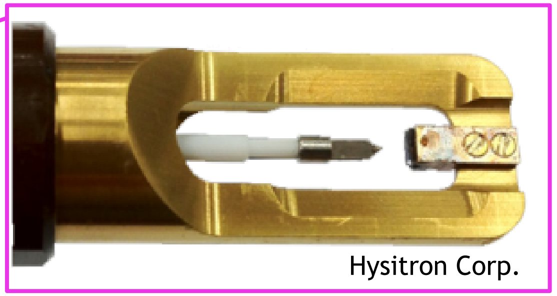
SAND2022-3860C

Fatigue-crack Healing in Pure Nanocrystalline Pt Enabled by Boundary Evolution

Chris Barr, Ta Duong, Dan Bufford, Nathan Heckman, Michael Demkowicz, Khalid Hattar, Brad Boyce

Sandia National Labs; Texas A&M

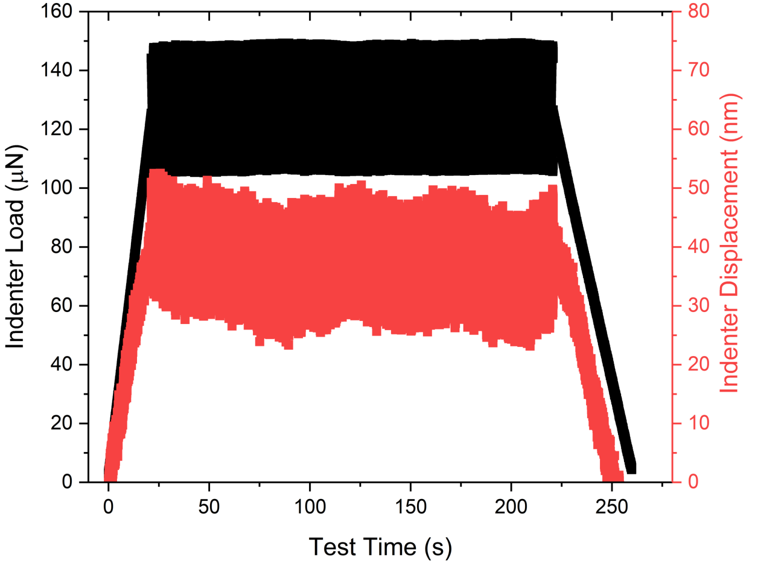
High-cycle fatigue in the TEM



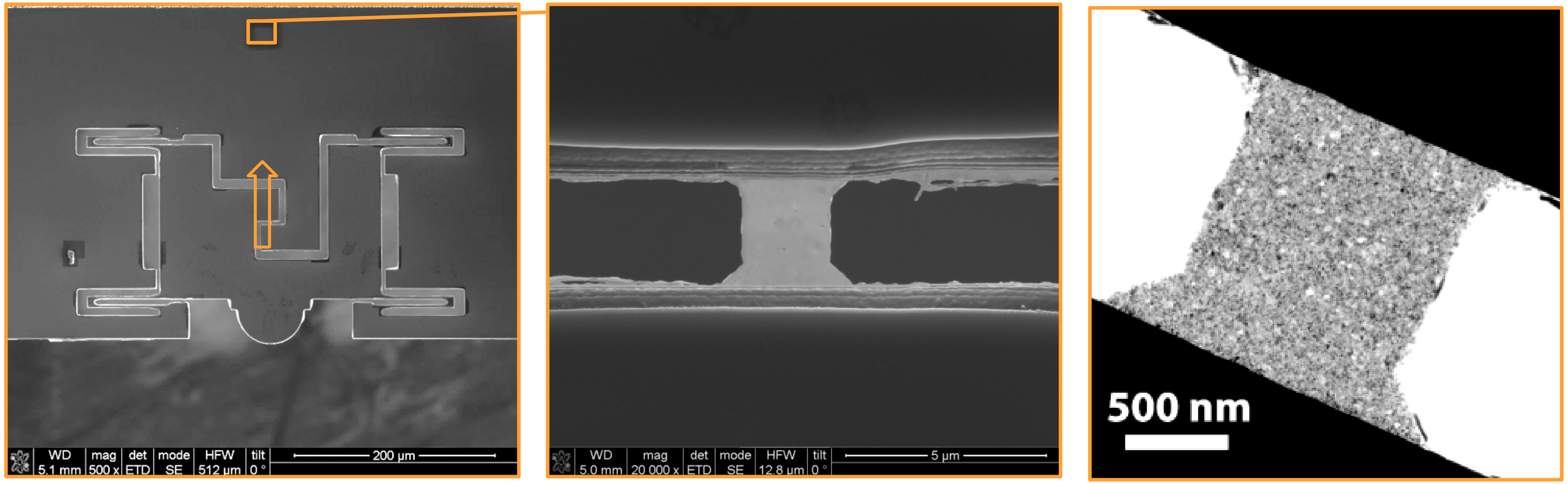
Hysitron PI95 *In Situ* Nanoindentation TEM Holder

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

Fatigue frequencies up to ~300 Hz

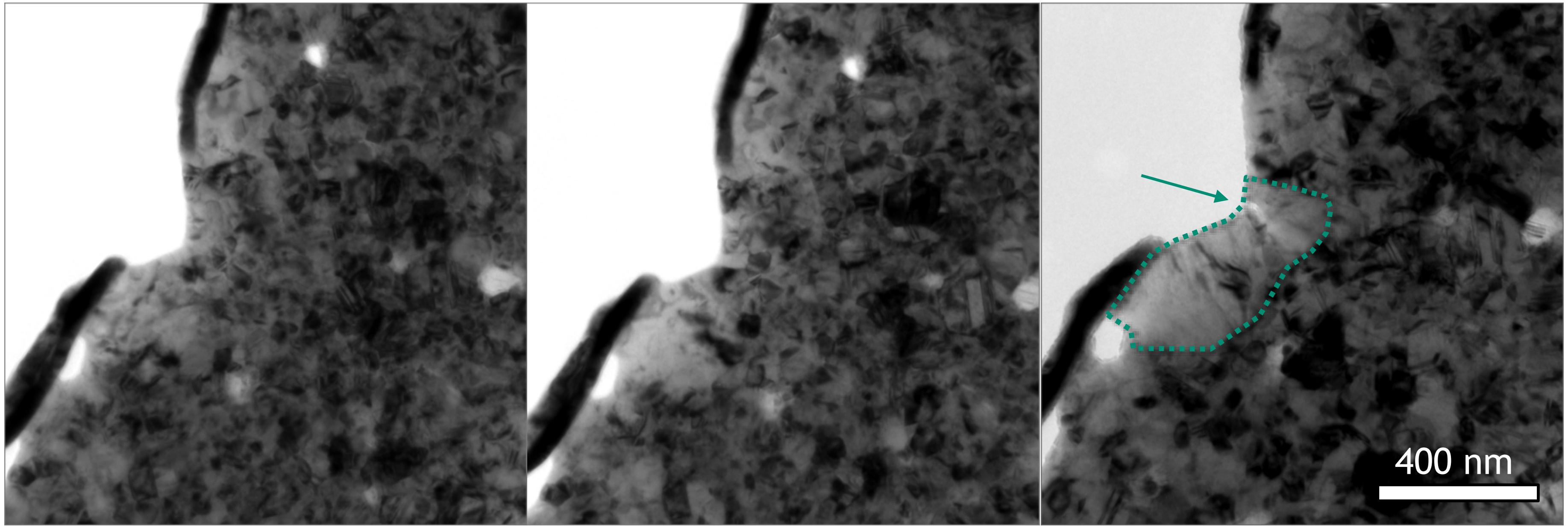


60,000 cycles in 3 minutes



- Nearly pure tension, uniform cross sectional area, stable load frame
- Fragile, sensitive to shape of edges, issues with magnetic materials

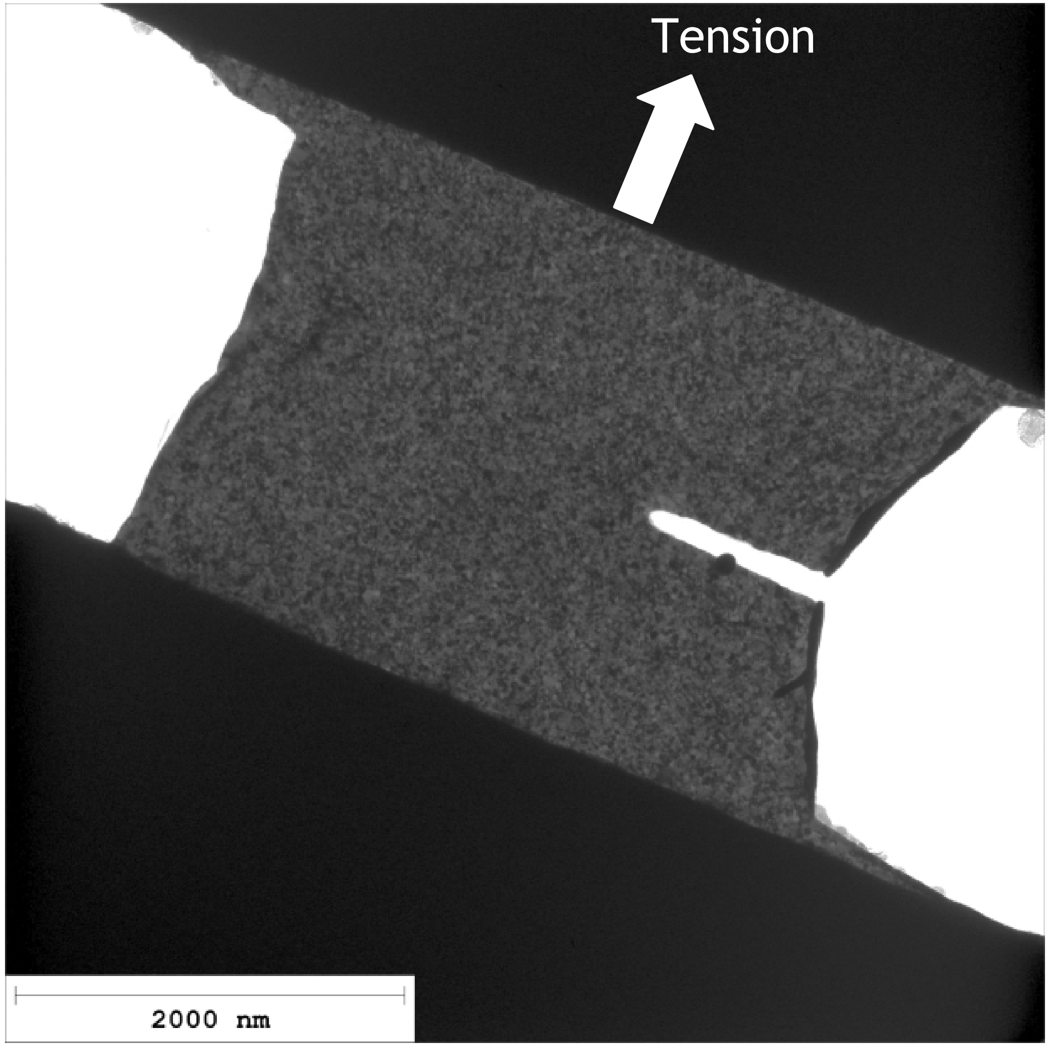
An example of prior observations in pure nanocrystalline Cu

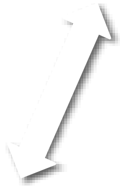


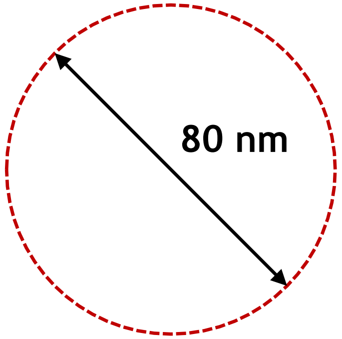
0 cycles 180,000 cycles 840,000 cycles

Bufford et al, “High cycle fatigue in the transmission electron microscope” *Nano Letters*, 2016

Direct TEM observation: In-situ cyclic loading of nanocrystalline Pt

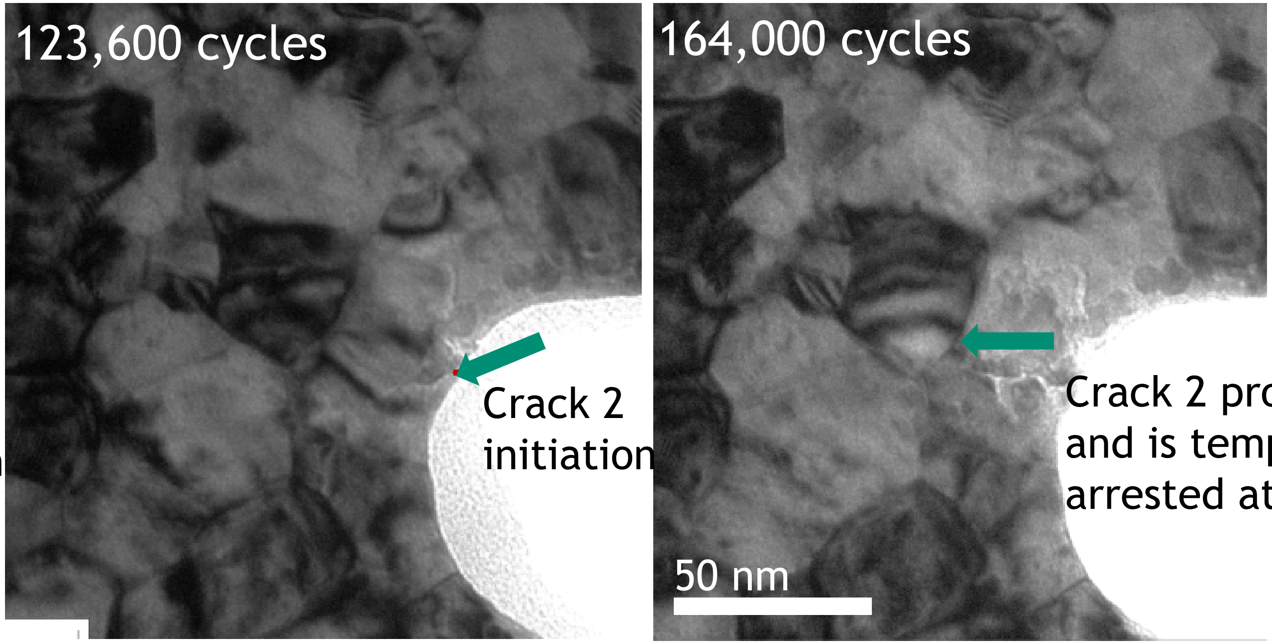






Crack initiation and early growth

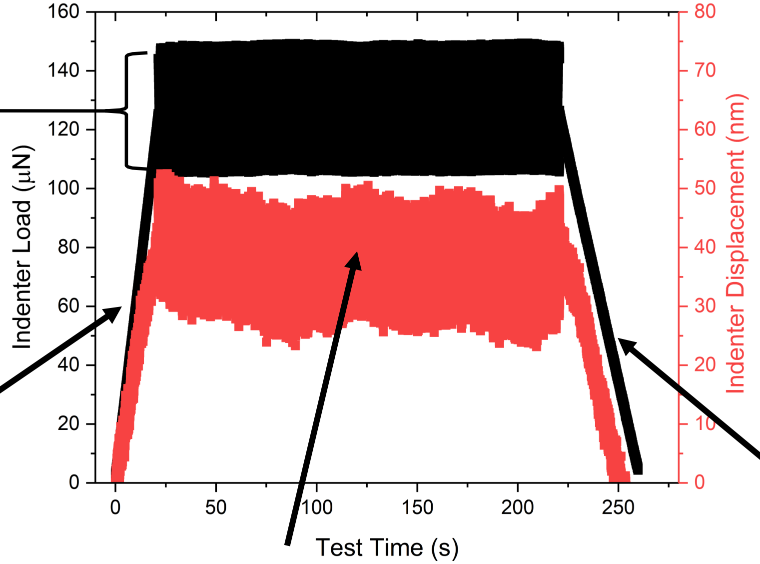
P_m = 80 uN
P_{amp} = 30 uN



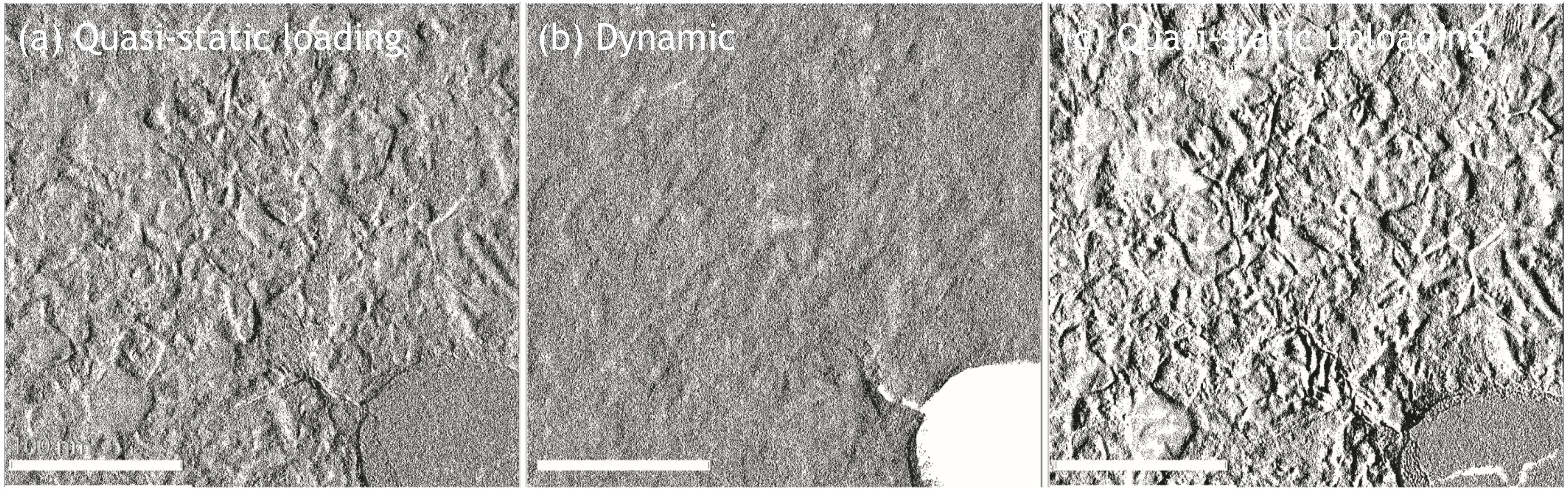
- Crack initiation
- Second crack initiates at ~ 90° to first crack, both 45° to notch tip normal
- Transgranular crack propagates until reaching initial grain boundary (25.7° [13 2 8] misorientation) and is subsequently arrested for over 250,000 additional cycles

Cyclic Loading in TEM Protocol

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

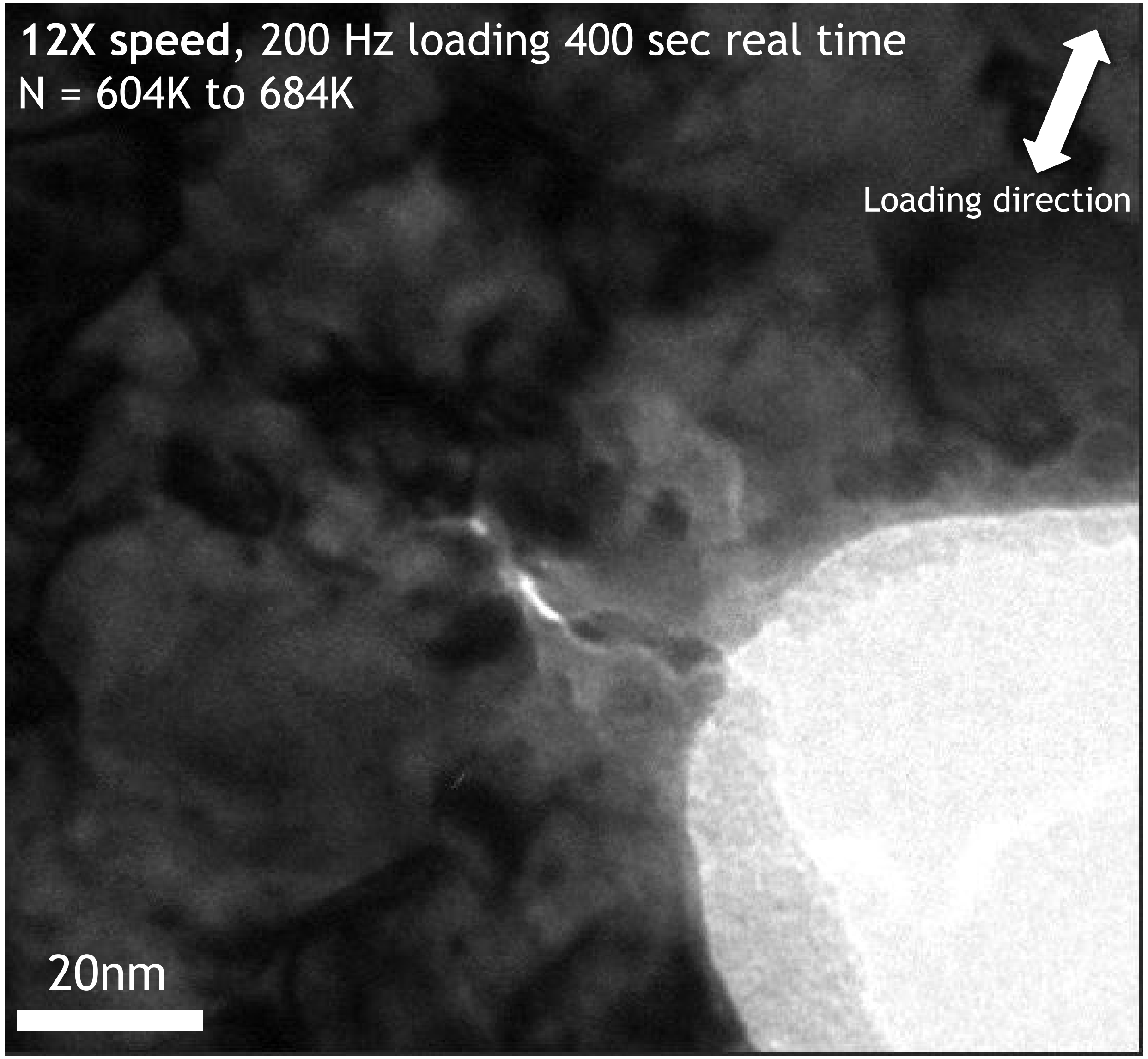


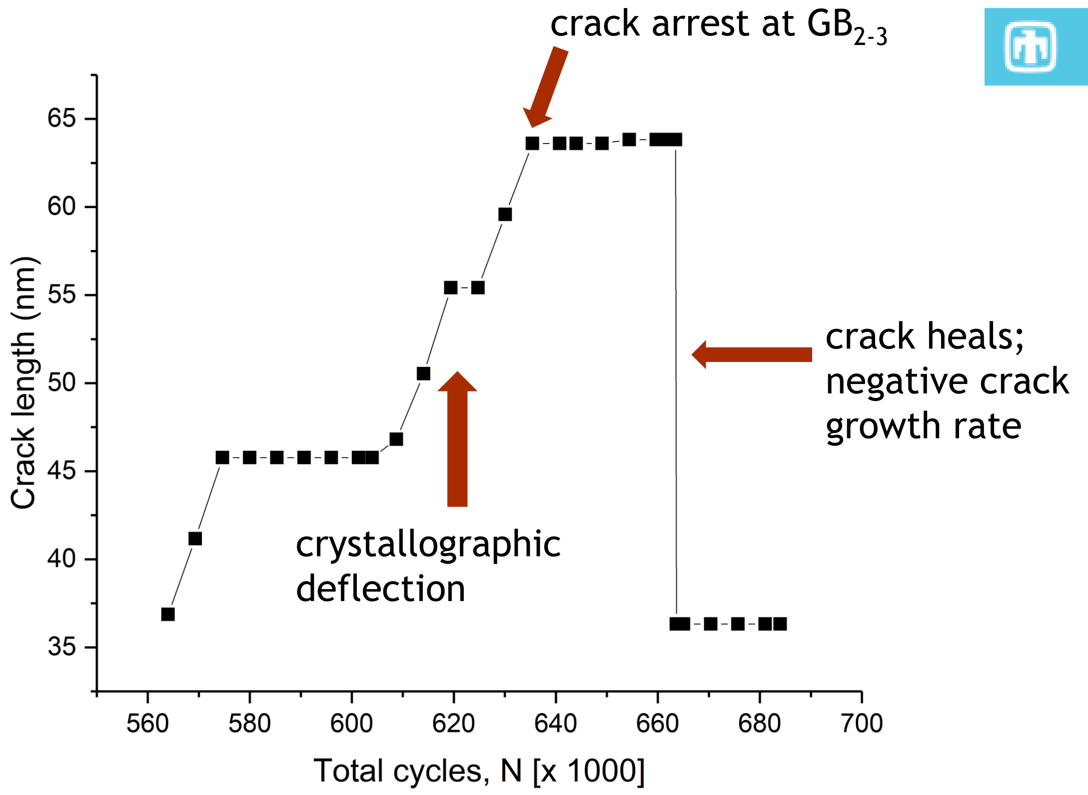
- **200 Hz Frequency**
- **40,000 cycles in 200s**

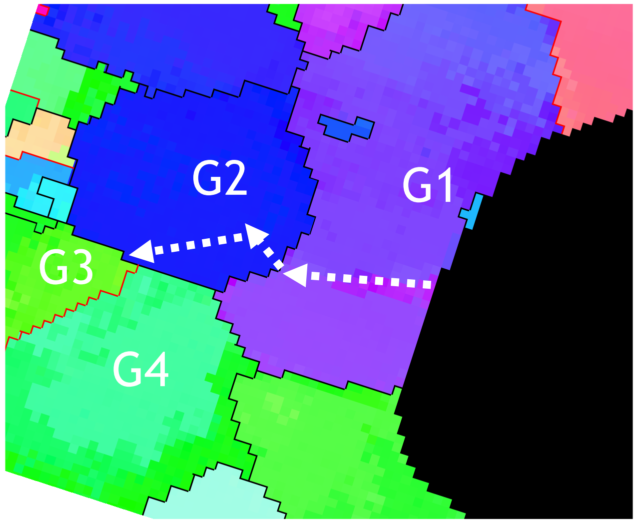


- Motion blur → loading frequency exceeded the frame rate (15 frames/s →**13 cycles per frame**)

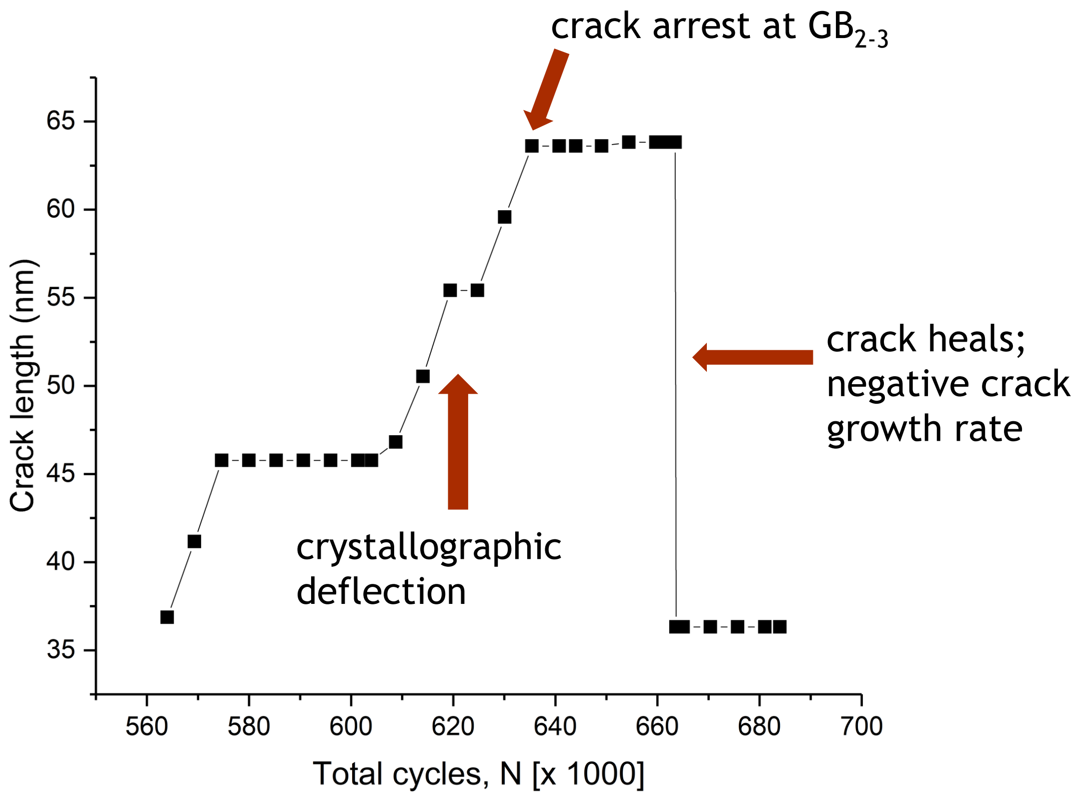
Crack Healing: In-situ TEM Fatigue

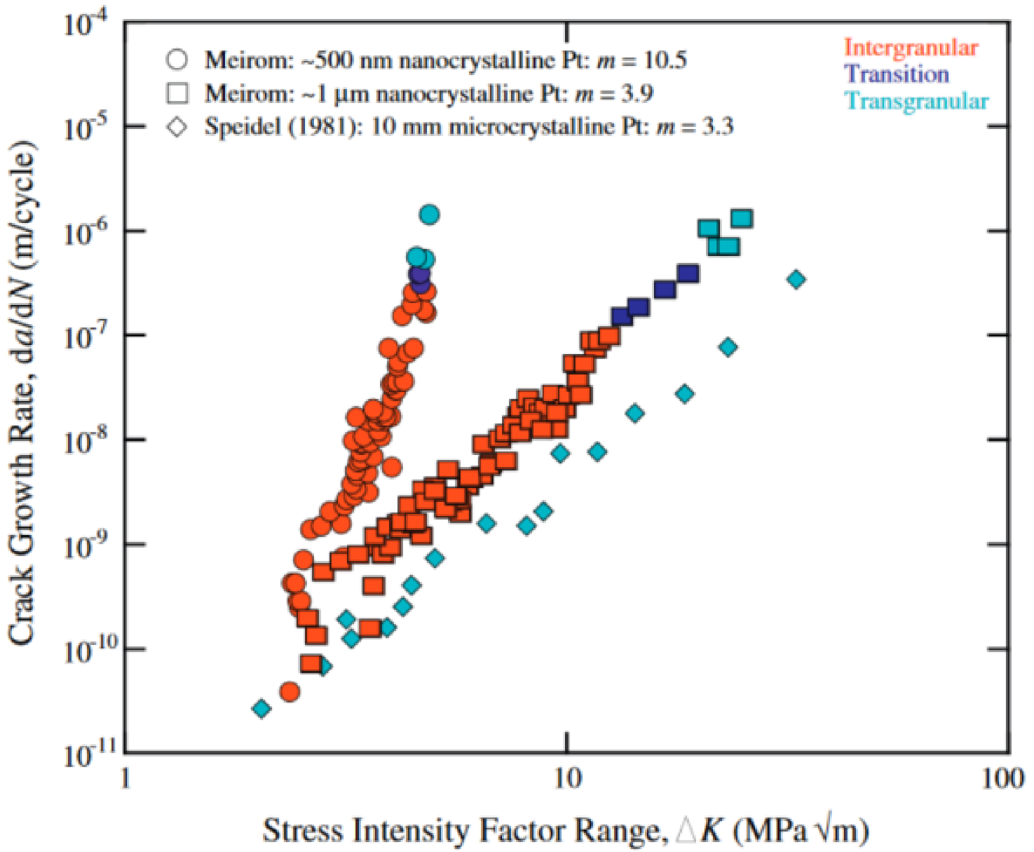






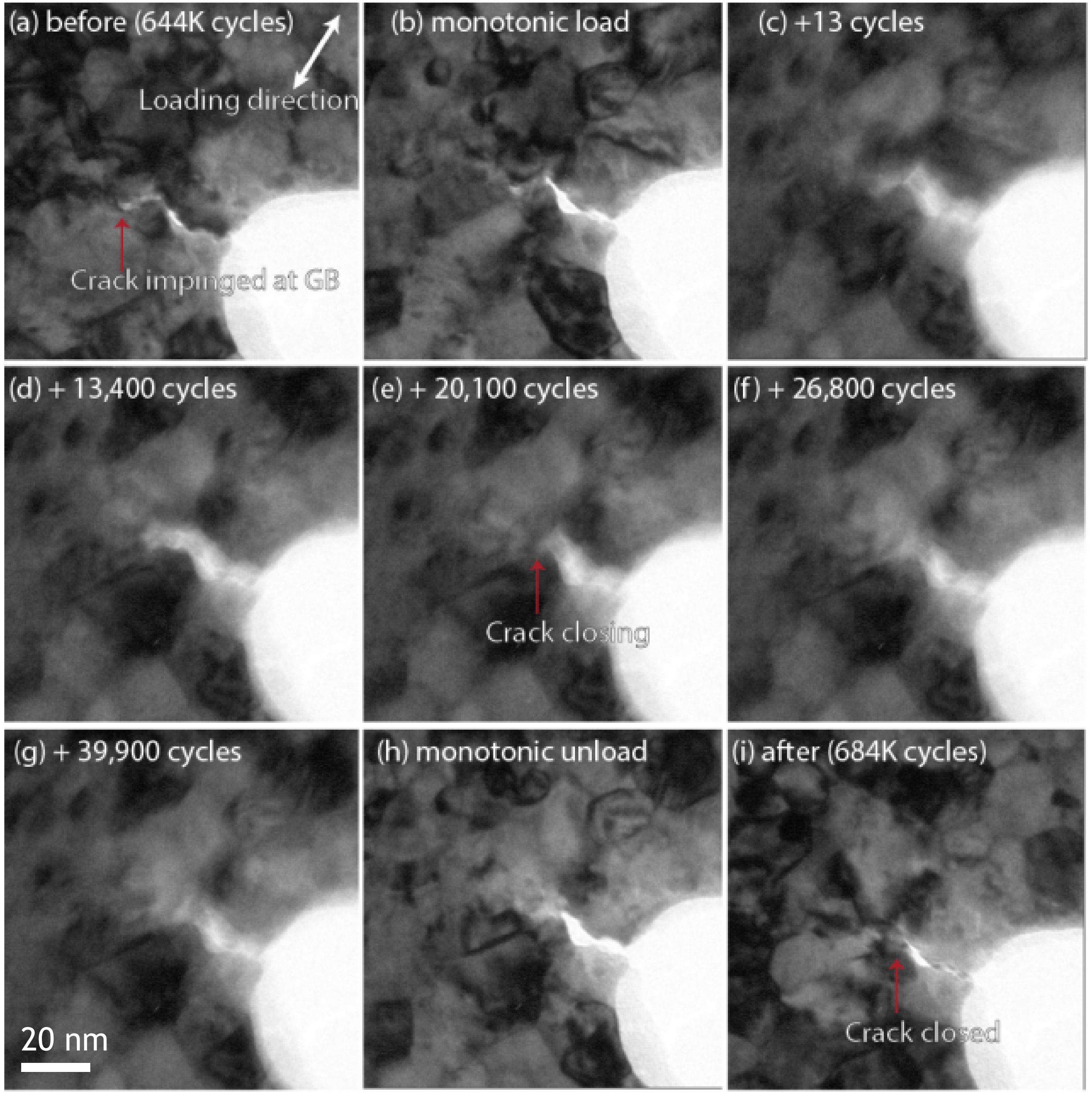
Negative crack growth rates: an impossibility?!





Mierom, Clark, and Muhlstein, Acta Materialia, 2012

Image Sequence: Crack Healing

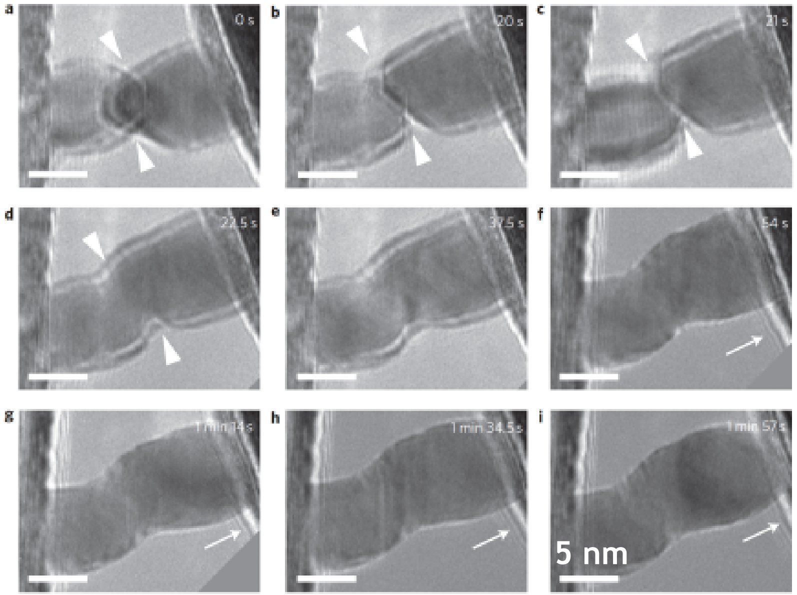


- Crack tip observes mixed mode loading: high and local shear during test
- Roughness-induced crack closure observed on loading and loading (side-to-side contact)
- Surface asperities on crack flank implies atomic fresh surfaces created and destroyed during cyclic loading
- Contrast changes after healing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

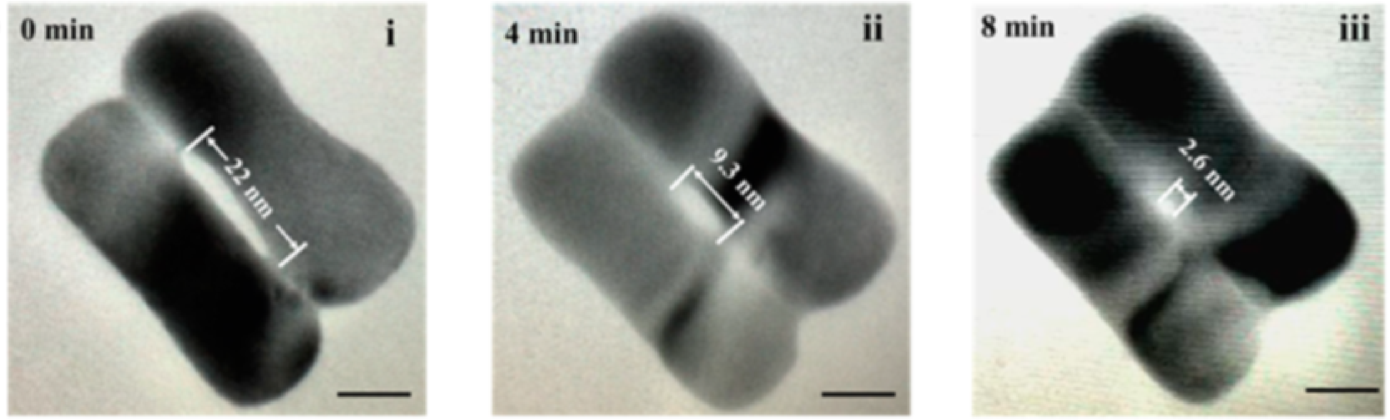
Cold Welding?
✓ Orientation the same on both sides of crack
✓ Vacuum, noble metal
✓ Atomically fresh surfaces

Cold welding …

Gold nanorods



Gold nanodumbells



1. **G. Dai, B. Wang, S. Xu, Y. Lu, Y. Shen, Side-to-Side Cold Welding for Controllable Nanogap Formation from "Dumbbell" Ultrathin Gold Nanorods. *ACS Appl Mater Interfaces* 8, (2016).**
2. G. S. Ferguson, M. J. Chaudhury, G. B. Sigal, G. M. Whitesides, Contact Adhesion of Thin Gold Films on Elastomeric Supports: Cold Welding Under Ambient Conditions. *Science* 253, (1991).
3. **Y. Lu, J. Y. Huang, C. Wang, S. Sun, J. Lou, Cold welding of ultrathin gold nanowires. *Nat Nanotechnol* 5, 218-224 (2010).**
4. D. V. Wagle, G. A. Baker, Cold welding: a phenomenon for spontaneous self-healing and shape genesis at the nanoscale. *Materials Horizons* **2**, 157-167 (2015).

But how did the crack flanks come into contact with each other?

A brief comment on load ratio and crack closure…

CONTACT SHIELDING

— wedging:

corrosion debris-induced crack closure

crack surface roughness-induced closure

— bridging:

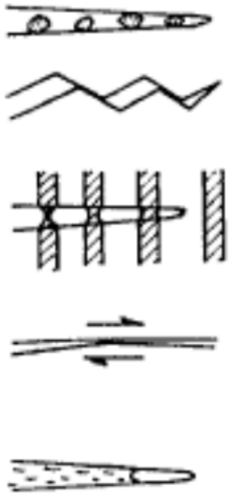
ligament or fiber toughening

— sliding:

sliding crack surface interference

— wedging + bridging:

fluid pressure-induced crack closure



R.O. Ritchie, *Int. J. Fracture*, 1999

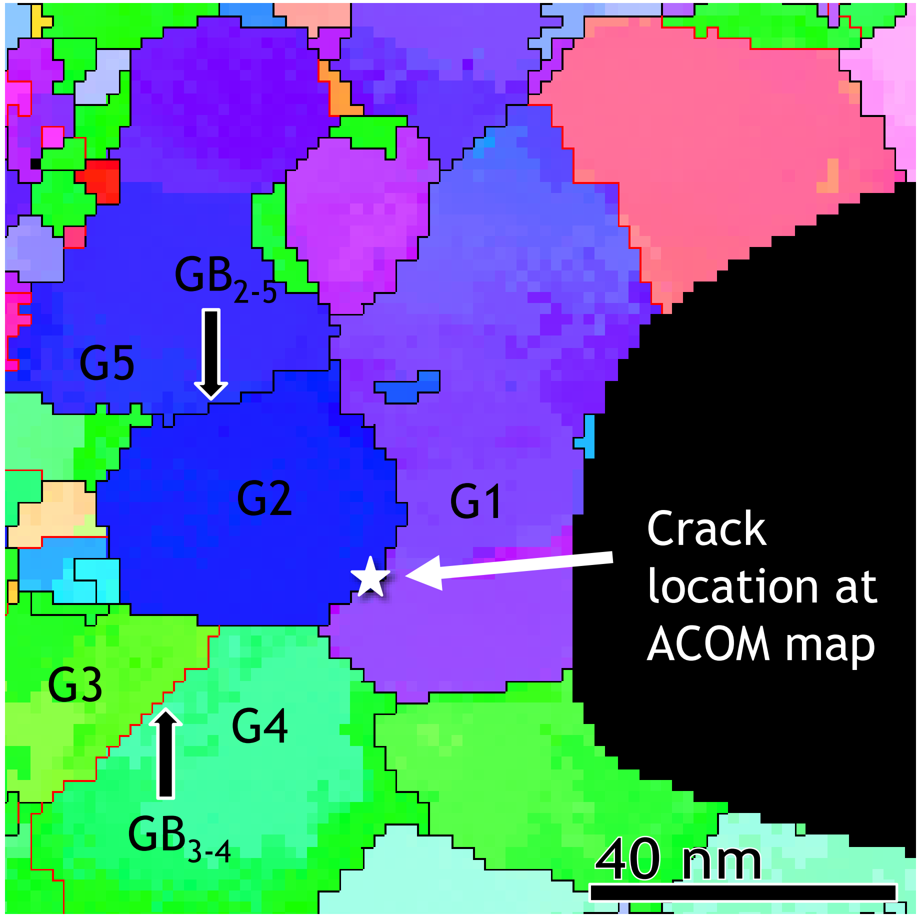
In the current experiment, the far-field applied loads were strongly tensile:

MINIMUM LOAD = +85 μN
MAXIMUM LOAD = +115 μN

Load ratio = 85/115 = 0.74

So… how did the crack flanks come into contact with each other?

Cyclic Loading: Local GB Misorientation Changes

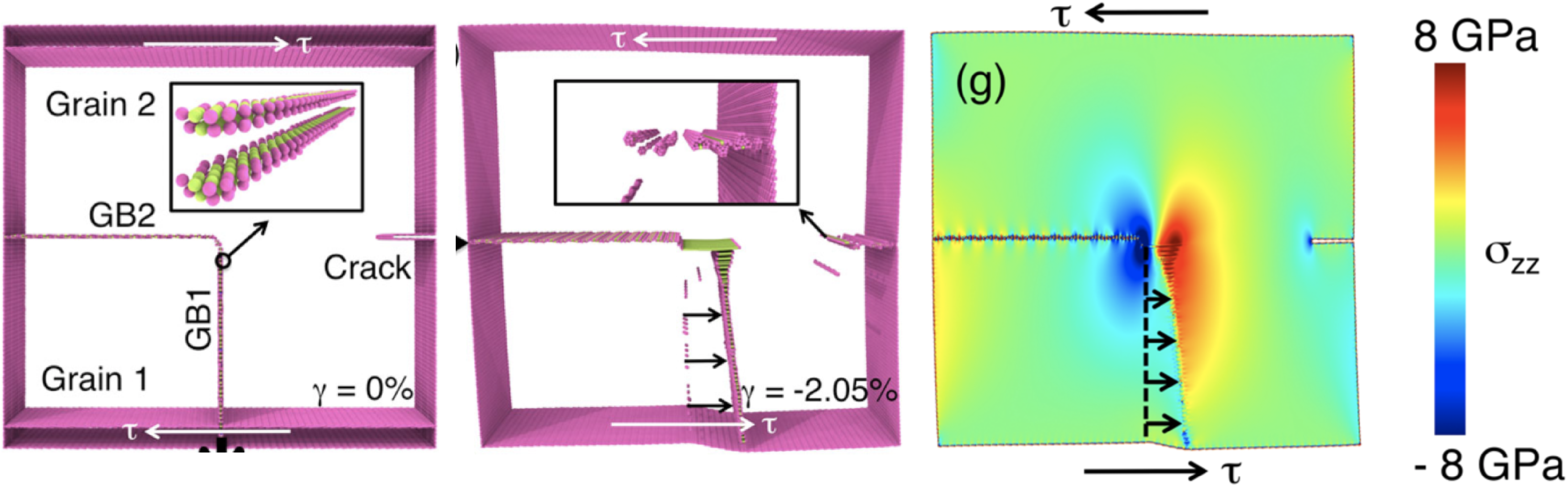


- Deformation from cyclic loading and crack transgranular crack propagation → clear change in relative GB misorientation
- **Likely active mechanisms:**
  - Grain rotation and associated dislocation-GB impingement associated with dislocation activity ahead of fatigue notch and crack path

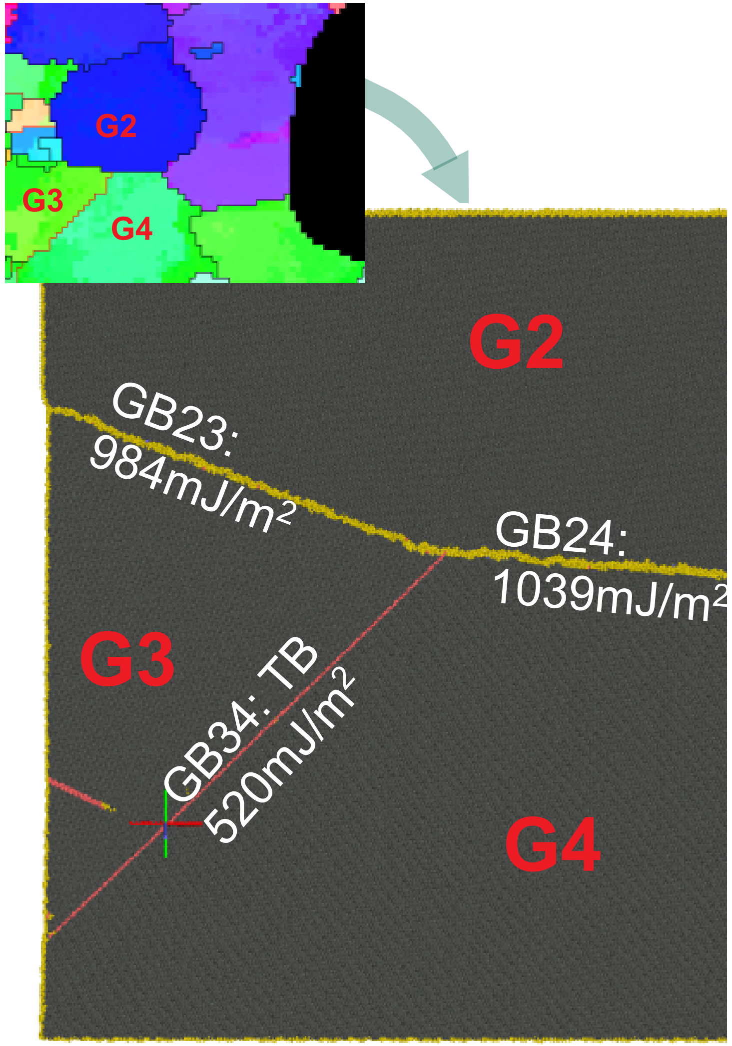
	As-deposited	After pre-cracking
GB_{3-4}	59.3° [1 1 1]; Σ3 Dev. 0.9°	56.9° [7 7 6]; Σ3 Dev. 4.9°

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

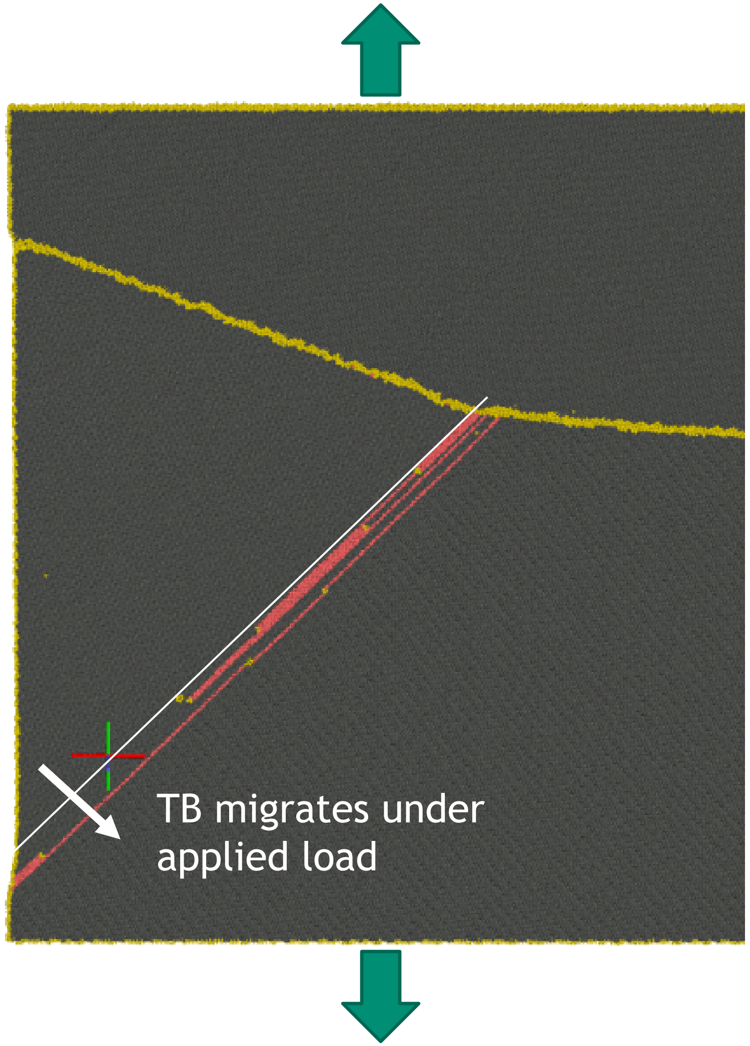
Toy model of crack healing from the perspective of Xu & Demkowicz (*PRL*, 2013)



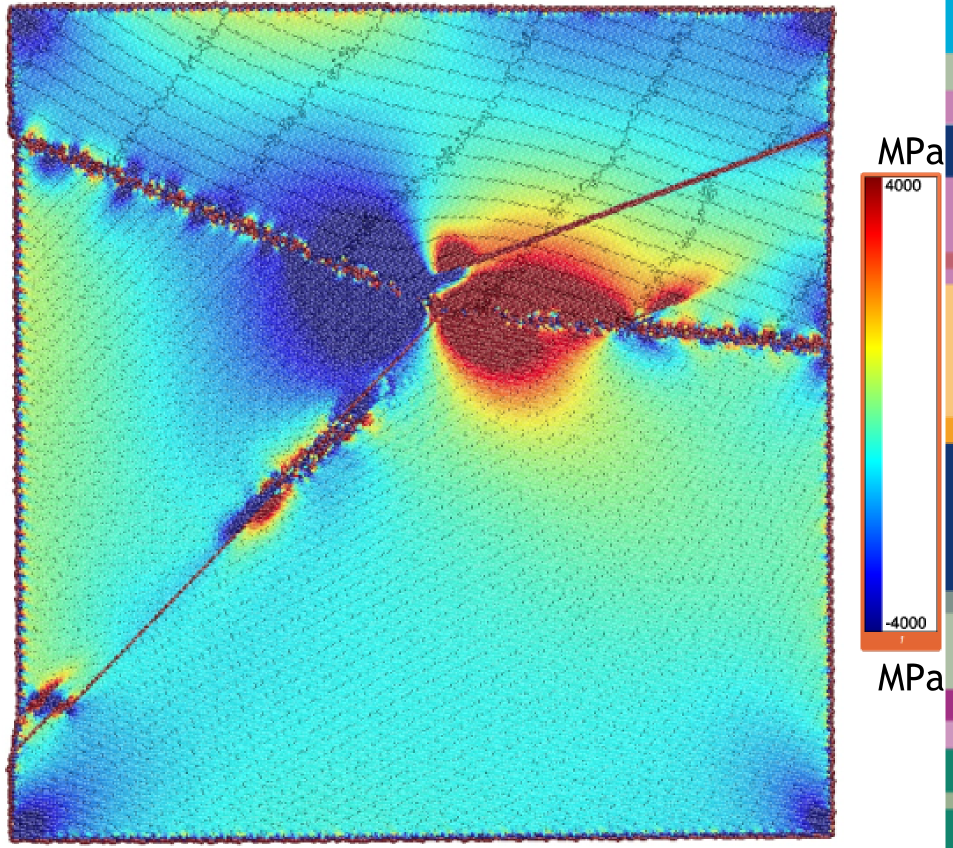
Strain-induced boundary migration in the absence of a crack…



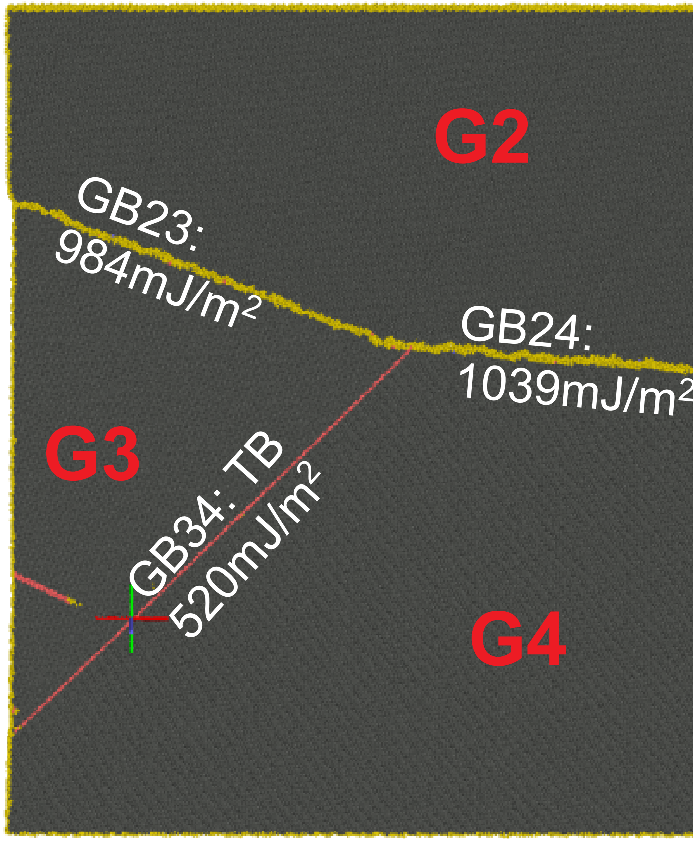
Model based on actual experimental configuration

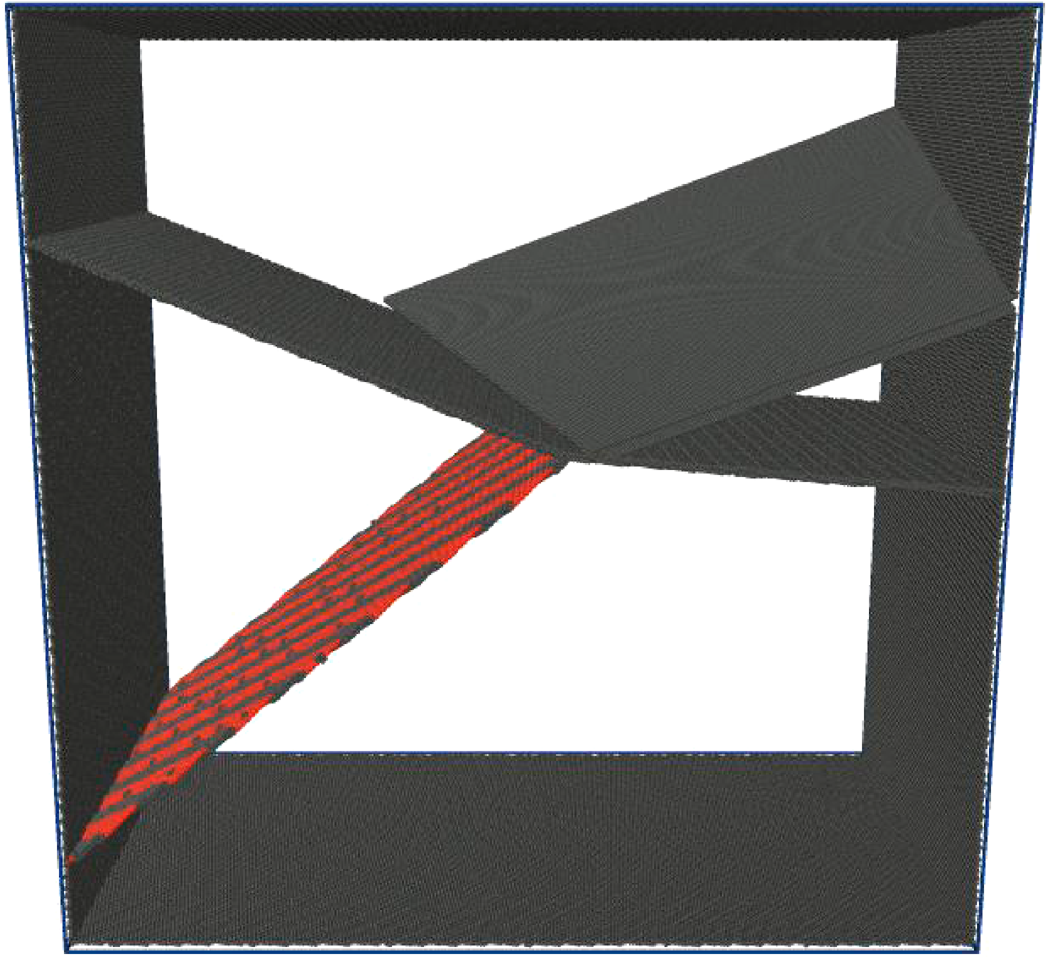


Stress due to TB migration



Crack healing in the presence of grain boundary migration…



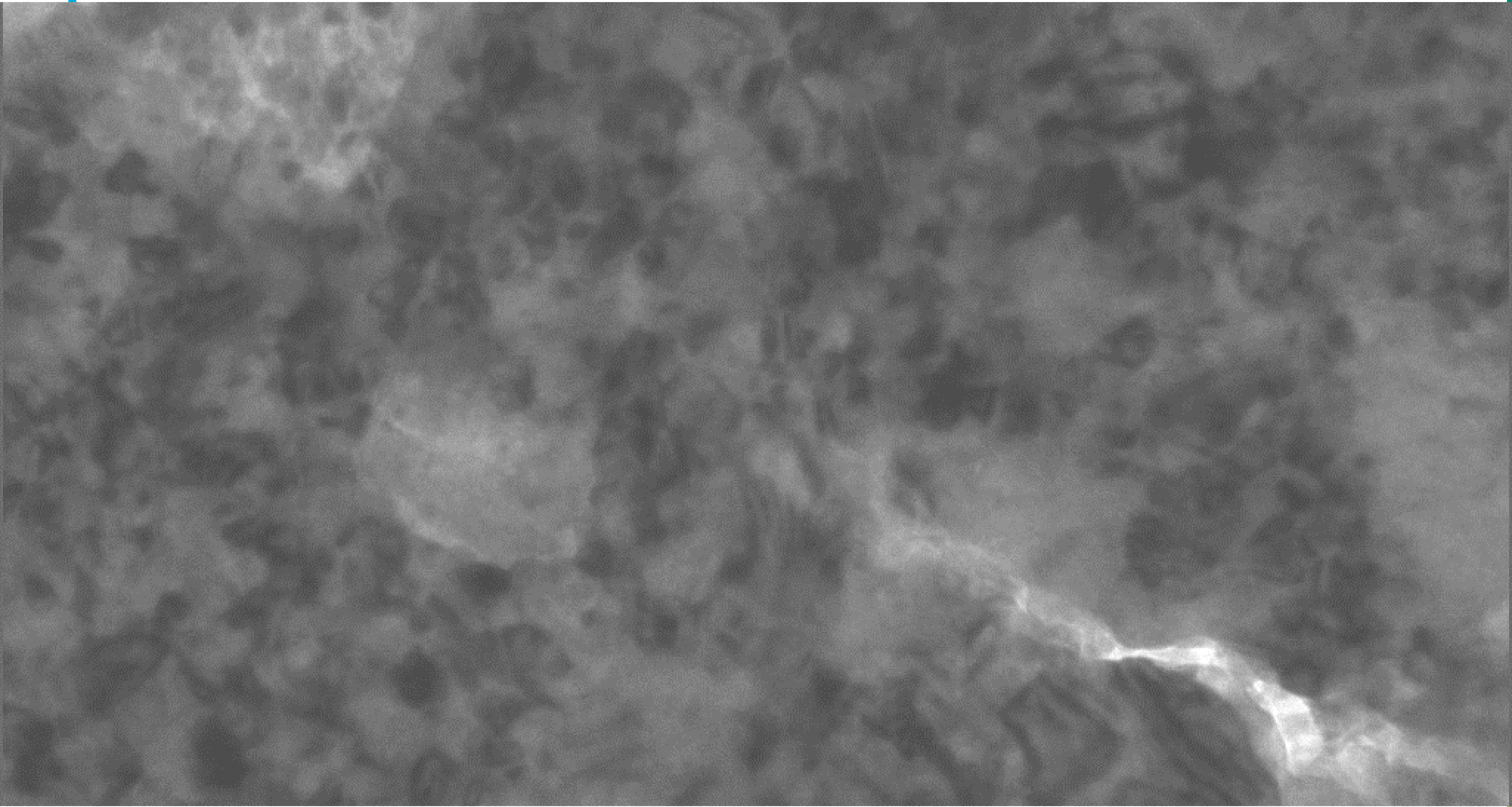


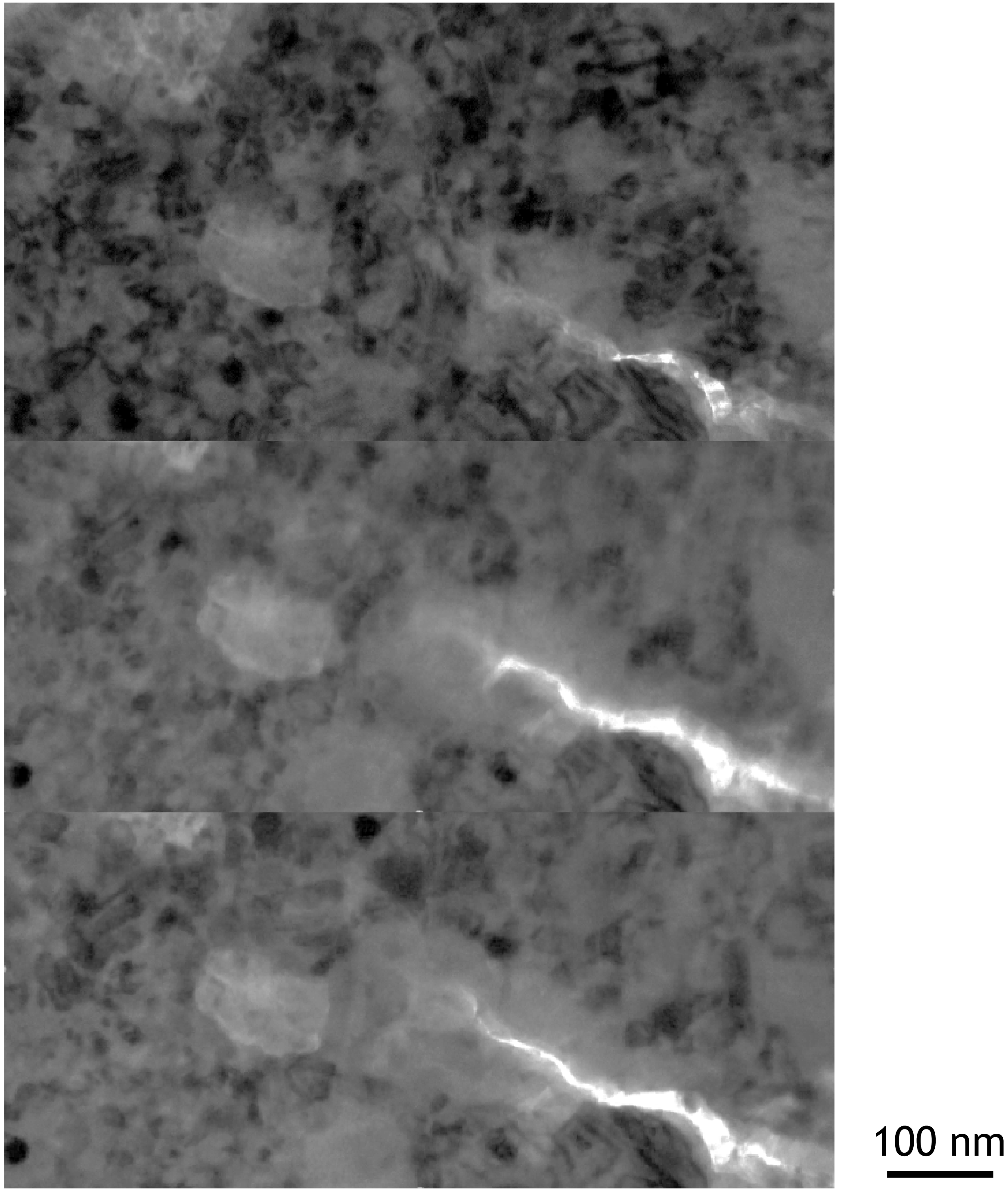
If the boundary is a perfect CTB, no migration occurs, and healing is delayed

Is this crack healing phenomena peculiar or common?

1. The electron-transparent specimen was thin (~50 nm), with little/no constraint
2. The twin boundary is not perfectly coherent- tilted slightly from the (111) plane, thereby providing twinning dislocations to facilitate migration.
3. The twin boundary is tilted at nearly 45° to the far-field tensile load, aiding shear coupling.
4. The crack had deflected substantially from mode-I, promoting shearing on the crack flanks
5. The TEM environment minimizes exposure to oxygen
6. Beam heating effects can occur in the TEM
7. The crack was microstructurally small (only involving a few grains).
8. The crack was transgranular: crack flanks were crystallographically aligned
9. The crack was growing very slow, averaging <<10^{-12} m/cycle
10. Pt is a noble metal, resistant to oxidation

Was this really just an extremely rare 'black swan' event, as a result of the confluence of all these conditions?




100 nm

Come perform your own experiments!

at the Center for Integrated Nanotechnologies

Extensive capabilities for nanomechanics including 6 in-situ platforms

2-Page user proposals due March 31

Cint.sandia.gov

Summary

1. Under high-cycle fatigue conditions, a fatigue crack in nanocrystalline Pt was observed to undergo healing.

2. Cold welding from crack flank contact could have been facilitated, at least in part, by grain boundary migration.

3. Additional observations in Cu suggest that this phenomena is not uncommon, at least in nanocrystalline metals under the conditions of the TEM experiment.