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In-situ TEM High Cycle Fatigue Response of Nanocrystalline Pt

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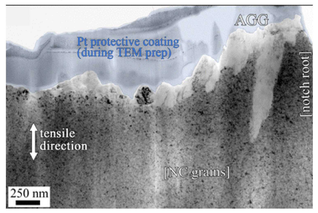
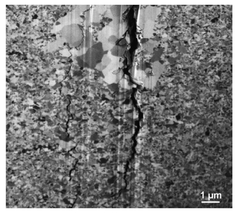
Introduction:

- Nanocrystalline (NC) metals are typically have improved fatigue properties compared to coarse grain counterparts with higher endurance limit
- Nanoscale microstructural changes however occur with high cyclic fatigue loading
- Fatigue in NC metals can show deleterious and significant grain boundary migration, rotation, grain growth, and localized plasticity

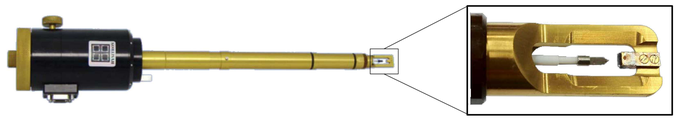


Furnish et al., J. Mat. Science, 52 (2017)



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

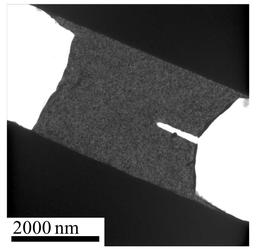
- In-situ TEM mechanical testing provides unique platform to explore underlying deformation mechanisms

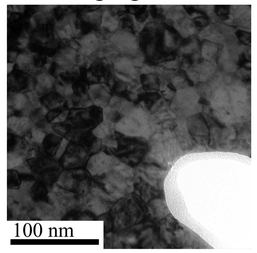


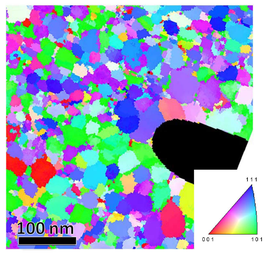
- Quantitative mechanical testing with cyclic loading capabilities available via Bruker/Hysitron PI-95 stage at Sandia's I^{3}TEM CINT facility

Notched Fatigue Conditions:

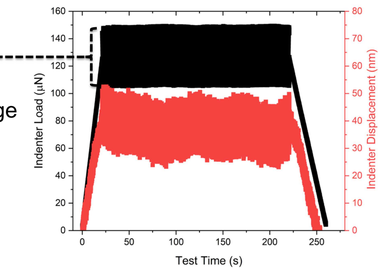
- FIB notch creates localized deformation and allows for higher resolution in-situ TEM imaging







- Precession electron diffraction (PED) provides ability to examine grain rotation and grain boundary misorientation evolution during fatigue propagation
  - Mean load (P_{mean}) = 135 uN
  - Amplitude load (P_{amp}) = 35 uN
  - 200 Hz dynamic loading → image motion blur with standard 15 frames/s camera
  - 13 cycles per individual frame

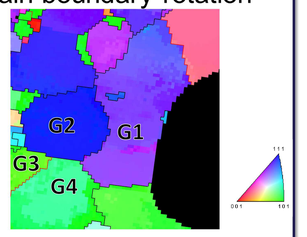


- PED orientation maps taken prior to test and during intermediate loading condition (120,000 total cycles)

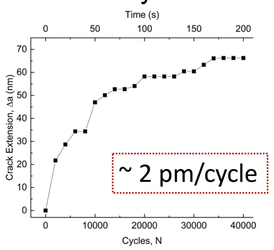
Fatigue Crack Growth Rate:

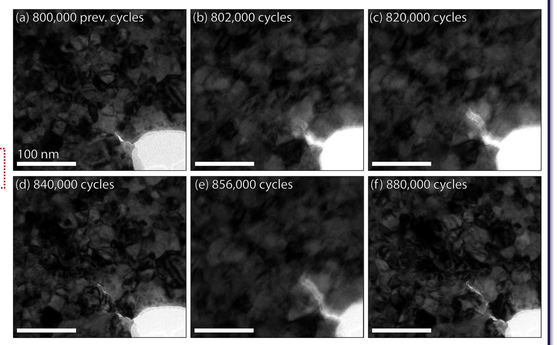
- Intermediate PED map (120,000 total cycles) indicates significant and quantifiable grain and grain boundary rotation

GB_{3-4}(Twin Boundary Σ3)	Misorientation	Deviation from Ideal Σ3
Prior to Cyclic Loading	59.9° [1 1 1]	0.8 °
Crack Impinges GB at GB_{1-2}	56.9 [7 7 6]	4.9 °



- Rapid propagation – transgranular between 800,000 to 854,000 total cycles
- After 854K cycles, cyclic loading crack impinges grain boundary

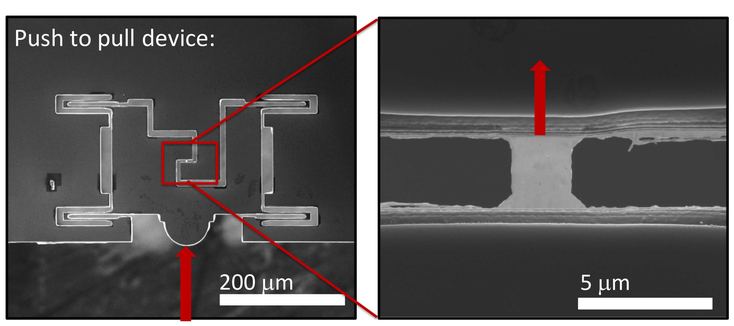




  - da/dN = 1.7 x 10^{12} m/cycle
  - Non-linear crack extension rate
- No indications of previous deflected crack path – propagation continues closer to a mode I (normal to loading) direction
- Transgranular crack propagation has characteristics of "classic" zig-zag crack deflection

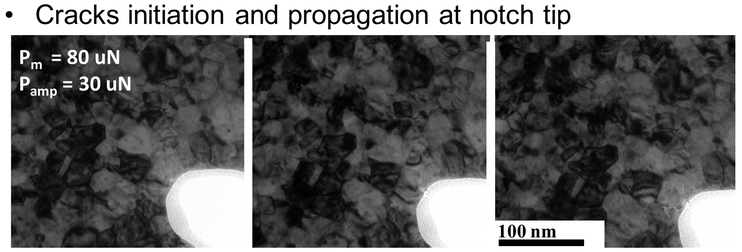
High Cycle Fatigue In-situ TEM Method:

- Push-to-Pull devices – microfabricated Si test frames
- $F_{applied}=F_{measured}-F_{spring}$
- Pt film (40 nm) sputter deposited on NaCl → floated onto devices
- FIB utilized for nanoscale machining → create small geometry for tension-tension fatigue → critical to minimize Ga imaging
- Indenter compression creates region of tension on sample

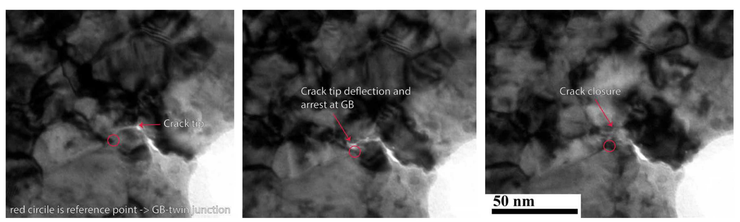


Crack Propagation and Deflection:

- Cracks initiation and propagation at notch tip

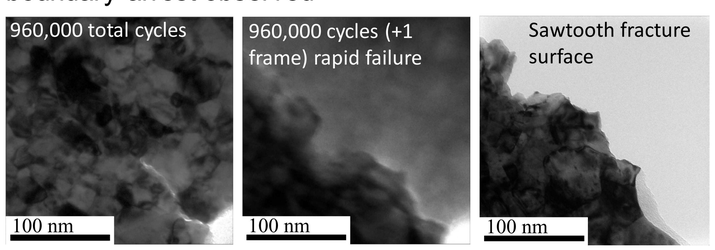


- Crack propagation transgranular
- Significant crack deflection away from mode I direction
- Deflected crack with mix-mode condition impinges grain boundary and appears to close (crack heal) upon loading



Key Outcomes and Future Directions:

- In-situ TEM fatigue tests utilizing the PI-95 nanoDMA setup provides a wealth of new opportunities for understanding nanoscale microstructure evolution under dynamic mechanical testing → 2D materials, novel architectures
- Crack initiation, transgranular propagation and grain boundary arrest observed



- Unique crack healing event observed during crack deflection away from mode I conditions
- Localized deformation (grain rotation and grain boundary misorientation change) quantified via PED orientation analysis

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