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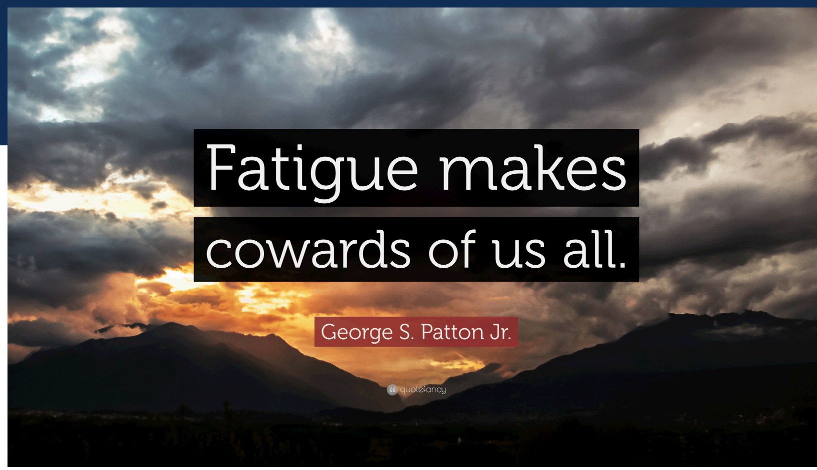

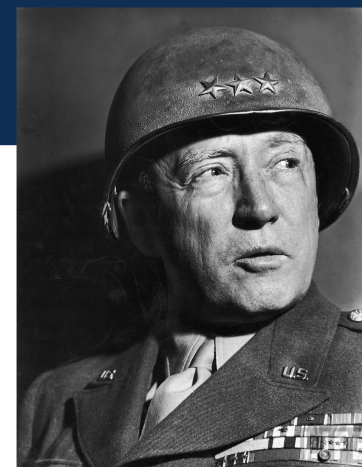

Nanocrystalline Alloys: Impervious to Fatigue Failure?

Brad L. Boyce

Sandia National Laboratories, Albuquerque, NM

U.S. DEPARTMENT OF ENERGY
NNSA National Nuclear Security Administration

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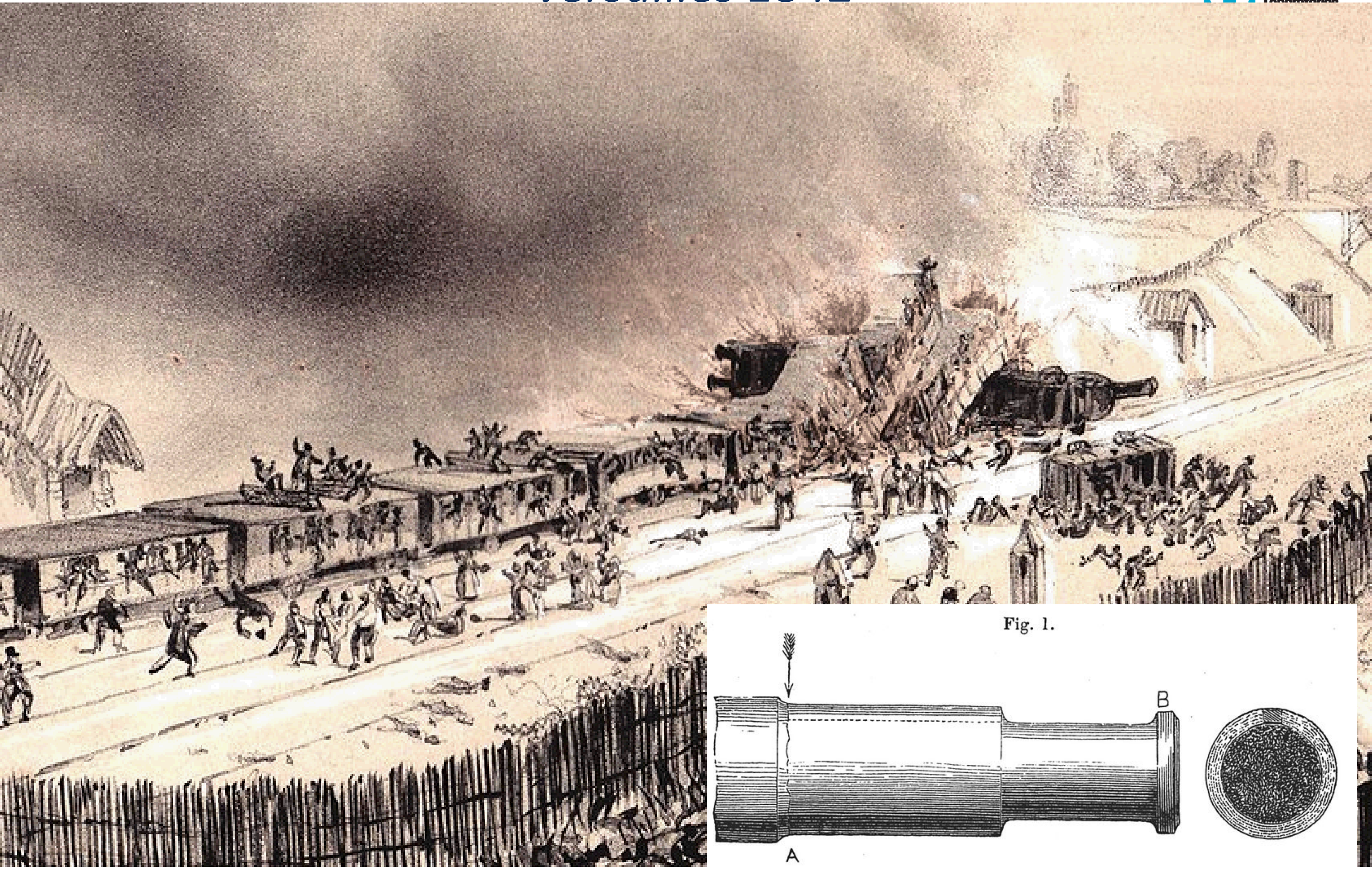
Versailles 1842



by Joseph Glynn, 1843

Aloha Airlines, 1988

4-28-1988 After 89,090 flight cycles on a 737-200, metal fatigue lets the top go in flight.

Fatigue in Aviation

What is fatigue?

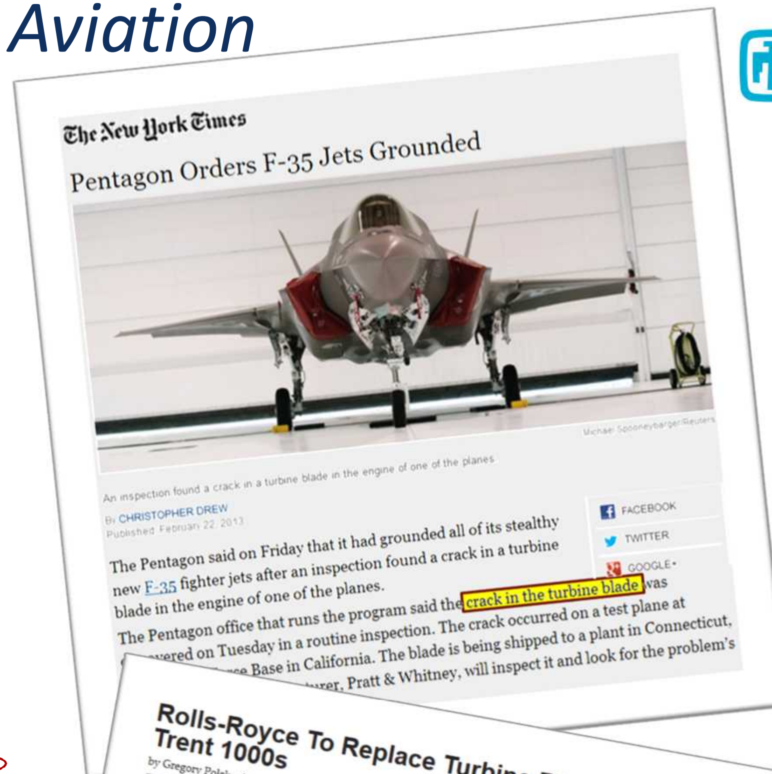
"…Changes in properties which can occur in a metallic material due to the repeated application of stresses or strains, although usually this term applies specially to those changes which lead to cracking or failure…"

General Principles for Fatigue Testing of Metals, International Organization for Standardization, Geneva, 1964.

Percentage of Failures in Aircraft

Corrosion	16
Fatigue	55
Brittle fracture	-
Overload	14
High temperature corrosion	2
SCC/Corrosion fatigue/HE	7
Creep	-
Wear/abrasion/erosion	6

[SJ Findlay and ND Harrison, "Why Aircraft Fail", *Materials Today*, 2002]



The New York Times

Pentagon Orders F-35 Jets Grounded

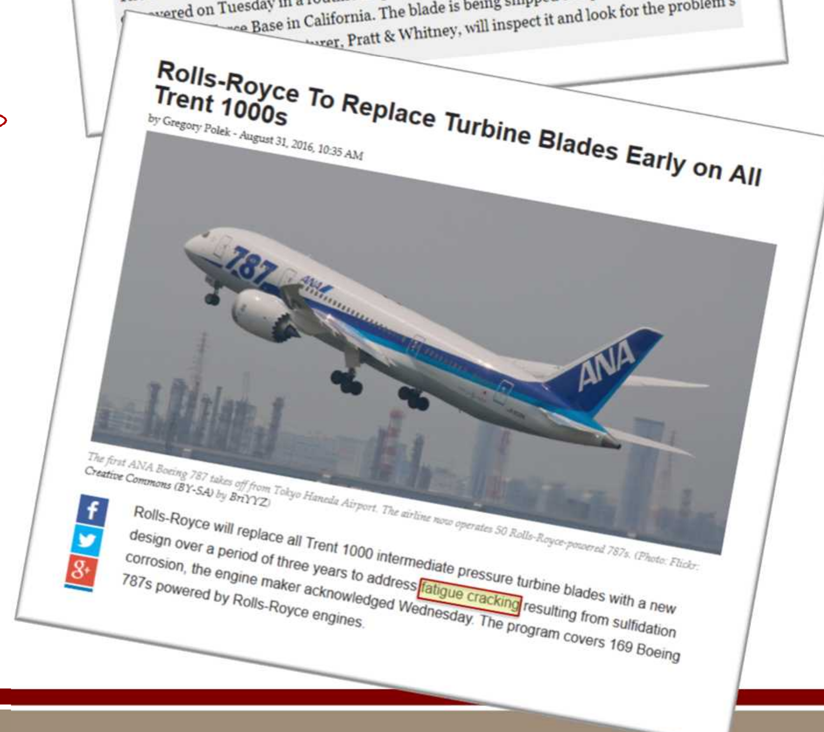
An inspection found a crack in a turbine blade in the engine of one of the planes

By CHRISTOPHER DREW

Published February 22, 2013

The Pentagon said on Friday that it had grounded all of its stealthy new F-35 fighter jets after an inspection found a crack in a turbine blade in the engine of one of the planes.

The Pentagon office that runs the program said the crack in the turbine blade was …vered on Tuesday in a routine inspection. The crack occurred on a test plane at …Base in California. The blade is being shipped to a plant in Connecticut, …rer, Pratt & Whitney, will inspect it and look for the problem's



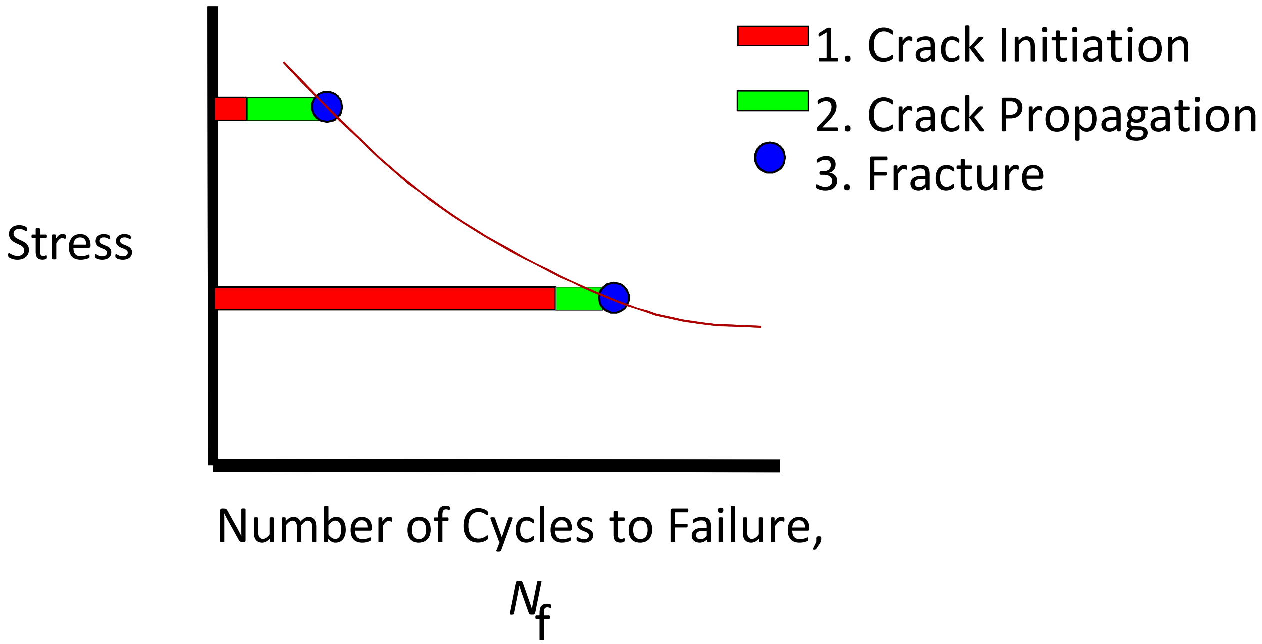
Rolls-Royce To Replace Turbine Blades Early on All Trent 1000s

by Gregory Polek - August 31, 2016, 10:35 AM

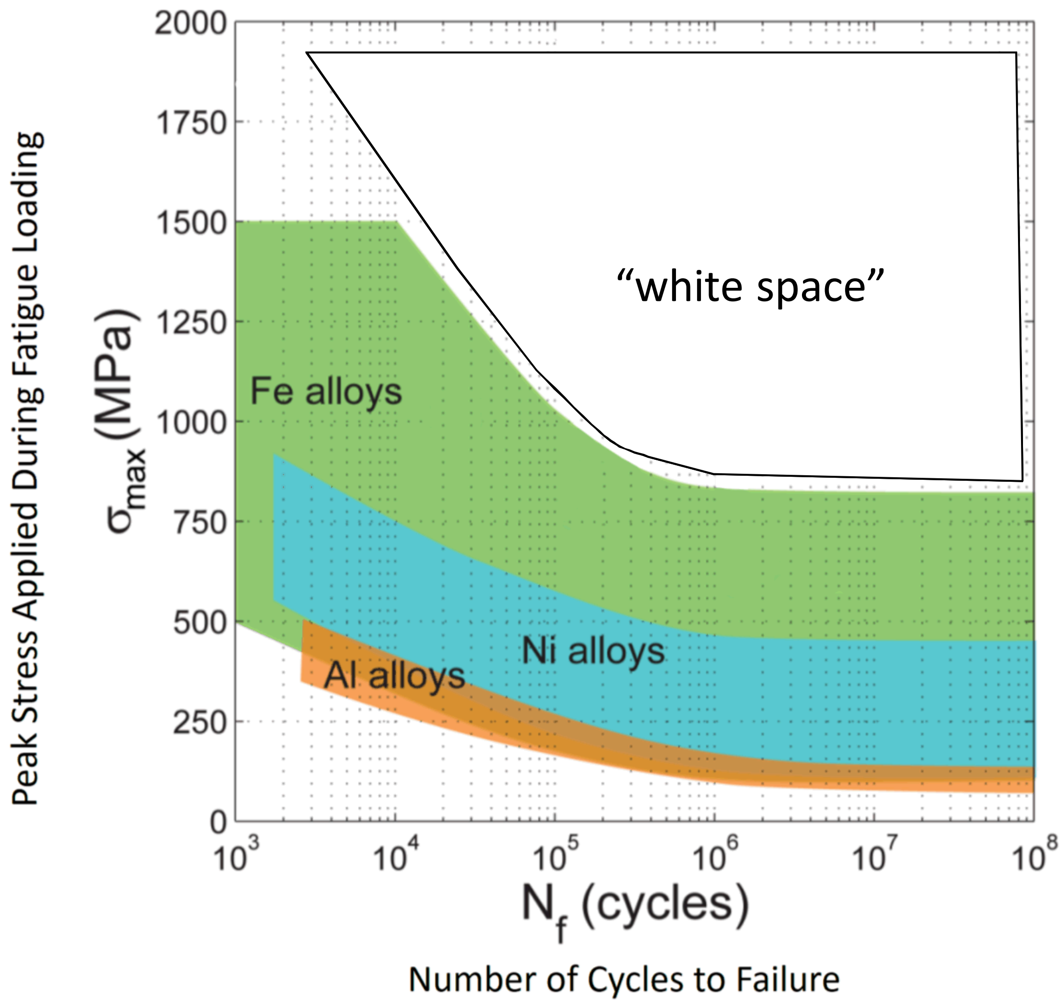
The first ANA Boeing 787 takes off from Tokyo Haneda Airport. The airline now operates 50 Rolls-Royce-powered 787s. (Photo: Flickr: Creative Commons (BY-SA) by BriYYZ)

Rolls-Royce will replace all Trent 1000 intermediate pressure turbine blades with a new design over a period of three years to address fatigue cracking resulting from sulfidation corrosion, the engine maker acknowledged Wednesday. The program covers 169 Boeing 787s powered by Rolls-Royce engines.

Background Concepts: Fatigue Life

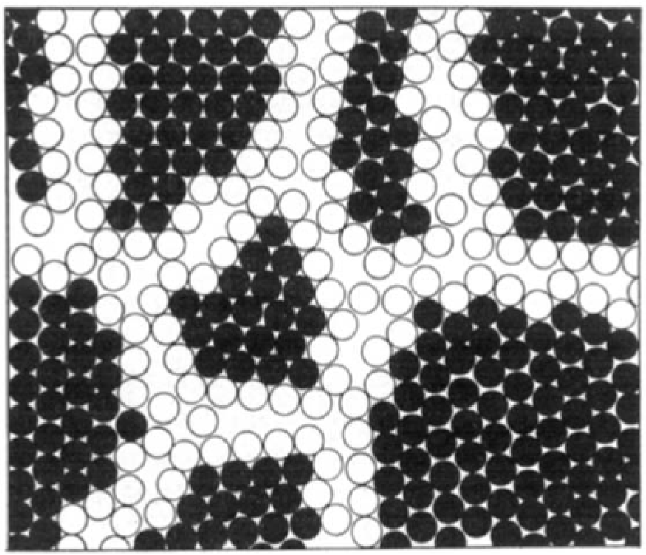


Fatigue Life in Engineering Alloys



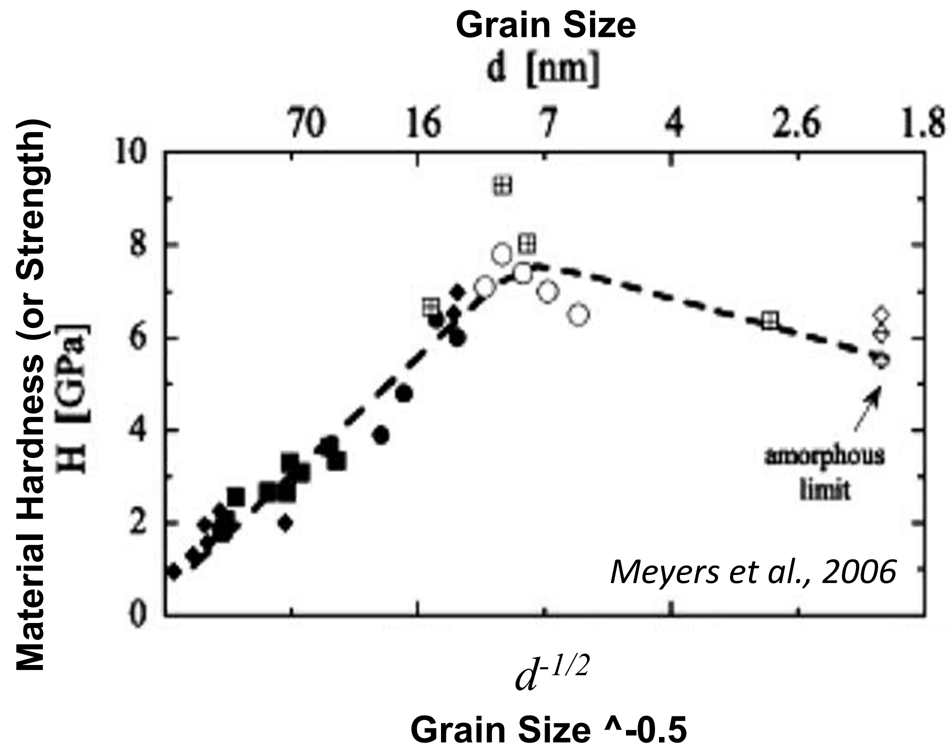
Nanocrystalline metals have excellent strength…

1989
Gleiter
Nanocrystalline
Materials



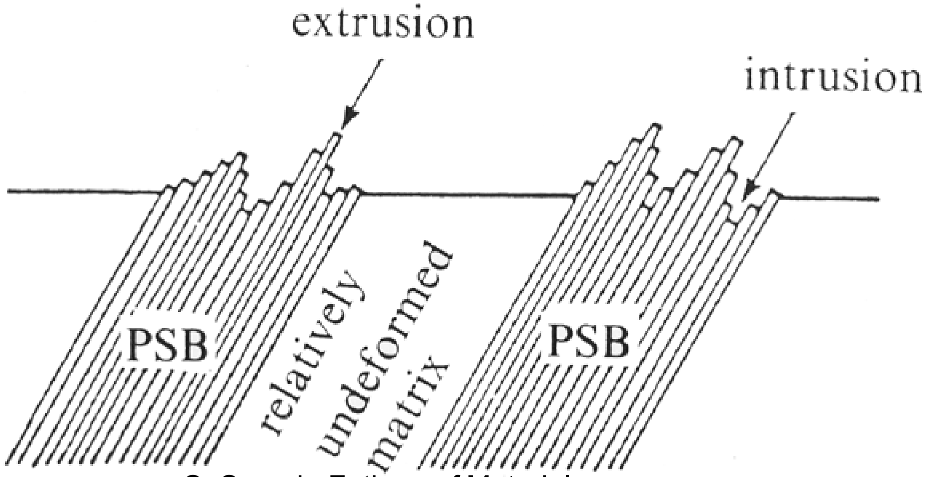
>4000 citations

2003
Kumar,
VanSwygenhoven
Suresh
Hall-Petch Breakdown

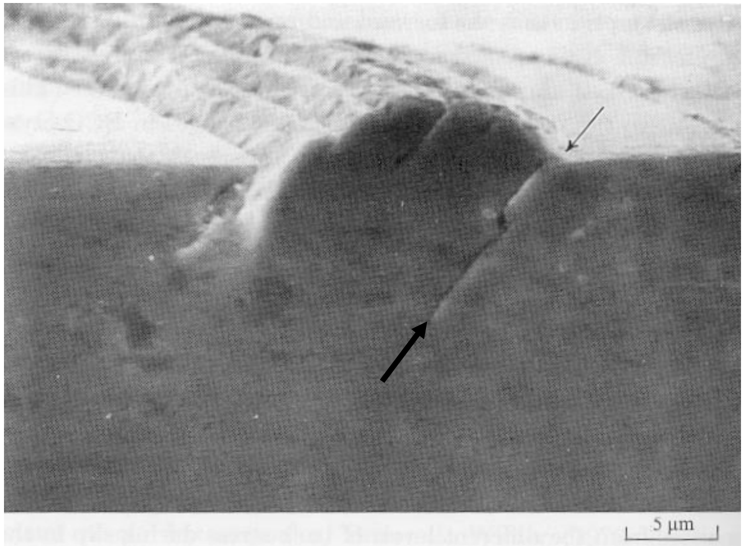


…what about their fatigue resistance???

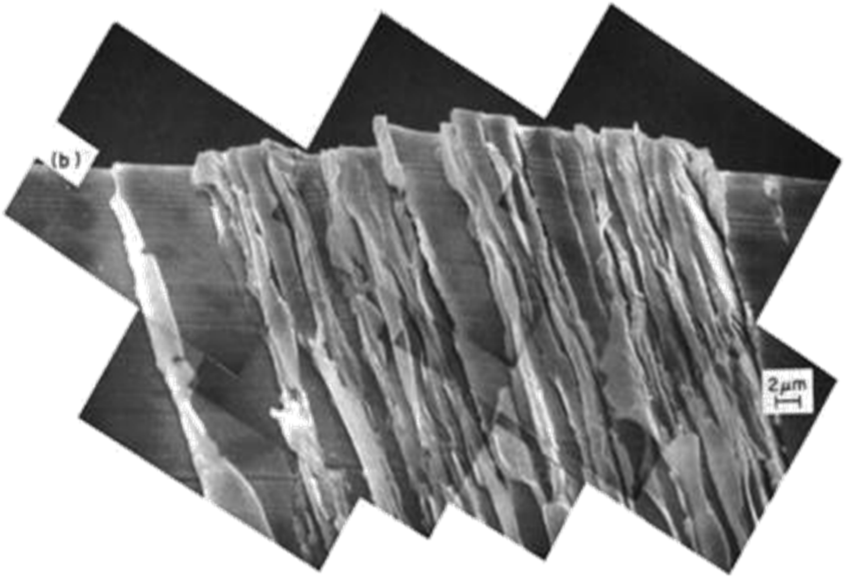
Is the fatigue mechanism suppressed in nanocrystalline metals?



S. Suresh, Fatigue of Materials



Ma and Laird, 1989



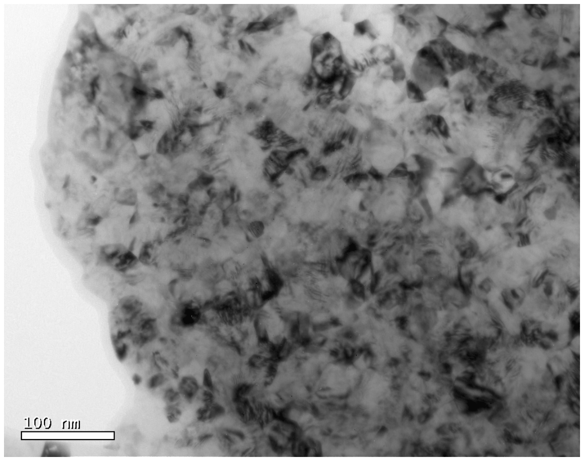
A billion nanocrystalline grains would fit inside a single microcrystalline grain of a traditional structural alloy.

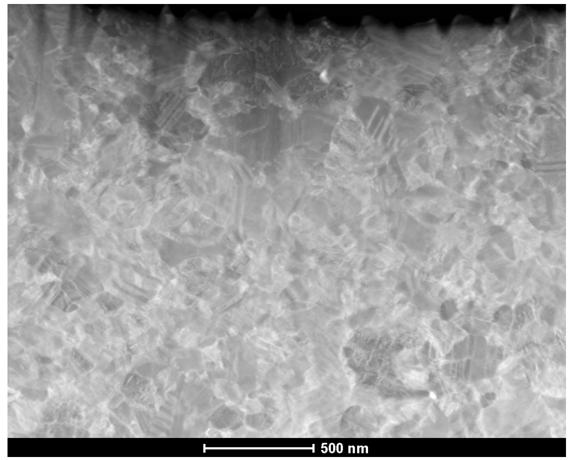
Does the fatigue mechanism change for such small grain sizes?

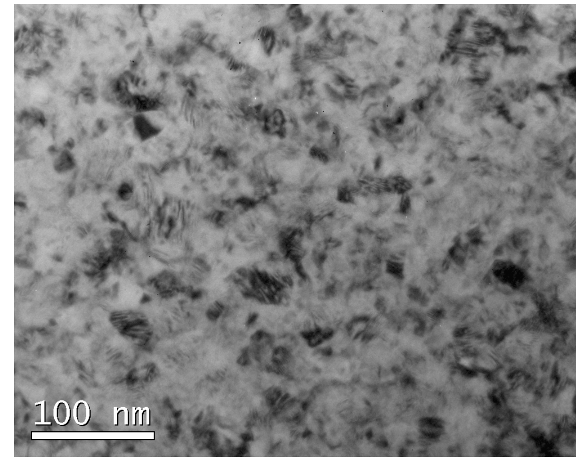
Three Nanocrystalline Ni-Based Alloys

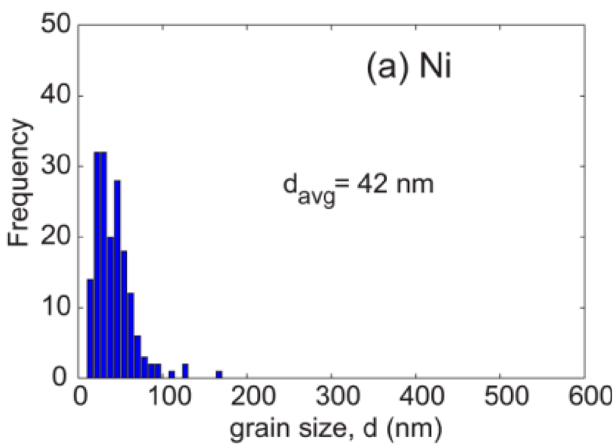
Sandia National Laboratories

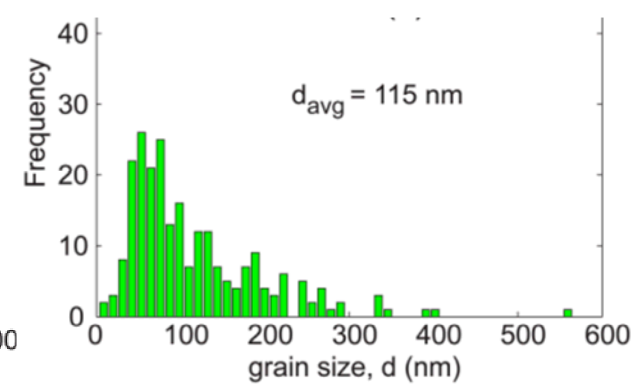
Ni (42 nm)	**Ni-0.5Mn (115 nm)**	**Ni-22Fe (25 nm)**

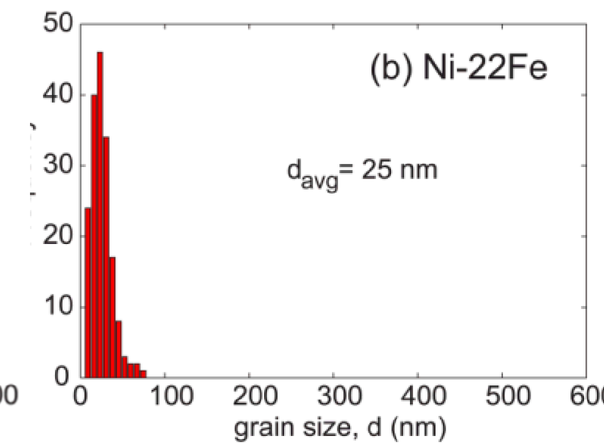




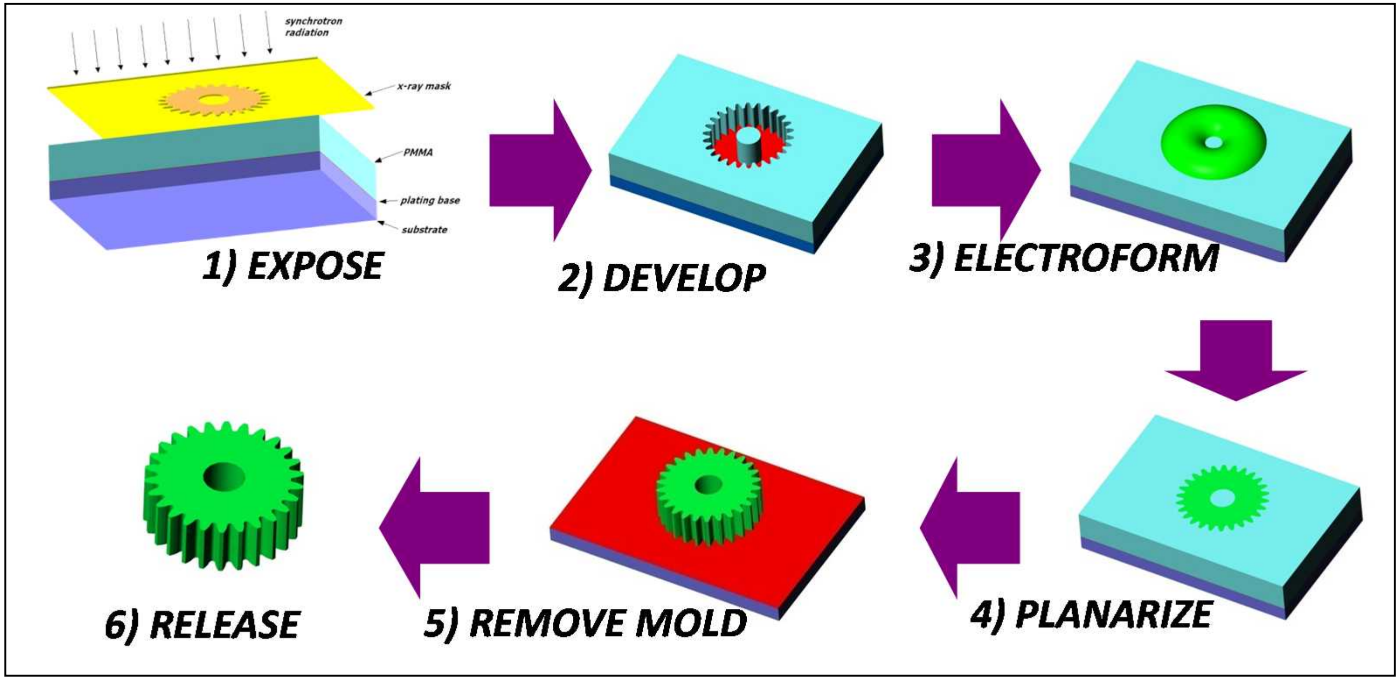




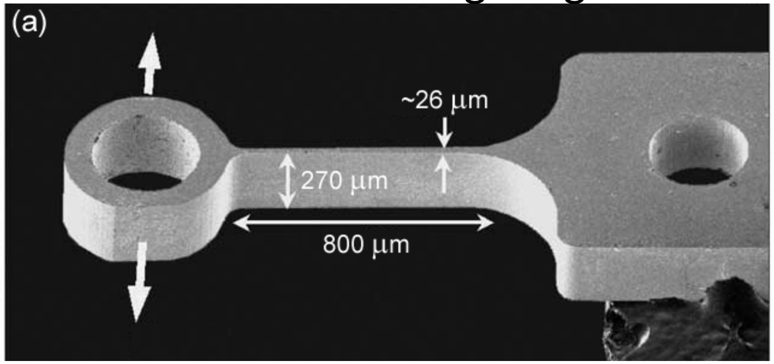




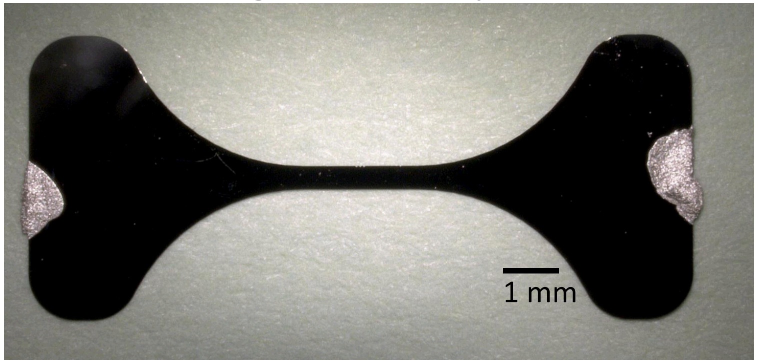
Electrodeposited nanocrystalline metal is lithographically patterned for fatigue testing



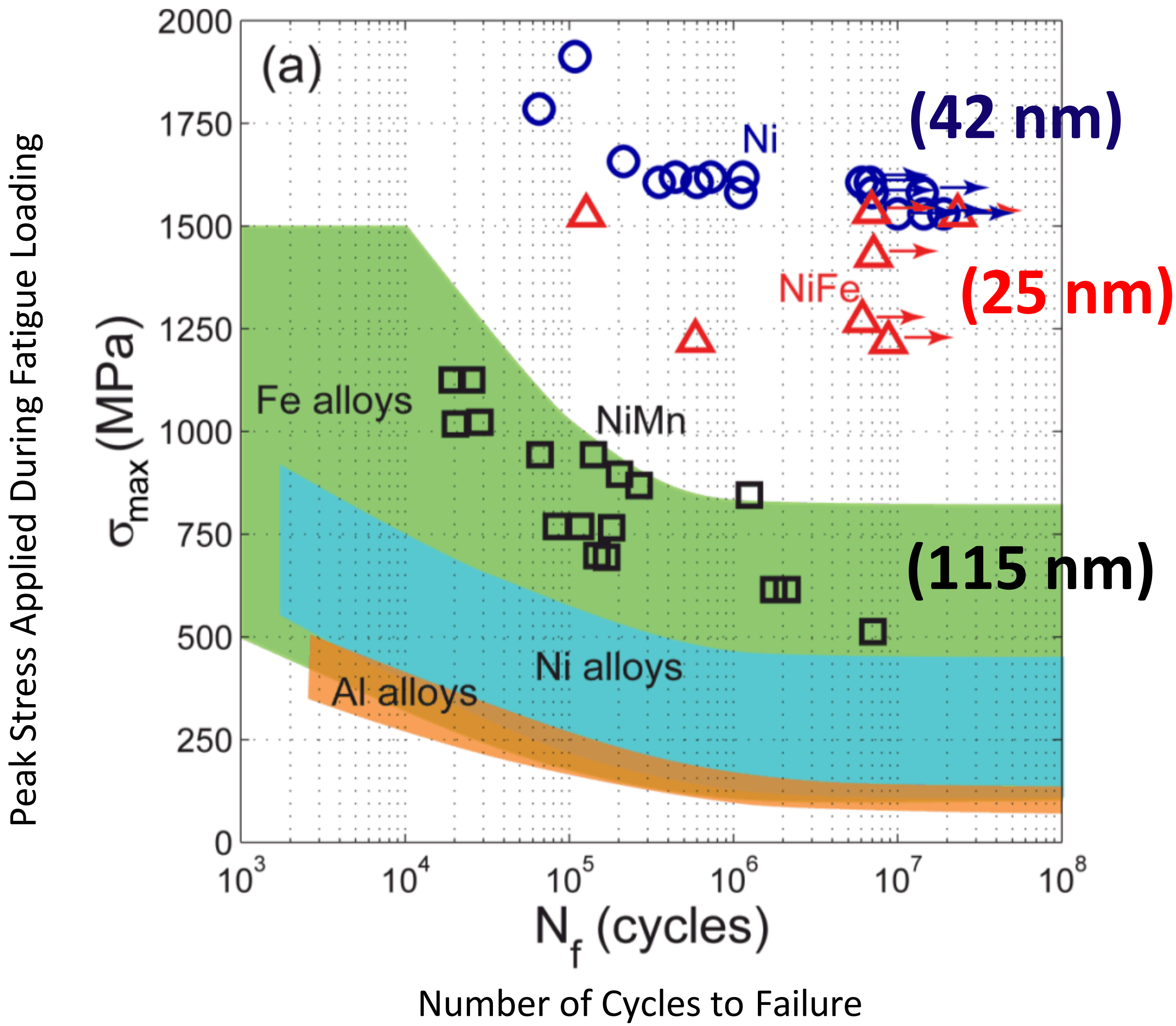
Cantilever beam bending fatigue bars



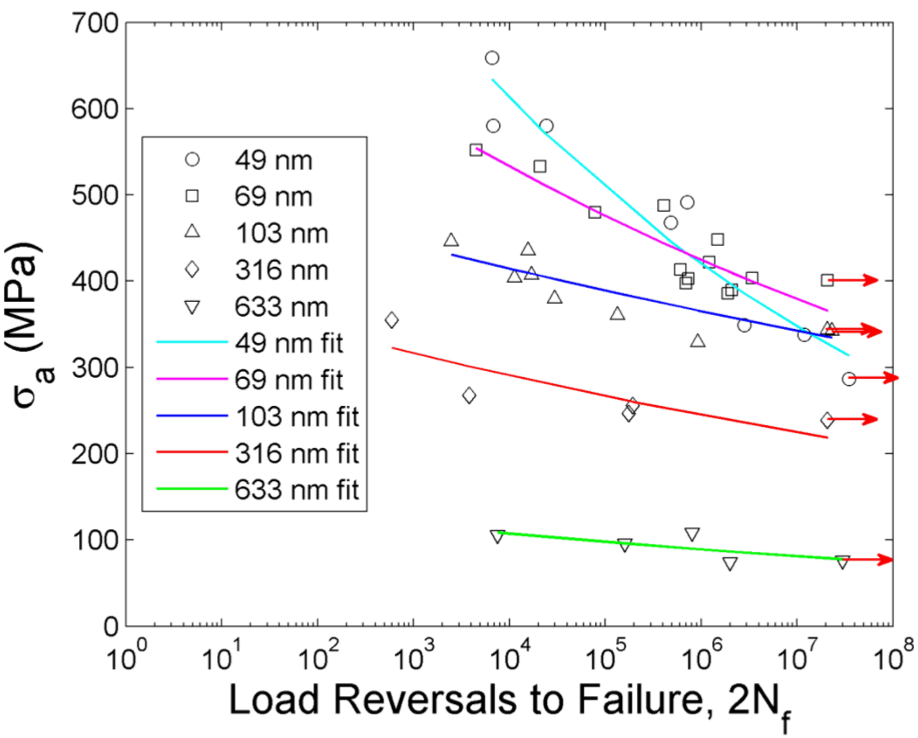
Tension fatigue bars (5 µm thick)

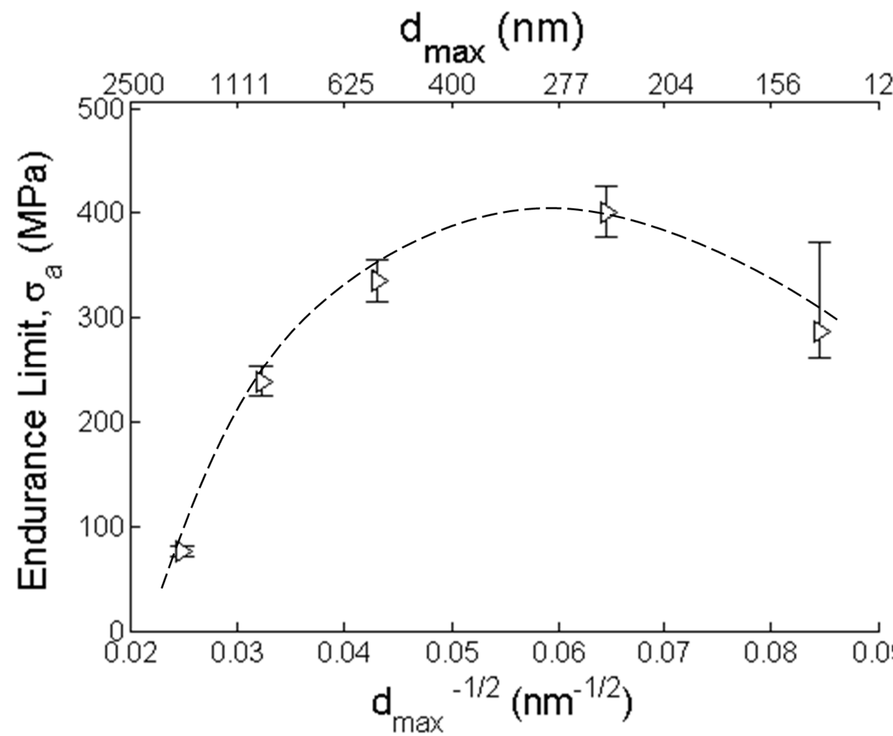


Superior Fatigue Resistance in Nanocrystalline Alloys



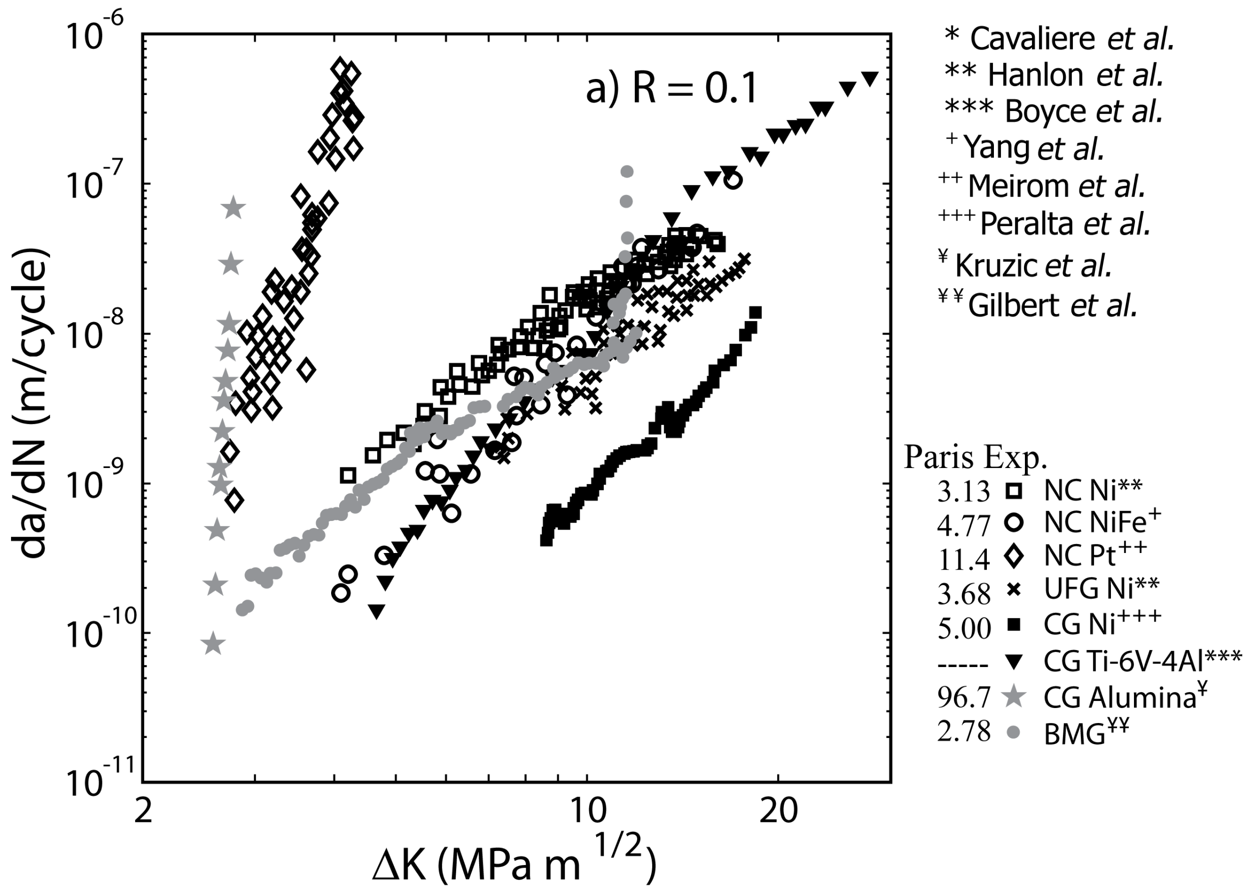
Endurance limit plateaus at average grain size ~70 nm! (max grain size ~400 nm)



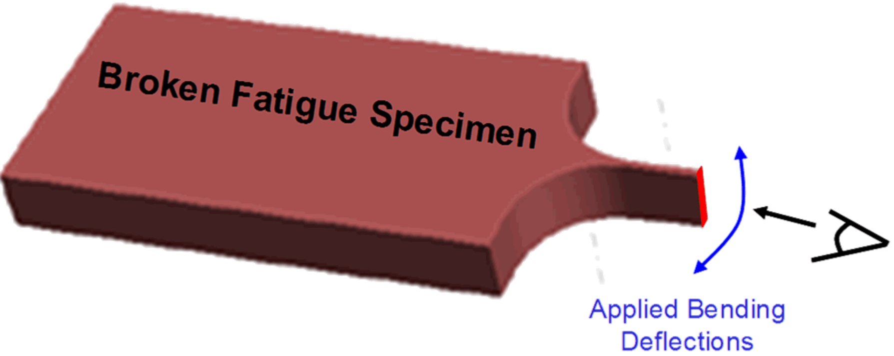


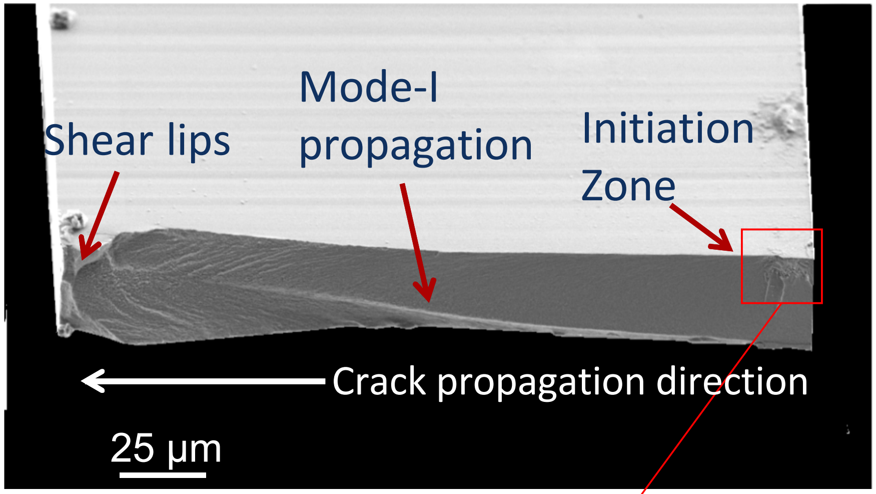
The impressive high-cycle fatigue resistance is likely due to suppressed crack initiation…

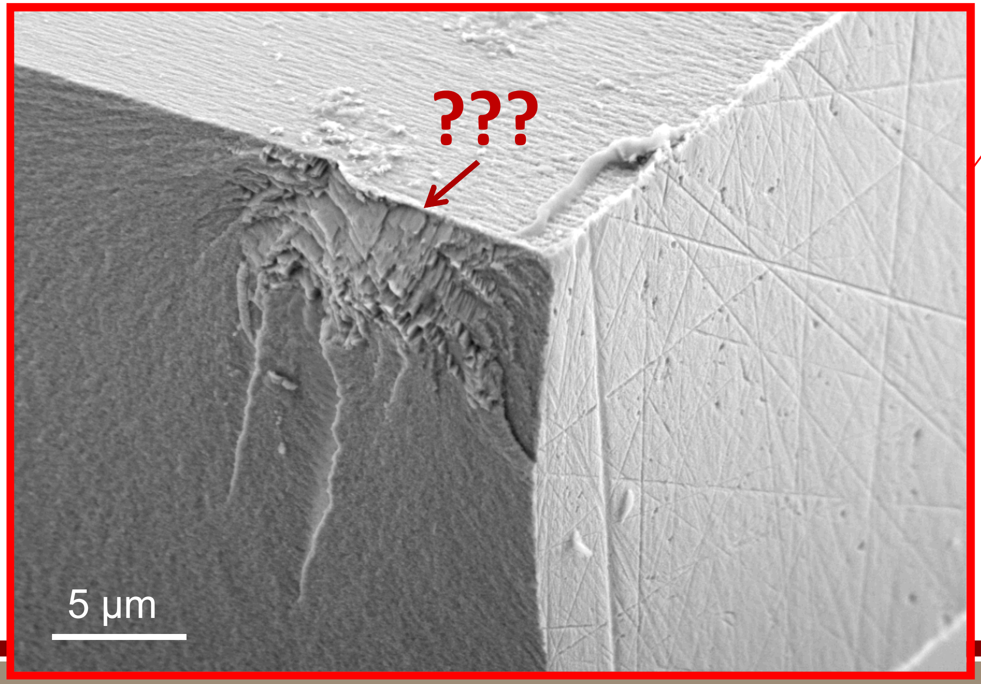
Crack growth resistance is worse than coarse-grained Ni, as expected

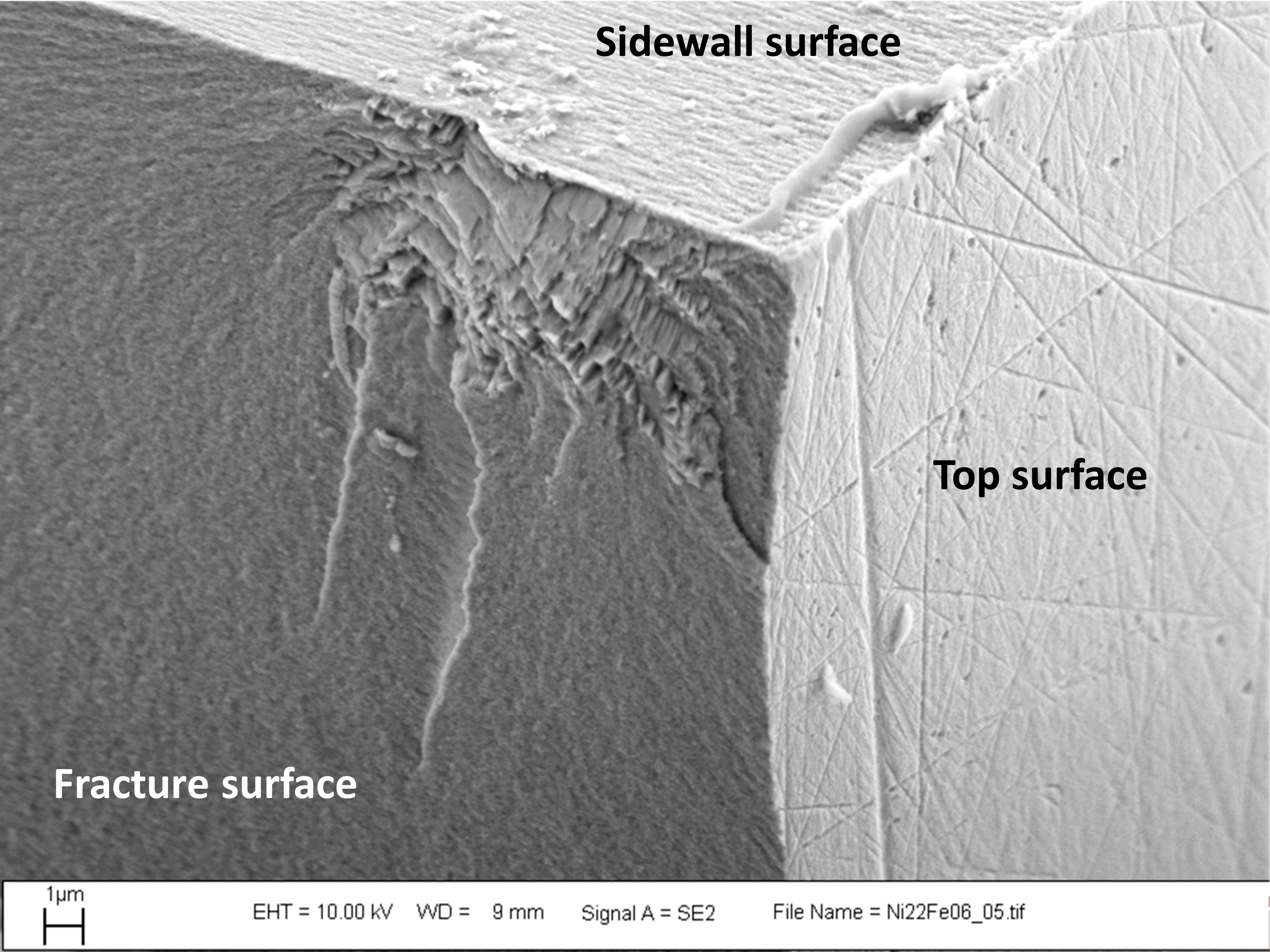


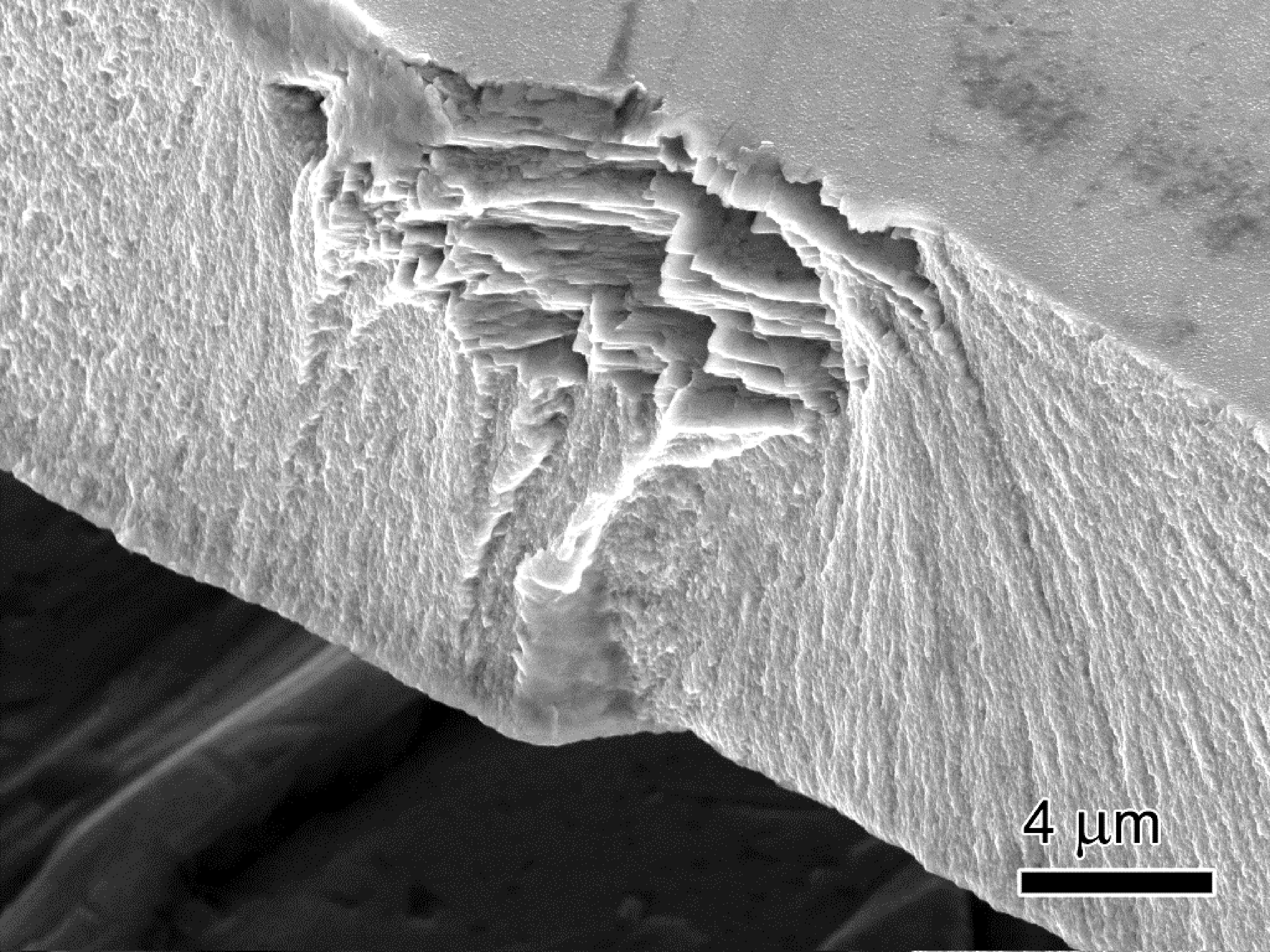
Unusual initiation zone in nanocrystalline Ni-Fe Alloy



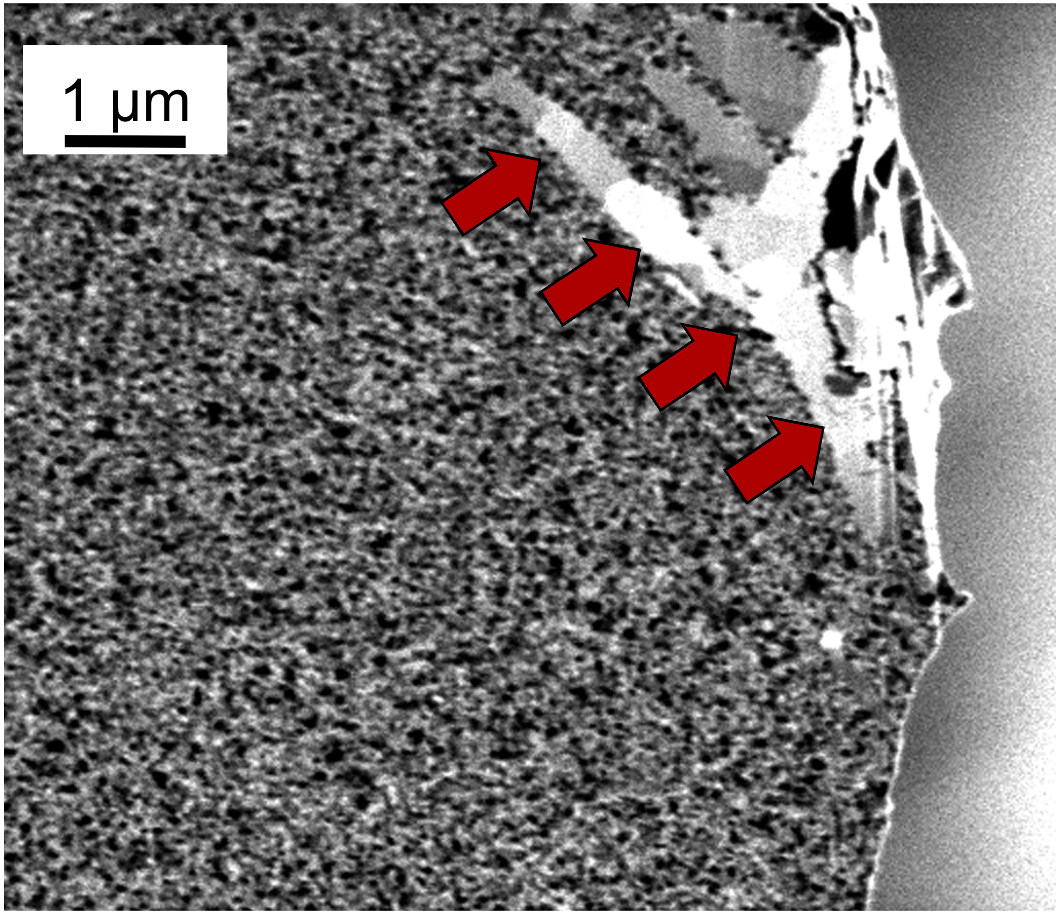





Sidewall surface
Top surface
Fracture surface
1µm
EHT = 10.00 kV
WD = 9 mm
Signal A = SE2
File Name = Ni22Fe06_05.tif


4 μm

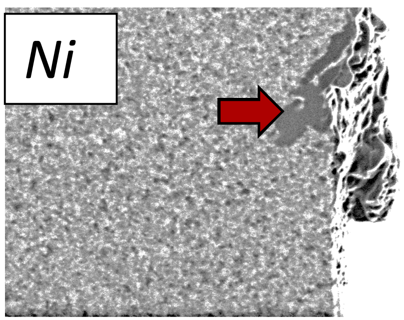
A cross-section through the 'blocky' zone reveals Very large subsurface grains

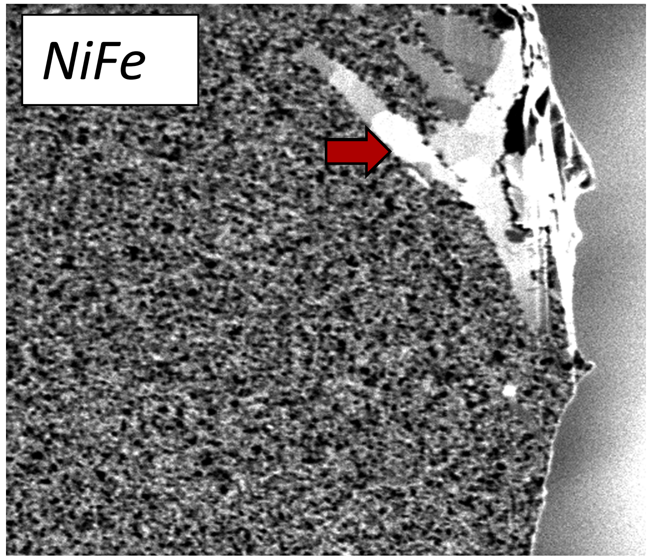


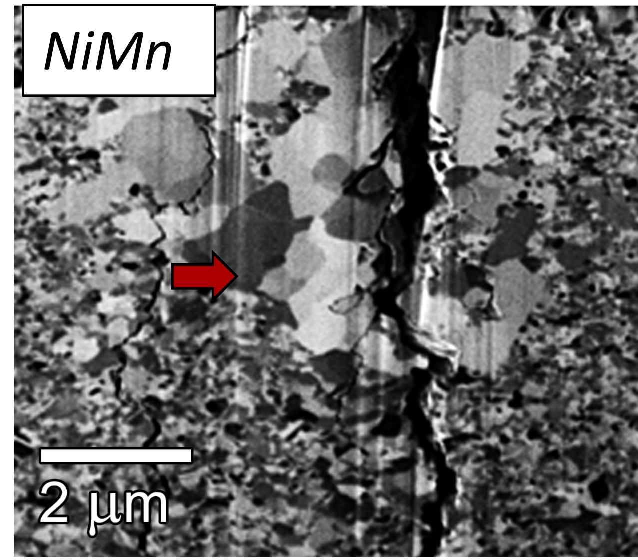
- Fatigue-induced coarsened zone *at room temperature!*
- Coarsened zone only forms locally (abnormal grain growth) in regions of maximum stress, prior to crack initiation

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

Sandia National Laboratories

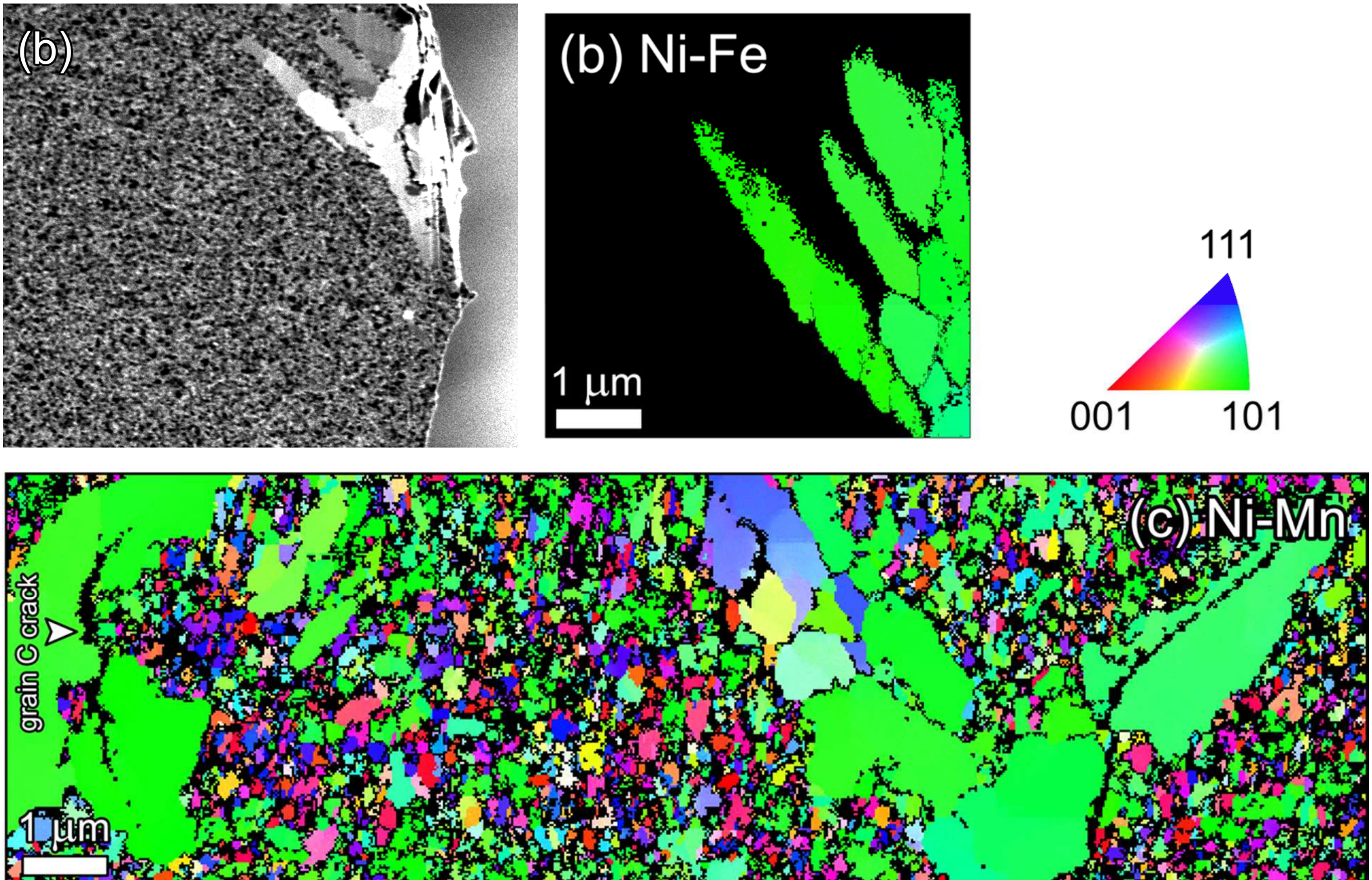






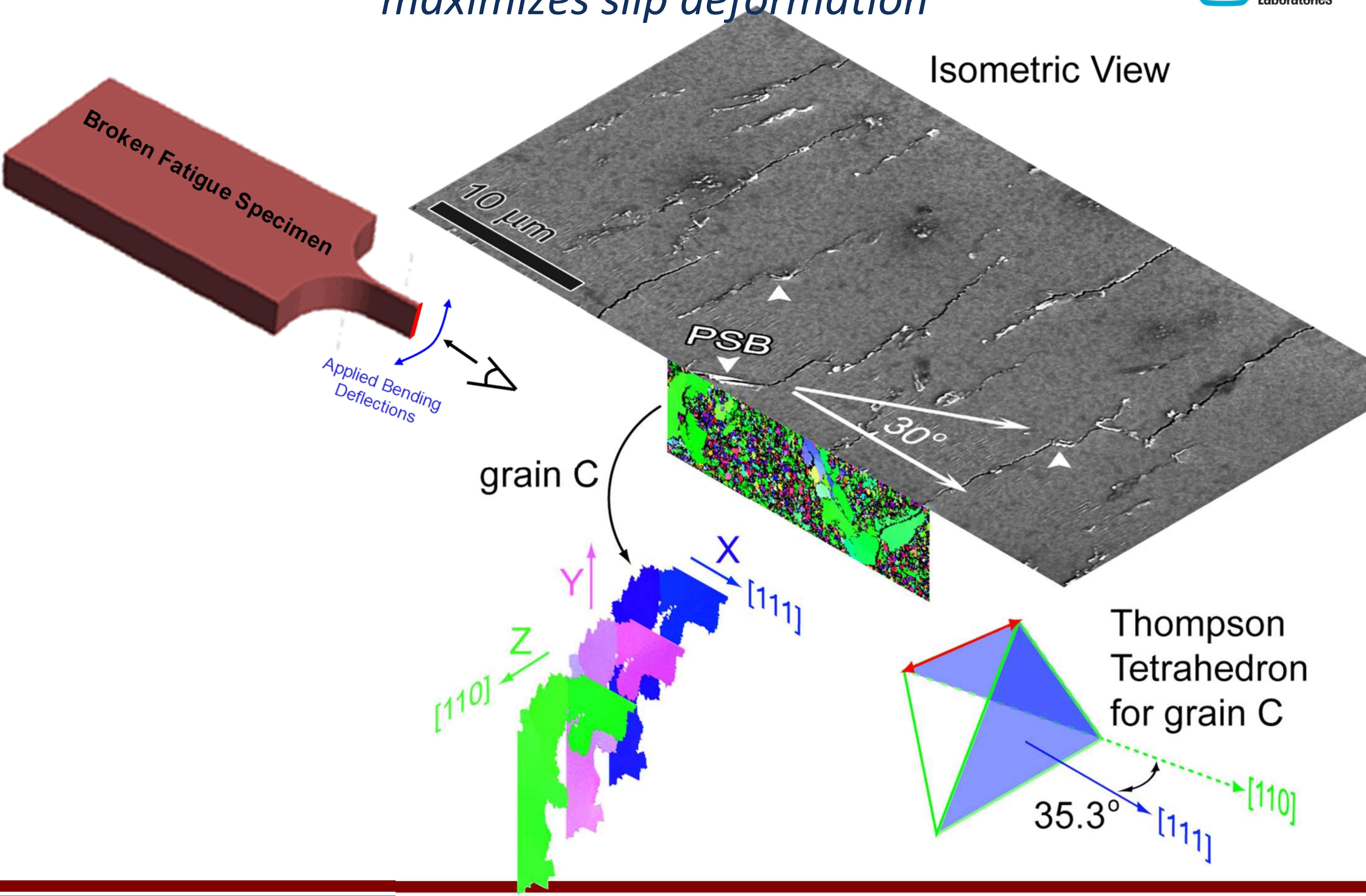
Boyce and Padilla, *Metall. And Mater. Trans. A,* 2011

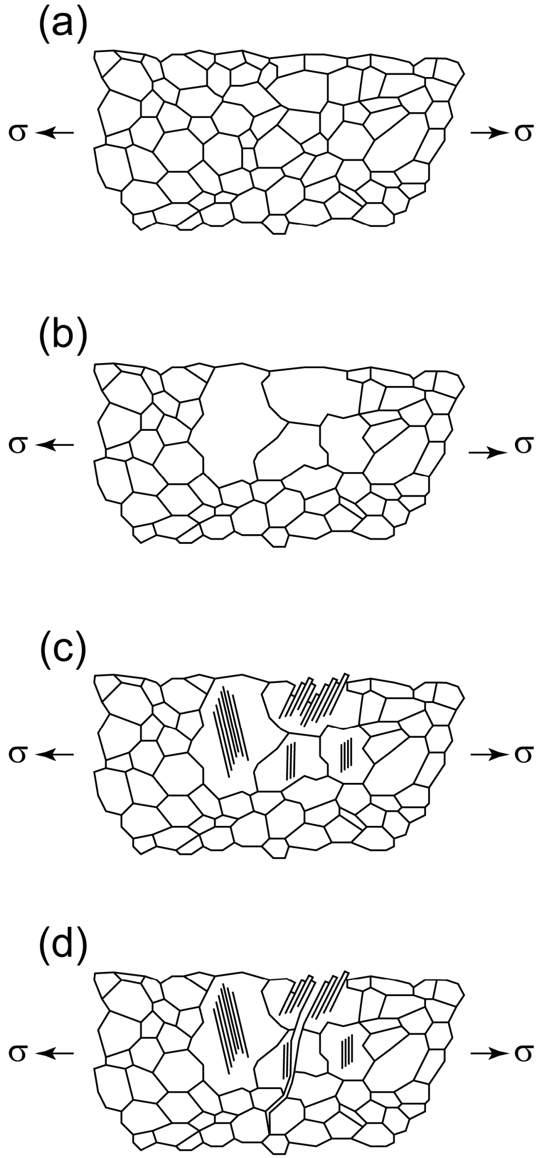
Crystal orientation of the abnormally large grains



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

The crystalline orientation of the large grains maximizes slip deformation





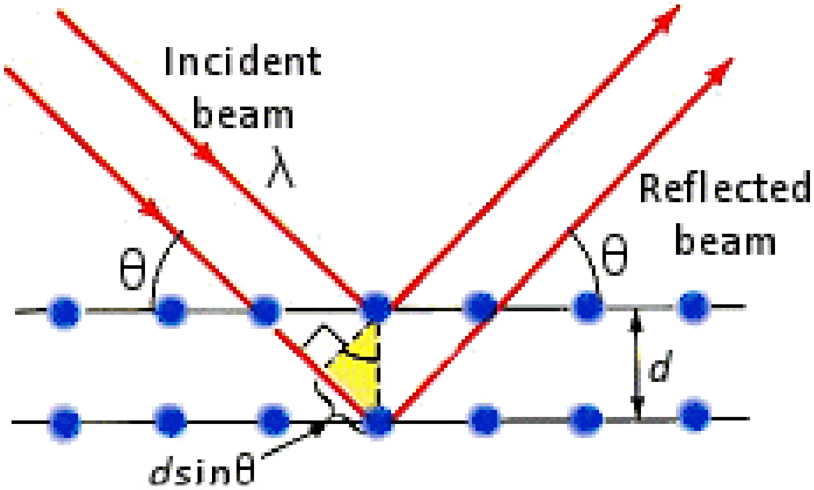
We posulate that in nanocrystalline metals (d_{gb} < ~70 nm), the fatigue process induces grain growth as a precursor to gross slip and eventual crack initiation

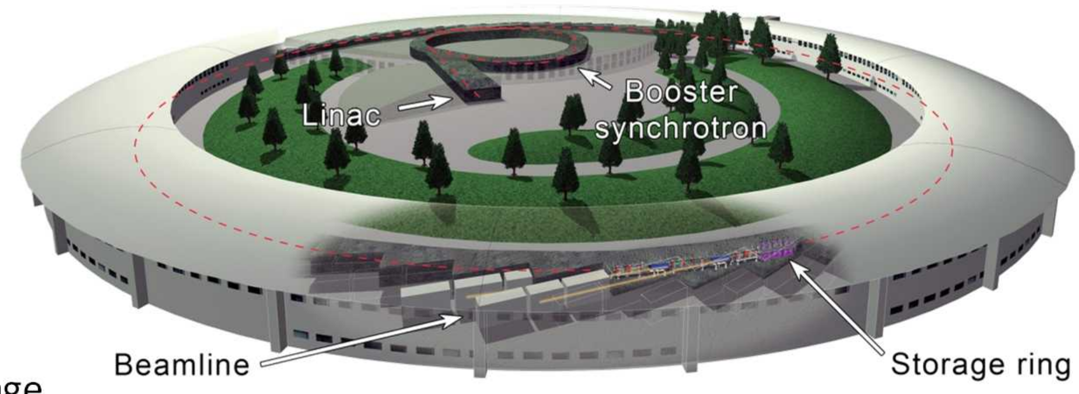
1. Were these few large grains formed during deposition, not fatigue loading?
2. Does the large grains facilitate cracking, or do the high stresses at the crack drive grain growth?
3. If the grains grow during fatigue, how many cycles does it take? [what are the kinetics of grain growth]

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

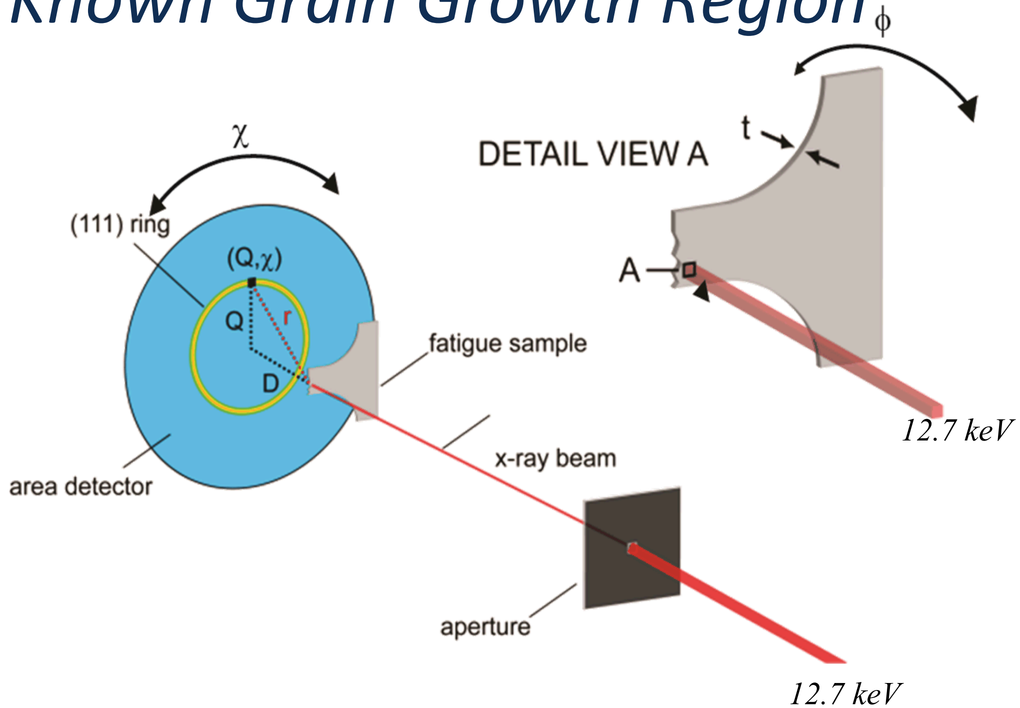
X-rays, Diffraction, & Synchrotrons

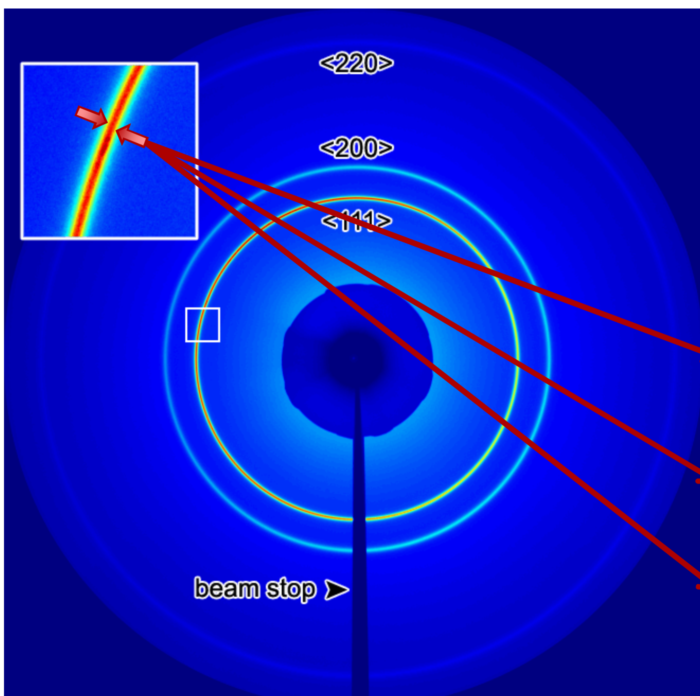
Wilhelm Konrad Roentgen's 1896 x-ray image of his wife's hand, Nobel Prize 1901





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





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

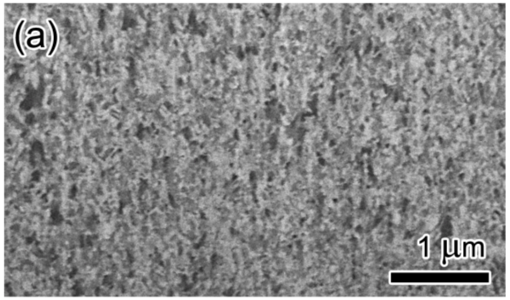
$$B\cos\theta = \frac{K\lambda}{d_{avg}} + \eta\sin\theta$$ *Williamson-Hall (1953)*

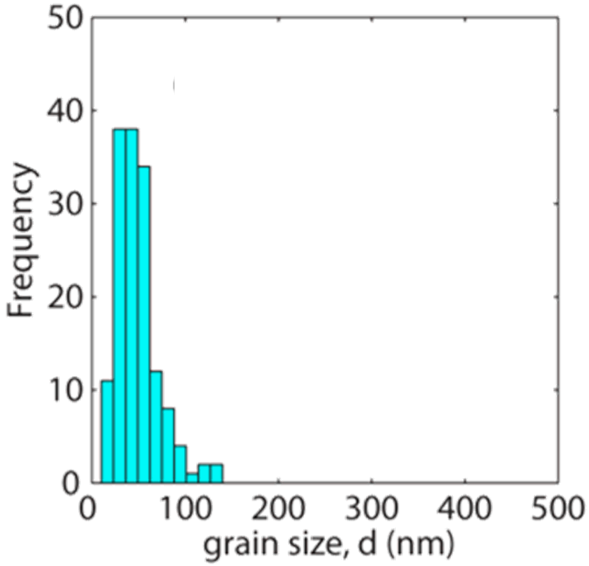
$$A_L = A_L^S A_L^D = A_L^S \exp[-2\pi^2 L^2 g^2 \langle \varepsilon_{g,L}^2 \rangle]$$ *Warren-Averbach (1959)*

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

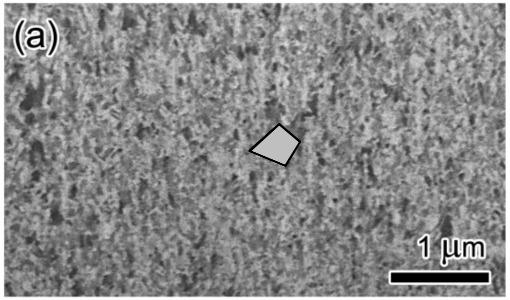
The crux of the problem: needle-in-a-haystack: the onset of abnormal grain growth has an imperceptible effect on the average grain size

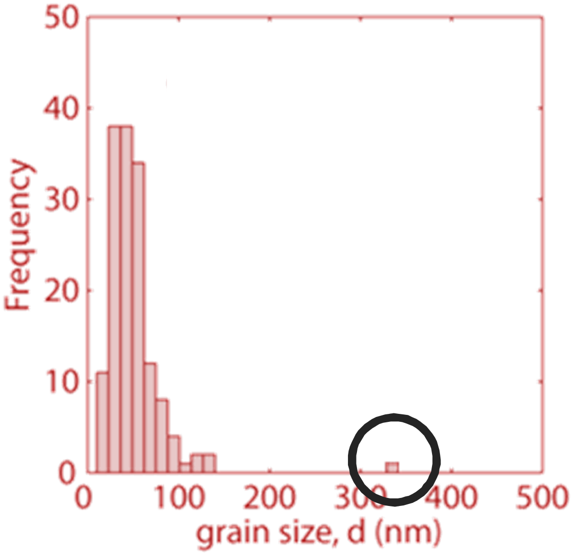
Before abnormal grain growth



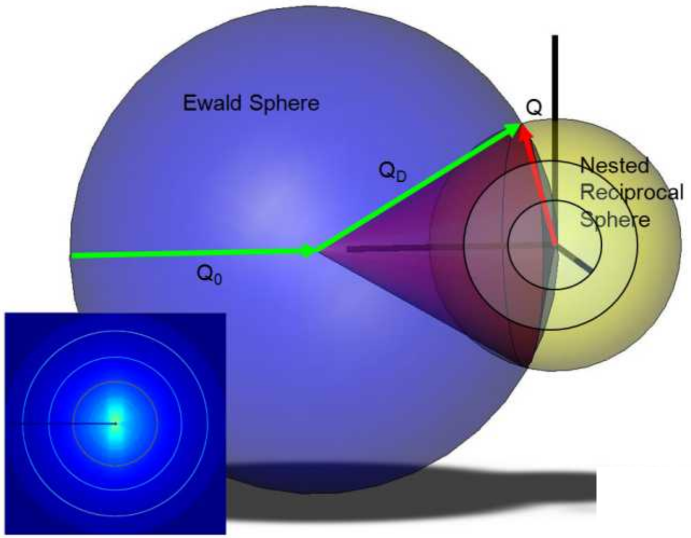


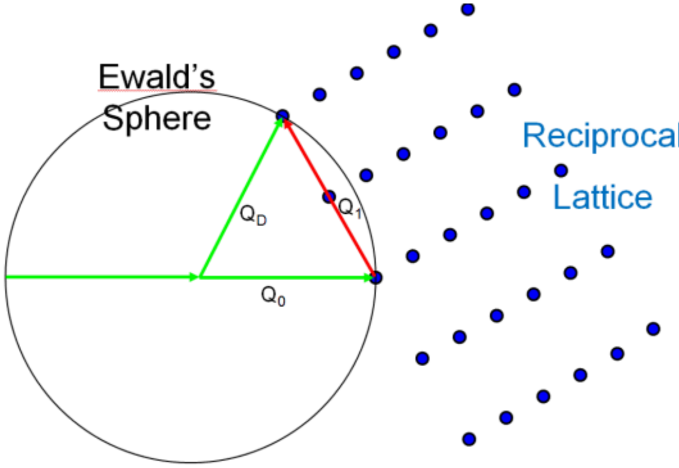
After abnormal grain growth

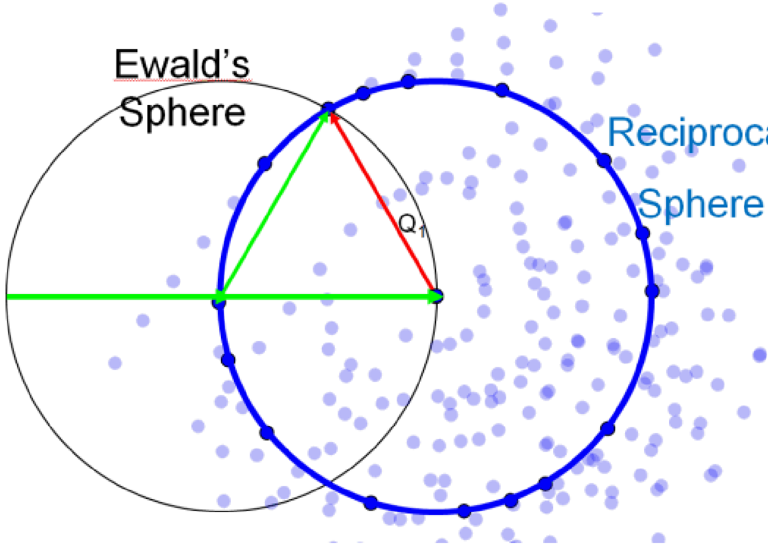




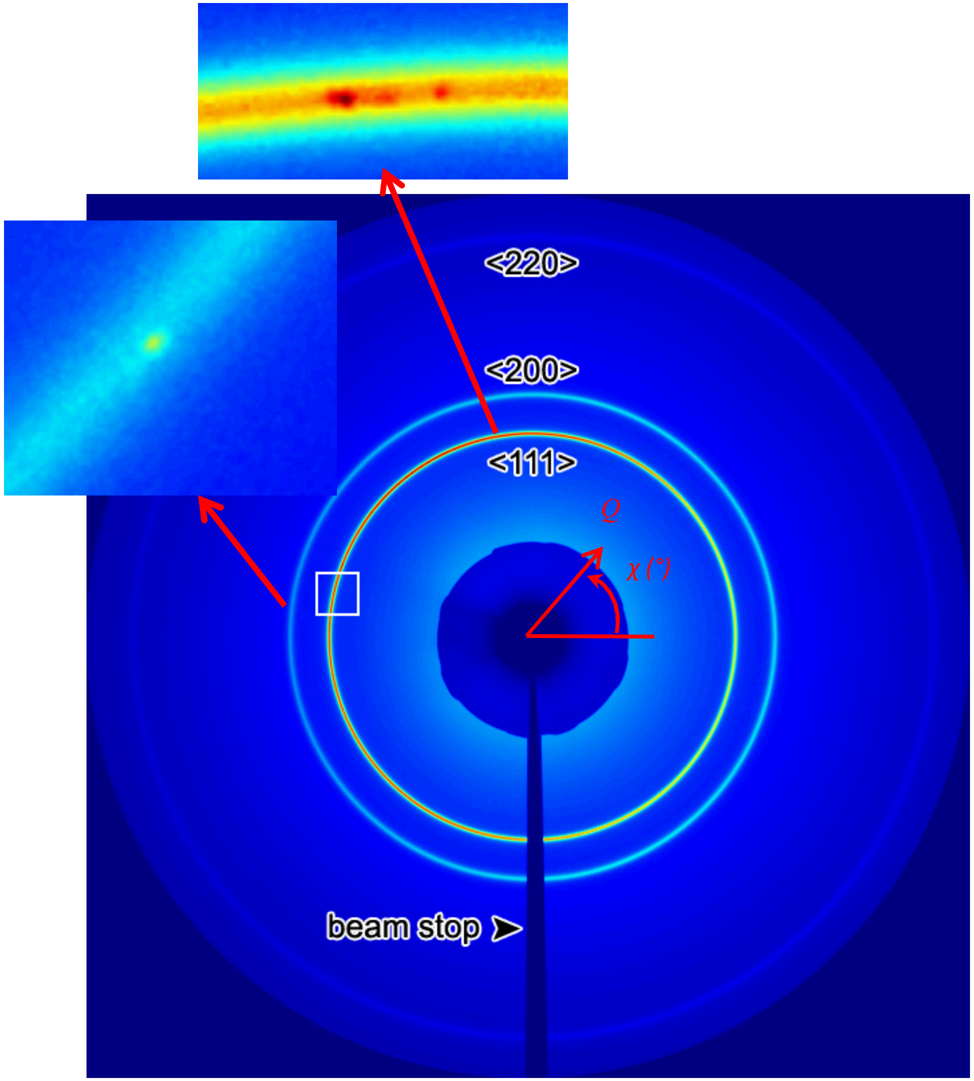
What happens in diffraction when One grain is large and the rest are small?

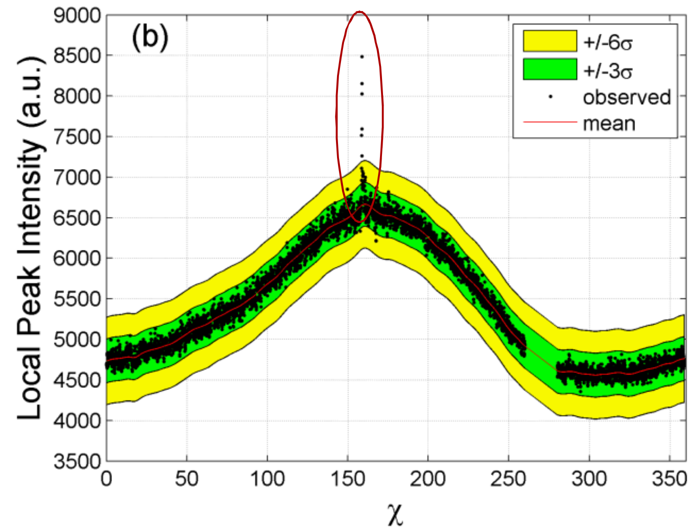






Preliminary Observation: A ‘spike’ in the Debye ring.

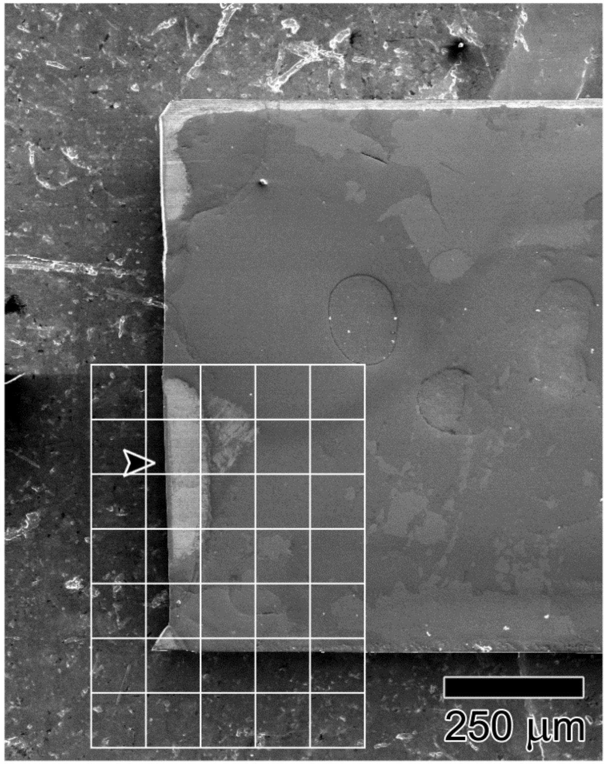


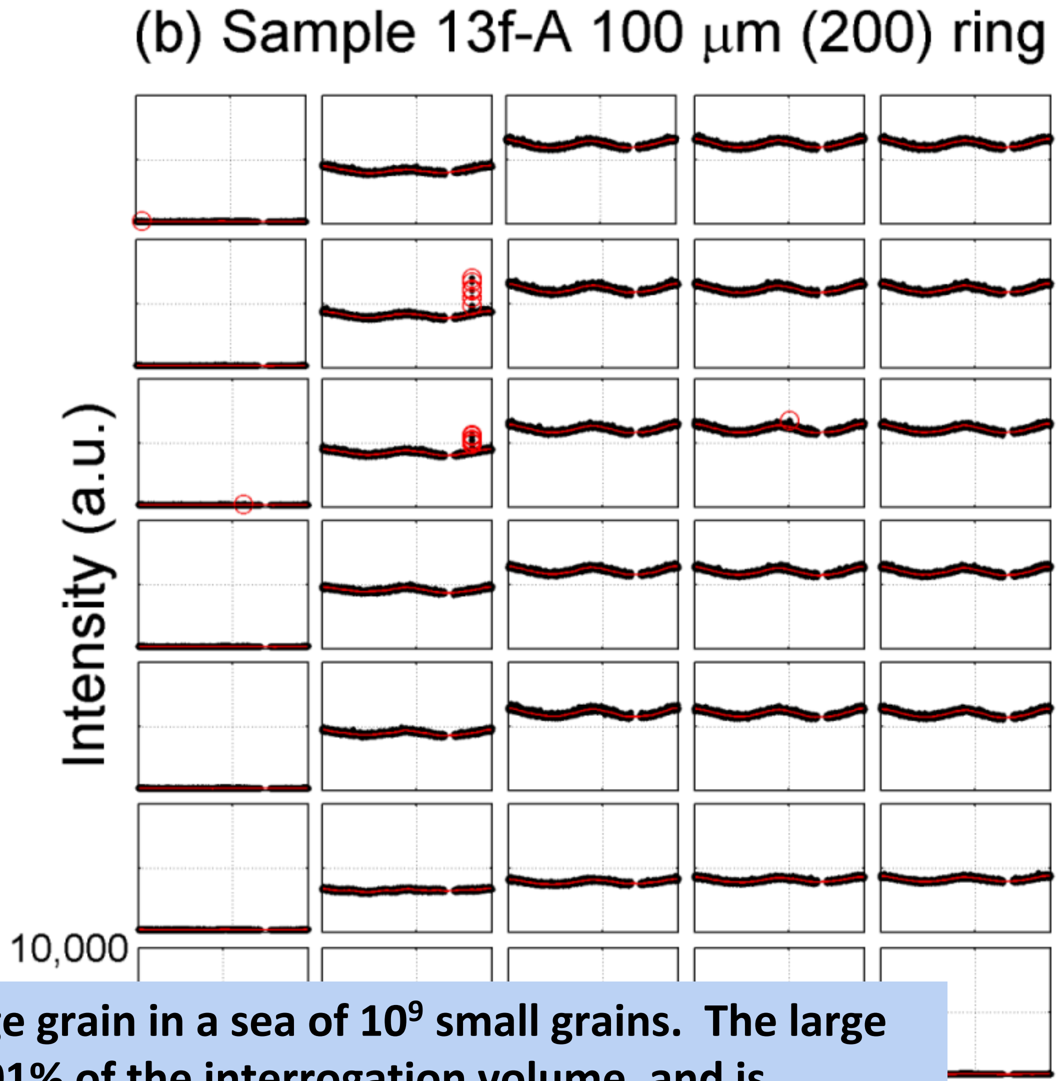


How do we know these spikes are truly statistically significant anomalies and not just noise?

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

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



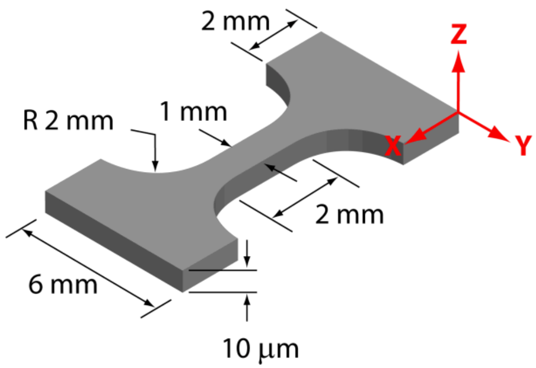


We've identified 1 large grain in a sea of 10^9 small grains. The large grain occupies ~0.00001% of the interrogation volume, and is identified with a statistical confidence >> 99.9999998% (6σ).

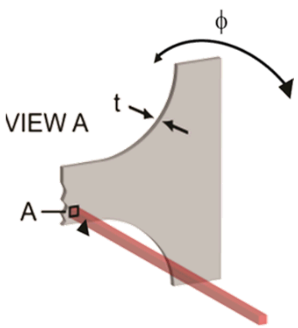
Boyce, Furnish, Padilla, Van Campen, Mehta, *J. Mater. Sci.*, 2015

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

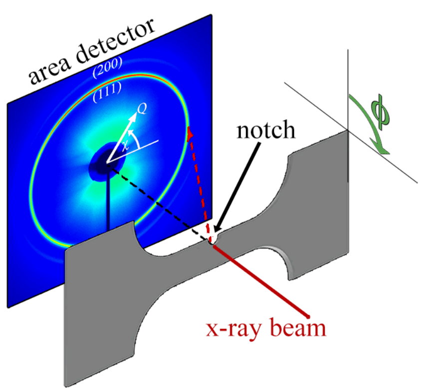
the needle-in-a-haystack challenge…
Rapidly detecting 1 abnormal grain in 10^{12}



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

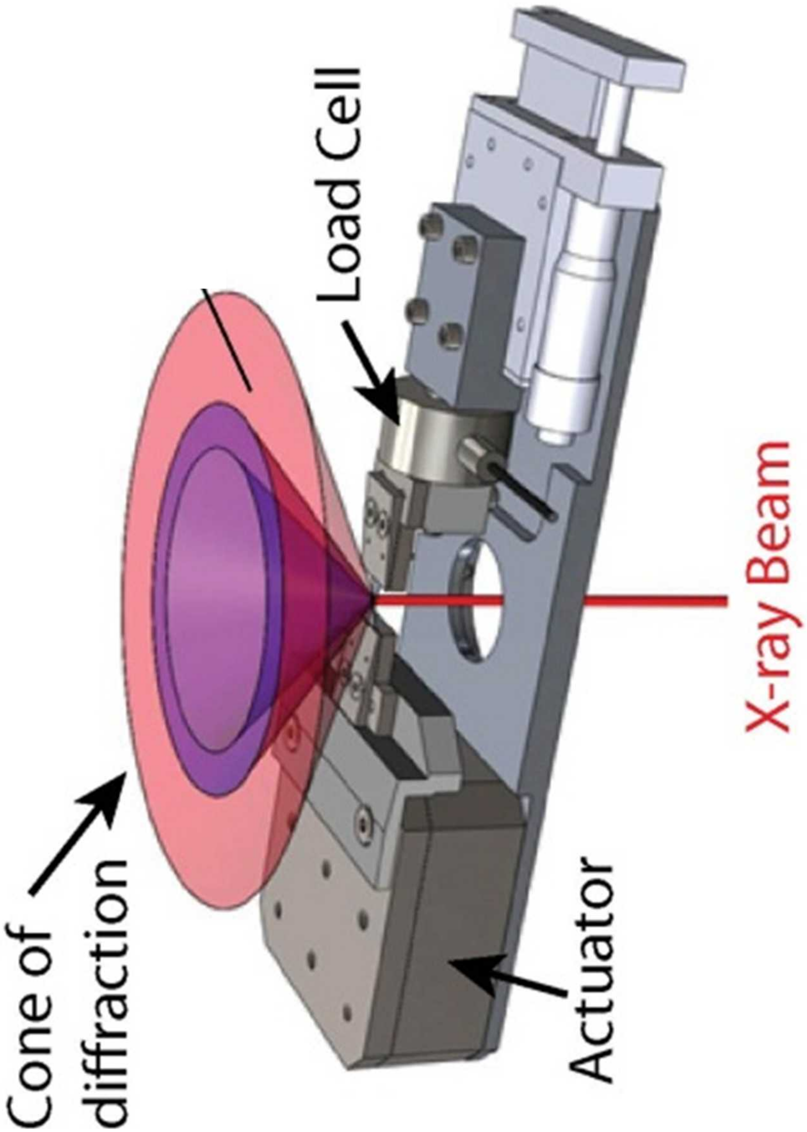


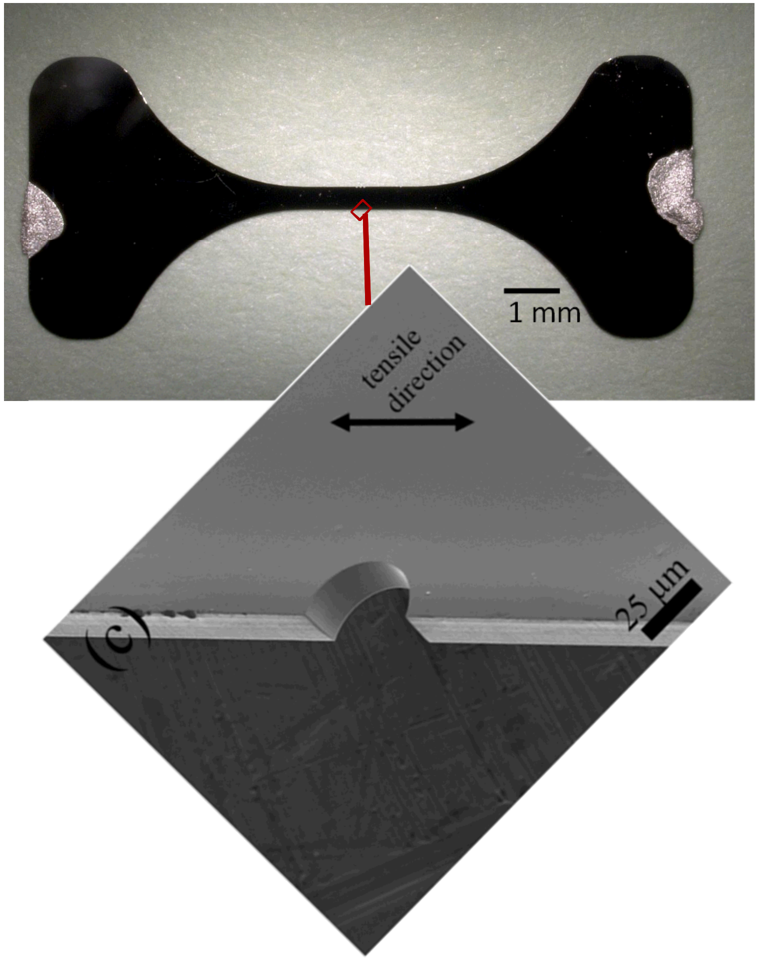
A 100x100 μm x-ray spot interrogates
~1 x10^{9} grains



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

Piezo-actuated thin film fatigue tester for the synchrotron

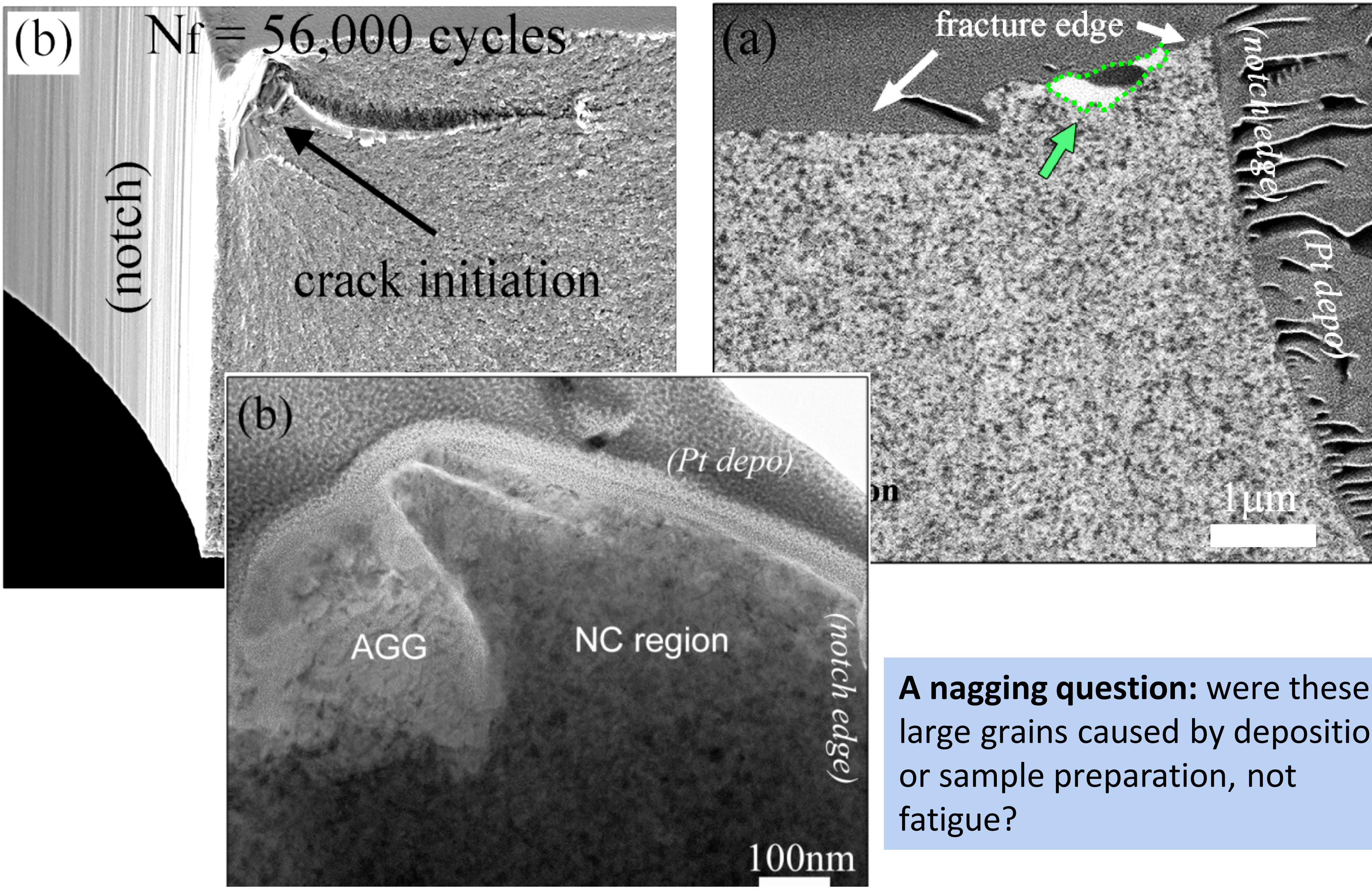




Elastic stress concentration factor:
K_t=2.8

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

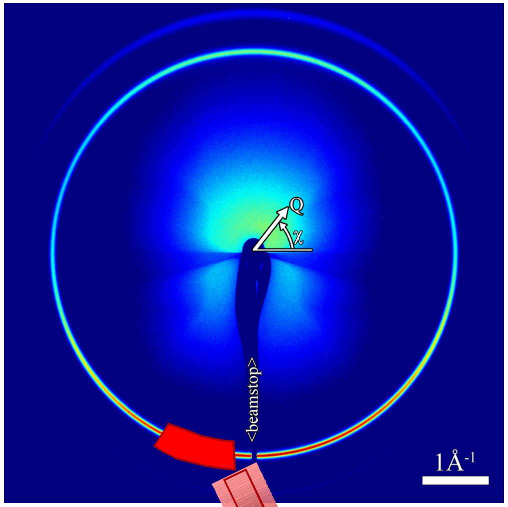


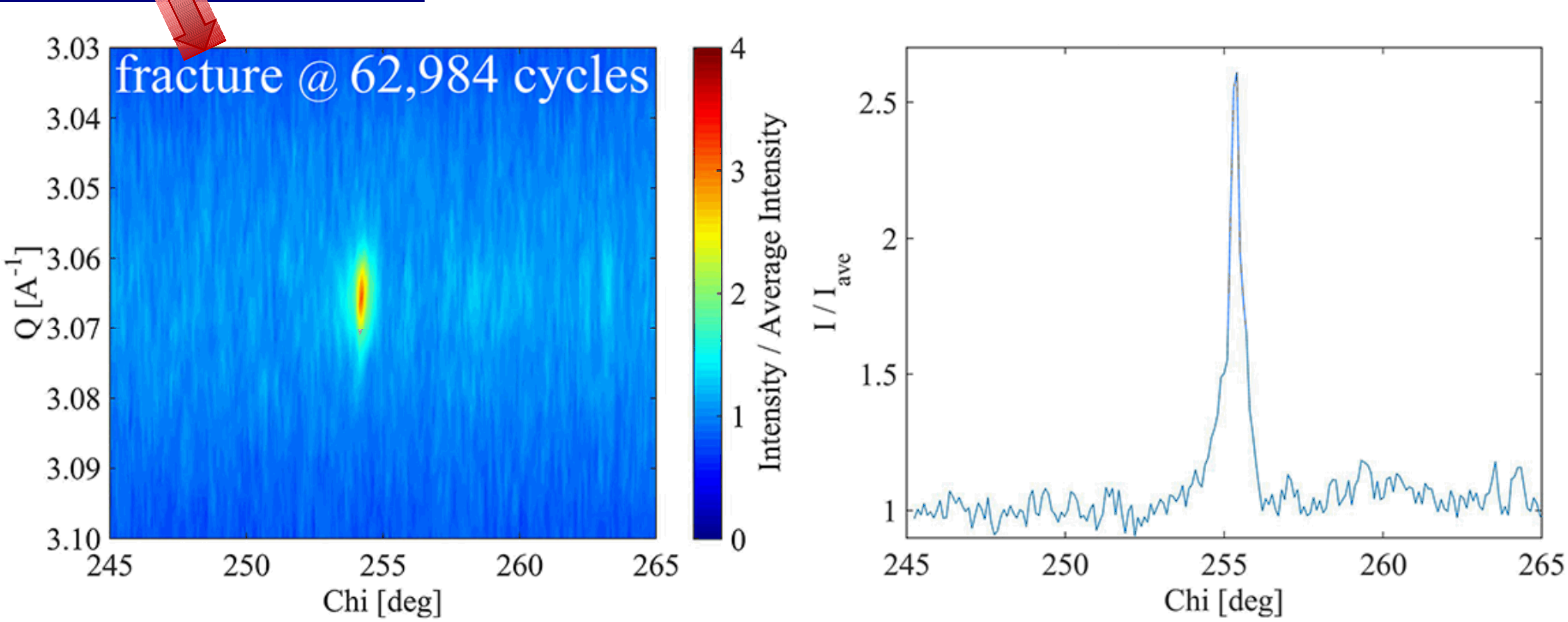
A nagging question: were these large grains caused by deposition or sample preparation, not fatigue?

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

Detecting the onset of grain growth during fatigue

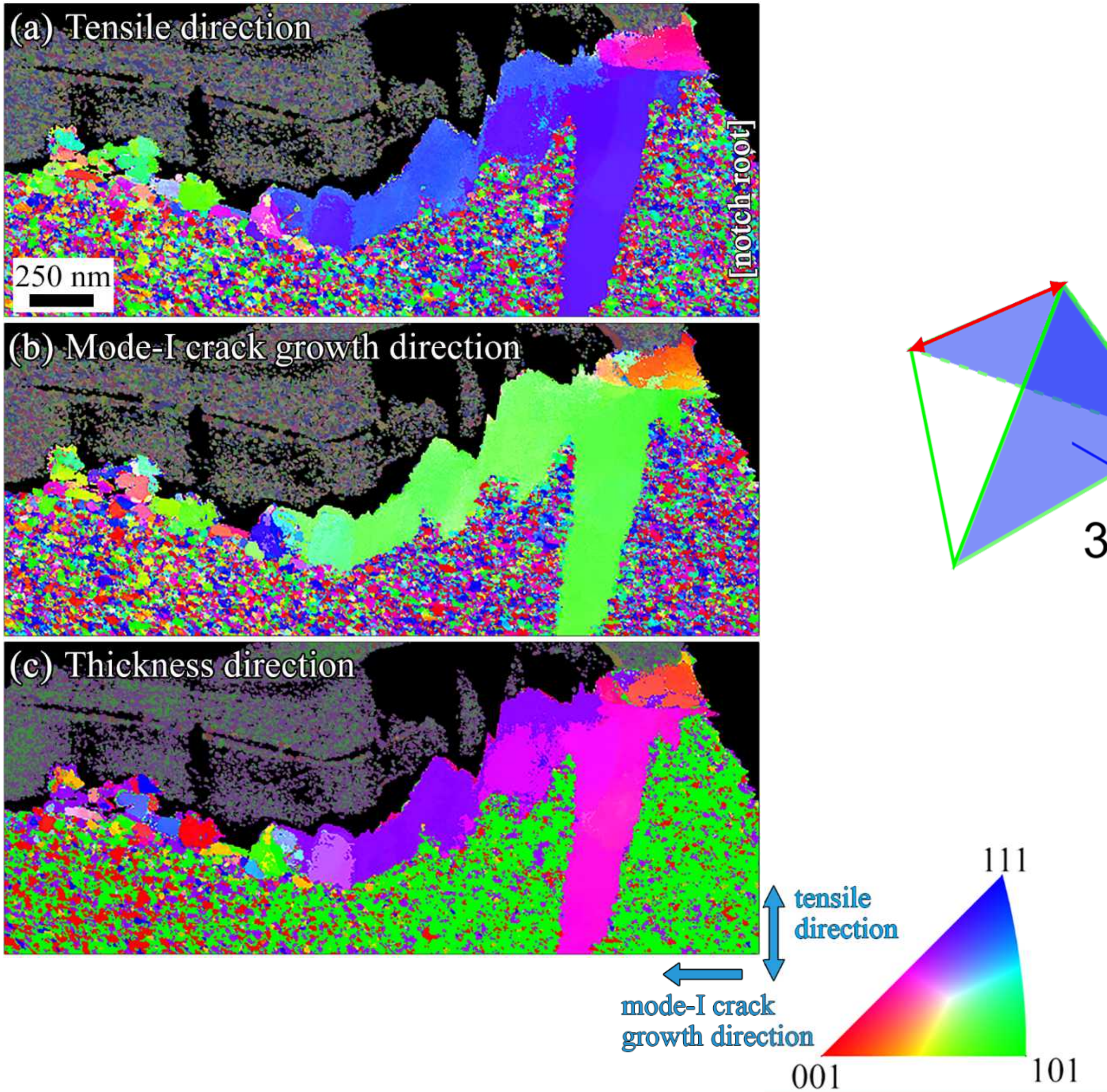
Looking at a 20º arc of the (111) diffraction ring…

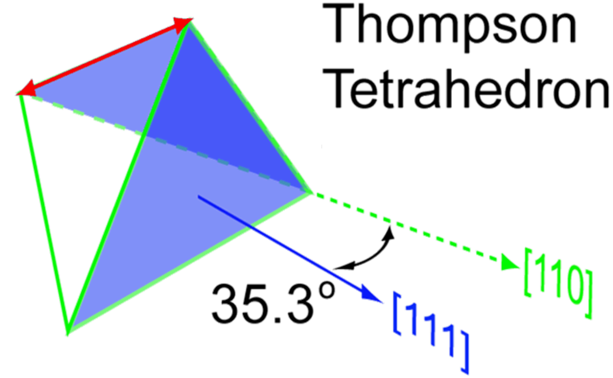




Furnish, Mehta, Van Campen, Bufford, Hattar, Boyce, *J. Mater. Sci.*, 2016

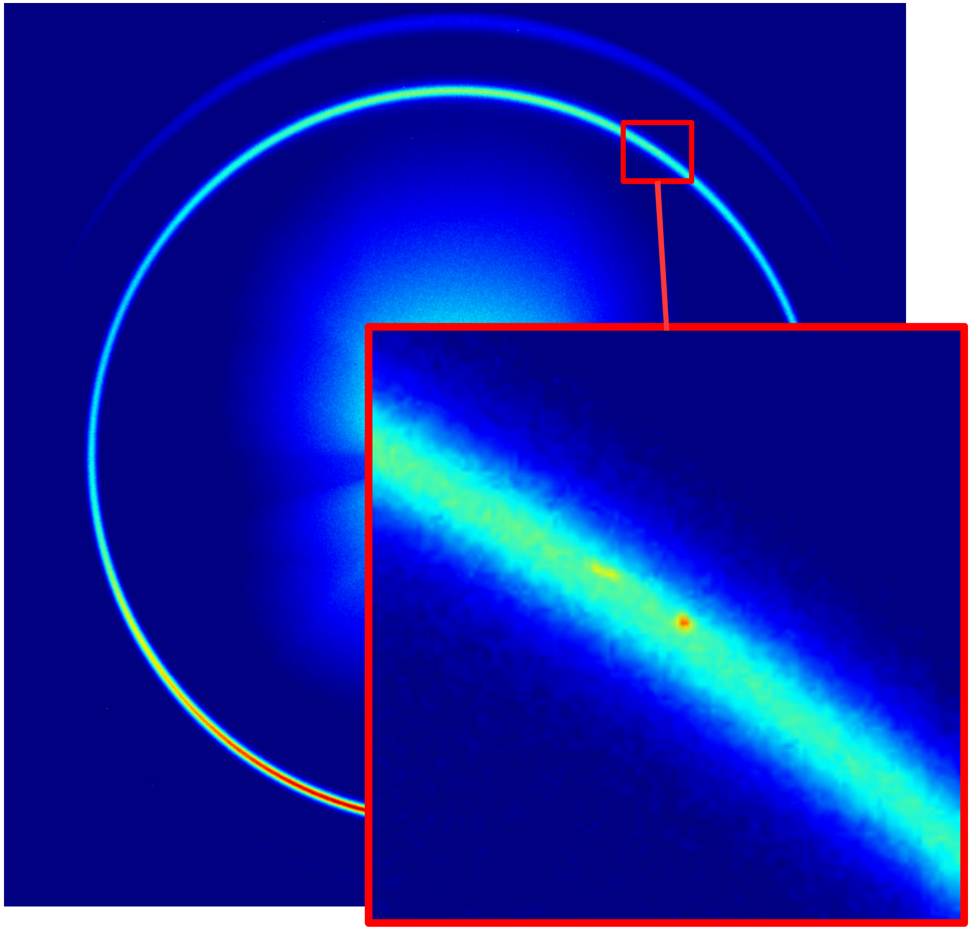
After x-ray detection, the abnormally large grains are confirmed by FIB dissection + precession electron diffraction

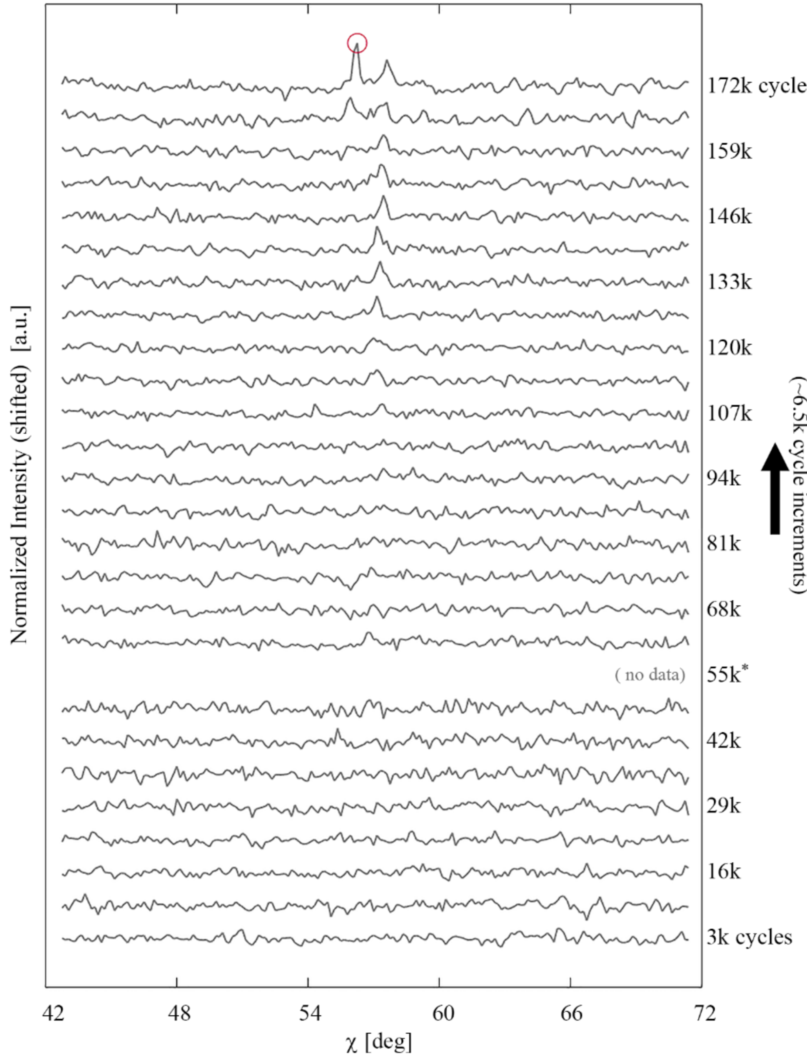




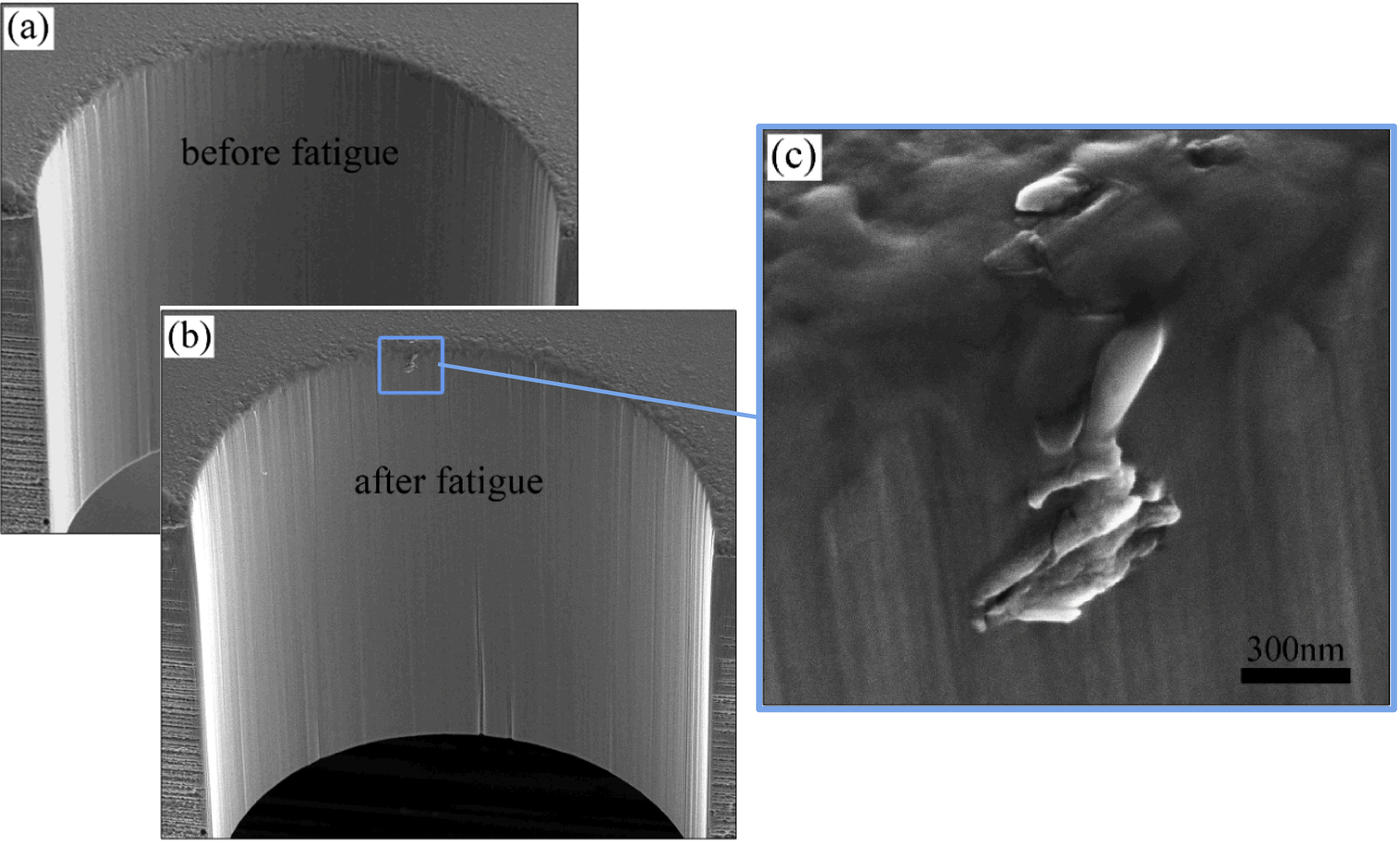
Furnish, Mehta, Van Campen, Bufford, Hattar, Boyce, *J. Mater. Sci.*, 2016

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

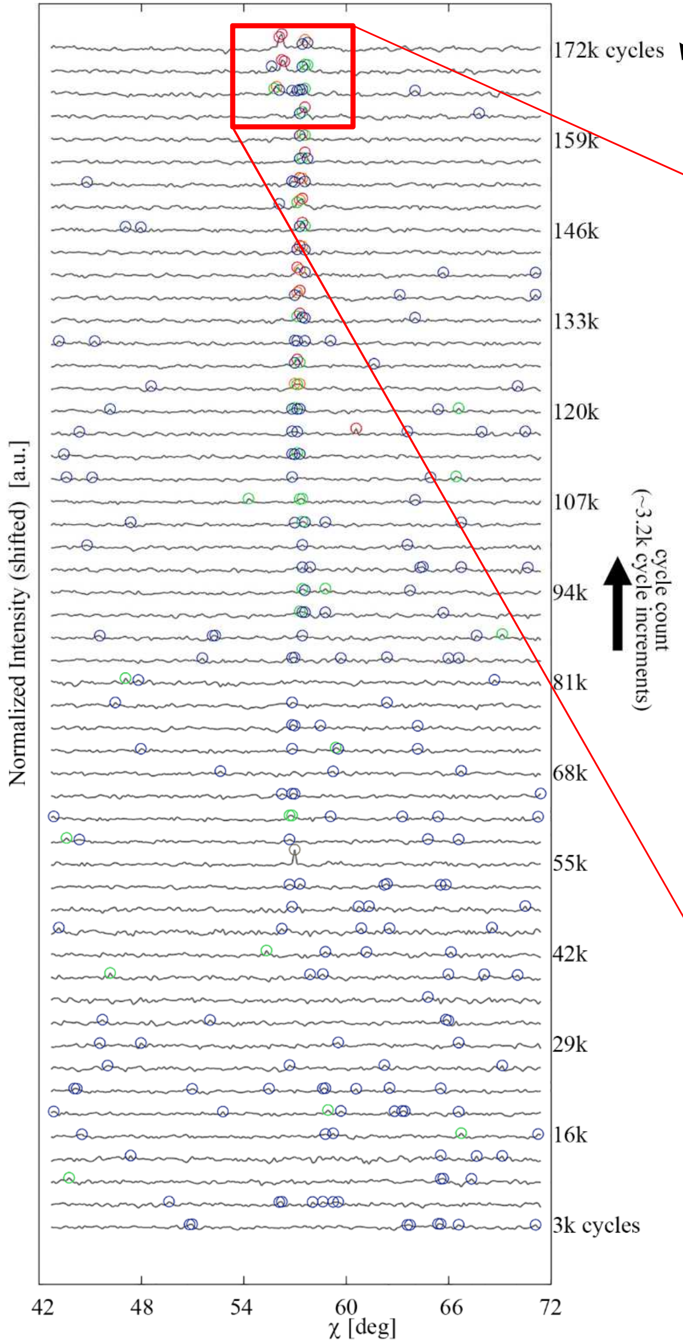




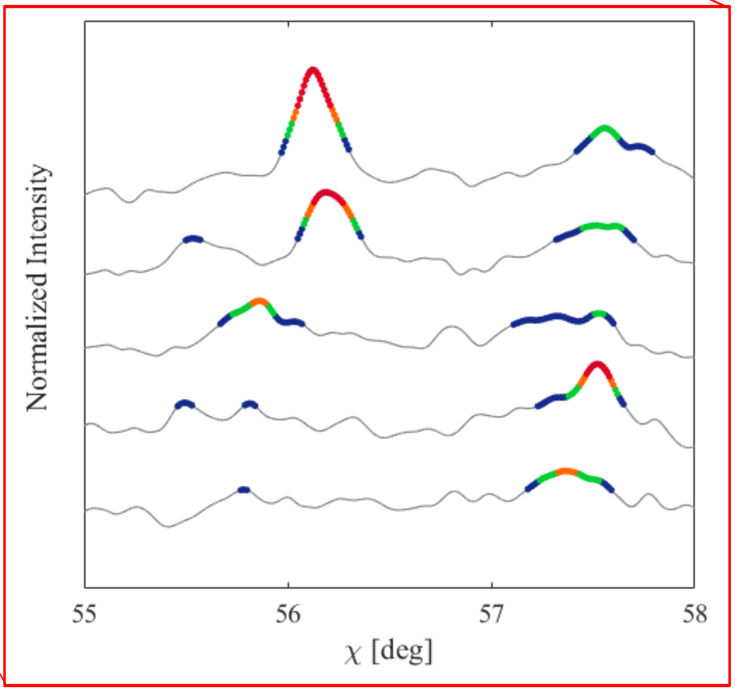
Interrupting based on fatigue testing.



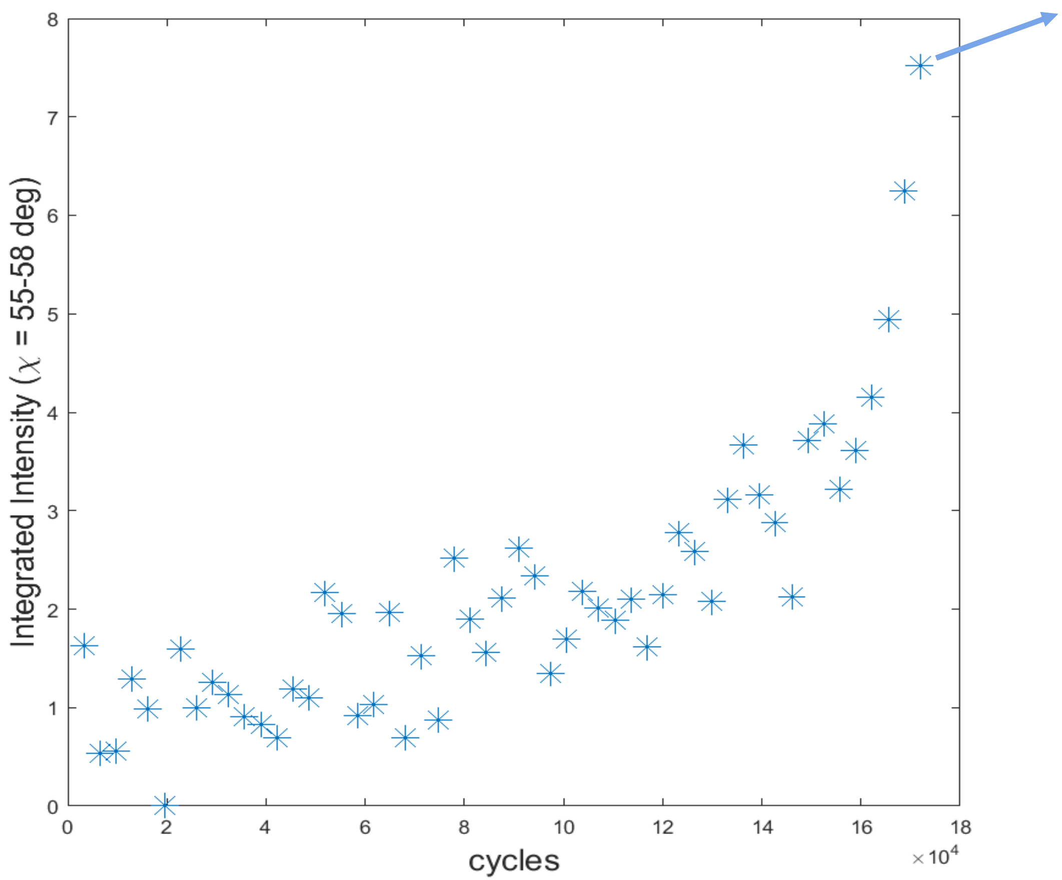
Furnish, Bufford, Mehta, Ren, Hattar, Boyce, *unpublished*

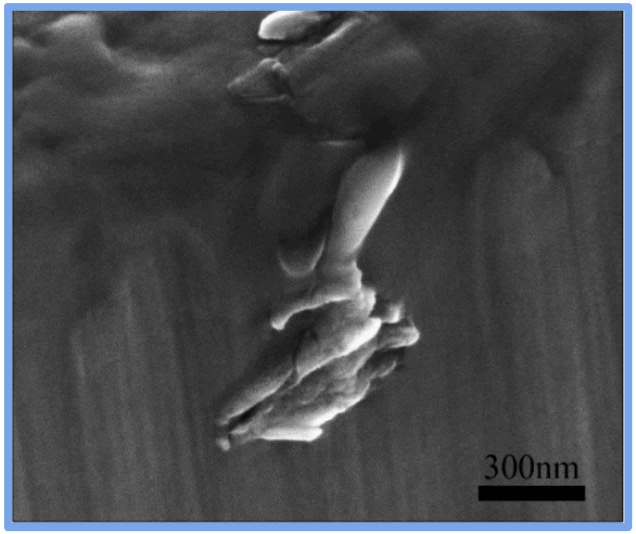


Evolution can be traced back to early in the fatigue life, well before crack initiation



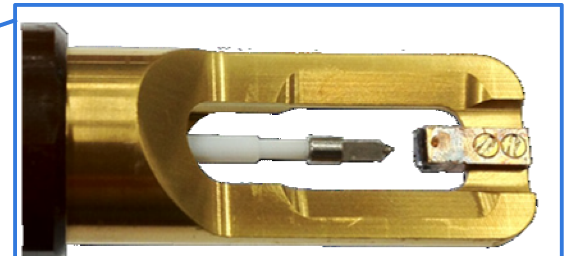
Kinetics of evolution… leading up to fatigue-crack initiation





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

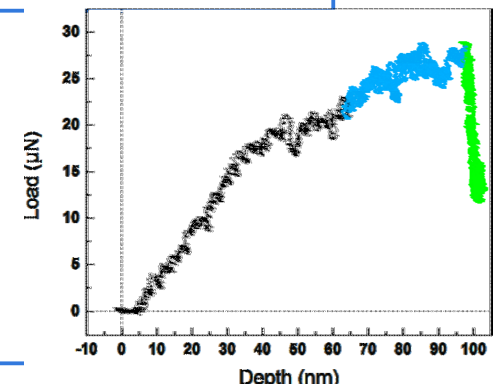
In-Situ Fatigue in the TEM is not a slam-dunk…

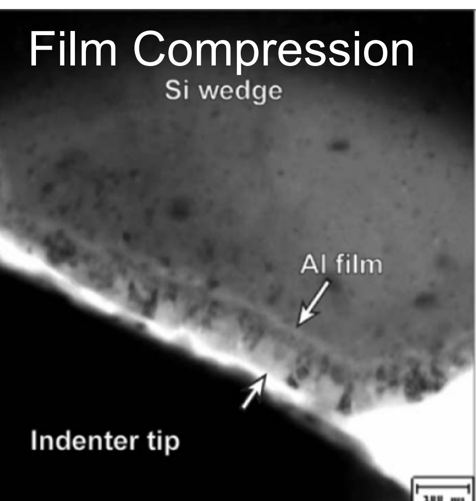


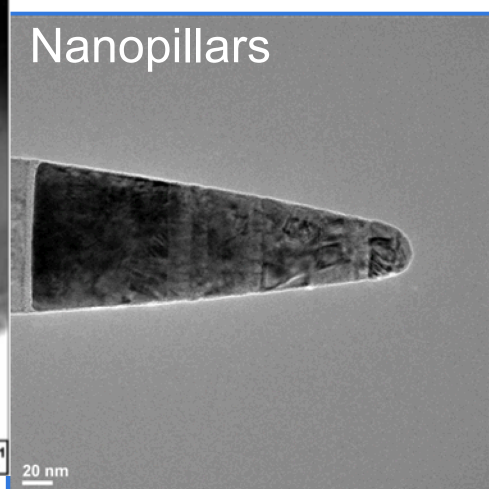
Hysitron Corp.

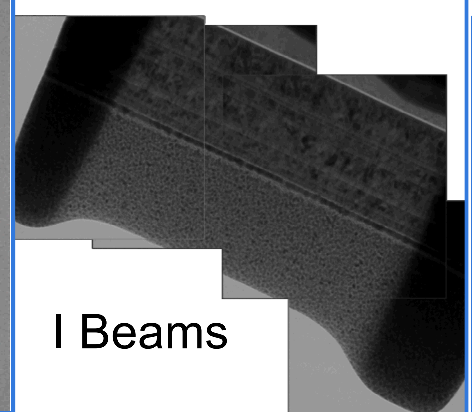
Hysitron PI95 *In Situ* Nanoindentation TEM Holder

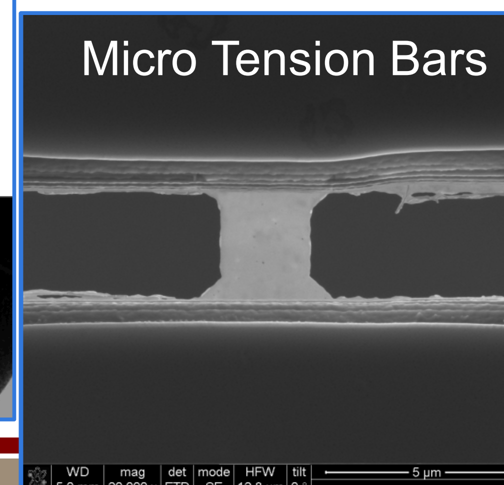
- Sub nanometer displacement resolution
- Quantitative force information with µN resolution
- Concurrent real-time imaging by TEM



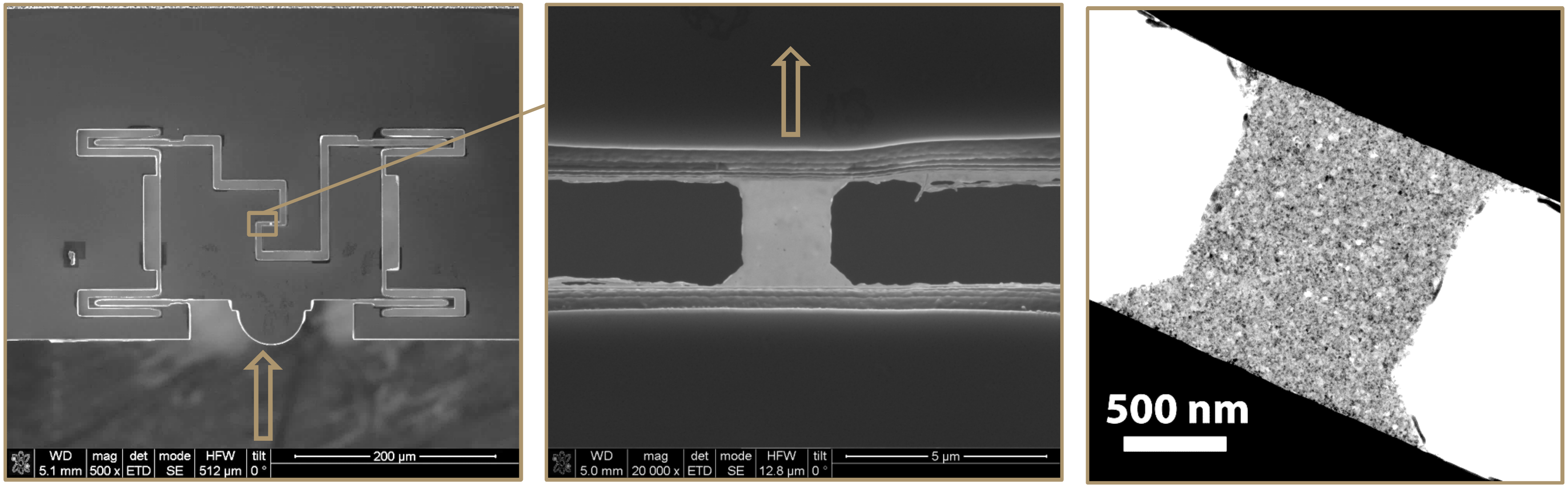








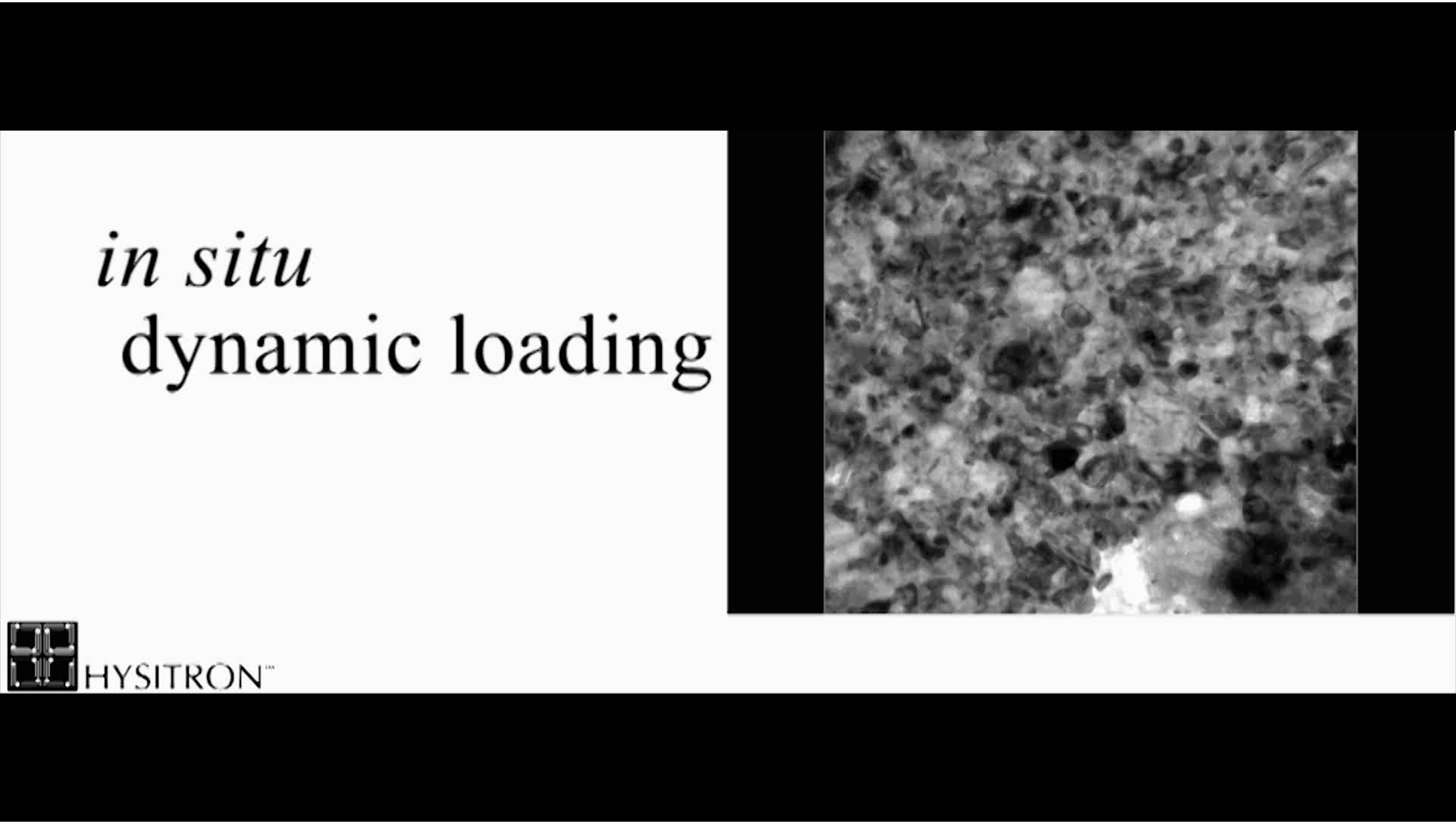
- Hysitron "Push-to-Pull" devices
  - Microfabricated Si test frame
  - Cu film (75 nm) floated onto device, then FIB milled



- Nearly pure tension, uniform cross sectional area, stable load frame
- Fragile, sensitive to shape of edges, issues with magnetic materials

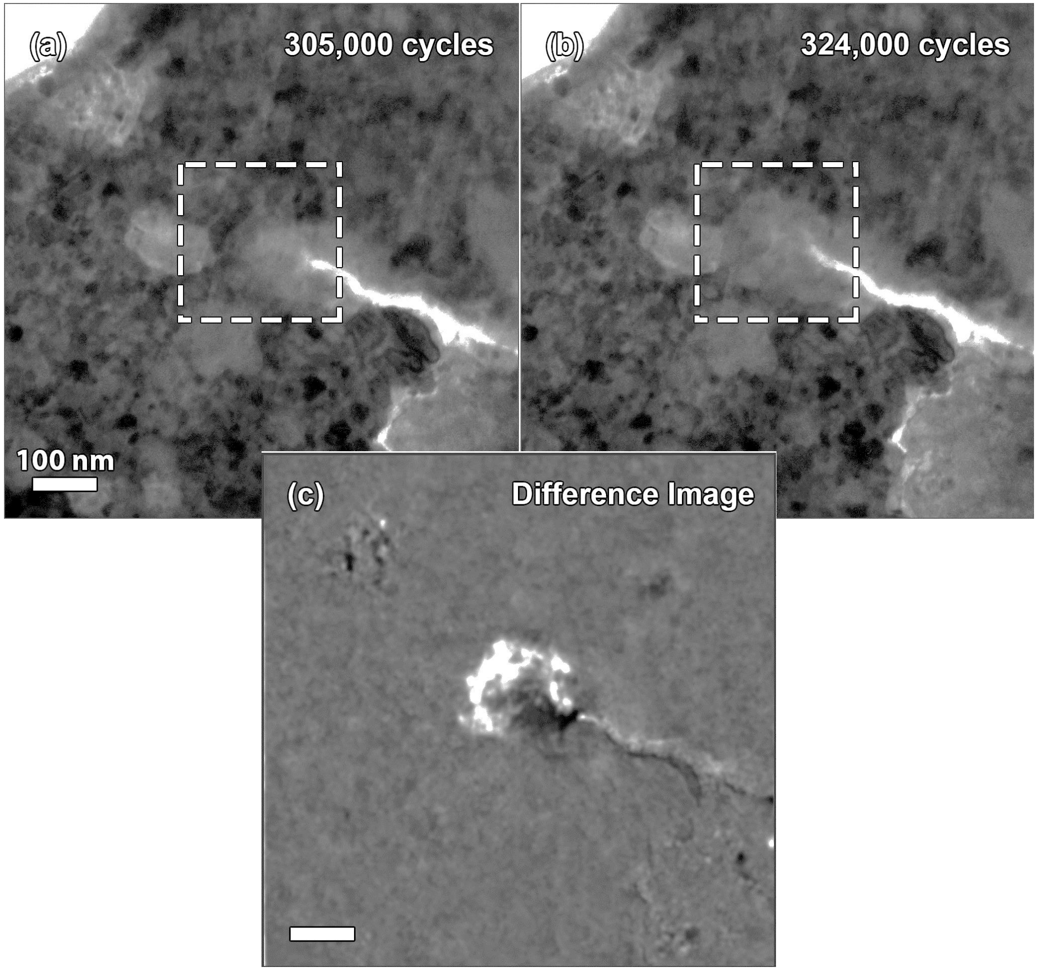
Watching fatigue-induced grain evolution directly….

Sandia National Laboratories



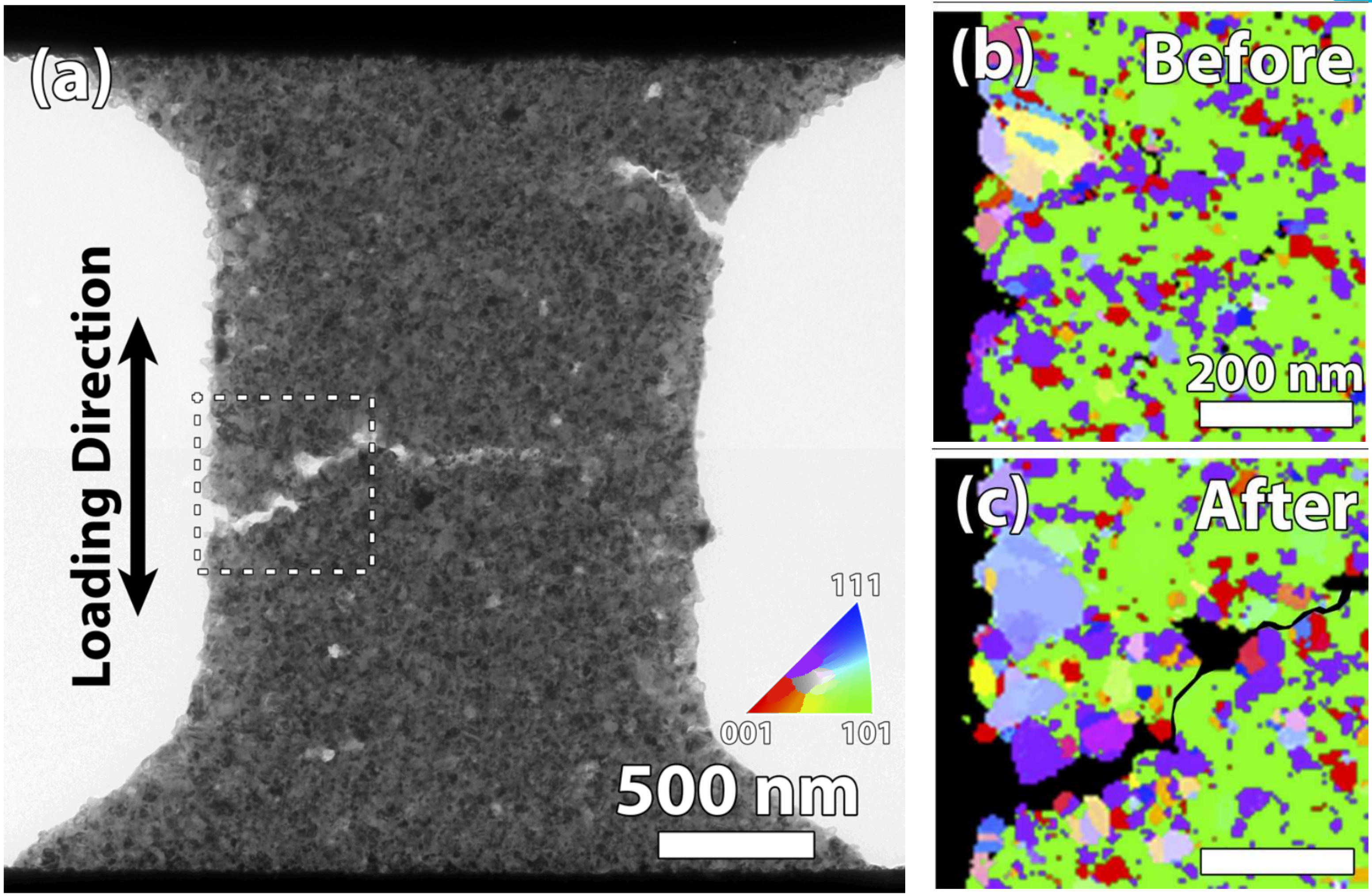
Bufford, Stauffer, Mook, Asif, Boyce, & Hattar, *Nano Letters*, 2016

Evolution during in-situ High Cycle Fatigue....



First ever high-cycle fatigue experiment in a TEM!
>300,000 cycles in ~20 minutes!

Evolution during in-situ High Cycle Fatigue….



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

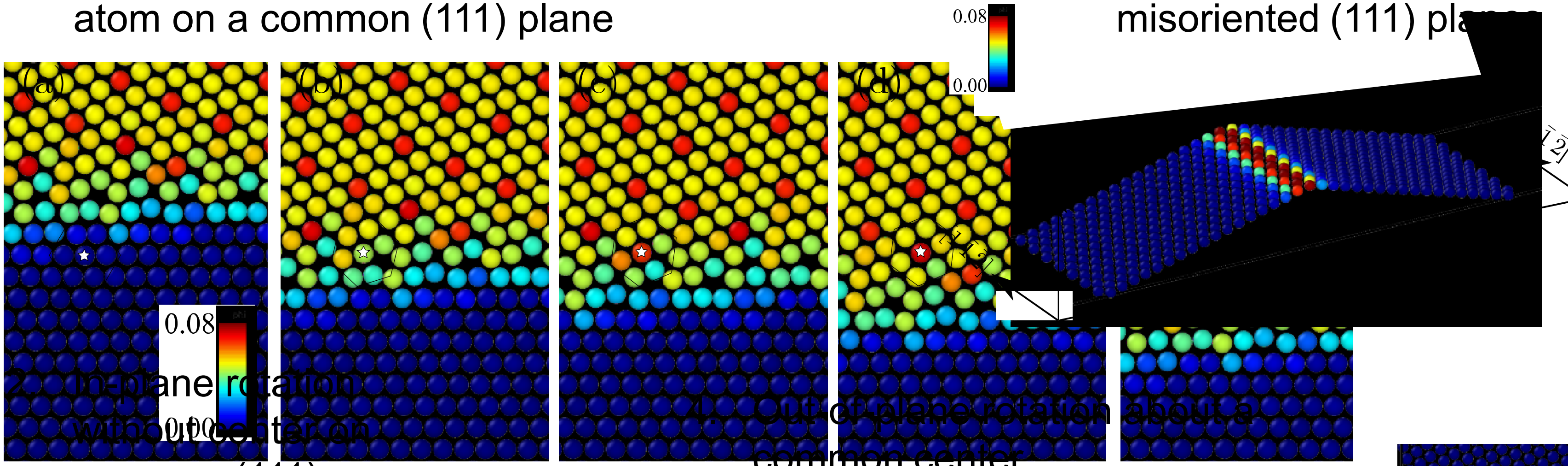
Hypothesis: a few grain boundary types have a distinct mobility advantage

Bufford, Stauffer, Mook, Asif, Boyce, & Hattar, *Nano Letters*, 2016

Mechanisms of antithermal grain boundary motion

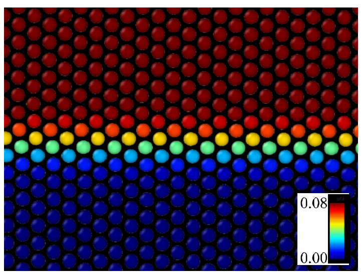
1. In-plane rotation about a fixed atom on a common (111) plane

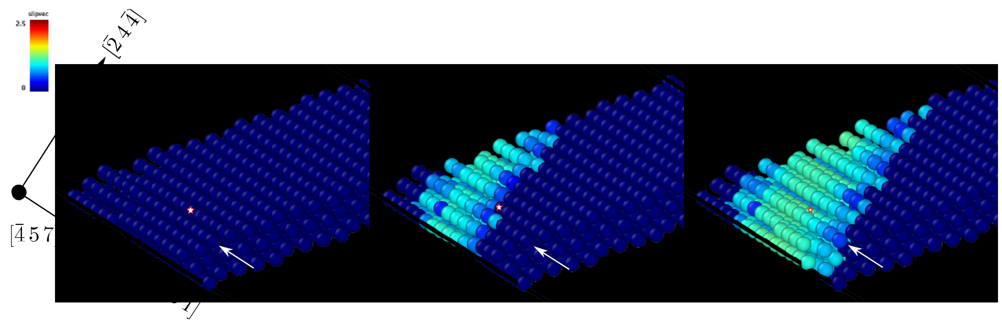
3. Rotation between two misoriented (111) pla

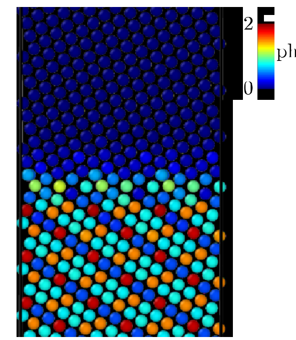


2. In-plane rotation without center on common (111) plane

4. Out of plane rotation about a common center

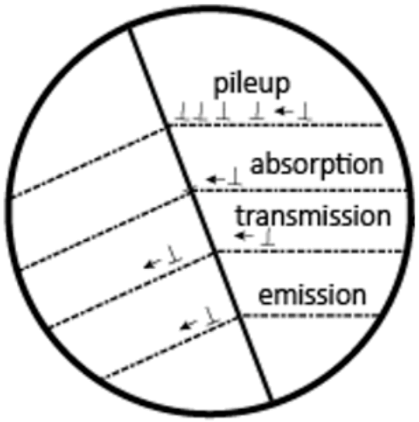






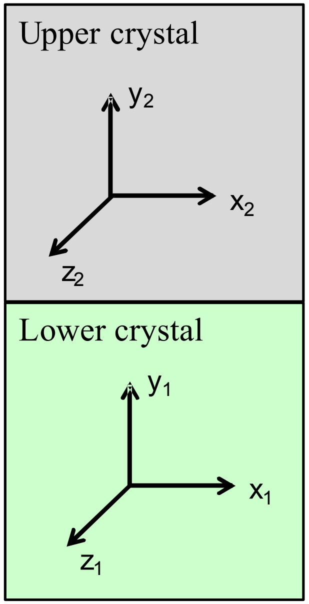
The mechanisms for anti-thermal boundary motion involve a coordinated shuffling or rotation about a common plane, typically (111). Because of the apparent coordinated motion, it bears similarity to a martensitic/military motion rather than a diffusive motion.

Some rare boundaries are triggered by dislocations



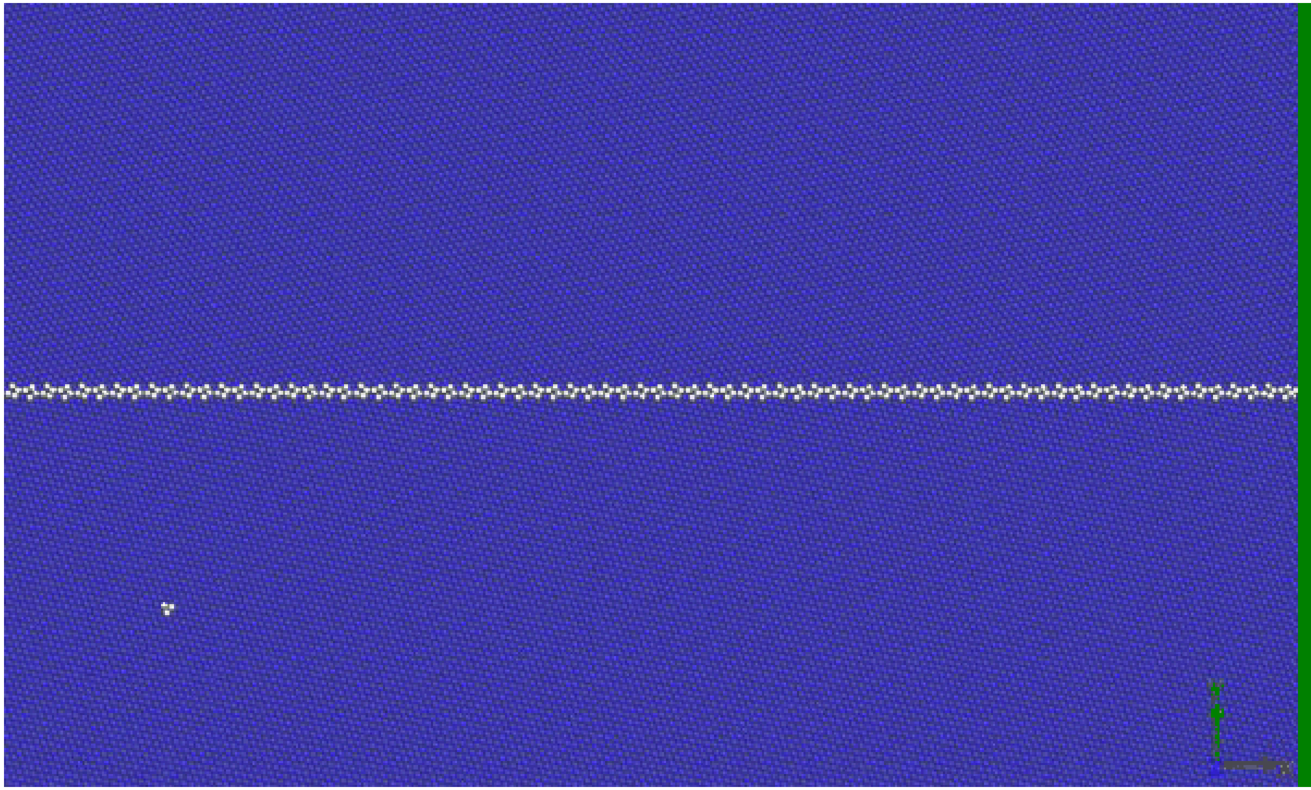
An example of dislocation ***absorption***
+ propagating GB structural changes
+ emission

S 7 <111> {1 4 5} GB plane normal

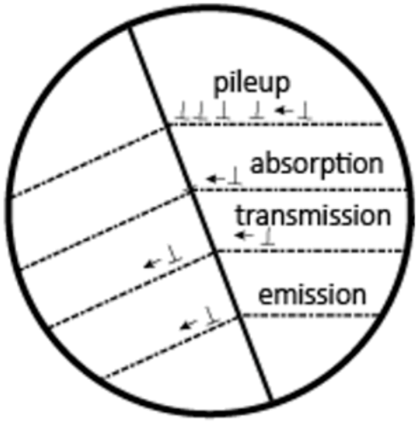


x1 = [2 1 -3]
y1 = [-4 5 -1]
z1 = [1 1 1]

x1 = [3 -1 -2]
y1 = [-1 5 -4]
z1 = [1 1 1]

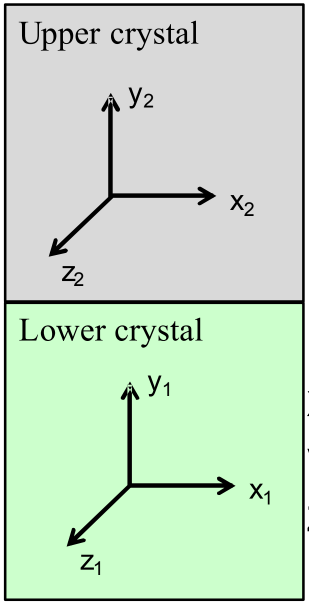


Some rare boundaries are triggered by dislocations



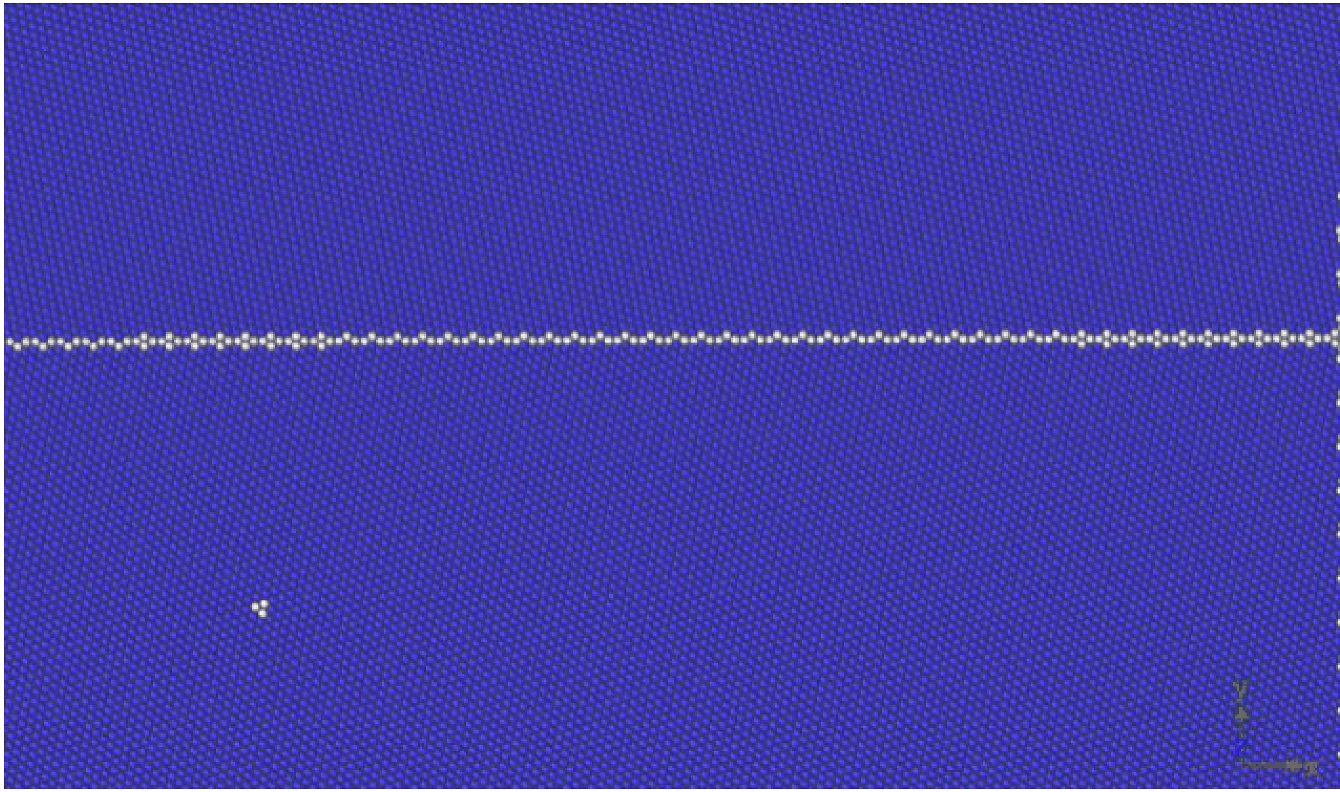
An example of dislocation absorption,
+ defect formation
+ **GB migration**

S 7 <111> {1 2 3} GB plane normal



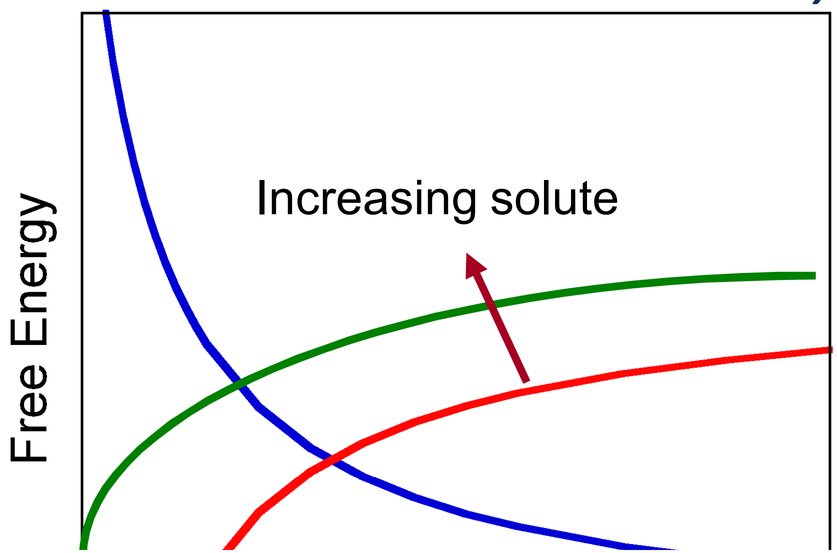
x1 = [5 -1 -4]
y1 = [-1 3 -2]
z1 = [1 1 1]

x1 = [5 -4 -1]
y1 = [1 2 -3]
z1 = [1 1 1]



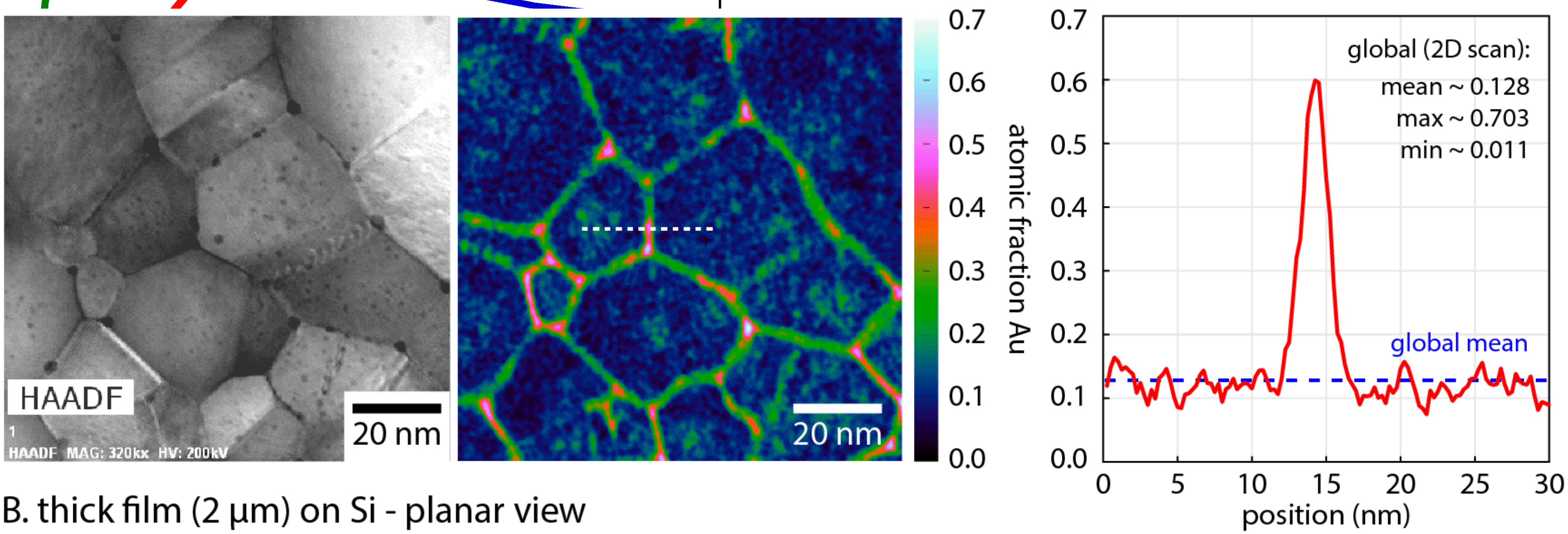
Collaboration with Stephen Foiles, Fadi Abdeljawad

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



$$dG = \gamma dA$$

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



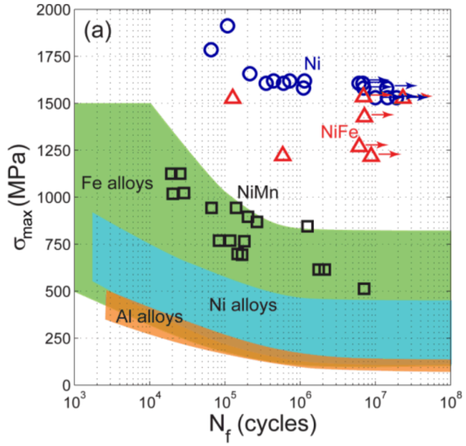
B. thick film (2 μm) on Si - planar view

Chookajorn, Murdoch, and Schuh, *Science*, 2012

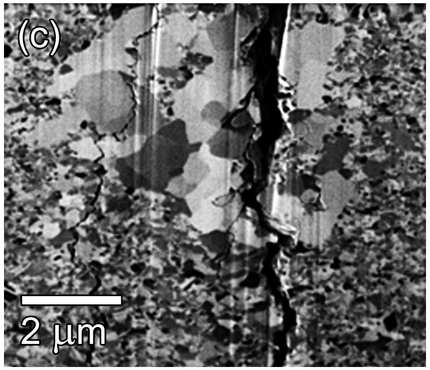
Recap…


4-28-1988 After 89,090 flight cycles on a 737-200, metal fatigue lets the top go in flight.

Fatigue is a failure process which continues to challenge structural metals.



Nanocrystalline metals offer outstanding strength, and enhanced fatigue resistance



Nanocrystalline grains grow under fatigue loading and these larger grains cause crack initiation, limiting fatigue enhancement.

Backup…

The New York Times

Sandia National Laboratories

Pentagon Orders F-35 Jets Grounded

Michael Spooneybarger/Reuters

An inspection found a crack in a turbine blade in the engine of one of the planes.

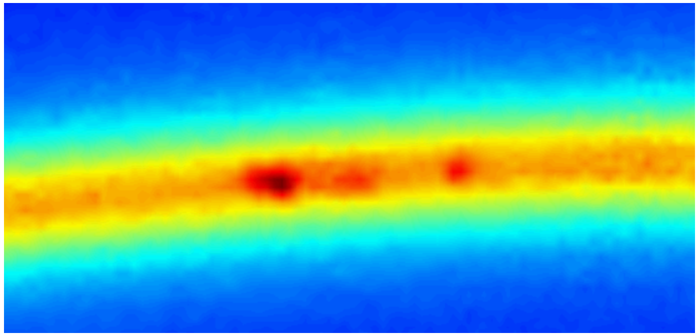
By CHRISTOPHER DREW
Published: February 22, 2013

FACEBOOK
TWITTER
GOOGLE+

The Pentagon said on Friday that it had grounded all of its stealthy new F-35 fighter jets after an inspection found a crack in a turbine blade in the engine of one of the planes.

The Pentagon office that runs the program said the crack in the turbine blade was discovered on Tuesday in a routine inspection. The crack occurred on a test plane at Edwards Air Force Base in California. The blade is being shipped to a plant in Connecticut, where the engine manufacturer, Pratt & Whitney, will inspect it and look for the problem's cause.

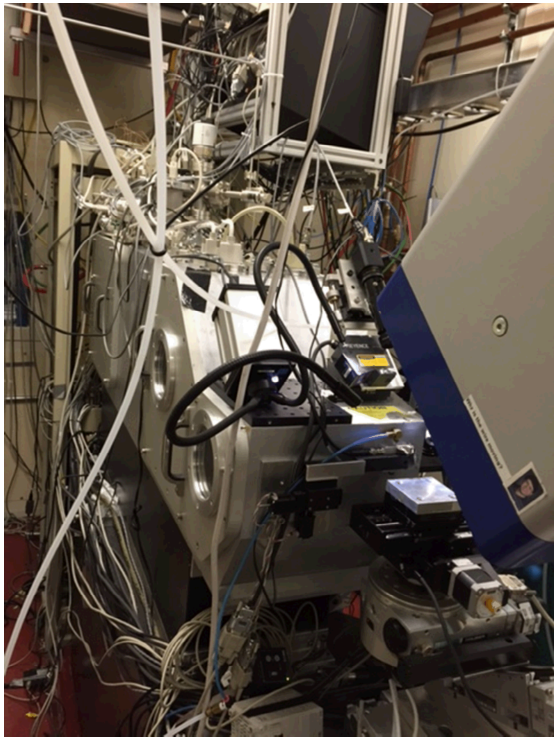
Summary

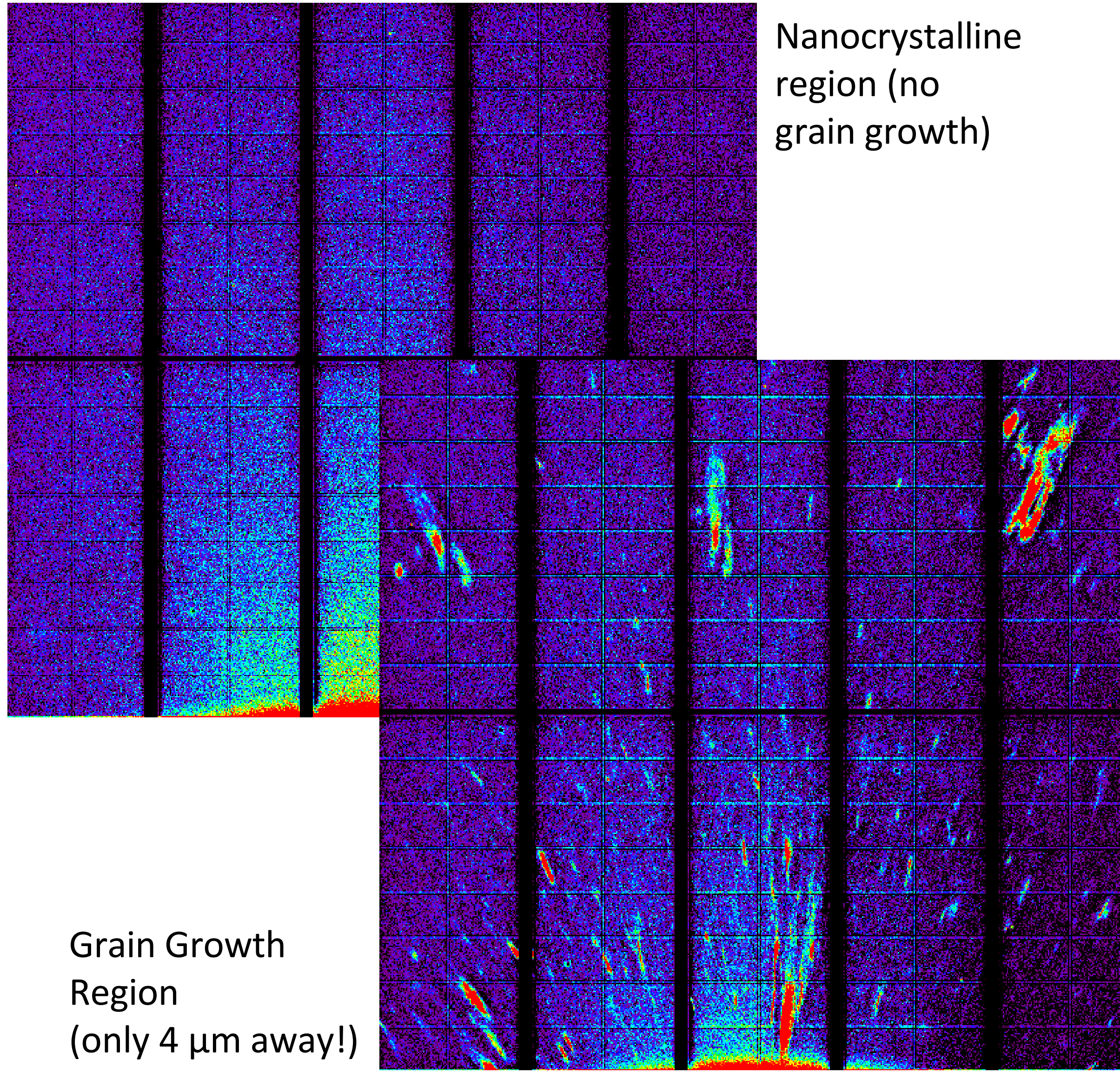


1. A new x-ray diffraction modality allows the observation of **dynamic abnormal grain growth during fatigue testing**.

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!)