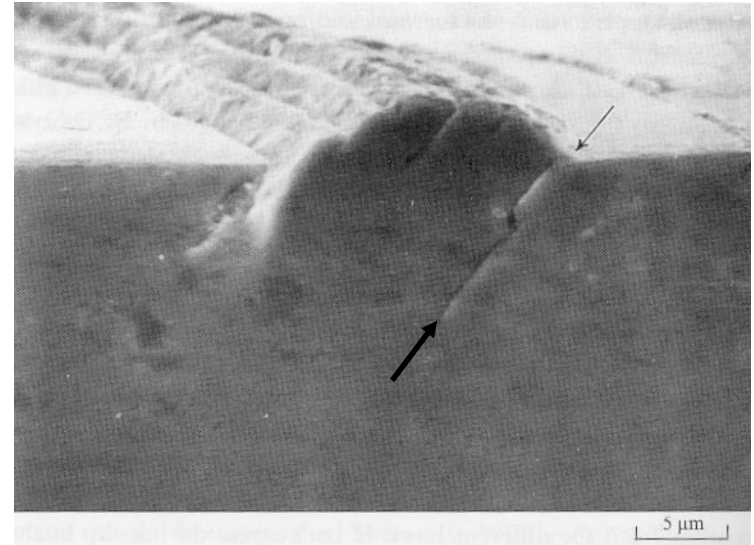
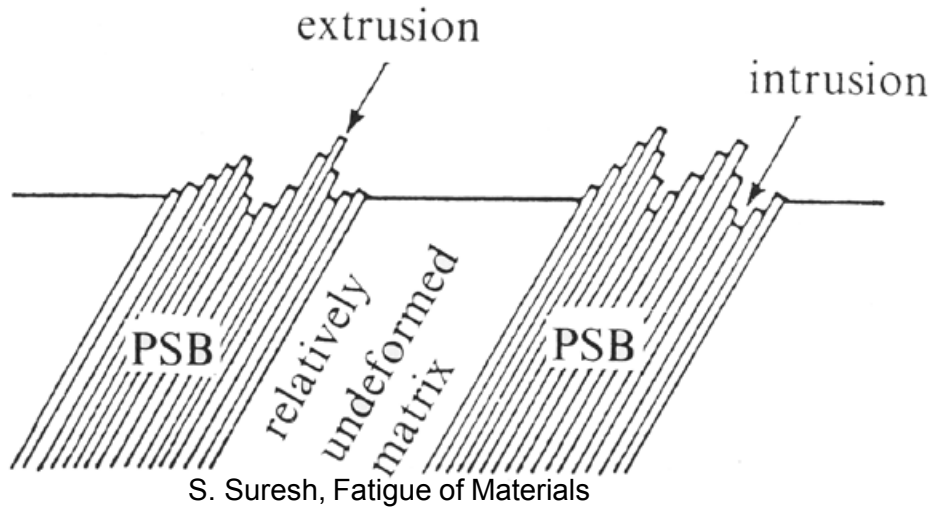


## *Catching the Embryo of Fatigue Crack Nucleation in Nanocrystalline Metals*

Timothy A. Furnish, Daniel C. Bufford, Khalid Hattar, Christopher J. O'Brien, Stephen M. Foiles, Brad L. Boyce

Sandia National Laboratories, Albuquerque, NM

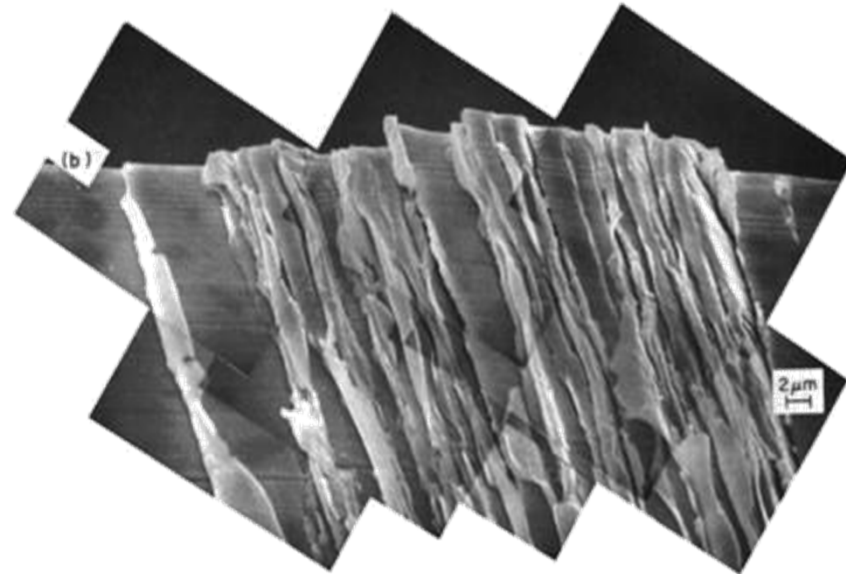
# Is the fatigue mechanism suppressed in nanocrystalline metals?



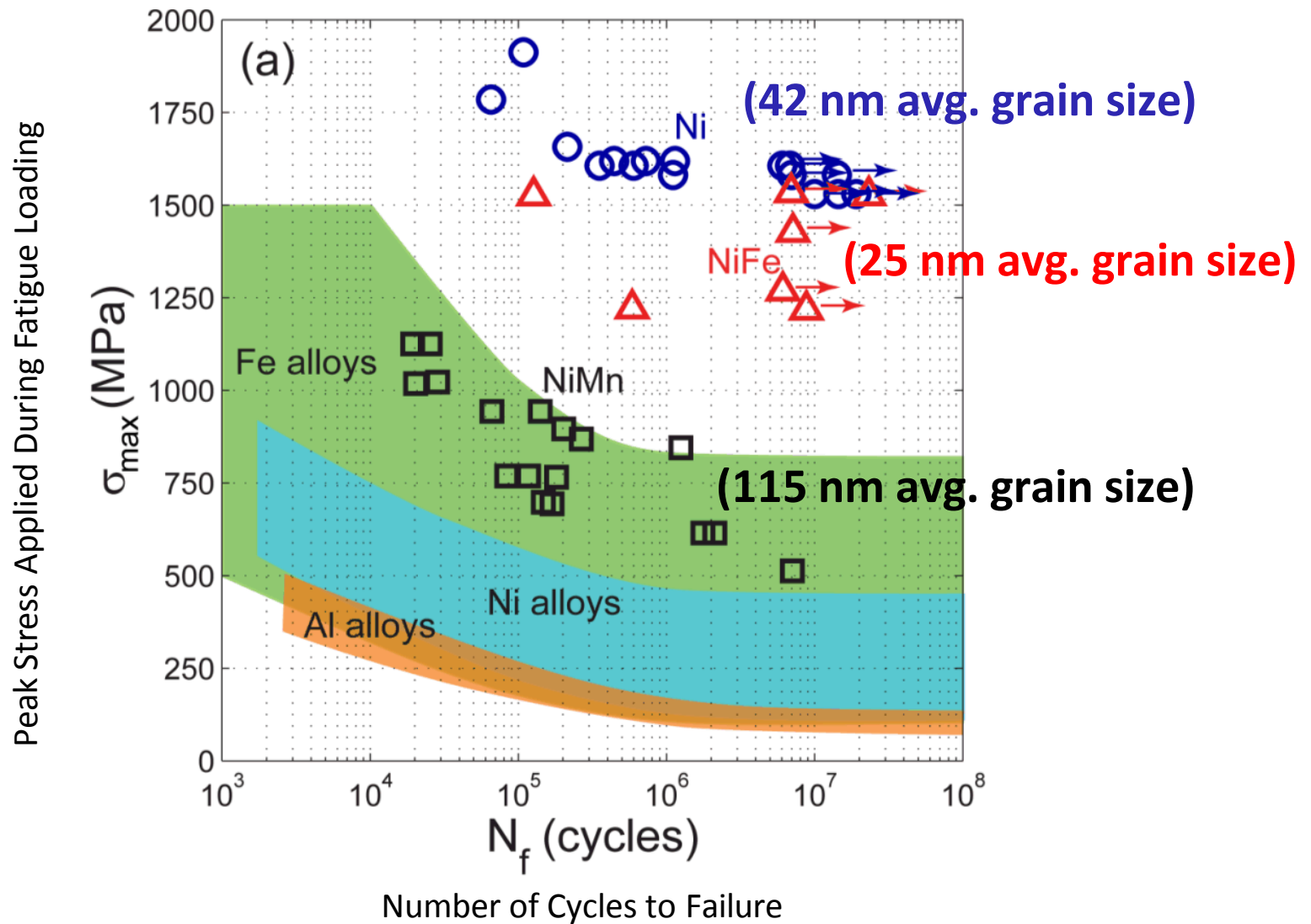
Ma and Laird, 1989

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

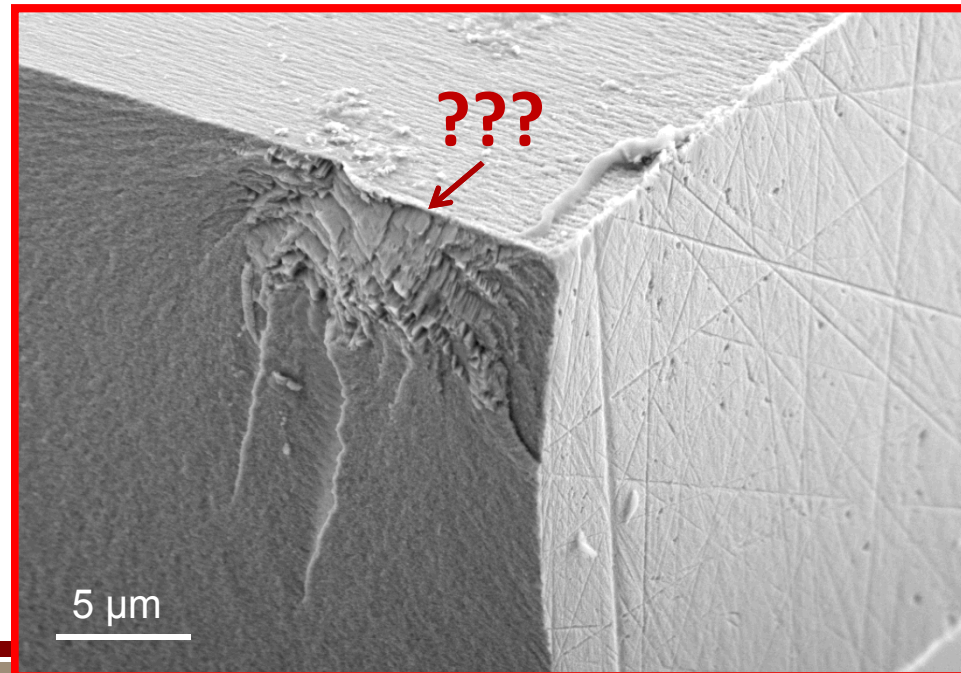
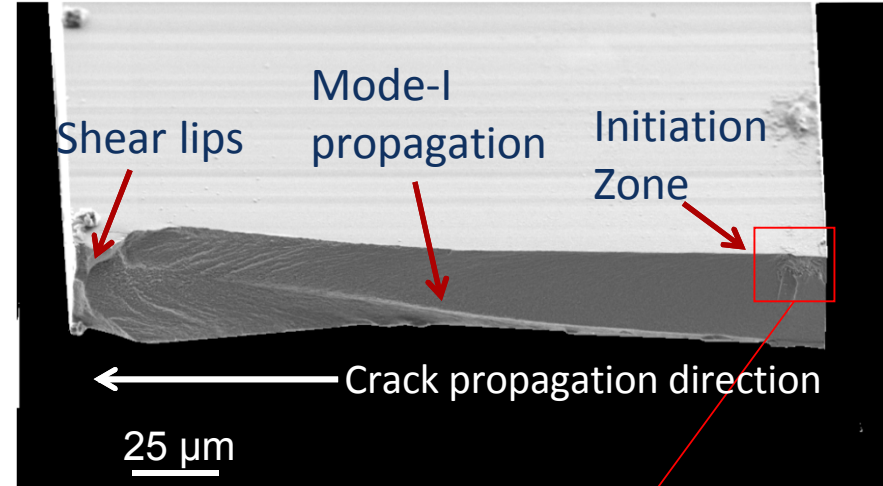
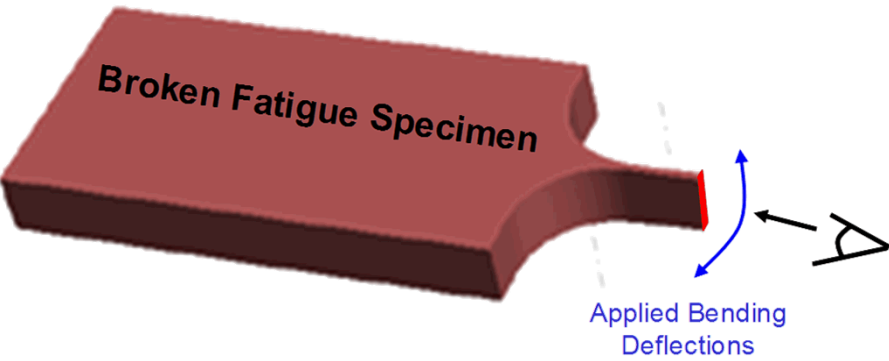
Does the fatigue mechanism change for such small grain sizes?

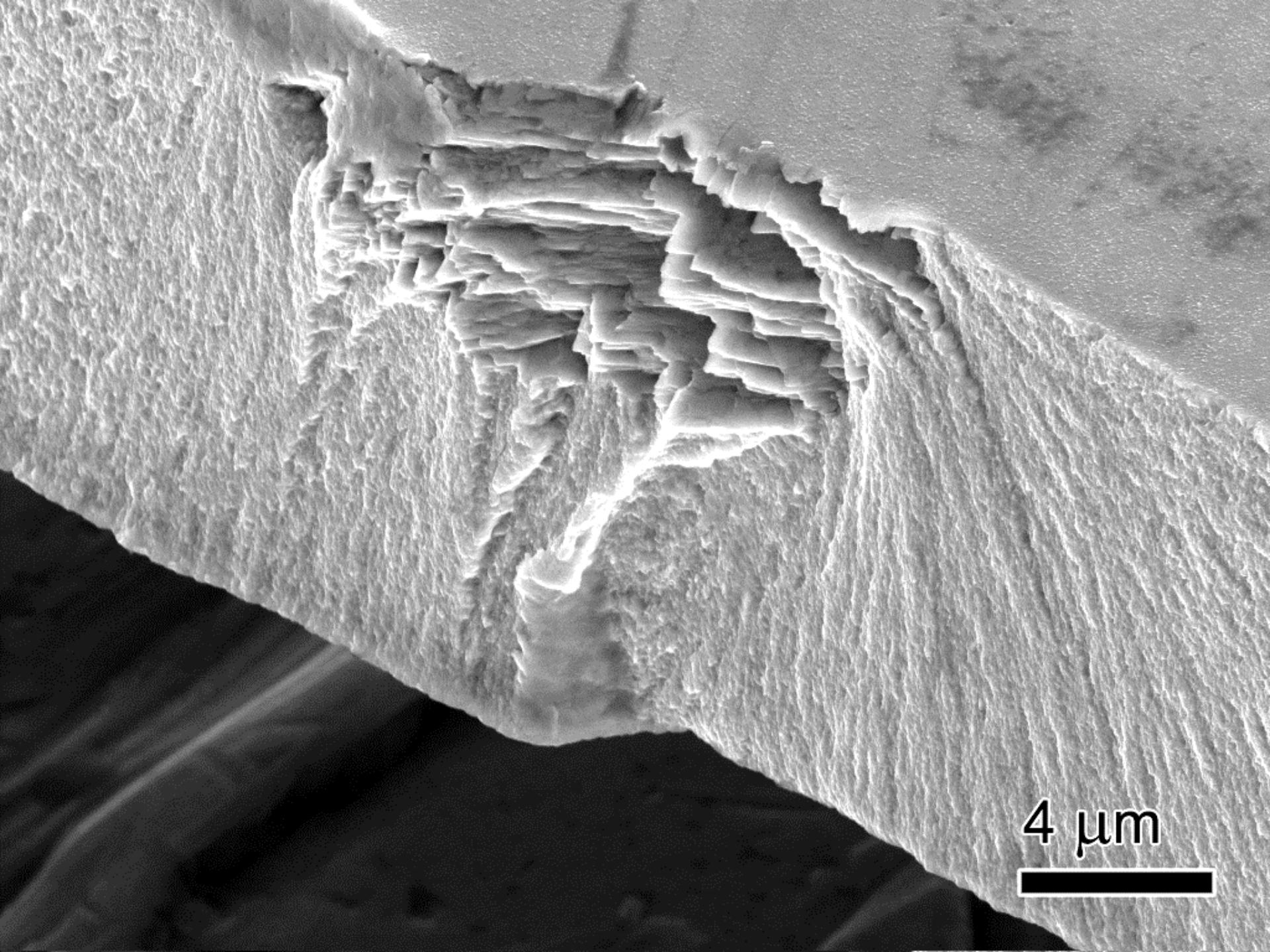


# Superior Fatigue Resistance in Nanocrystalline Alloys



# Unusual initiation zone in nanocrystalline Ni-Fe Alloy

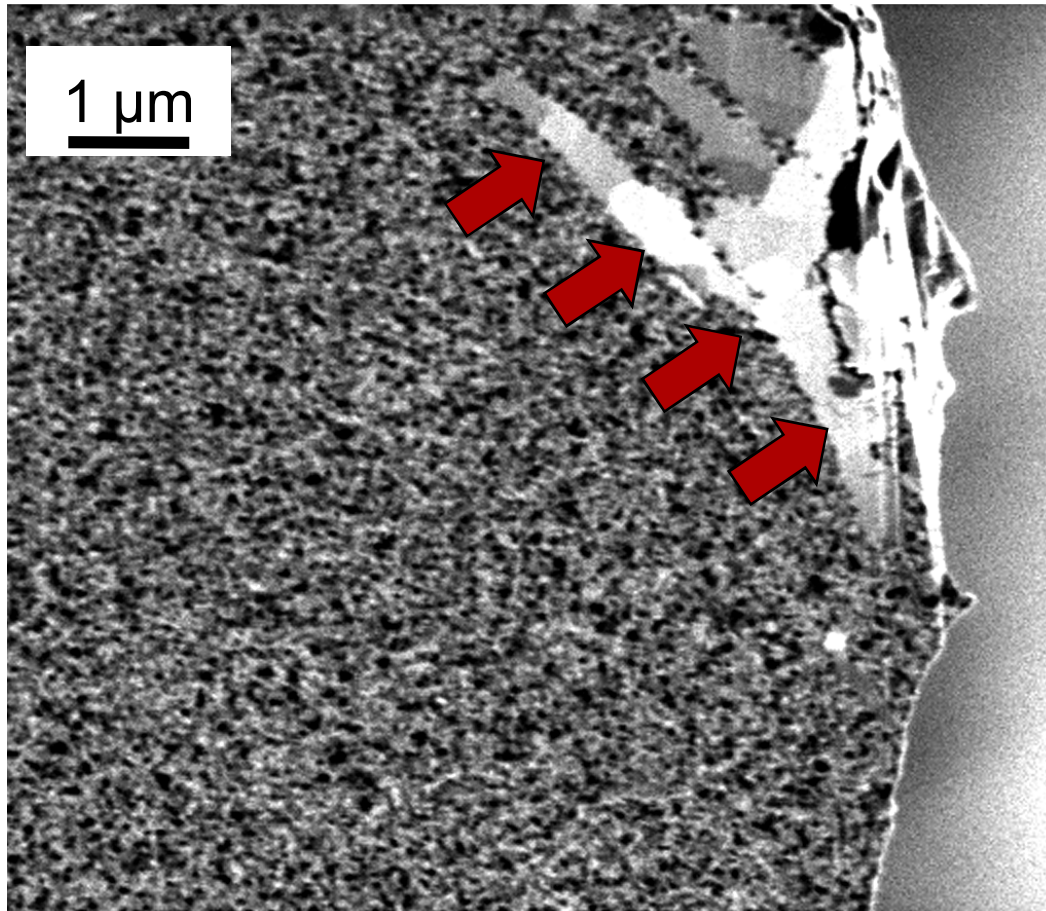




4  $\mu\text{m}$

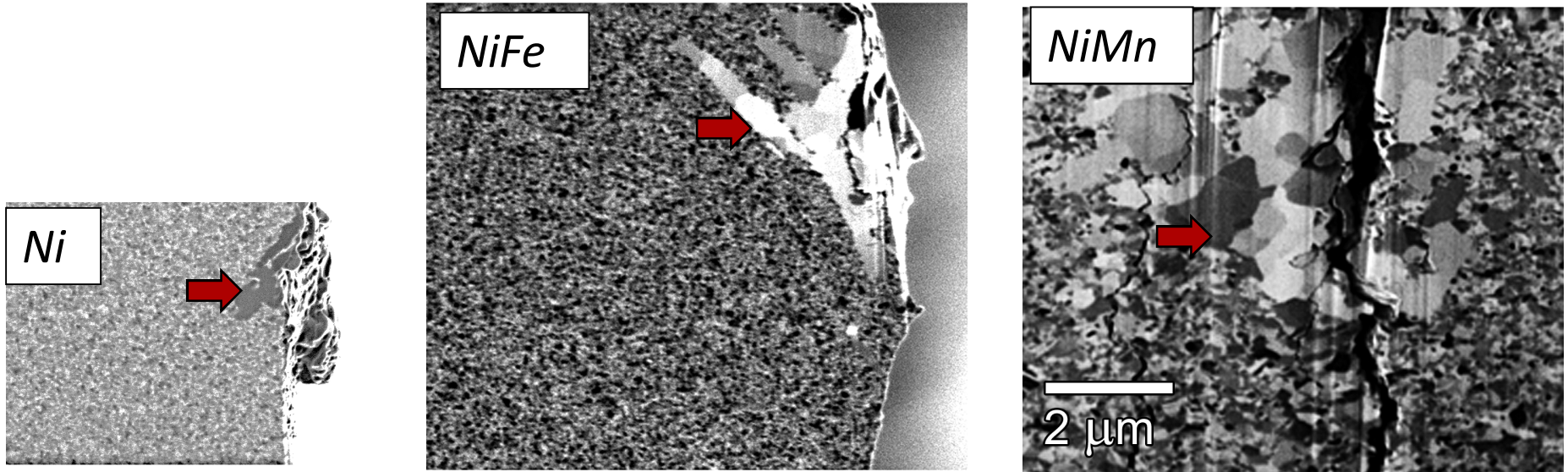


# A cross-section through the 'blocky' zone reveals Very large subsurface grains

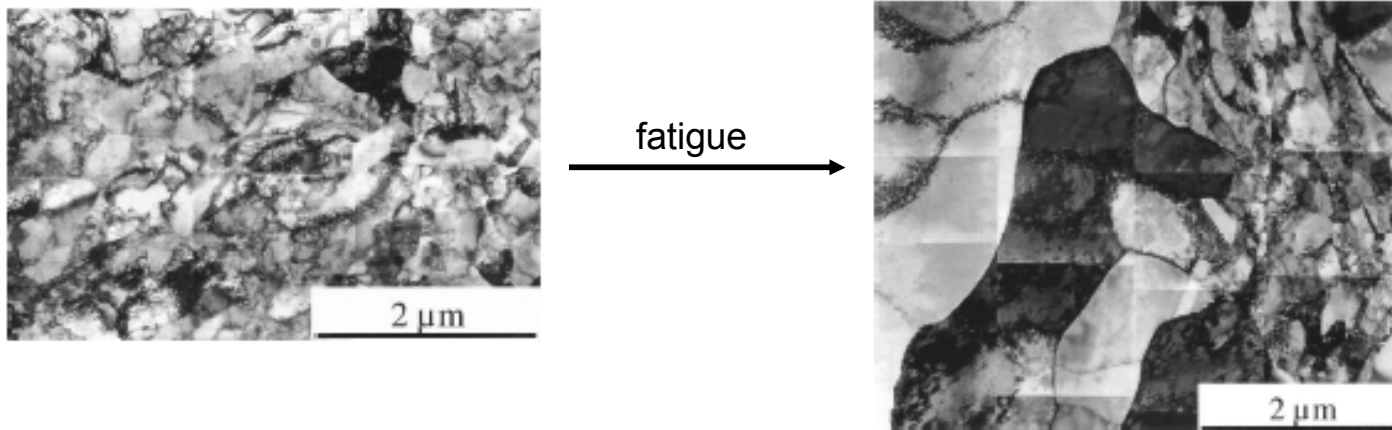


- Fatigue-induced coarsened zone *at room temperature!*
- Coarsened zone only forms locally (abnormal grain growth) in regions of maximum stress, prior to crack initiation

# Fatigue-induced abnormal grain growth occurs at the site of crack initiation in all 3 alloys...

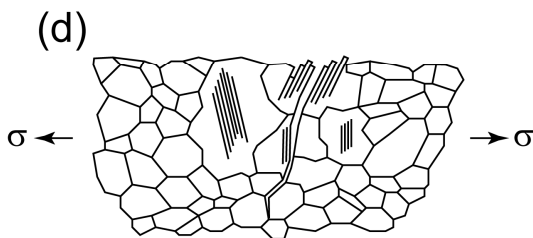
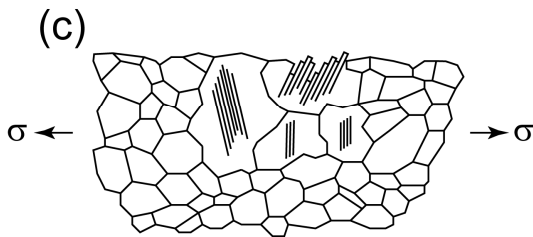
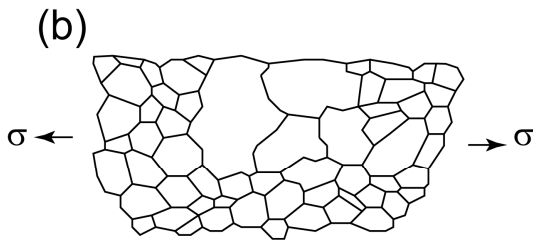
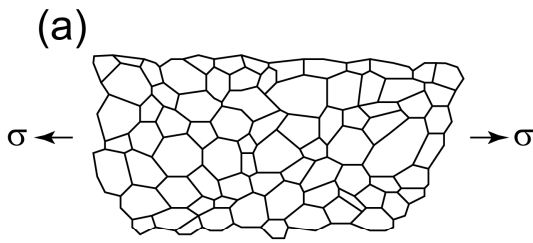


Boyce and Padilla, *Metall. And Mater. Trans. A*, 2011



- Highly defected (fabricated by severe plastic deformation)
- No connection noted between grain growth and crack initiation

Höppel, Zhou, Mughrabi, and Valiev, *Philosophical Magazine*, 2002

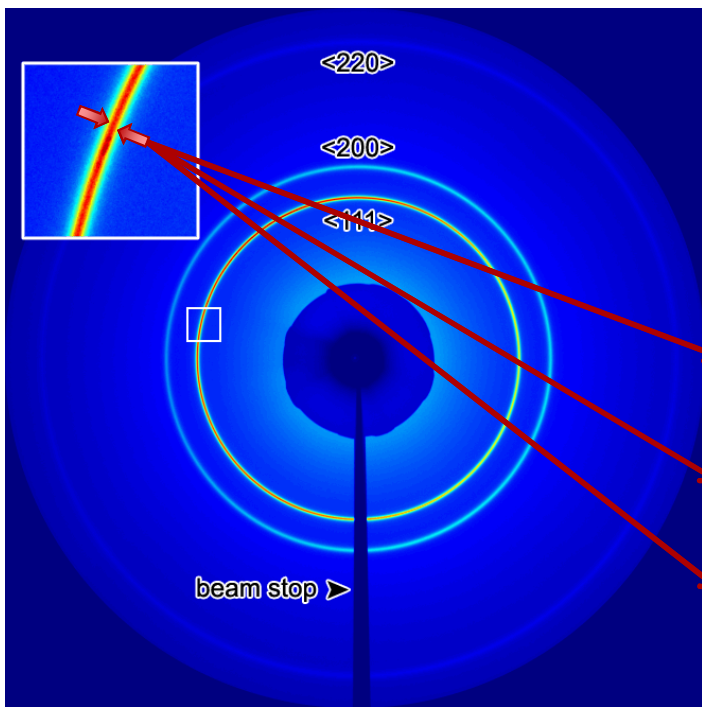
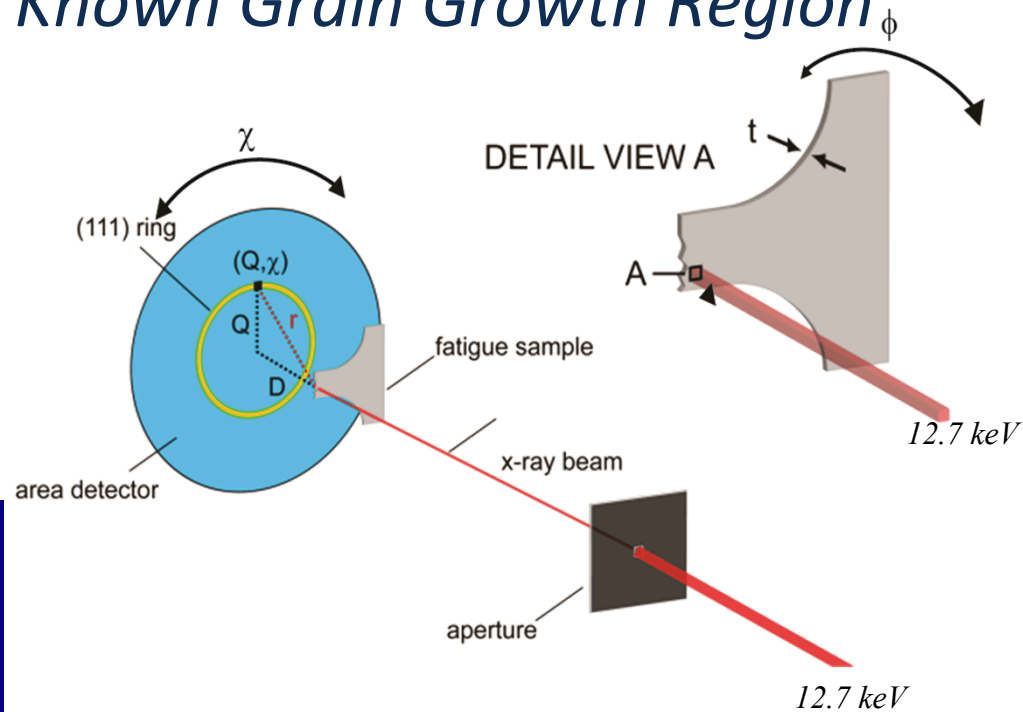


**We postulate that in nanocrystalline metals ( $d_{gb} < \sim 70$  nm), the fatigue process induces grain growth as a precursor to gross slip and eventual crack initiation**

1. Were these few large grains formed during deposition, not fatigue loading?
2. Does the large grains facilitate cracking, or do the high stresses at the crack drive grain growth?
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



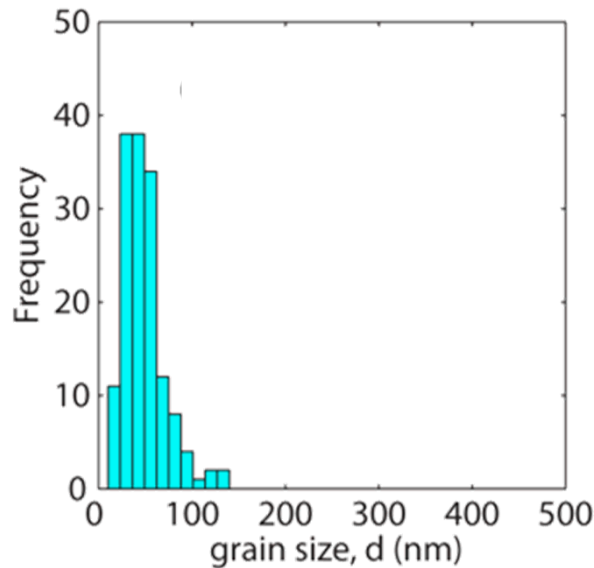
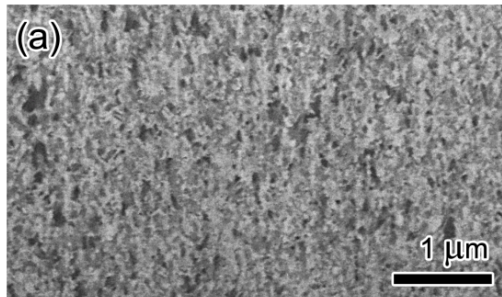
$$B \cos \theta = \frac{K \lambda}{d_{avg}} \quad \text{Scherrer Formula (1918)}$$

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

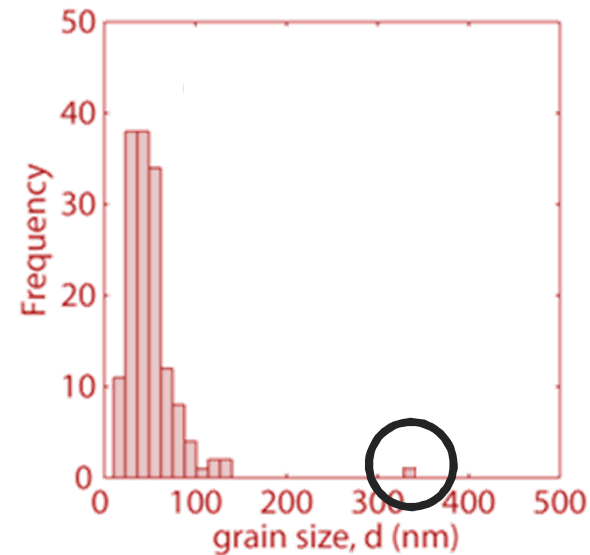
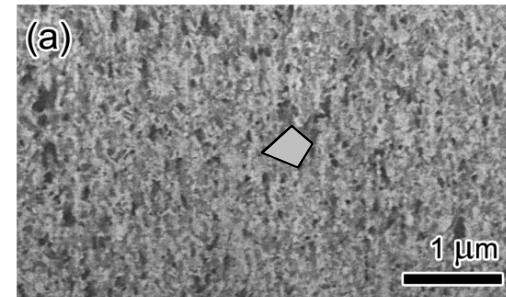
$$A_L = A_L^S A_L^D = A_L^S \exp[-2 \pi^2 L^2 g^2 \langle \varepsilon_{g.L}^2 \rangle] \quad \text{Warren-Averbach (1959)}$$

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

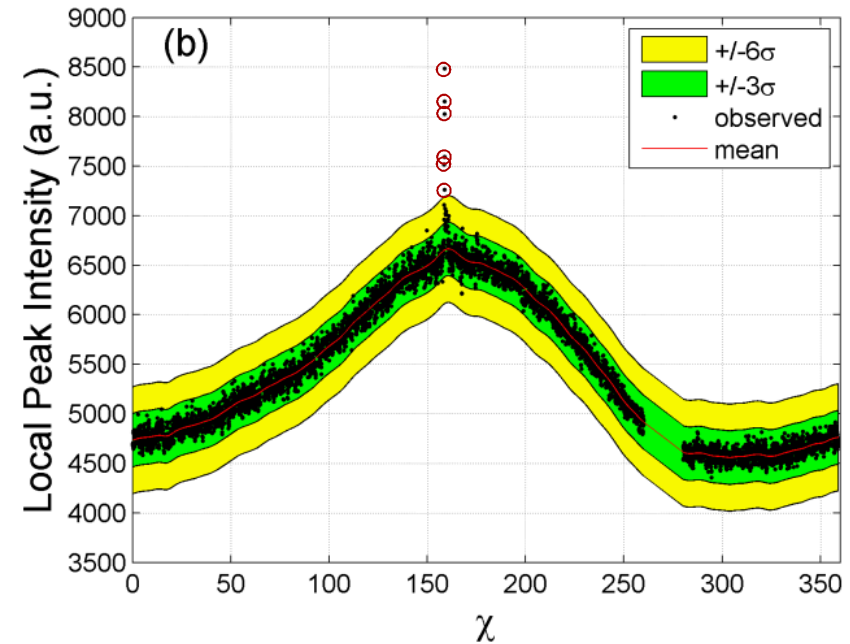
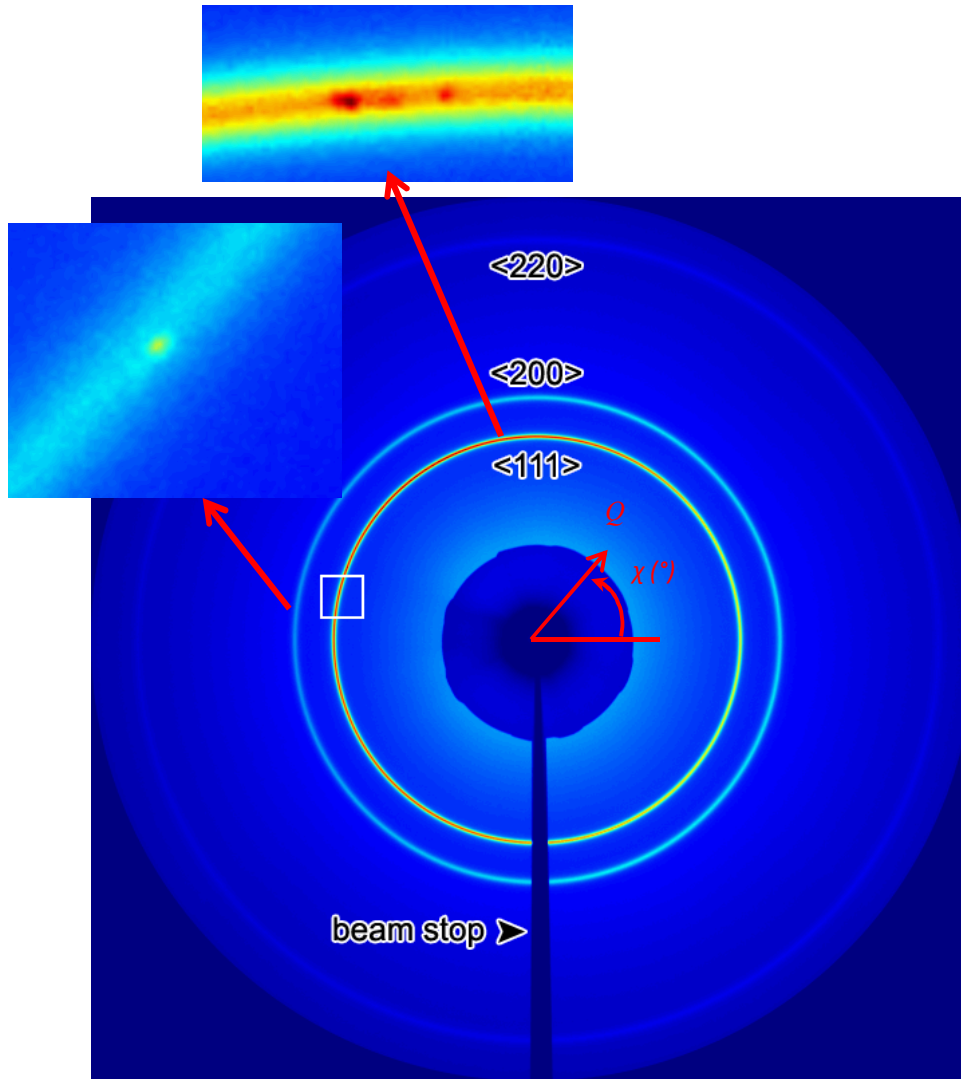
Before abnormal grain growth



After abnormal grain growth

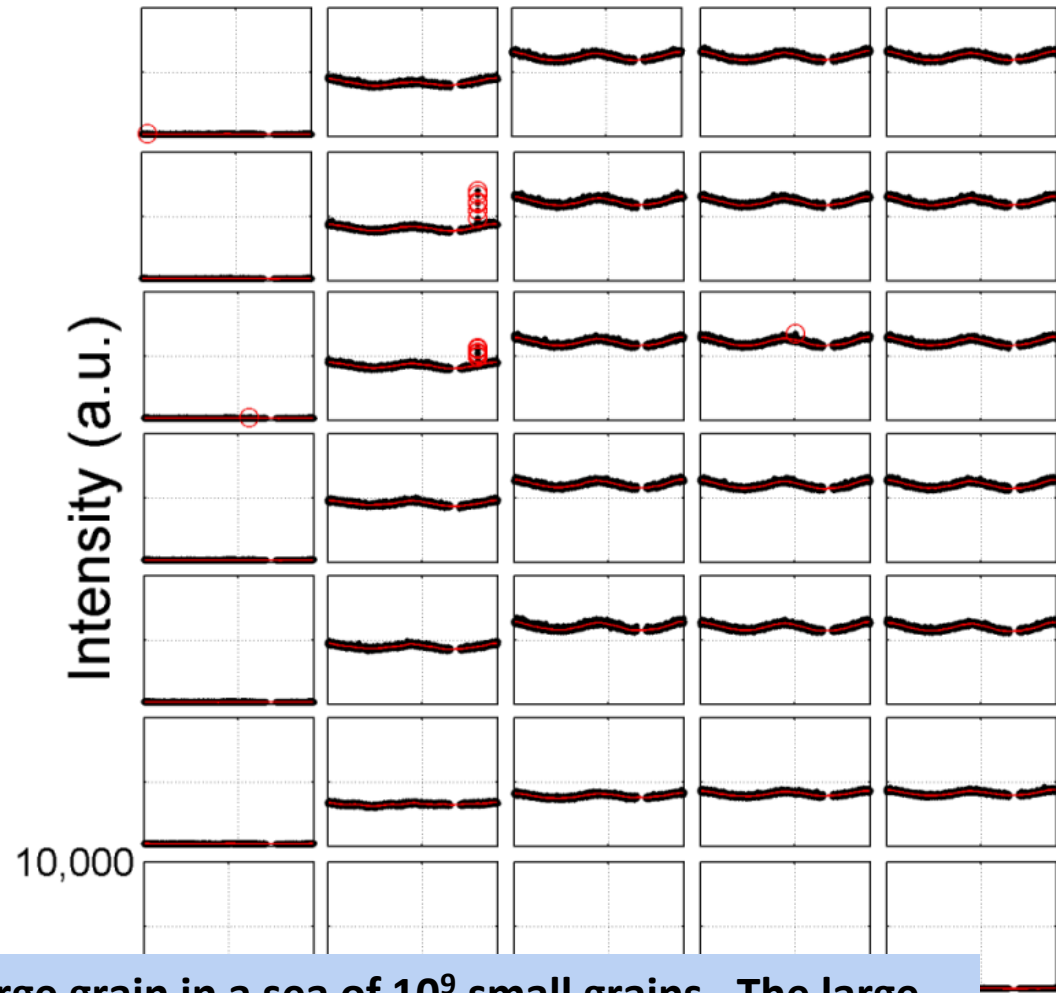
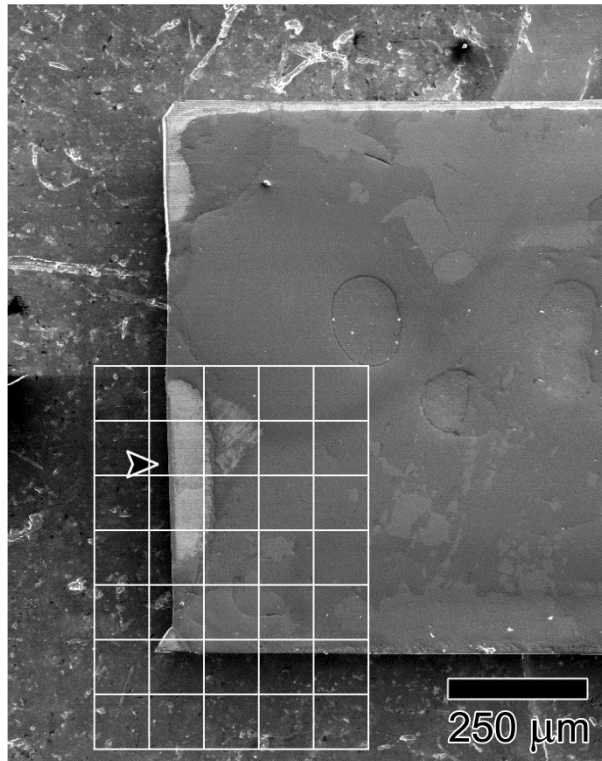


# Preliminary Observation: A 'spike' in the Debye ring.



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

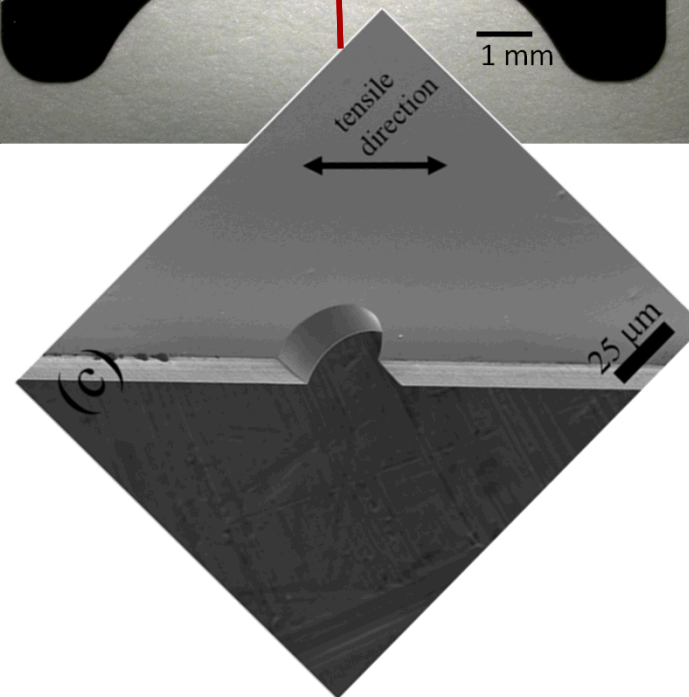
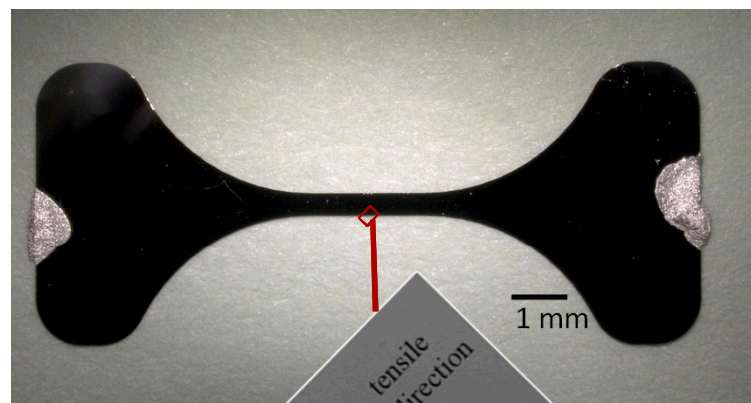
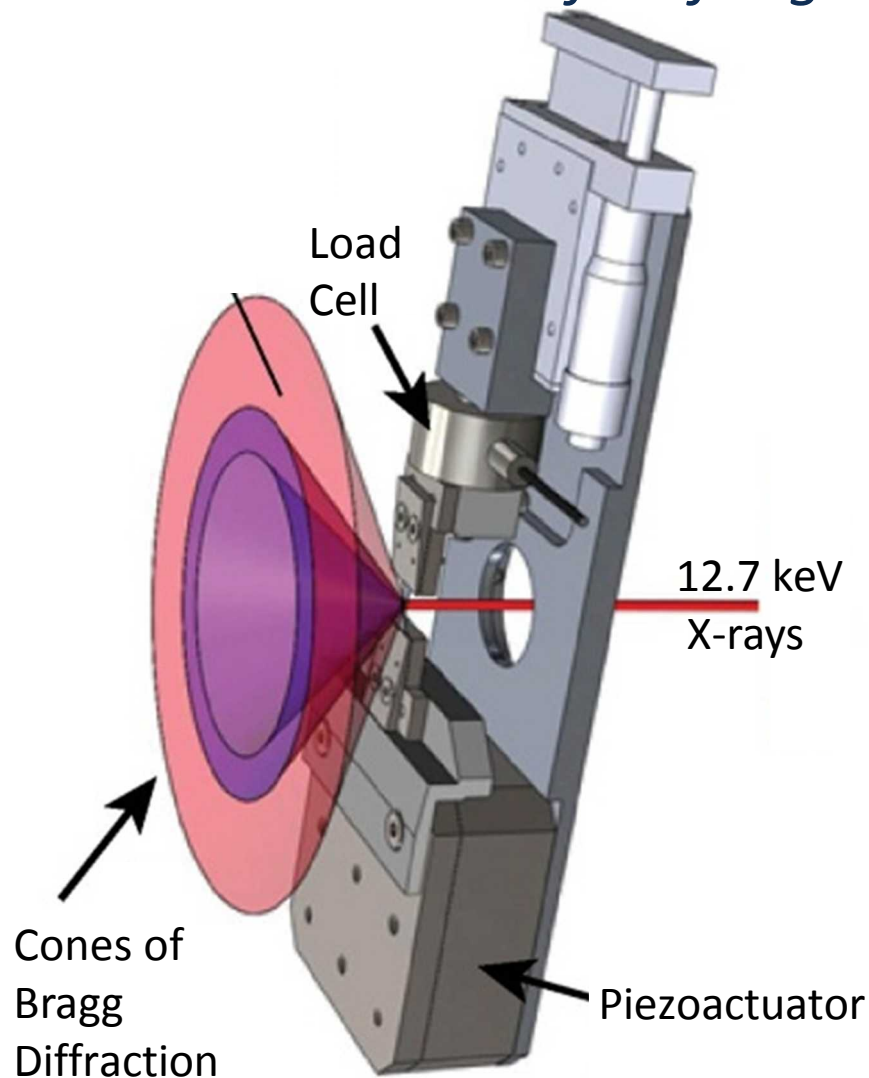
# Confirmation: the intensity spike occurs in the known grain growth location and nowhere else



**We've identified 1 large grain in a sea of  $10^9$  small grains. The large grain occupies  $\sim 0.00001\%$  of the interrogation volume, and is identified with a statistical confidence  $\gg 99.9999998\%$  ( $6\sigma$ ).**

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

# Piezo-actuated thin film fatigue tester for the synchrotron



Elastic stress concentration factor:

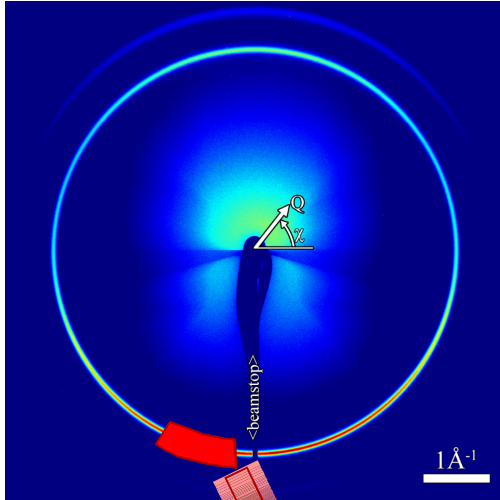
$$K_t=2.8$$

Fatigue stress concentration factor:

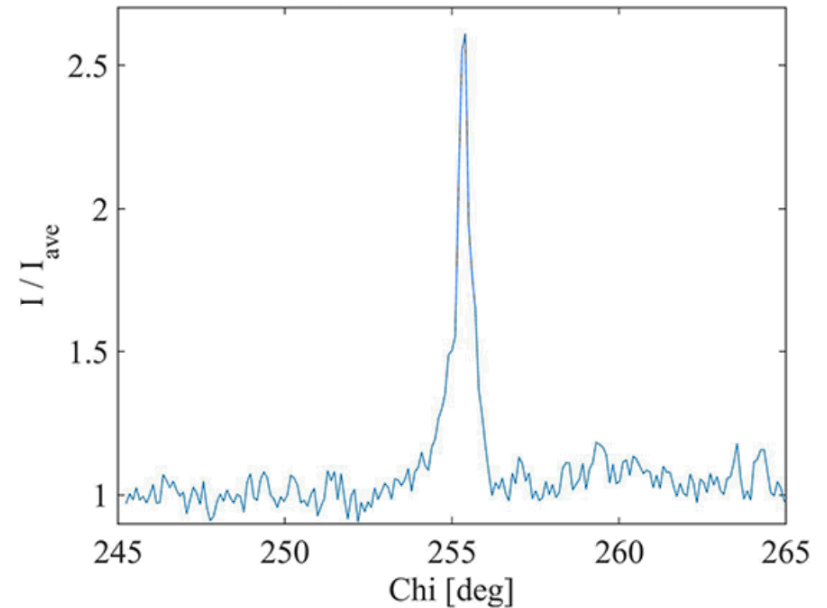
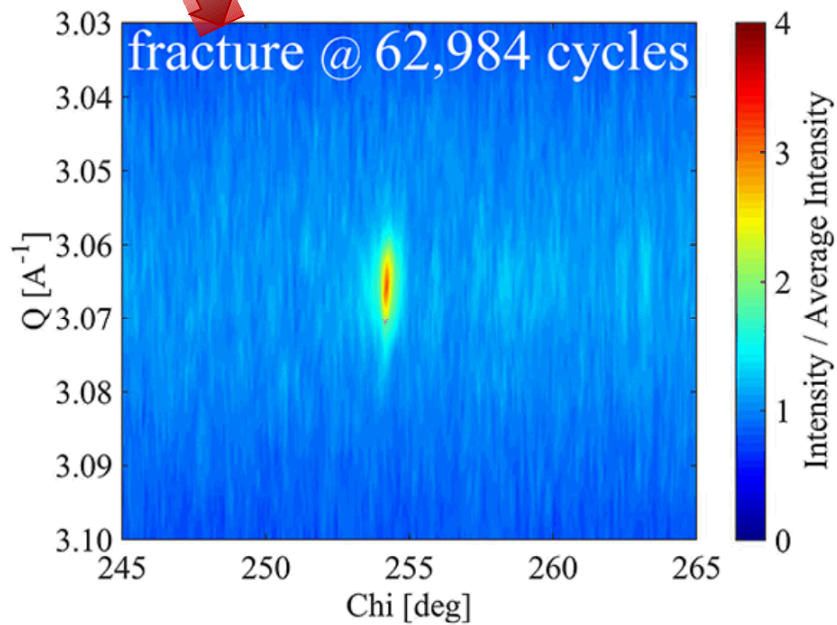
$$K_f=1.5$$

***There are  $\sim 10^8$  grains within 1 radius of the notch tip***

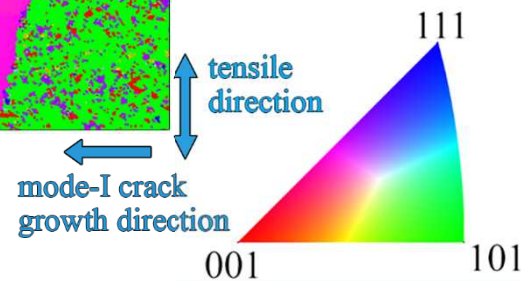
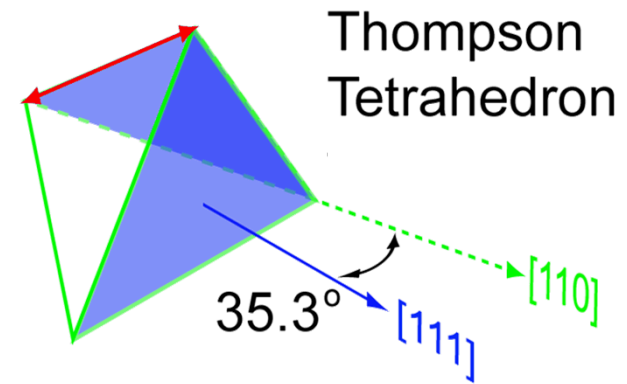
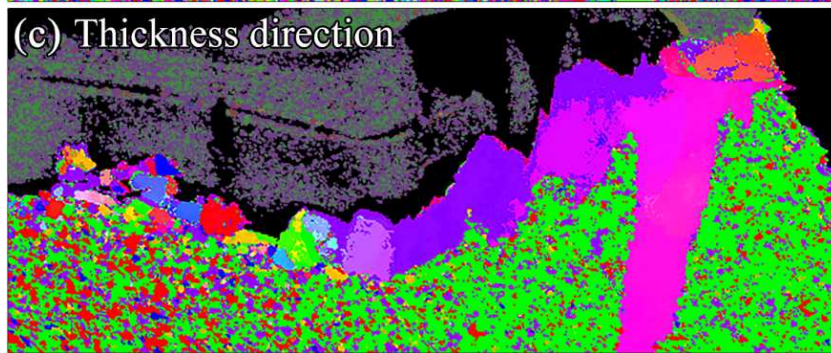
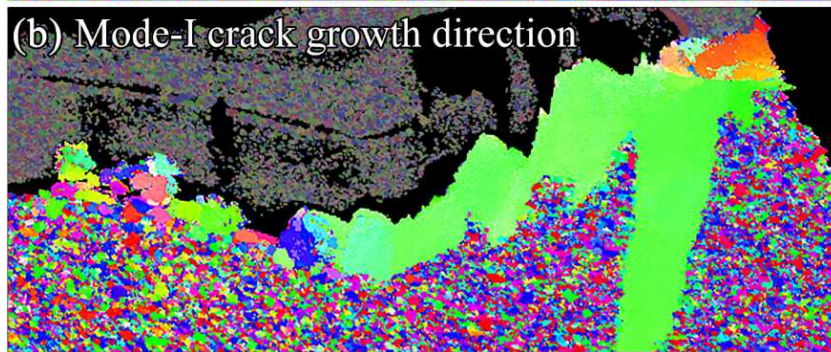
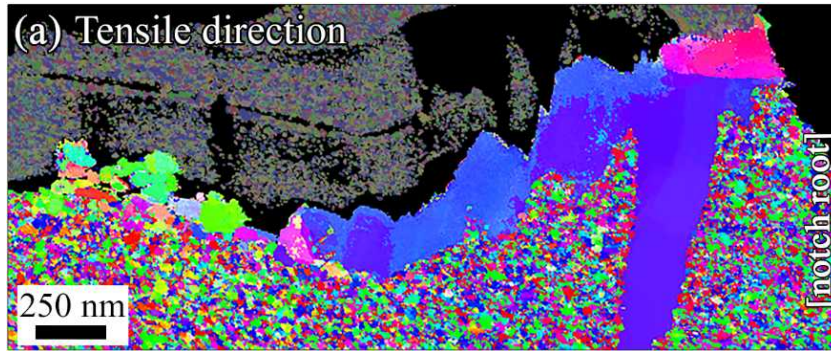
# Detecting the onset of grain growth during fatigue



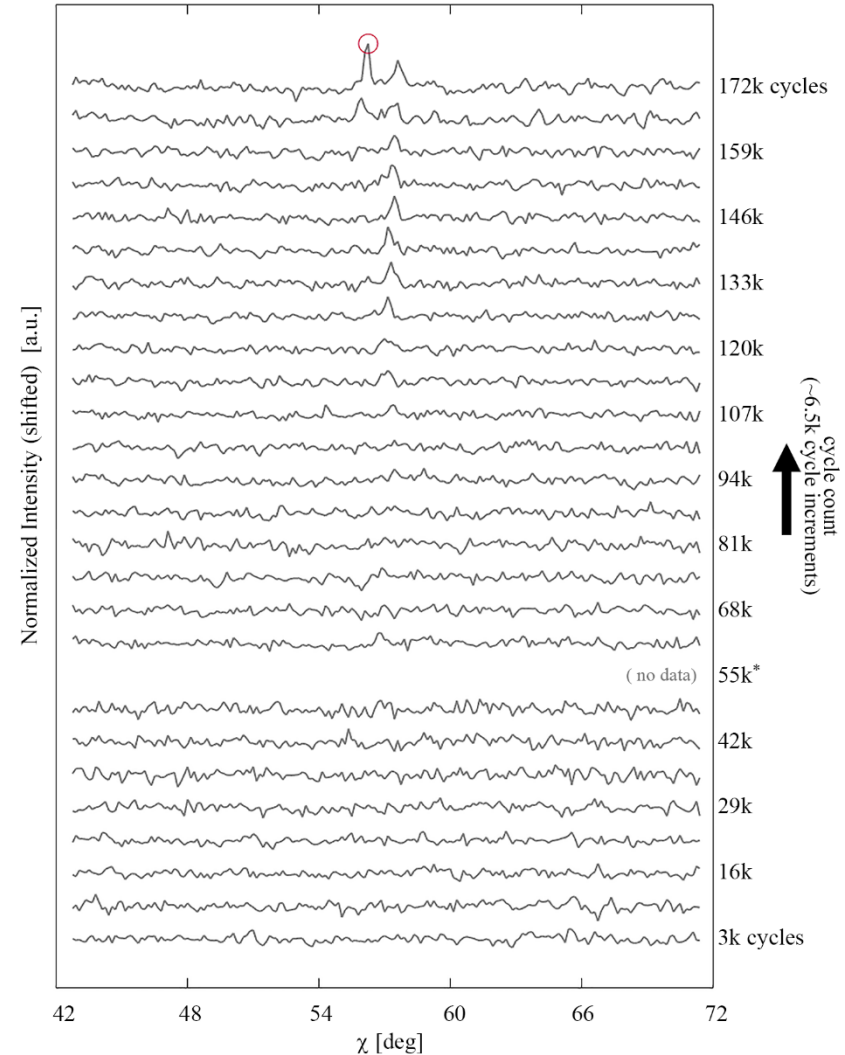
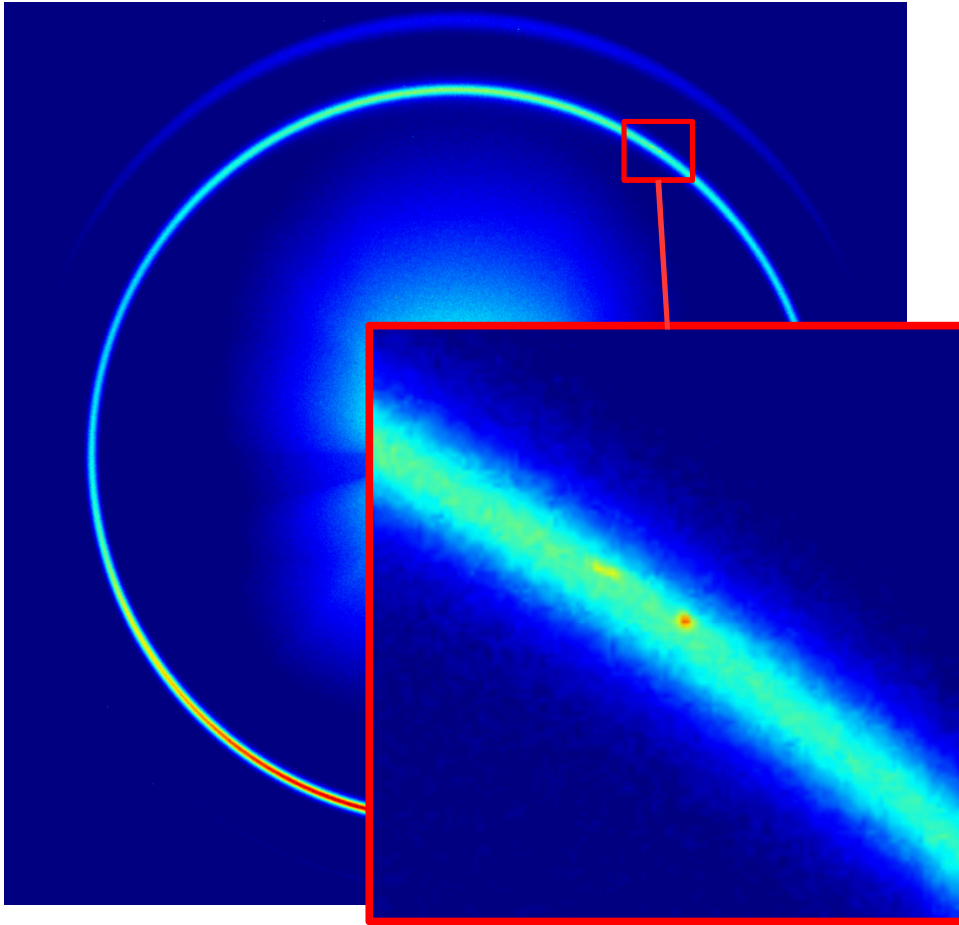
Looking at a  $20^\circ$  arc of the (111) diffraction ring...



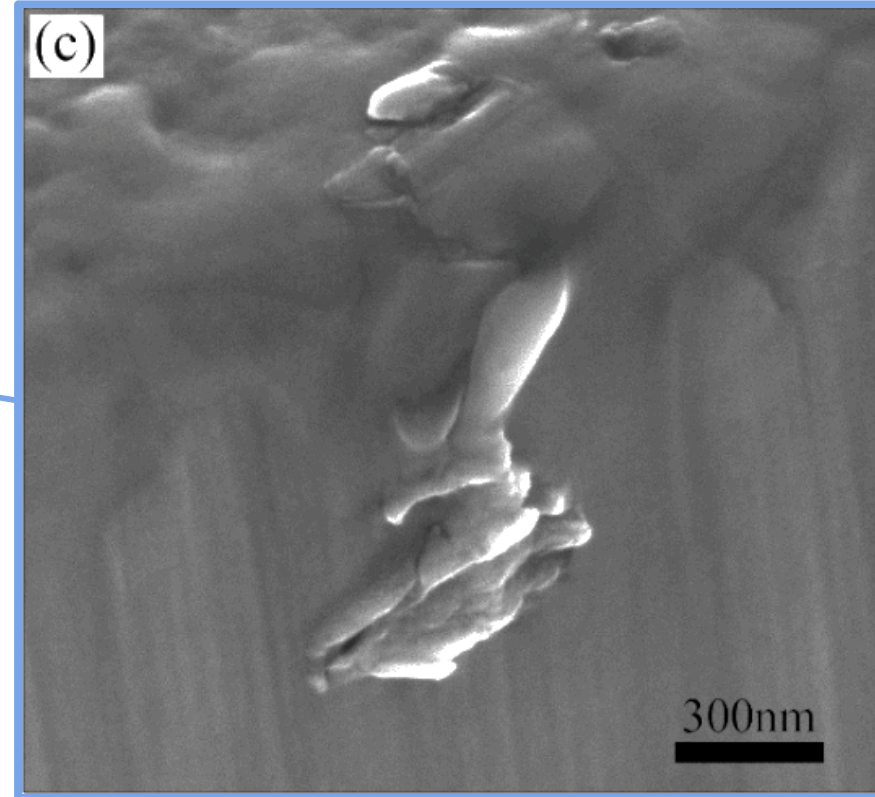
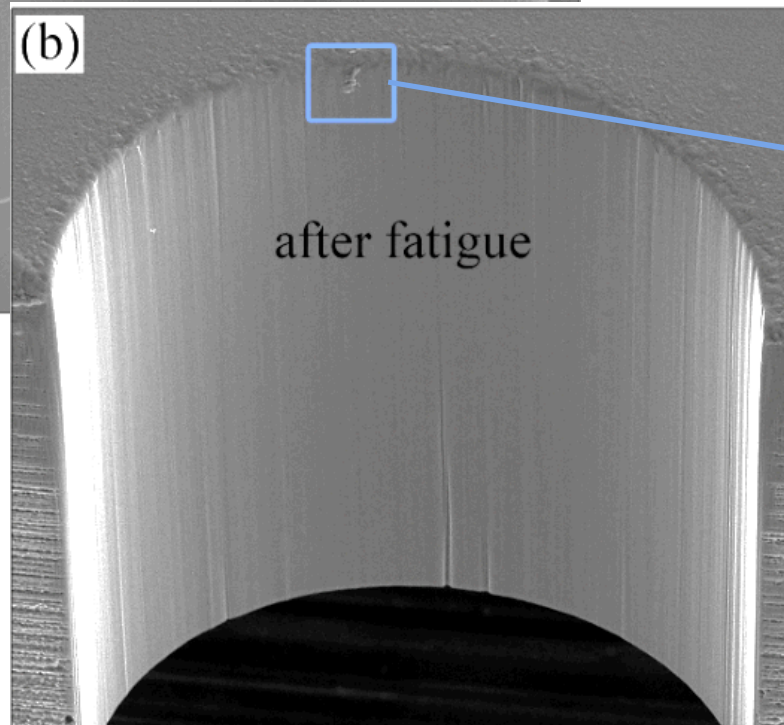
After x-ray detection, the abnormally large grains are confirmed by FIB dissection + precession electron diffraction



# Can we interrupt a fatigue test at the onset of Abnormal grain growth?

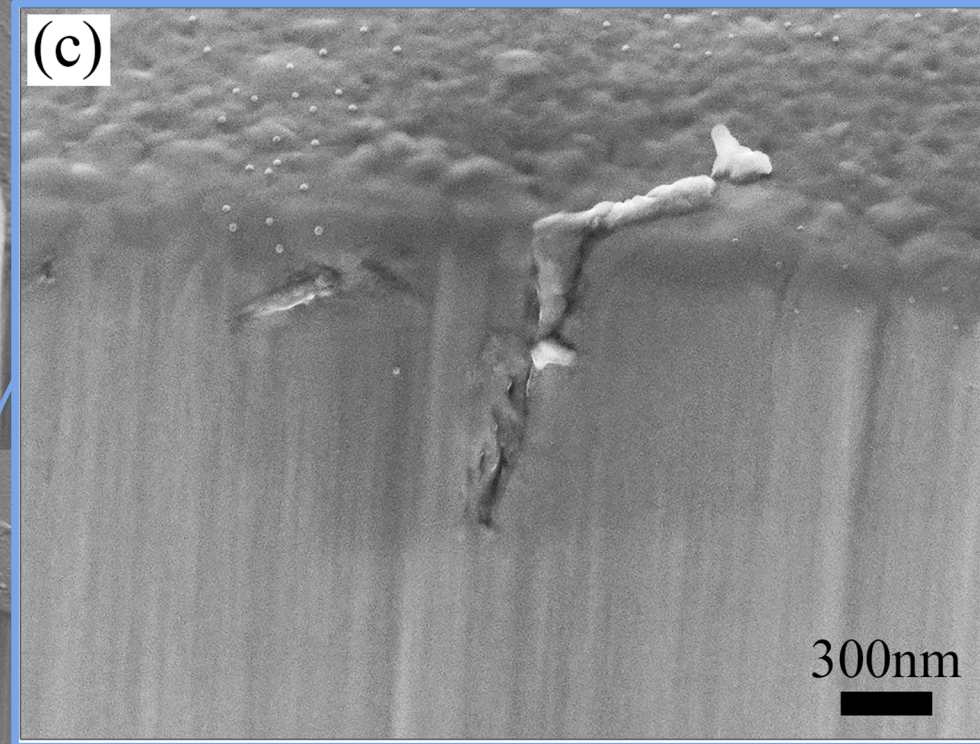
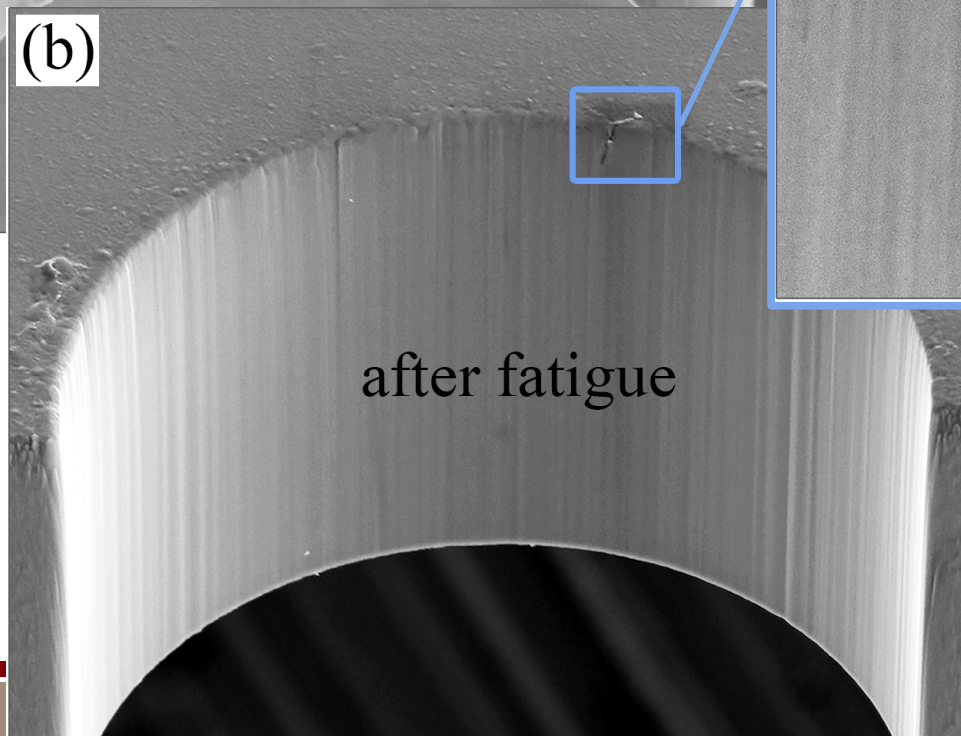
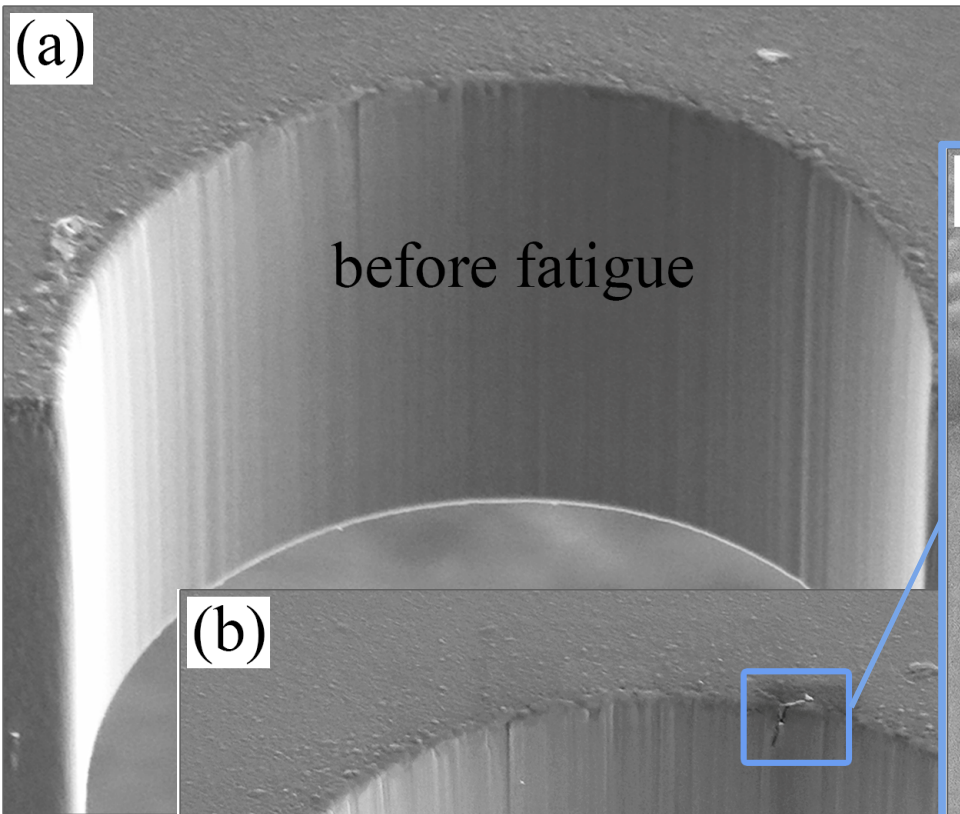


# Interrupting based on fatigue testing.

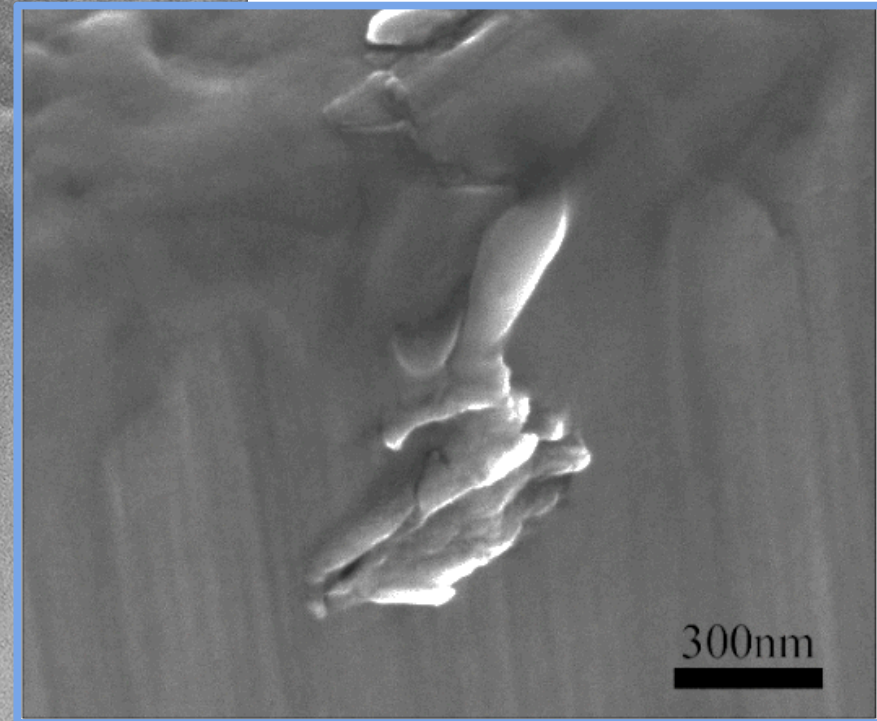
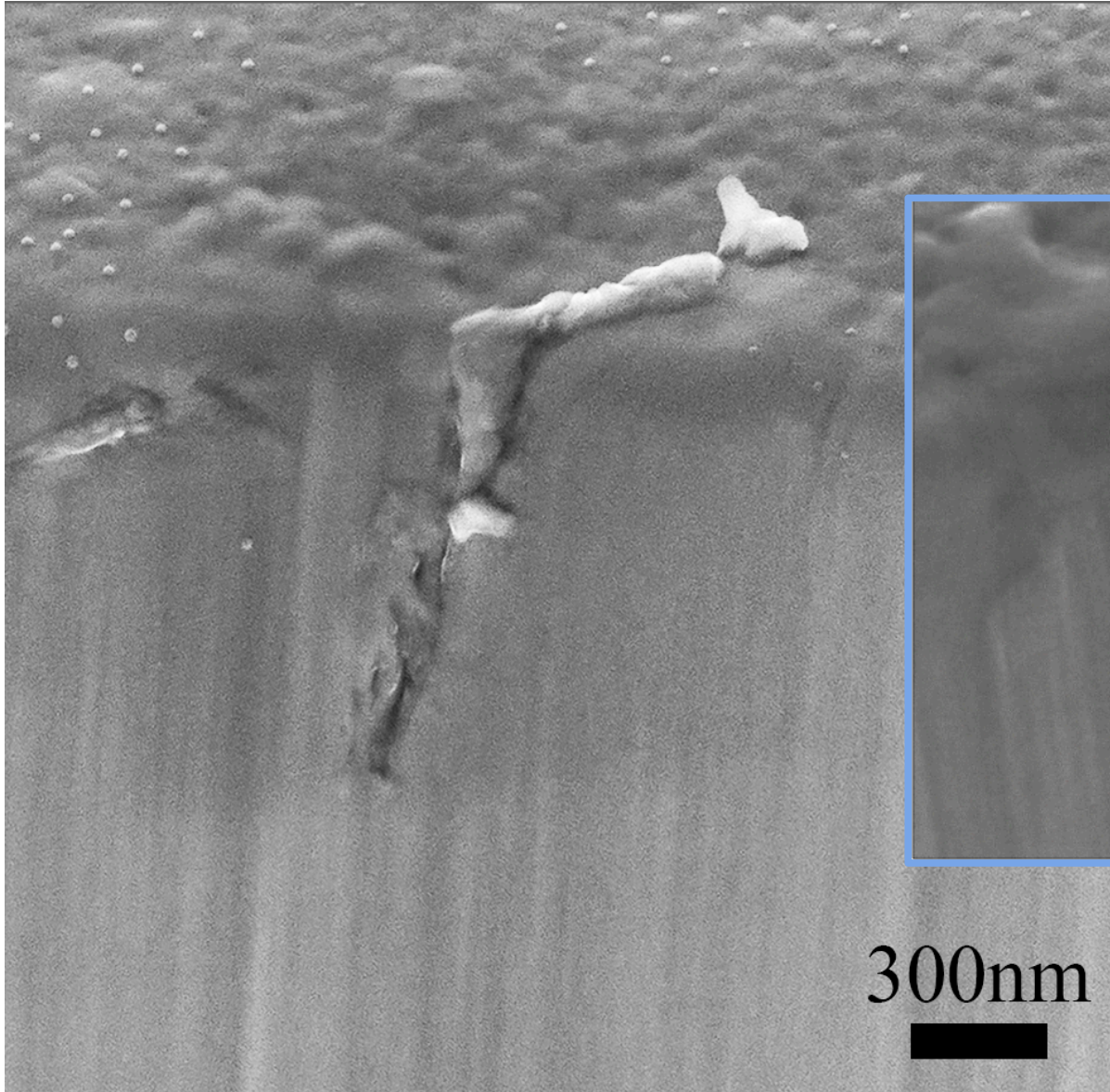


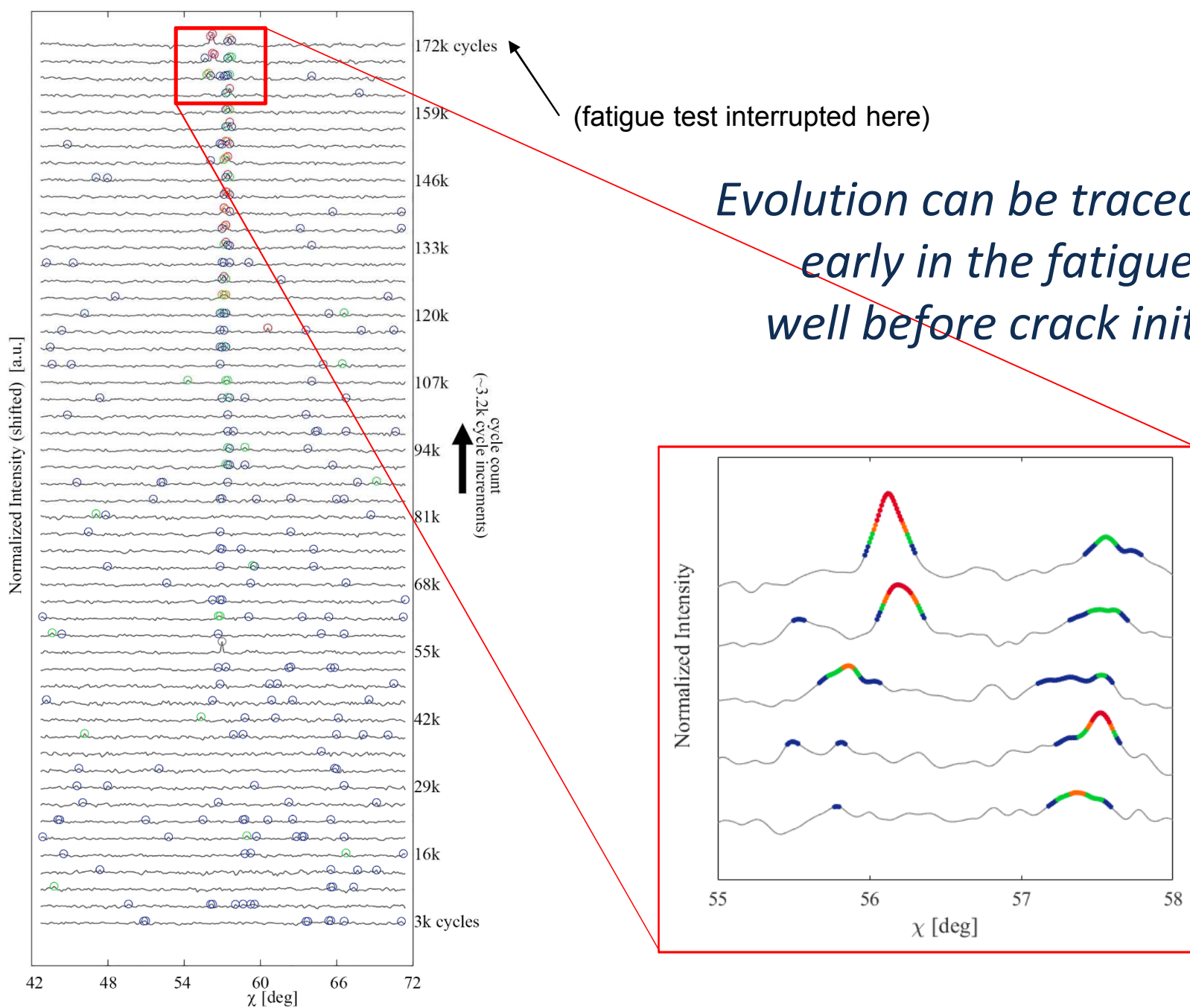
**Interrupted at 172k cycles**

*Another sample, slightly larger, with crack?*

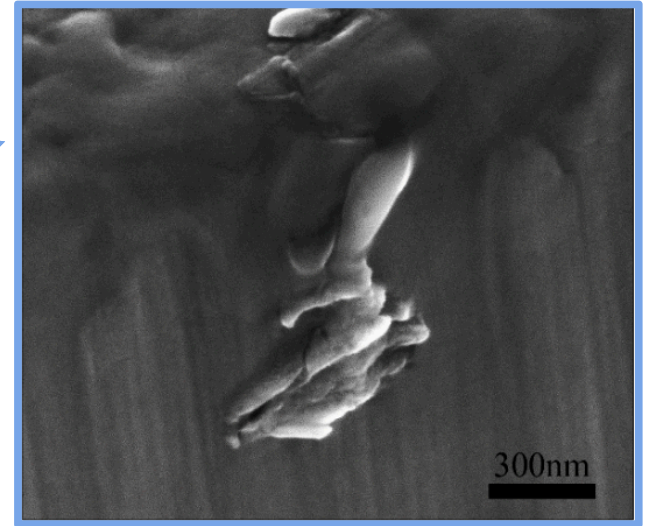
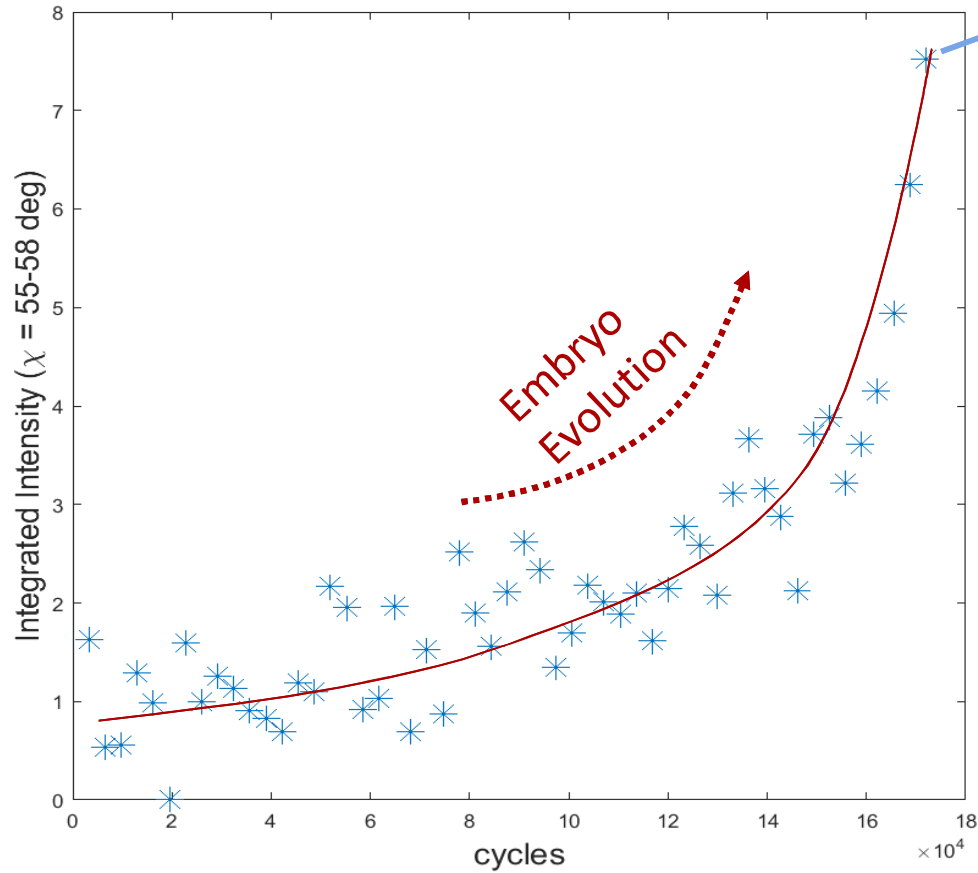


# *A comparison of the “embryo” initiation site*



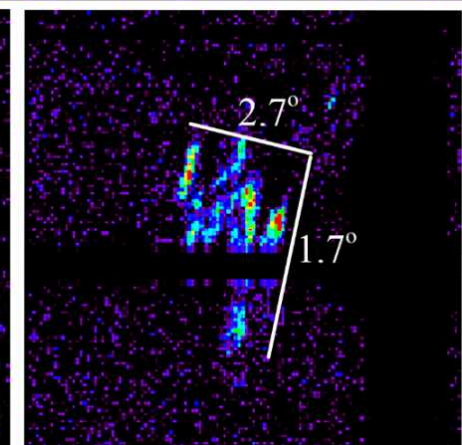
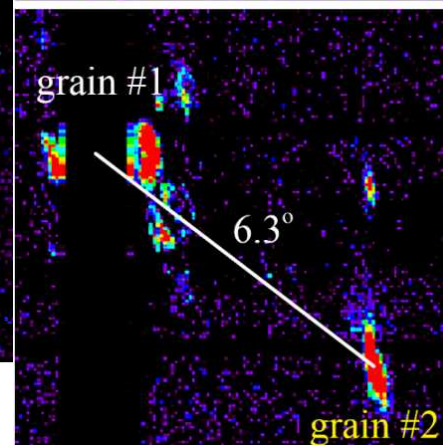
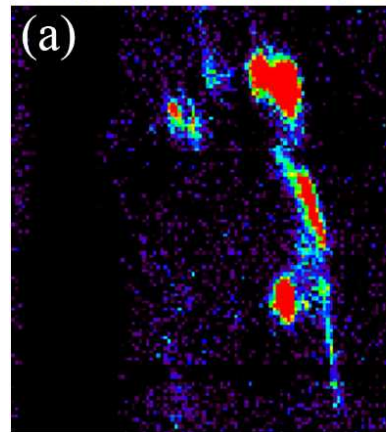
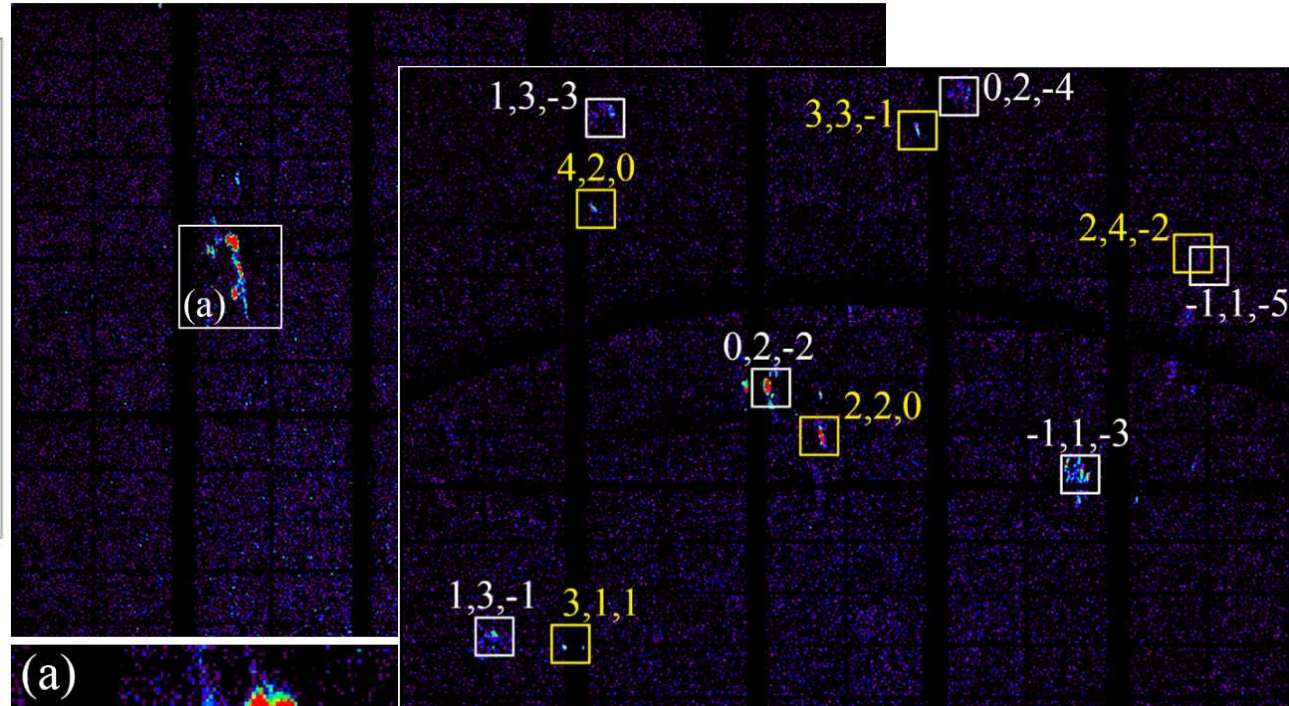
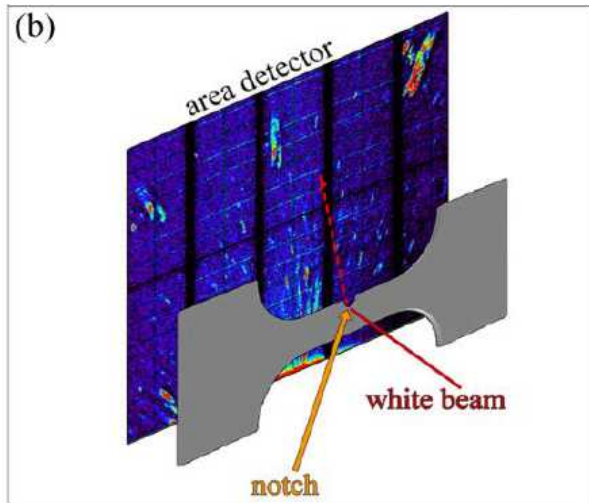


# Kinetics of evolution... leading up to fatigue-crack initiation



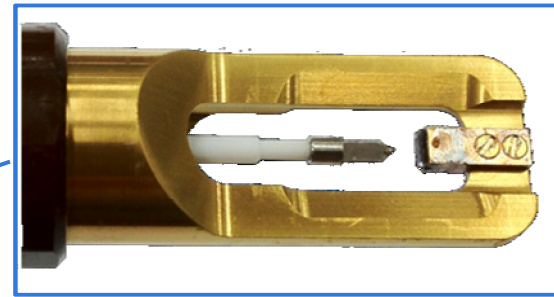
*White Light?*

# Polychromatic microdiffraction reveals orientation of the abnormal grain, subgrain structure, misorientation spread.



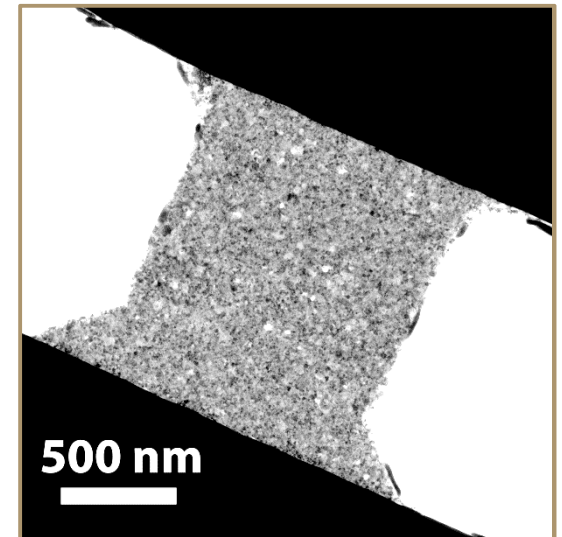
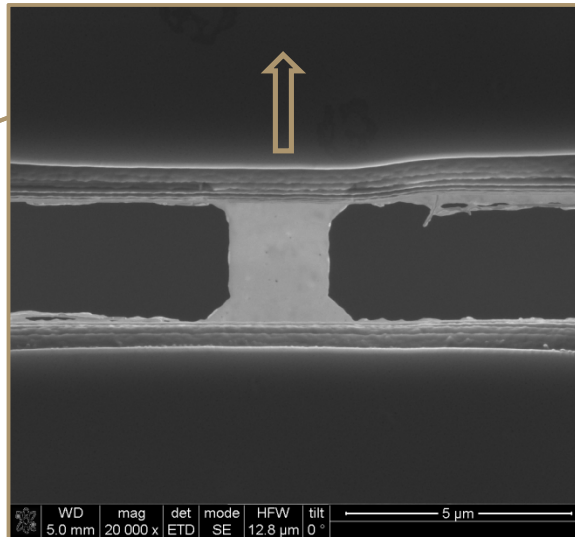
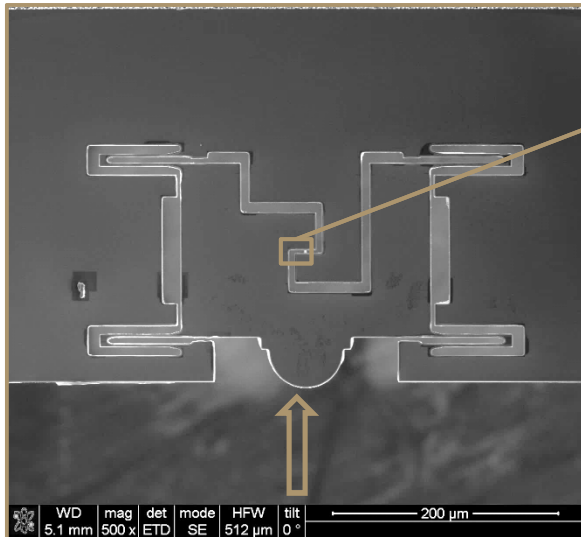
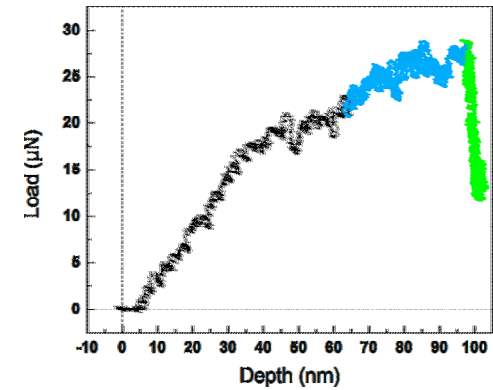
*Can we watch fatigue-induced grain growth  
**directly** in an electron microscope?*

# Quantitative high-cycle fatigue tensile testing in a TEM

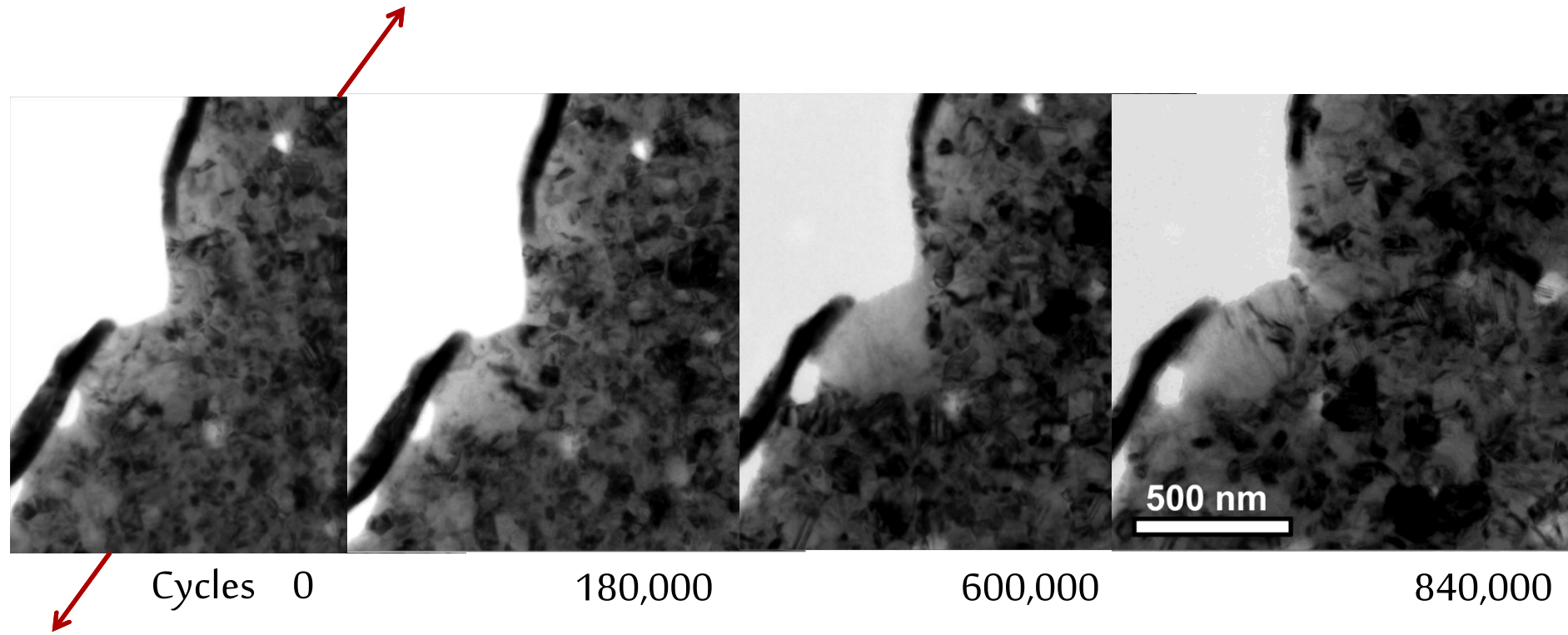


Hysitron PI95 *In Situ* Nanoindentation TEM Holder

- Sub nanometer displacement resolution
- Quantitative force information with  $\mu\text{N}$  resolution
- Concurrent real-time imaging by TEM



*Direct evidence of nano-scale crack initiation after grain growth*



# *What is the mechanism for abnormal grain growth at room temperature during fatigue loading?*

## Strongly shear-coupled grain boundaries

Cahn, Mishin, Suzuki, Acta Materialia, 2006

Homer, Foiles, Holm, Olmsted, Acta Materialia, 2013

## Highly mobile “anti-thermal” grain boundaries

Homer, Holm, Foiles, Olmsted, JOM, 2014

Cantwell, Holm, Harmer, Hoffmann, Scripta Materialia, 2015

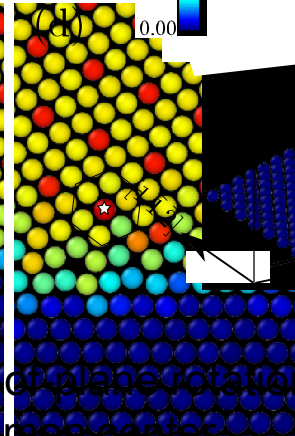
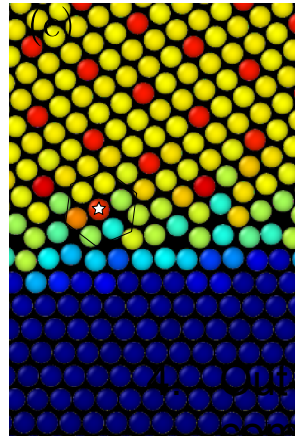
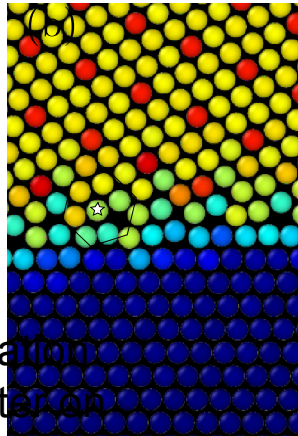
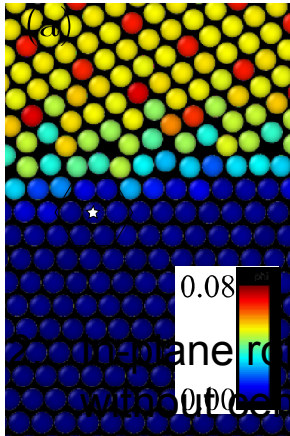
O’Brien and Foiles, J. Materials Science, 2016

## Motion facilitated by defects in the grain boundary

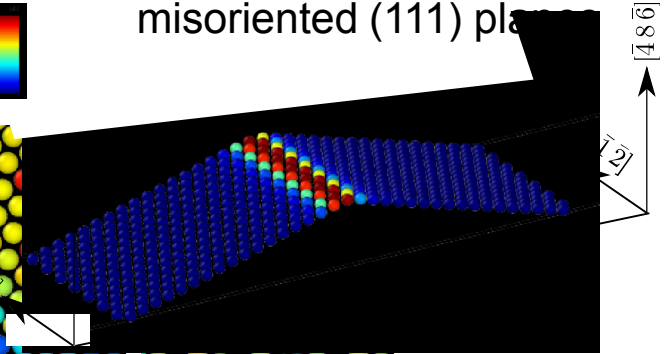
Rajabzadeh, Legros, Combe, Momprou, Molodov, Phil. Mag., 2013

# Mechanisms of antithermal grain boundary motion

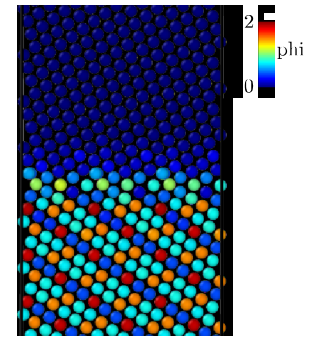
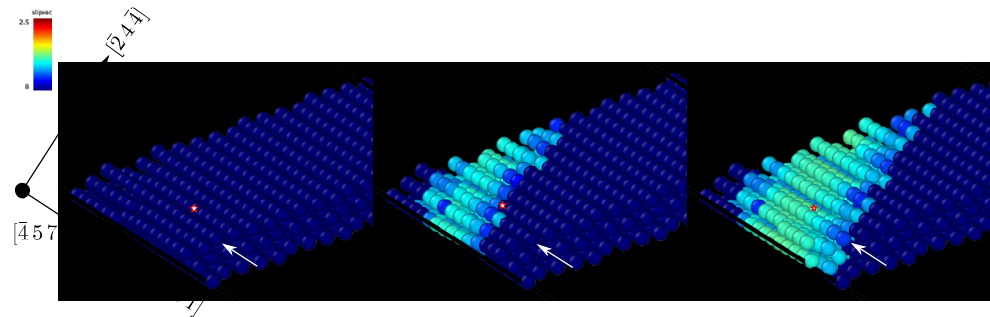
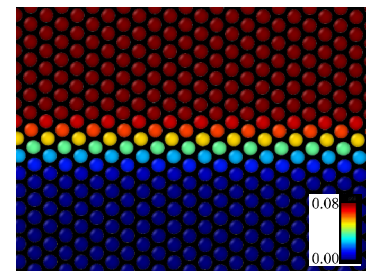
1. In-plane rotation about a fixed atom on a common (111) plane



3. Rotation between two misoriented (111) planes

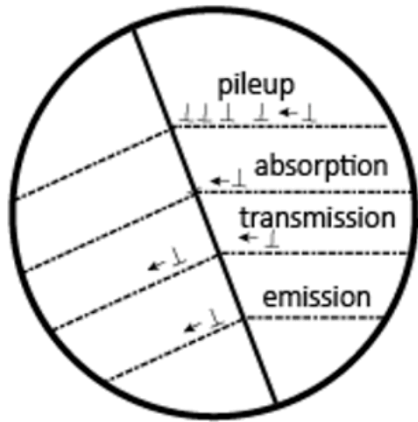


2. In-plane rotation without rotation about a common (111) plane



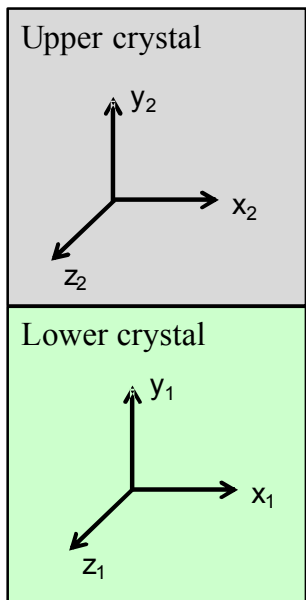
The mechanisms for anti-thermal boundary motion involve a coordinated shuffling or rotation about a common plane, typically (111). Because of the apparent coordinated motion, it bears similarity to a martensitic/military motion rather than a diffusive motion.

# An example (among many) of dislocation-GB interactions



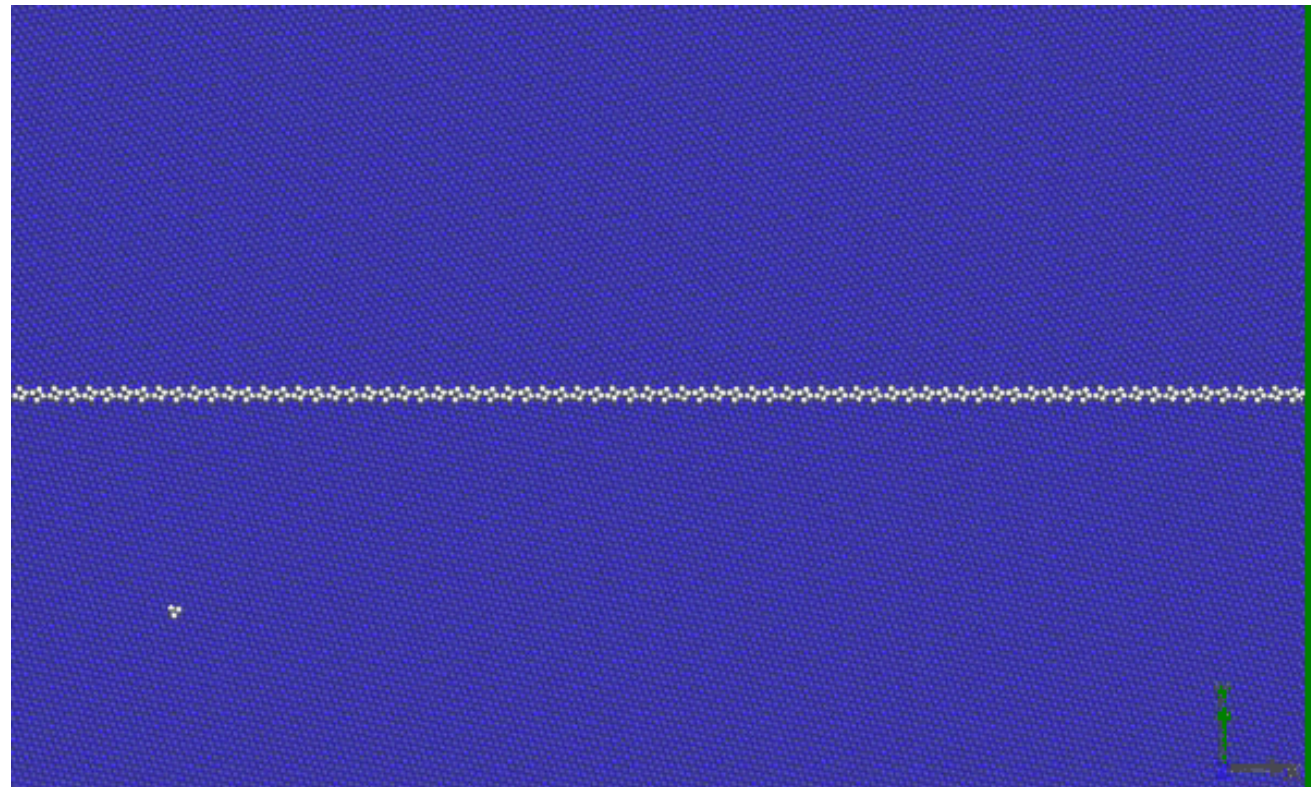
An example of dislocation **absorption**  
 + propagating GB structural changes  
 + emission

$\Sigma 7 \langle 111 \rangle \{1\ 4\ 5\}$  GB plane normal

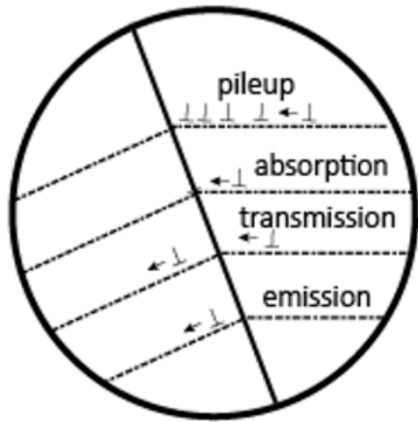


$$\begin{aligned} x_2 &= [2\ 1\ -3] \\ y_2 &= [-4\ 5\ -1] \\ z_2 &= [1\ 1\ 1] \end{aligned}$$

$$\begin{aligned} x_1 &= [3\ -1\ -2] \\ y_1 &= [-1\ 5\ -4] \\ z_1 &= [1\ 1\ 1] \end{aligned}$$

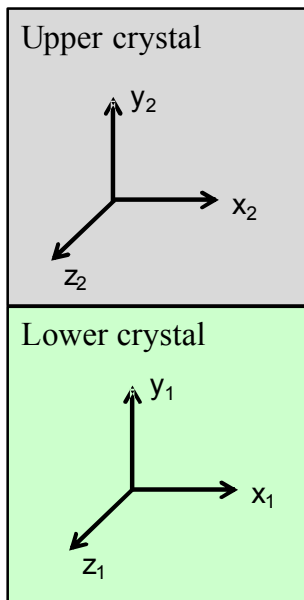


# Some rare boundaries are triggered by dislocations



An example of dislocation absorption,  
+ defect formation  
+ **GB migration**

$\Sigma 7 \langle 111 \rangle \{1 2 3\}$  GB plane normal



$$x_2 = [5 \ -1 \ -4]$$

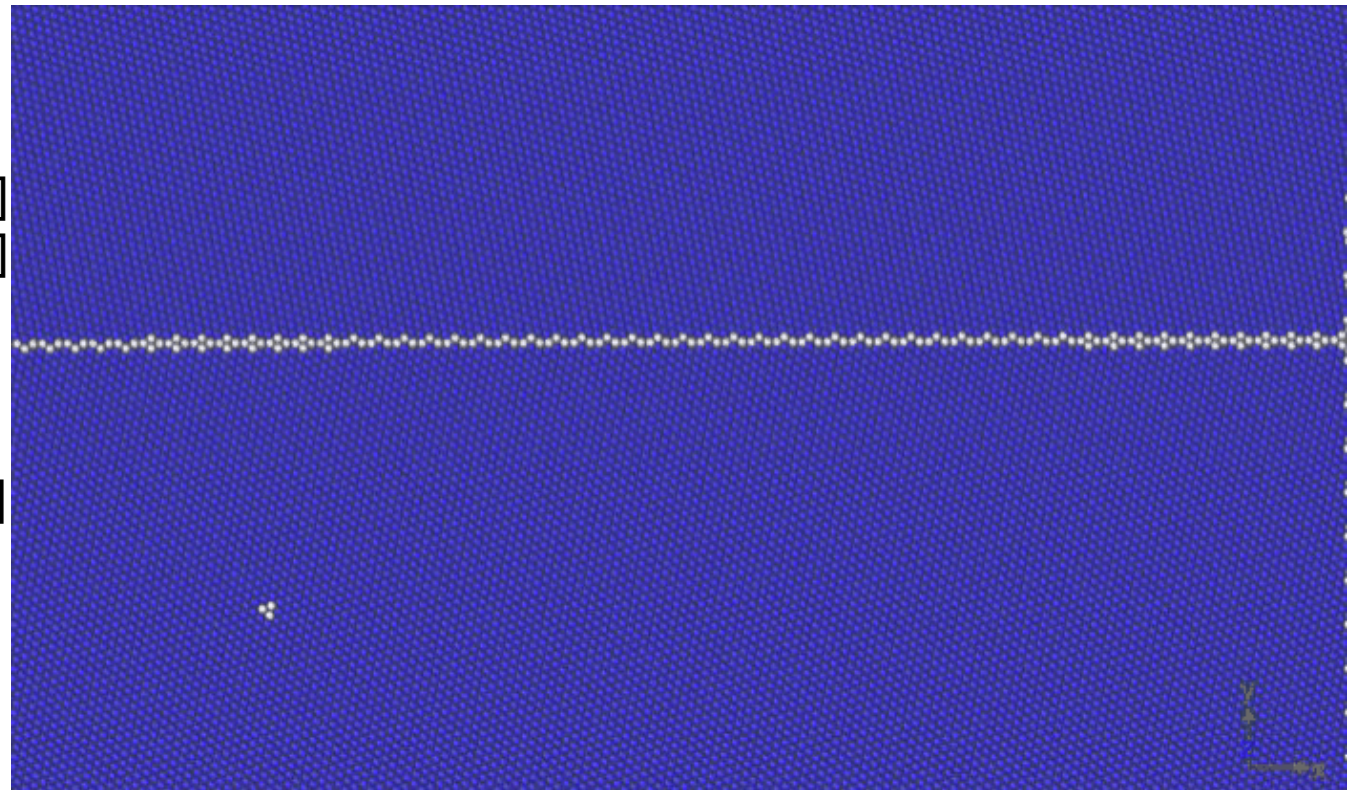
$$y_2 = [-1 \ 3 \ -2]$$

$$z_2 = [1 \ 1 \ 1]$$

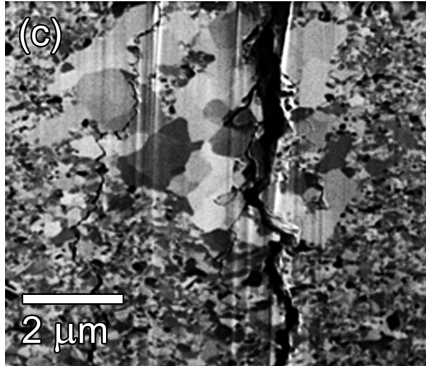
$$x_1 = [5 \ -4 \ -1]$$

$$y_1 = [1 \ 2 \ -3]$$

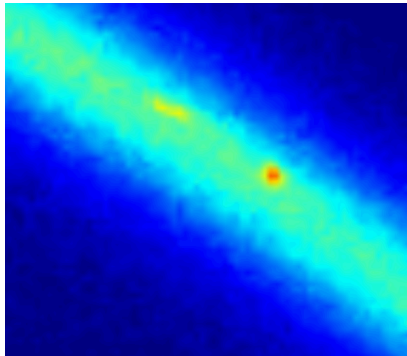
$$z_1 = [1 \ 1 \ 1]$$



## Recap...

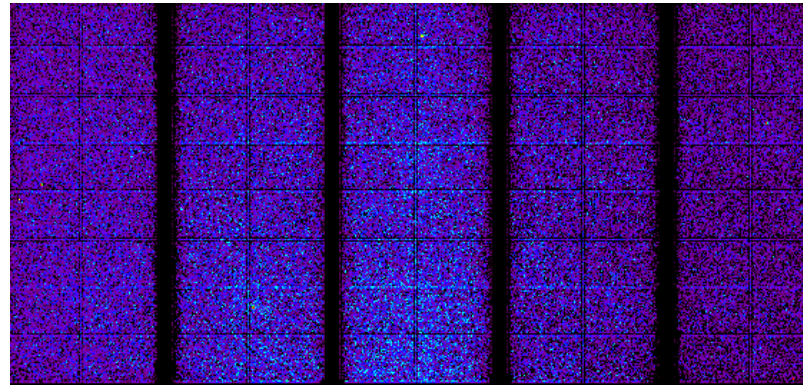


*In nanocrystalline Ni alloys (and presumably others), crack initiation involves abnormal grain growth!*

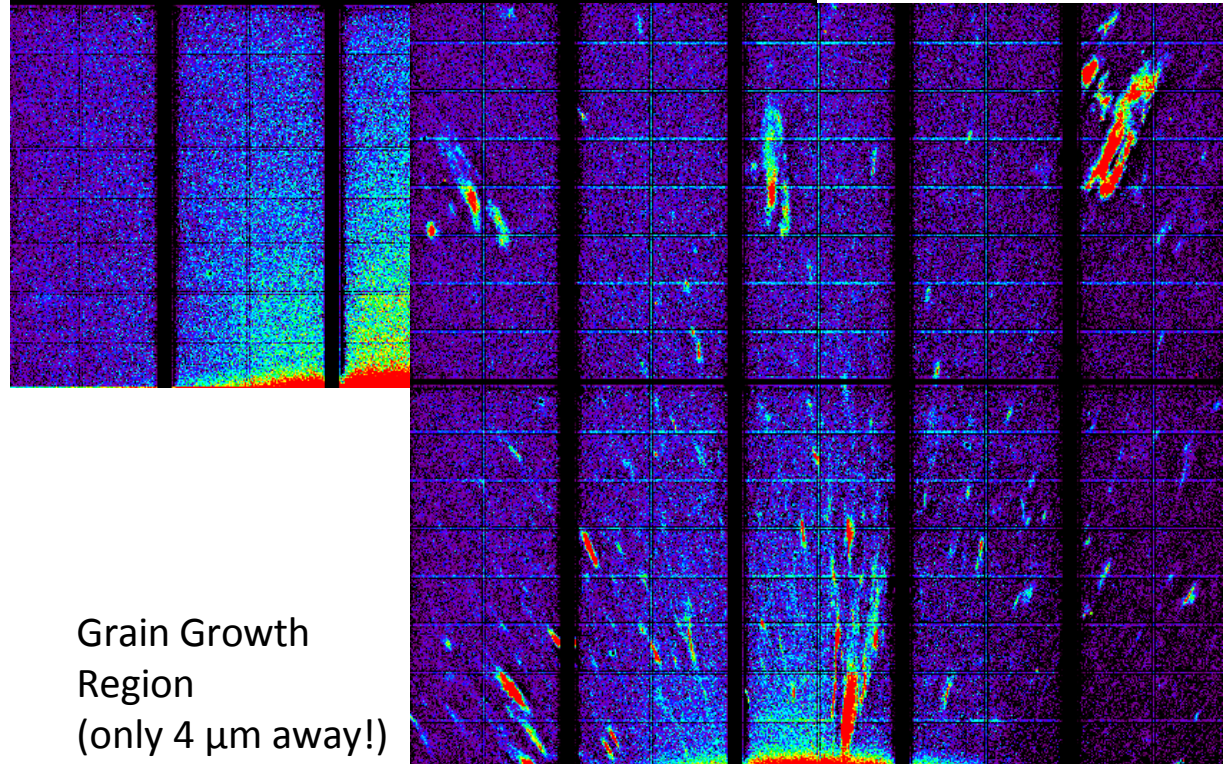


*Synchrotron XRD allows us to detect the grain growth precursor "embryo" as it evolves prior to crack initiation.*

# Polychromatic Microdiffraction (Advanced Light Source)

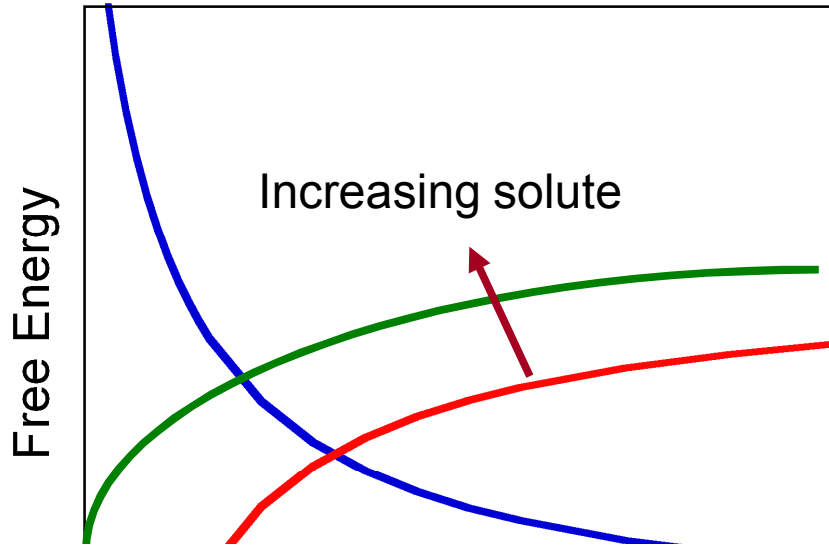


Nanocrystalline region (no grain growth)



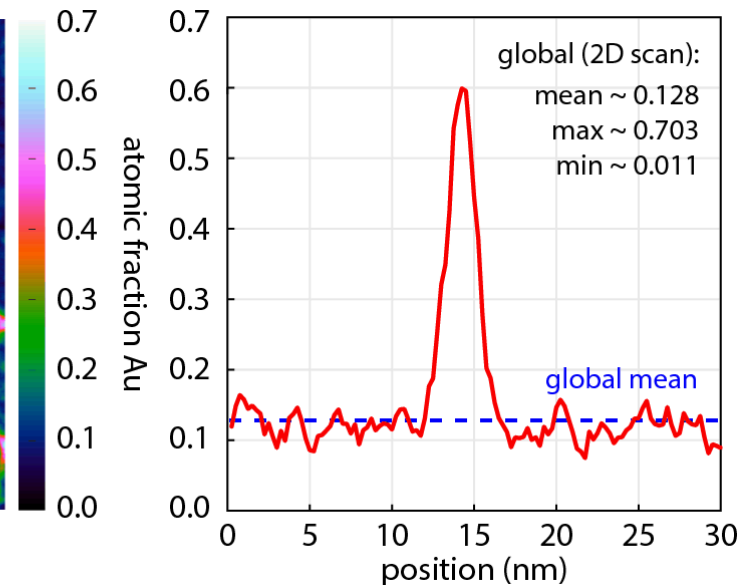
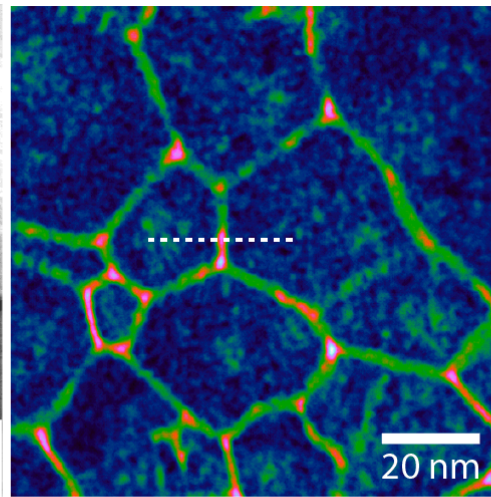
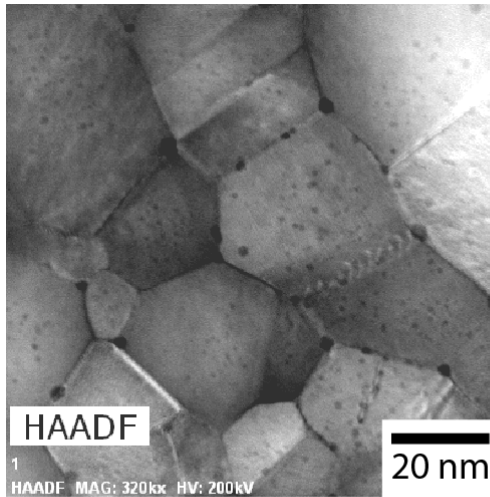
Grain Growth Region  
(only 4  $\mu\text{m}$  away!)

One last thought: Can we stabilize the grain boundaries thermodynamically???



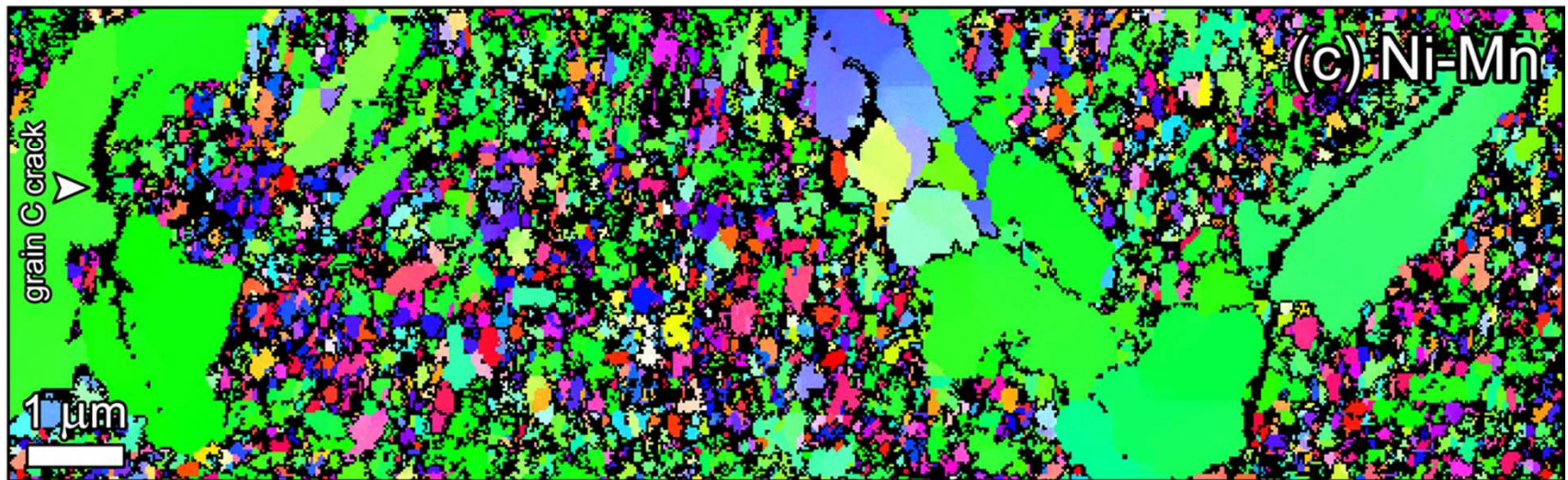
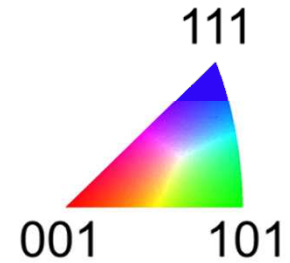
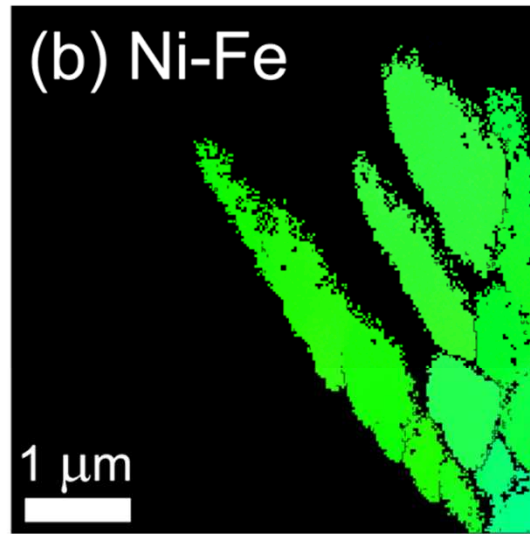
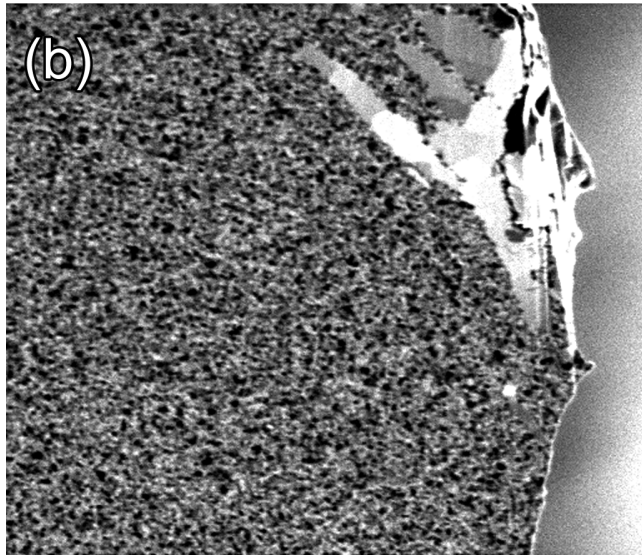
$$dG = \gamma dA$$

$$dG = \left[ \gamma - \frac{N_{\beta}}{A} \Delta G_{seg} \right] dA$$



B. thick film (2  $\mu\text{m}$ ) on Si - planar view

# Crystal orientation of the abnormally large grains



# The crystalline orientation of the large grains maximizes slip deformation

