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Detecting the Precursor to Fatigue Crack Initiation in Nanocrystalline Ni-Fe Using Synchrotron Diffraction

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U.S. DEPARTMENT OF ENERGY

NNSA National Nuclear Security Administration

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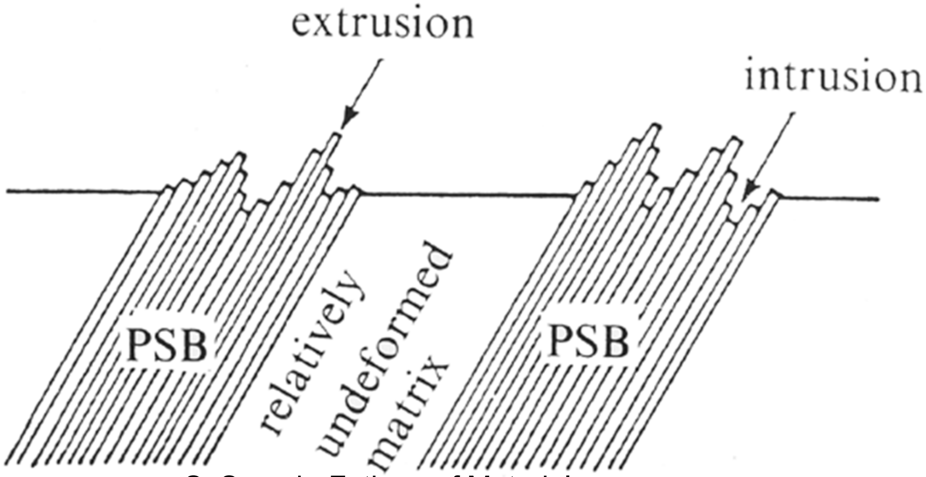
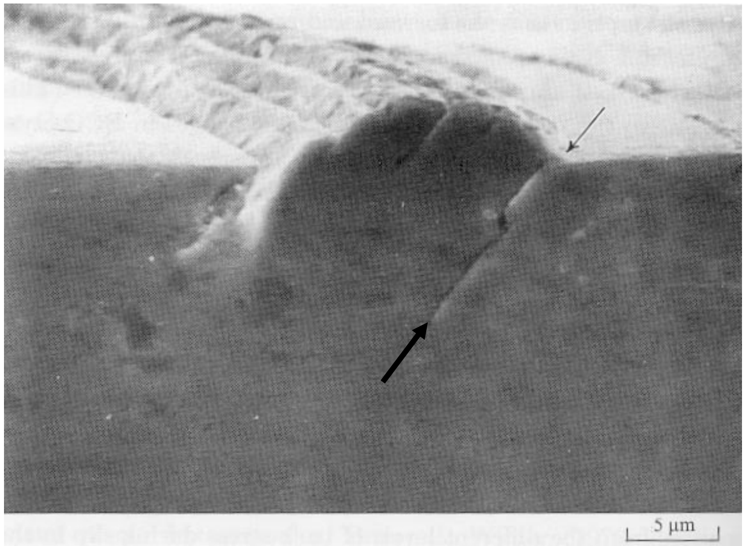
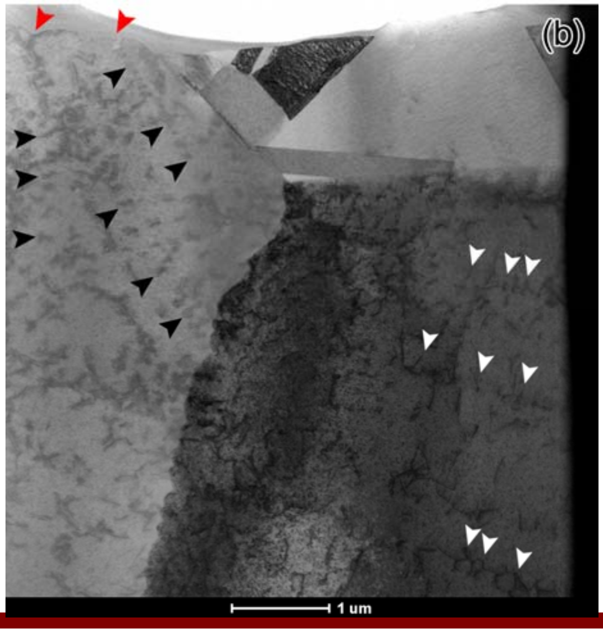
Hypothesis: the small grain size of nanocrystalline metals suppresses persistent slip crack initiation processes



S. Suresh, Fatigue of Materials



Ma and Laird, 1989

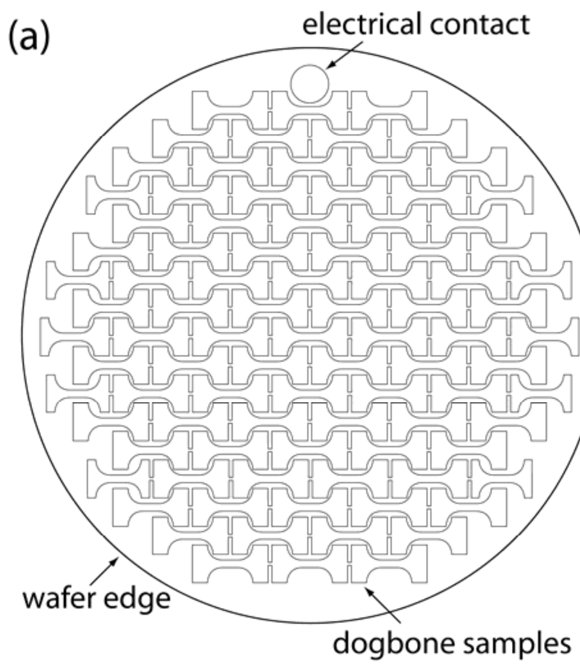


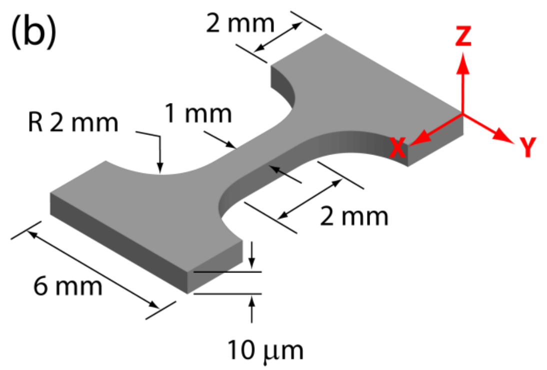
A billion nanocrystalline grains would fit inside a single microcrystalline grain of a traditional structural alloy.

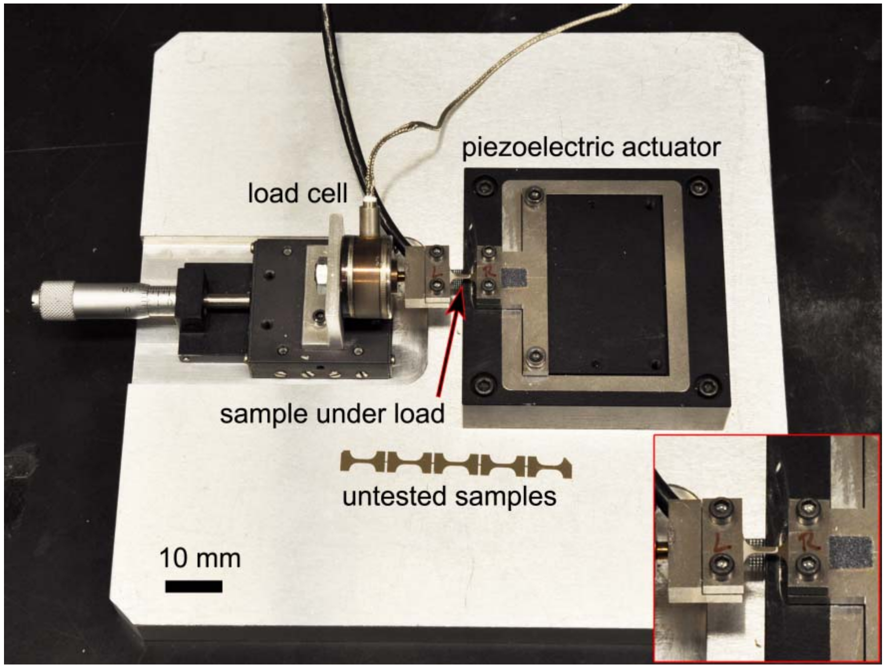
Does the fatigue mechanism change for such small grain sizes?

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

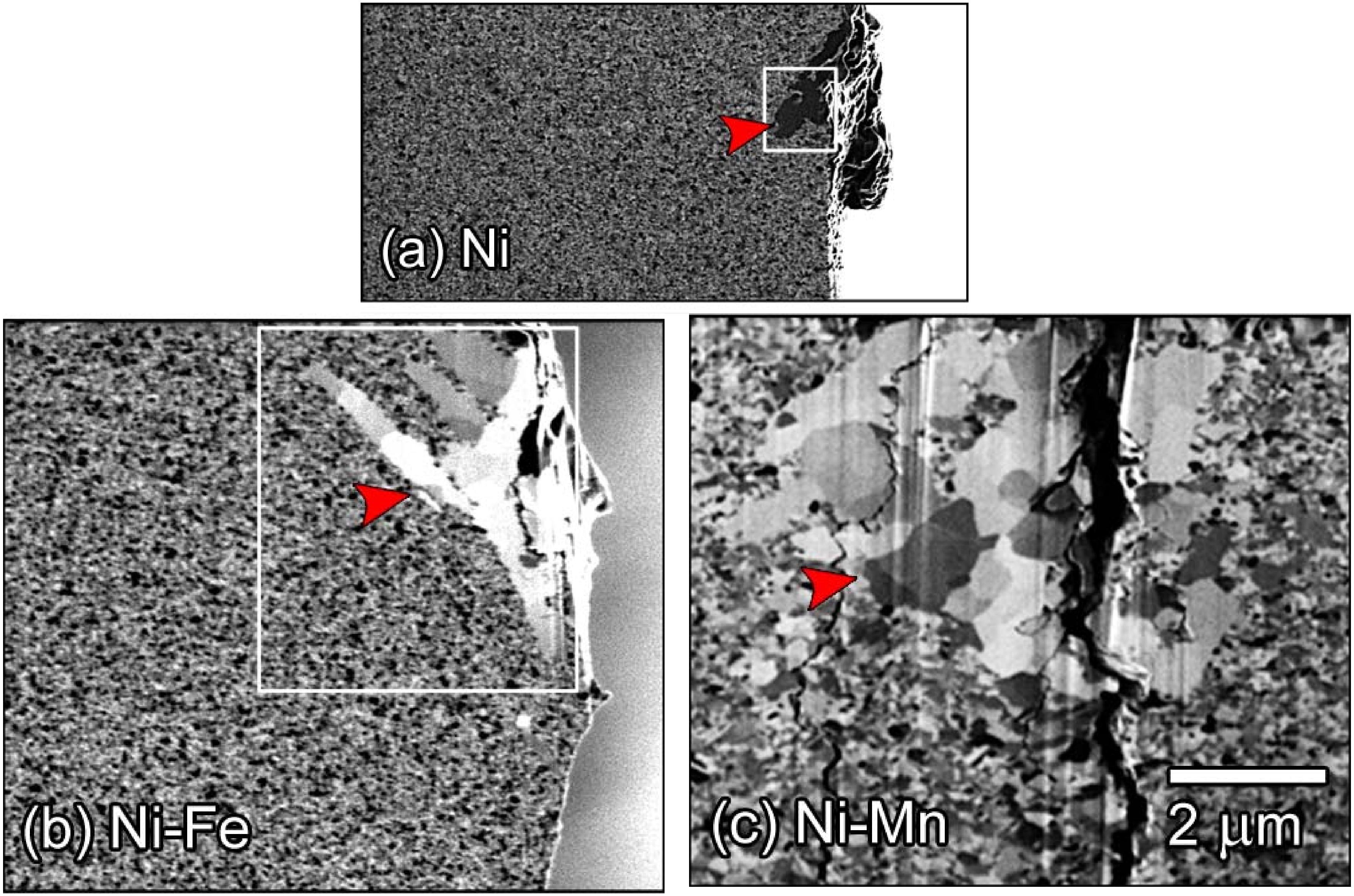
Thin film fatigue of electrodeposited Ni-Fe (permalloy)



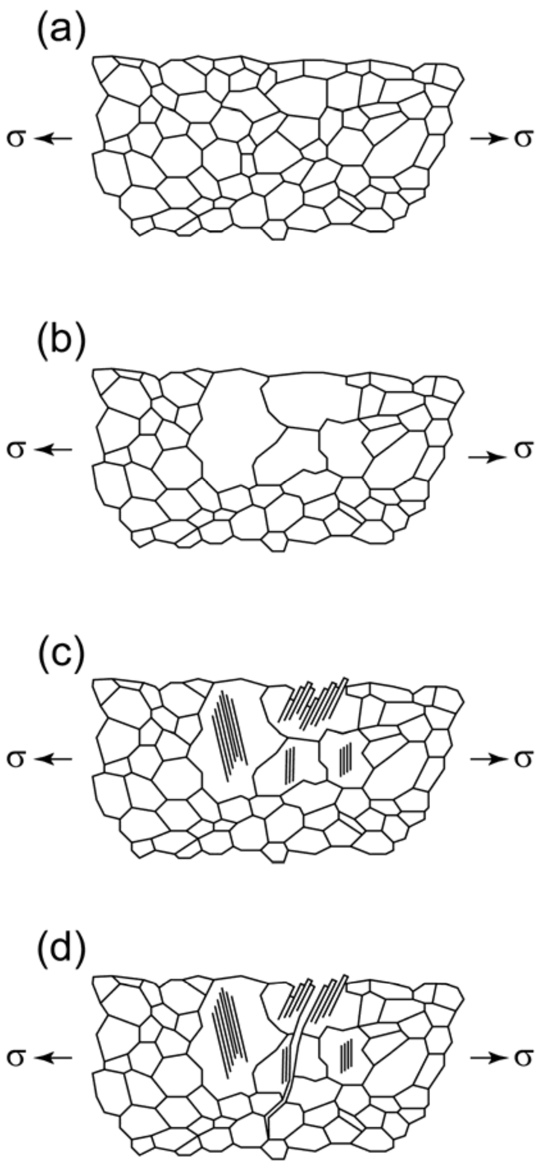




Blocky fracture surface corresponds to very large subsurface grains at point of crack initiation



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



We posulate that in nanocrystalline metals (d_{gb} < ~70 nm), the fatigue process induces grain growth as a precursor to gross slip and eventual crack initiation

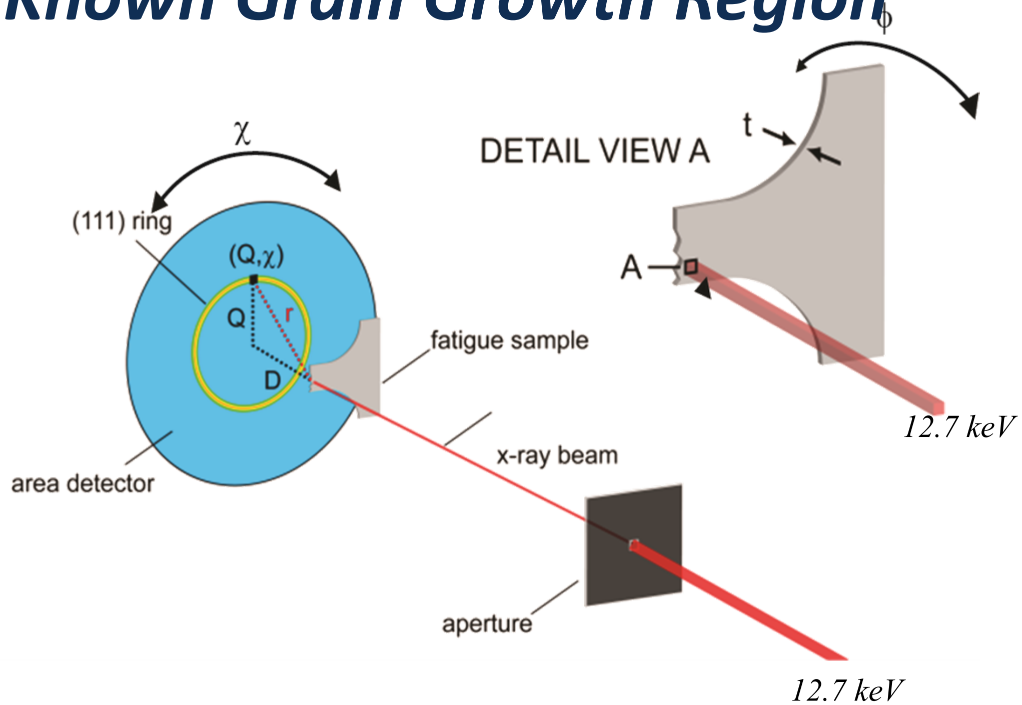
1. Is this really just a dynamic recrystallization mechanism?

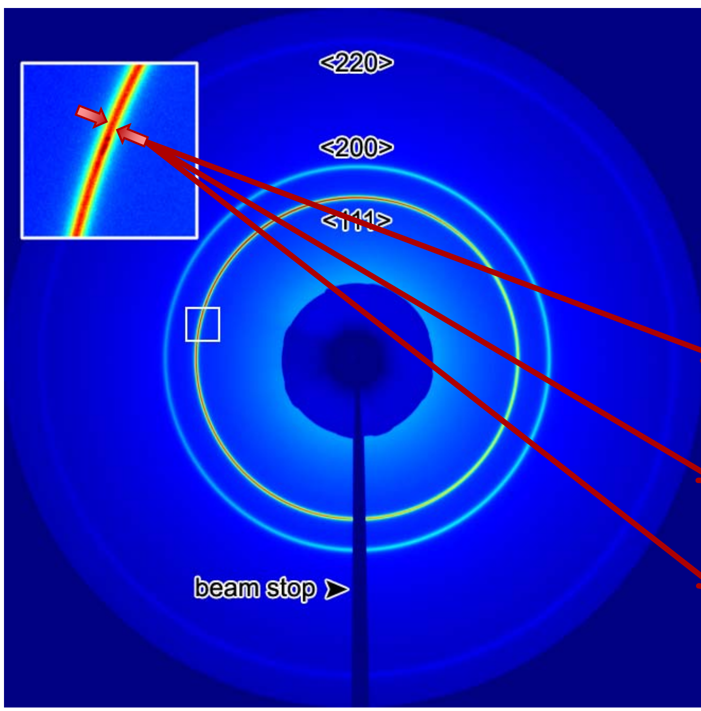
2. Were these few large grains formed during deposition, not fatigue loading?

3. If the grains grow during fatigue, how many cycles does it take? [what are the kinetics of grain growth]

Can we observe fatigue-driven grain evolution directly using *in-situ* techniques?

Proof of Concept… Interrogate Broken Fatigue Sample with Known Grain Growth Region





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

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

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

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

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

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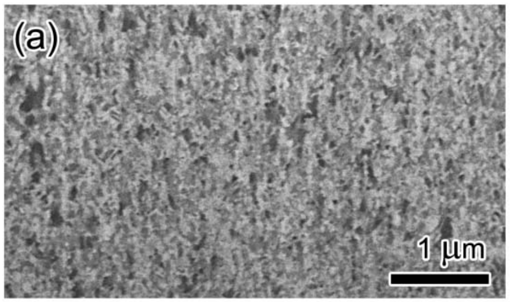
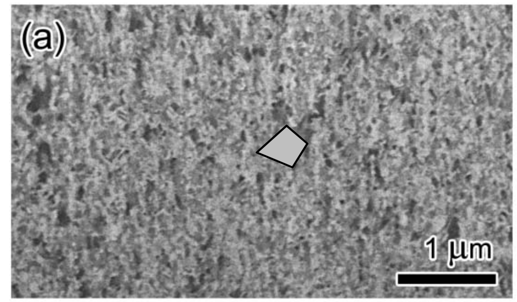
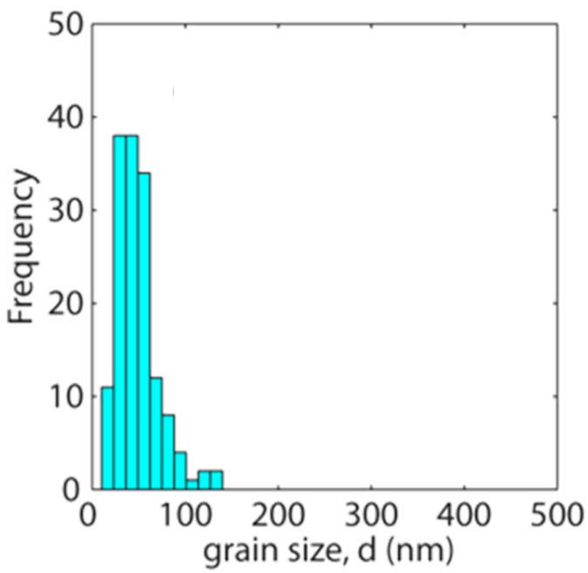
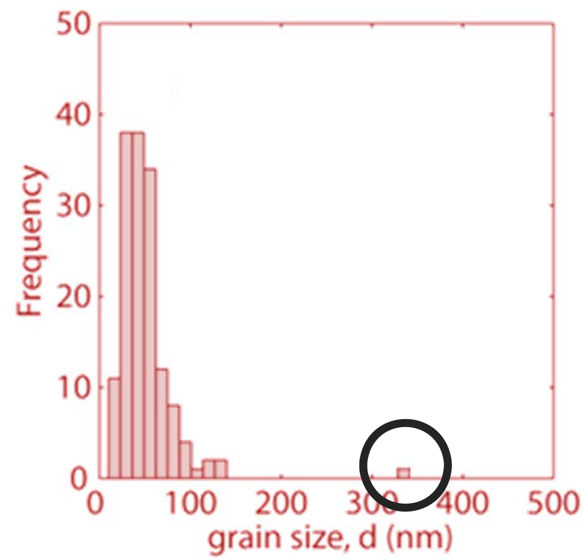
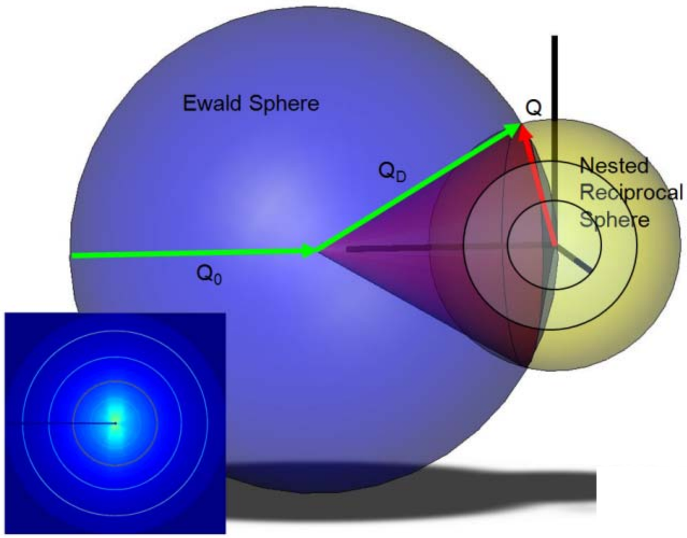
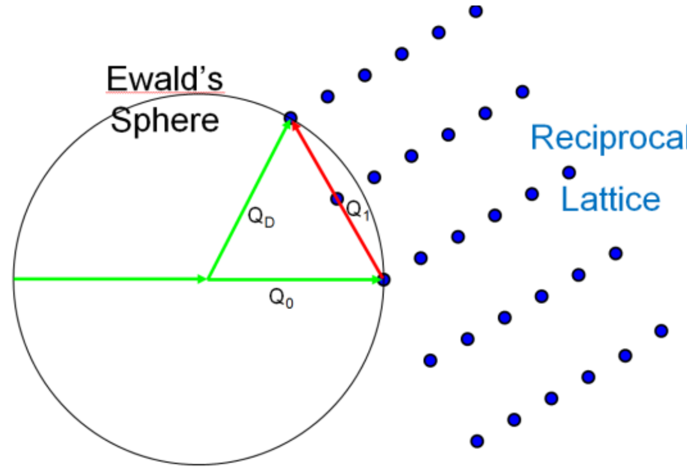
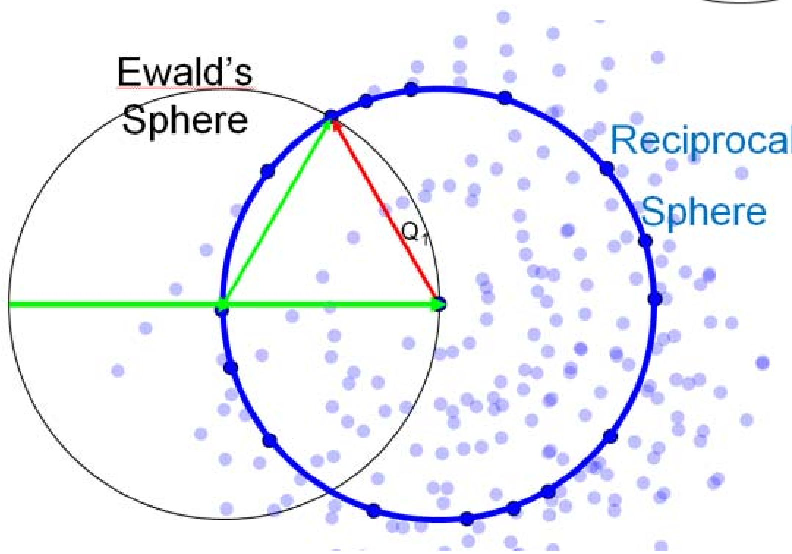
Before abnormal grain growth	After abnormal grain growth
	
	

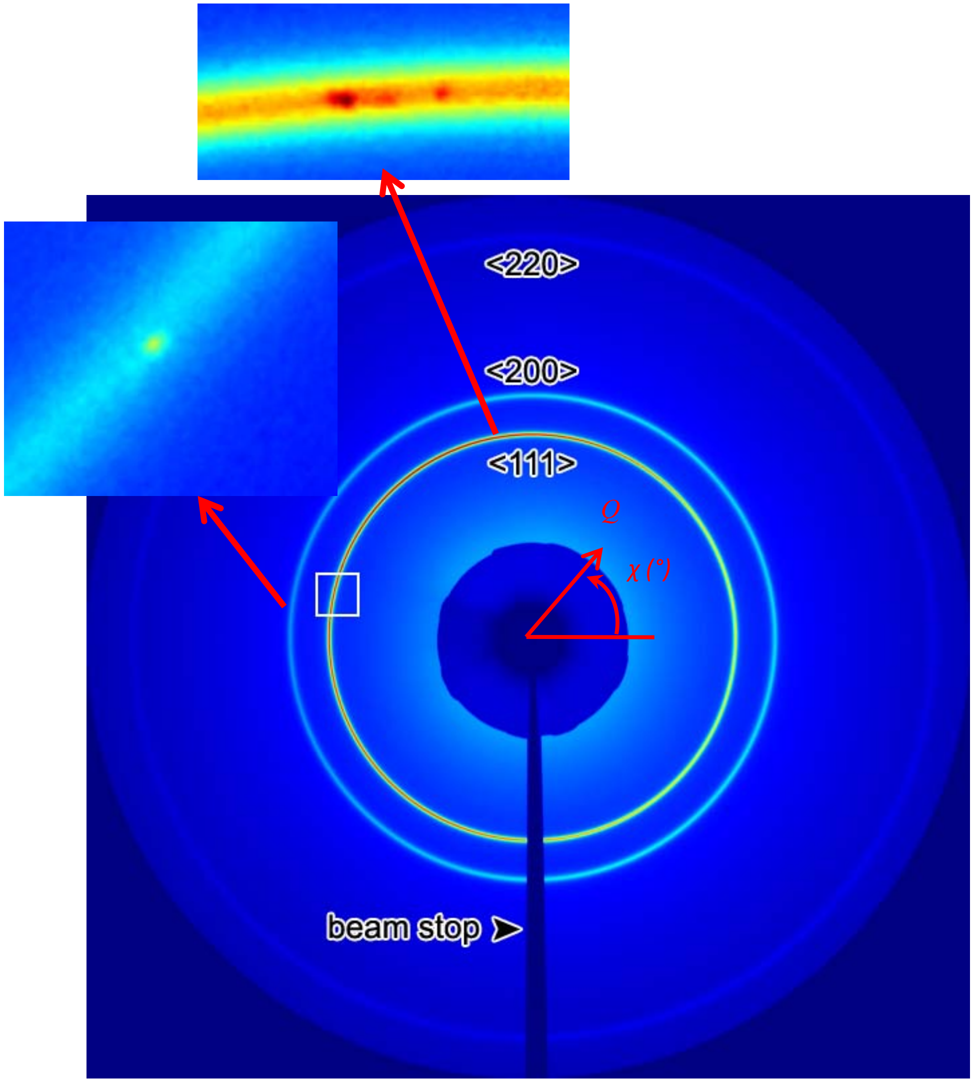
What happens in diffraction when One grain is large and the rest are small?

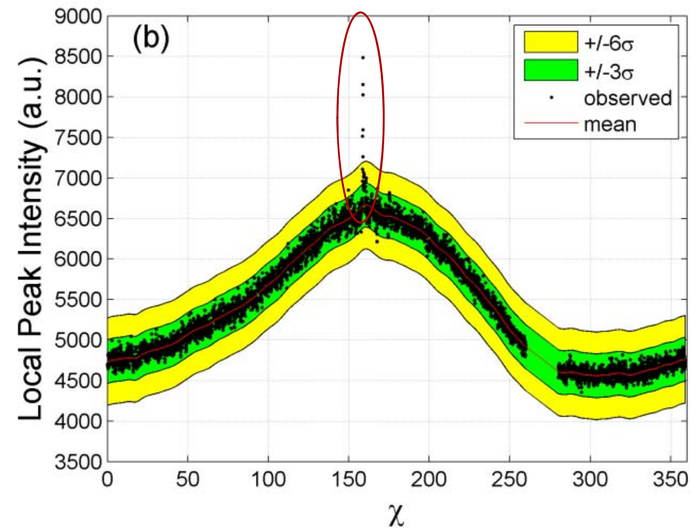






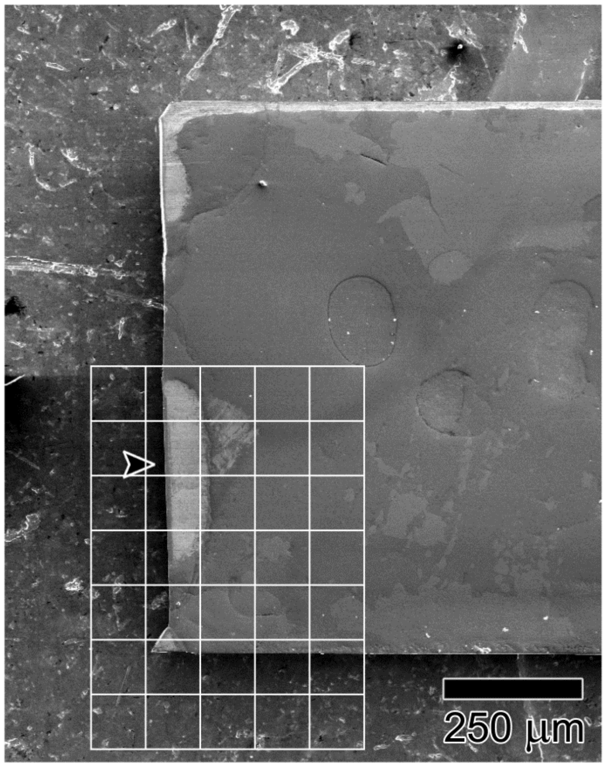
Preliminary Observation: A ‘spike’ in the Debye ring.

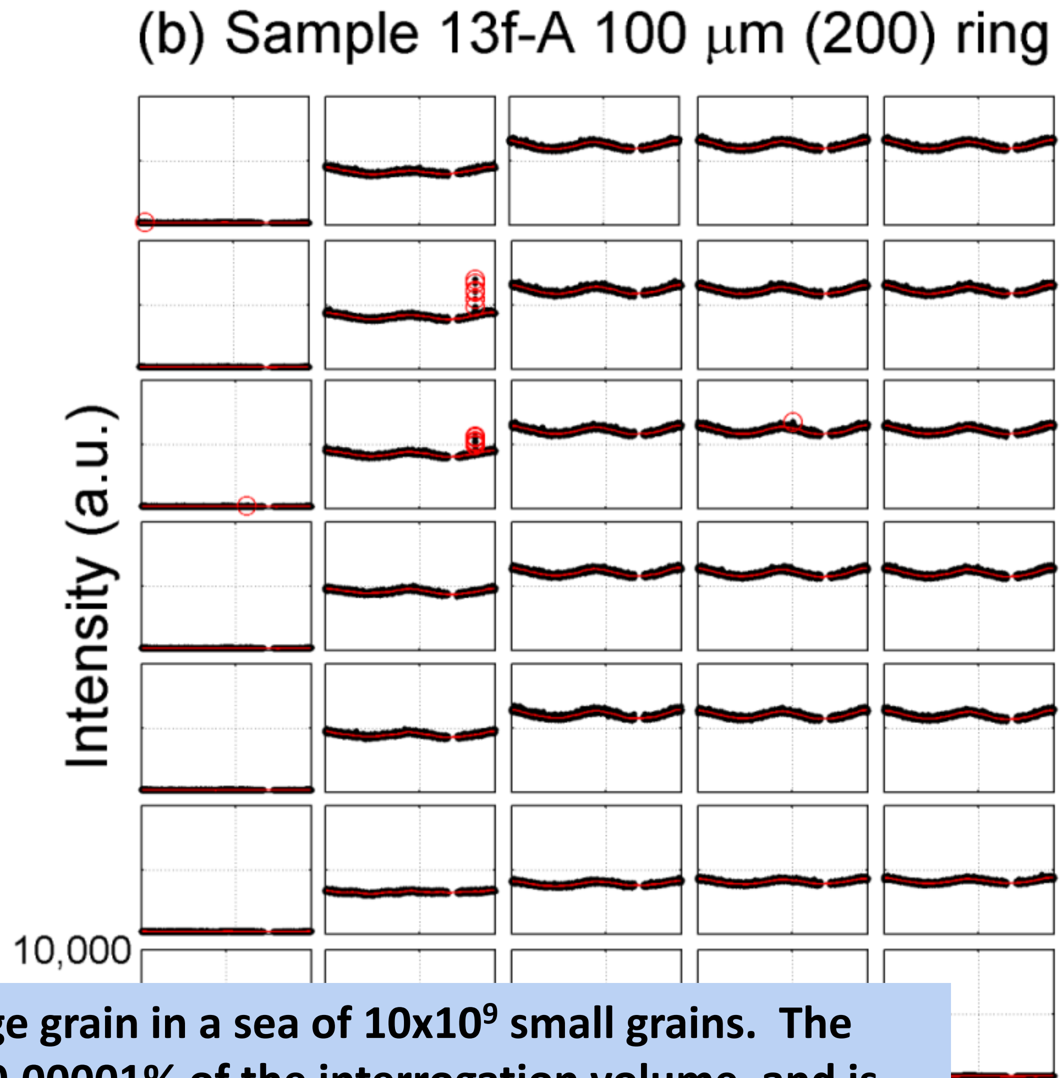




How do we know these spikes are truly statistically significant anomalies and not just noise?

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

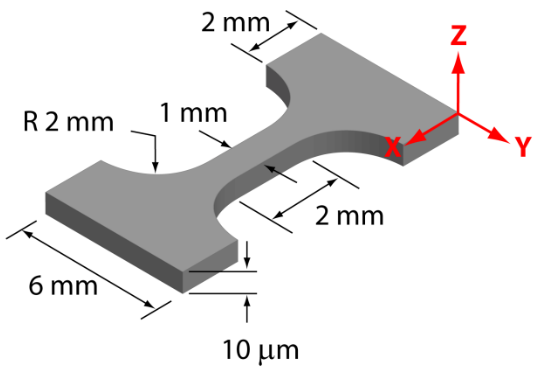




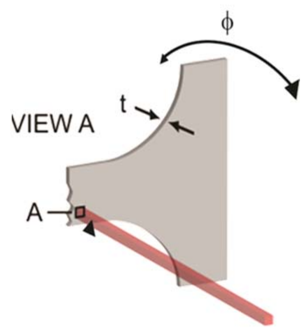
We've identified 1 large grain in a sea of 10x10^9 small grains. The large grain occupies ~0.00001% of the interrogation volume, and is identified with a statistical confidence >> 99.9999998% (6σ).

*Now…. Can we observe active grain growth **during** a fatigue test?*

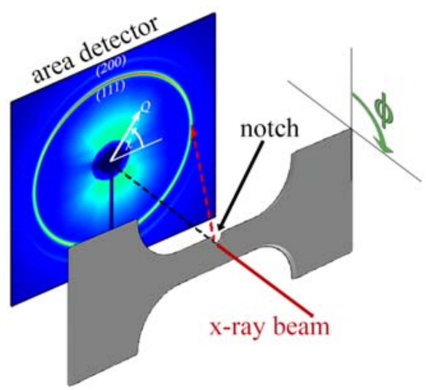
the needle-in-a-haystack challenge…
Rapidly detecting 1 abnormal grain in 10^{12}



The gage section contains
~1 x10^{12} grains

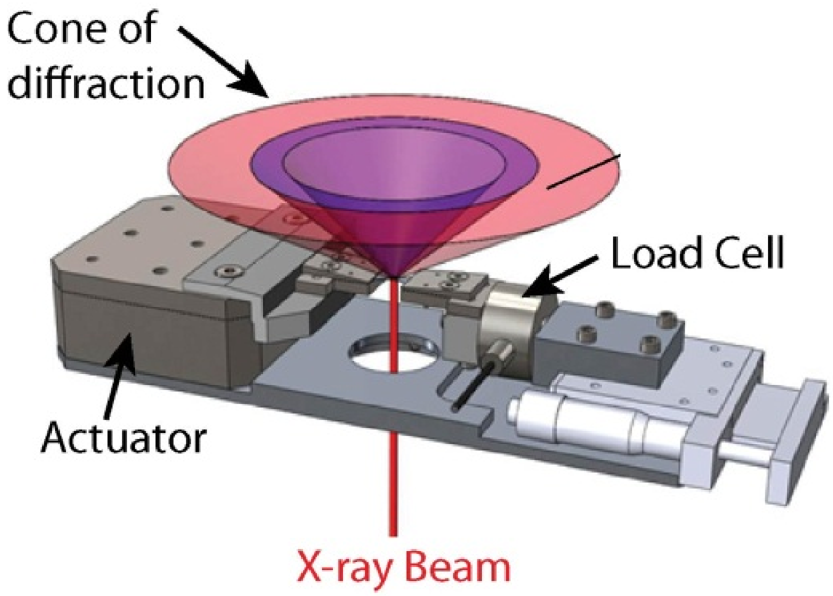


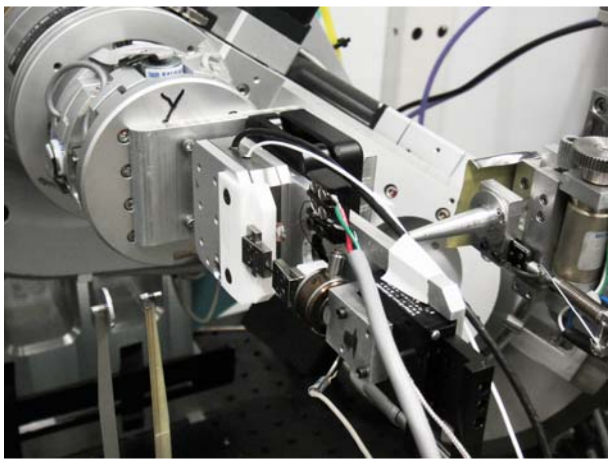
A 100x100 μm x-ray spot interrogates
~1 x10^{9} grains

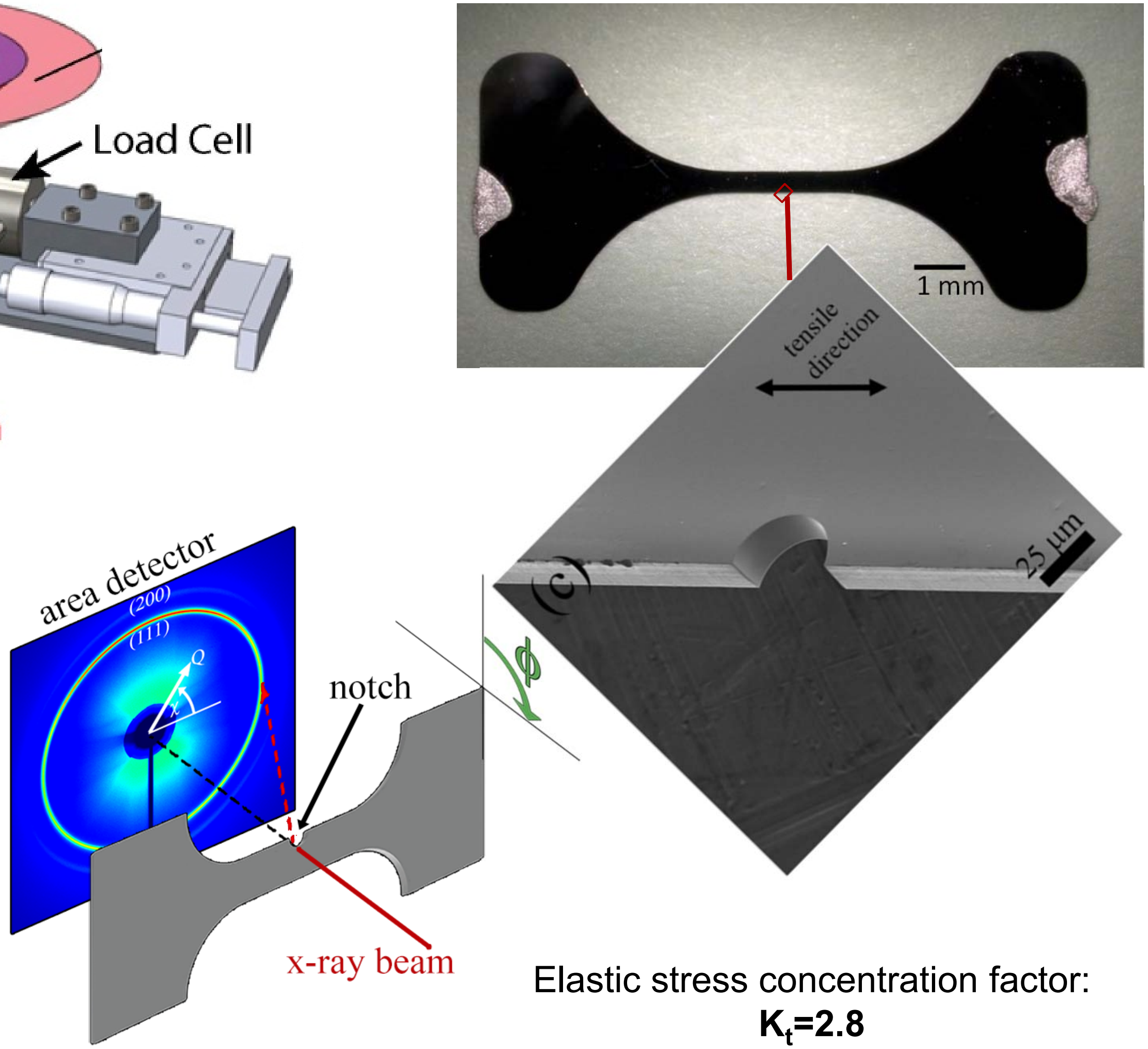


A 10 μm notch localizes the peak stress to
<<1 x10^{7} grains

In-situ Notch Fatigue



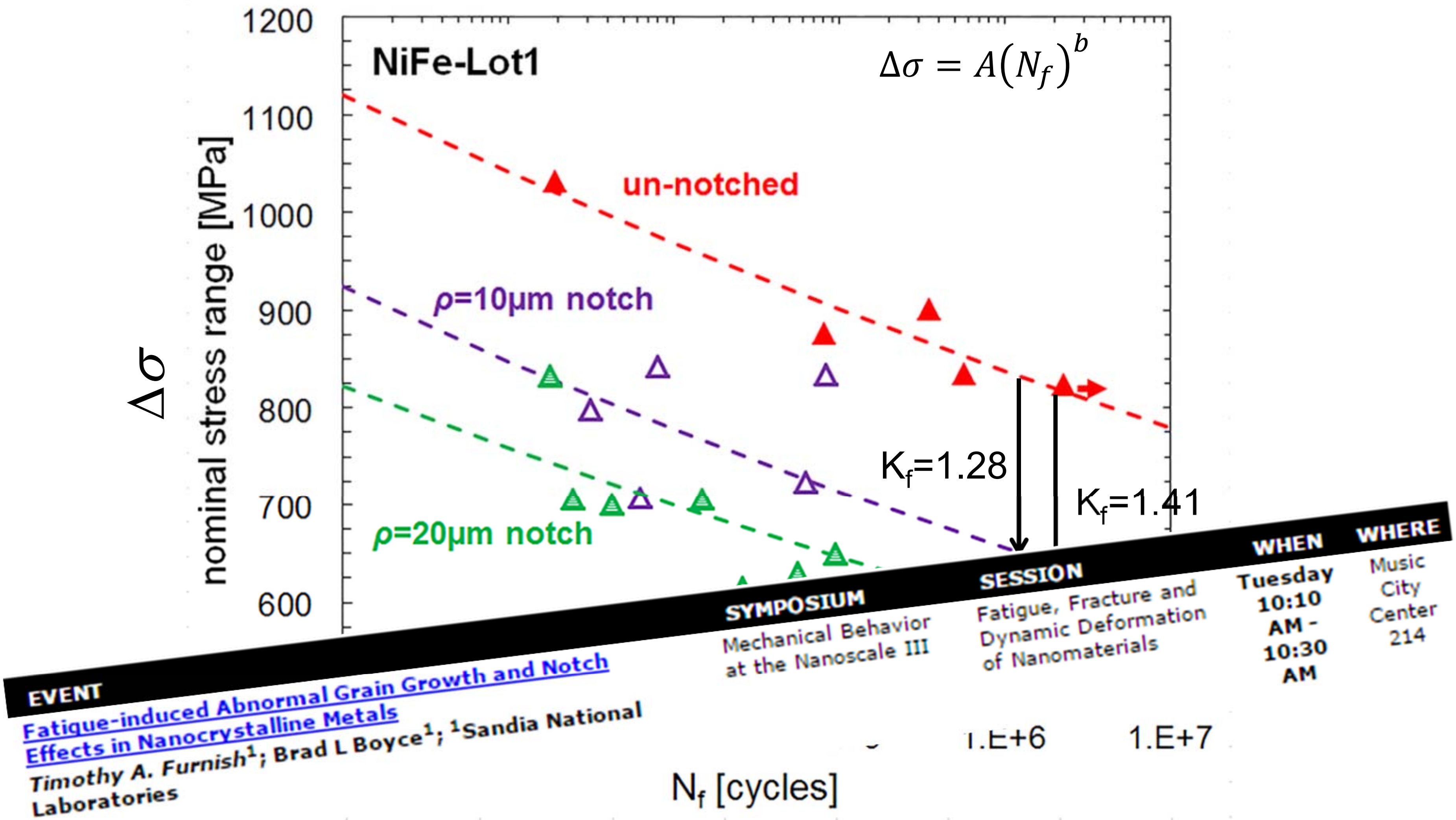




Elastic stress concentration factor:
K_t=2.8

Furnish, Sharon, Boyce, Arrington, Pillars and Clark, *J. Mater. Res.*, 2016

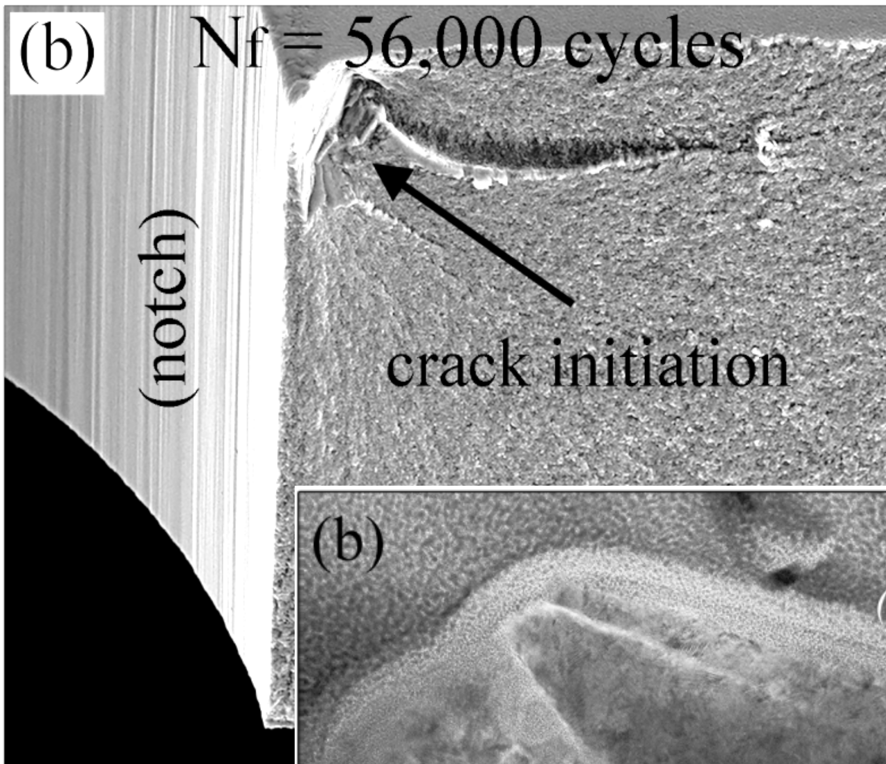
Result: Effect of notch on S-N fatigue

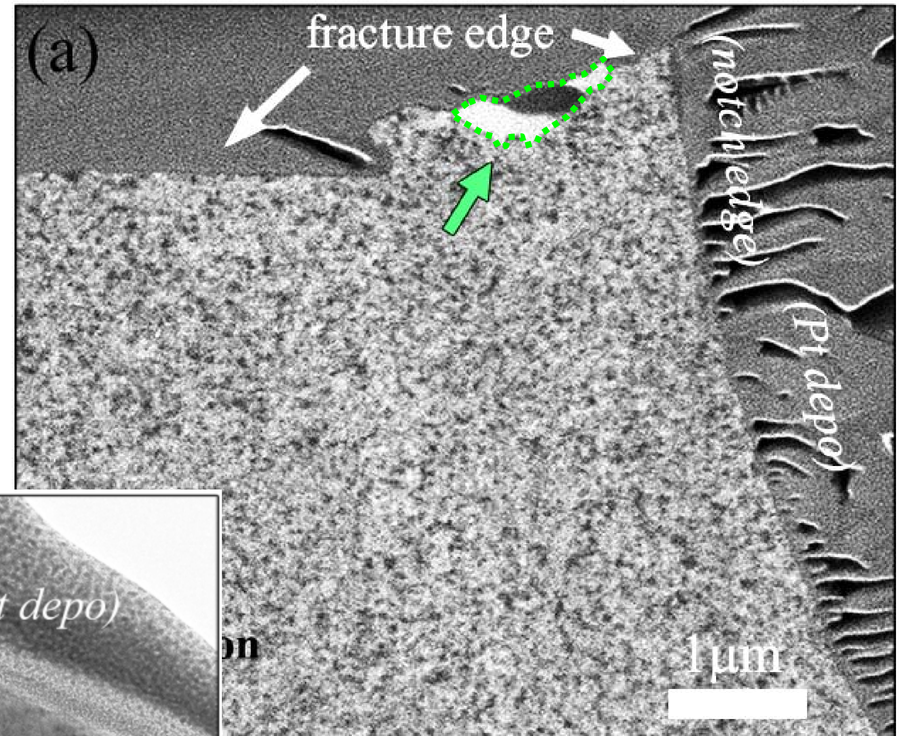


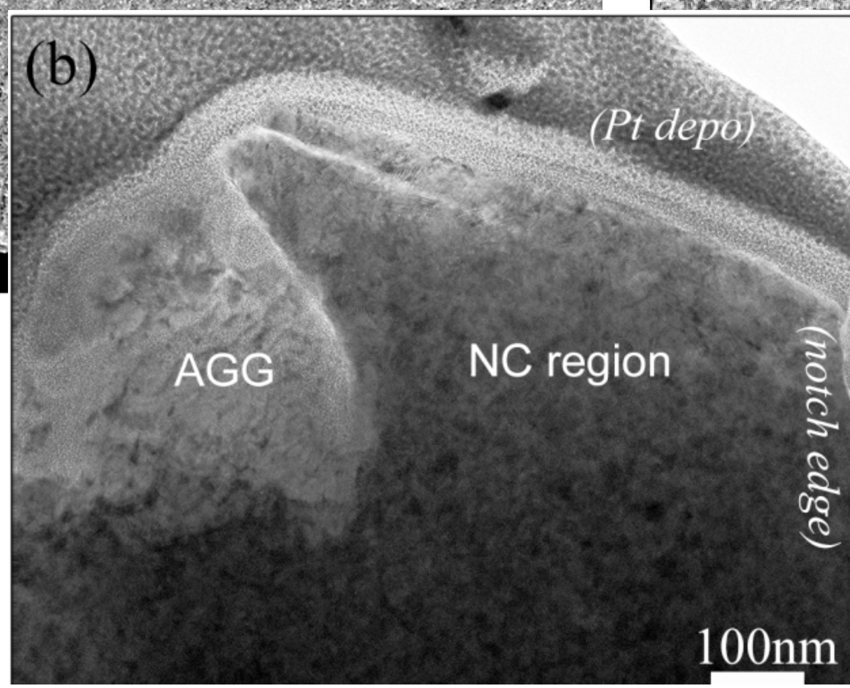
number of cycles to failure

Furnish, Sharon, Boyce, Arrington, Pillars and Clark, *J. Mater. Res.*, 2016

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





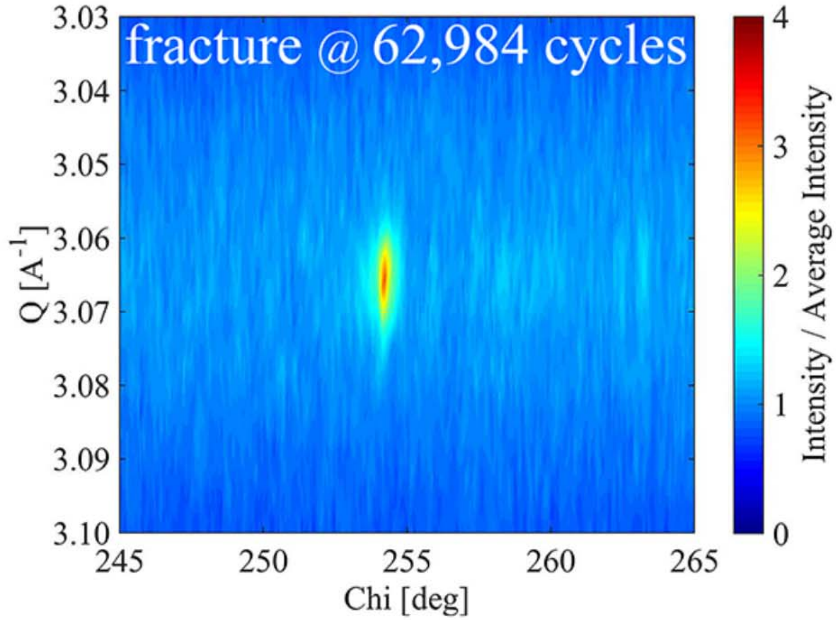


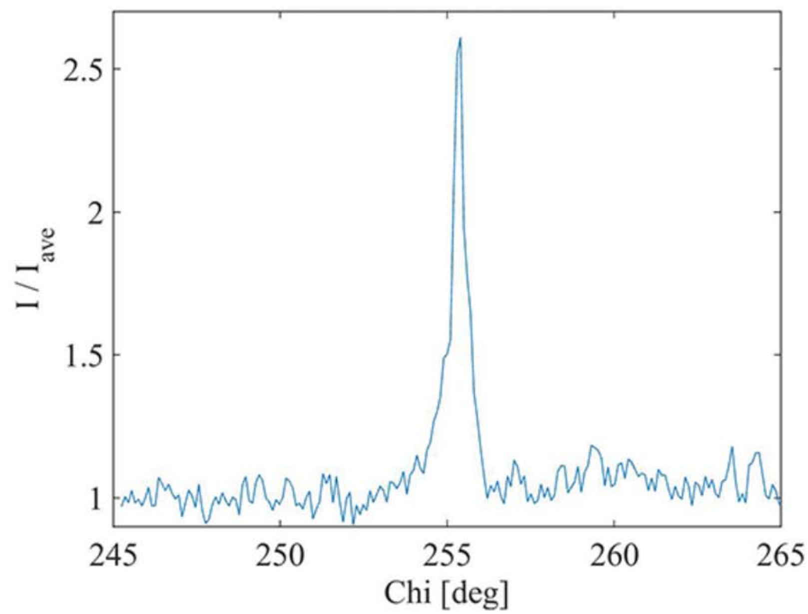
A nagging question: were these large grains caused by deposition or sample preparation, not fatigue?

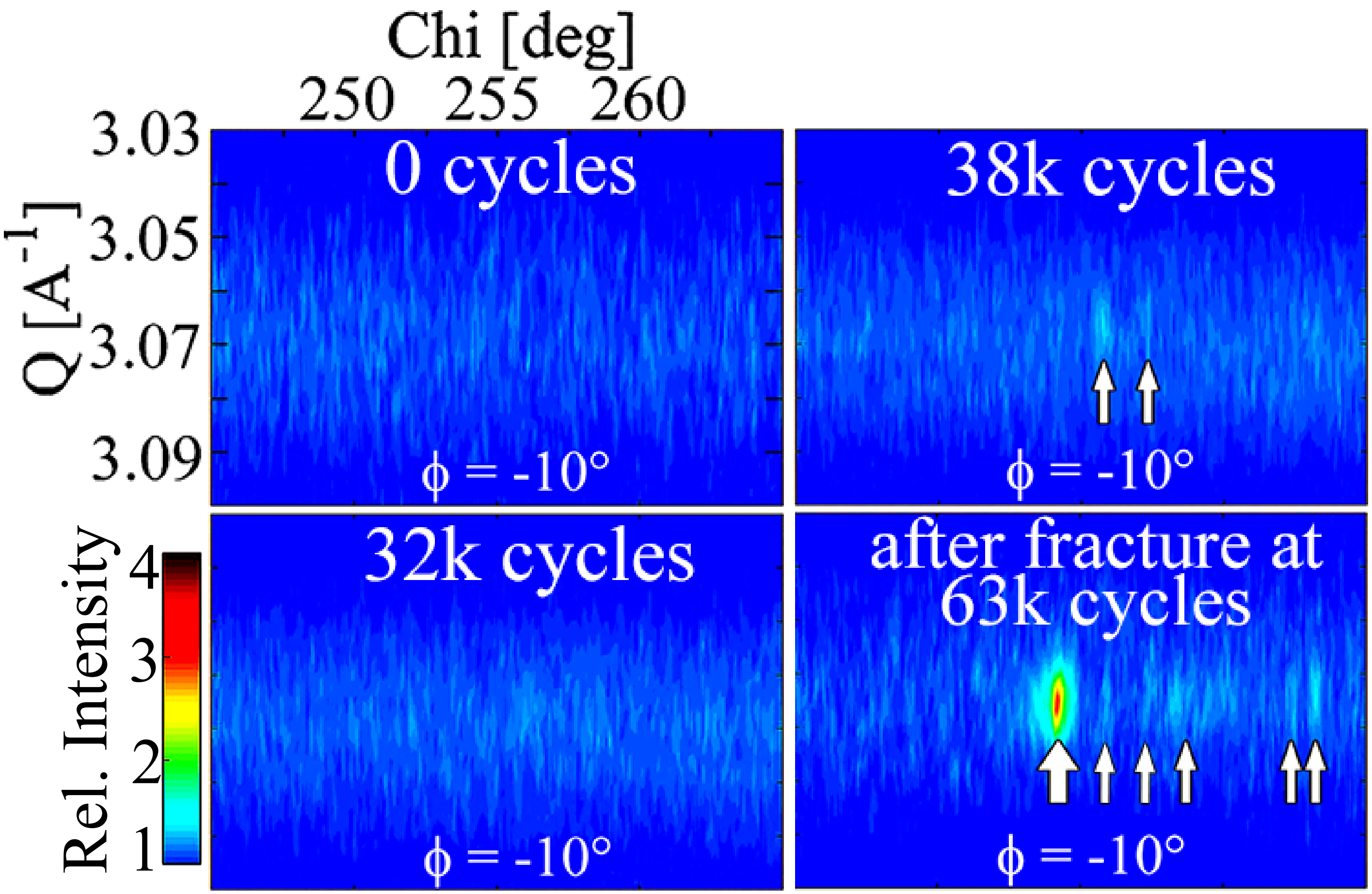
Furnish, Sharon, Boyce, Arrington, Pillars and Clark, *J. Mater. Res.*, 2016

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

Looking at a 20º arc of the (111) diffraction ring…

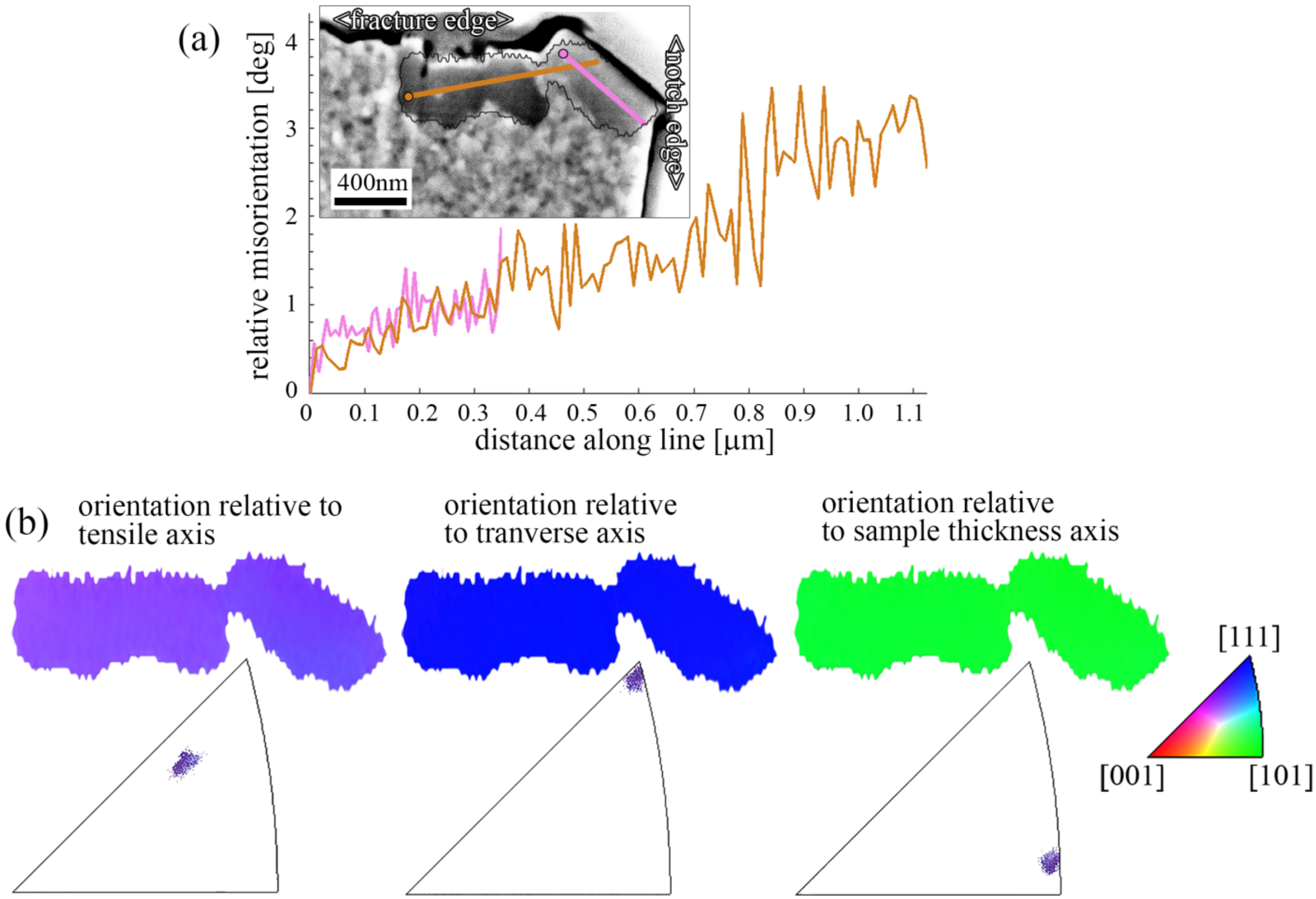




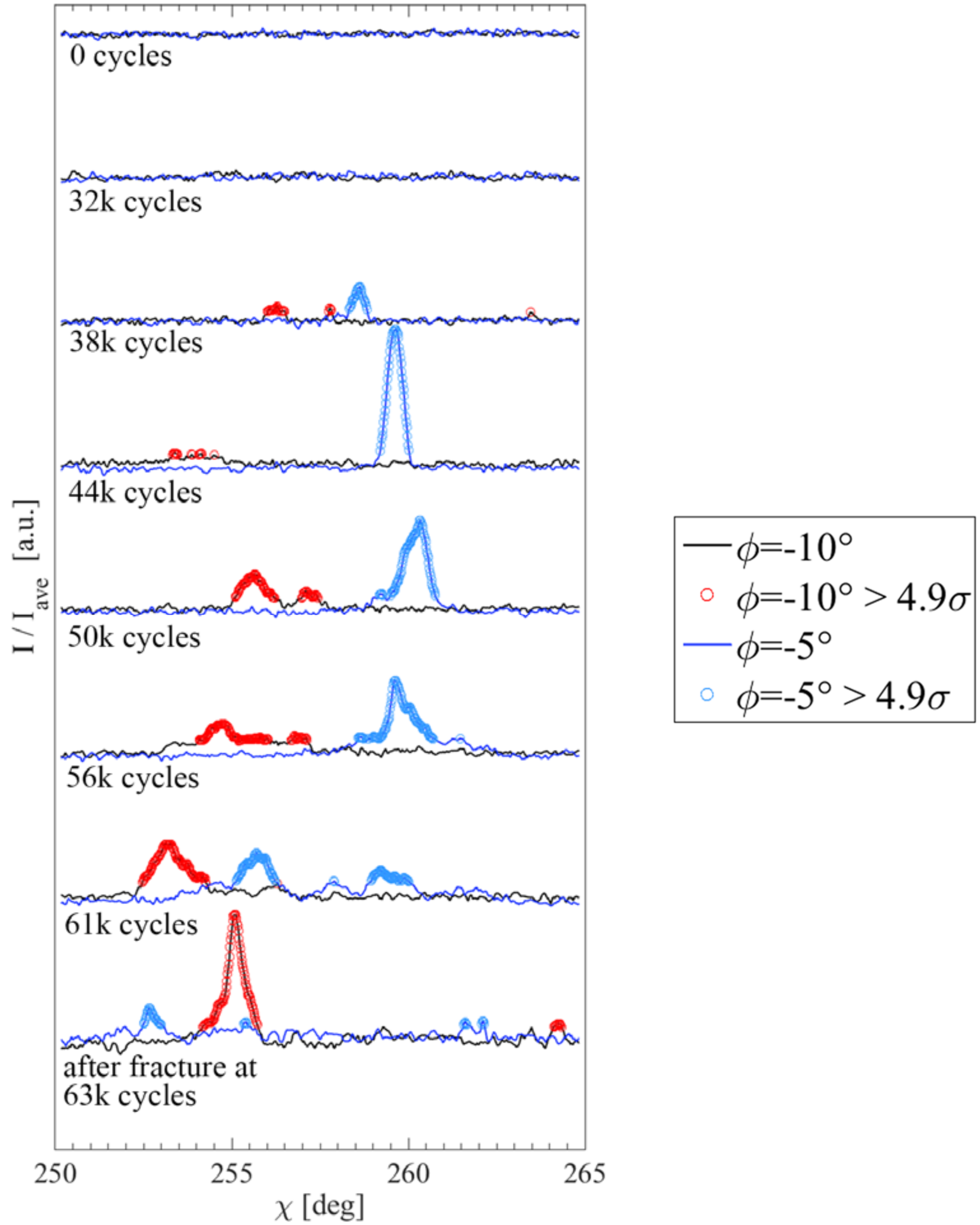


The X-ray technique detects rare events but lacks spatial details…
→What about the TEM?

Not recrystallization… EBSD shows that there is orientation spread of >3º within a growing grain

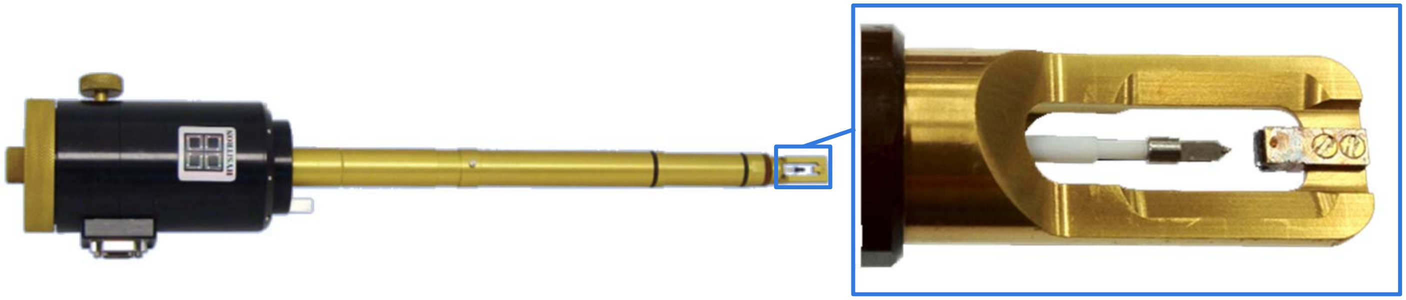


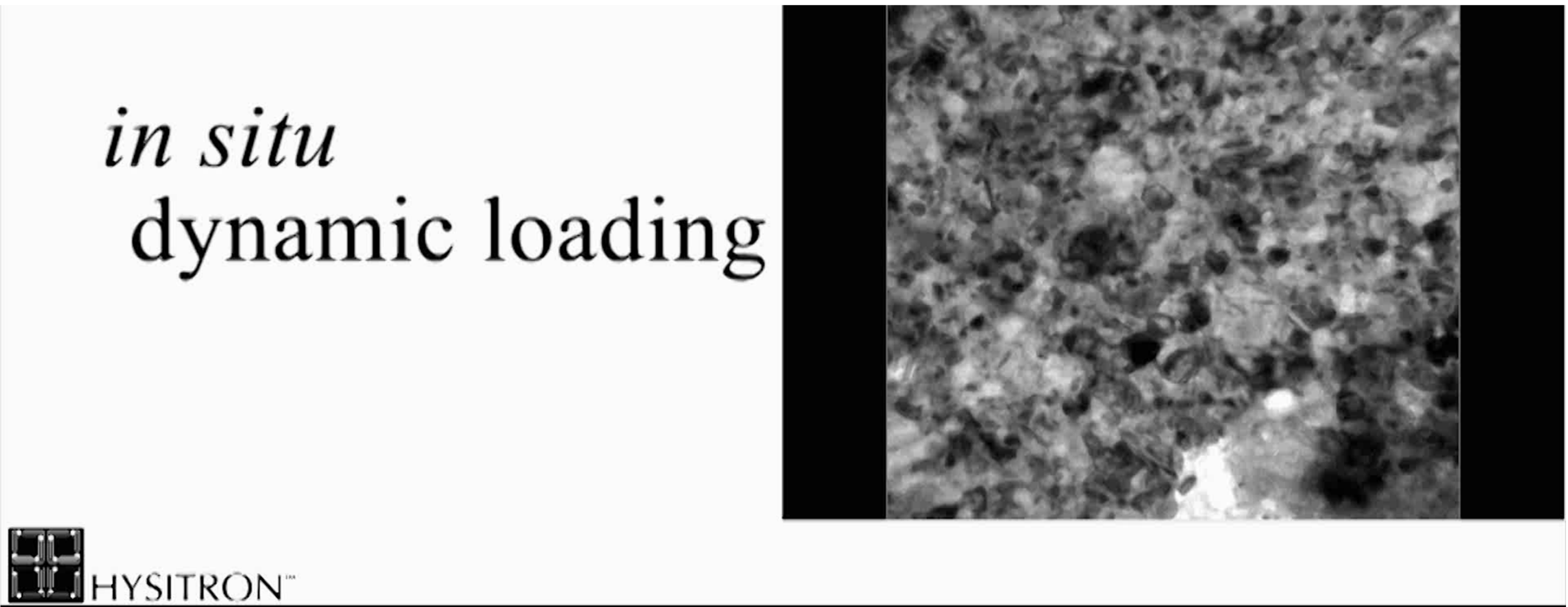
The large grains don't just emerge, they rotate…



Can we also image the grain evolution directly in the TEM?

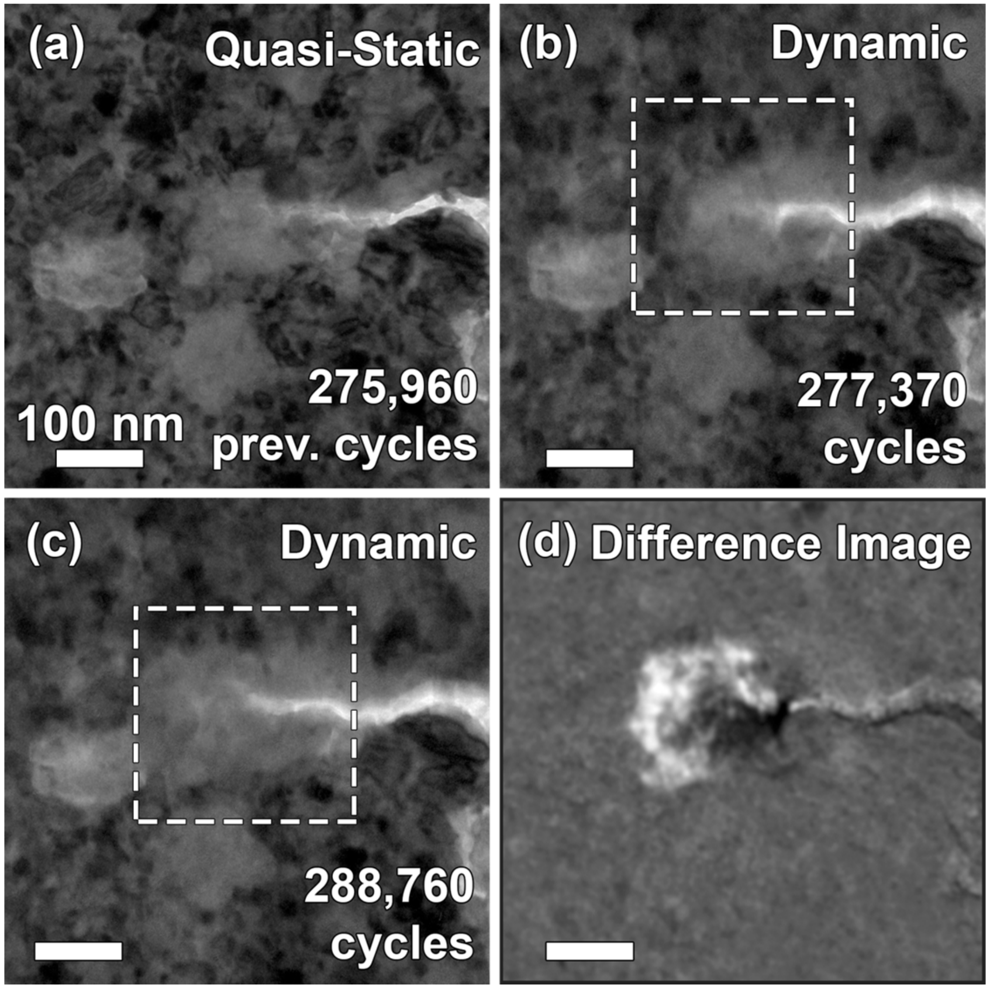
Watching fatigue-induced grain evolution directly….





Collaboration with Khalid Hattar, Dan Bufford, Bill Mook, Doug Stauffer (Hysitron)

Evolution during in-situ High Cycle Fatigue….



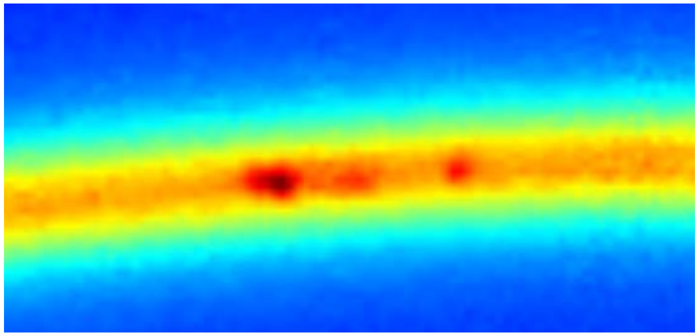
First ever high-cycle fatigue experiment in a TEM!

>300,000 cycles in ~20 minutes!

Fatigue crack growth rate of ***da/dN* = 6 × 10^{-12} m/cycle**

Collaboration with Khalid Hattar, Dan Bufford, Bill Mook, Doug Stauffer (Hysitron)

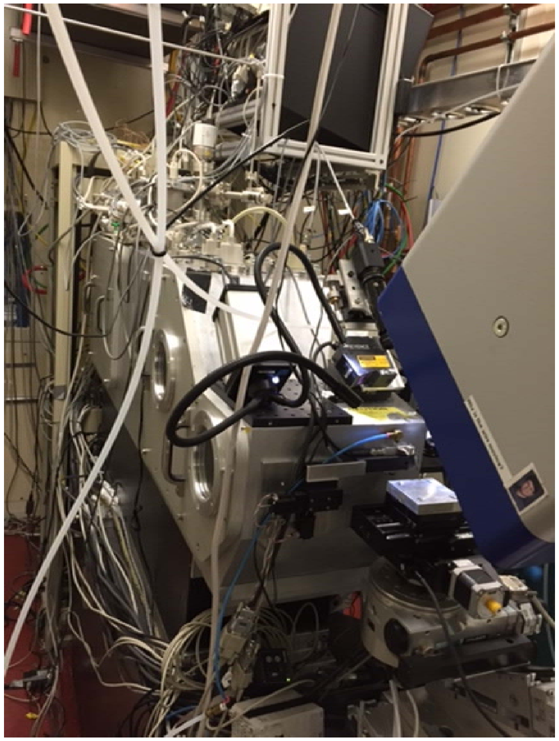
Summary

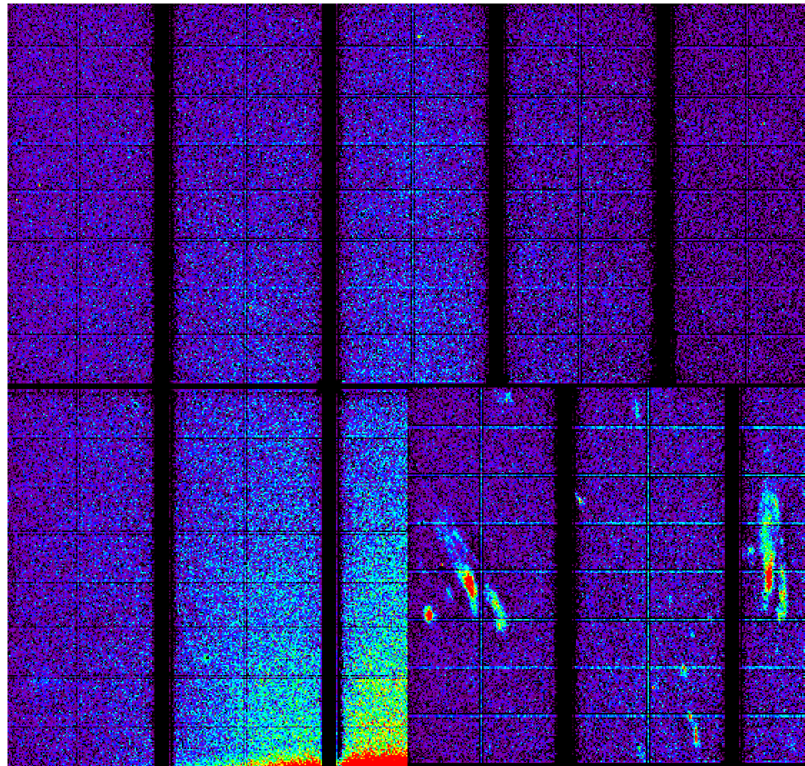


1. A new x-ray diffraction modality allows the observation of **dynamic abnormal grain growth during fatigue testing**.

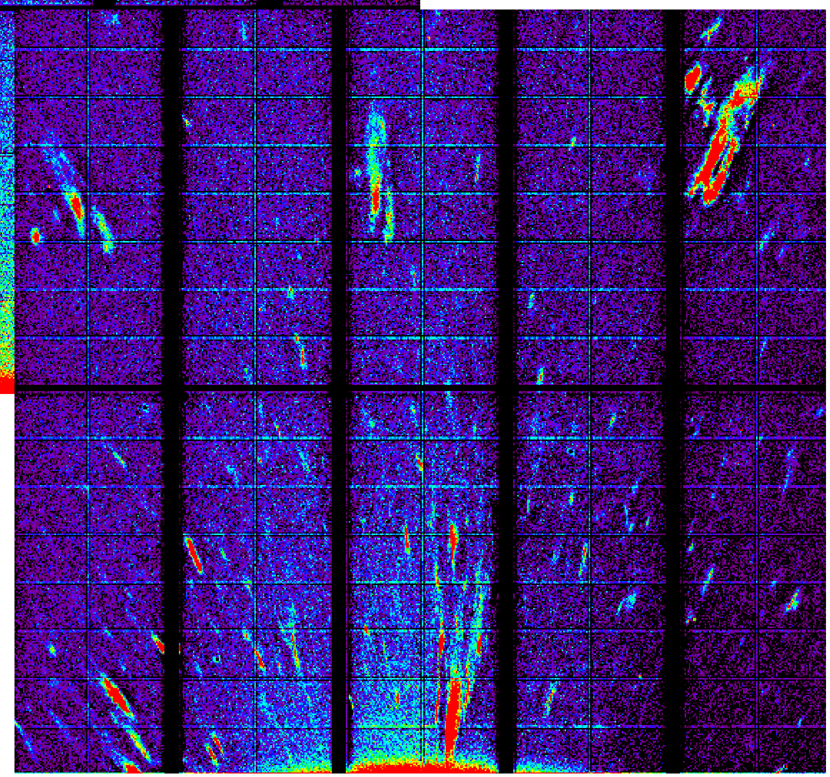
2. This new technique may also be relevant to:
   * detecting other abnormal grain growth events such as Goss grains in electrical steels
   * detecting the onset of recrystallization

Polychromatic Microdiffraction (Advanced Light Source)



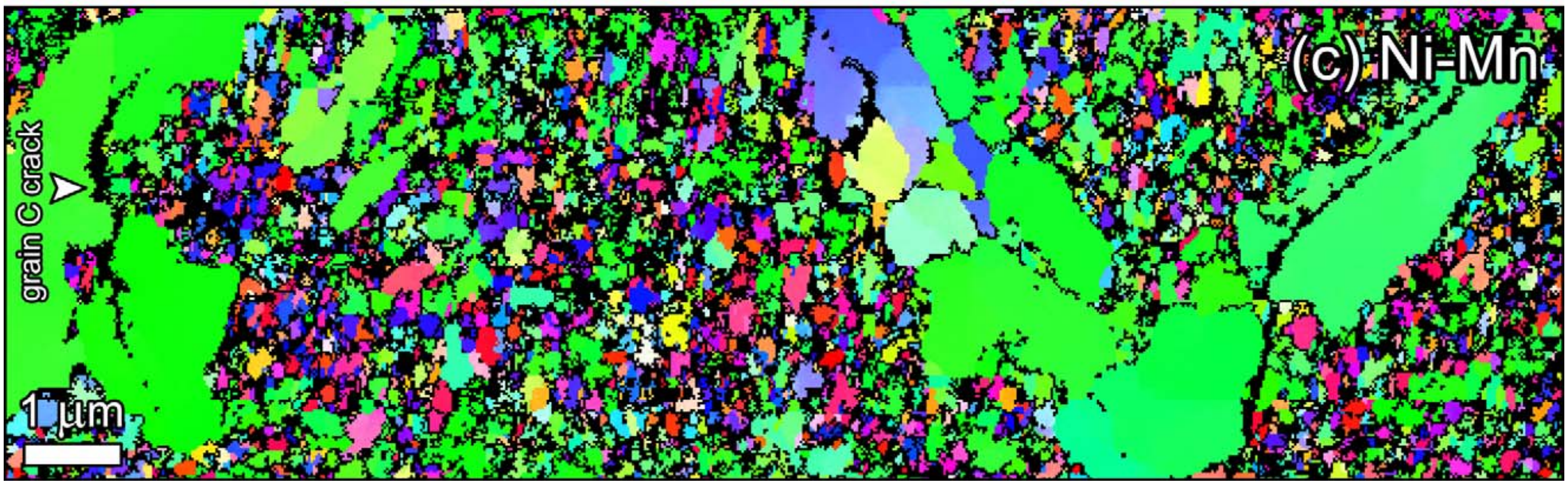


Nanocrystalline region (no grain growth)



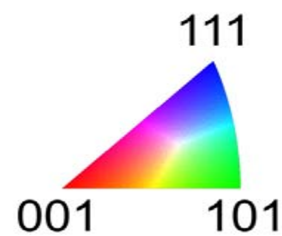
Grain Growth Region (only 4 µm away!)

How could abnormal grain growth occur at such low temperatures???



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

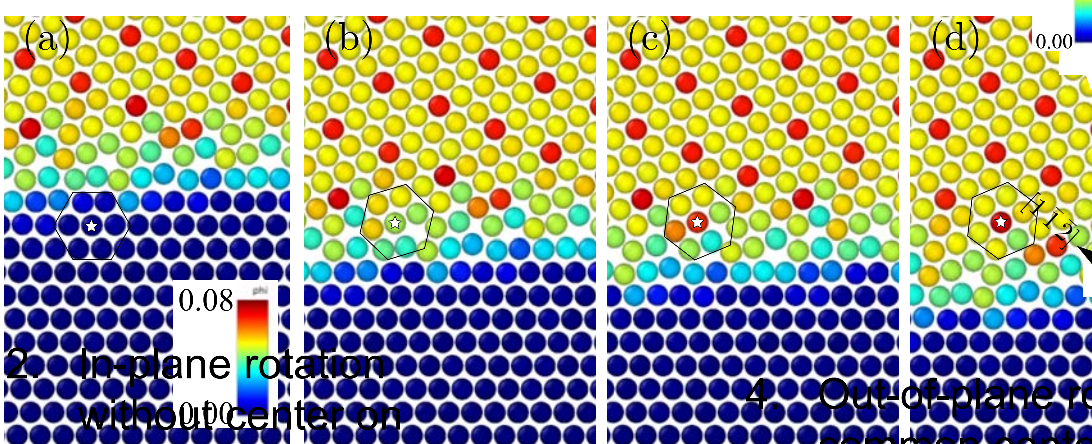
Hypothesis: a few grain boundary types have a distinct mobility advantage

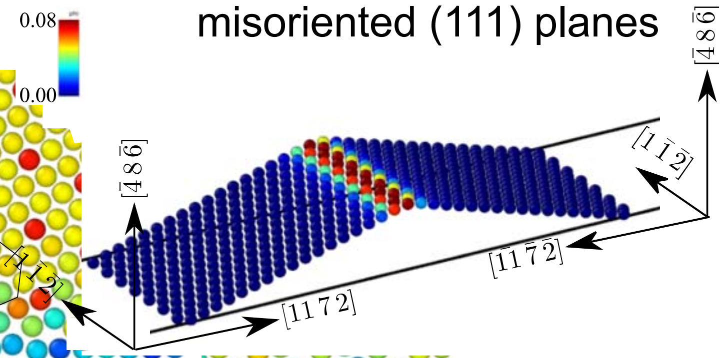


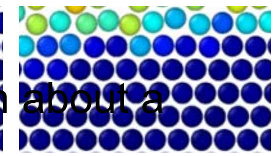
Mechanisms of antithermal grain boundary motion

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

3. Rotation between two misoriented (111) planes

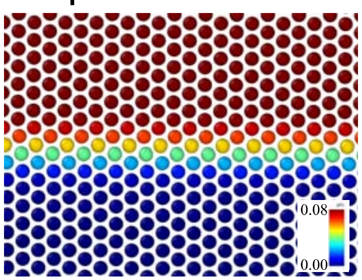


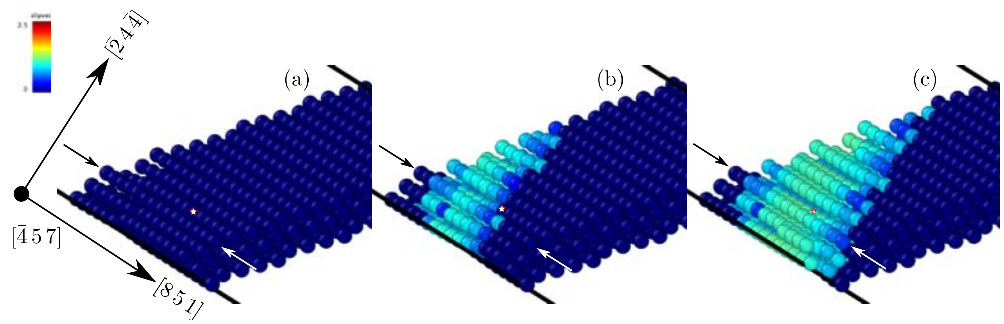


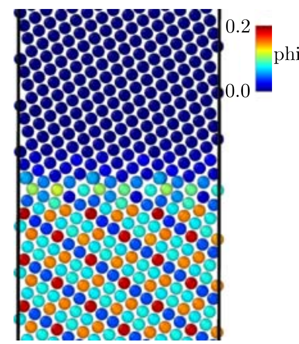


2. In-plane rotation without center on common (111) plane

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







The mechanisms for anti-thermal boundary motion involve a coordinated shuffling or rotation about a common plane, typically (111). Because of the apparent coordinated motion, it bears similarity to a martensitic/military motion rather than a diffusive motion.

Collaboration with Stephen Foiles, Chris O'Brien