SAND2013-2396C

Nanomechanics and Nanometallurgy of Boundaries

Sandia National Laboratories

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Today's topic: **Grain growth in nanocrystalline alloys under cyclic loading conditions**

Funded by DOE Office of Basic Energy Sciences

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Team: Nanomechanics and Nanometallurgy of Boundaries

TASK 1
Experimental Observations
of Boundary Processes

Investigators
Brad L Boyce
Blythe Clark
Khalid Hattar

Postdocs
Henry Padilla (now staff)
John Sharon
Shreyas Rajasekhara

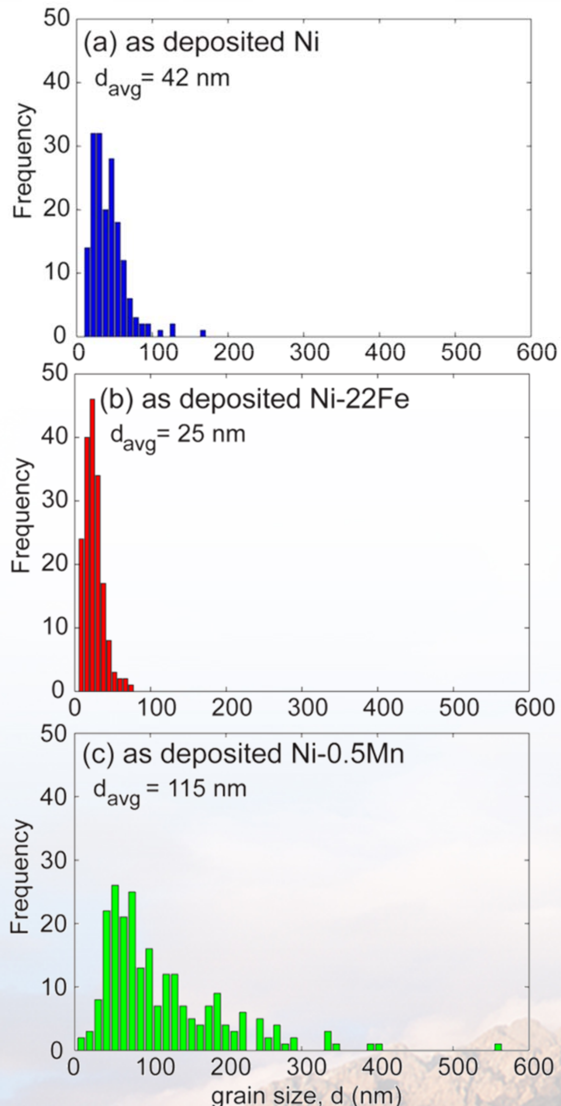
***TASK** 2*
Theory and Modeling
Of Boundary Processes

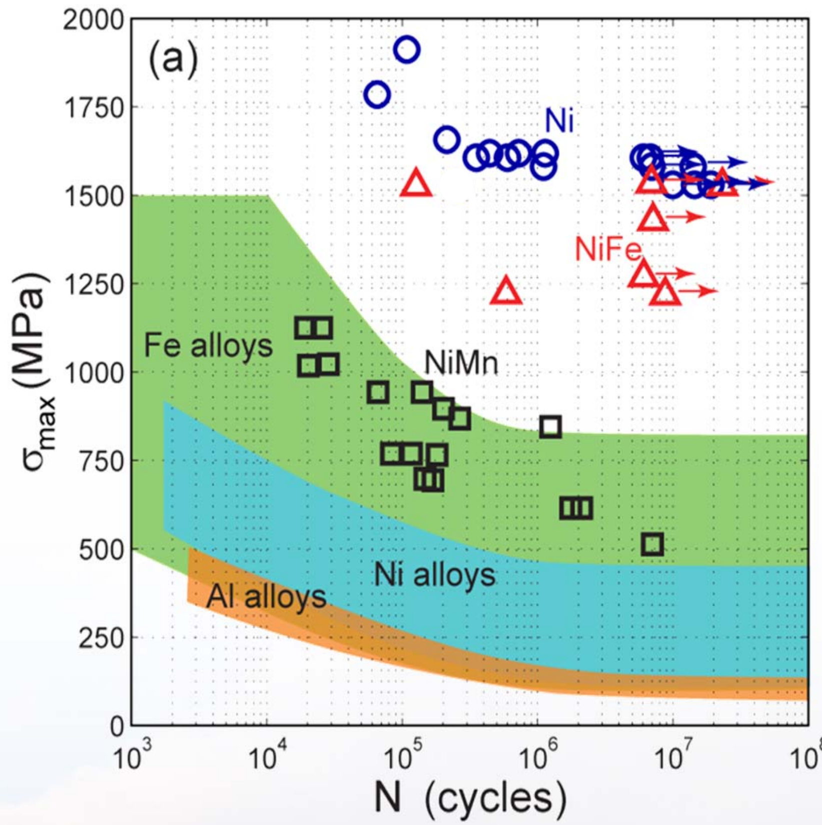
Investigators
Stephen Foiles
Liz Holm (now at CMU)

Postdocs
Eric Homer (now at BYU)
Garritt Tucker
Ben Winchester

External Collaborators, Co-conspirators, & Overall Decent People (mostly): Ian Robertson (UIUC), Tony Rollett & Greg Rohrer (CMU), Greg Thompson (Alabama), Paulo Ferreira (UT), Amit Misra (LANL), Mitra Taheri (Drexel), Daniel Keiner (Leoben, Austria), Chris Schuh (MIT), Douglas Spearot (Arkansas), Xinghang Zhang (Texas A&M), Julia Greer (Cal Tech), Dan Gianola (U Penn), Andy Minor & Peter Hosseman (Berkeley), Kevin Hemker (Johns Hopkins), Apurva Mehta (Stanford Synchrotron), O. El-Atwani (Purdue), Tom LaGrange (LLNL), Emmanuelle Marquis & Sam Daly (Michigan),

S-N curves for nanocrystalline nickel alloys show extraordinary fatigue resistance





Boyce & Padilla, *Metall. Mater. Trans. A*, 2011

While ultrafine grained Ni-0.5Mn only exhibited modest Improvement in S-N fatigue performance, nanocrystalline Ni and Ni-Fe both showed dramatic improvement in fatigue performance. Why such a large difference? How does fatigue performance scale with grain size and strength?

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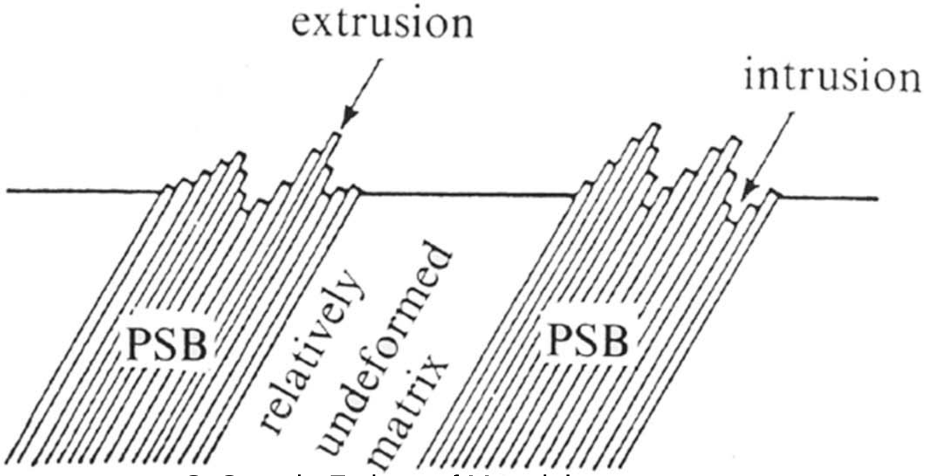
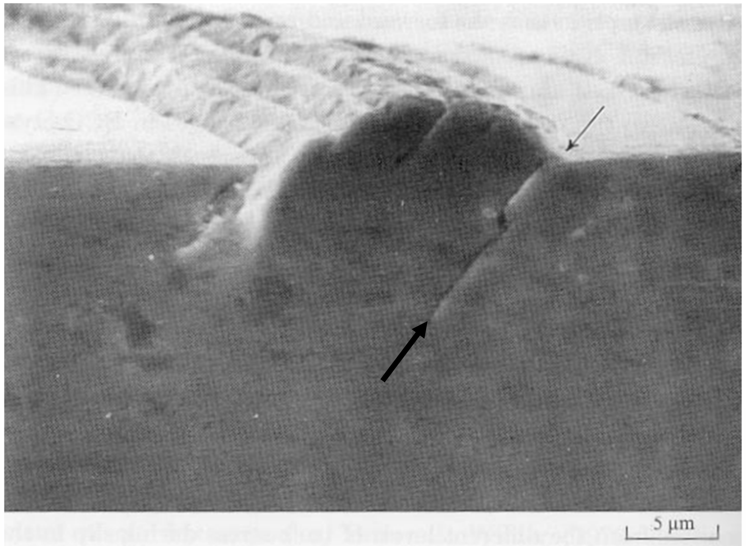
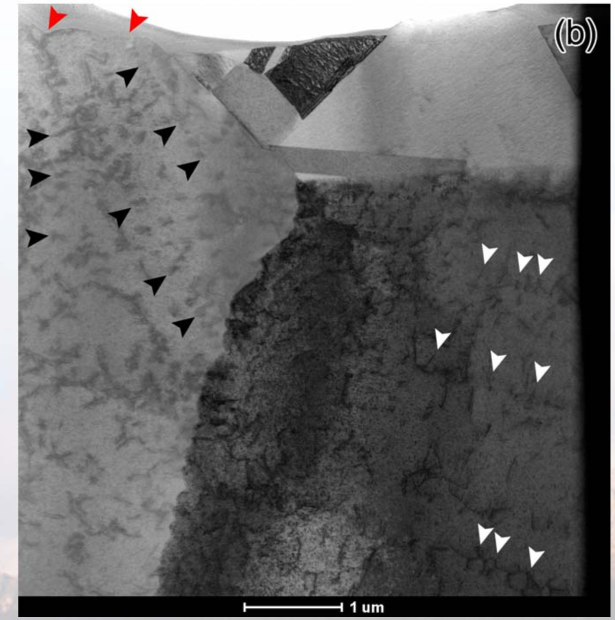
Hypothesis: the small grain size of nanocrystalline metals suppresses persistent slip crack initiation processes



S. Suresh, Fatigue of Materials

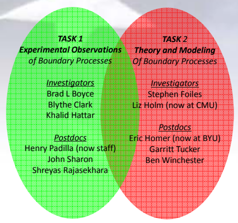


Ma and Laird, 1989



Boyce & Padilla, *Metall. Mater. Trans. A*, 2011

If persistent slip bands operate over dimensions of 100's of nm to microns, how does fatigue response change when there is not sufficient path length for PSB operation?



Core Theme & Key Questions: Nanomechanics and Nanometallurgy of Boundaries

- **Motivating Questions**
  - *Is mechanically-driven grain growth rate limited as a diffusional process controlled by Arrhenius kinetics? [Is it a thermally-activated process?]*
  - *Do impurities, precipitates, defects, and compositional heterogeneities impact mechanically-induced grain growth in the same way that they affect conventional thermally-induced growth?*
  - *How do polycrystalline grain boundary networks serve to assist or impede grain boundary motion?*
  - *Why does grain growth occur abnormally for certain microstructures and environments?*
  - *Are there special boundary types or boundary-defect interactions that give rise to mechanically-induced grain growth?*
  - *How does mechanically-induced grain growth accelerate or suppress failure under monotonic and cyclic loading conditions?*
  - *Can grain growth be harnessed as a means to dissipate plastic work?*

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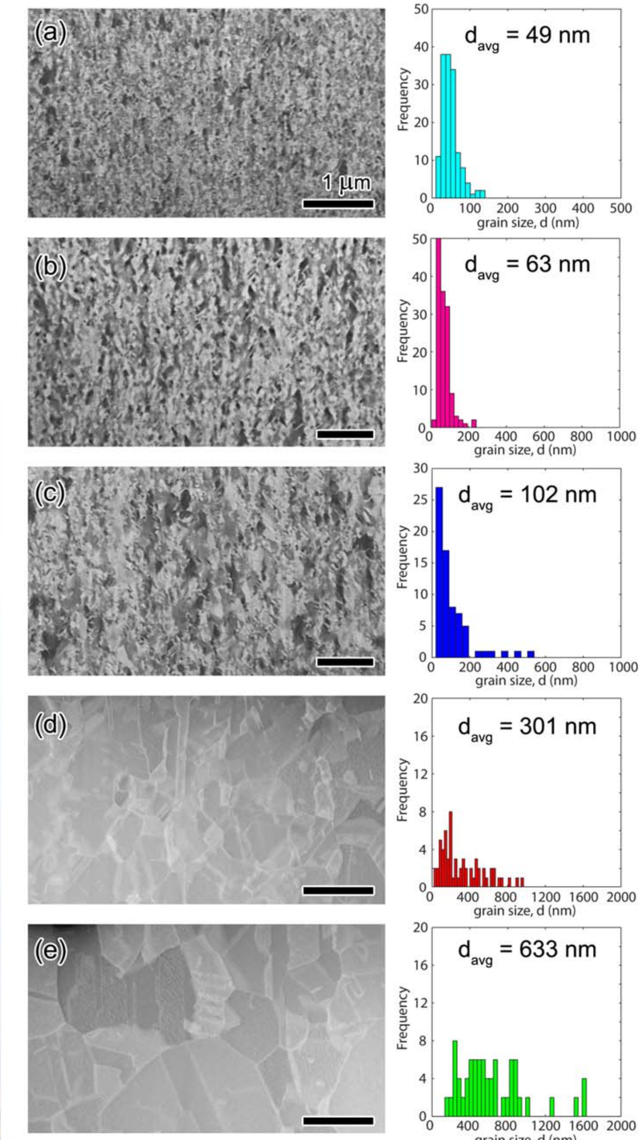
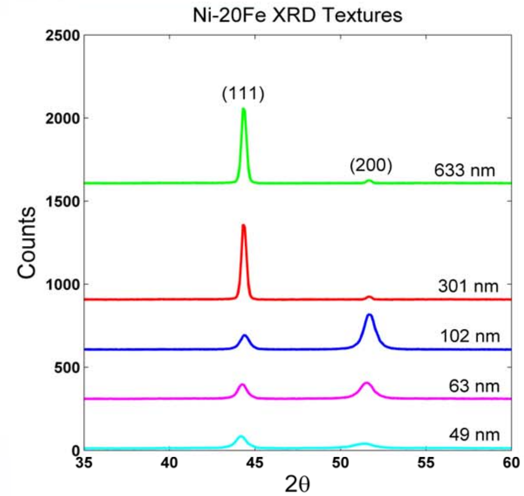
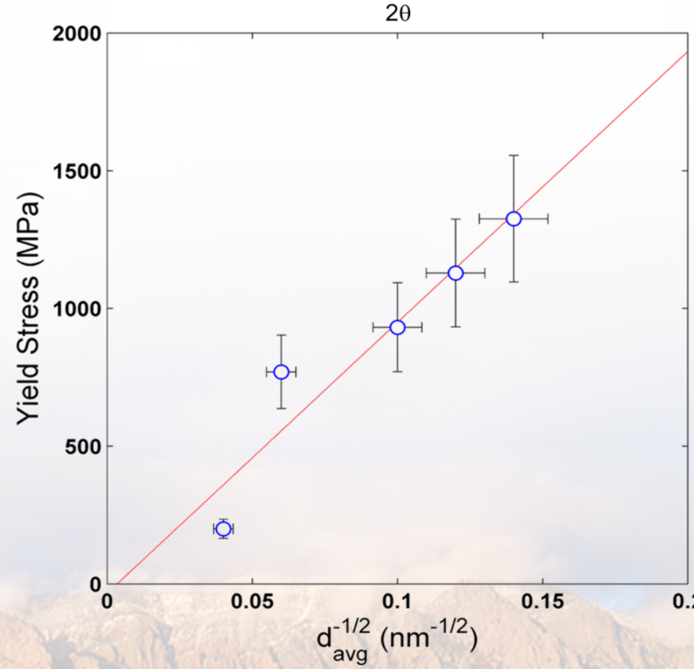
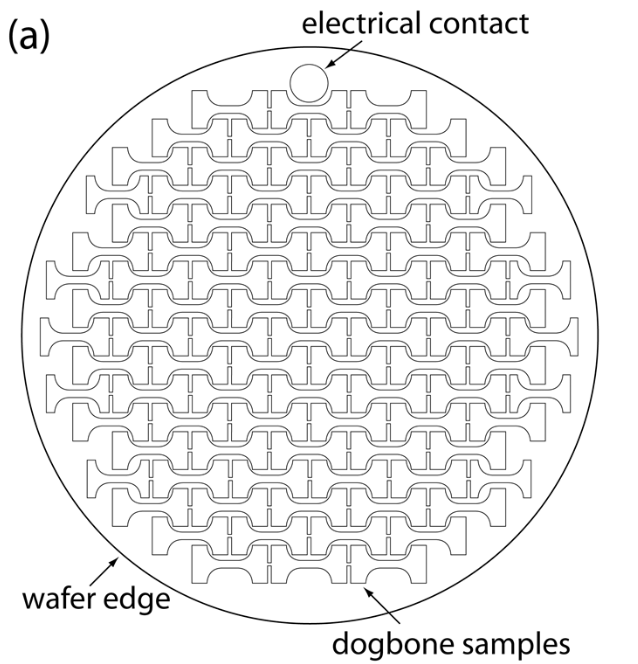
One alloy (Ni-10Fe) produced with 5 Grain Sizes

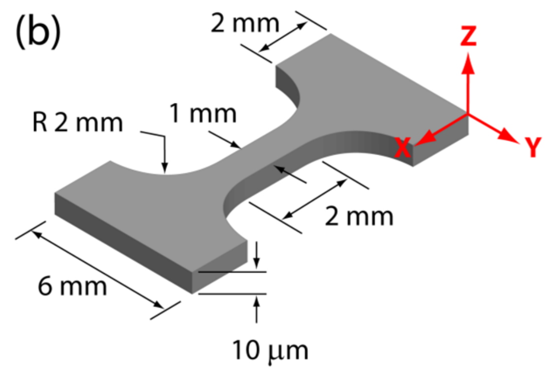


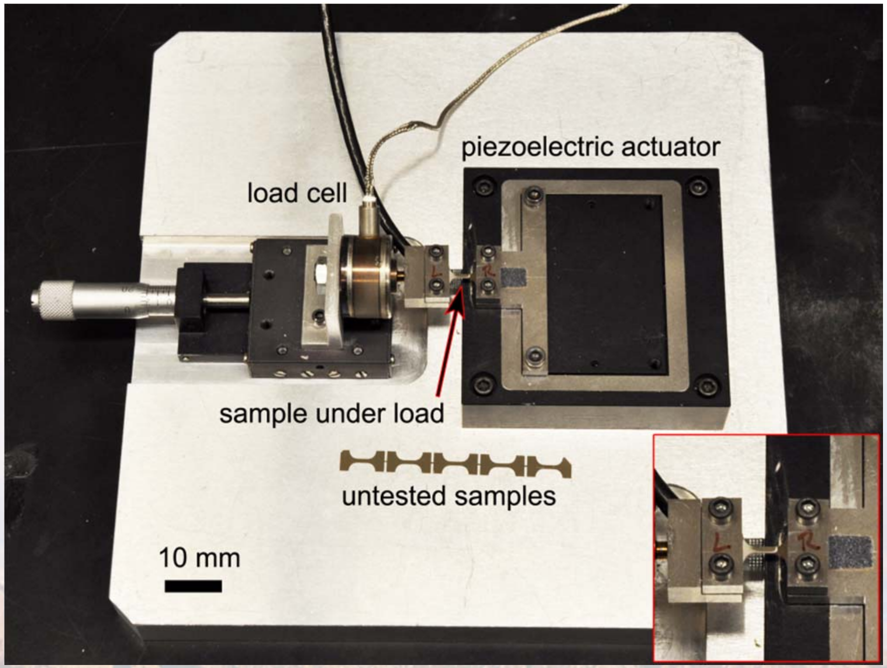




Thin film fatigue of electrodeposited Ni-Fe (permalloy)







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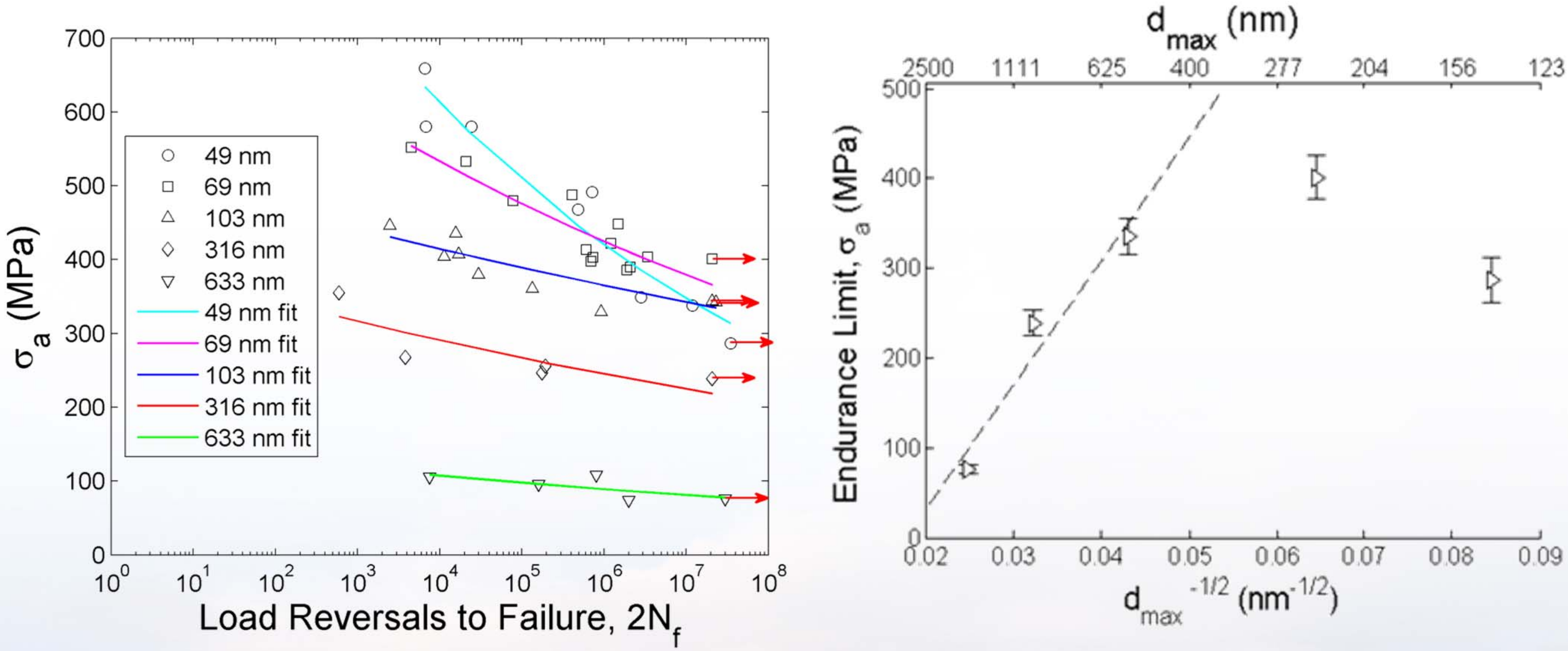
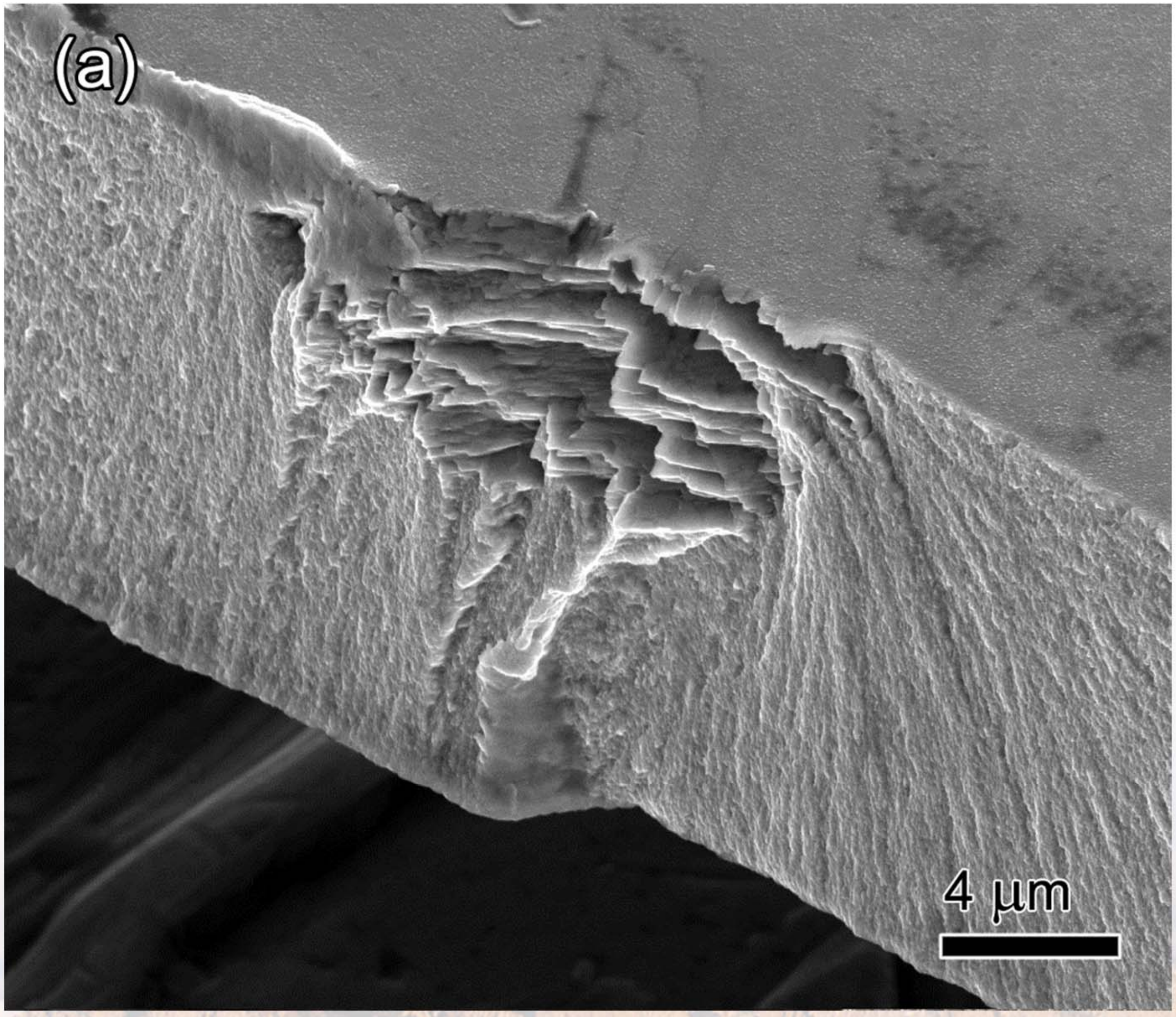
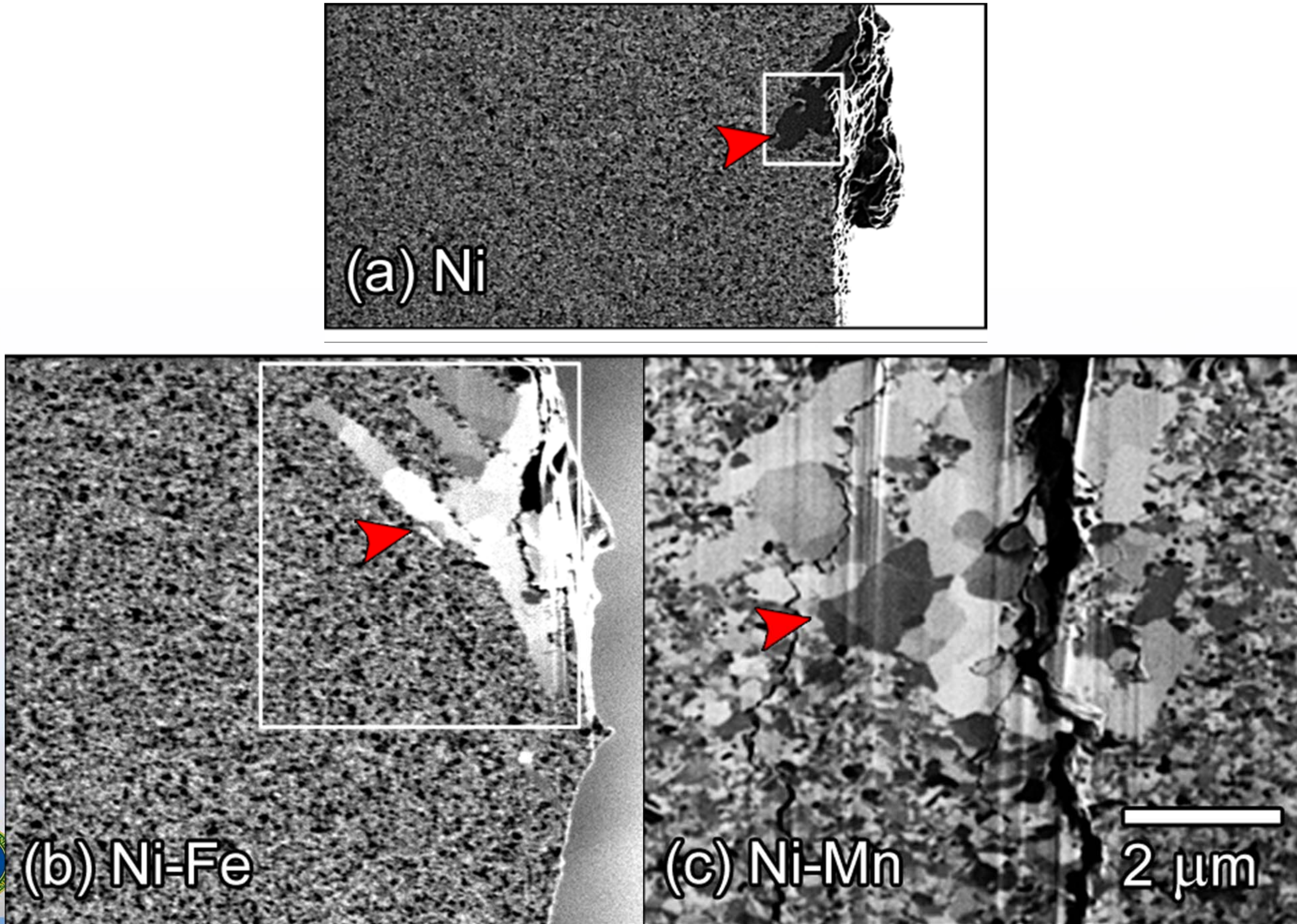
Endurance limit plateaus at average grain size ~70 nm! (max grain size ~400 nm)



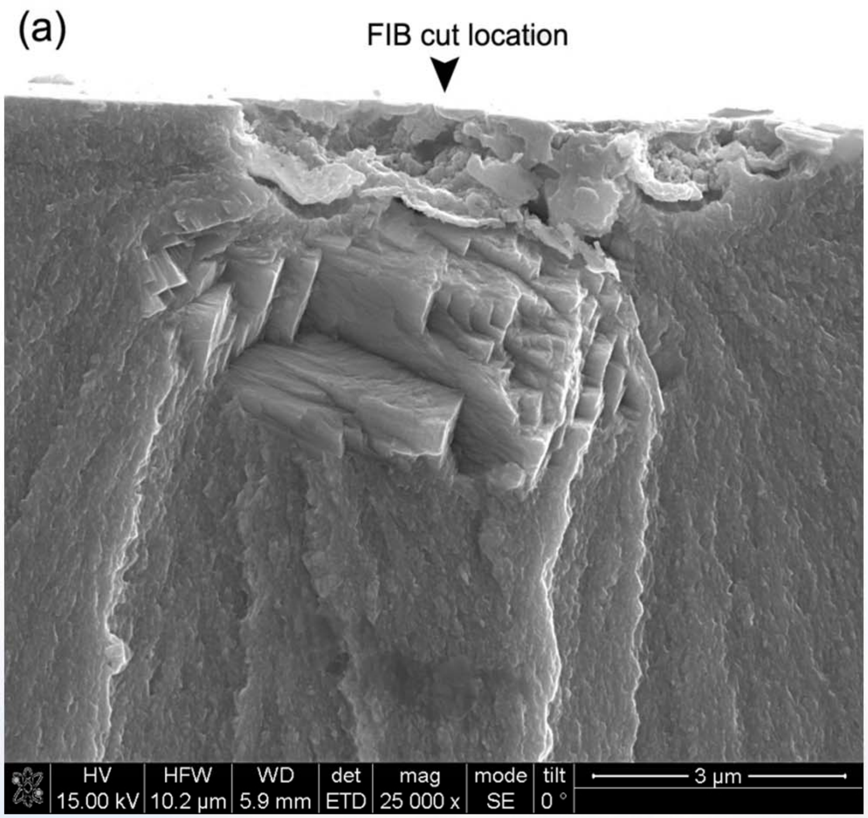
For the nanocrystalline samples (d_{gb} < ~70 nm), a block initiation zone is apparent

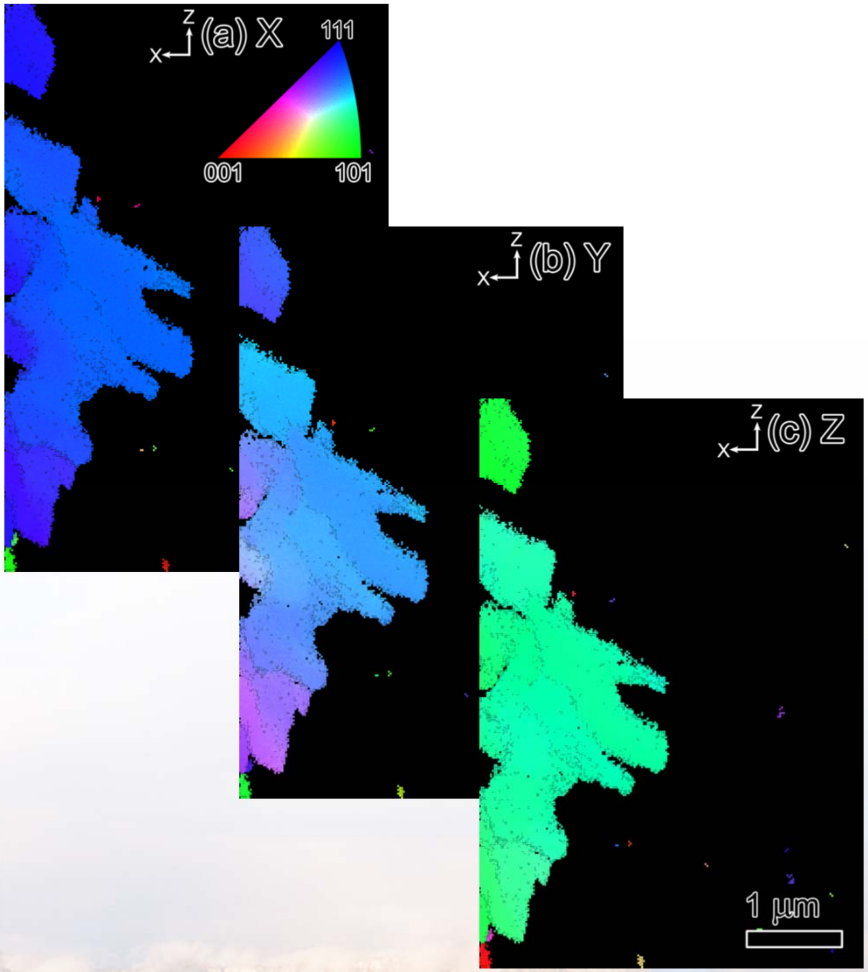


Blocky fracture surface corresponds to very large subsurface grains at point of crack initiation



Same morphology of highly textured, fatigue-coarsened grains form in samples with grain sizes of 100 nm or less.





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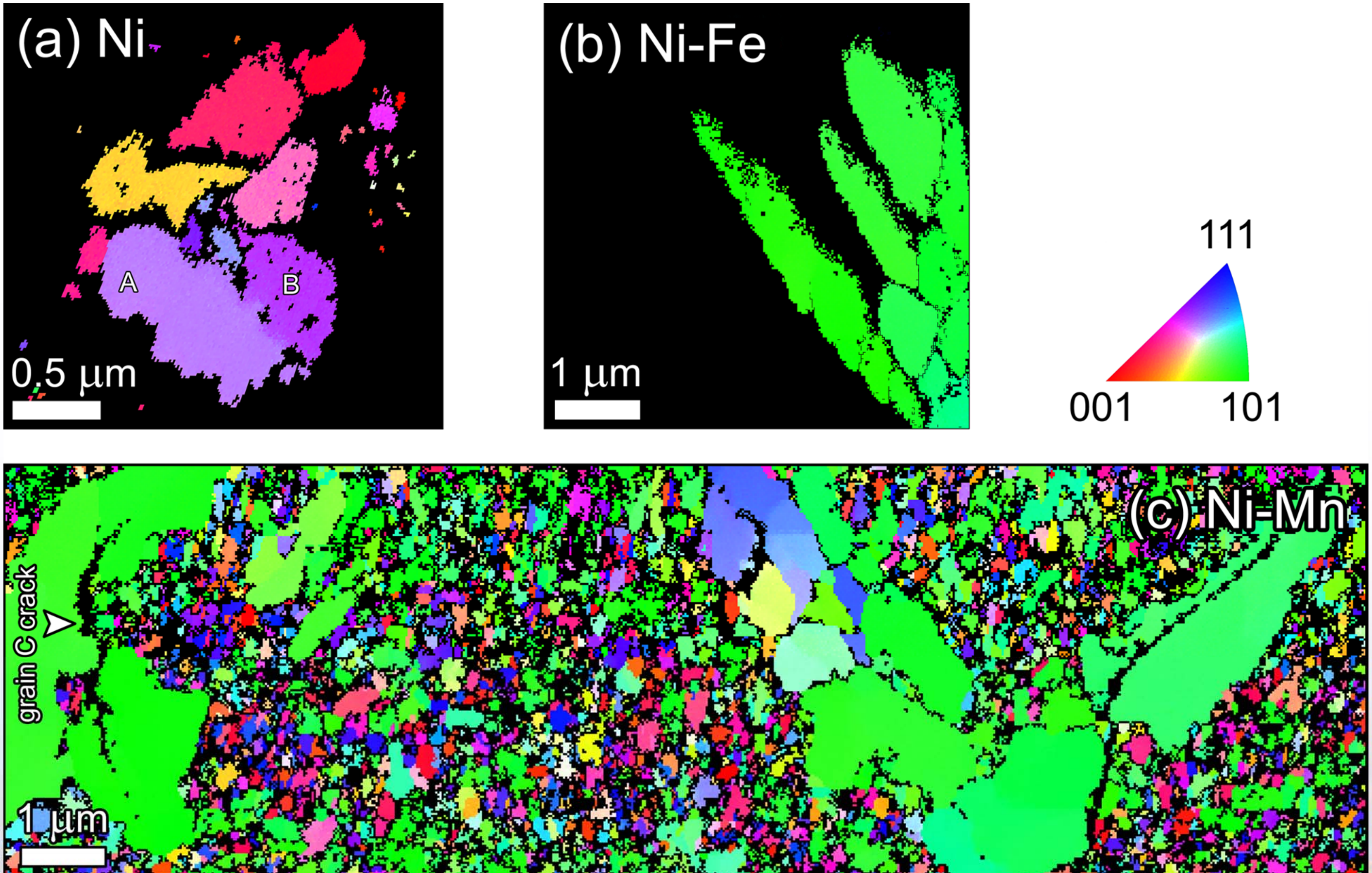
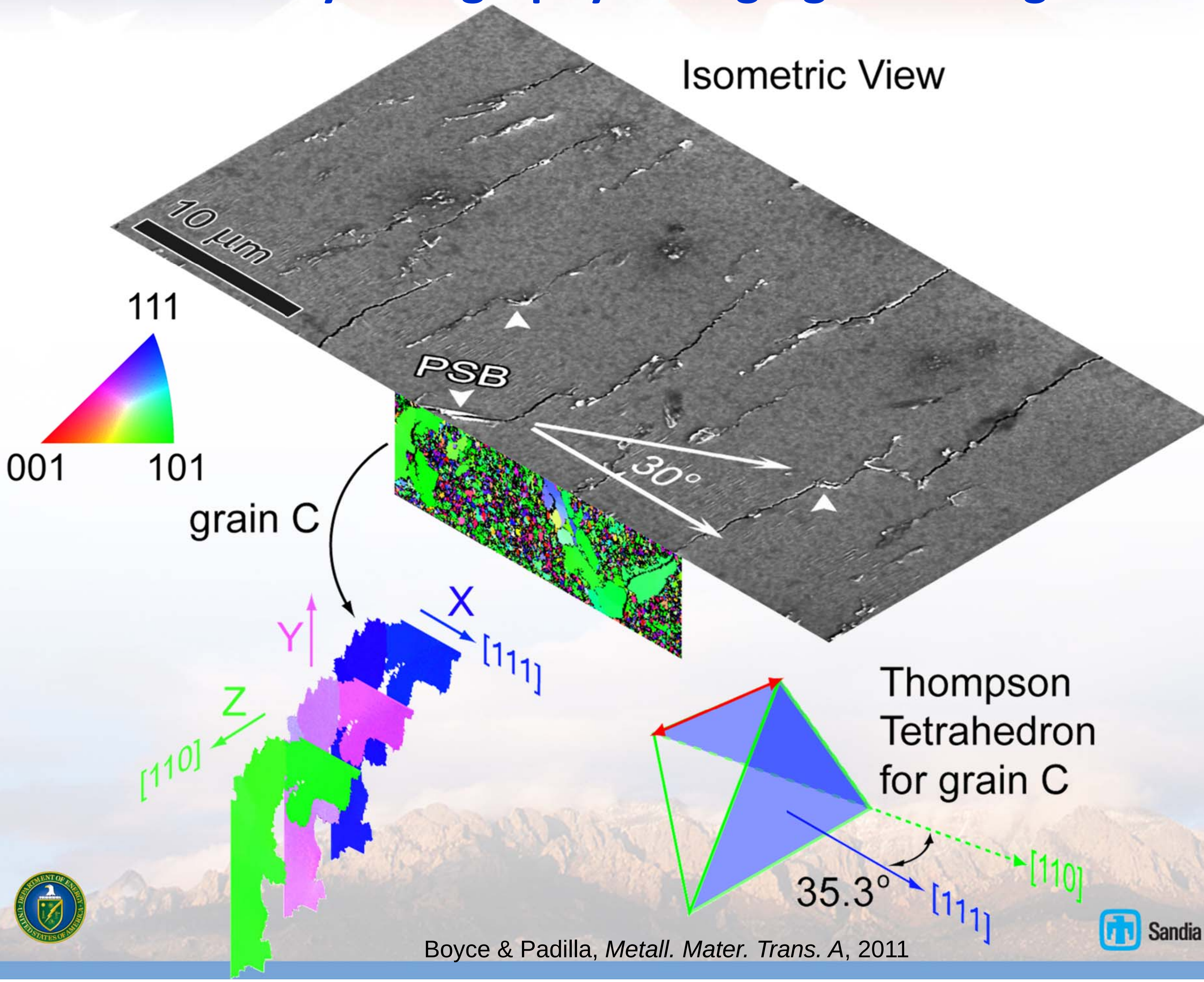
EBSD of large grain region



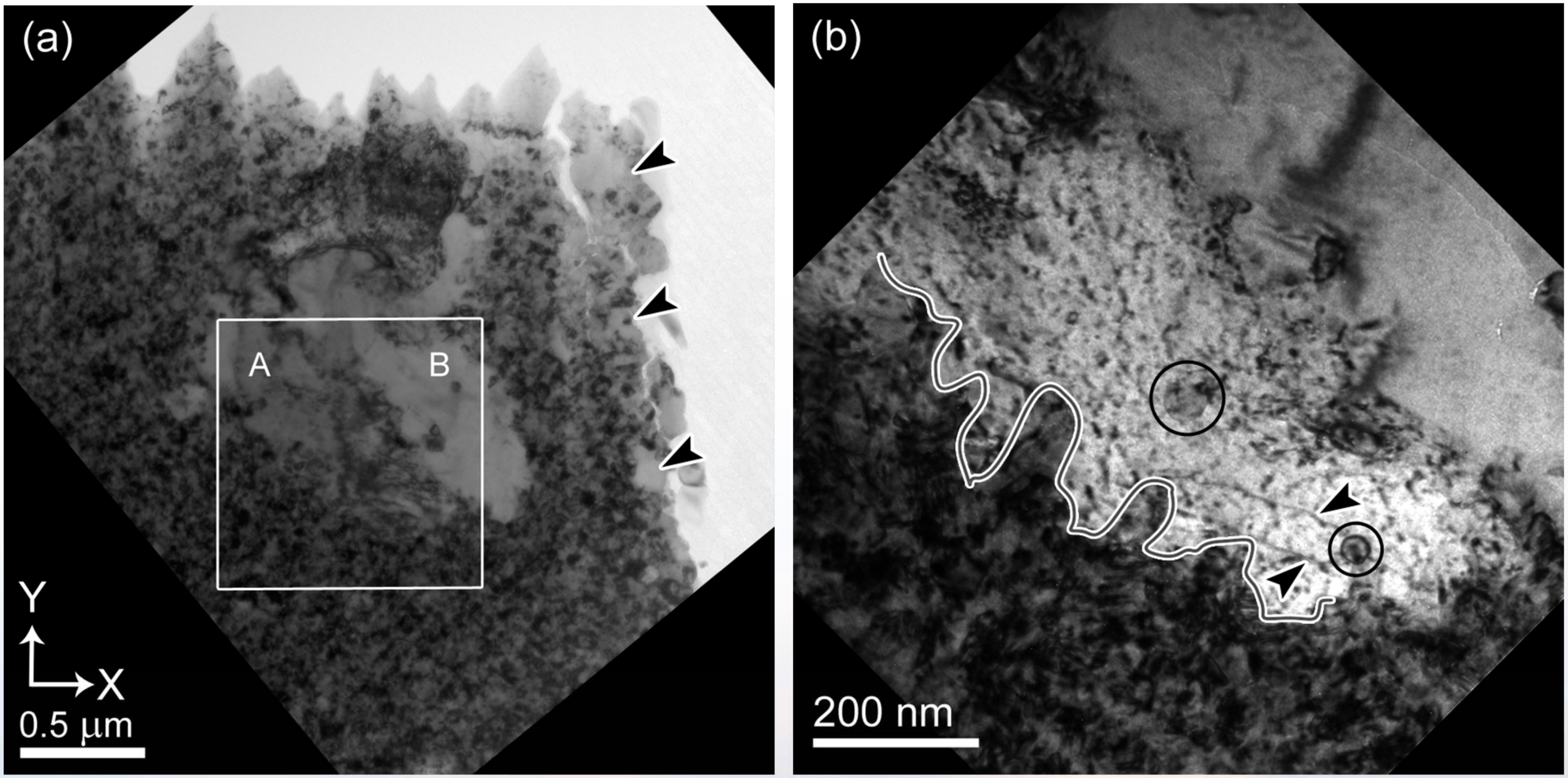
Boyce & Padilla, *Metall. Mater. Trans. A*, 2011

Crystallography of large-grained regions



Boyce & Padilla, *Metall. Mater. Trans. A*, 2011

Defect residue and grain boundary morphology of an abnormal grain.



Boyce & Padilla, *Metall. Mater. Trans. A*, 2011

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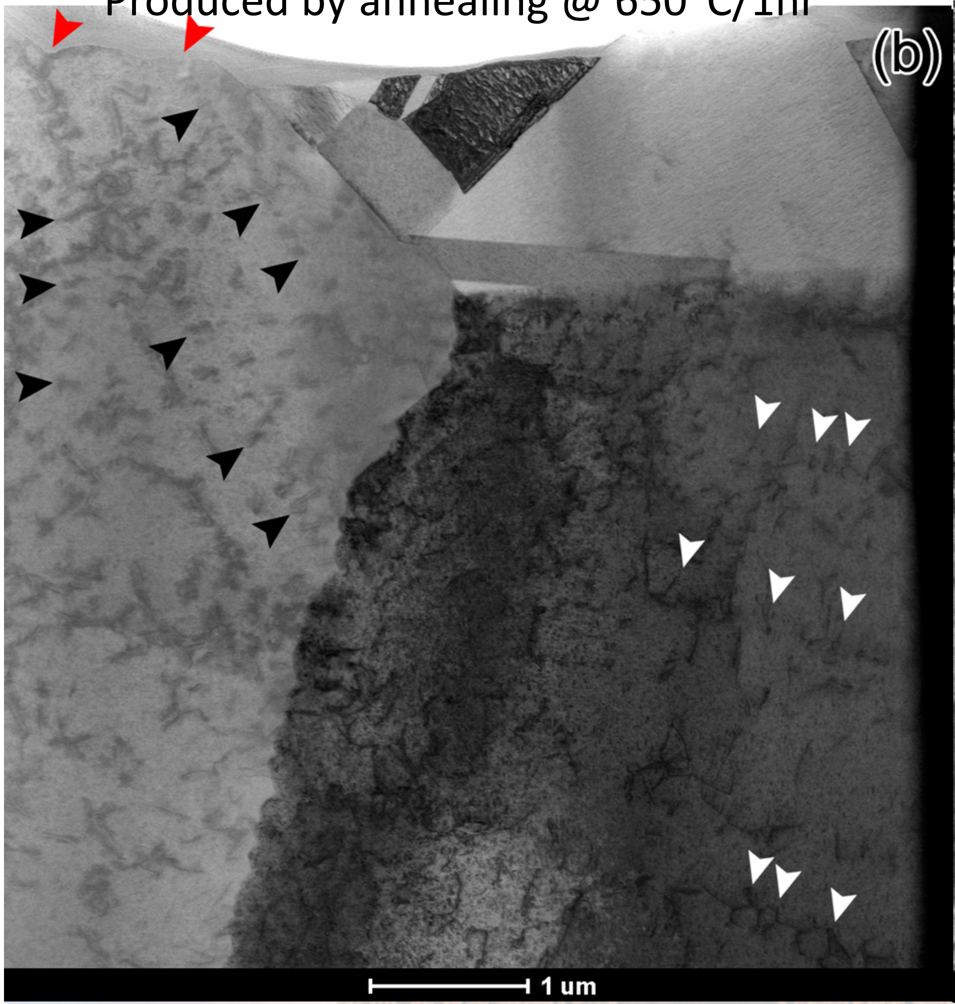
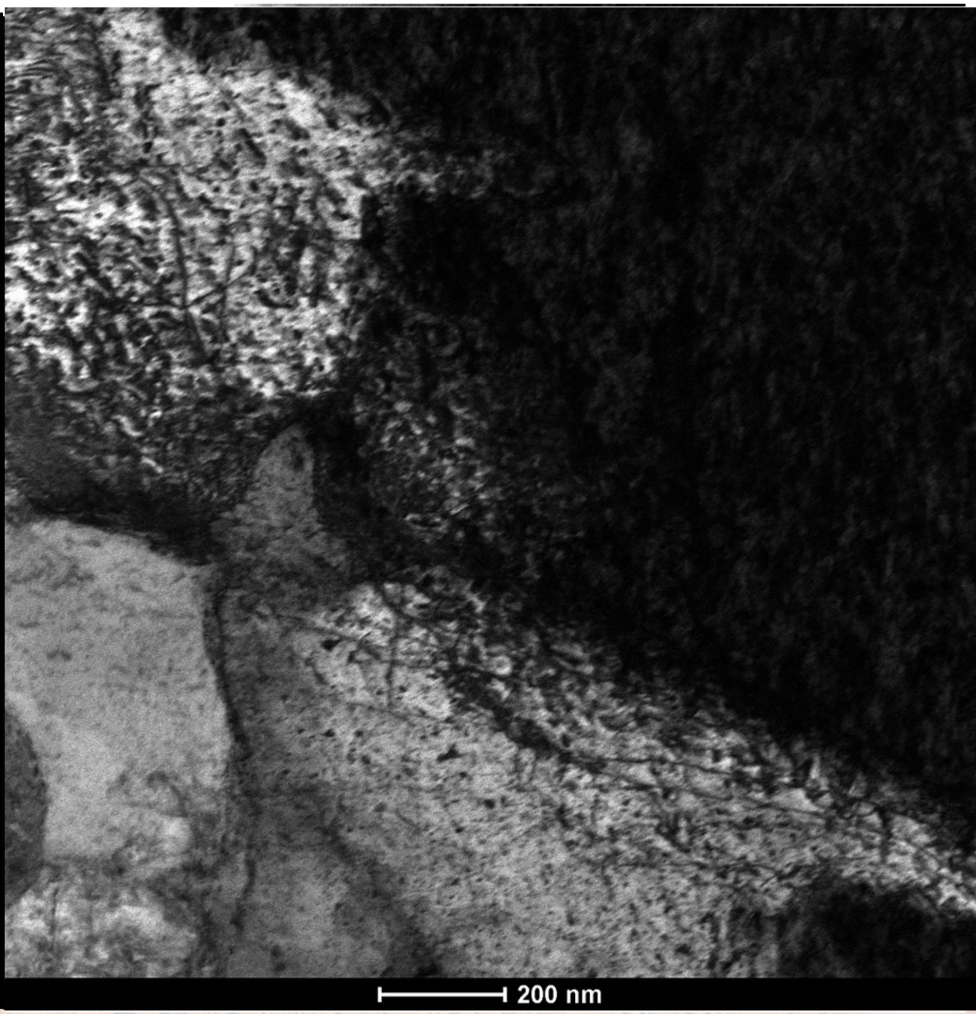
Persistent slip ladders are apparent in coarse grained metals, but have not yet been found in fatigue-coarsened grains

Coarse Grained Ni-Fe
Produced by annealing @ 650°C/1hr

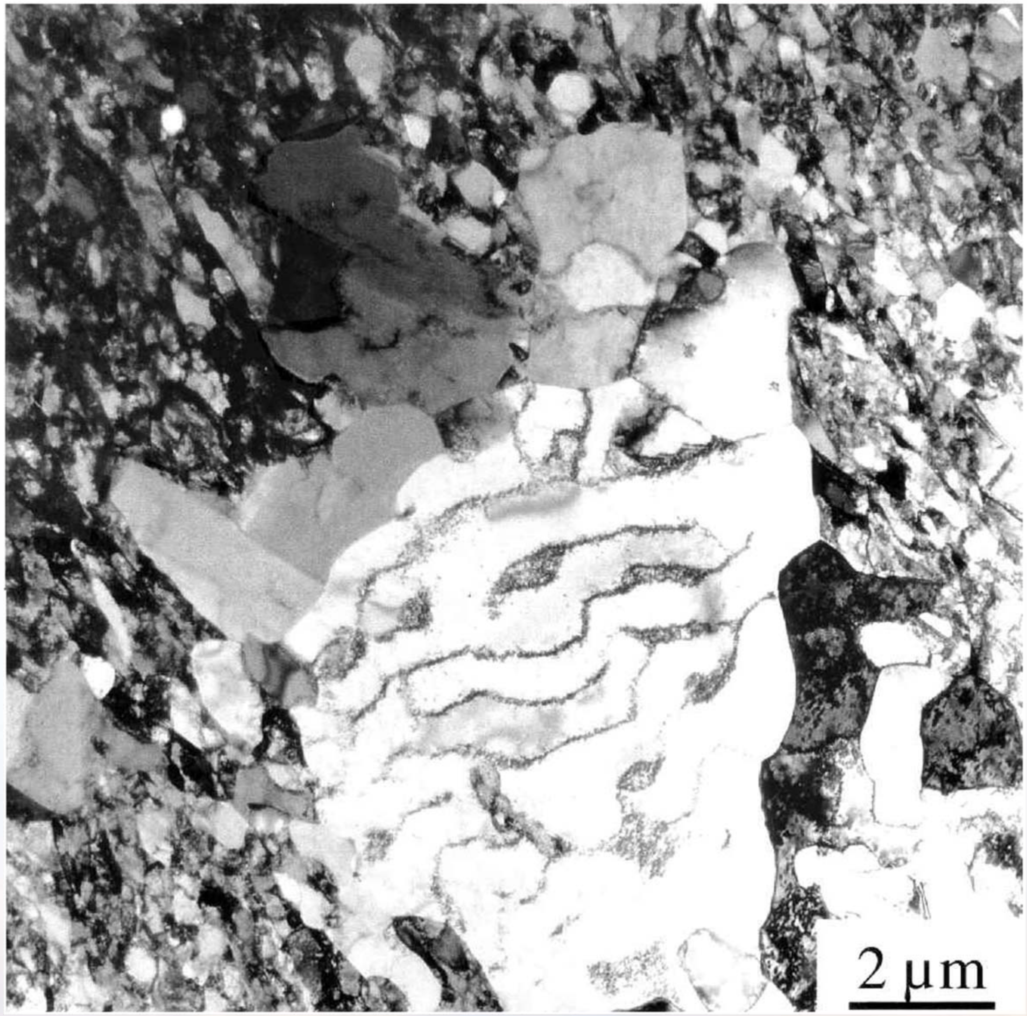


Nanocrystalline Ni-Fe



Boyce & Padilla, *Metall. Mater. Trans. A*, 2011

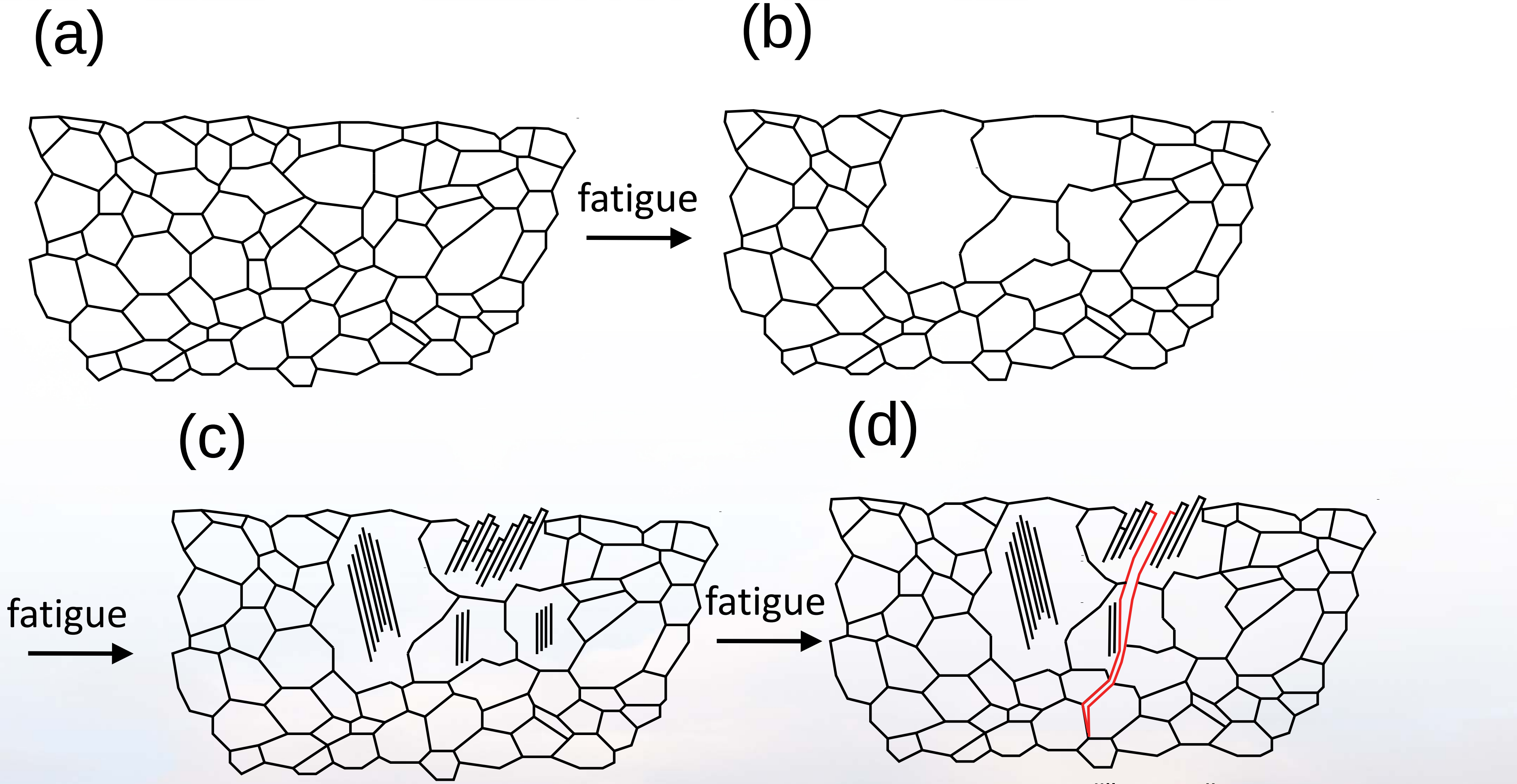
Others have observed fatigue-induced grain coarsening too…



LCF of UFG Cu: H.W. Hoppel, et al., *Phil Mag*, 2002

Coarsening on crack flanks during HCF of NC Ni-Fe: Cheng, Zhao, Lavernia, *PRL*, 2010

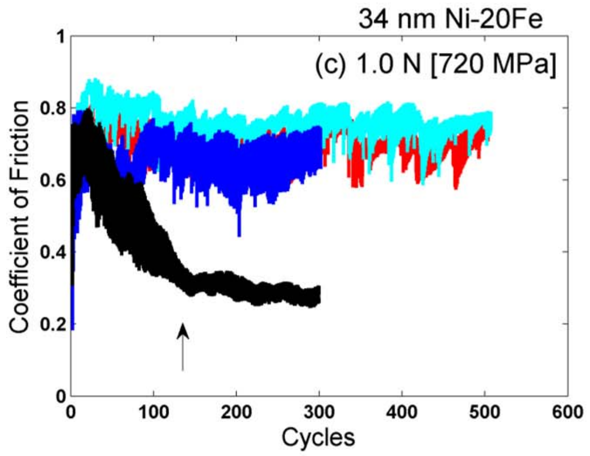
A new crack initiation process involving mechanically-induced grain growth?

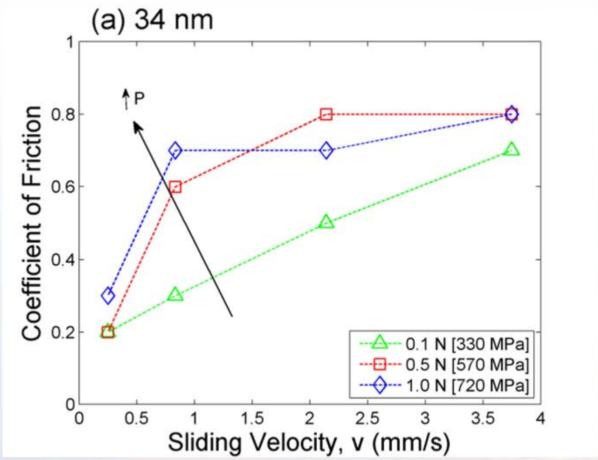


Boyce & Padilla, *Metall. Mater. Trans. A*, 2011

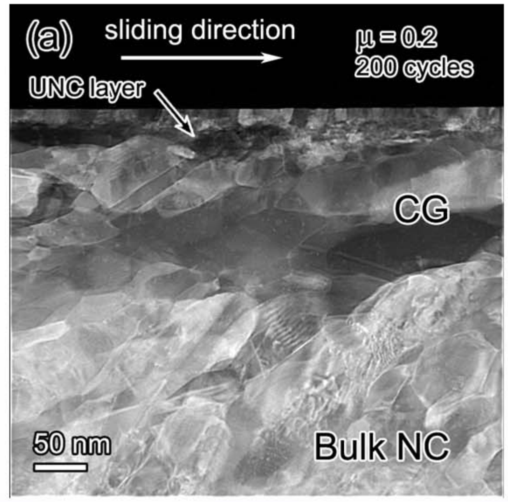
If this is a standard mechanism for nanocrystalline degradation under cyclic loading, then is it observable in other scenarios, such wear?

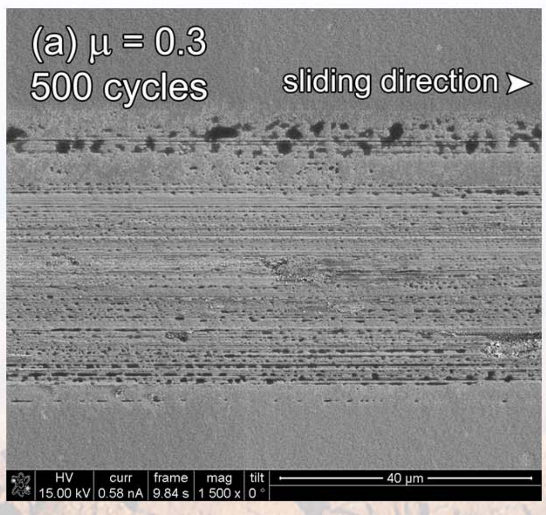
Inhomogeneous stress distributions under wear tip lead to both grain refinement and grain growth



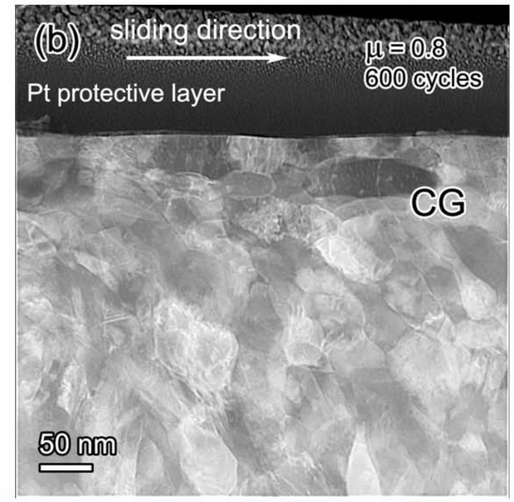


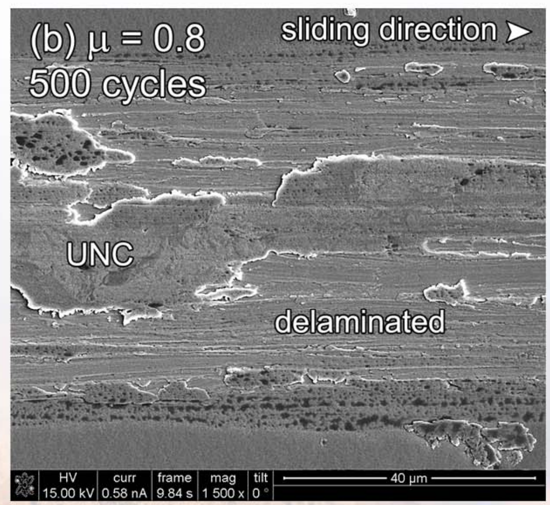
Low friction behavior





High friction behavior





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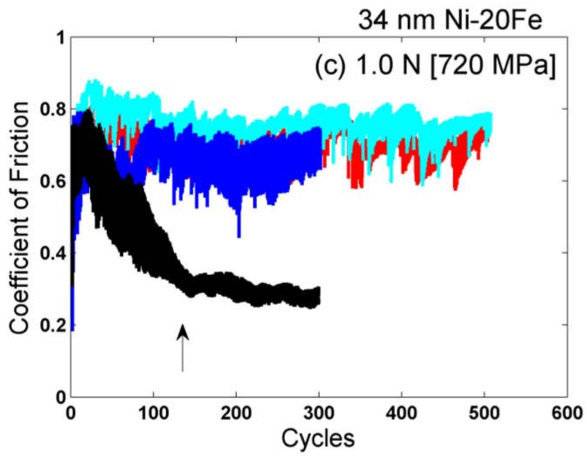
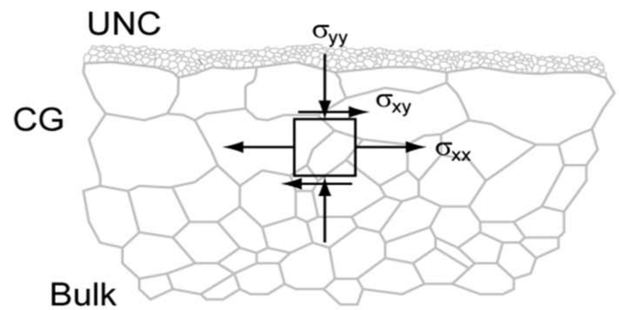
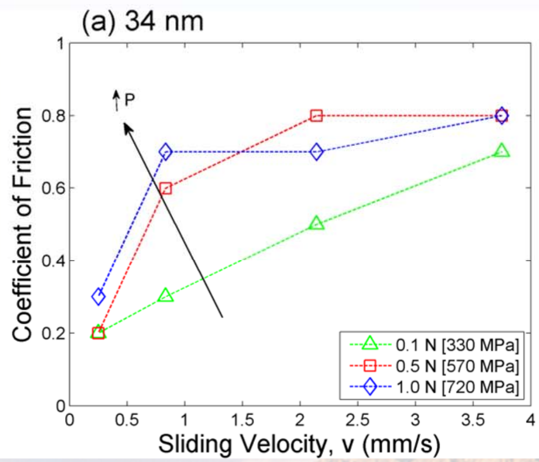
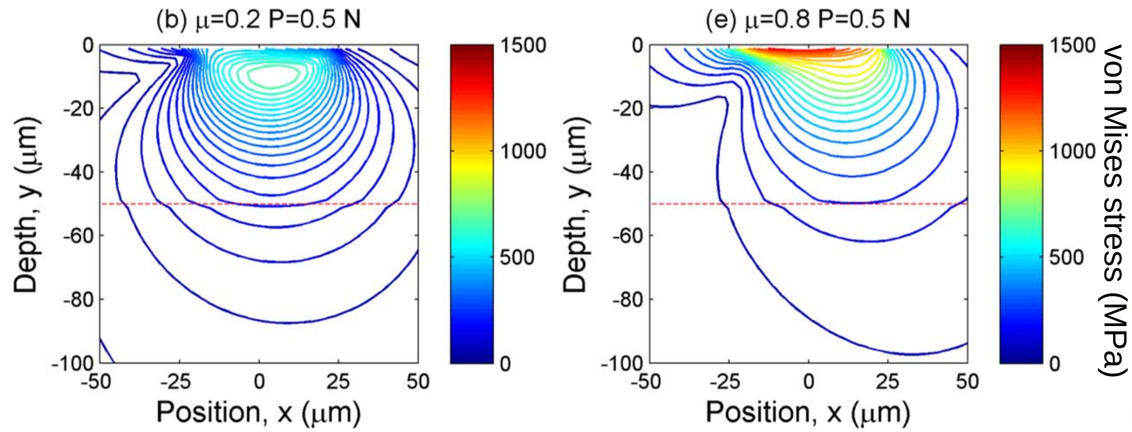
CATCH 22: High friction coefficient results in high stresses at the interface whereas low friction coefficient drives the peak stresses away from the interface.









Low friction behavior is associated with weaker peak stresses, far below the UNC-CG interface.

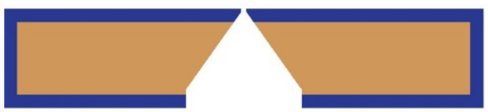
High friction behavior is associated with elevated peak stresses immediately at the UNC-CG interface

How and why does cycle-driven grain growth occur?
$\Rightarrow$ This question begs for new in-situ observations

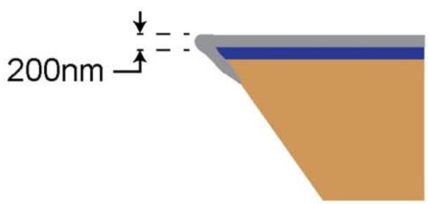
Sample Fabrication

1. Start with Si wafer coated with nitride

2. Open window in nitride



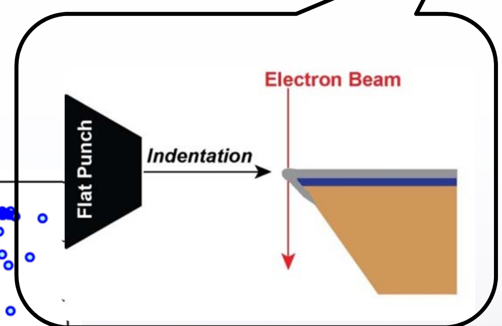
3. Anisotropic wet etch (TMAH) exposes 111 plane to create a wedge

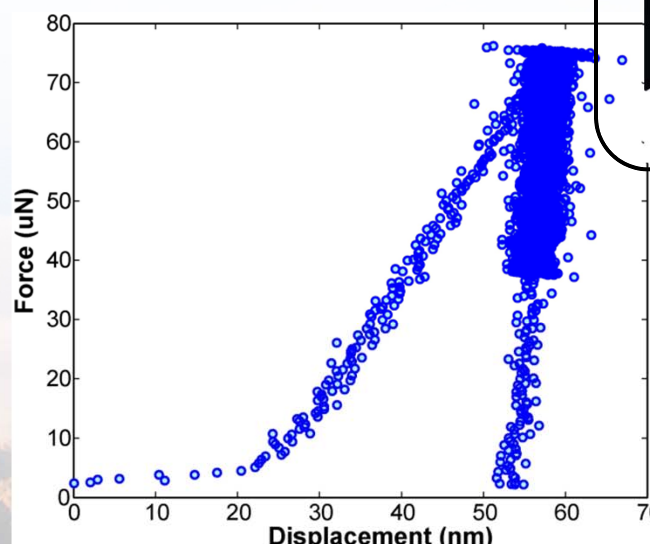


4. Pulse laser deposition of Al onto wedge

In situ TEM fatigue

- Microscope: JEOL JEM 2100 LaB_6 TEM
- Holder: Hysitron PI-95 PicoIndenter with a 1μm flat tip punch





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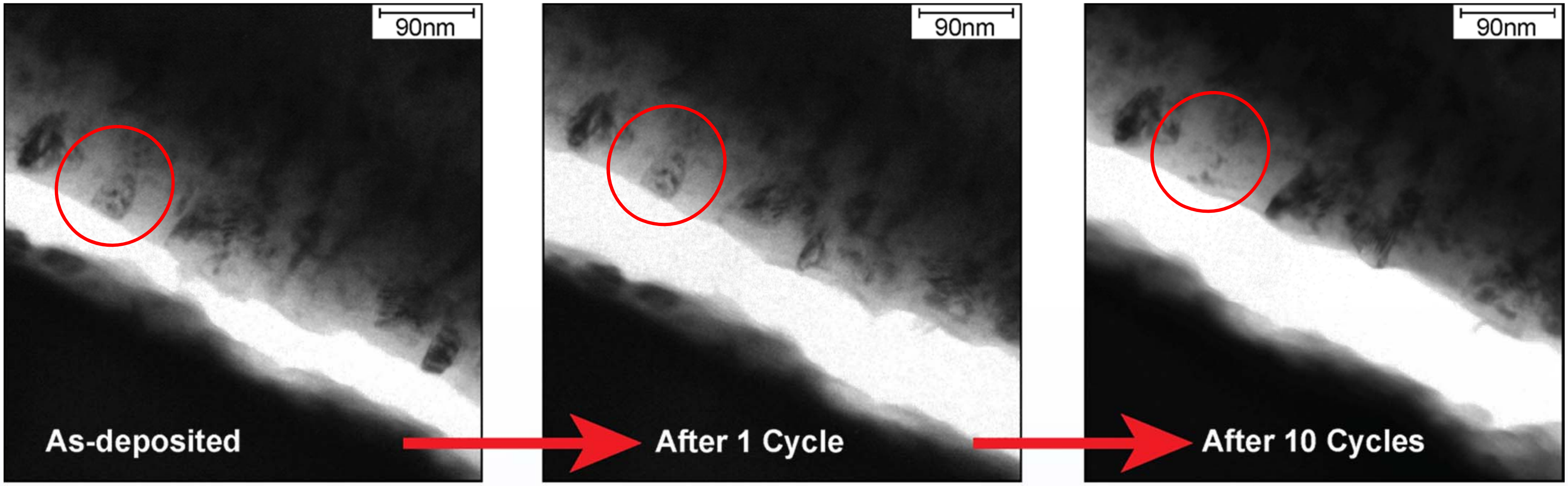
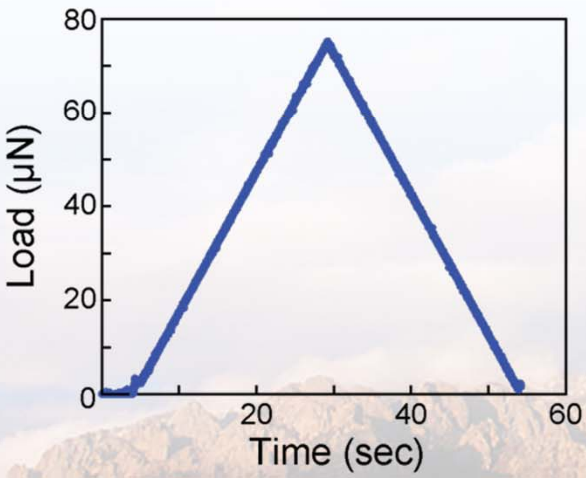
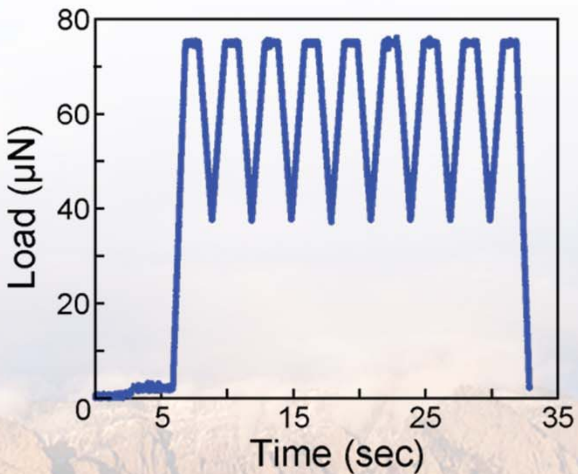
A first experiment appears to show cycle-driven boundary collapse (work ongoing)







375 MPa nominal contact stress

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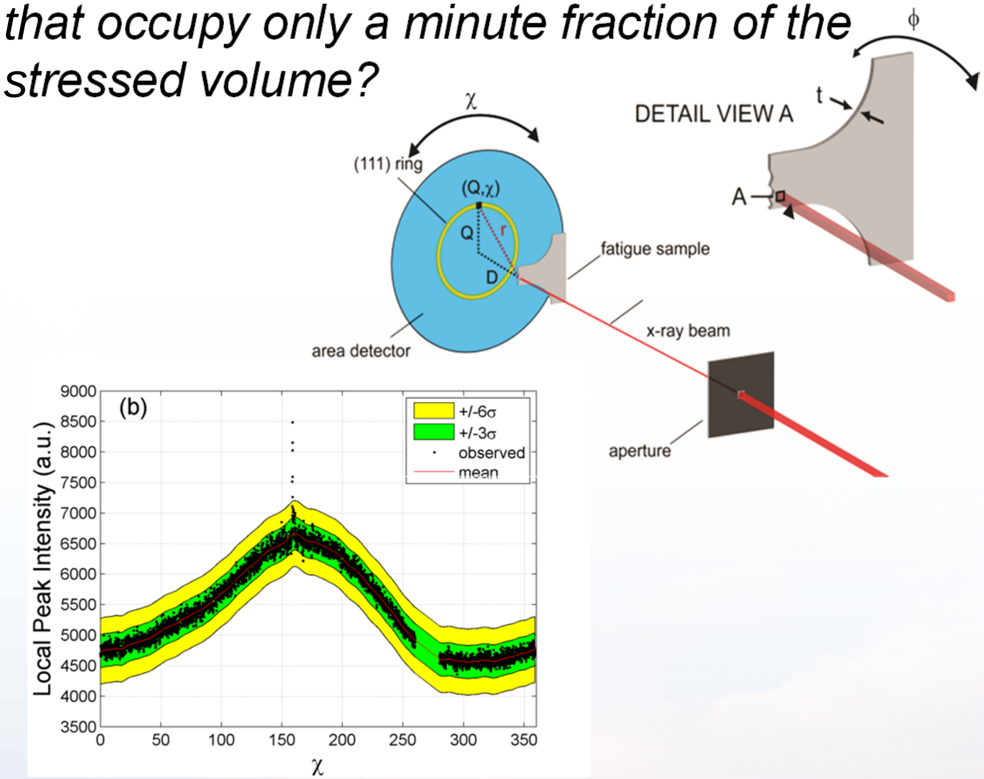
Other Experiments to Elucidate Aspects of Mechanically-Induced Grain Growth in Nanostructured Metals…

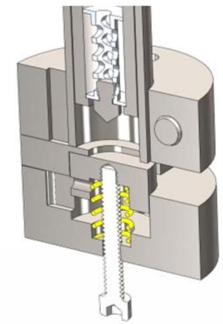
How do we detect the first abnormal grains that occupy only a minute fraction of the stressed volume?

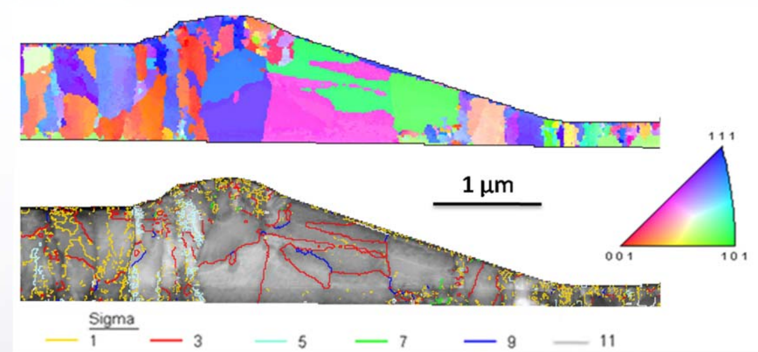


We can identify a single abnormally large grain that occupies <0.00001% of the interrogation volume

{Talk at Spring MRS}

Is mechanically-induced grain growth a thermally-activated process?





Nanotwinned Cu shows more indentation-induced grain growth at 4K than at room temperature.

Brons et al, Scripta Mater, 2013

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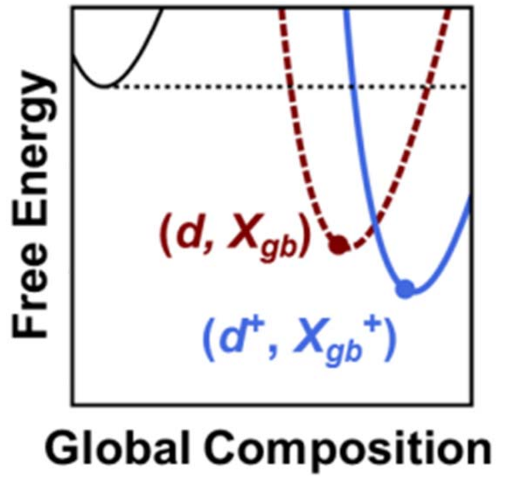
An exciting result from the literature…

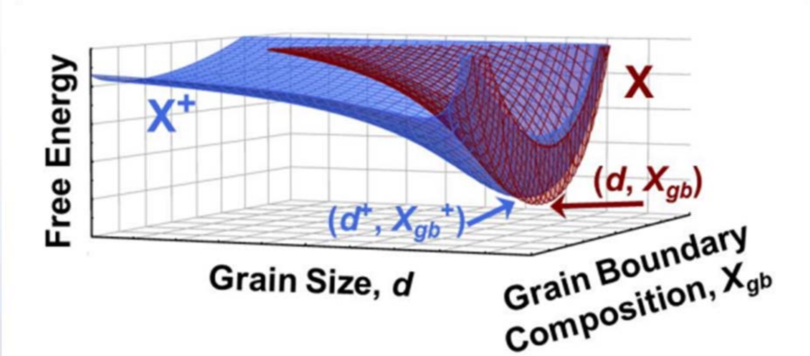
Design of Stable Nanocrystalline Alloys (?)

Tongjai Chookajorn, Heather A. Murdoch, Christopher A. Schuh*, *Science*, 2012



$$\gamma = \gamma_0 - \Gamma(\Delta H^{seg} + kT\ln X)$$

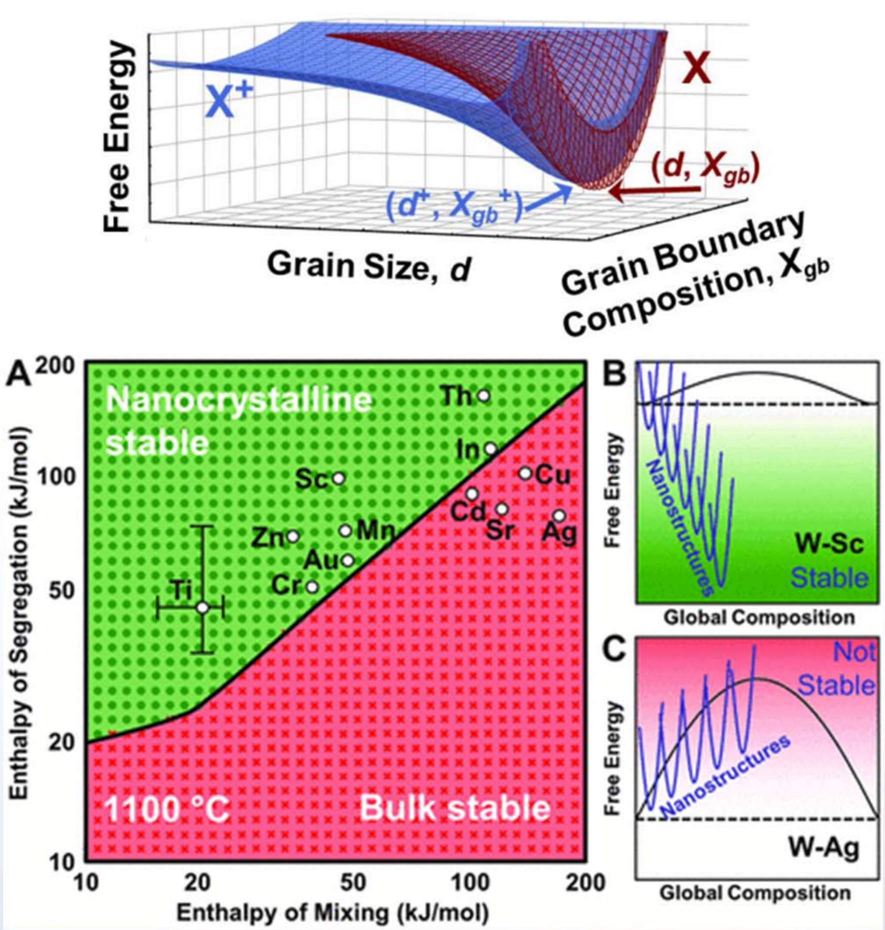
Solute at boundary lowers the enthalpy and raises the entropy

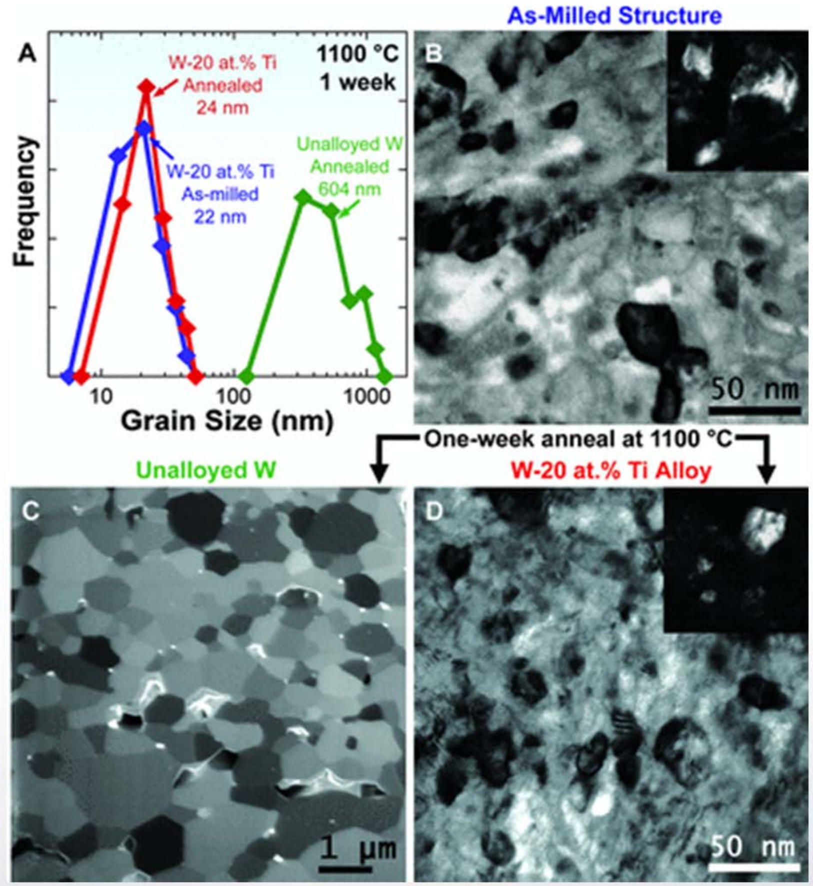


$$\Delta G^{mix} = (1 - f_{gb})\Delta G_c^{mix} + f_{gb}\Delta G_{gb}^{mix} + z\nu f_{gb}(X_{gb} - X_c)\left[(2X_{gb} - 1)\omega_{gb} - \frac{1}{zt}(\Omega^B\gamma^B - \Omega^A\gamma^A)\right] \quad (2)$$

Design of Stable Nanocrystalline Alloys (?)

Tongjai Chookajorn, Heather A. Murdoch, Christopher A. Schuh*, *Science*, 2012





Does thermodynamic stability bear any relevance to stability against mechanically-induced grain growth?

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We are currently seeking new postdocs for this project

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Conclusions

- *Grain-growth in nanocrystalline metals can occur by repeated application of stress, in scenarios where equivalent monotonic stress does not induce grain growth.*
- *When grain sizes are below a ~70 nm, fatigue-induced grain growth appears to be a necessary precursor to crack initiation in Ni-based nanocrystalline alloys.*
- *Under sliding wear conditions, grain growth occurs below the surface whereas grain refinement occurs at the surface. At high strain-rates, delamination occurs between these two layers leading to high friction response.*
- *The precise mechanism behind cycle-induced grain growth is not yet fully understood. Ongoing experiments investigate the defect evolution and temperature dependence.*