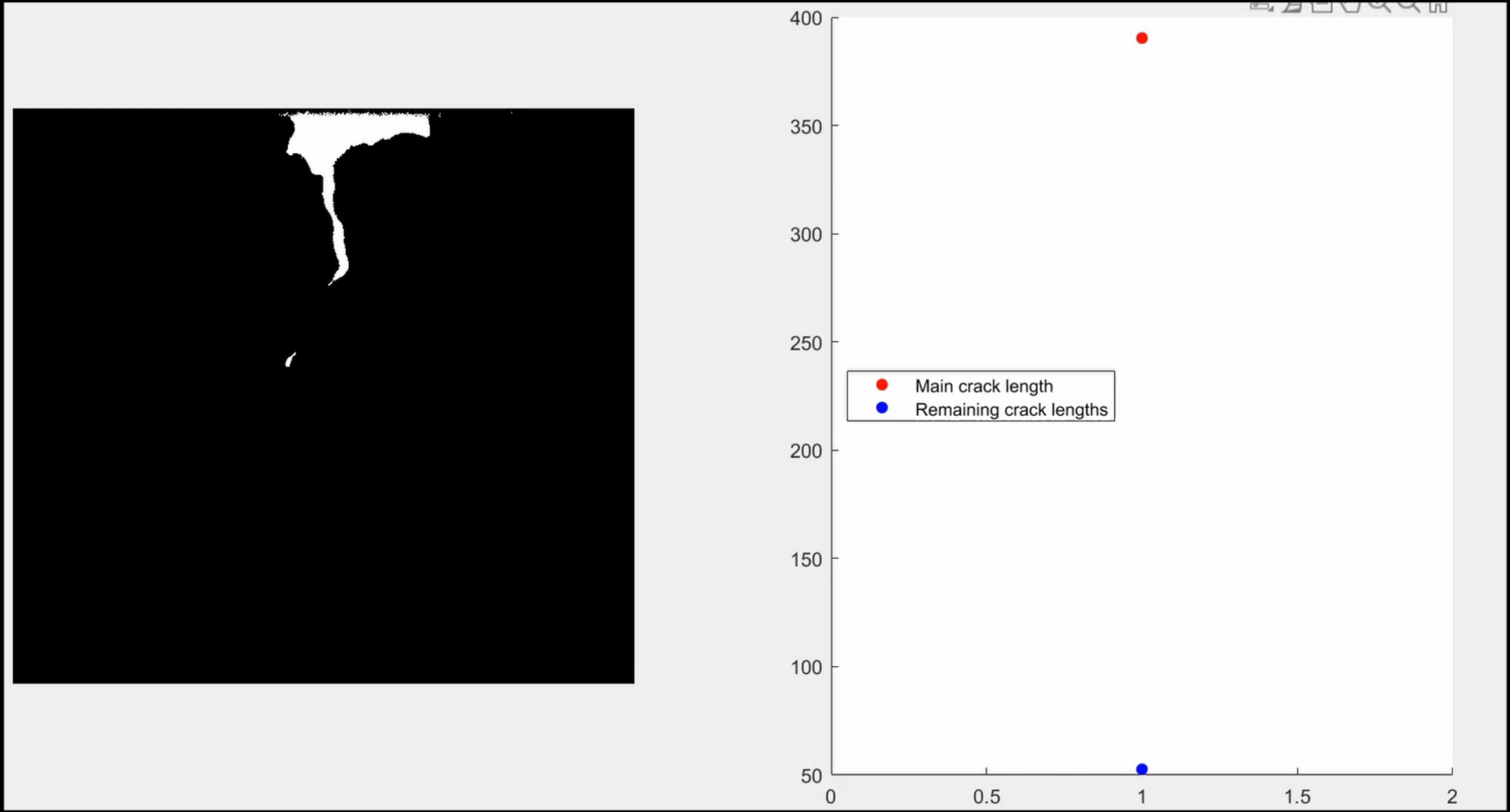




Can Apply AI/ML in the Search for More Events

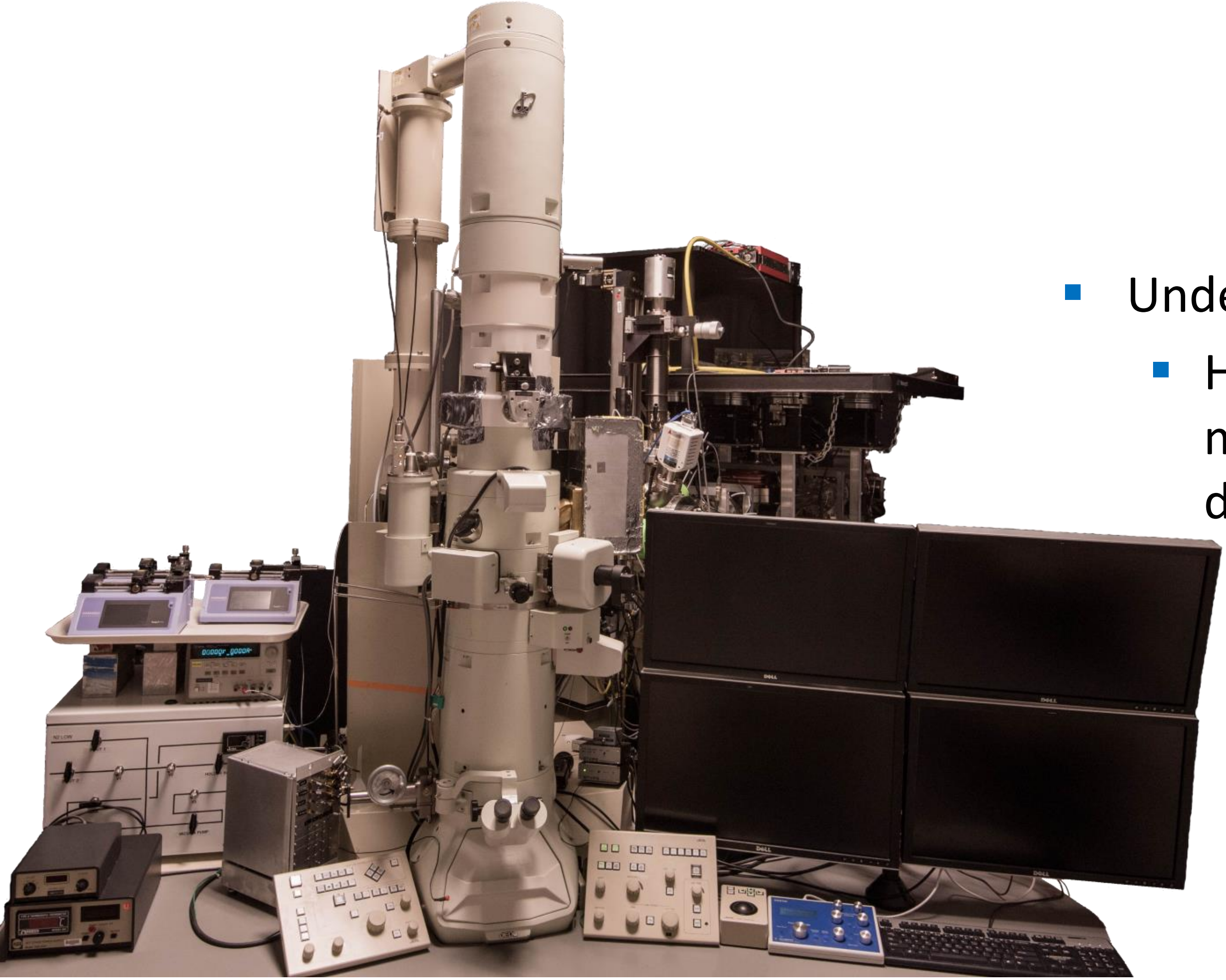


AI/ML Can Assist in Sorting the Data

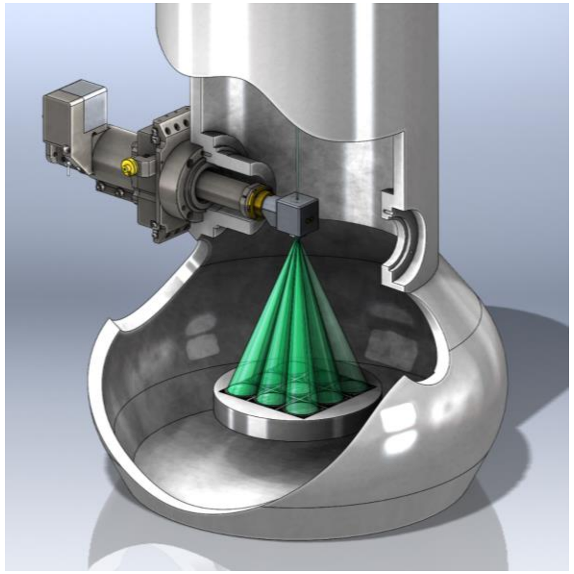


Black = sample, White= crack and “maybe crack”
DE Camera. 30fps. This video is sped up to ~450fps

Faster Imaging During In-Situ Mechanical Testing

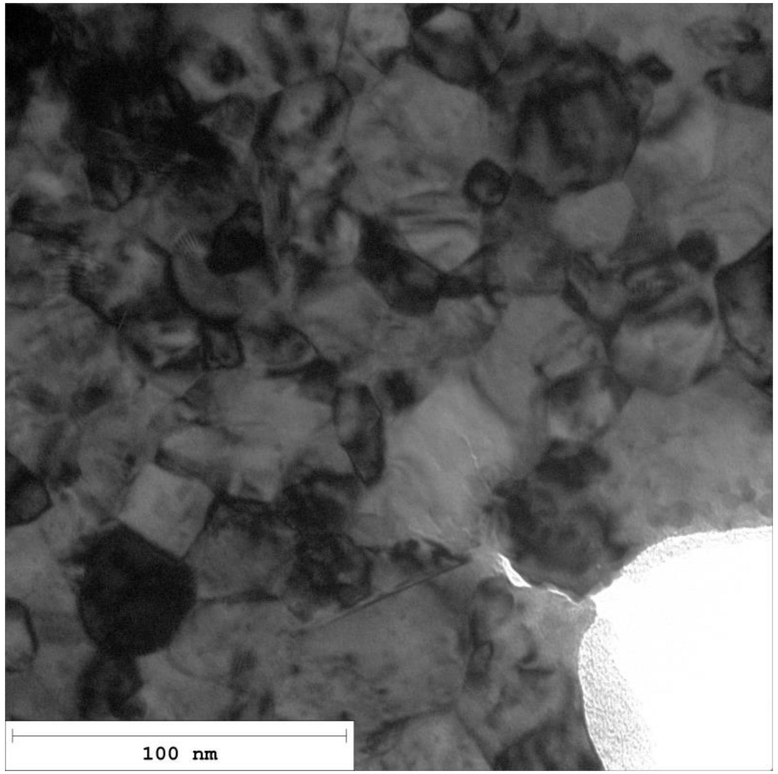


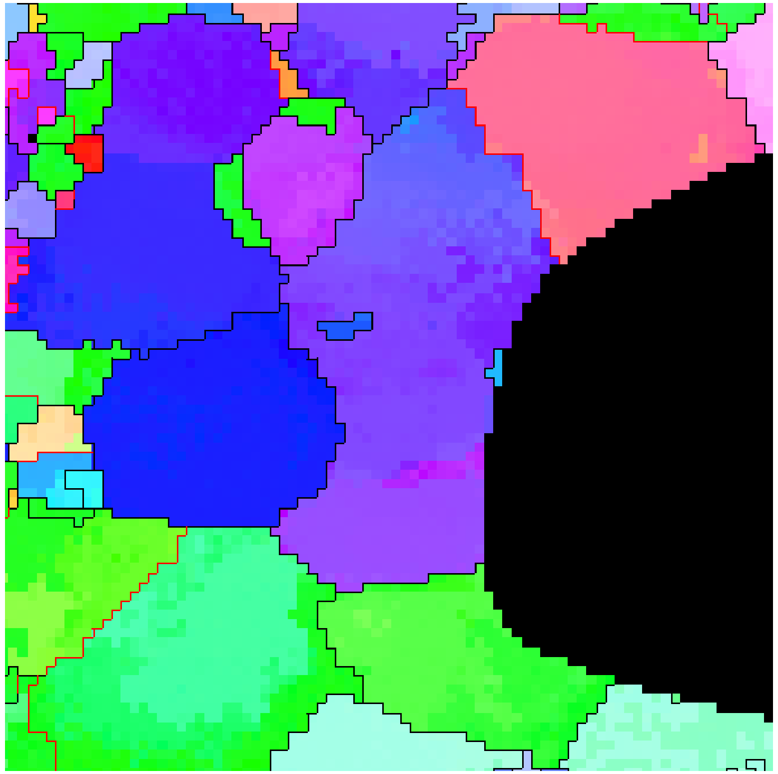
- Under development capabilities:
  - High temporal camera capabilities – remove motion blur → actual dislocation migration during loading

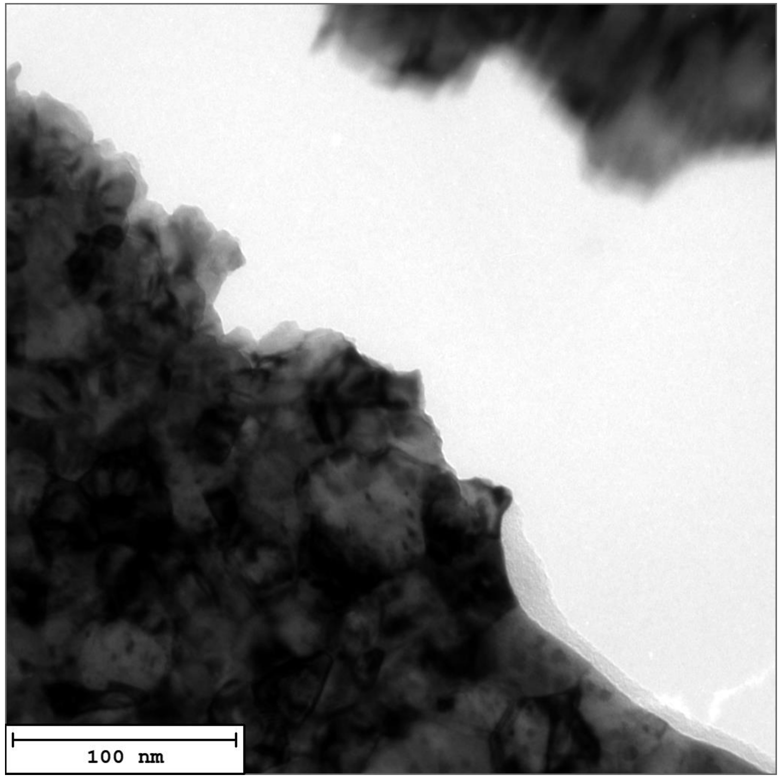


Images from Integrated Dynamic Electron Solutions (IDES):

Quantitative dynamic mechanical testing in NC Pt has provided new information regarding potential fatigue toughening mechanisms including cold welding induced crack healing!







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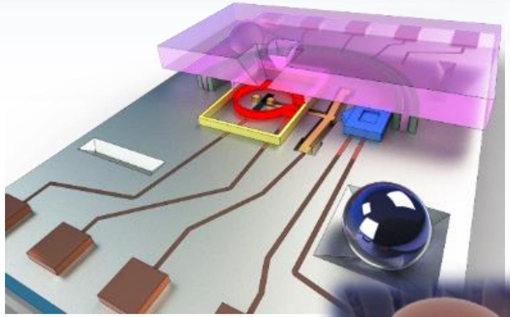
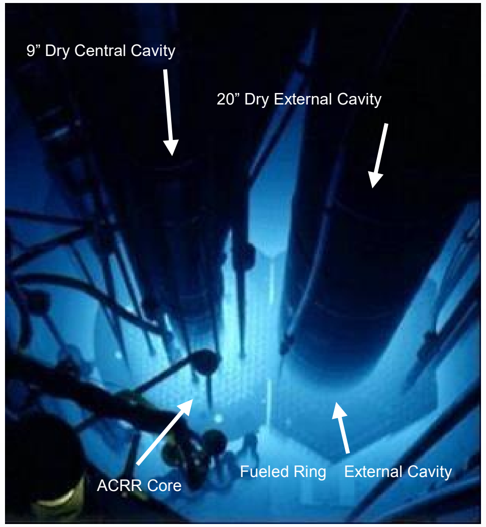
Sandia's User and Position Opportunities

D. Hanson, W. Martin, M. Wasiolek

www.cint.lanl.gov
- Spring and Fall proposals for 18 months
- Rapid Access proposal anytime for 3 months

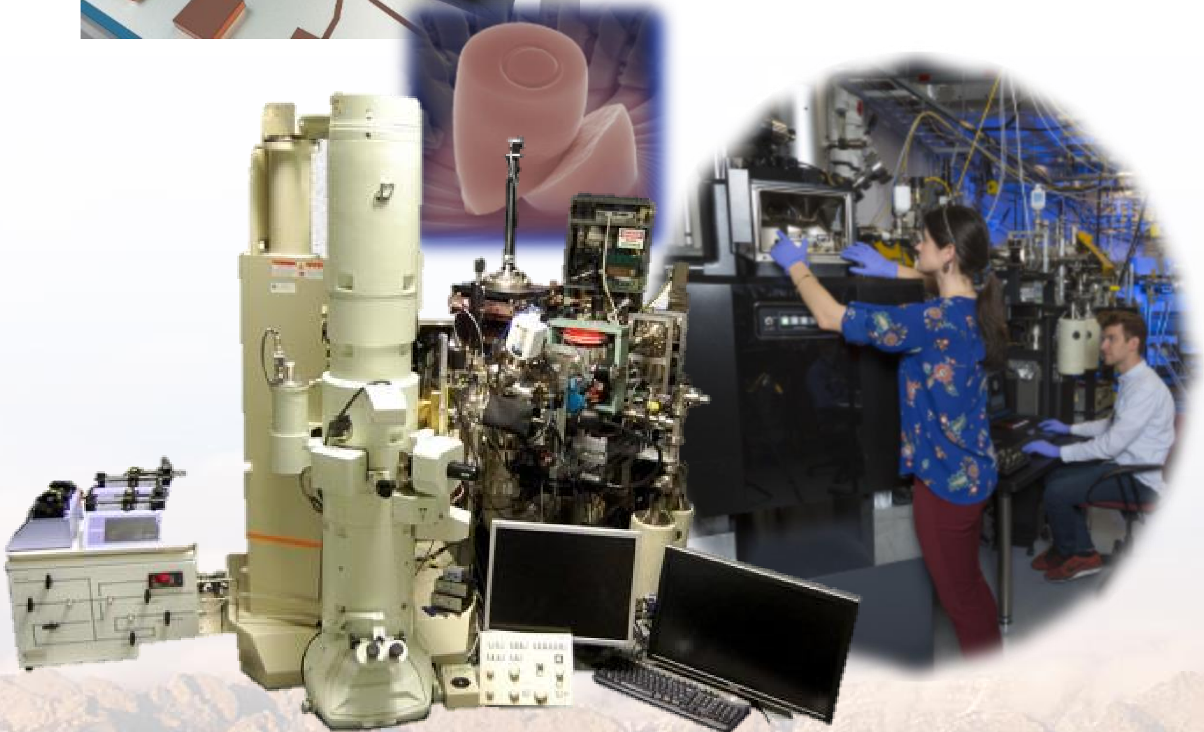


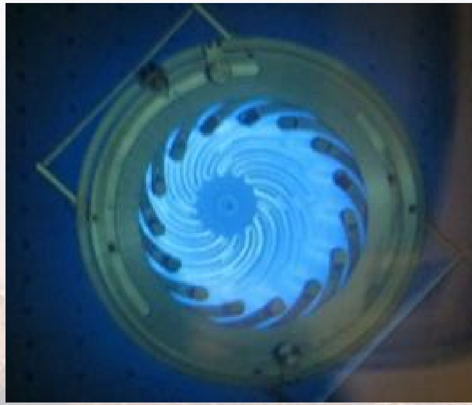
www.nsuf.inl.gov
- Three proposal a year for 9 months



Core Facility - SNL

Gateway Facility - LANL





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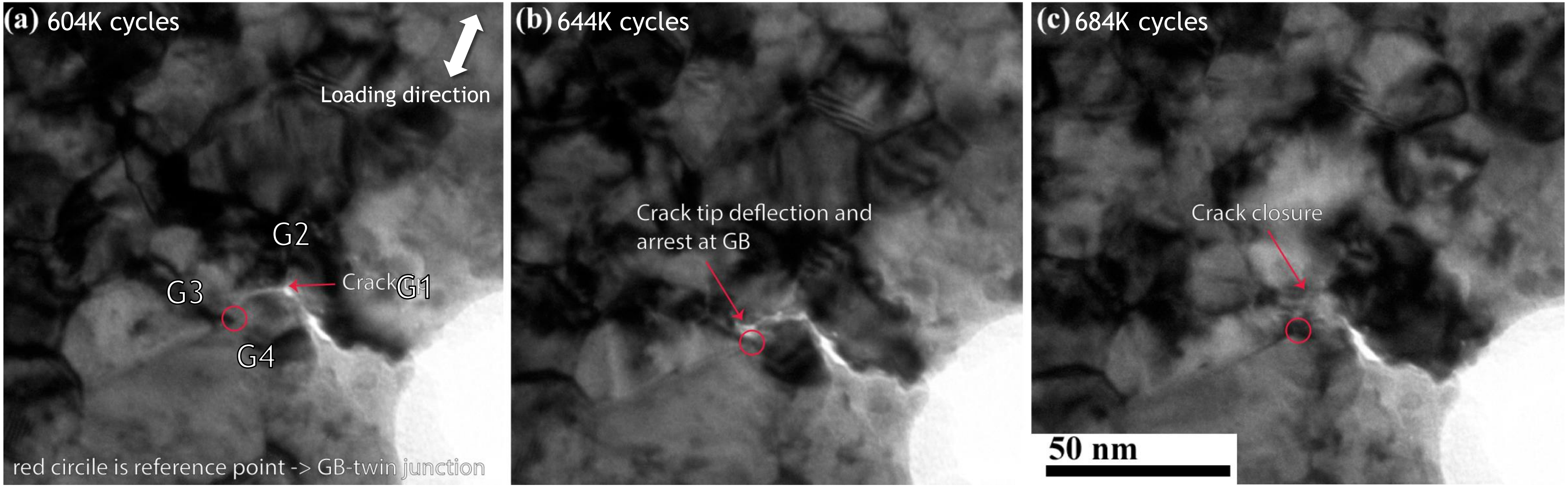
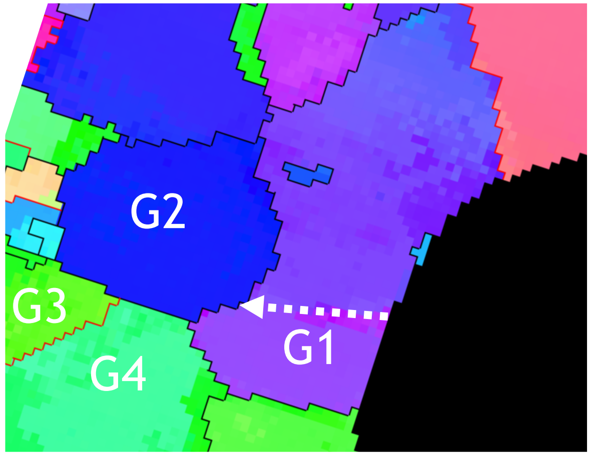
EXTRA AFTER THIS POINT

Fatigue Crack Healing: Local Crack Evolution



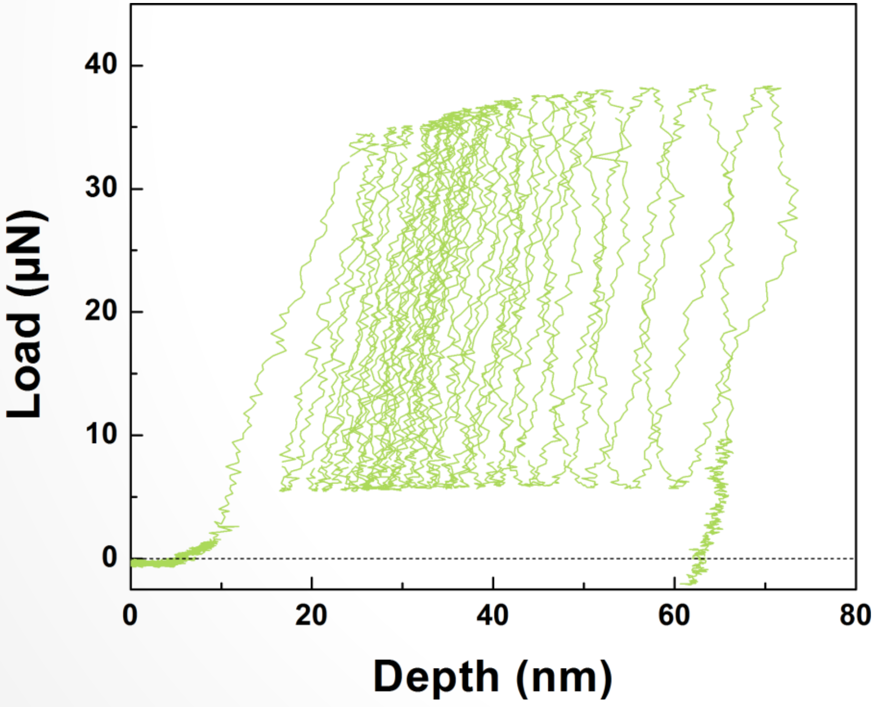
Intermediate PED
Crack at GB between G1 and G2

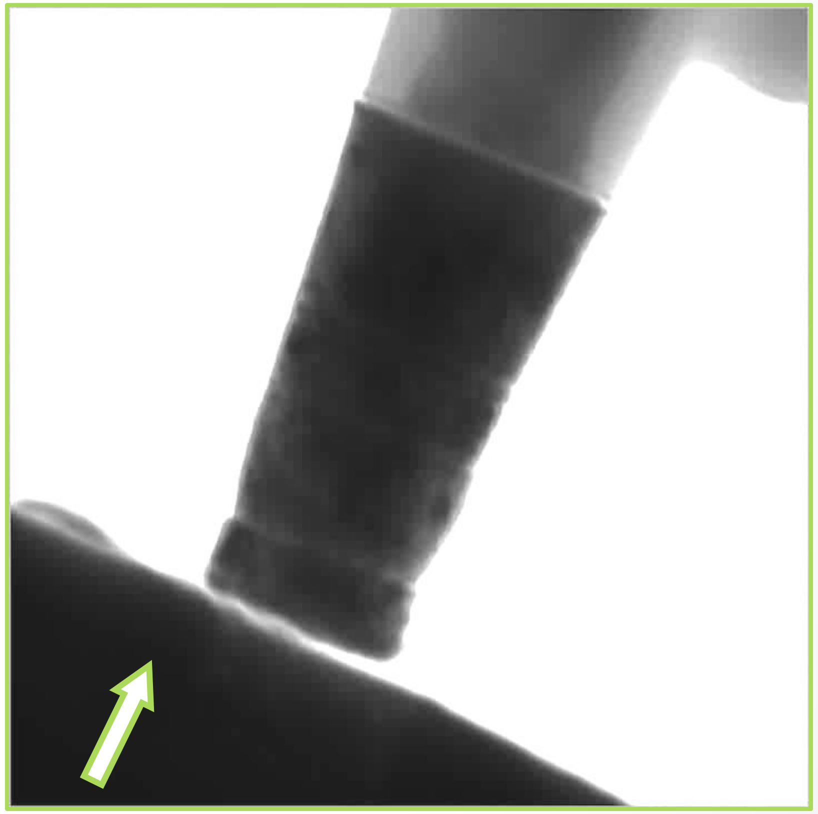


1. Fatigue crack propagates into Grain G2
2. In G2, crack deflection away from Mode I: Doubly-kinked crack deflection with local tip stress state k2 (shear) > k1 (tension)
3. Crack temporarily arrested at GB between G2 and G3
4. Next test (between 644K and 684K, roughness induced crack closure and full healing of deflected crack!

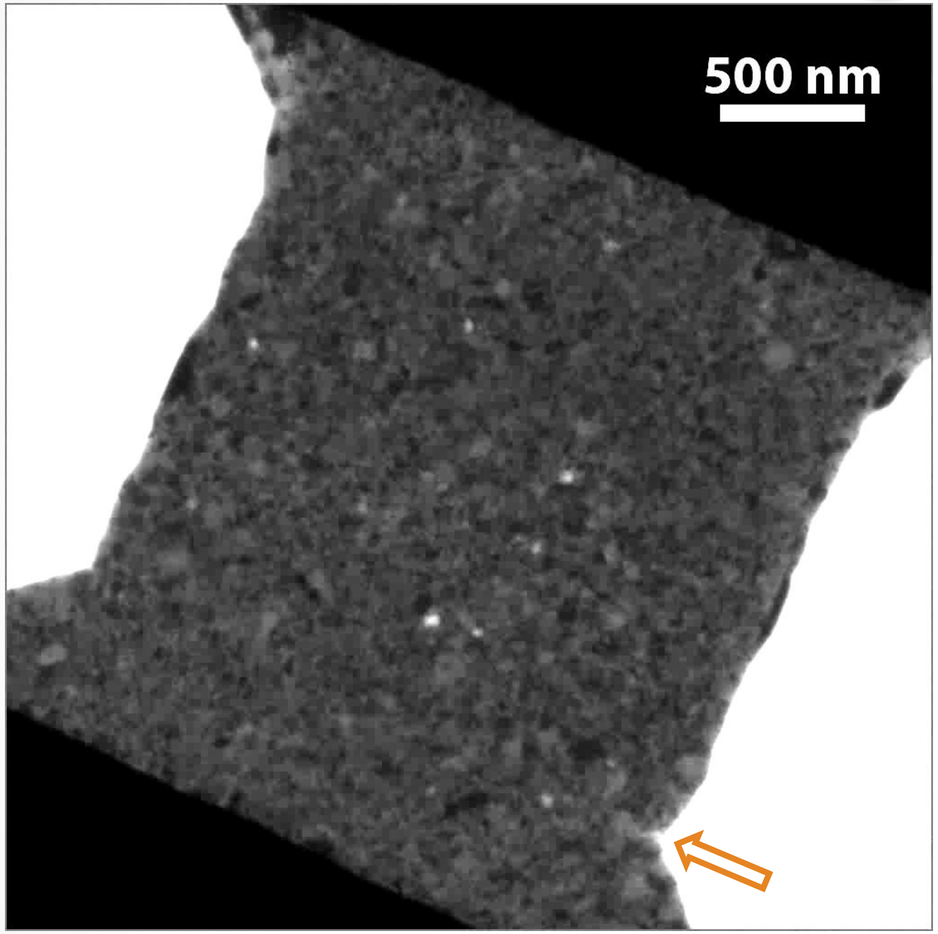
Pillar Compression

- Cyclic loading with increasing force amplitude
  - After a previous monotonic compression
  - 23 cycles to failure
- Failure initiated at notches



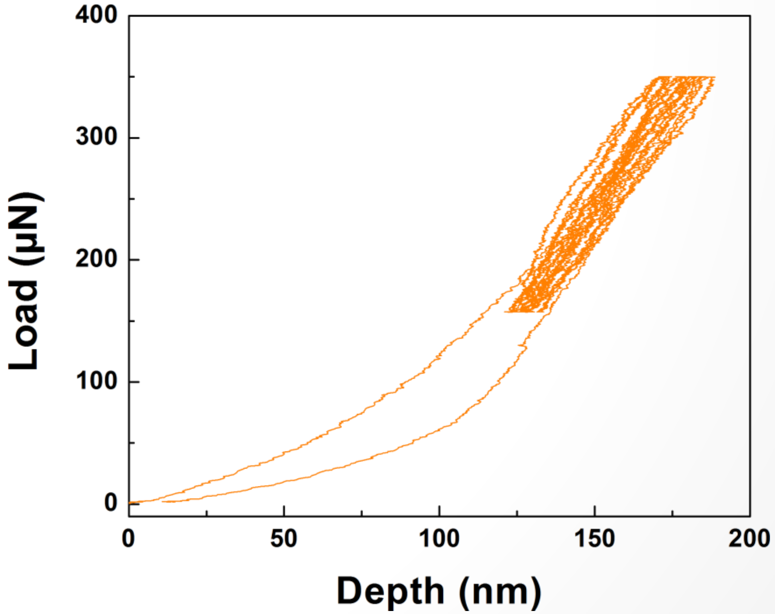


Tension Testing

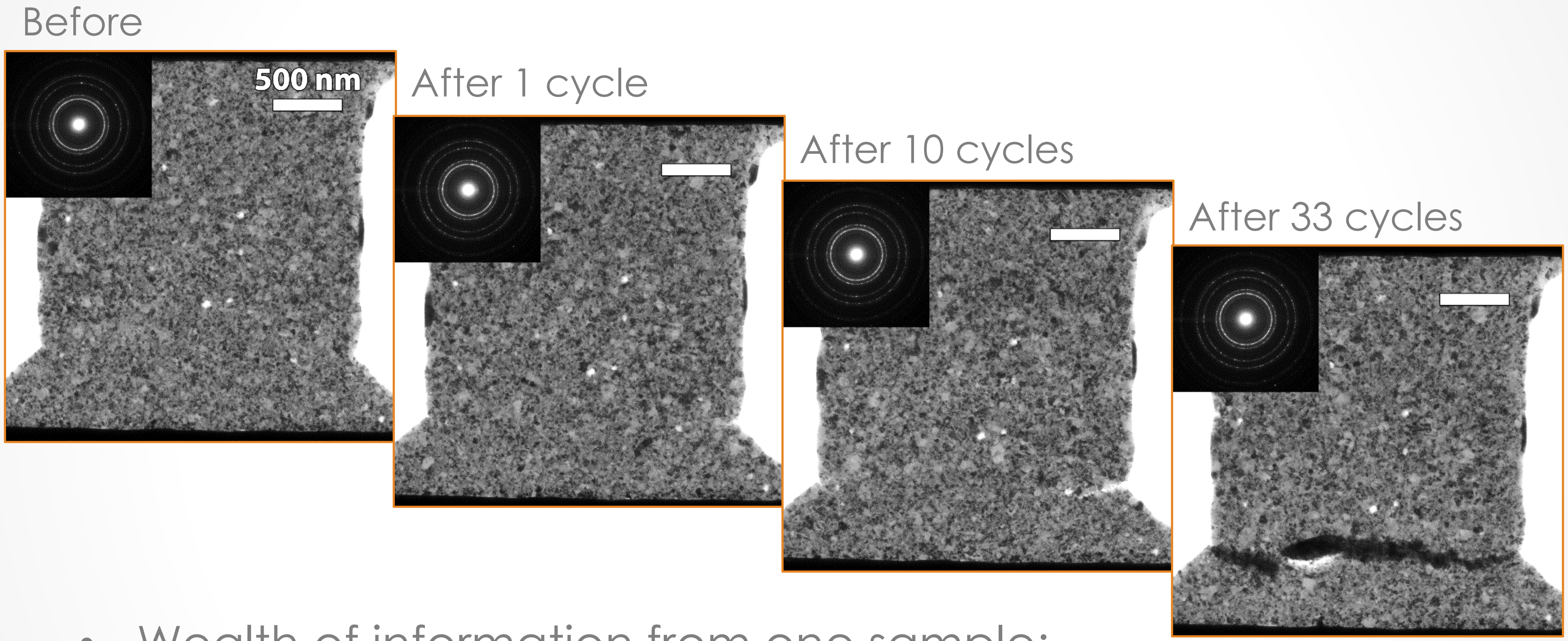


- Slow crack propagation
- Evidence of grain growth

- Cyclic loading:
  - Crack initiated in previous monotonic test
  - 9 cycles to ~87.5% of that load
  - 50 % unloading
  - Slow crack propagation



Cyclic Tension *In Situ*



- Wealth of information from one sample:
  - Images and electron diffraction at each stage
  - Video and force/displacement during load cycles
- Microstructural change still elusive
  - Difficult to confirm and quantify

Quantifying Microstructural Change

- Combining orientation mapping with deformation
- EBSD-like capability in the TEM
  - Powerful analytical tools available