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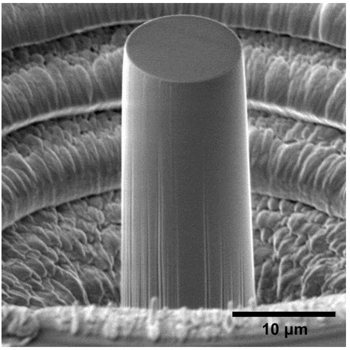

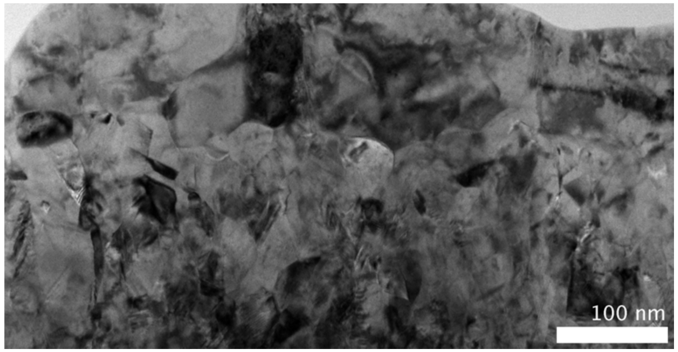

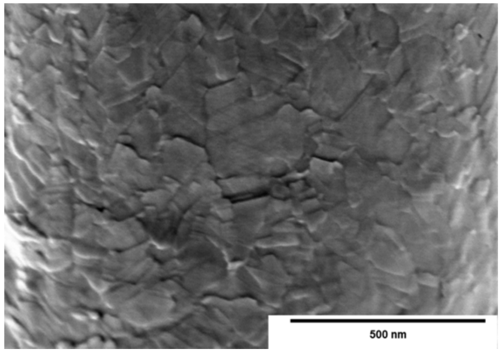

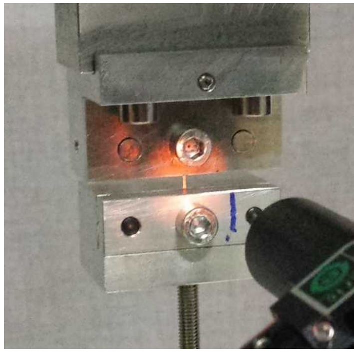

Nanomechanics and Nanometallurgy of Boundaries

Brad Boyce, Stephen Foiles, Khalid Hattar, Blythe Clark,
Fadi Abdeljawad, Dan Bufford, Tim Furnish, Chris O'Brien
Sandia National Laboratories, Albuquerque, NM

U.S. DEPARTMENT OF ENERGY

NNSA National Nuclear Security Administration

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Team:
Nanomechanics and Nanometallurgy of Boundaries

TASK 1
Experimental Observations
of Boundary Evolution

Investigators
Brad L Boyce
Blythe Clark
Khalid Hattar

Postdocs
Tim Furnish
Dan Bufford

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

Investigator
Stephen Foiles

Postdocs
Fadi Abdeljawad
Christopher O'Brien

De Havilland Comet, 1953-4

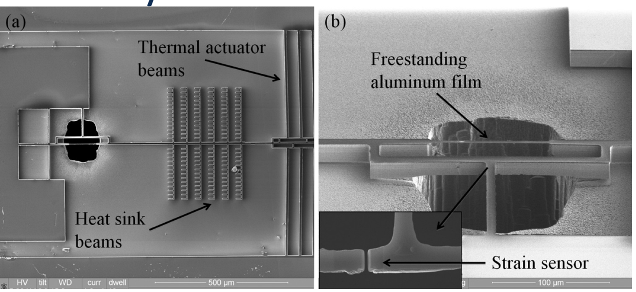
NANO LETTERS
2009
pub

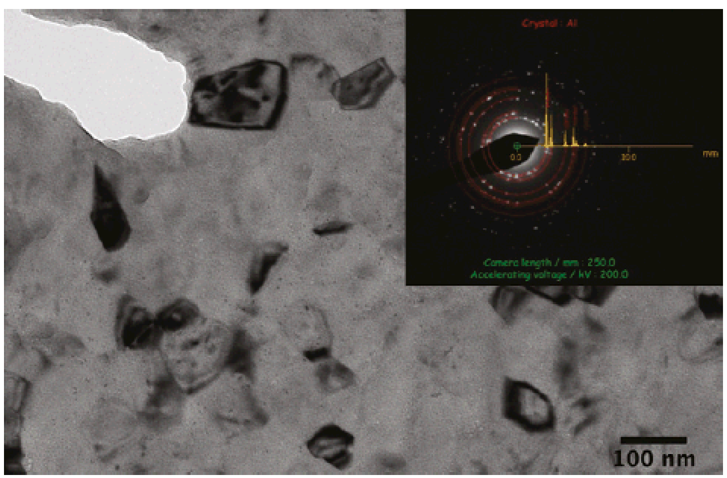
Sandia National Laboratories

Is Stress Concentration Relevant for Nanocrystalline Metals?

Sandeep Kumar,[†] Xiaoyan Li,[‡] Aman Haque,[*,†] and Huajian Gao[*,‡]

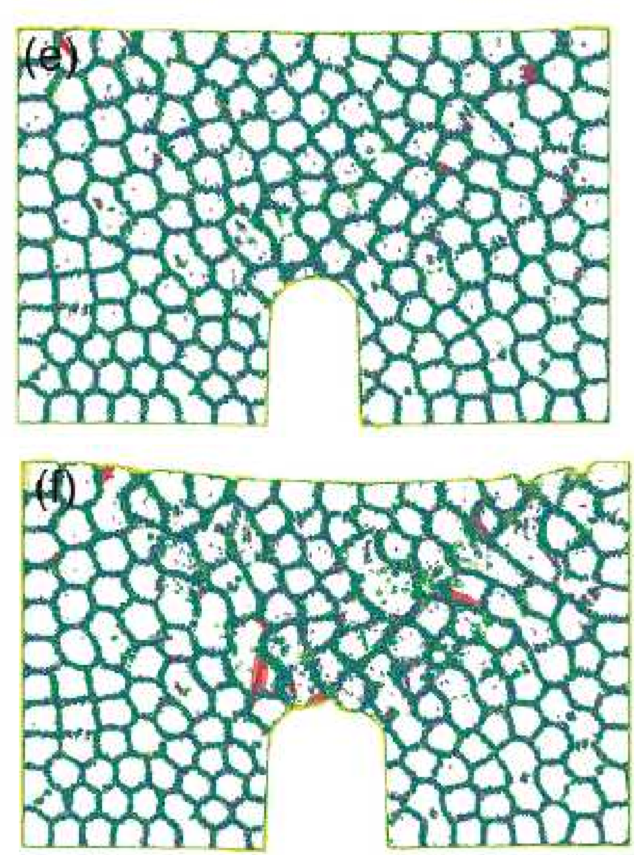
Nanocrystalline Al





5.13% strain

10.52% strain



“experimental evidence of extreme stress homogenization”

2013

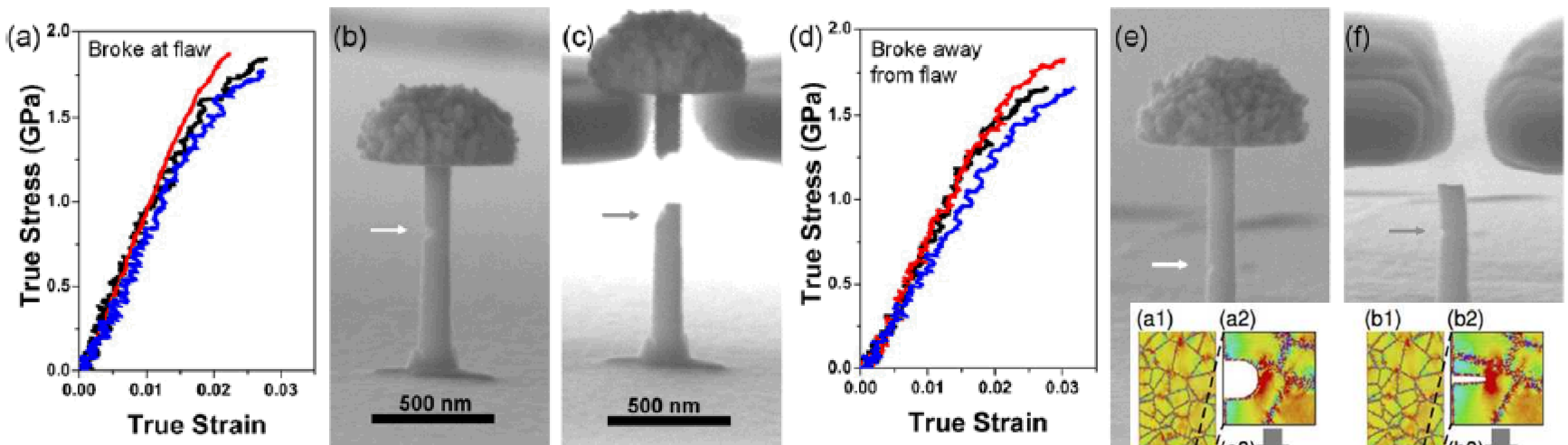
NANO LETTERS
Letter
pubs.acs.org/NanoLett

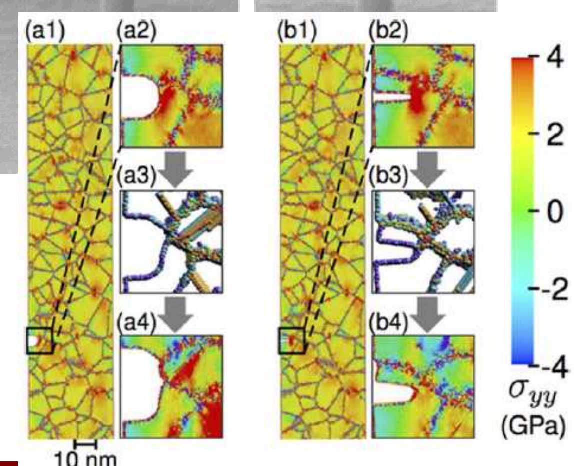
Sandia National Laboratories

Microstructure versus Flaw: Mechanisms of Failure and Strength in Nanostructures

X. Wendy Gu,[†] Zhaoxuan Wu,[§] Yong-Wei Zhang,[§] David J. Srolovitz,[‖] and Julia R. Greer[*,‡]

“no consensus exists on the effect of flaws on fracture at the nanocale”



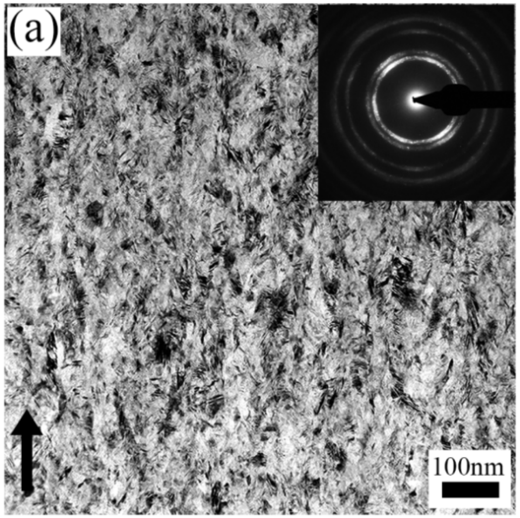


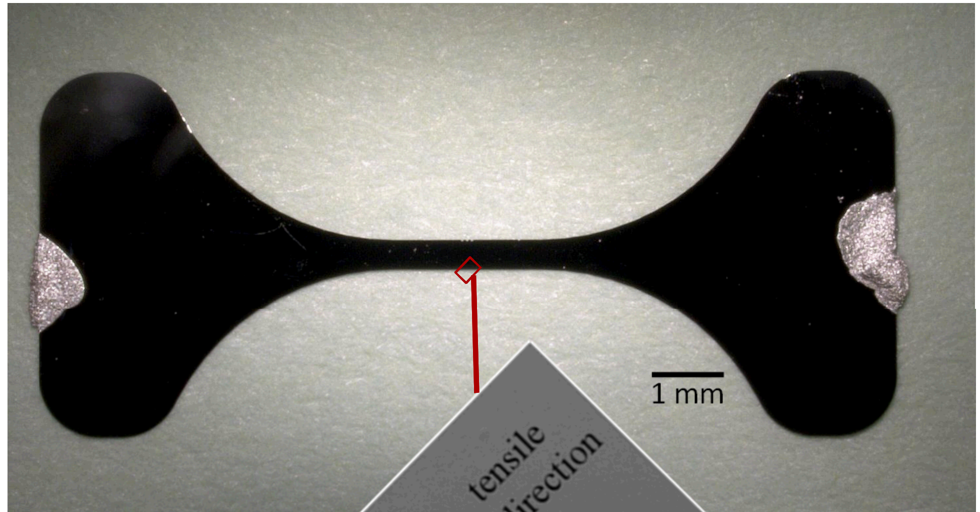
Nanocrystalline Pt

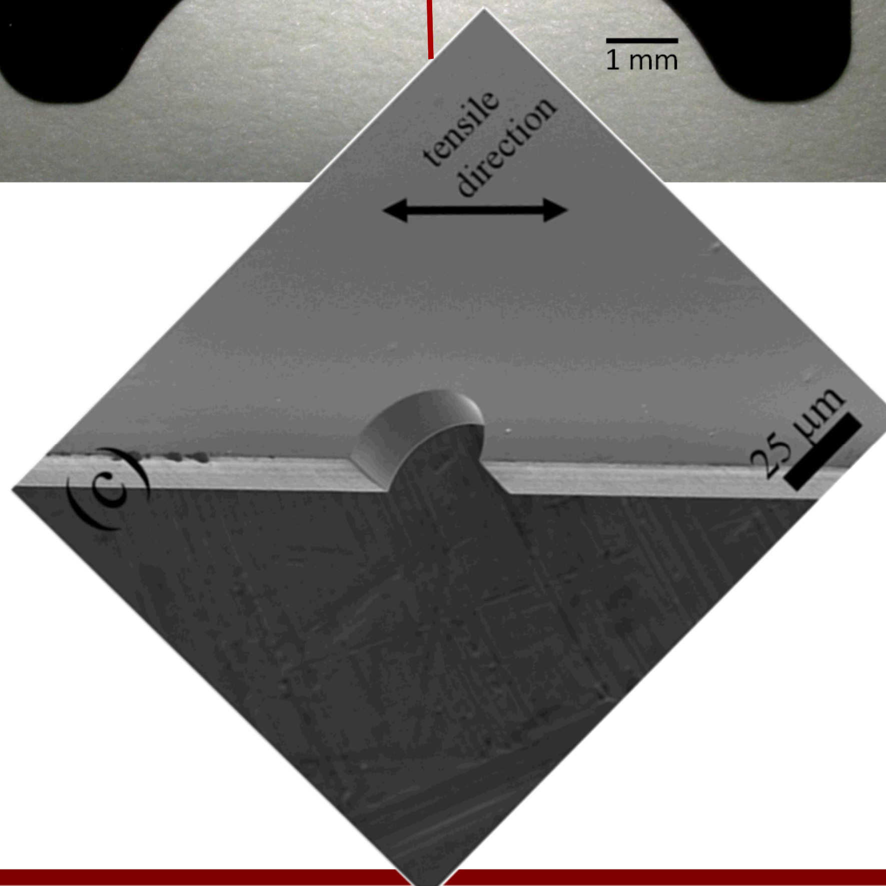
Notches are no worse than microstructure!

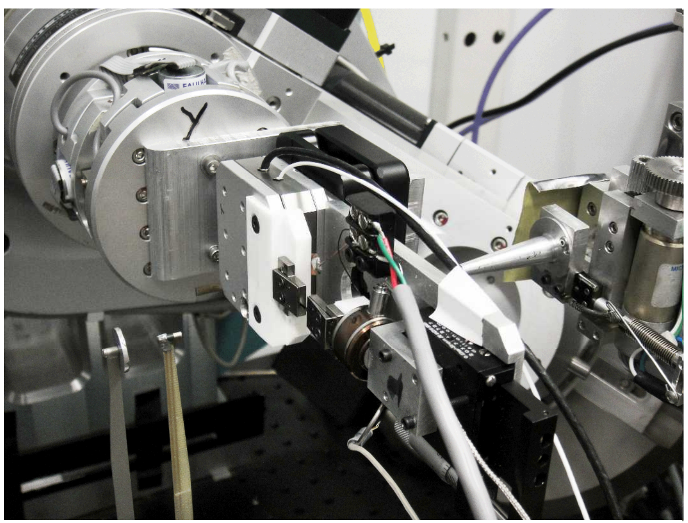
Neither of these studies mention the ratio of notch size to grain size, or the “sampling volume” of the notch…

Nanocrystal Thin Film Notch Fatigue



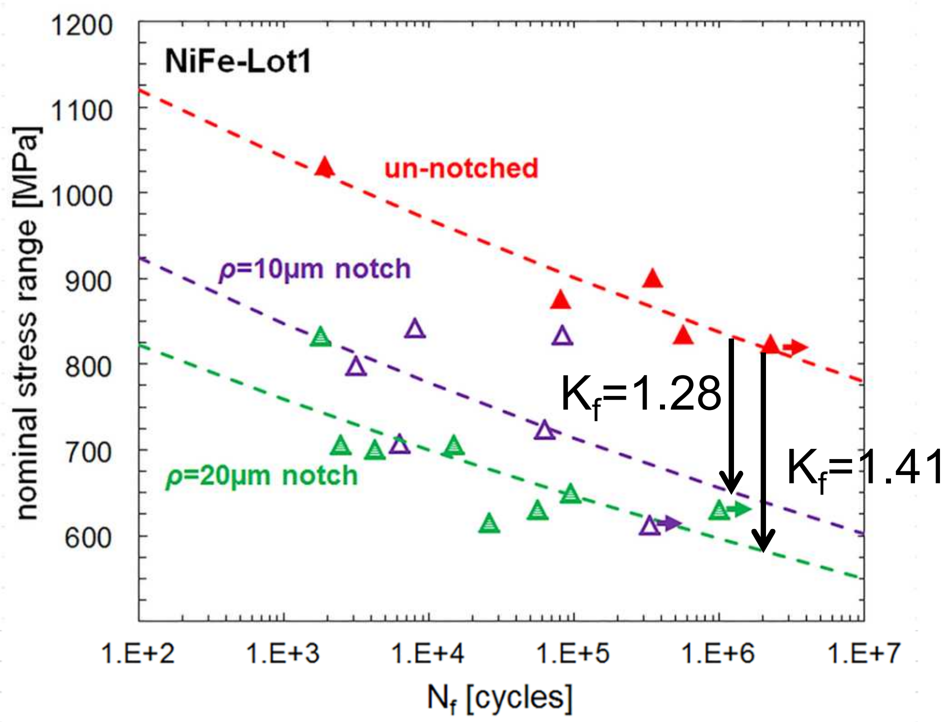






Furnish, Sharon, Boyce, Arrington, Pillars and Clark, *submitted*

Result: Effect of notch on S-N fatigue



$$\Delta\sigma = A\left(N_f\right)^b$$

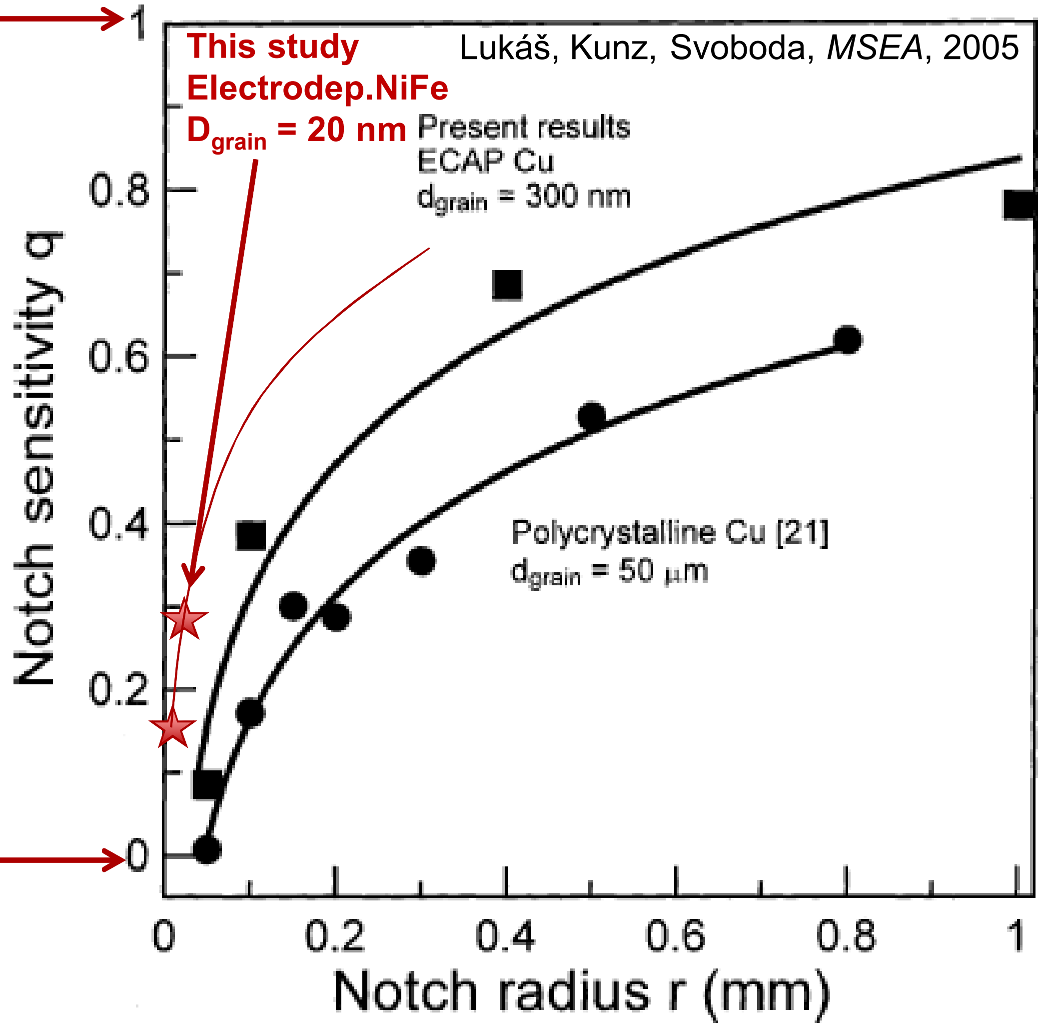
Furnish, Sharon, Arrington, Pillars Clark, and Boyce *submitted*

Notch effects diminish as two length scales converge

Notches obey elastic stress concentration factor

$$q = \frac{(K_f - 1)}{(K_t - 1)}$$

Notches are Irrelevant!!!



Nanocrystalline metals are ***more*** sensitive to stress concentrations than their coarse grained counterparts!!!

Furnish, Sharon, Arrington, Pillars Clark, and Boyce *submitted*

Models go where experiments can not:
nominally identical grain topology but different grain sizes

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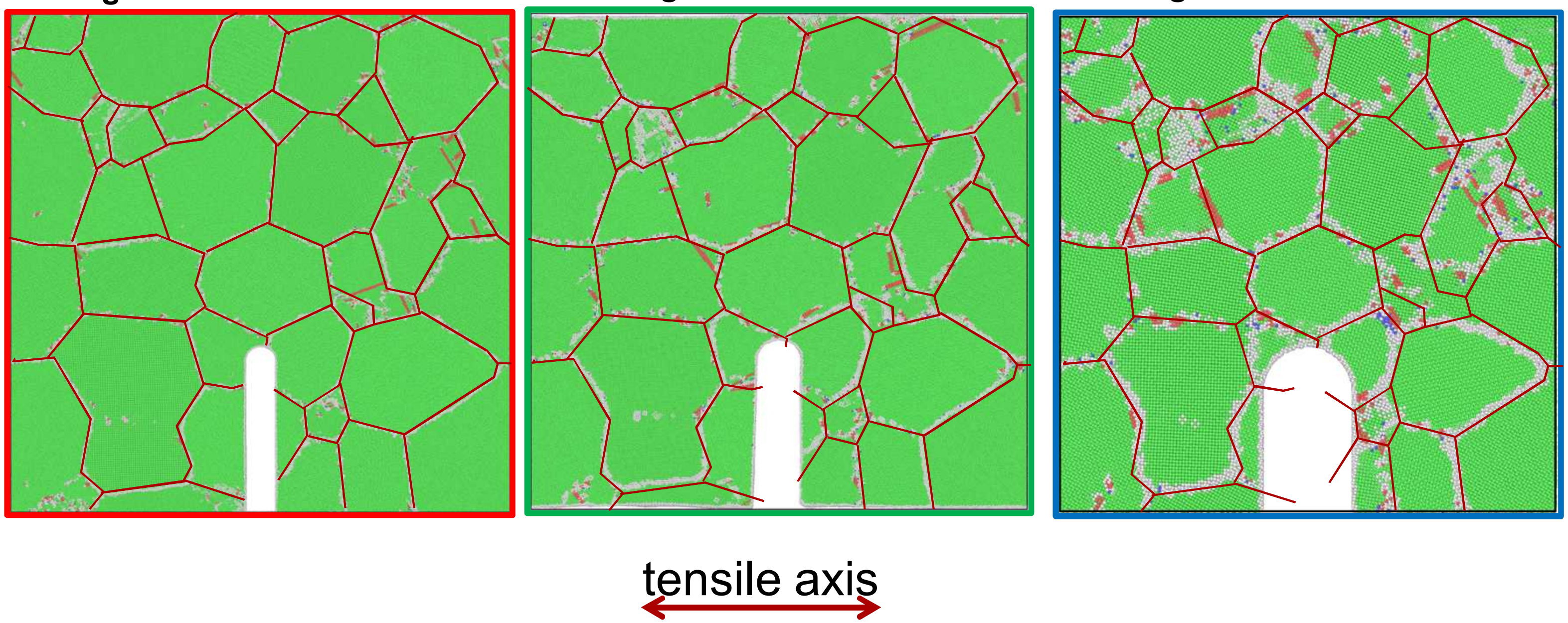
Grain size = 15 nm
Notch:grain = 1:3

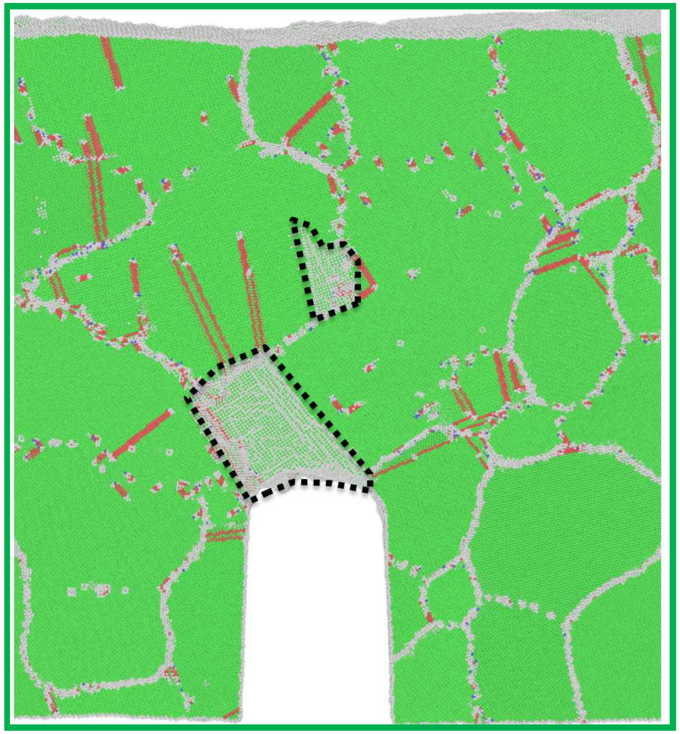
Grain size = 10 nm
Notch:grain = 1:2

Grain size = 5 nm
Notch:grain = 1:1

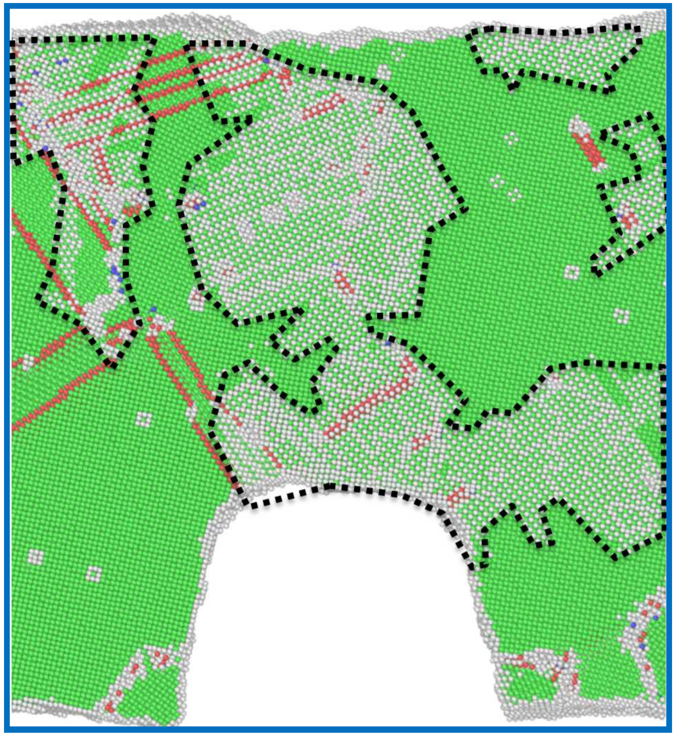


Unlike conventional Vornoi tesselation construction for virtual microstructure, a phase field approach to grain growth enables realistic grain curvature and triple junctions.

Do small grain sizes really provide "extreme stress homogenization"???



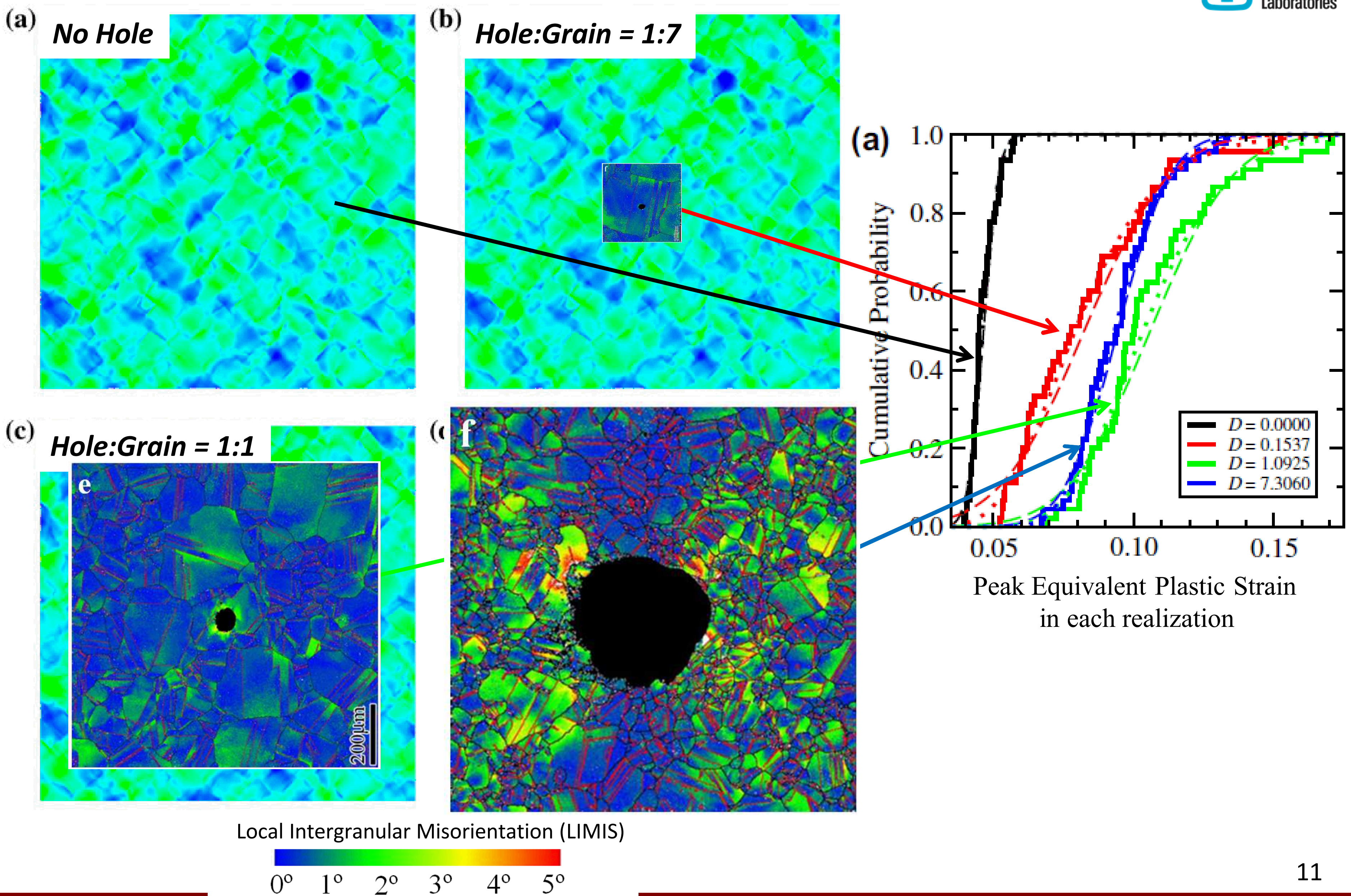
10 nm grain size



5 nm grain size

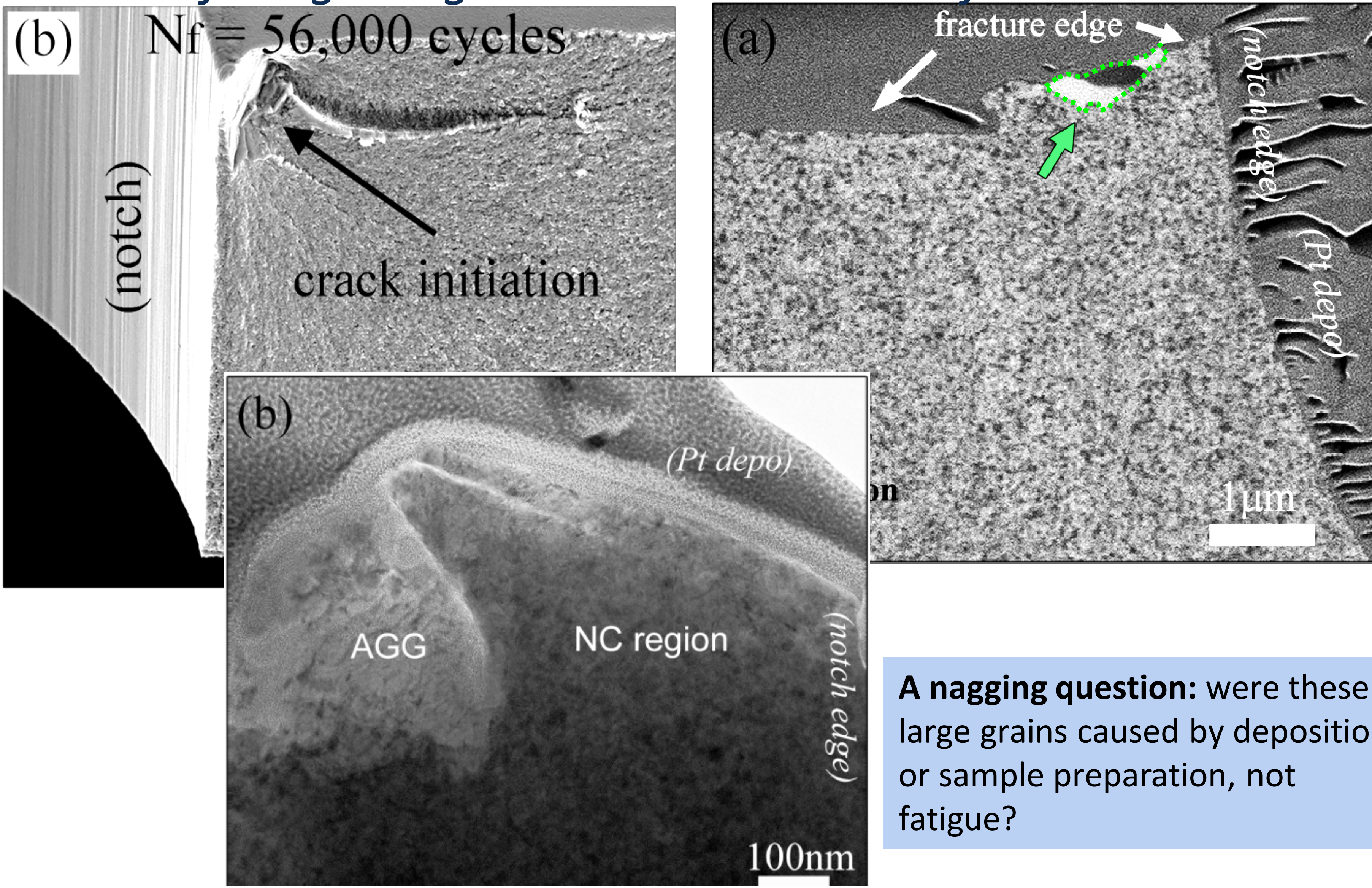
tensile axis

The ratio of two length scales matter!!!



Battaile, Brewer, Emery and Boyce, *Philosophical Magazine*, 2015

Fractography and FIB cross-section confirm grain growth at the source of crack initiation

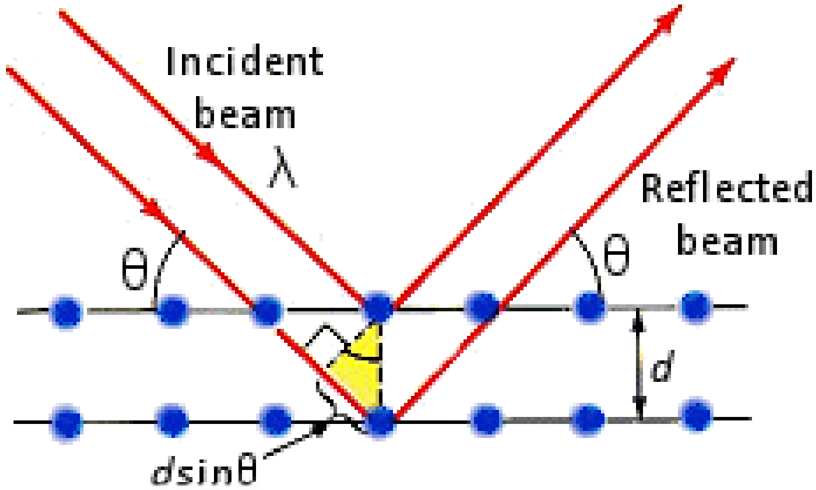


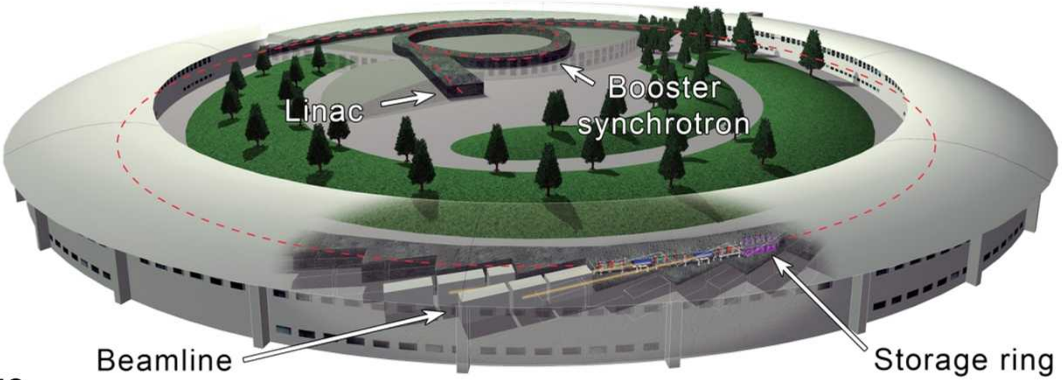
A nagging question: were these large grains caused by deposition or sample preparation, not fatigue?

Furnish, Sharon, Arrington, Pillars Clark, and Boyce *submitted*

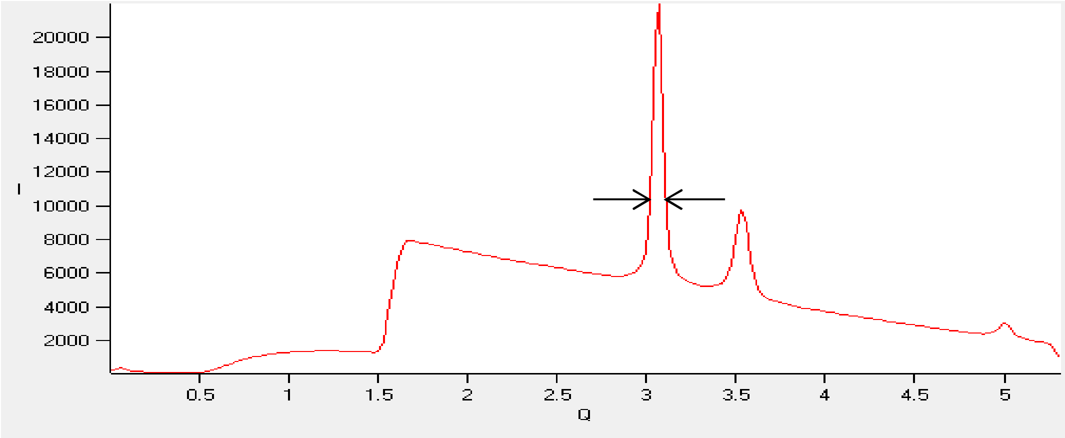
X-rays, Diffraction, & Synchrotrons

Wilhelm Konrad Roentgen's 1896 x-ray image of his wife's hand, Nobel Prize 1901





Diffraction-based grain size measurements rely on observation of peak broadening



Scherrer Formula (1918) $$B\cos\theta = \frac{K\lambda}{d_{avg}}$$

Williamson-Hall (1953) $$B\cos\theta = \frac{K\lambda}{d_{avg}} + \eta\sin\theta$$

Warren-Averbach (1959) $$A_L = A_L^S A_L^D = A_L^S \exp[-2\pi^2 L^2 g^2 \langle \varepsilon_{g,L}^2 \rangle]$$

Can we observe the large grains using in-situ transmission synchrotron x-ray diffraction?

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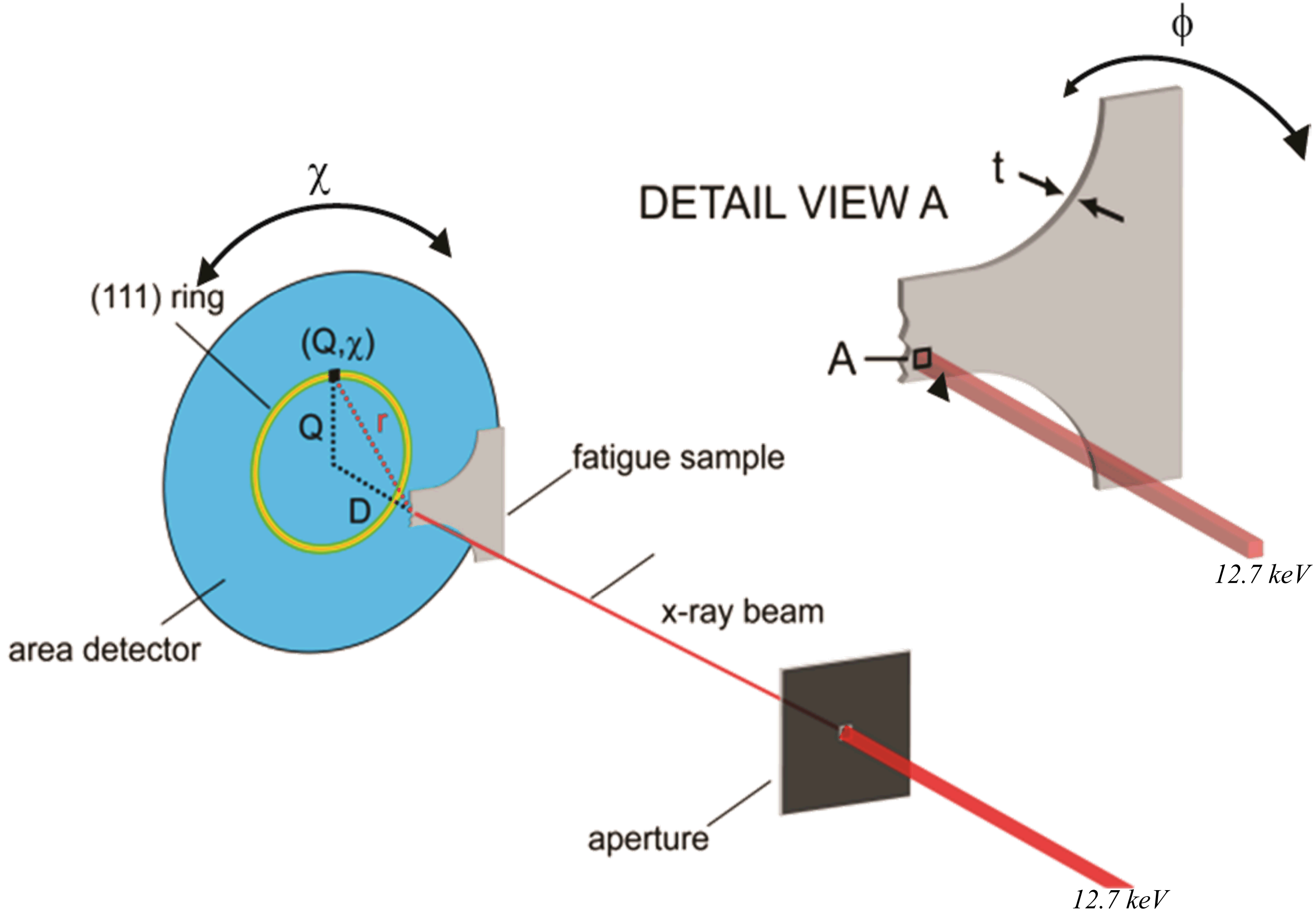
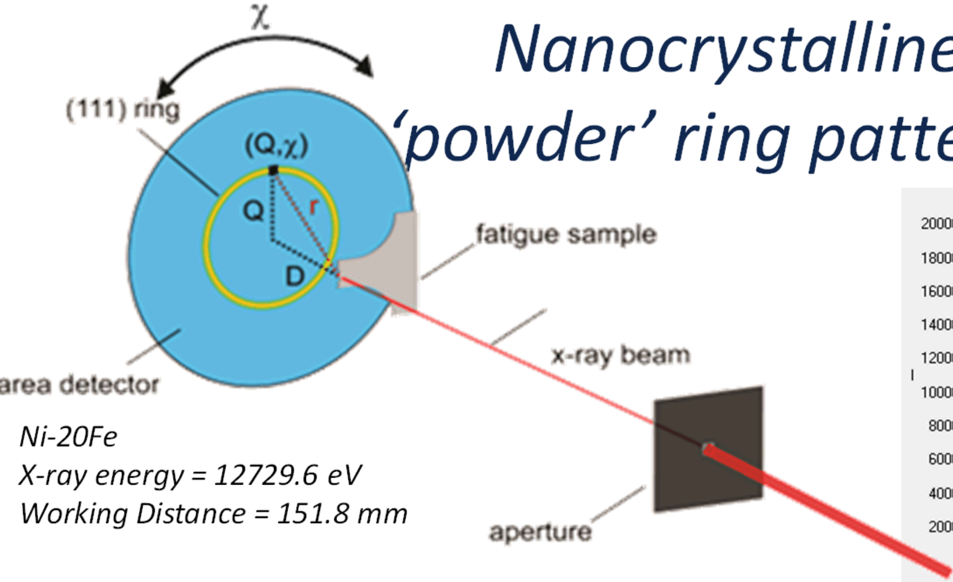
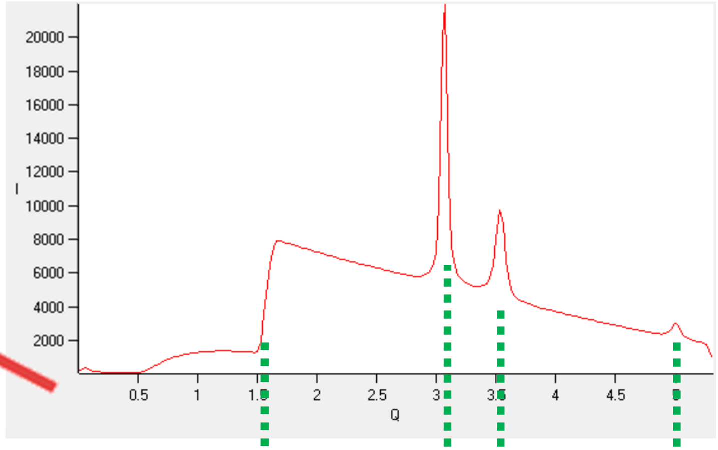


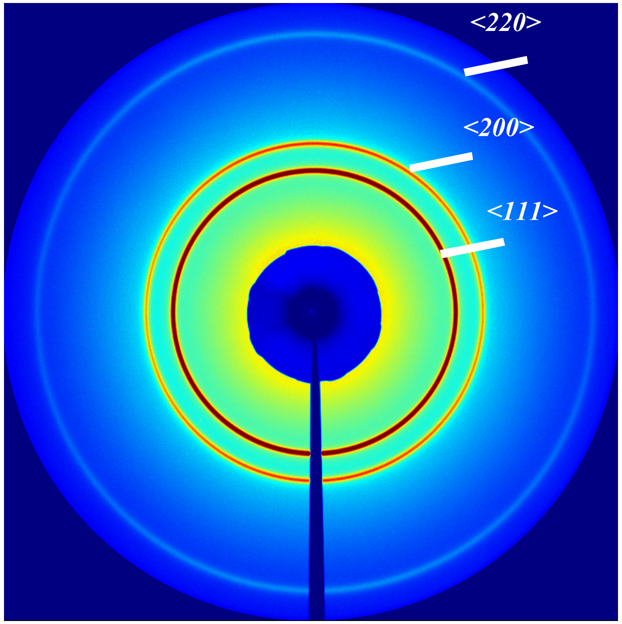
Boyce, Furnish, Padilla, Van Campen, Mehta, *J. Mater. Sci.*, 2015

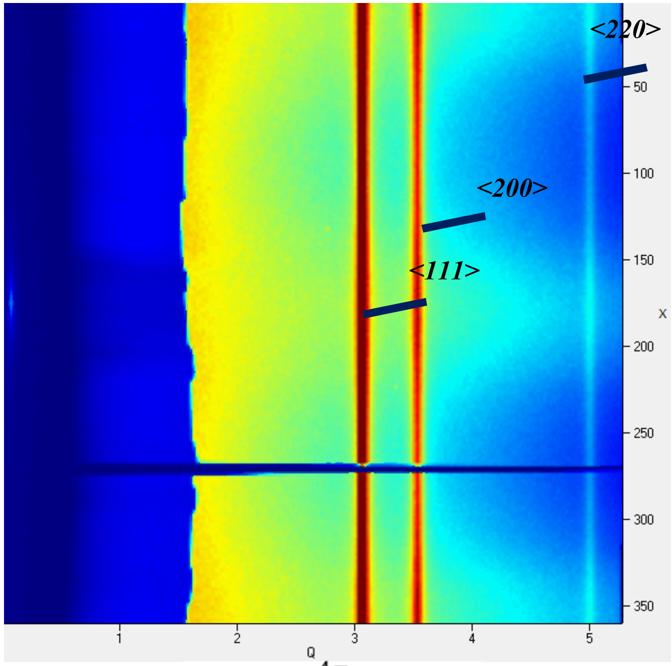
Nanocrystalline metals produce ‘powder’ ring pattern on area detector



Ni-20Fe
X-ray energy = 12729.6 eV
Working Distance = 151.8 mm



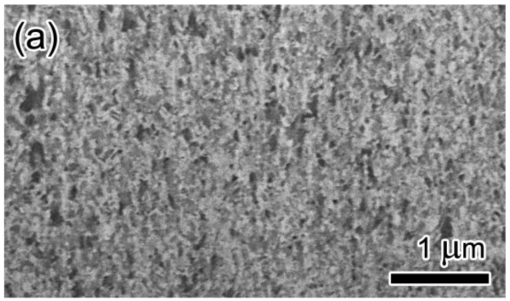


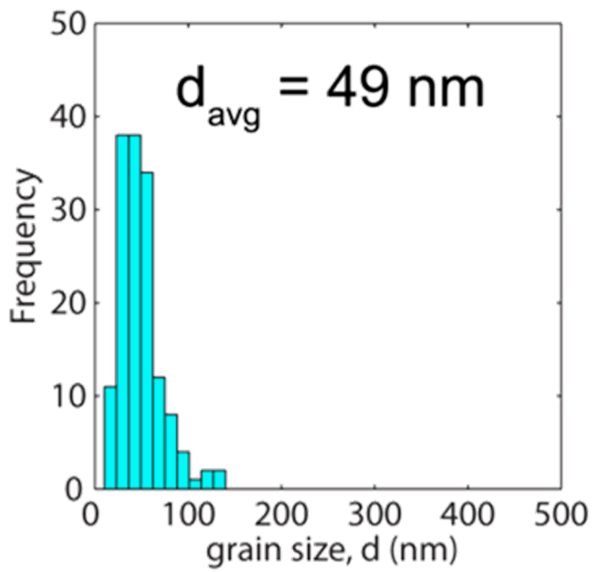


$$Q = \frac{4\pi}{\lambda}\sin\theta$$

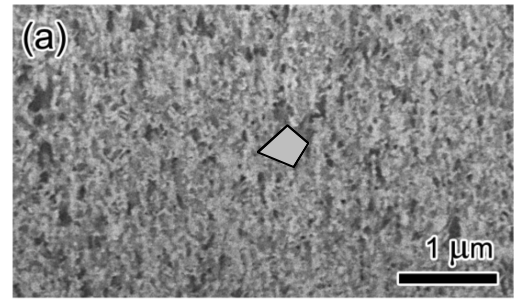
The crux of the problem: needle-in-a-haystack: the onset of abnormal grain growth has an imperceptible effect on the average grain size

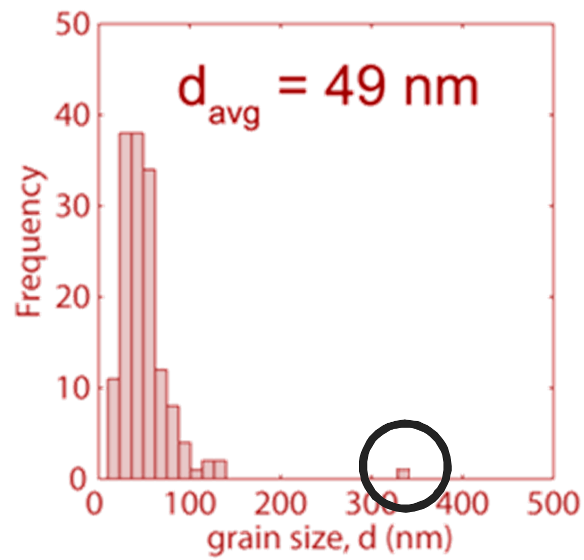
Before abnormal grain growth





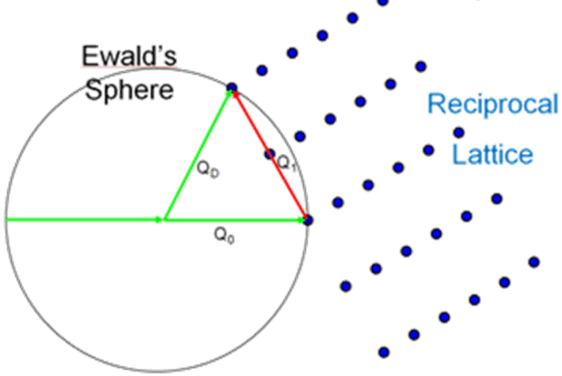
After abnormal grain growth



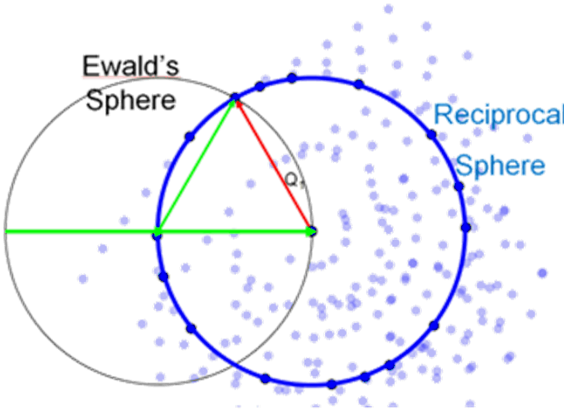


A hypothesis: a large grain in a sea of small grains should diffract like a single crystal superimposed on a polycrystal

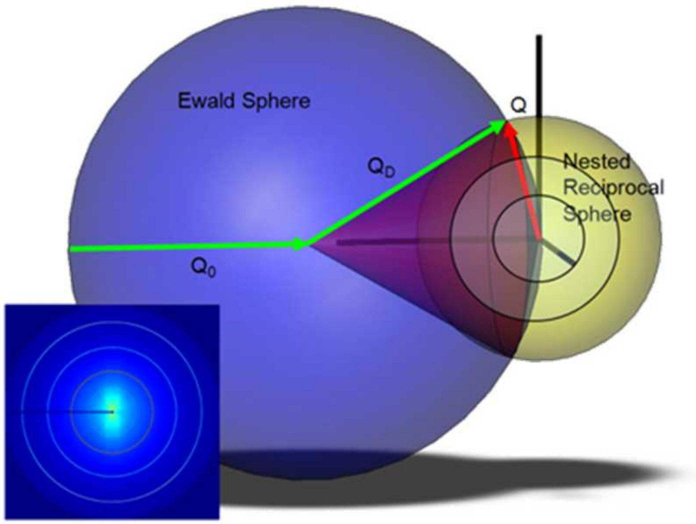
Single Crystal



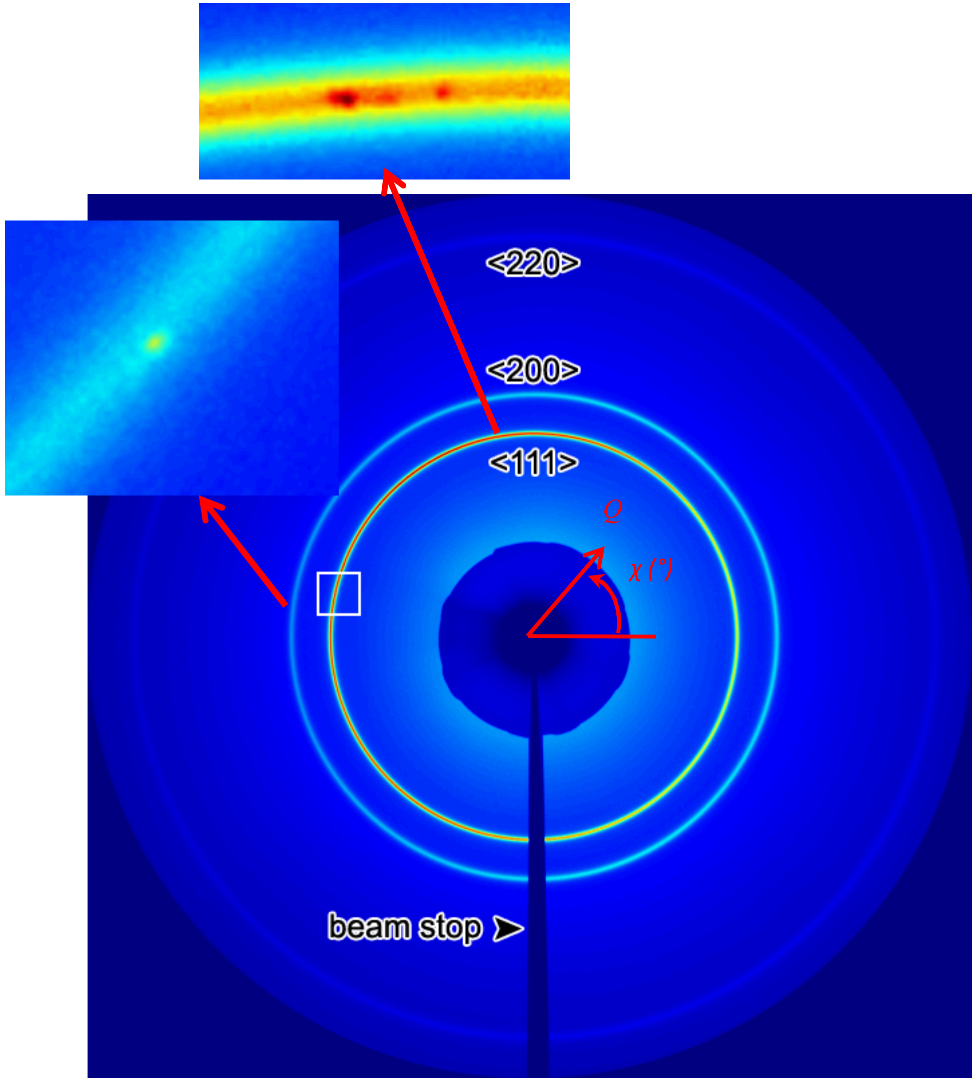
Oligocrystal



Polycrystal

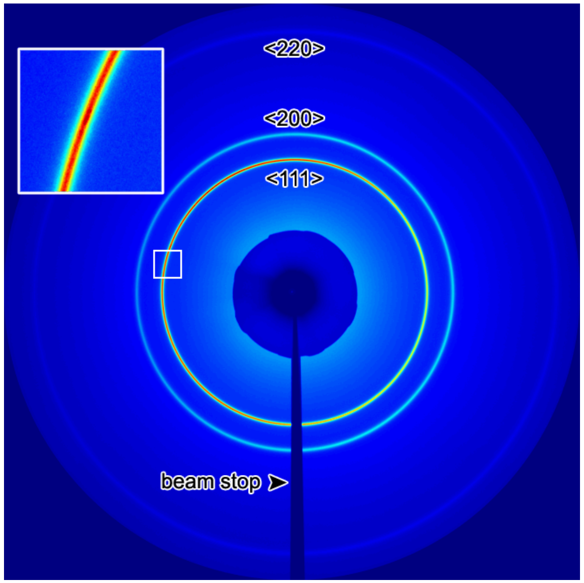


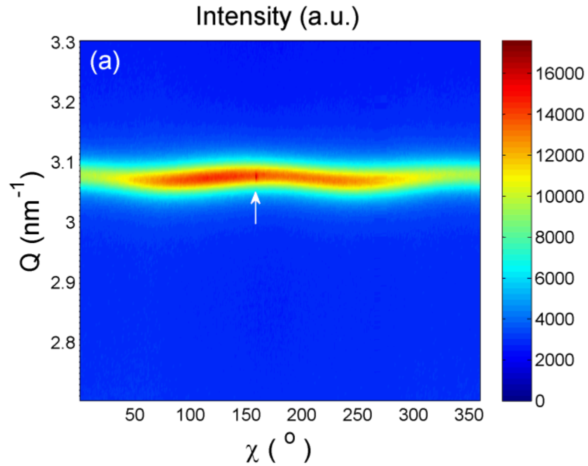
Preliminary Observation: A ‘spike’ in the diffraction ring.

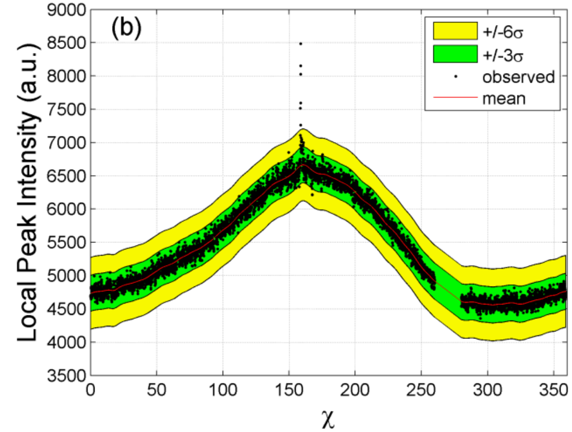


How do we know these spikes are truly statistically significant anomalies and not just noise?

An abnormal intensity spike is the single crystal signature that stems from an isolated large grain

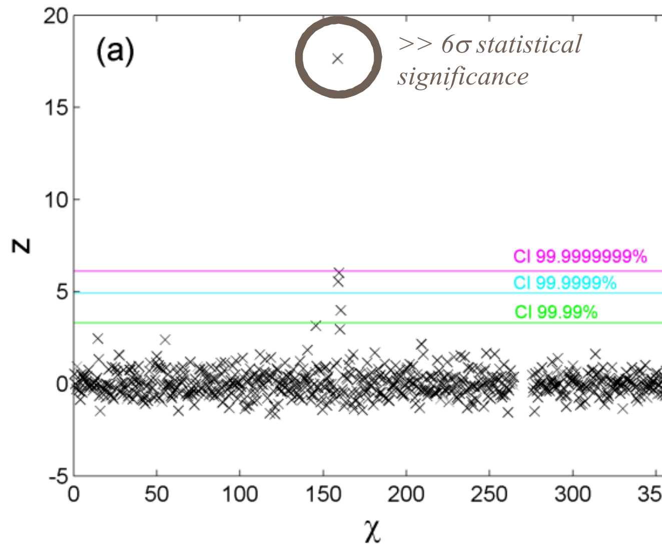






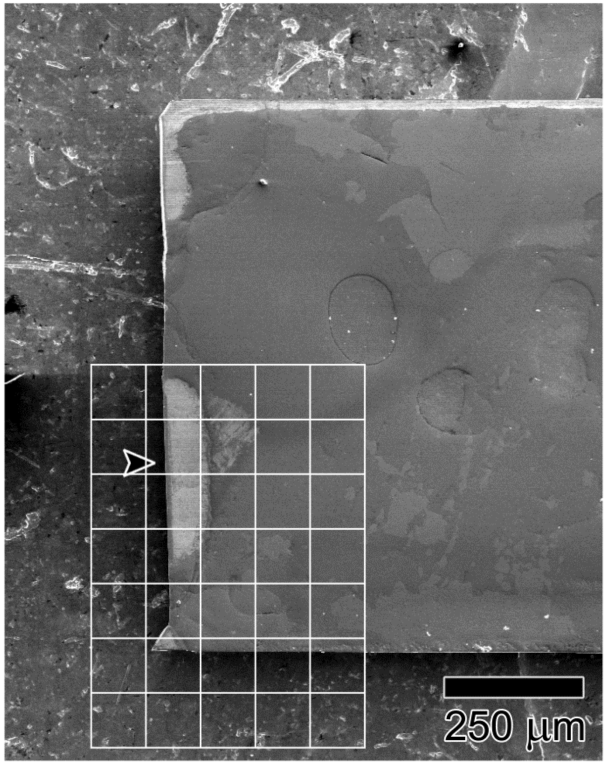
$$\sigma = \sqrt{\frac{1}{N}\sum_{i=1}^{N}(X_i - \mu_i)^2}$$

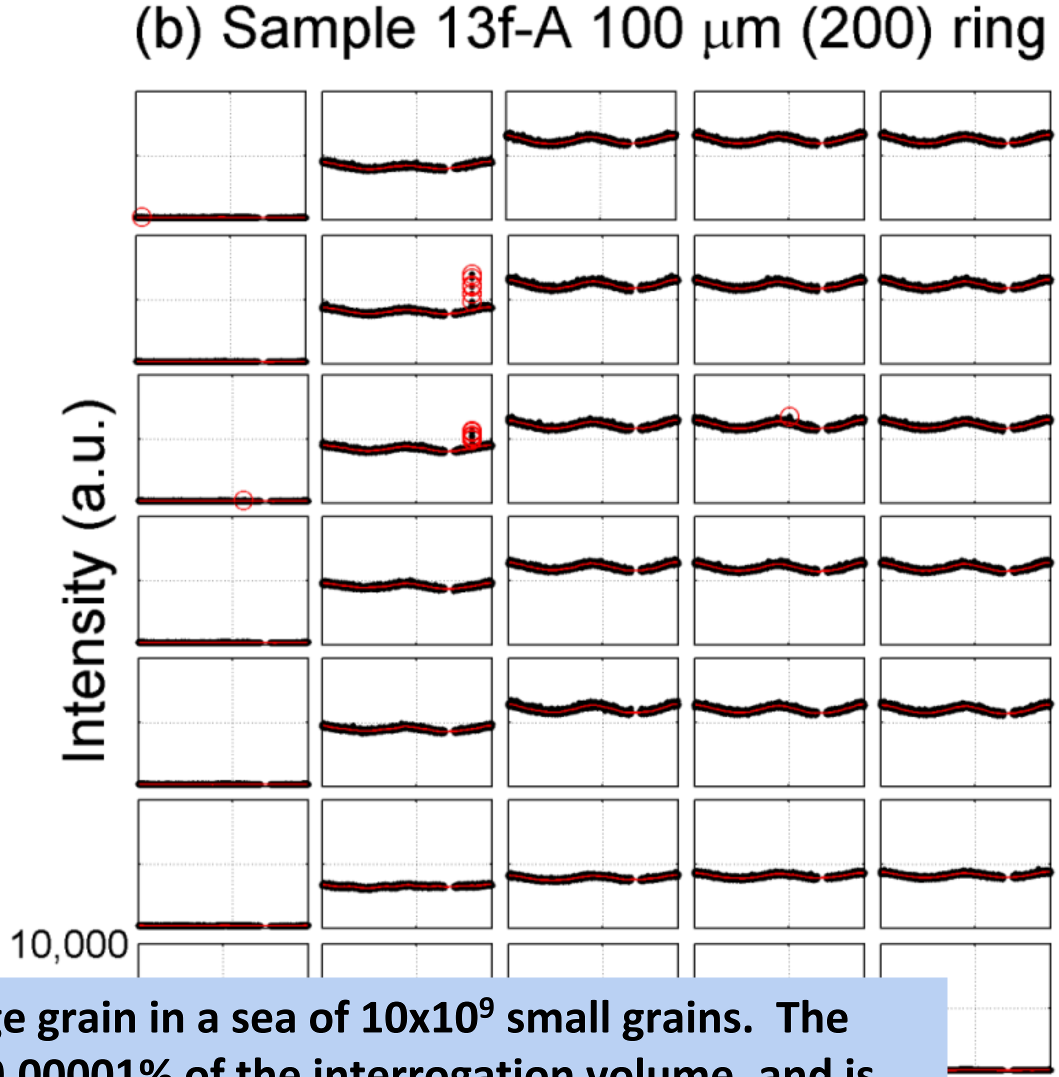
$$X_i = I(\chi_i) - \mu(\chi_i)$$



Boyce, Furnish, Padilla, Van Campen, Mehta, *J. Mater. Sci.*, 2015

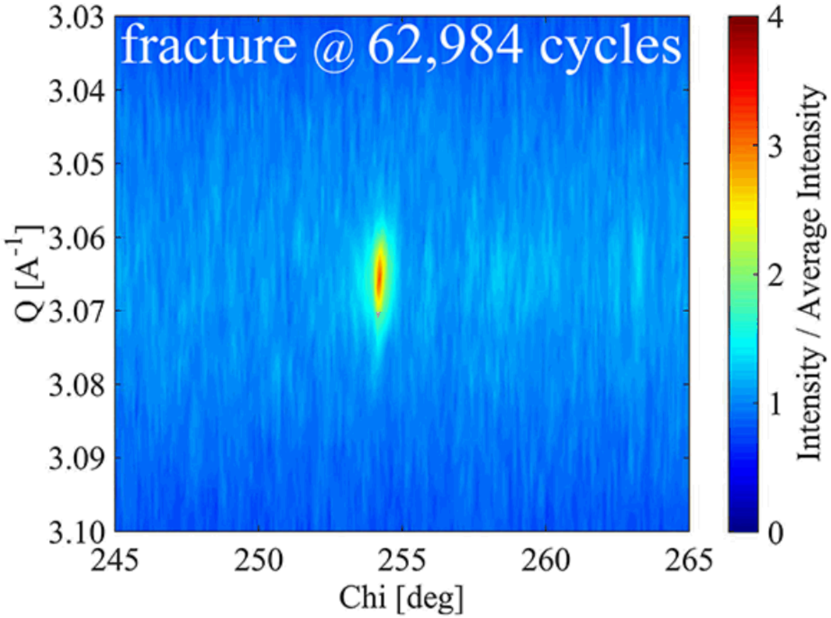
Confirmation: the intensity spike occurs in the known location of grain growth and nowhere else

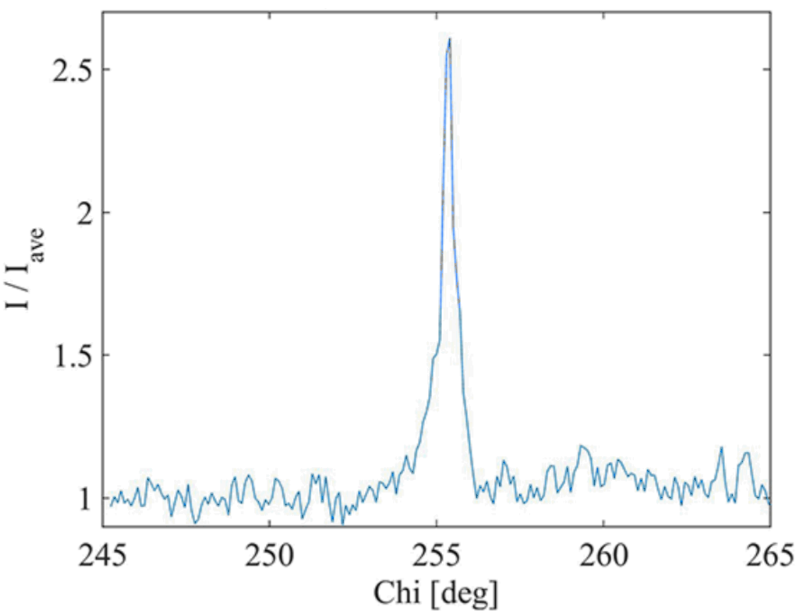




We've identified 1 large grain in a sea of 10×10^9 small grains. The large grain occupies ~0.00001% of the interrogation volume, and is identified with a statistical confidence >> 99.9999998% (6σ).

The definitive experiment: Detecting the onset of grain growth during fatigue





Watching fatigue-induced grain evolution directly....

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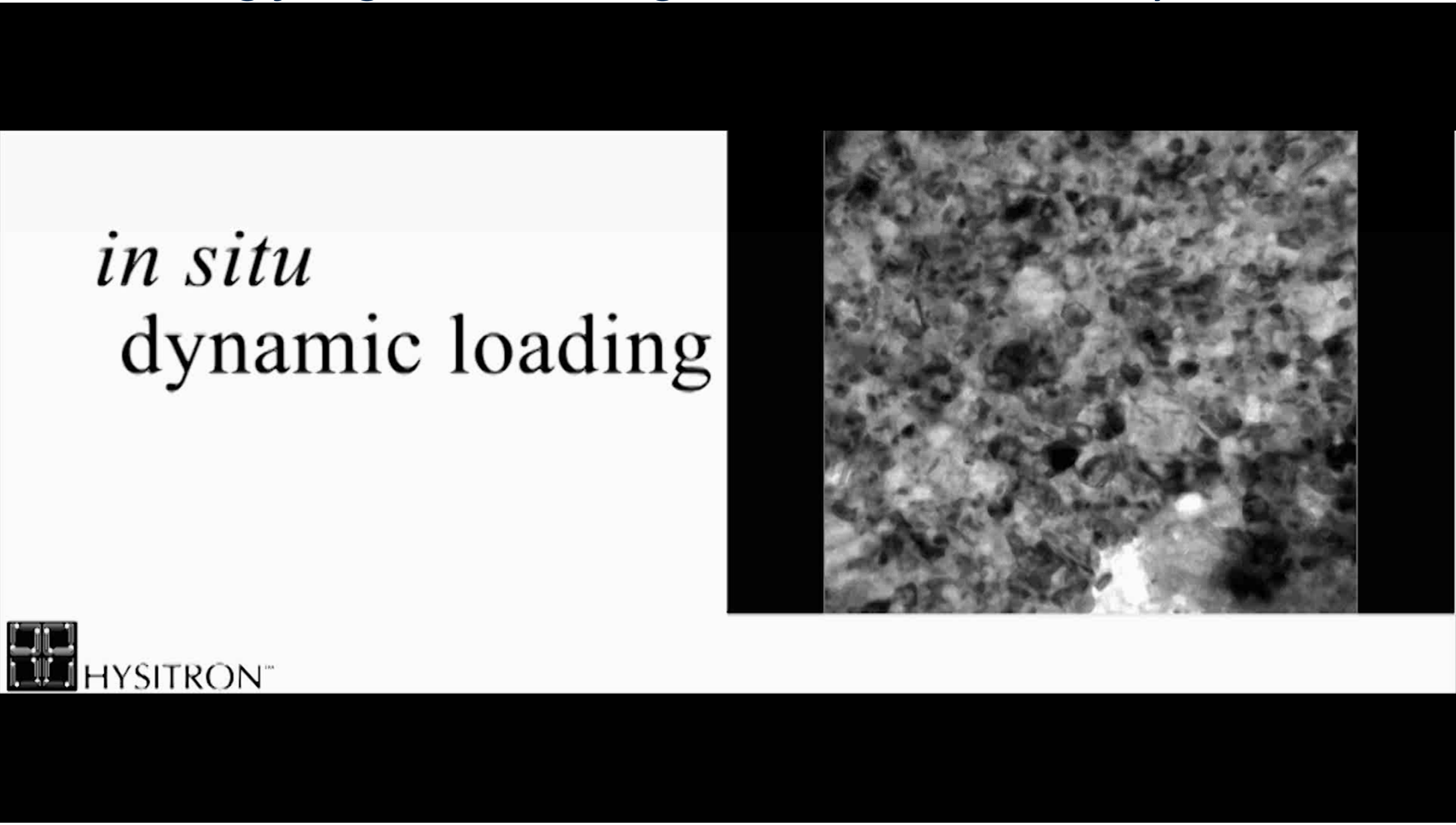

See Poster by Khalid Hattar: Observing grain growth through mechanical, thermal, and irradiation stimuli

How could <u>abnormal</u> grain growth occur at such low temperatures???

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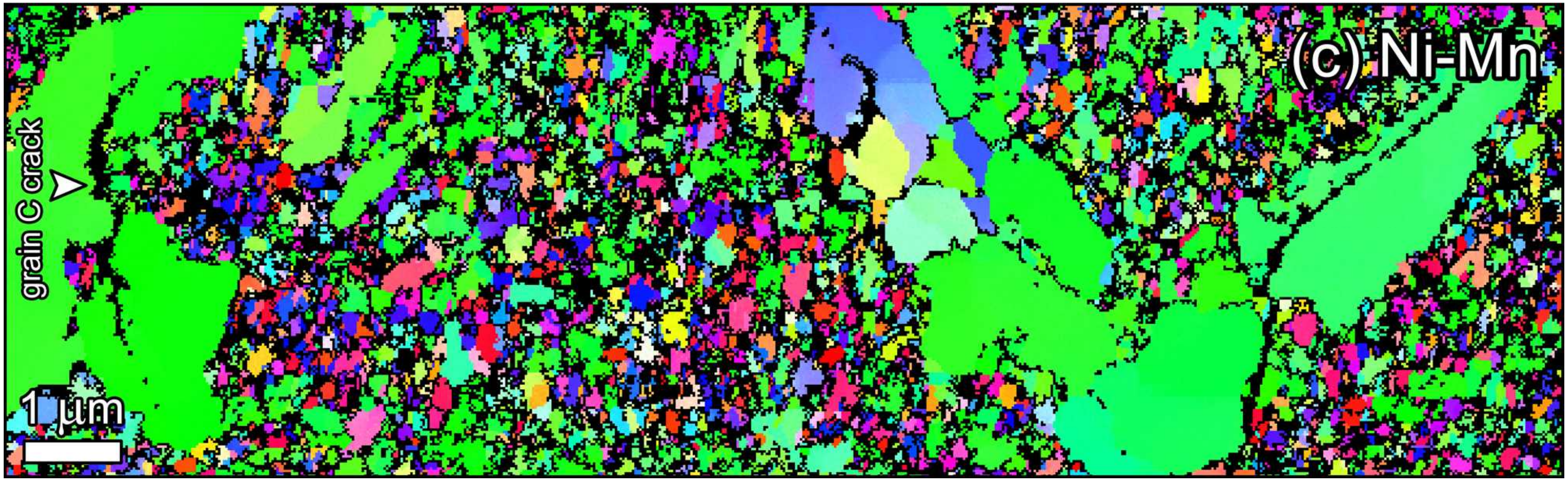
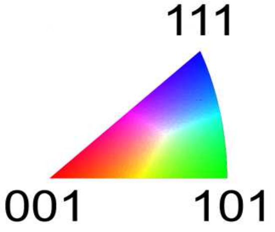
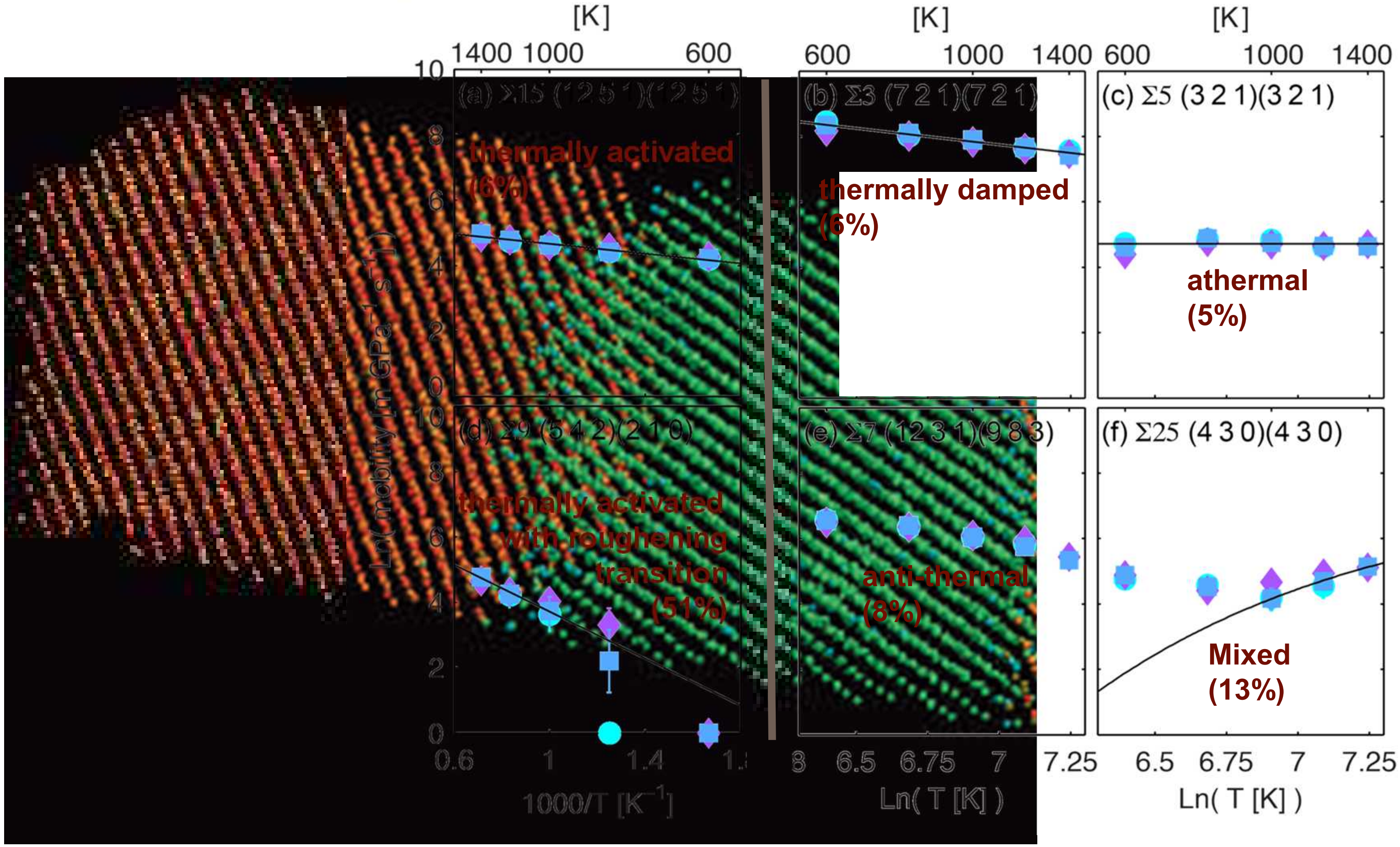


What causes these few grains to grow so quickly at room temperature?

Hypothesis: a few grain boundary types have a distinct mobility advantage



Hypothesis: a small fraction of grain boundaries have exceptional mobility



Mechanisms of antithermal grain boundary motion

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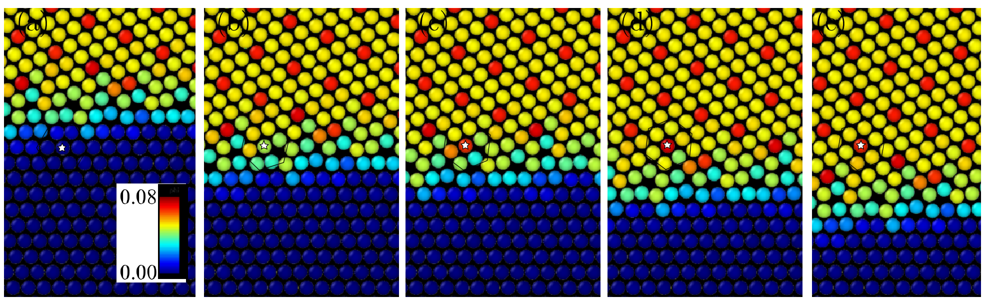
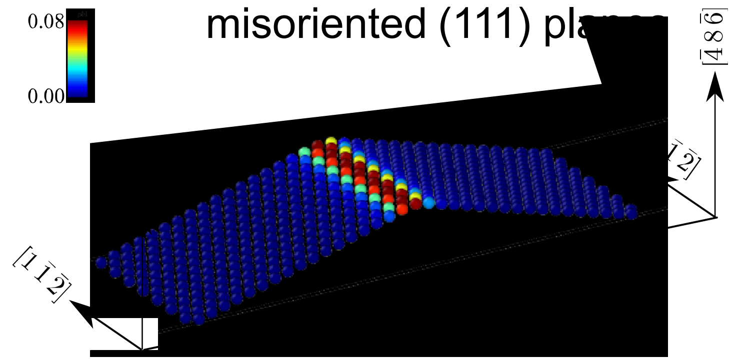
- Commonly present in ∑3 and ∑7 boundaries, which contain a common (111) plane, but not all behave antithermally, nor is this motion mechanism exclusive to these orientations
- The mechanism is qualitatively different from that encountered in thermally activated boundaries
  - Mechanism is based on the coordinated motion of many atoms
  - Often involves shuffling or rotation on a common (111) plane (or nearly co-planar)
  - Often involving rotation around a common center, even out-of-plane
- The existence of out-of-plane coordinated motion may make it difficult to establish a simple rule governing whether or not a boundary moves in a non-thermally activated manner.

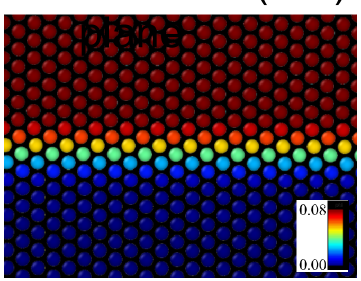
1. In-plane rotation about a fixed atom on a common (111) plane



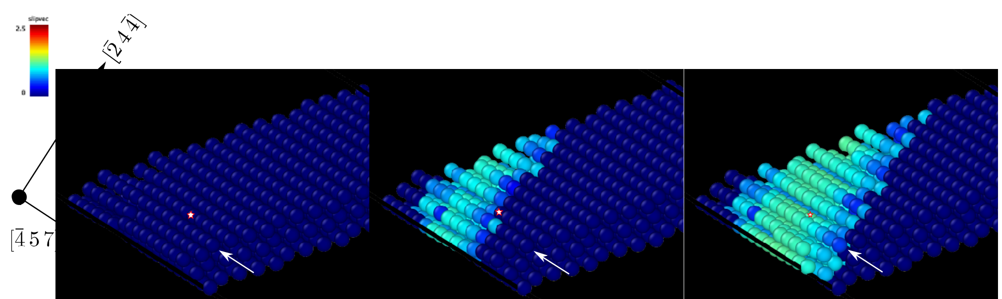
3. Rotation between two misoriented (111) pla

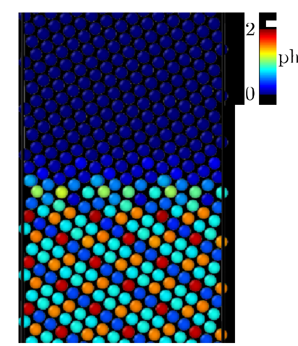


2. In-plane rotation without center on common (111) plane



4. Out-of-plane rotation about a common center





***From Someone Else's Work**: Srikanth Patala and Eric Homer's revelation on representing grain boundary properties.*

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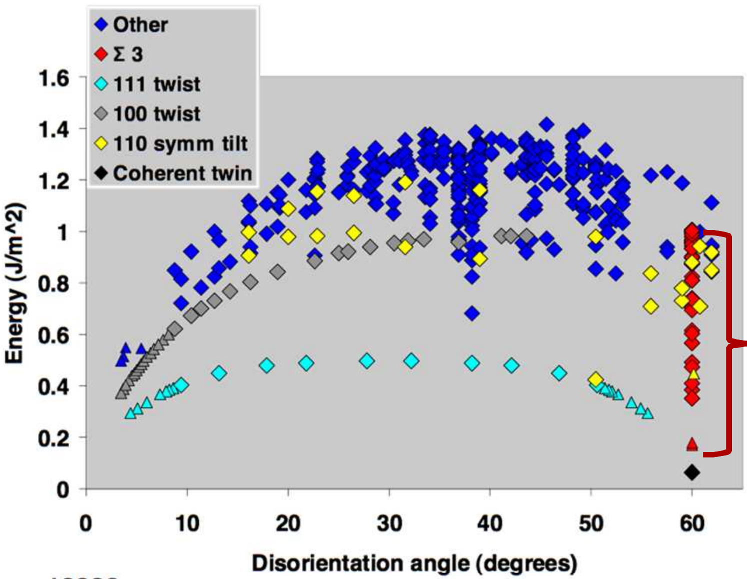

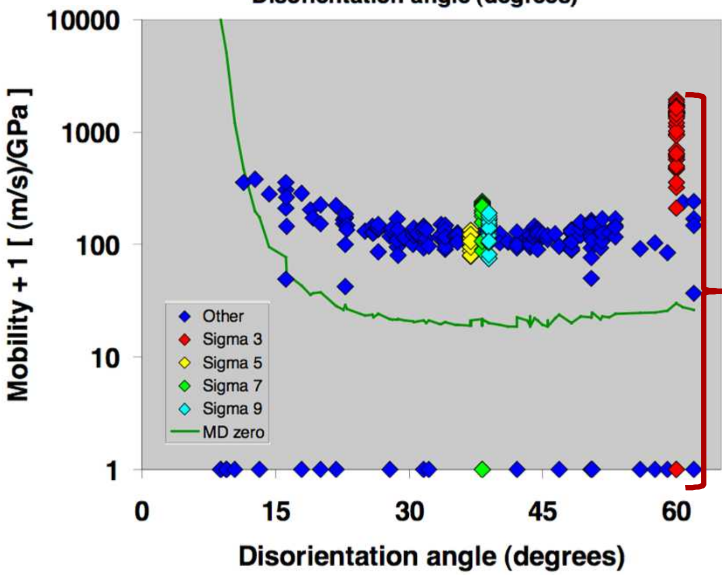

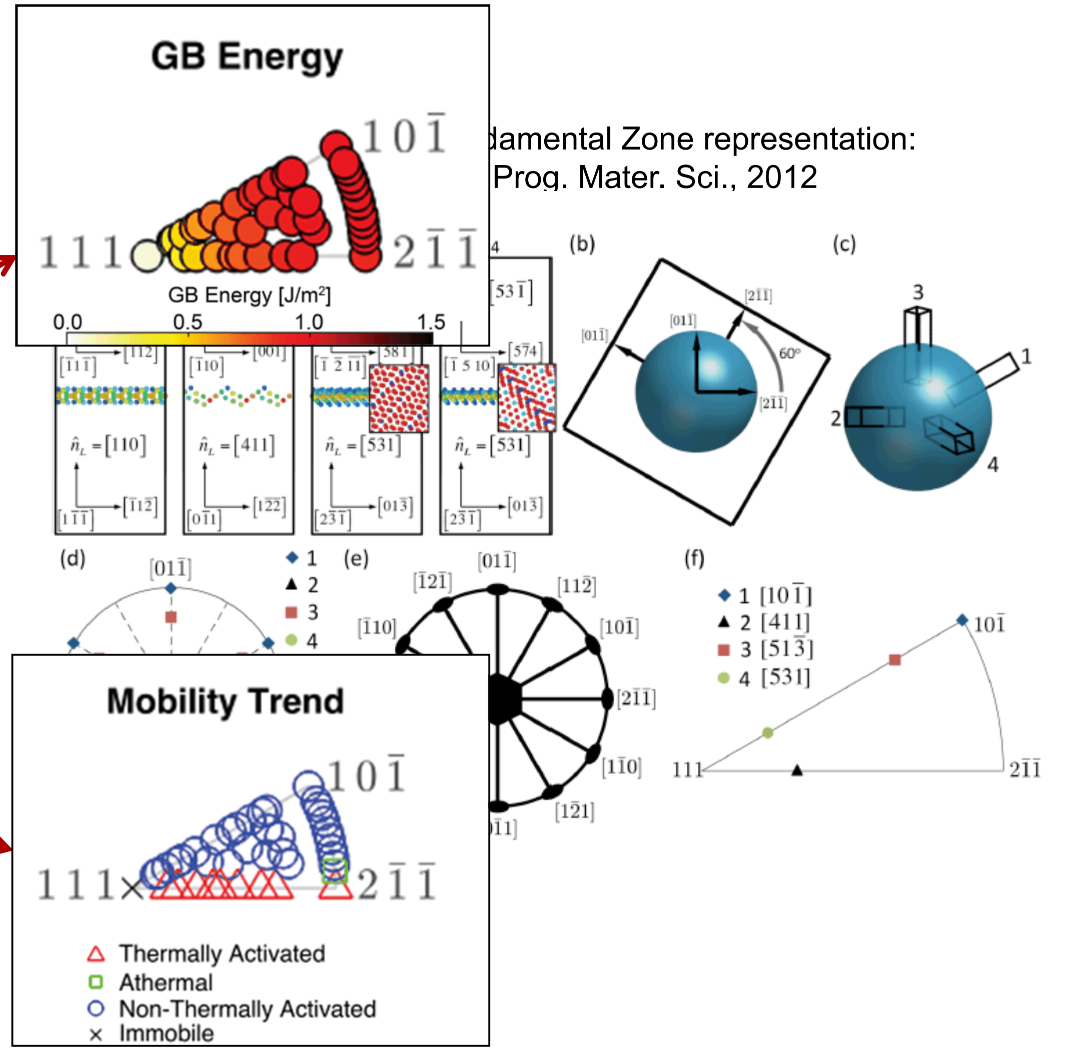

Homer, Patala, and Priedeman, *in review*

Summary

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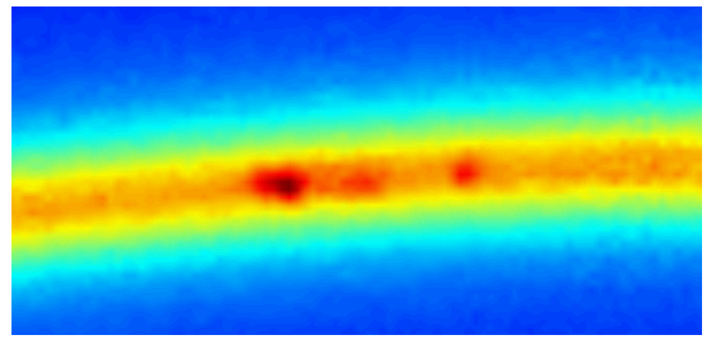
1. Stress concentrations ***are*** relevant for nanocrystalline metals, but when the feature size approaches the grain size, the effects can be swamped by microstructural effects.

For this reason, nanocrystalline metals actually have an **increased sensitivity** to notch effects!

NANO LETTERS

Is Stress Concentration Relevant for Nanocrystalline Metals?

Sandeep Kumar,[†] Xiaoyan Li,[‡] Aman Haque,[*,†] and Huajian Gao[*,‡]



2. A new x-ray diffraction modality combined with the localizing effect of notches allows the observation of dynamic abnormal grain growth during fatigue testing.

3. While there is still controversy about the mechanisms for low-temperature mechanically-driven abnormal grain growth, the cause is likely linked to the existence of a few 'special' anti-thermal grain boundary types which move by a coordinated motion of many atoms simultaneously.