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High-Cycle Fatigue *In Situ* in the Transmission Electron Microscope

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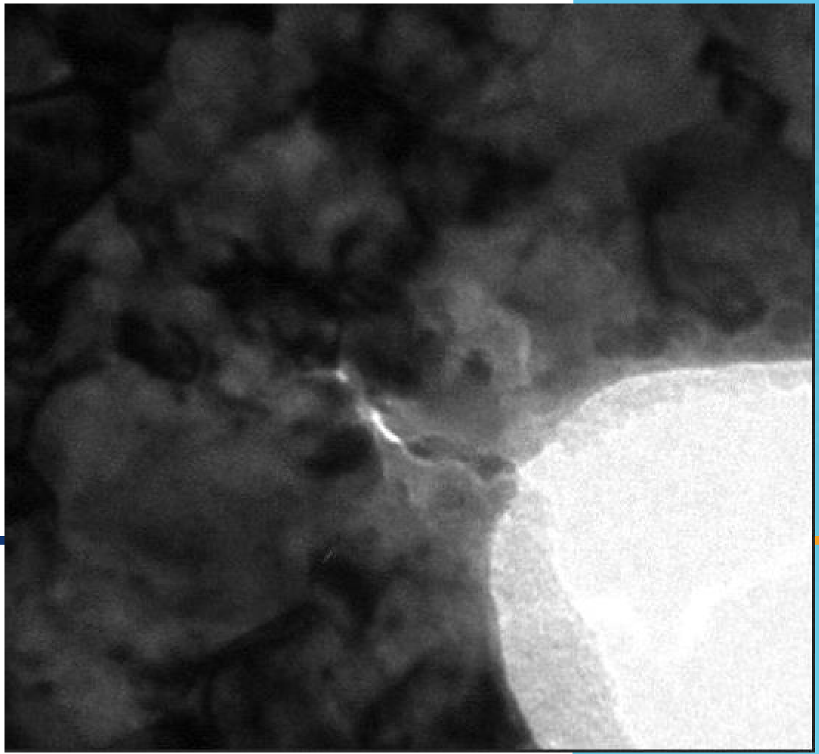

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Utilization In-situ TEM to Explore Fatigue in Nanocrystalline Metals

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

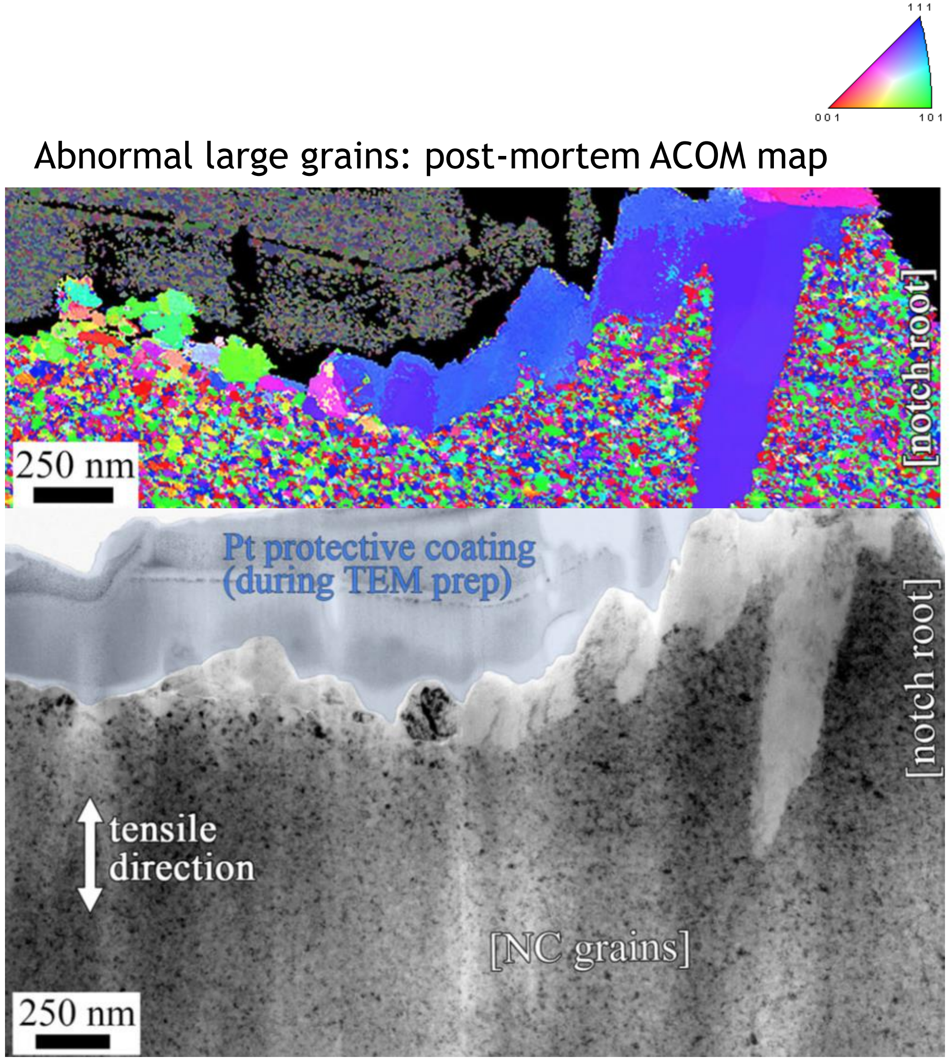
Fatigue behavior in nanocrystalline metals associated with:

- 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?

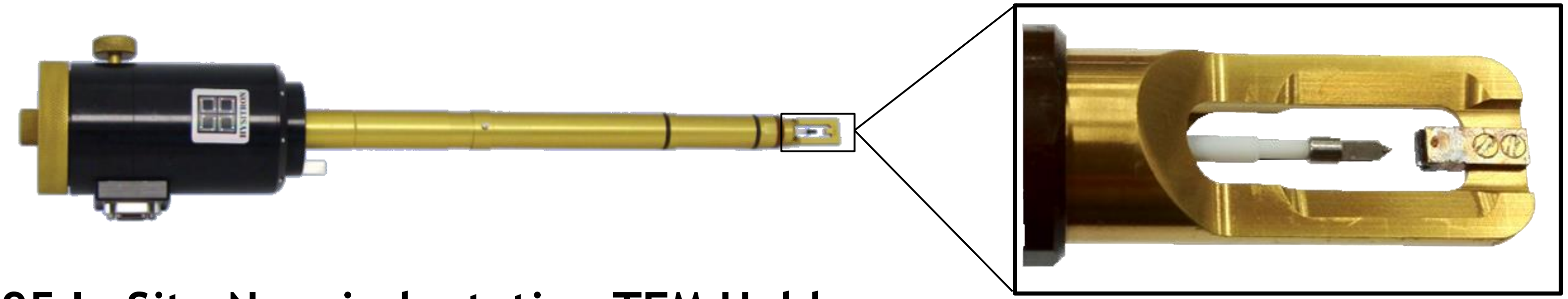
- **In situ TEM cyclic loading techniques provide the spatial resolution needed to investigate these questions**

Abnormal large grains: post-mortem ACOM map



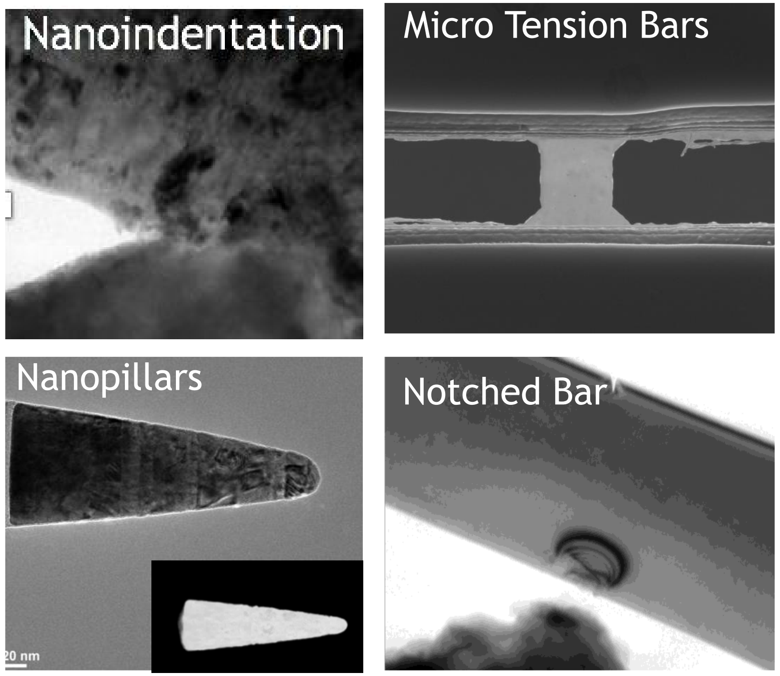
T.A. Furnish et al., J. Mat Sci 52:46-59 (2017)

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

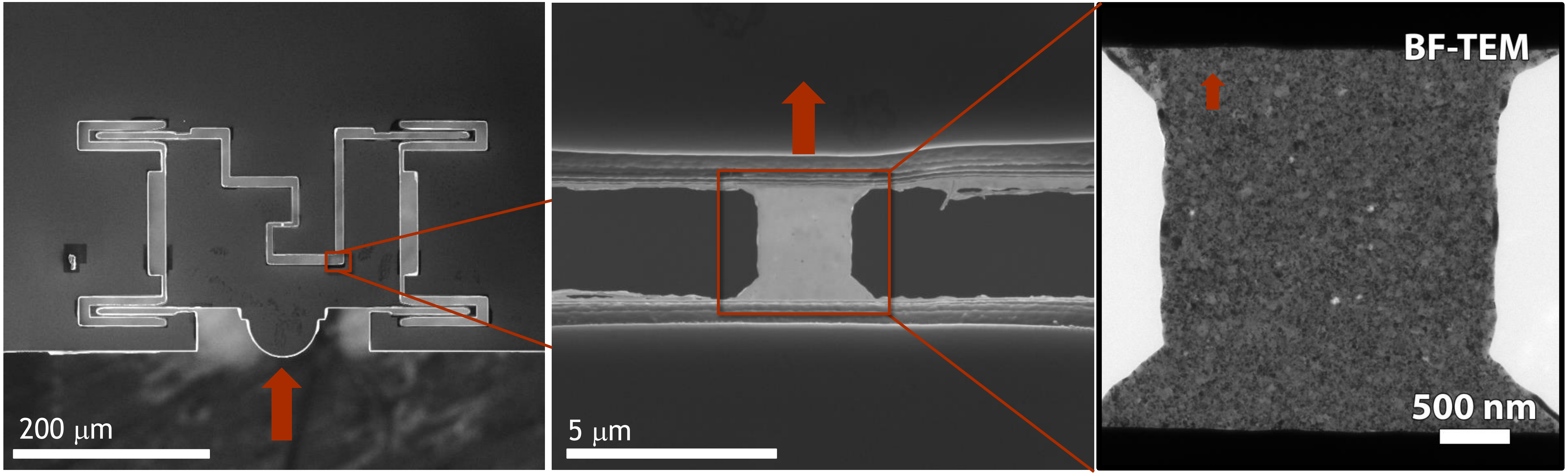
Can be combined with other external stimuli at I^{3}TEM (IR laser, heavy and light ion irradiation, and heating)

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

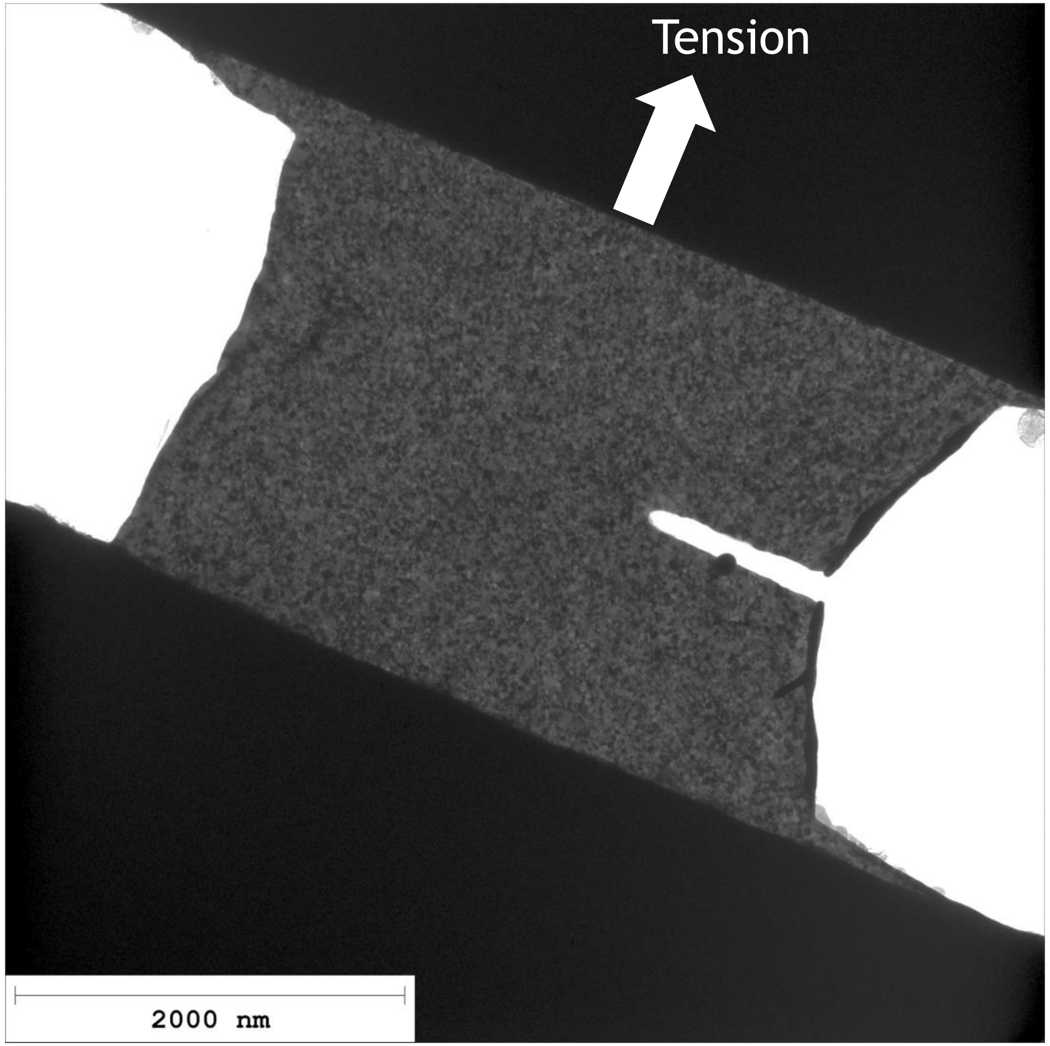
Nearly pure tension, uniform cross sectional area, stable load frame

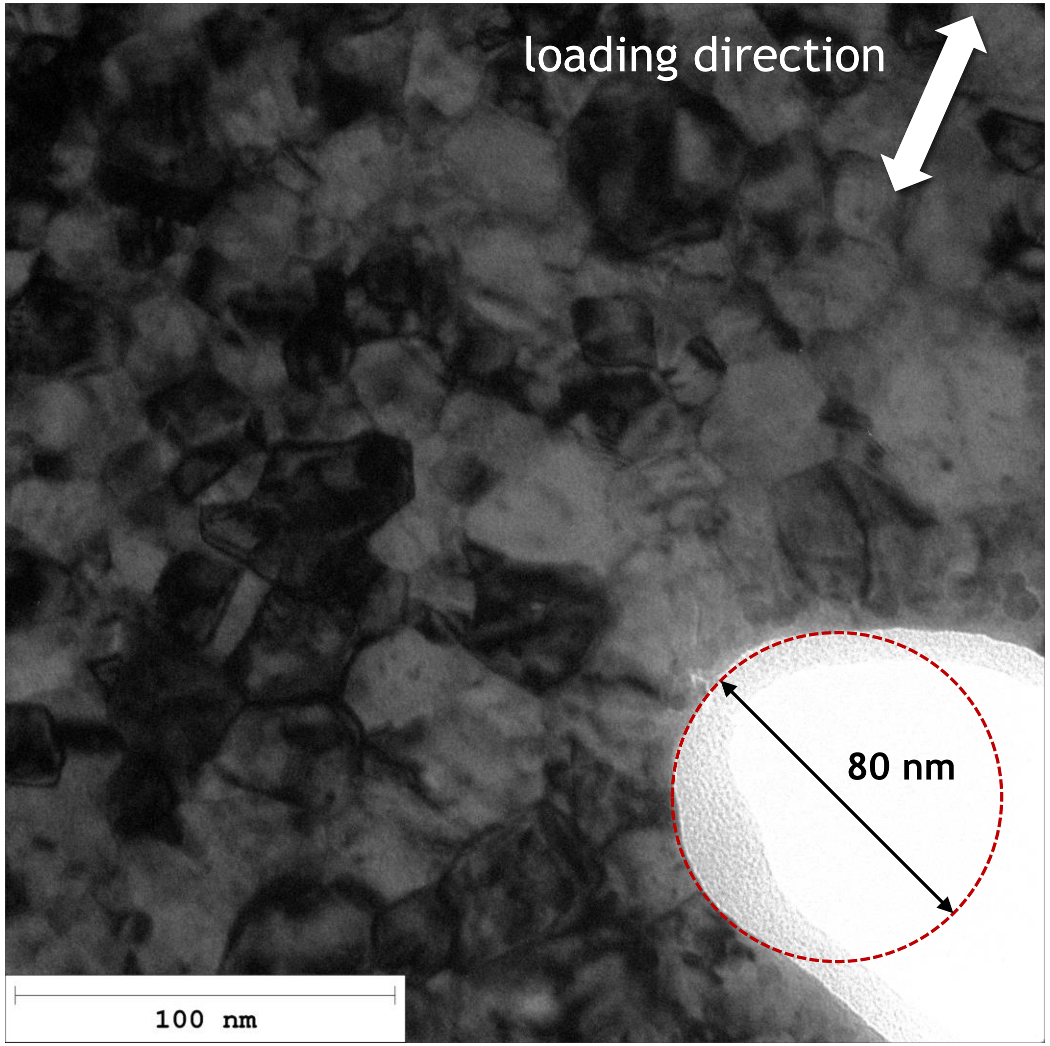
$$F_{applied} = F_{measured} - F_{spring}$$



Bufford et al. Nano Letters (2016)

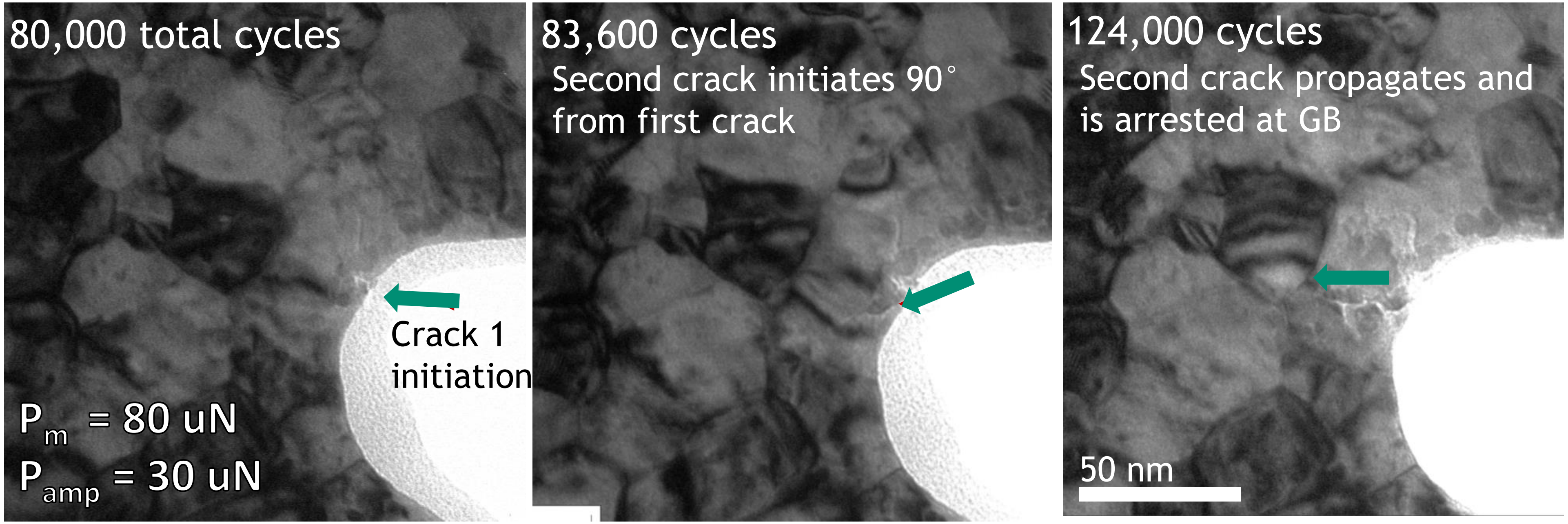
Notched Pt: In-situ Cyclic Loading





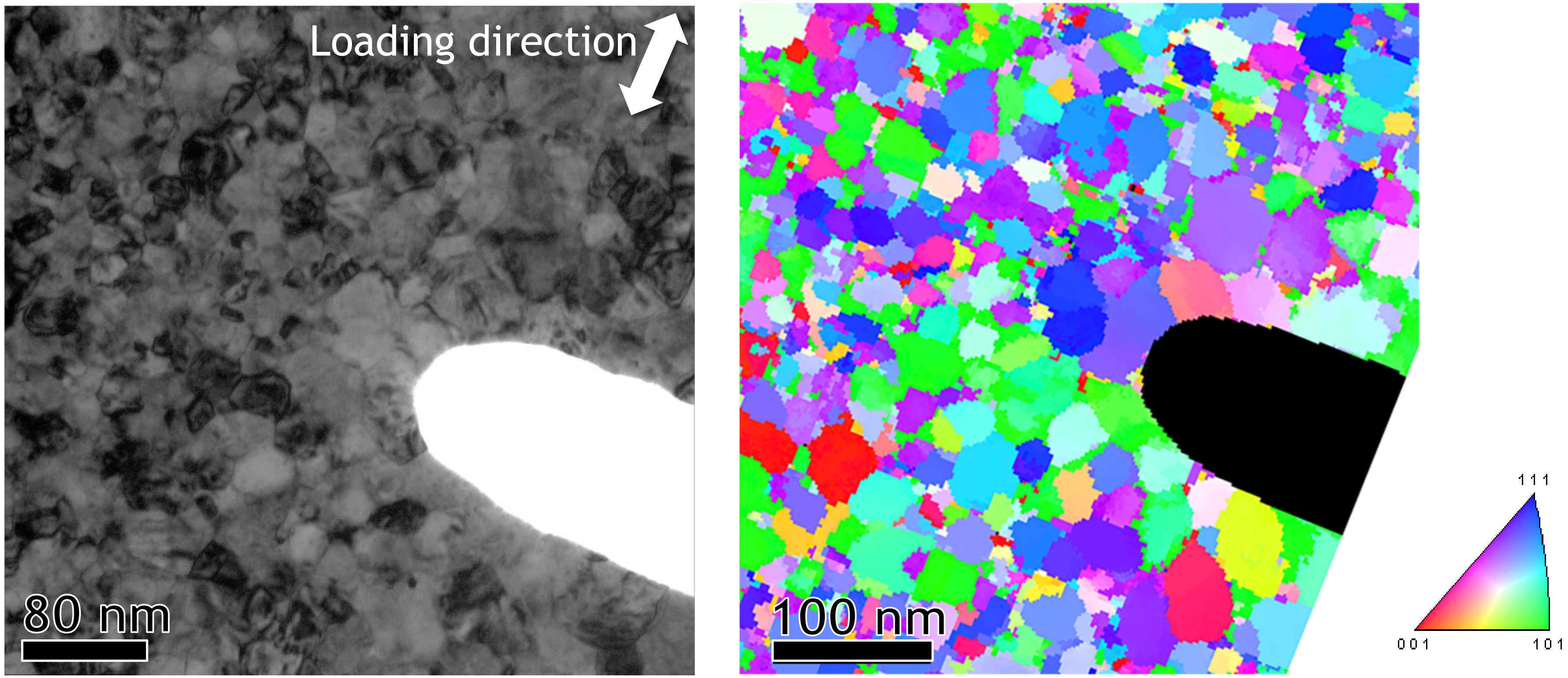
- Notch length = 950 nm, Gauge width = 3.3 um
- Notch created by FIB “line”

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

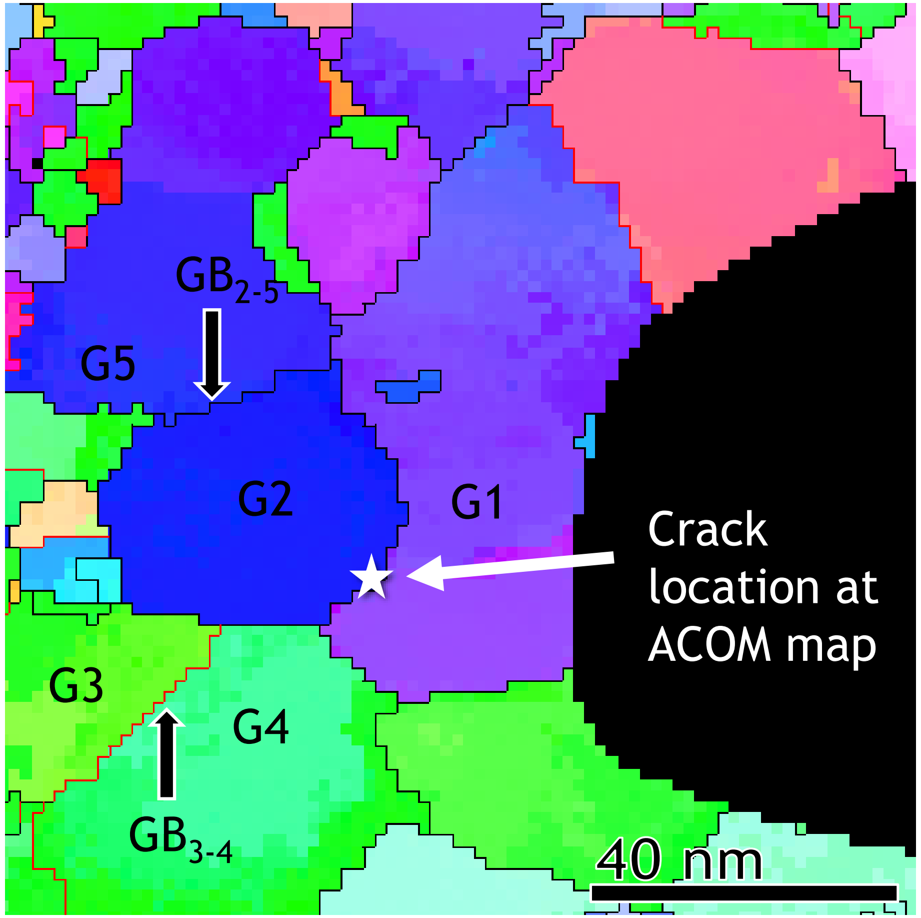
ACOM-PED coupled In-situ TEM Fatigue



- 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



- 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

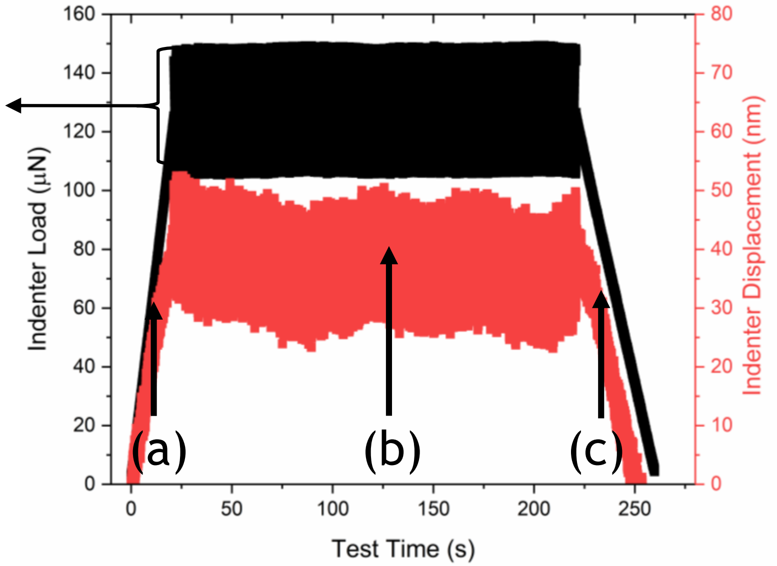
IDs:	Pre- Test ACOM	Intermediate ACOM
GB$_{3\text{-}4}$	59.3° [1 1 1]; Σ3 Dev. 0.9°	56.9° [7 7 6]; Σ3 Dev. 4.9°
GB$_{2\text{-}5}$	34.9° [12 10 11];Σ7 Dev. 4.1°	38.0° [12 11 13]; Σ7 Dev. 1.9°

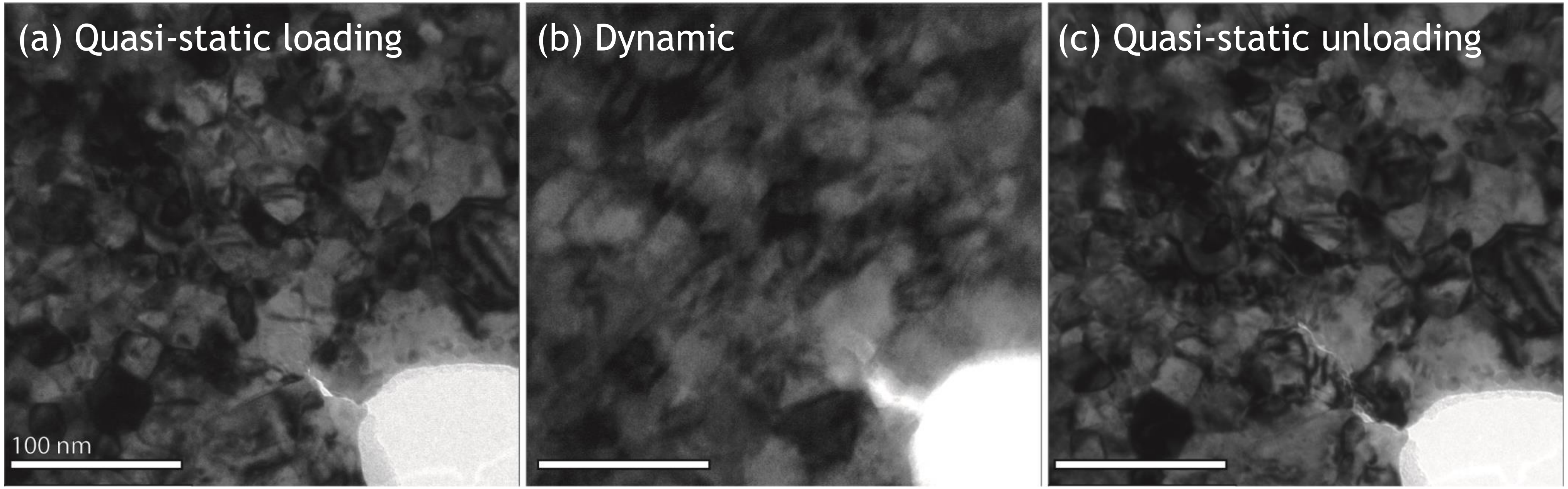
* Intermediate orientation map at 164,000 total cycles – crack at GB$_{1\text{-}2}$

Cyclic Loading in TEM Protocol

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

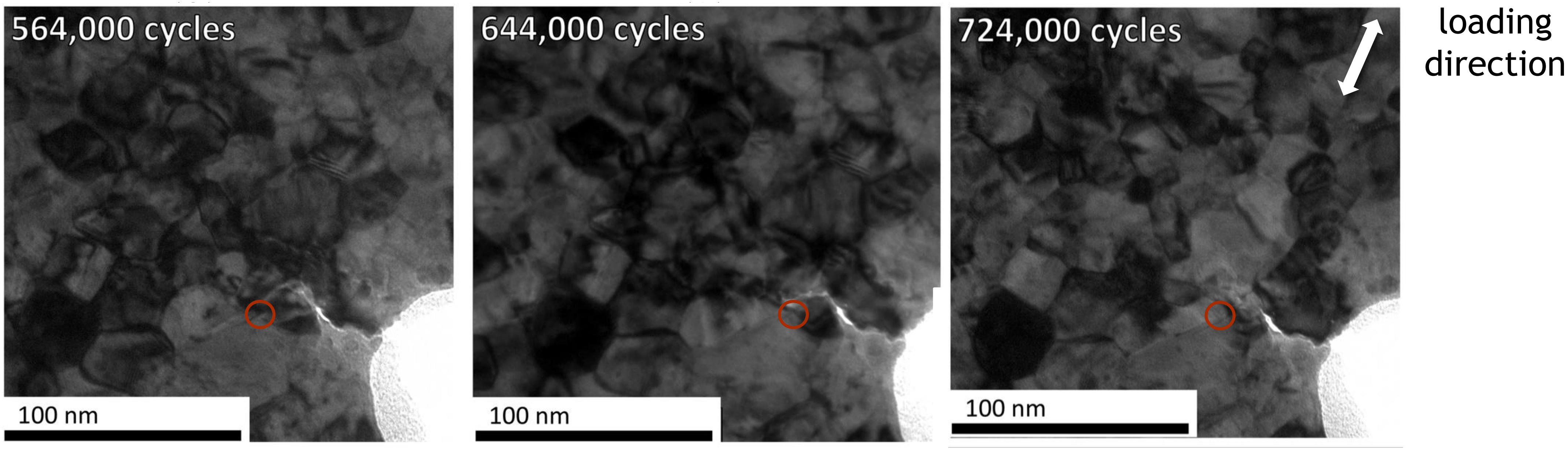
- **200 Hz Frequency**
- **40,000 cycles in 200s**





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

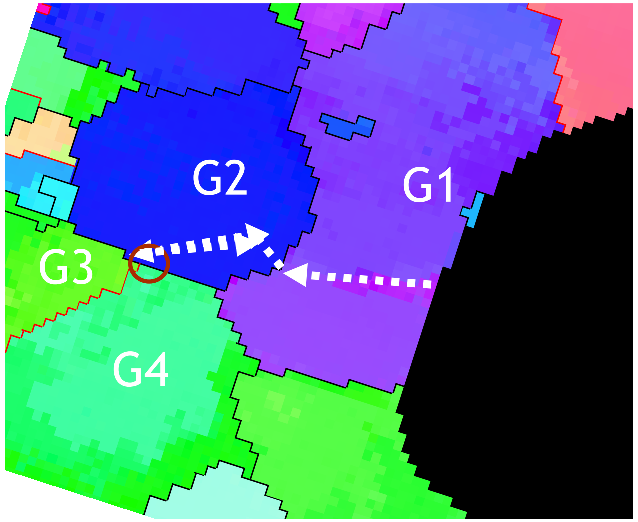
Crack Propagation, Deflection, Closure, and Healing



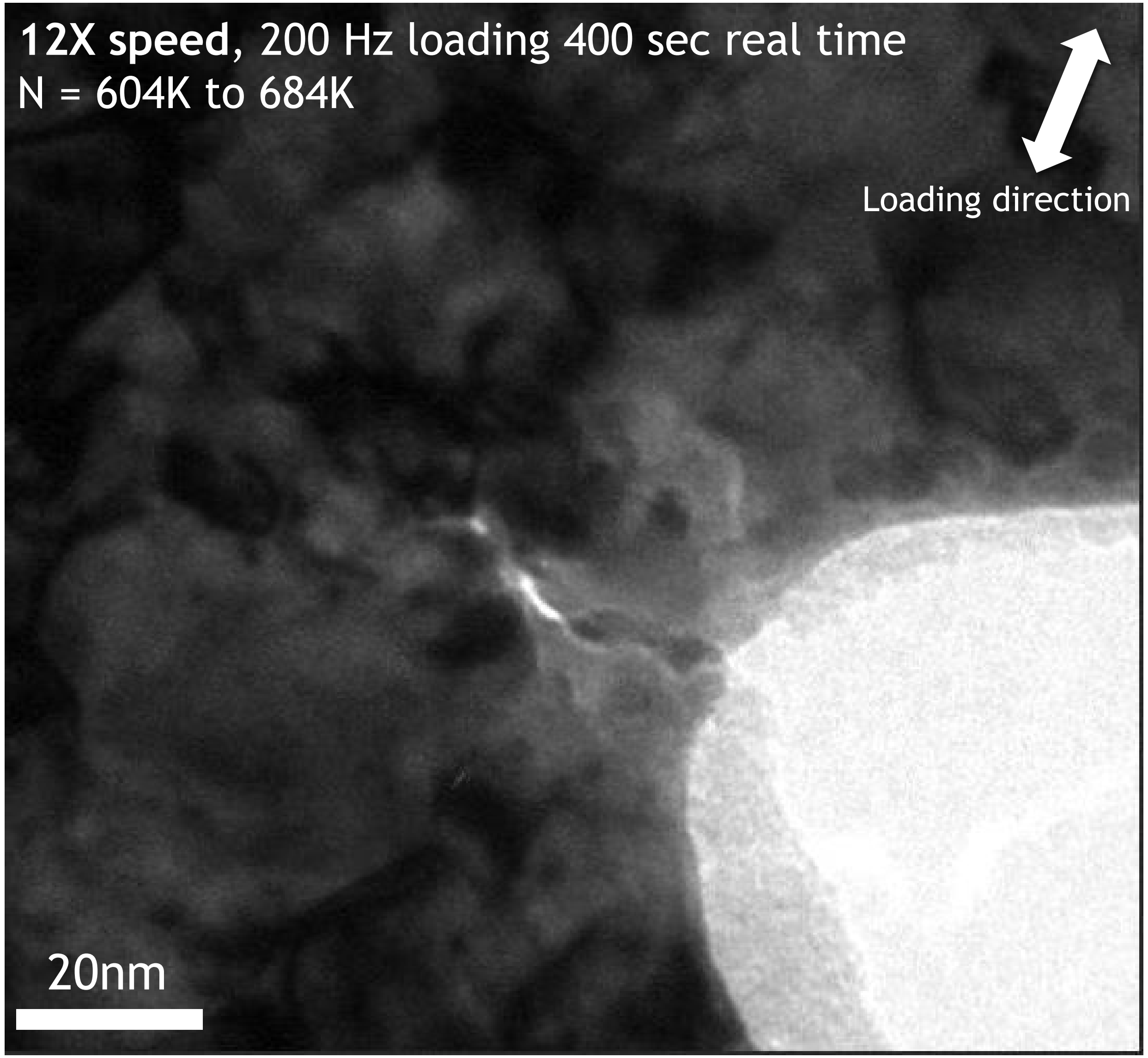
Crack follows two highest Schmid factor slip systems in G2

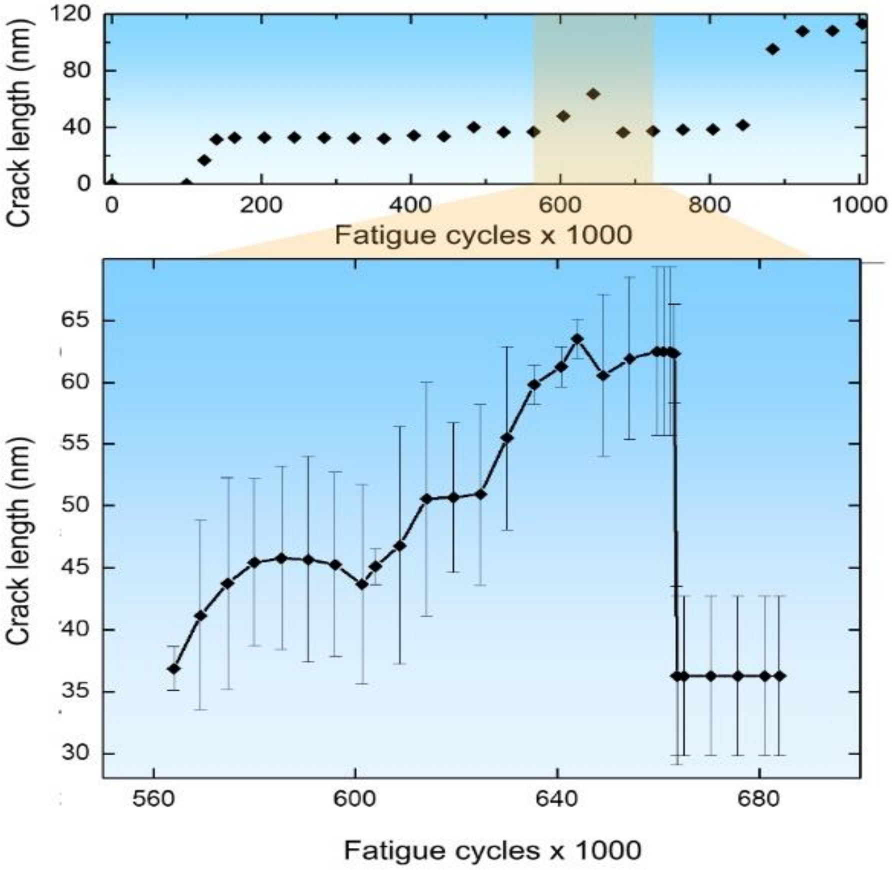
Near Mode I propagation:
(-1 1 -1)<1 1 0>, m = 0.420

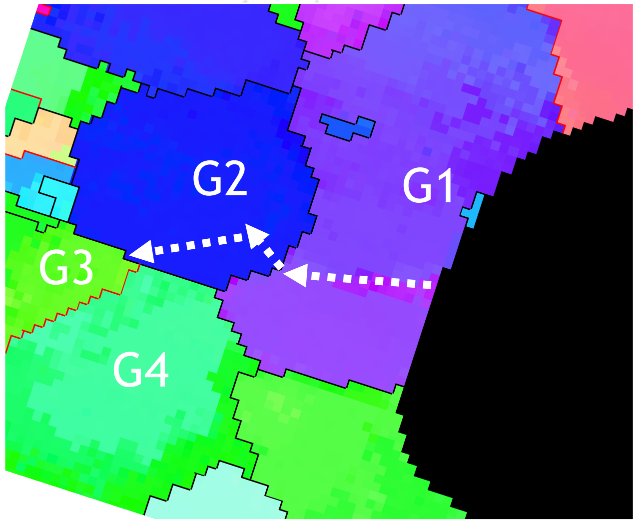
Deflected away from Mode I on:
(-1 -1 1)<1 -1 0), m = 0.408



Crack Healing: In-situ TEM Fatigue

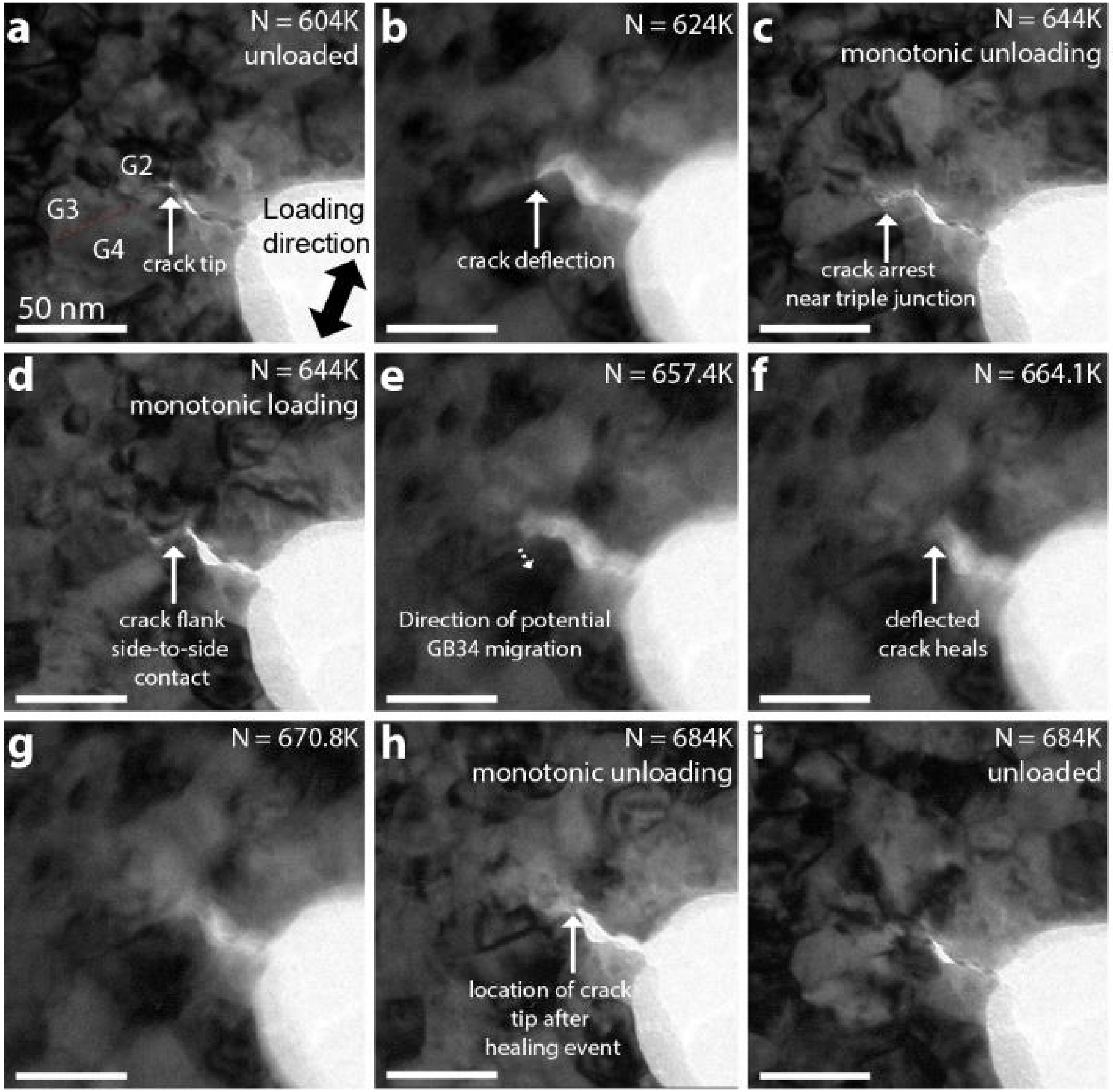






crack heals;
negative crack
growth rate

Image Sequence: Crack Healing

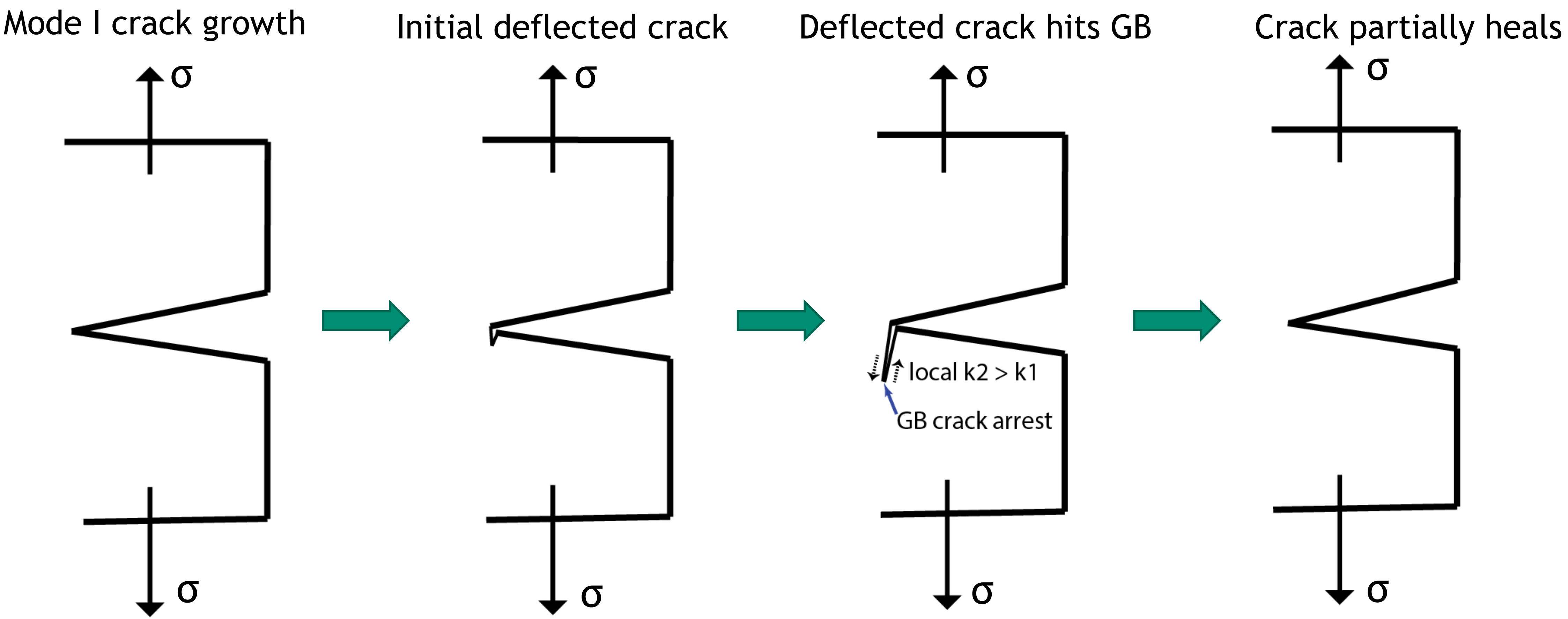


- Crack tip observes mixed mode loading: high and local shear during test
- Roughness-induced crack closure observed on loading and loading (side-to-side contact)
- Surface asperities on crack flank implies atomic fresh surfaces created and destroyed during cyclic loading
- Contrast changes after healing 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



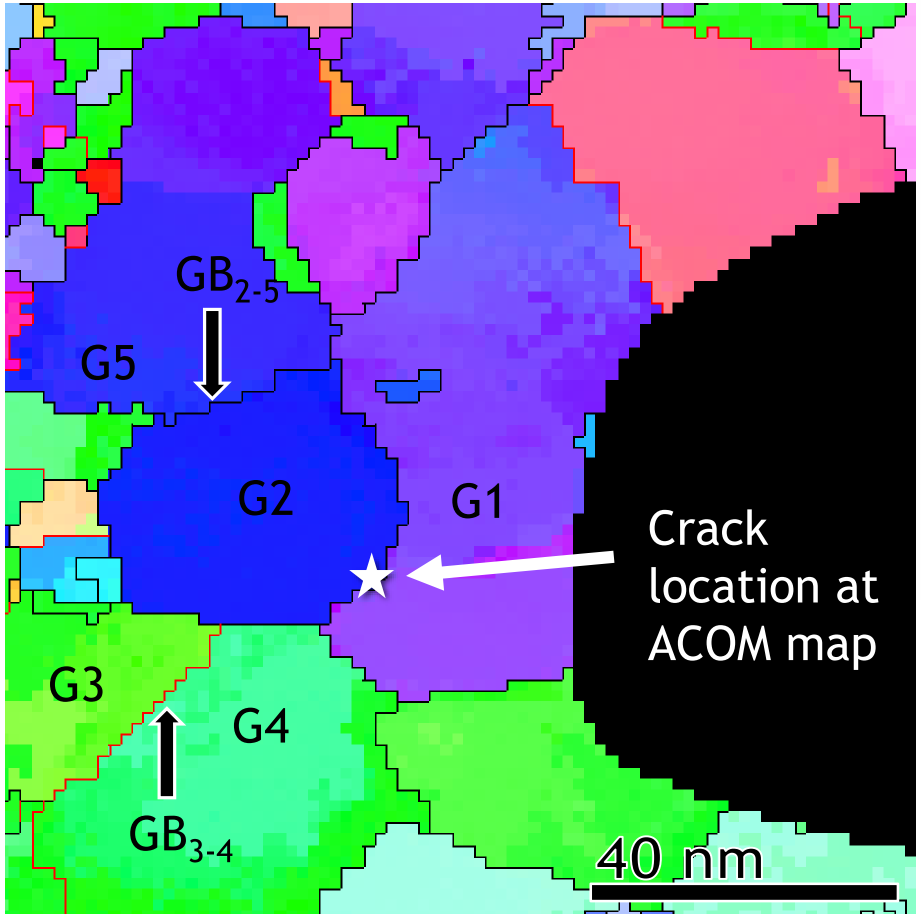
Key aspects of proposed crack healing:

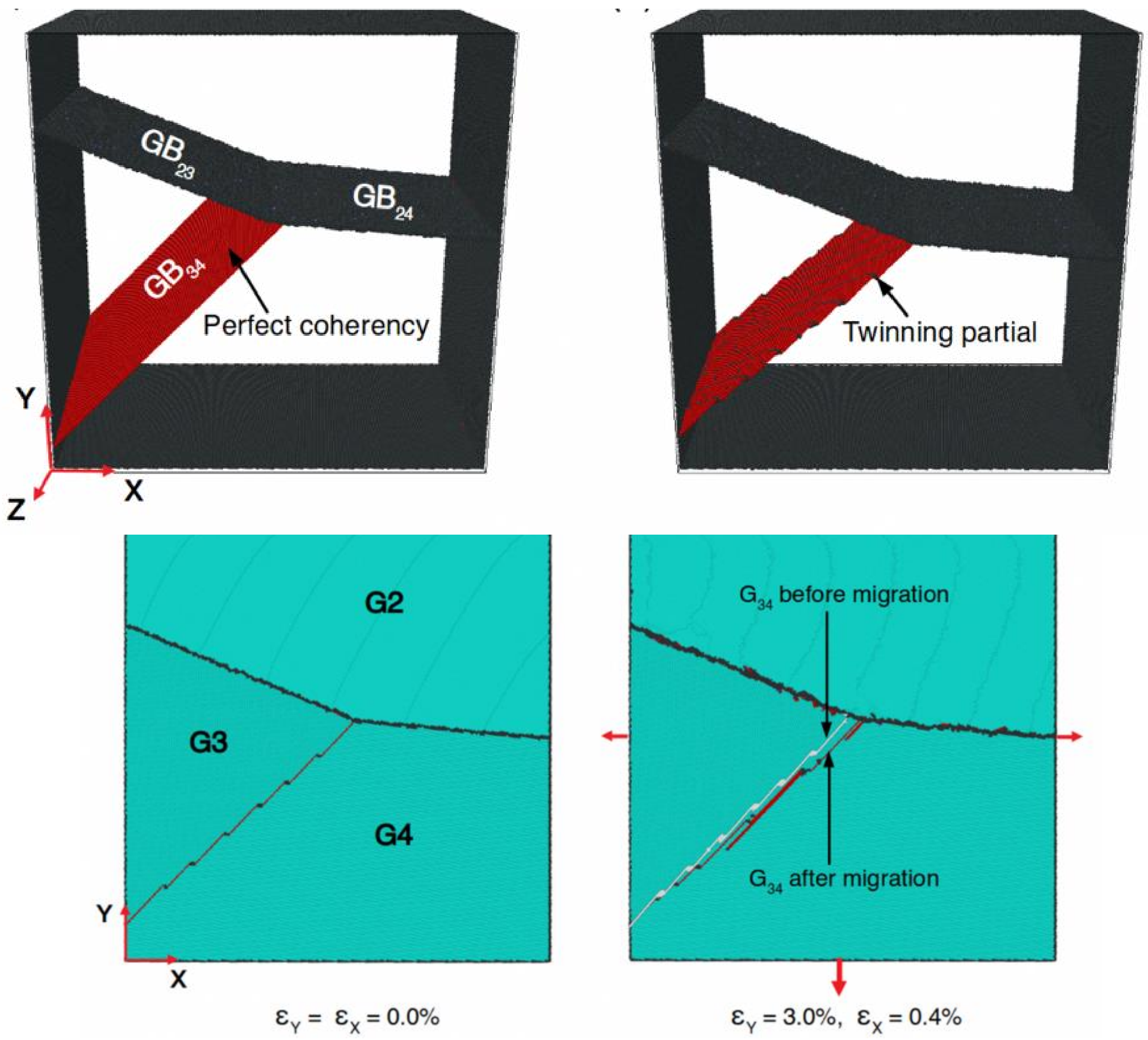
(1) small deflected crack tip opening

(2) roughness induced closure → crack flank contact

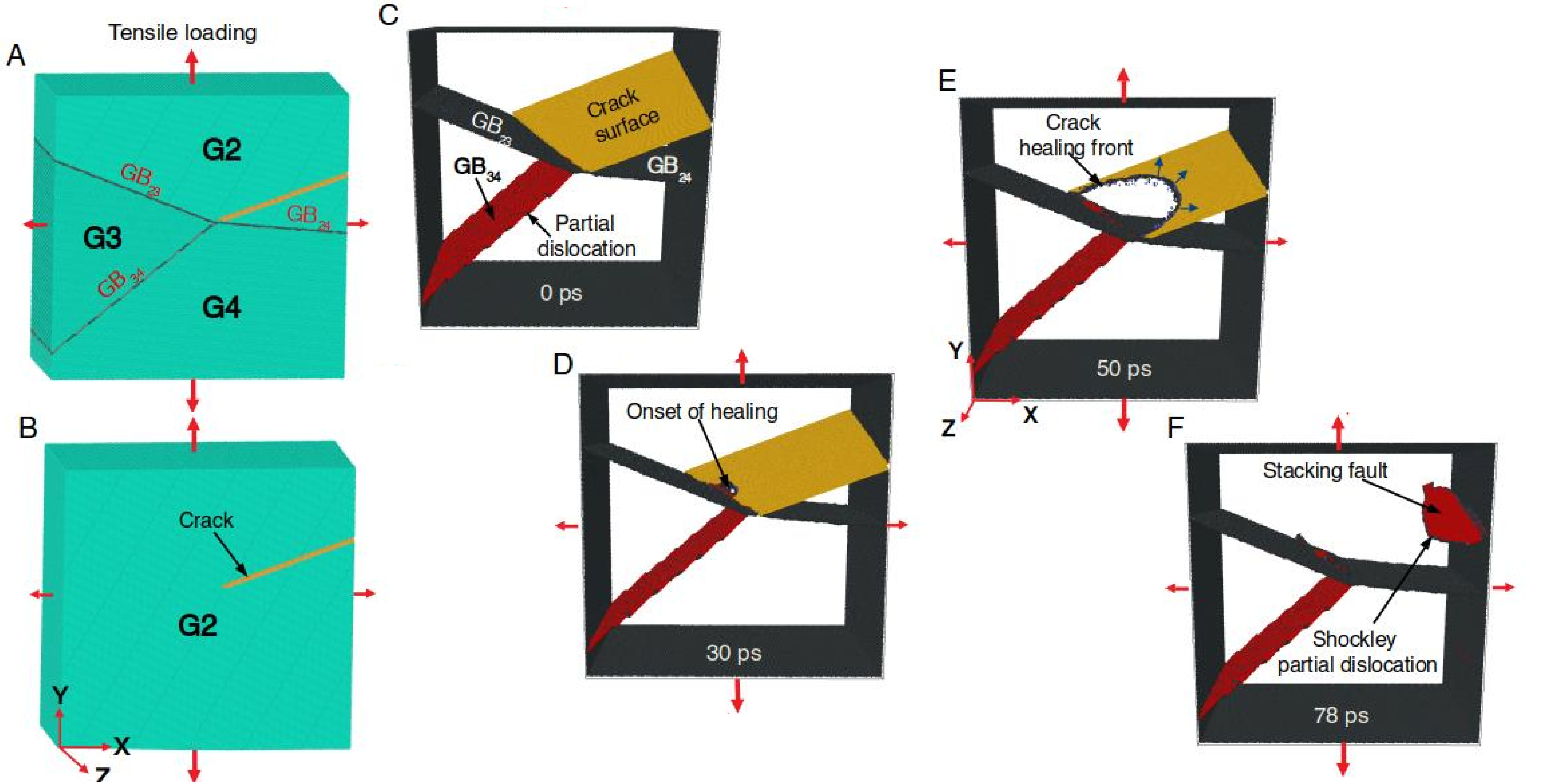
(3) Occurs in single grain – crack flanks share similar crystallography

Developing MD simulations from Reality via ACOM

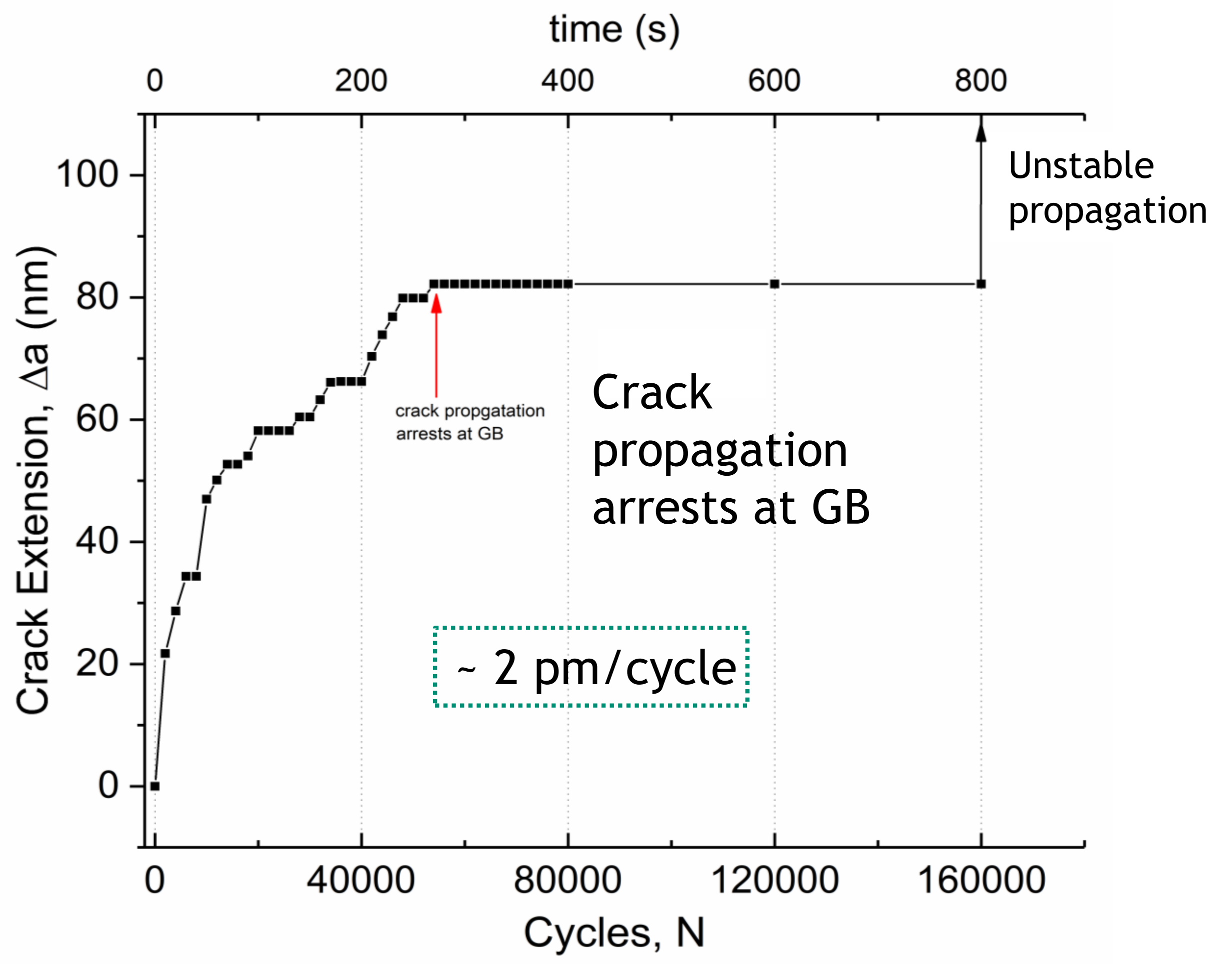


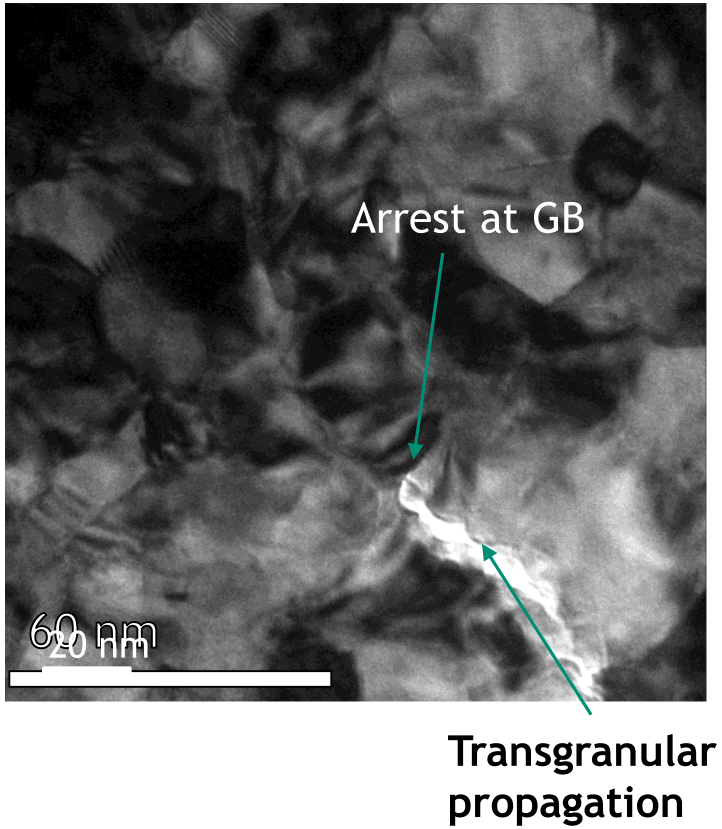


Elucidating Delayed Crack Healing



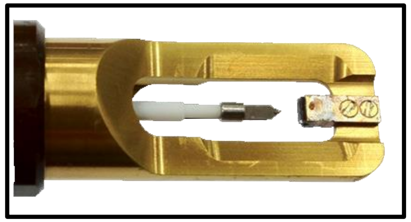
After Crack Healing: Transgranular Propagation Along Mode I





- 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



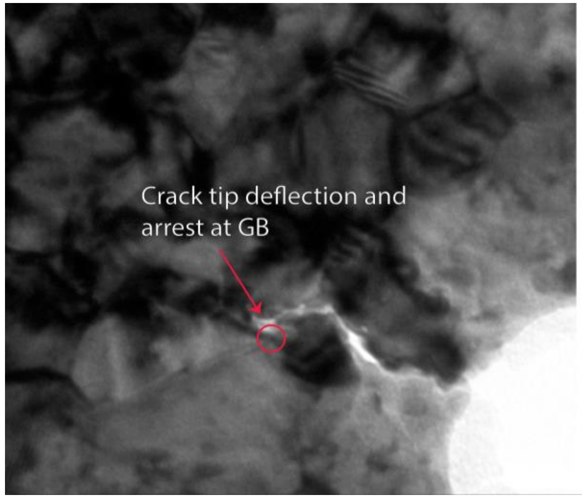
A few "first" TEM observations:

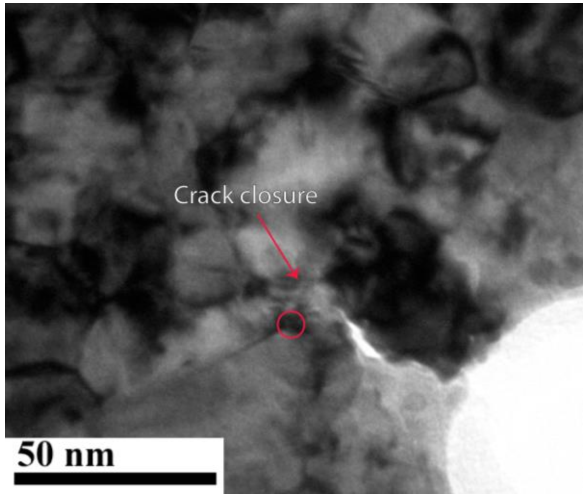
(1) Roughness induced crack closure – unlike coarse grain, deflected grain is arrested at GB

- 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!

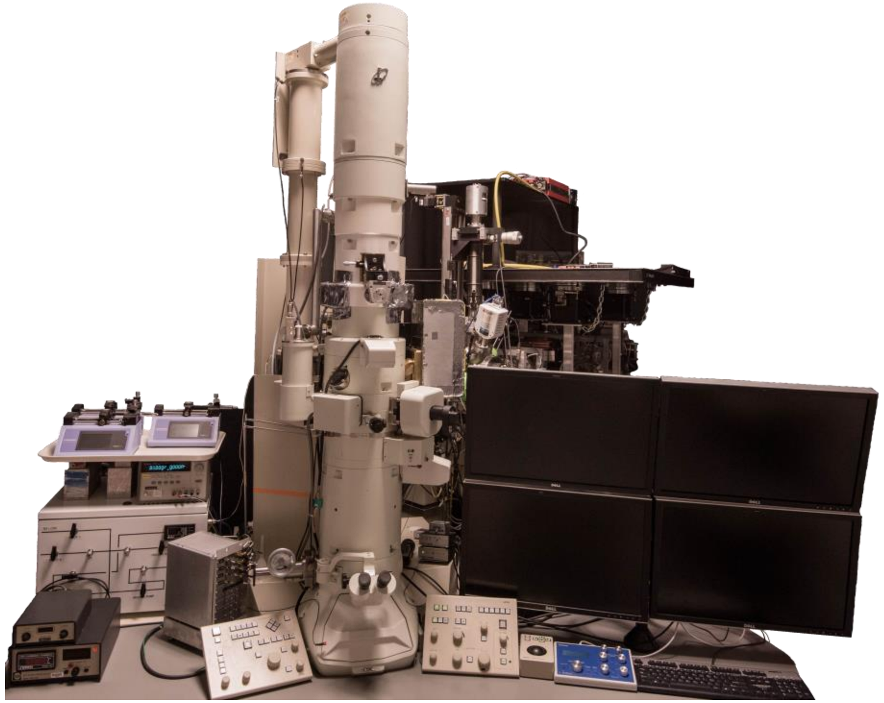
- Feasible in deflected cracks with shear and compressive components
- Vacuum and noble metal, fresh atomic surfaces
- Single grain – consistent with J. Lou orientated attachment crack healing mechanism in nanowires

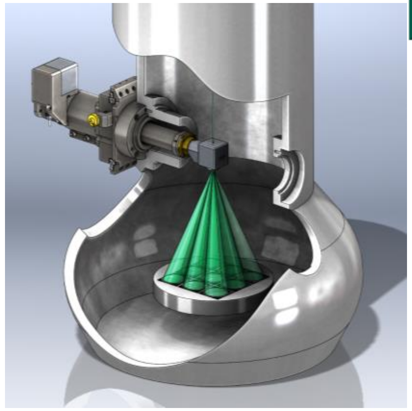




New implications for understanding nanoscale fatigue mechanism in NC metals through in-situ TEM

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

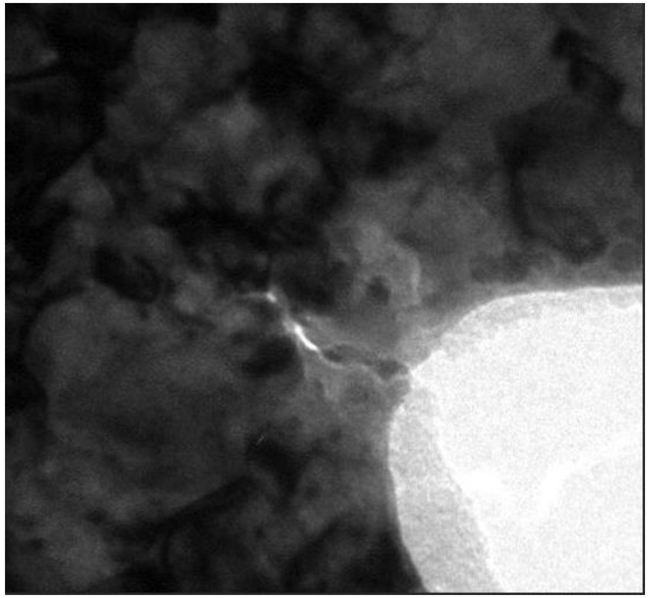


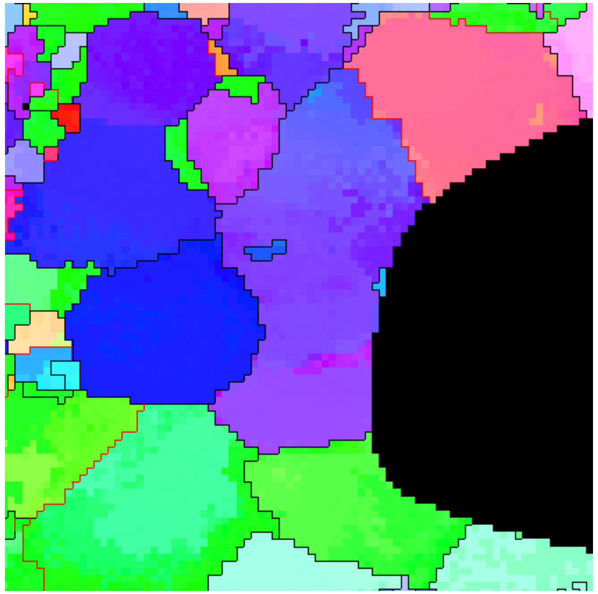


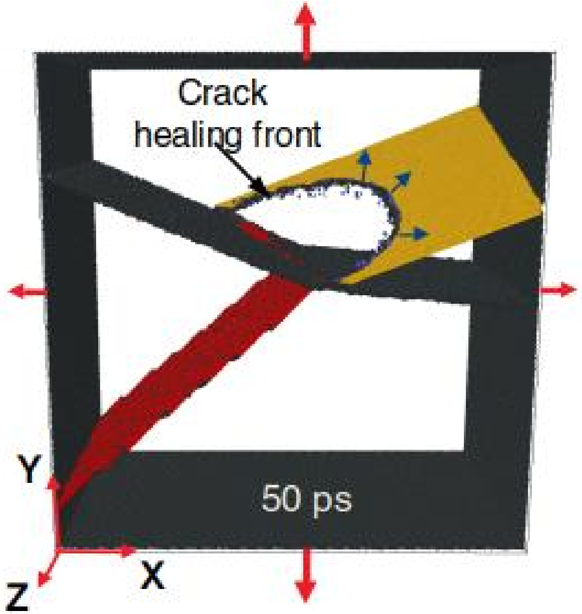
- Under development capabilities:
  - High temporal camera capabilities – remove motion blur → actual dislocation migration during loading
  - Coupling fatigue with other extreme environments:
    - Laser heating to 2100 ˚C
    - Electrical biasing
    - Ion Irradiation

Combining precision of TEM tension-tension fatigue with harsh environments capable in Sandia's In-situ Ion Irradiation TEM a wealth of previously impossible experiments are now feasible!

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







High-cycle Fatigue Experiments at the Nanometer Scale is Now Possible!

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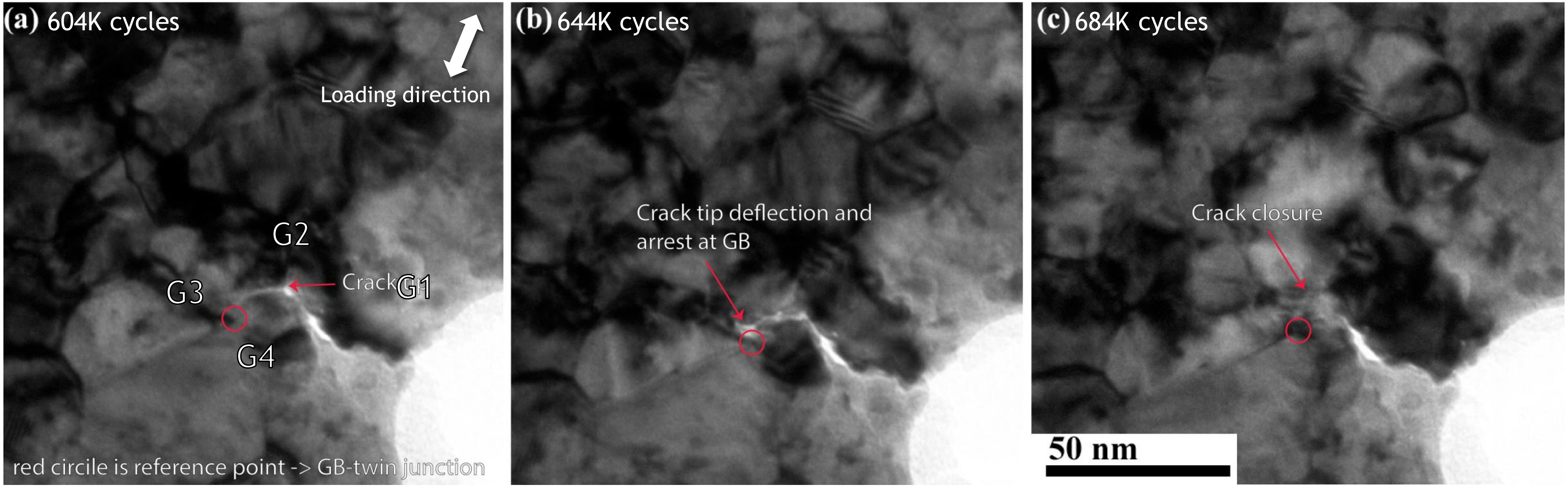
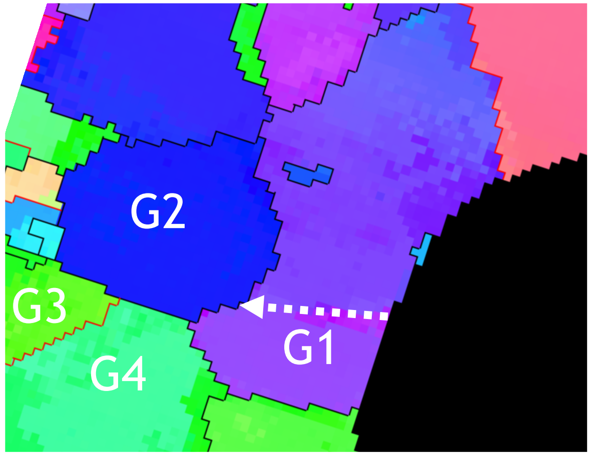
Thanks!

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!

Crack Healing Observations in Copper