

Nanocrystalline alloys: Impervious to Fatigue Failure?

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April 19th , 2013



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Versailles 1842

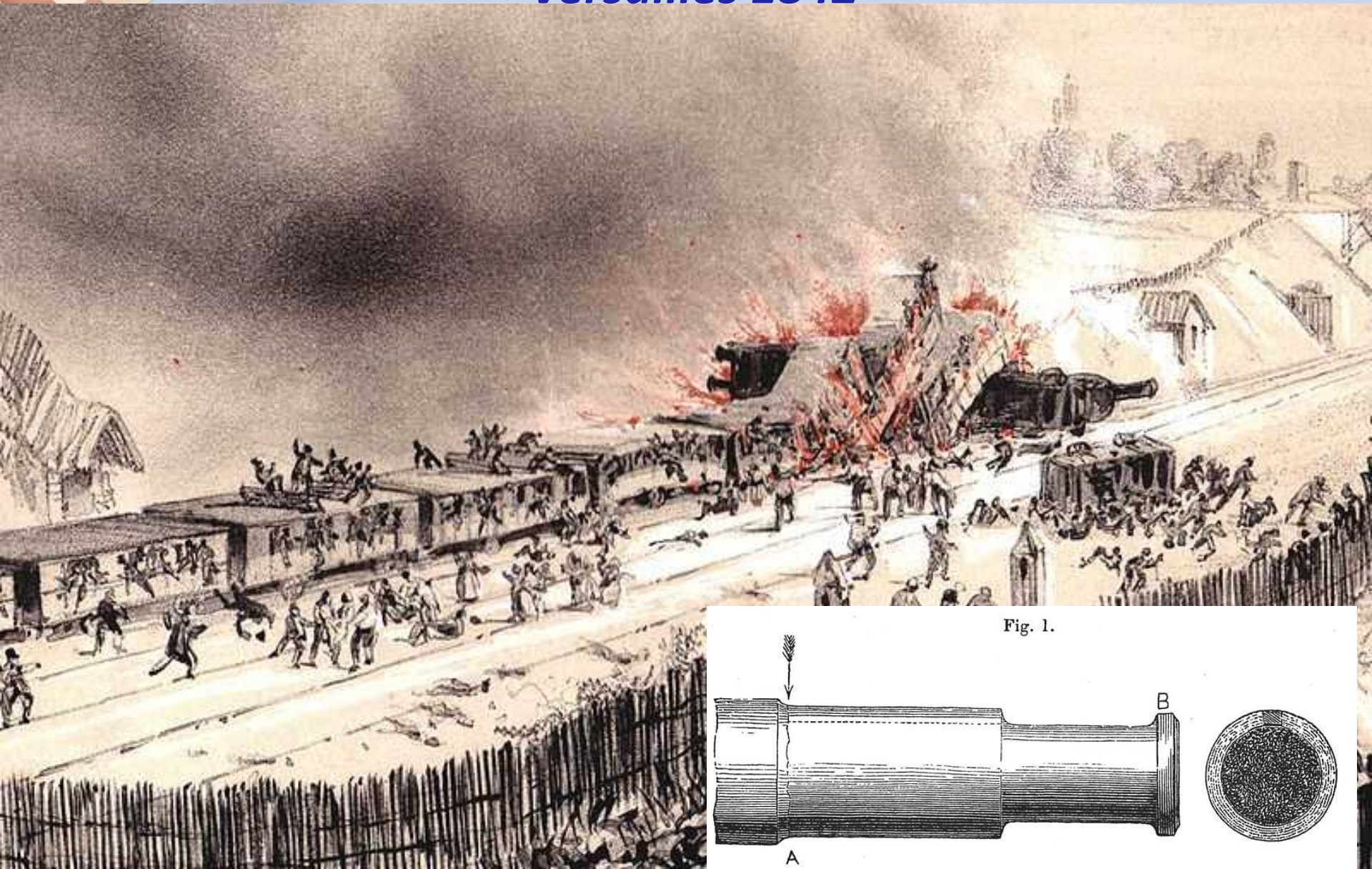
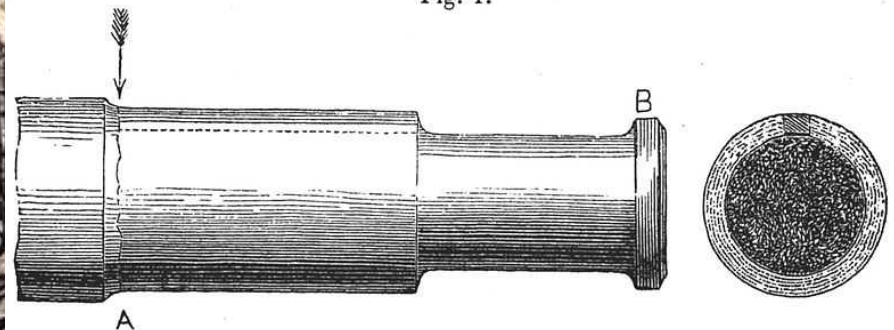
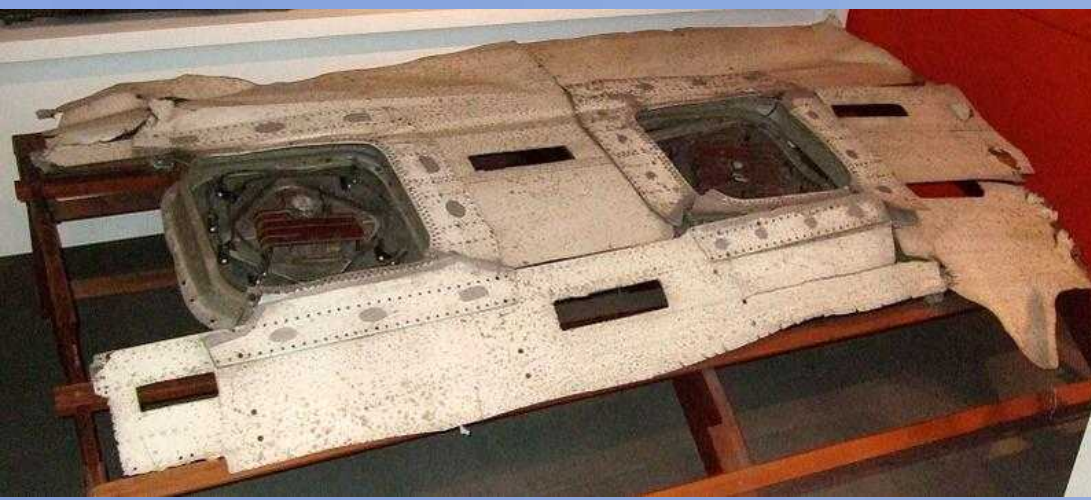


Fig. 1.



De Havilland Comet, 1953-4



Aloha Airlines, 1988



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

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

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.

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Background Concepts: 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.

Fracture and Fatigue cost U.S. economy an estimated \$119 billion.

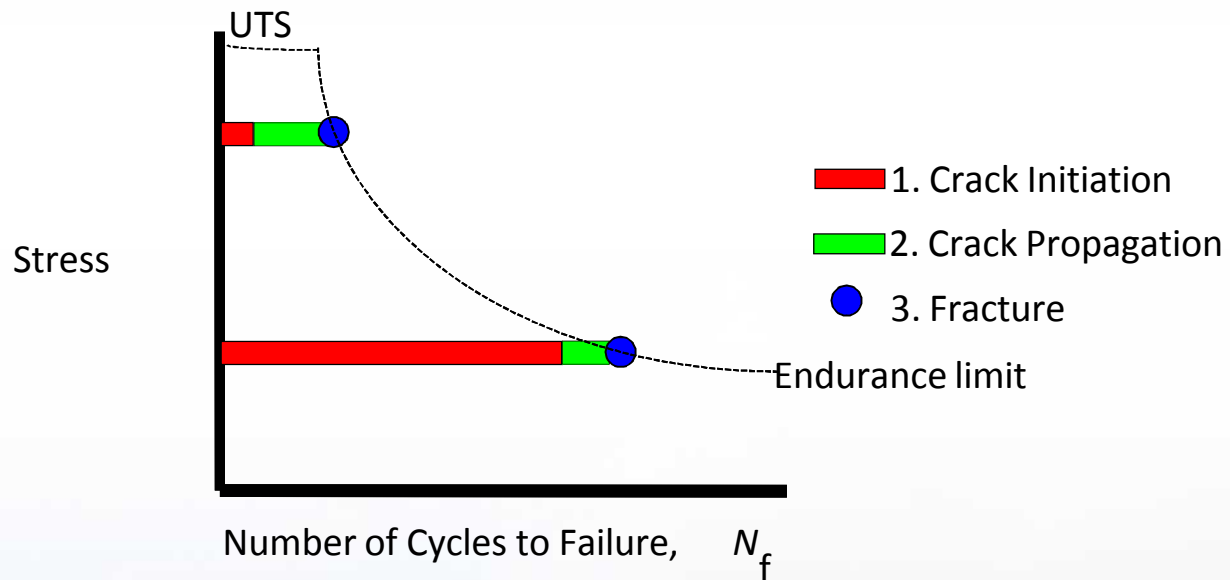
The Economics Effects of Fracture in the U.S., U.S. Dept. of Commerce, 1982.

70-90% of all structural fractures result from cyclic damage accumulation (fatigue).

Fatigue occurs in metals, polymers, ceramics, and composites, although the mechanisms can differ between classes.



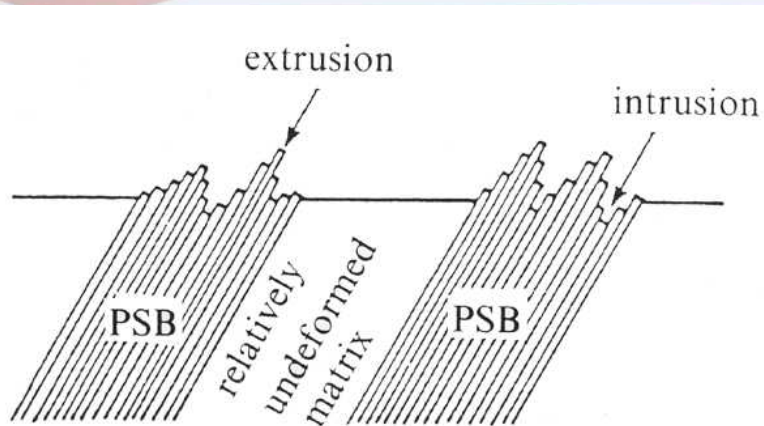
Background Concepts: Fatigue Life



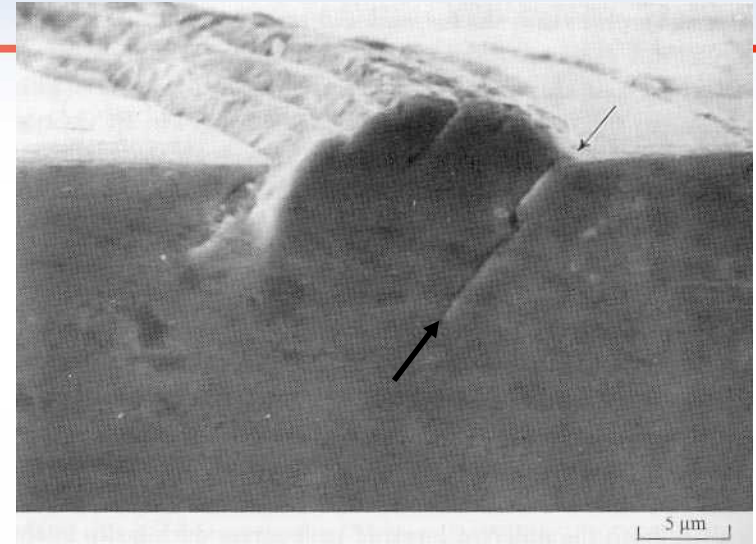
The S-N approach was originally described by Wöhler, 1860; Basquin 1910



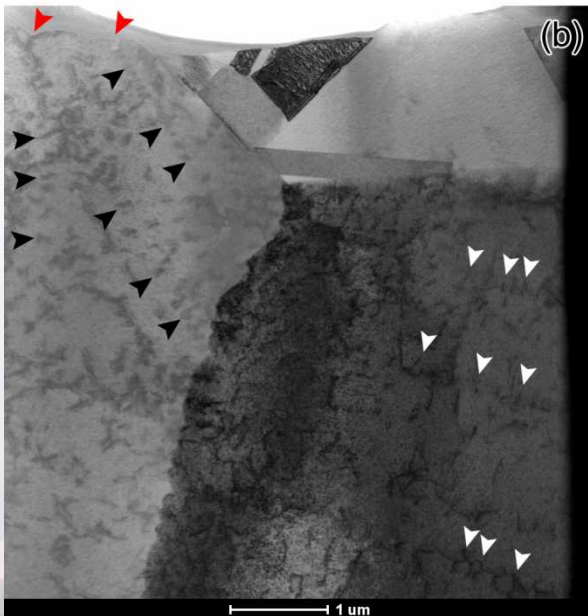
In the absence of pre-existing defects, fatigue crack nucleation occurs by a persistent slip band process



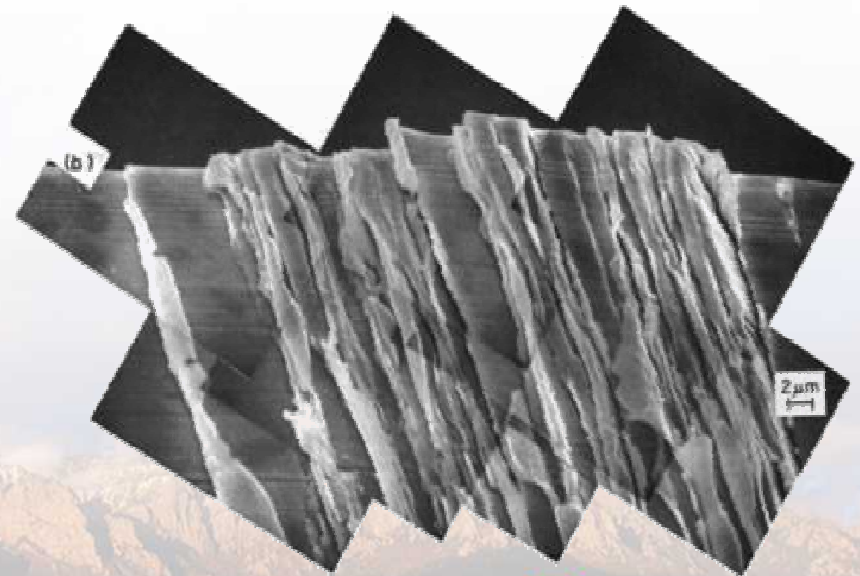
S. Suresh, Fatigue of Materials



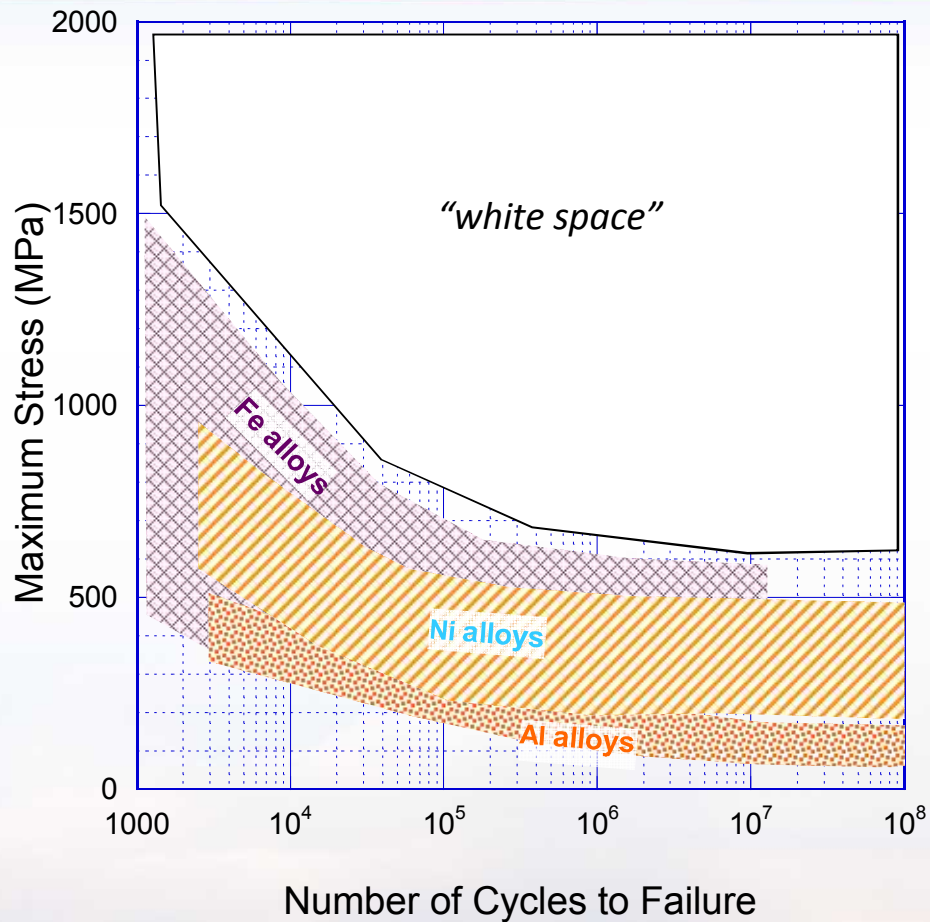
Ma and Laird, 1989



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



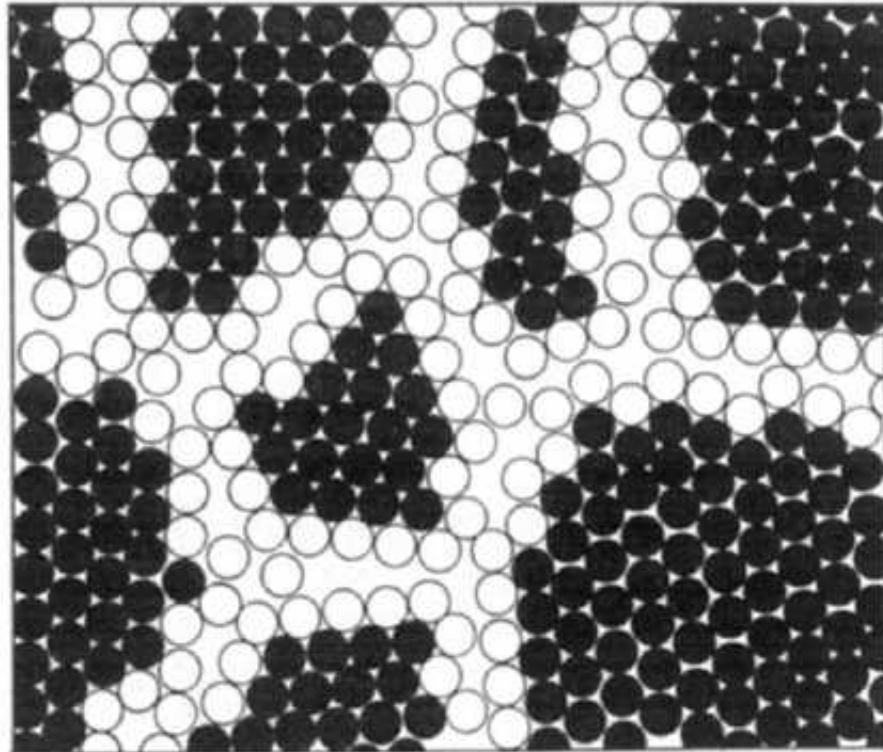
Fatigue Life in Engineering Alloys



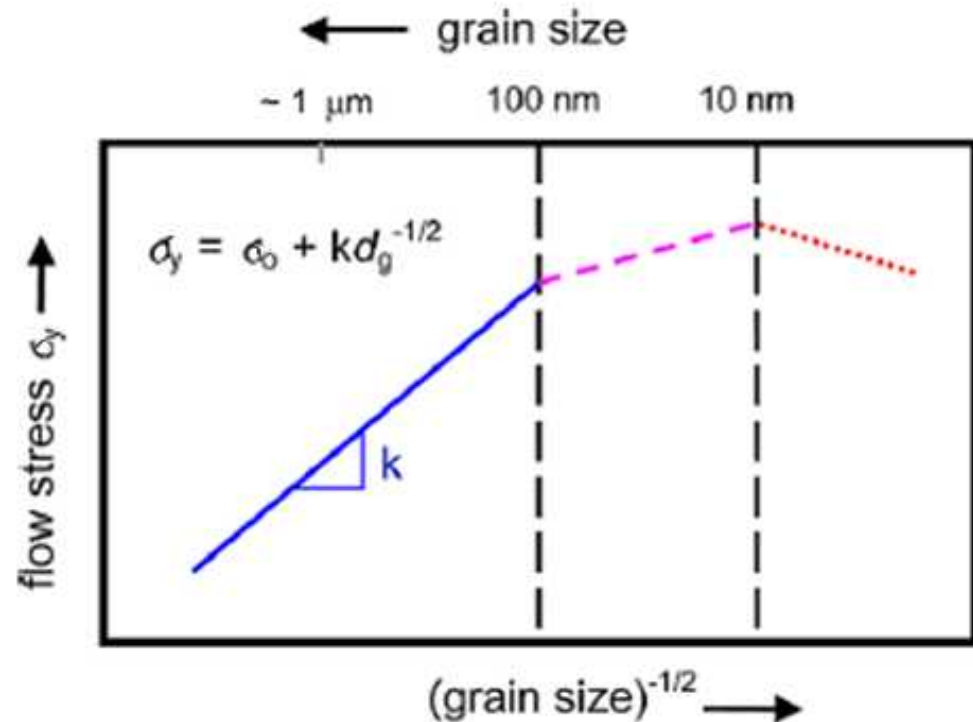
Nanocrystalline metals possess excellent strength

1989
Gleiter
Nanocrystalline
Materials

2003
Kumar,
VanSwygenhoven
Suresh
Hall-Petch Breakdown



>2000 citations



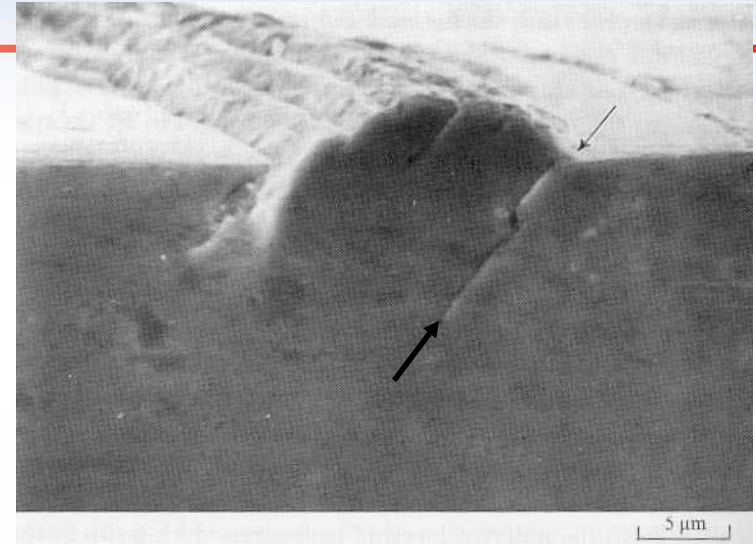
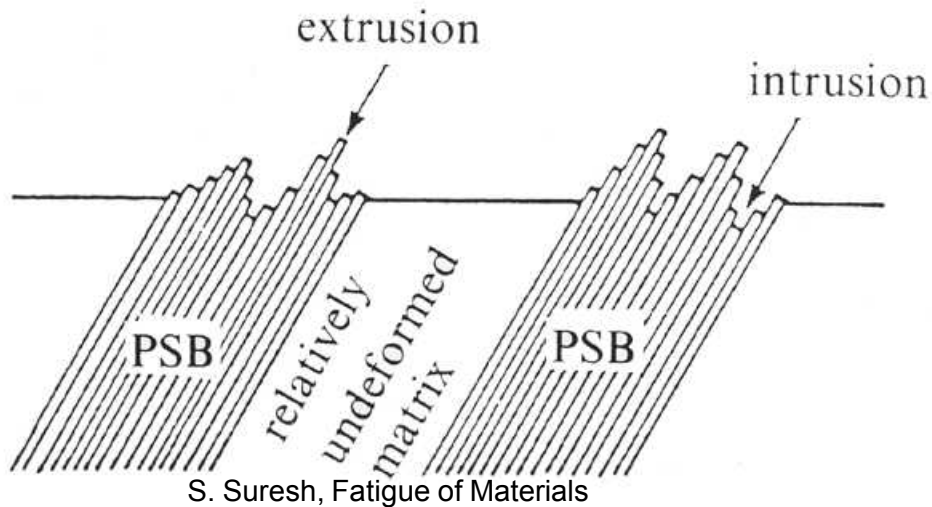
>400 citations

...what about their fatigue resistance???

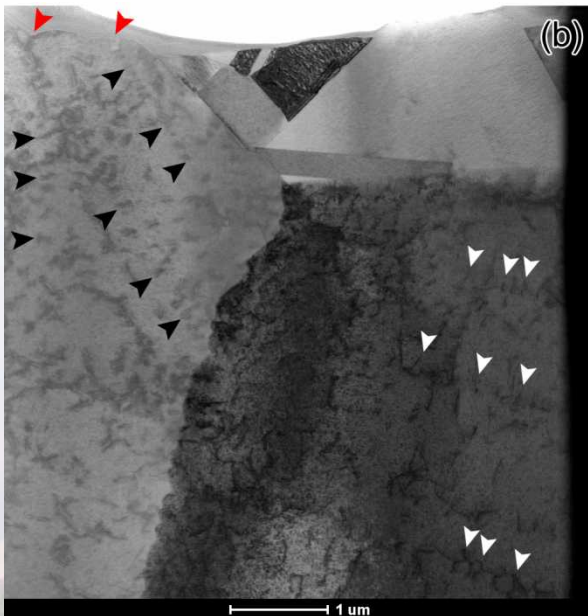


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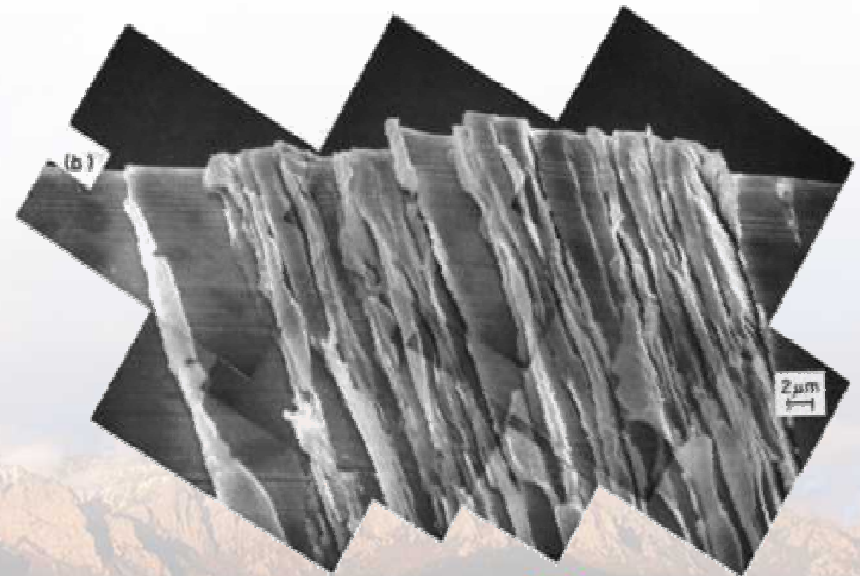
Hypothesis: the small grain size of nanocrystalline *metals* suppresses persistent slip crack initiation processes



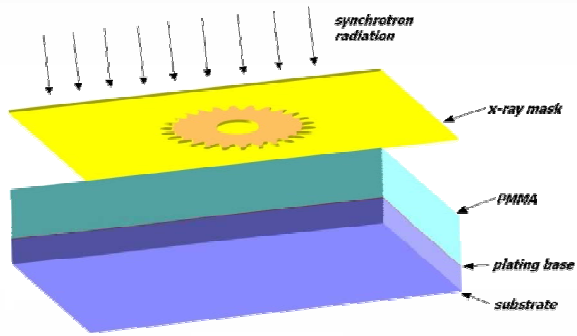
Ma and Laird, 1989



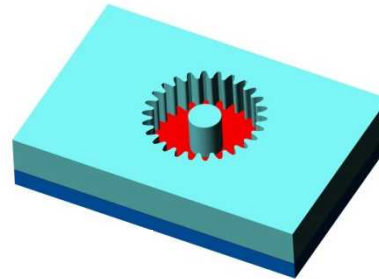
Boyce & Padilla, Metall. Mater. Trans. A, 2011



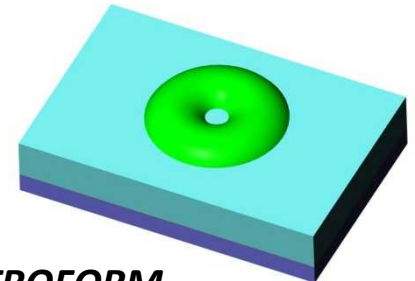
The Direct LIGA Process for Metallic Microdevices



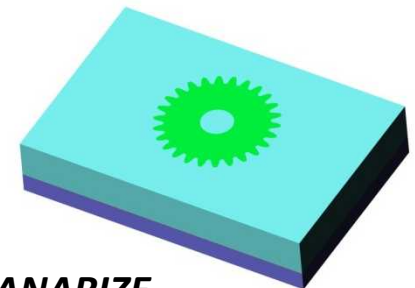
1) EXPOSE



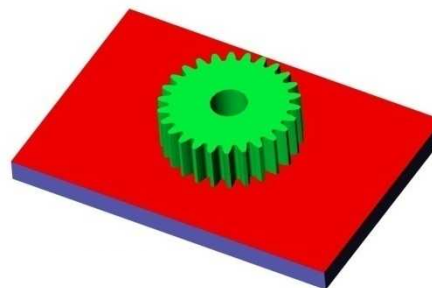
2) DEVELOP



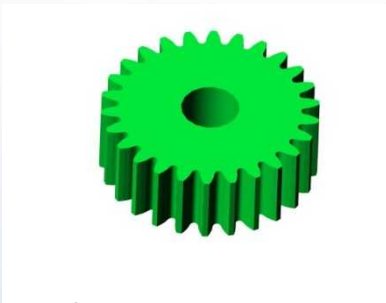
3) ELECTROFORM



4) PLANARIZE



5) REMOVE MOLD

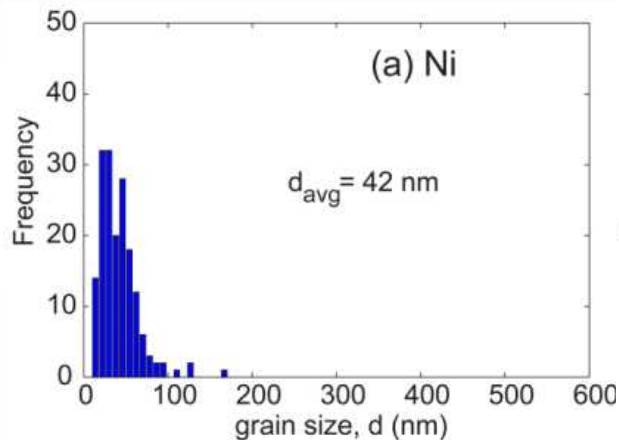


6) RELEASE



Three Nanocrystalline Ni-Based Alloys

Ni

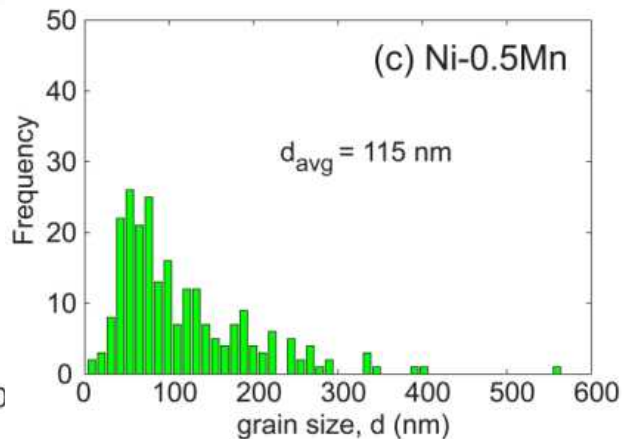


impurities

Yield ~ 1200 MPa

*$\langle 200 \rangle + \langle 111 \rangle$ very
weak texture*

Ni-0.5Mn

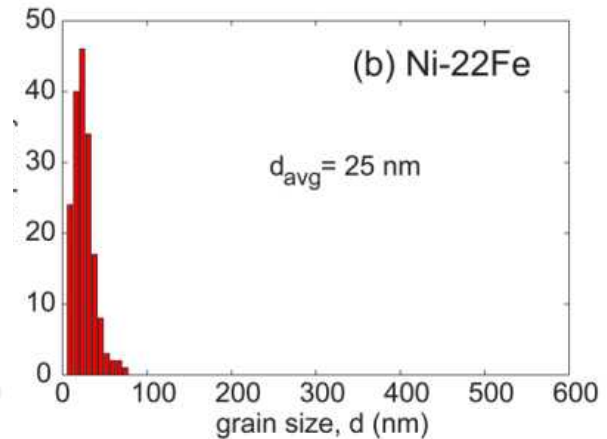


nanoprecipitates

Yield ~ 1200 MPa

*$\langle 220 \rangle$
texture*

Ni-22Fe



high solute content

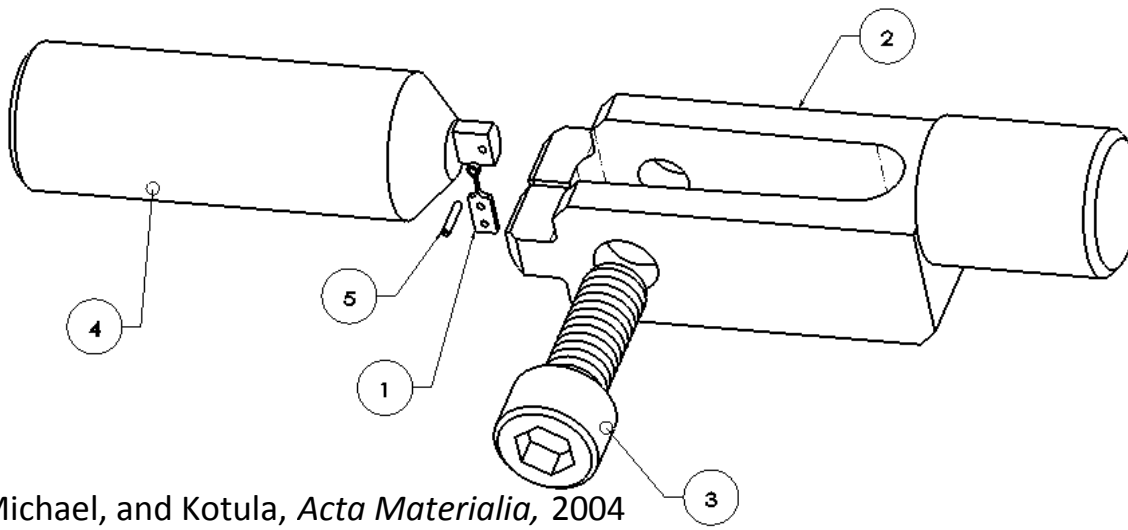
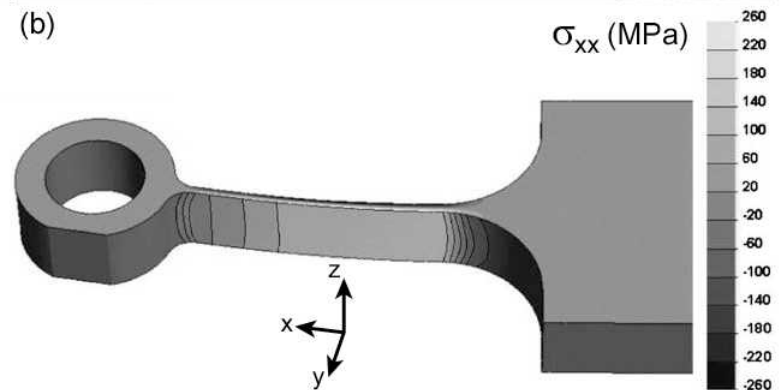
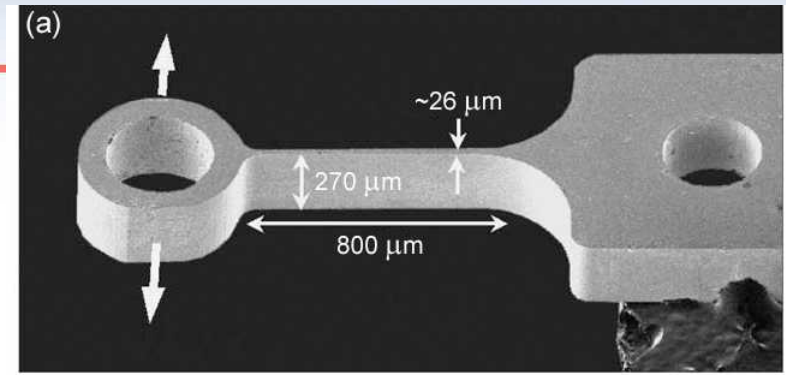
Yield ~ 1400 MPa

*$\langle 200 \rangle + \langle 111 \rangle$
texture*

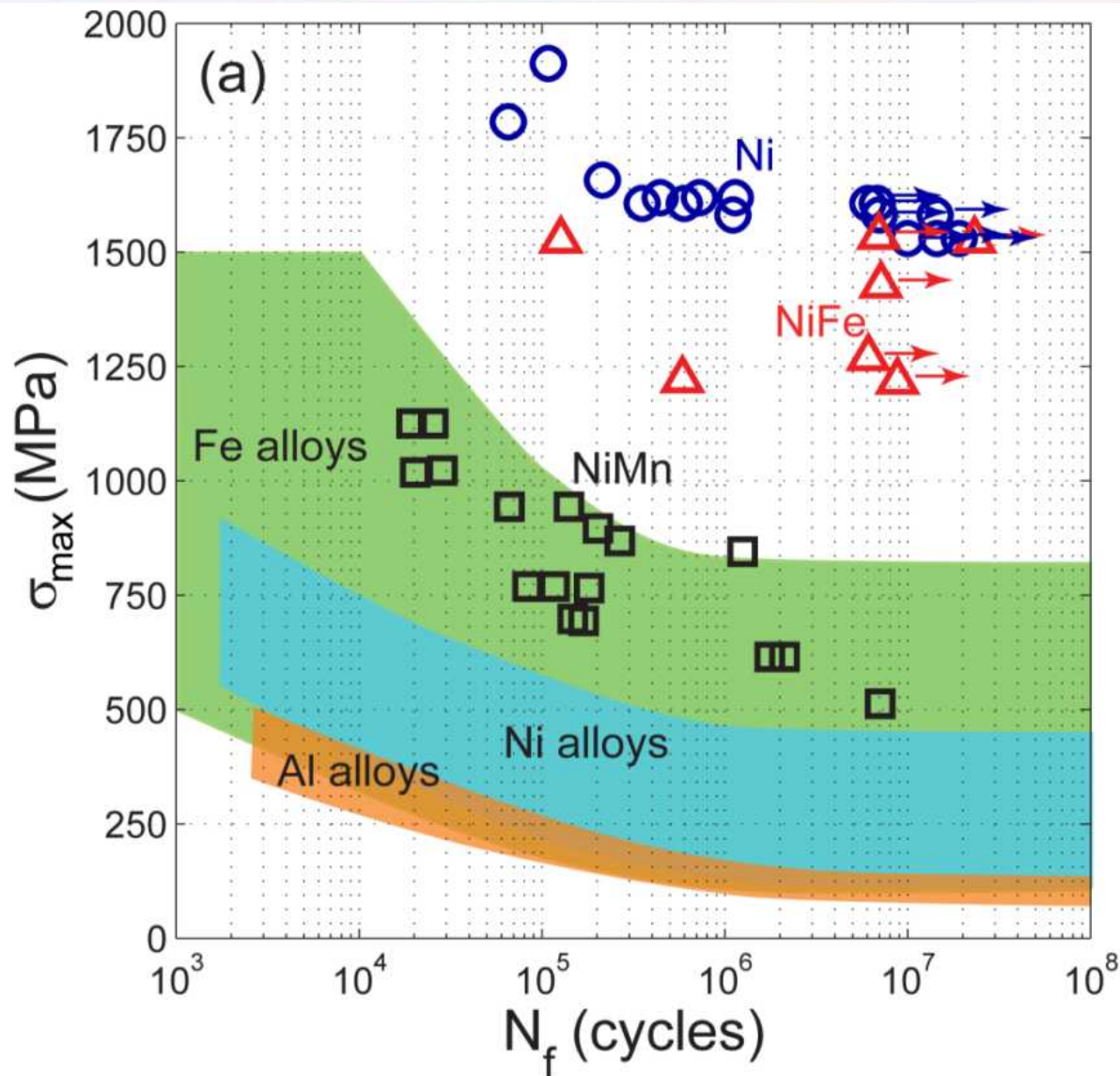


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LIGA Fatigue Testing



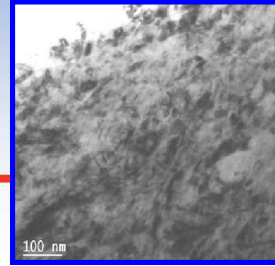
Fatigue Lifetime Results



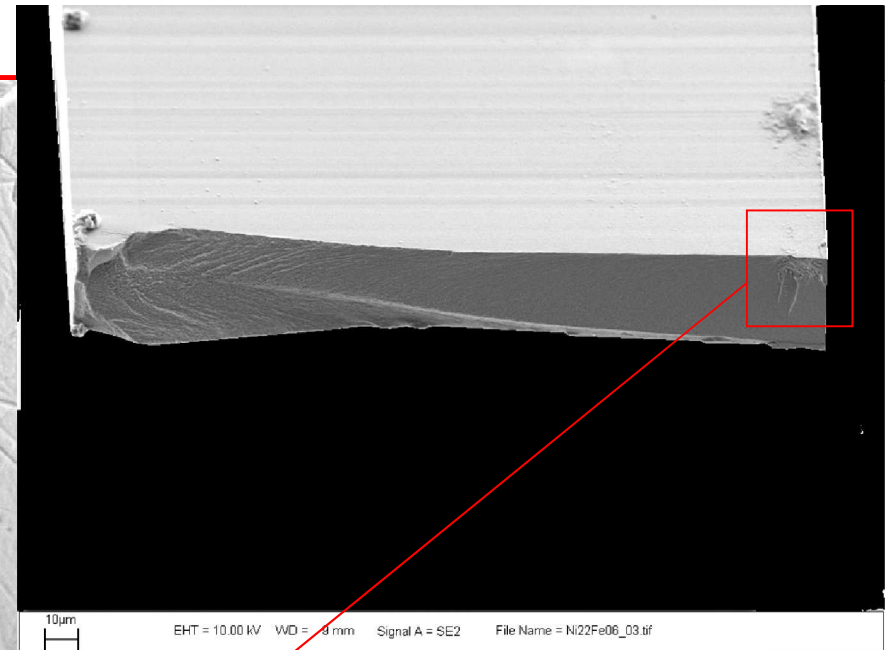
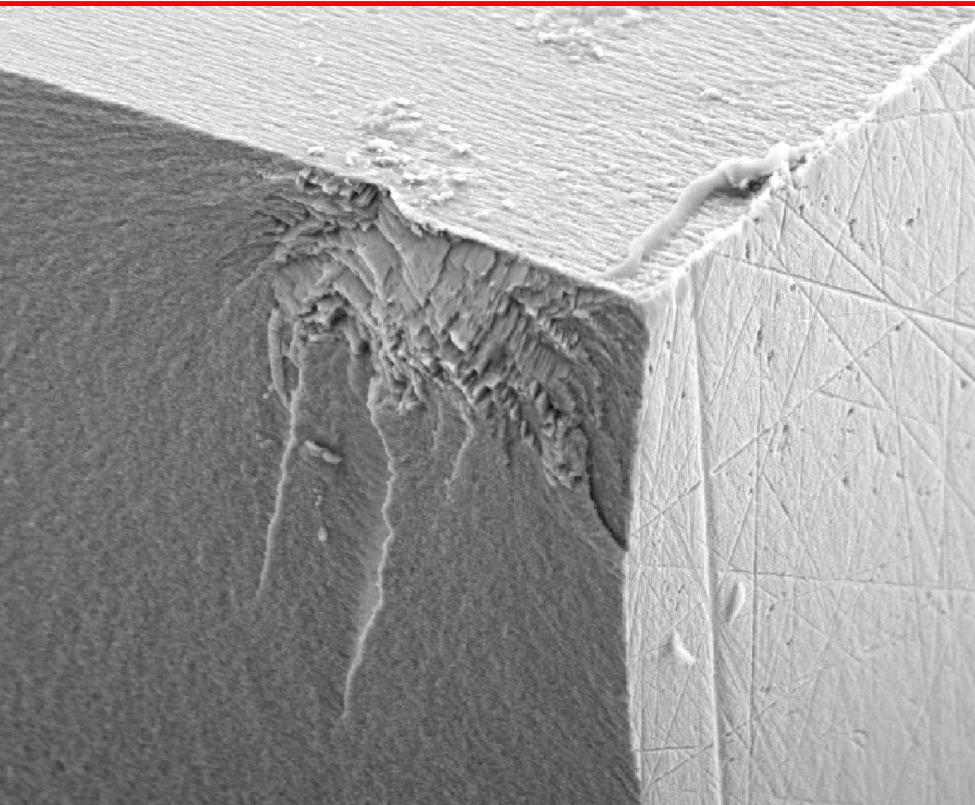
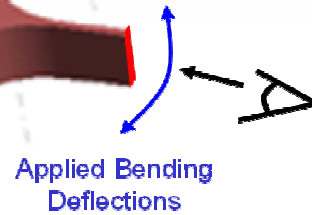
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Unusual Initiation Zone In Nanocrystalline Ni-Fe Alloy

NiFe

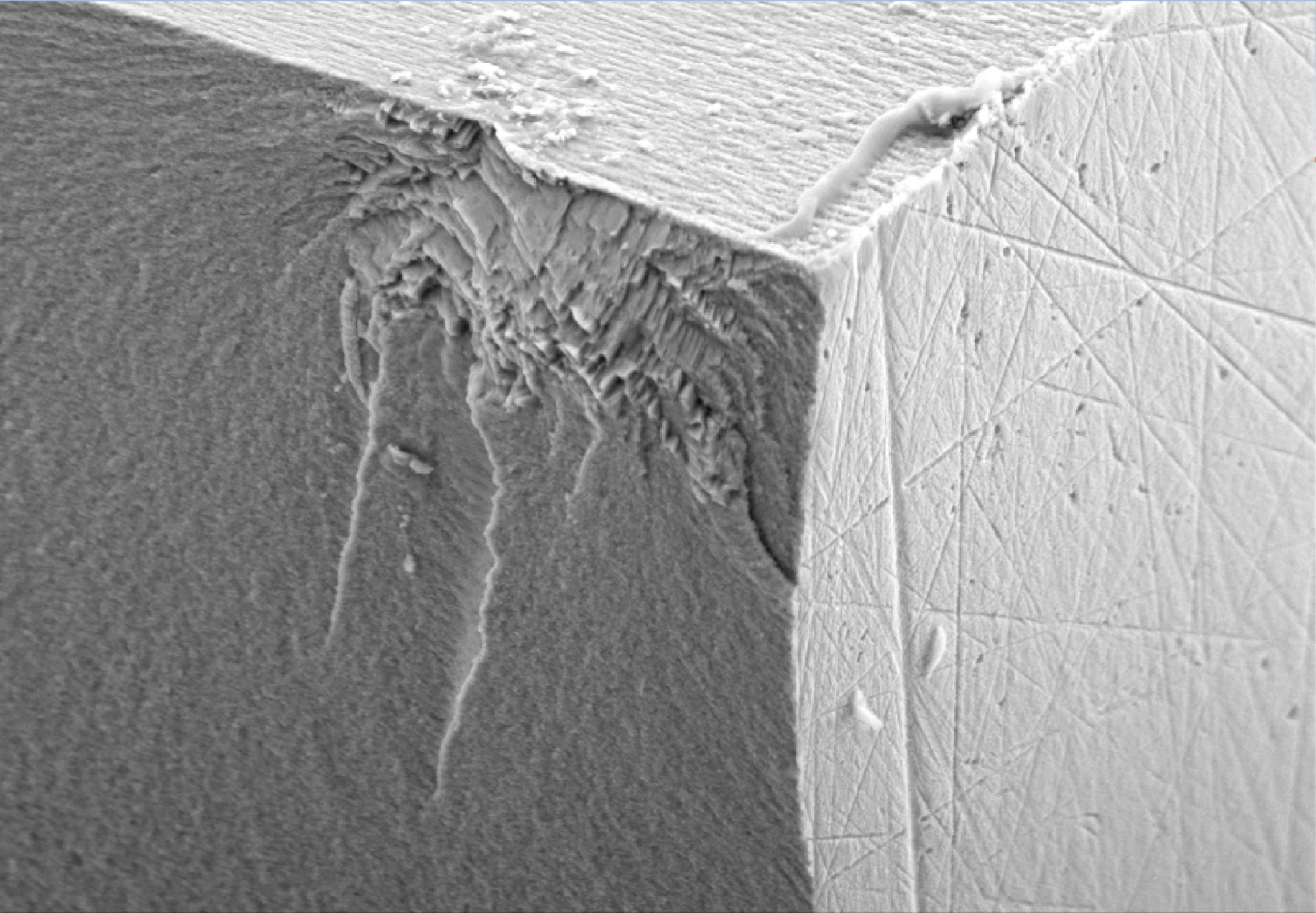


Broken Fatigue Specimen



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EHT = 10.00 kV WD = 8 mm Signal A = SE2 File Name = Ni22Fe08_05.tif

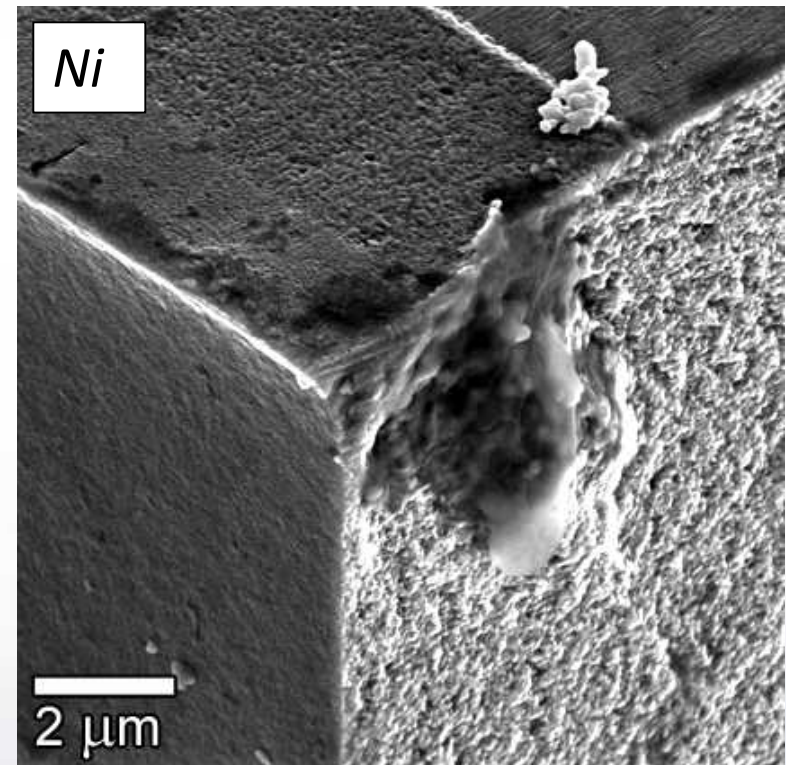
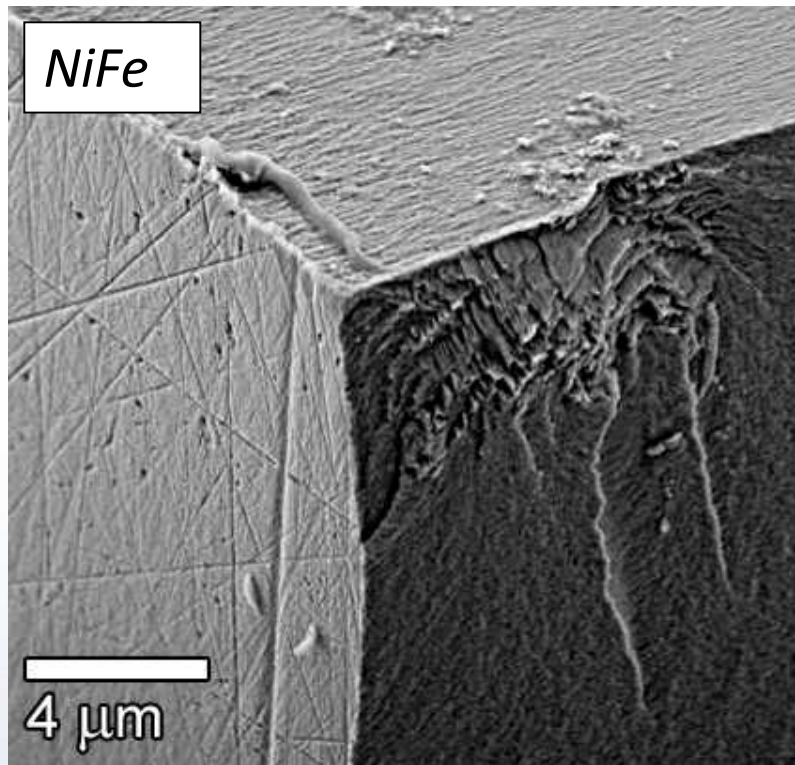
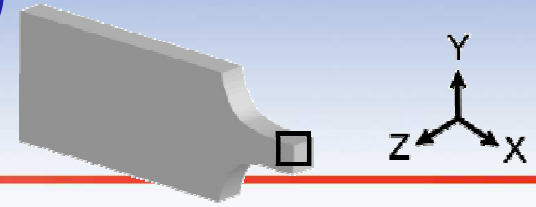


1 μ m
H

EHT = 10.00 kV WD = 9 mm Signal A = SE2

File Name = Ni22Fe06_05.tif

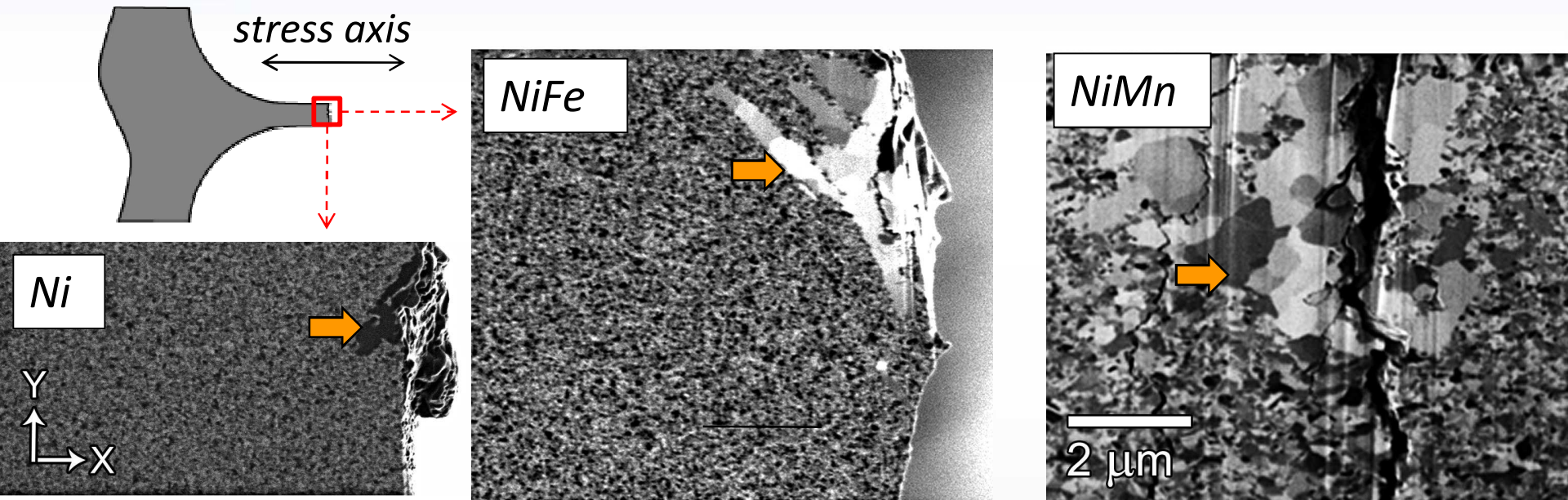
Typical Fracture Surface (SEM)



- Initiation zone marked by large blocky features
- Propagation zone morphology follows NC microstructure



Microstructural Analysis (FIB)

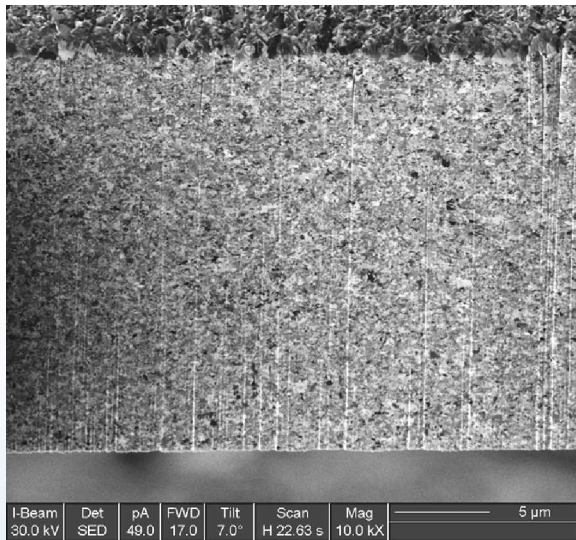


- Fatigue-induced coarsened zone *at room temperature!*
- Coarsened zone only forms locally (abnormal grain growth) in regions of maximum stress, prior to crack initiation
- No coarse grains found in as-deposited samples
- No coarse grains found in monotonically stressed samples

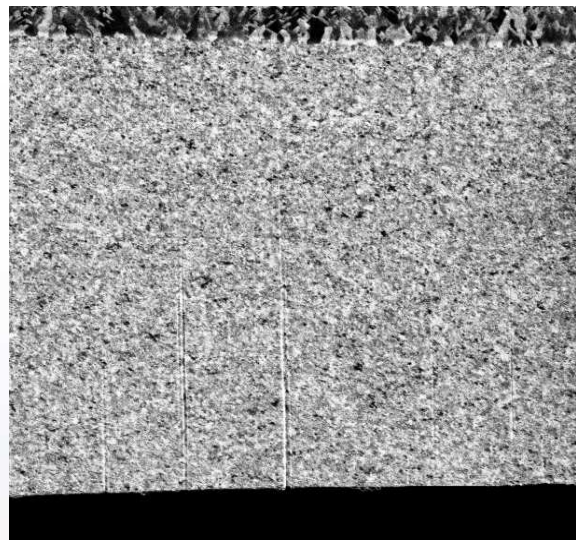


Grain growth is induced by fatigue

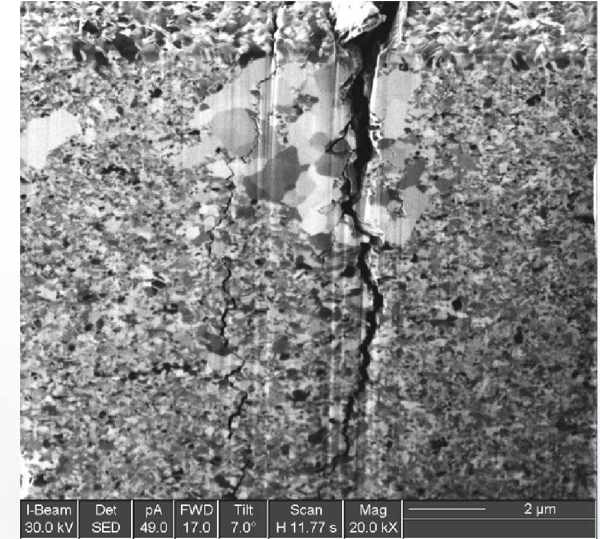
As-Deposited



After 1 Cycle

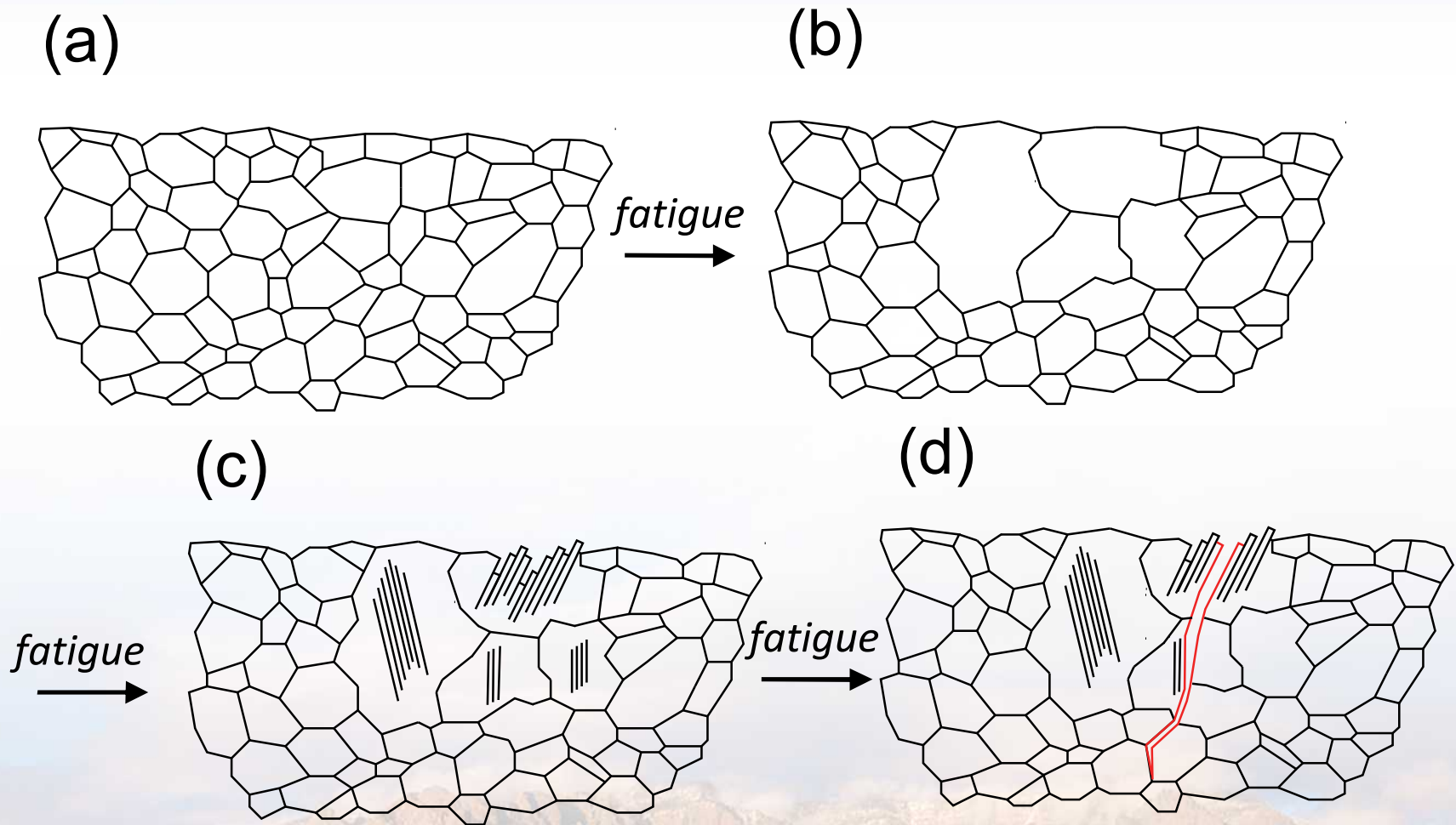


After ~100,000 Cycles



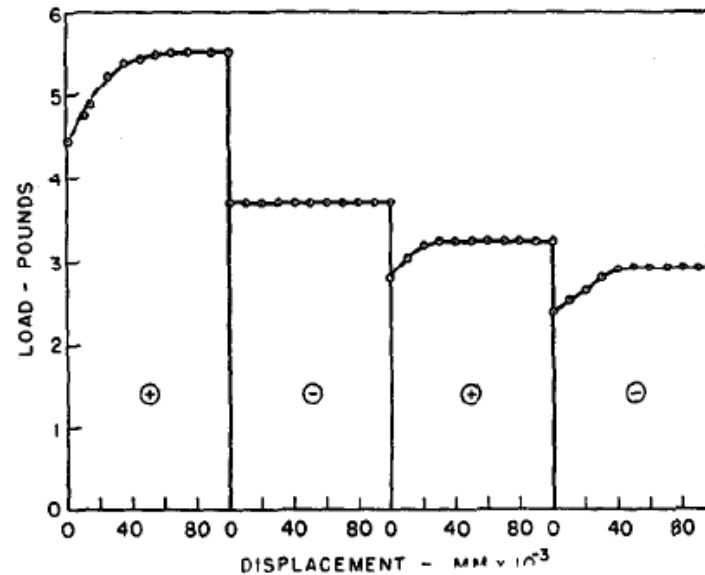
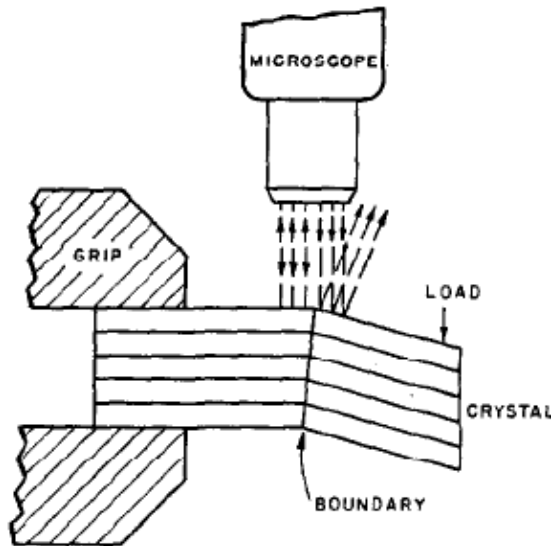
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New Fatigue Process in Nanocrystalline Ni Alloys



Mechanically-Induced Grain Boundary Motion

2° LAGB in 99.99% Zinc

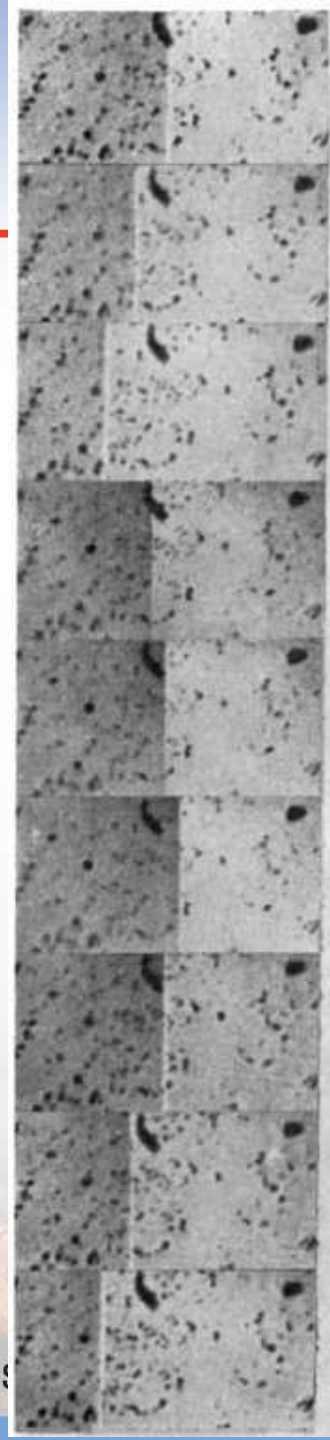


Li, Edwards, Washburn, and Parker, *Acta Metall.*, 1953

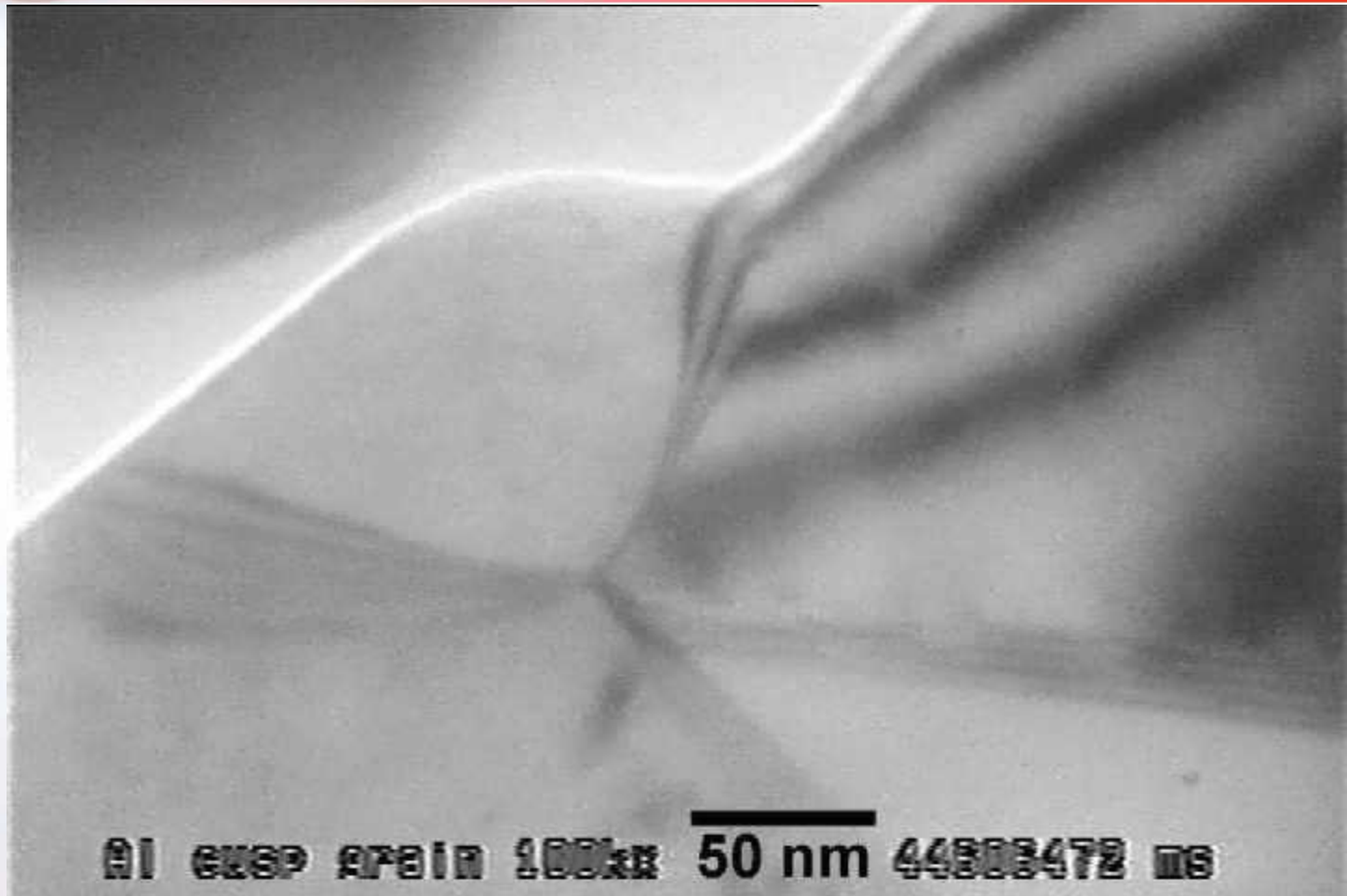
Recent Theoretical Models for Elastic Stress or Strain-Driven Grain Growth:

Cahn and Taylor, *Acta Materialia*, 52, 2004

Gutkin and Ovid'ko, *Applied Physics Letters* 85, 2005



Can Stress Really Move Grain Boundaries?



A. Minor, et al, National Center for Electron Microscopy, Lawrence Berkeley National Laboratory



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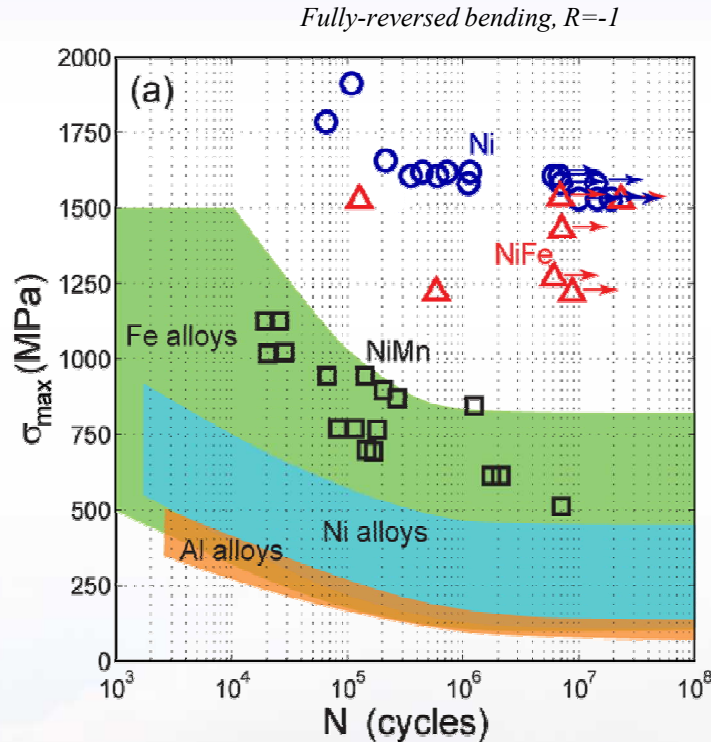
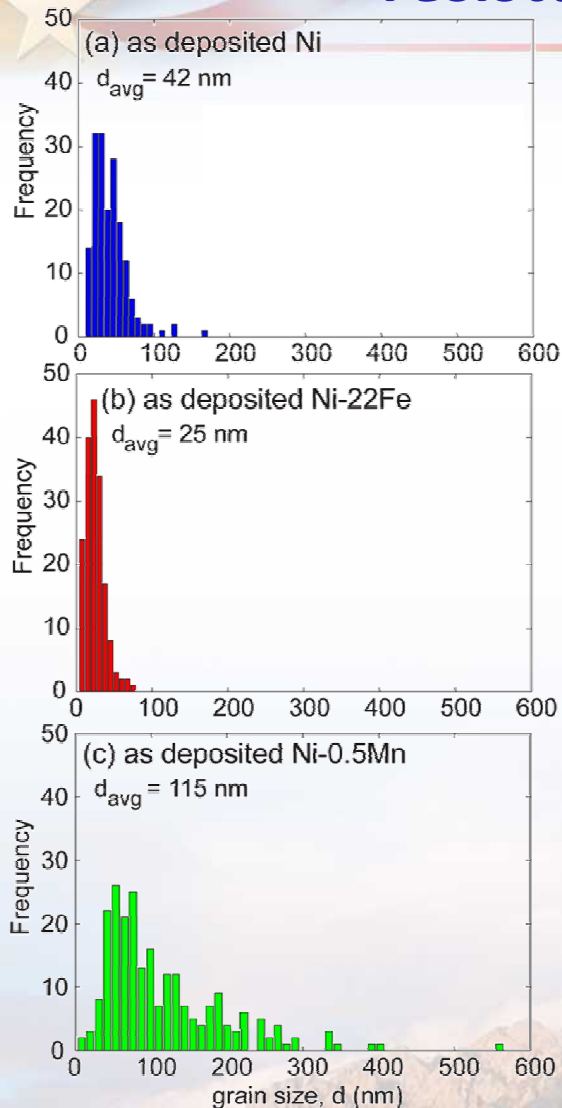
Summary so far: Fatigue of Nanocrystalline Ni alloys

Three nanocrystalline Ni alloys exhibit exceptional fatigue resistance, apparently due to the suppression of traditional dislocation-mediated crack initiation processes.

Only when nanocrystalline Ni is driven to undergo grain-growth does fatigue-crack initiation ensue.



A Question Left Unanswered... How does fatigue resistance scale with grain size?



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

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





Can we systematically vary grain size in a single alloy to see a fatigue-equivalent of Hall-Petch Breakdown?



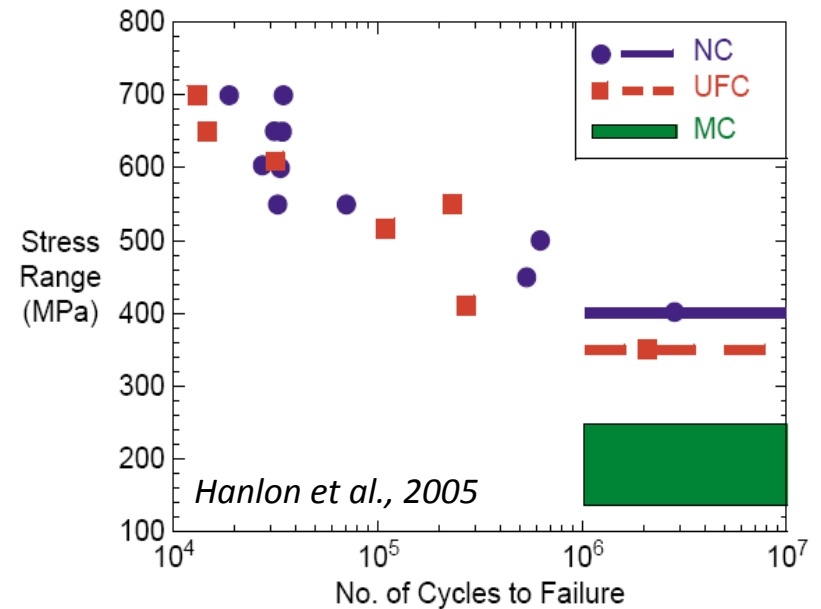
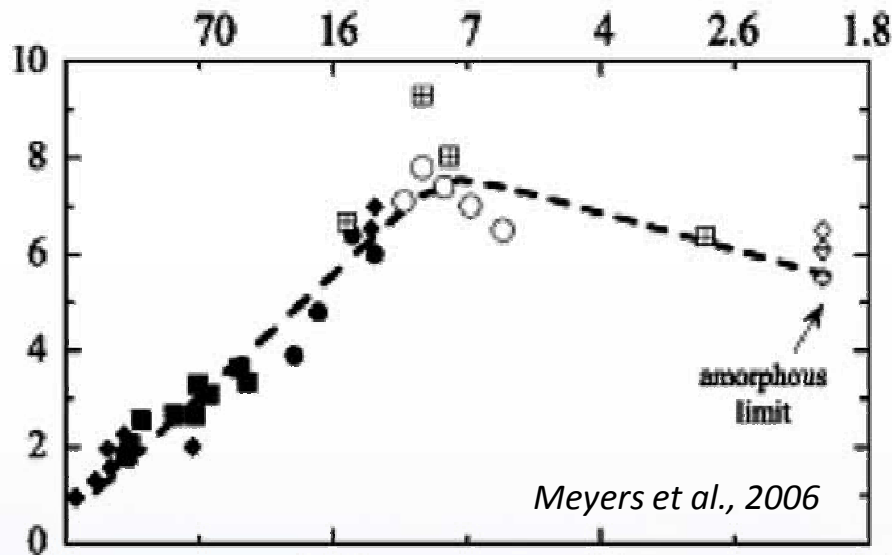
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Hall-Petch Grain Size Effects

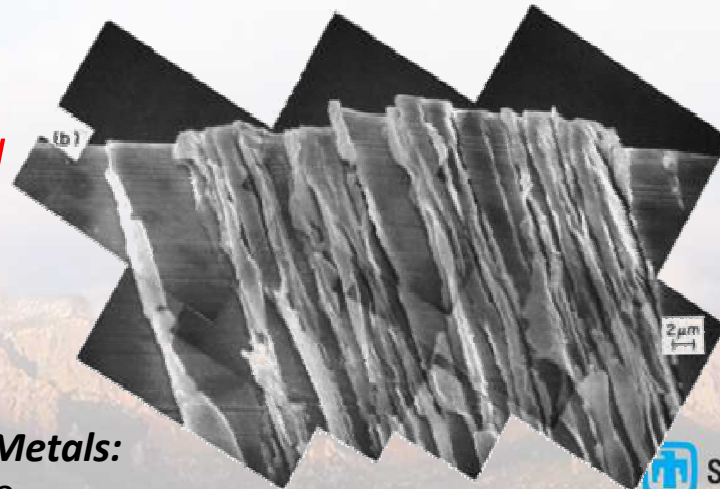
Material Hardness (or Strength)

Material Grain Size

d [nm]



At what length scale do conventional fatigue processes (i.e. persistent slip) begin to break down?



Ma and Laird, 1989



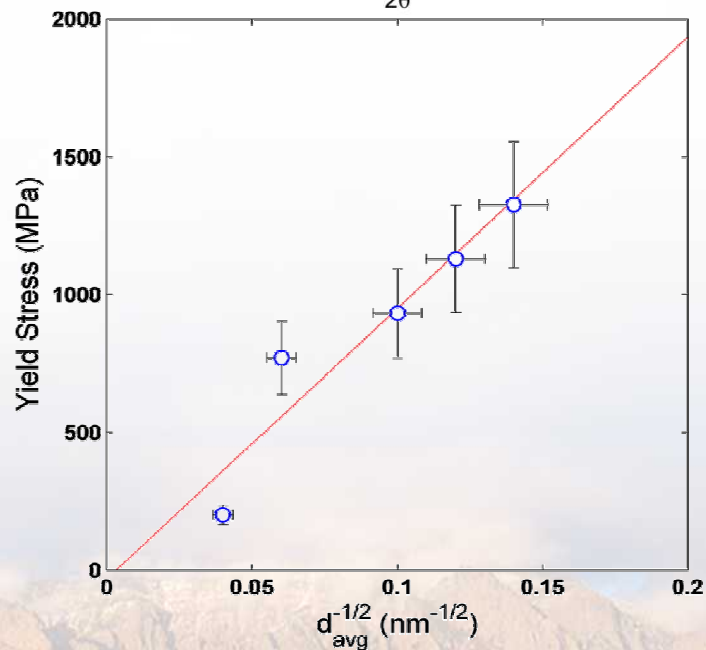
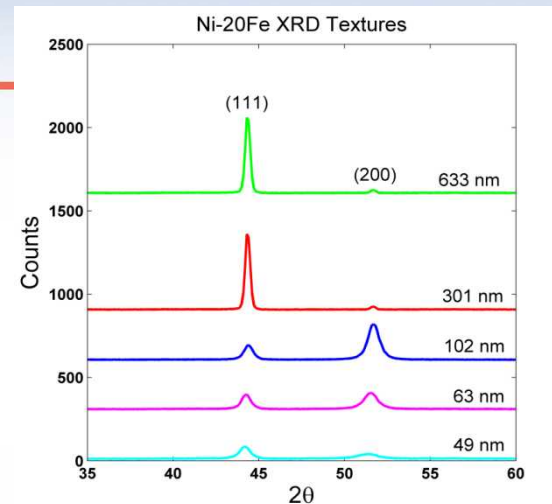
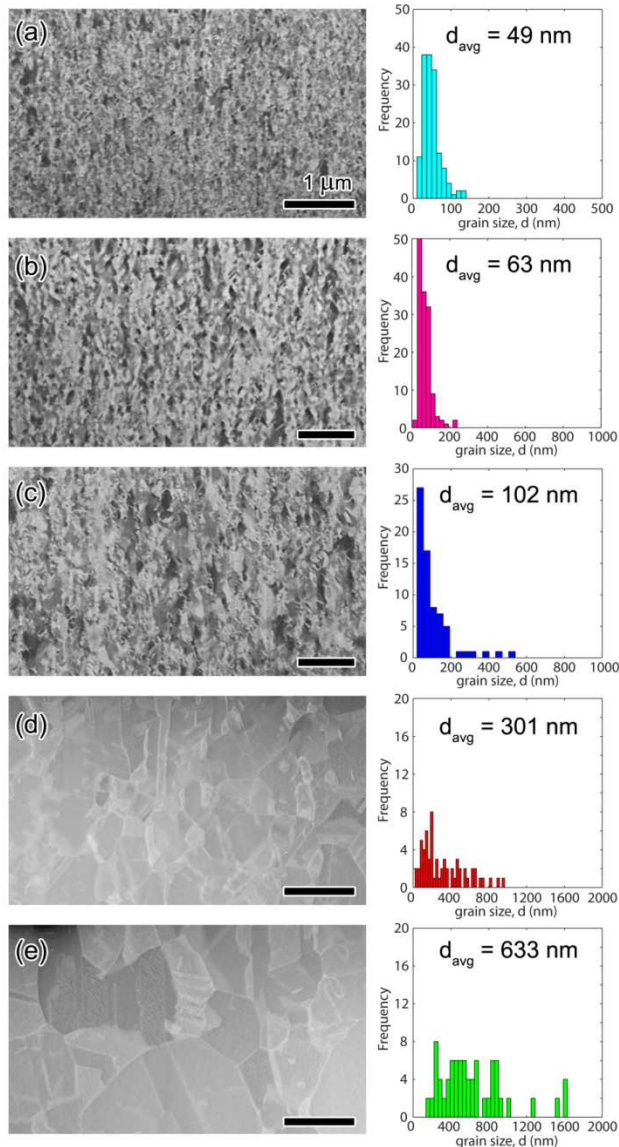
Review of Fatigue Behavior in Nanocrystalline Metals:

Padilla and Boyce, *Experimental Mechanics*, 2010

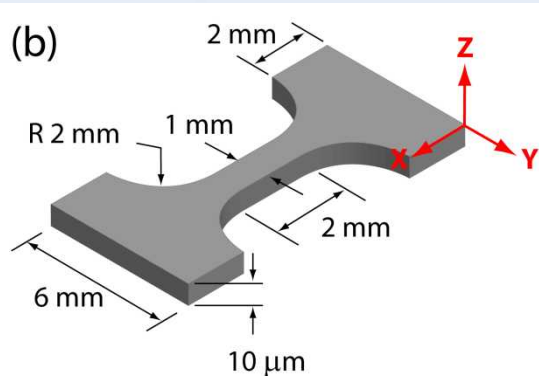
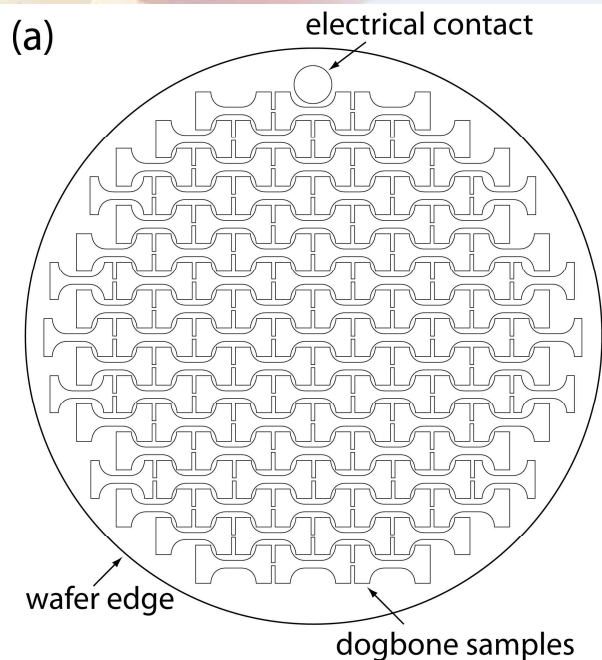


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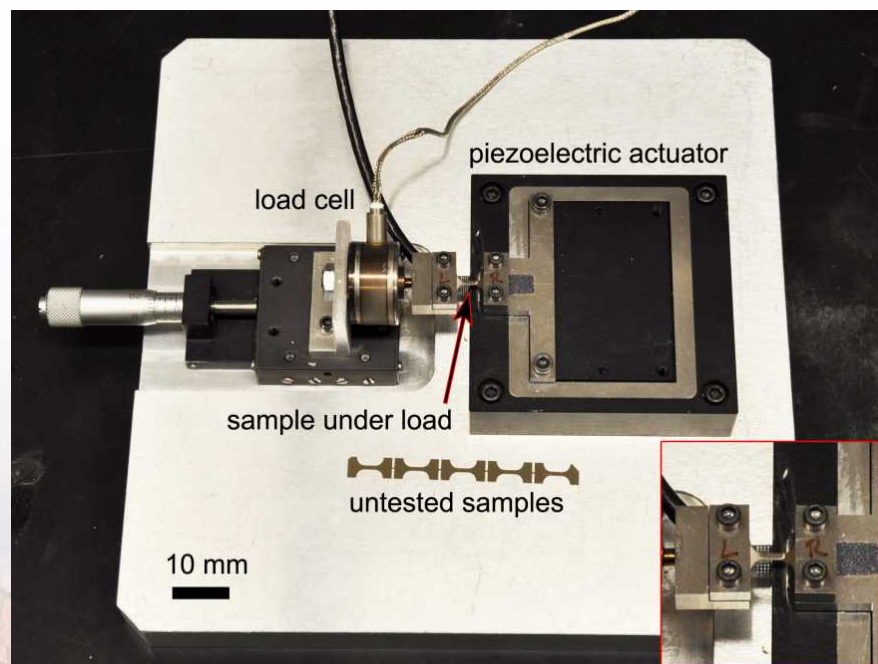
One alloy (Ni-10Fe) produced with 5 Grain Sizes



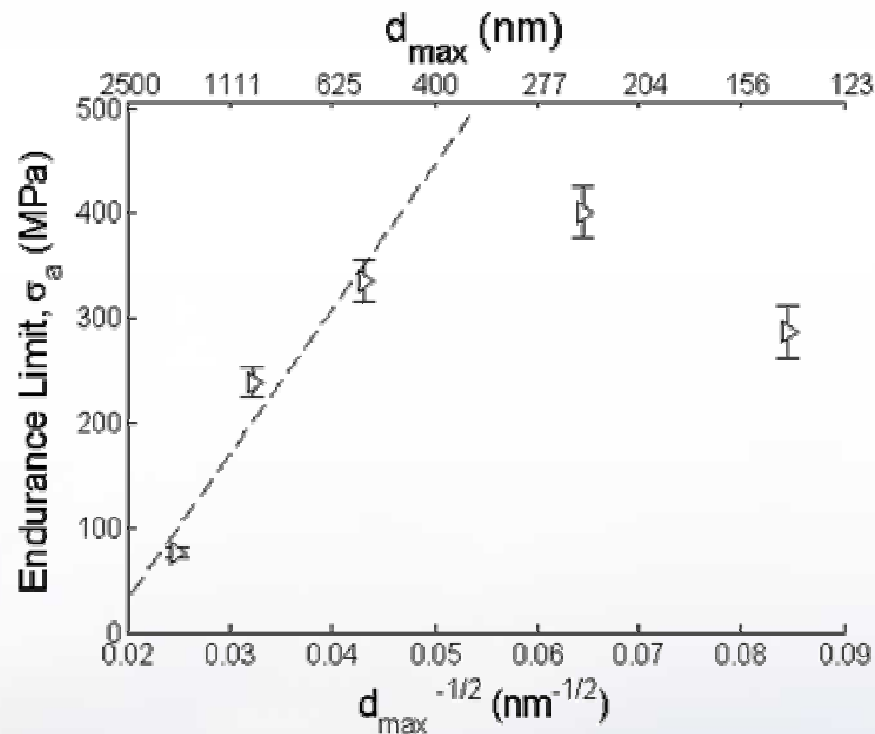
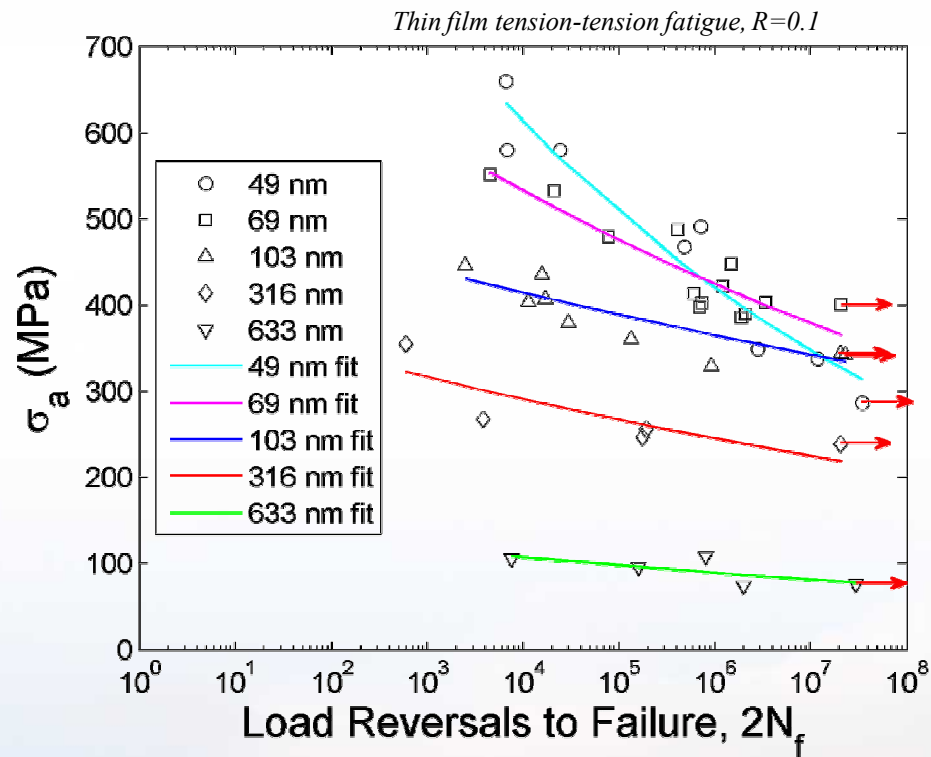
Thin film fatigue of electrodeposited Ni-Fe (permalloy)



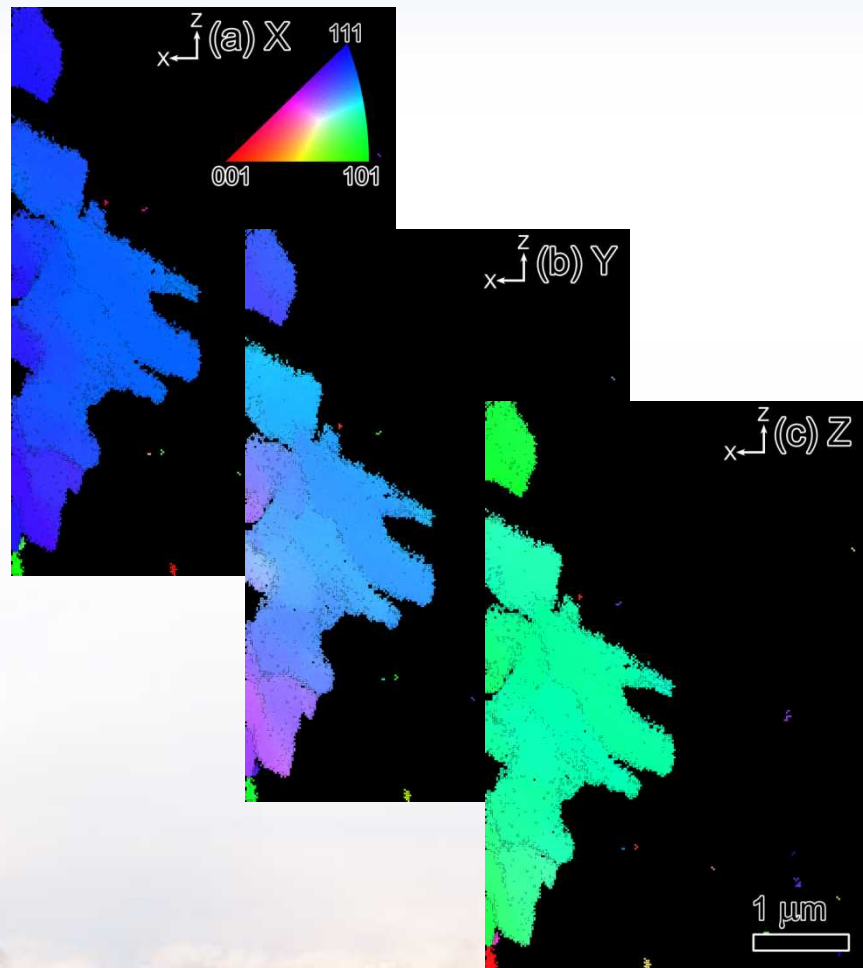
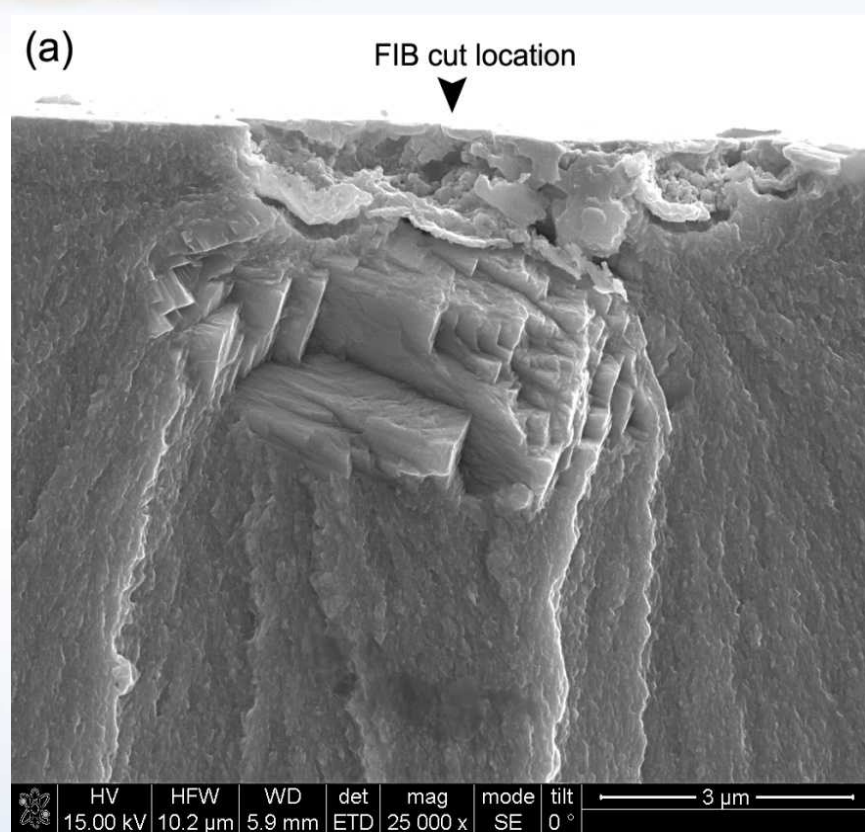
Thin film tension-tension fatigue, $R=0.1$



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



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



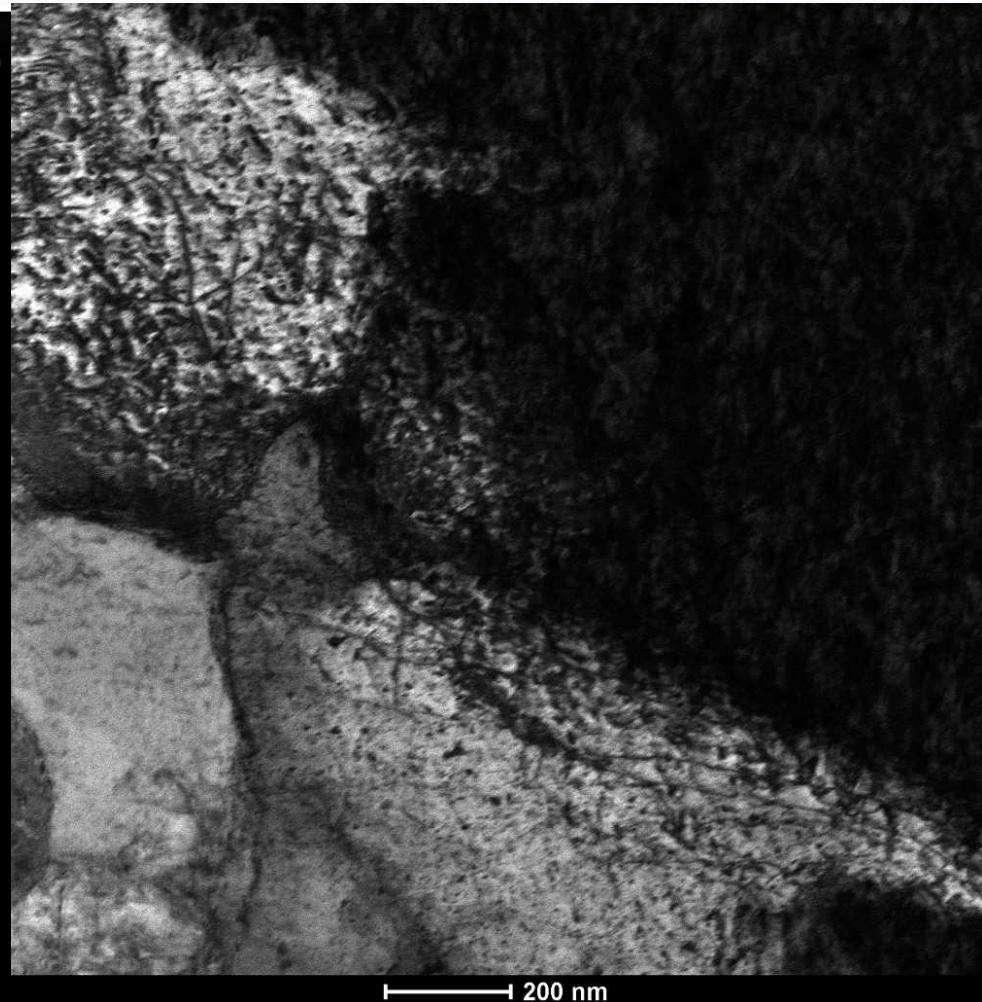
Persistent slip ladders are apparent in coarse grained metals, but have not yet been found in fatigue-coarsened grains

Coarse Grained Ni-Fe

Produced by annealing @ 650°C/1hr



Nanocrystalline Ni-Fe



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



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Can we observe the fatigue-driven grain evolution directly?



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Quantitative In-situ TEM fatigue of electron-transparent nanocrystalline films

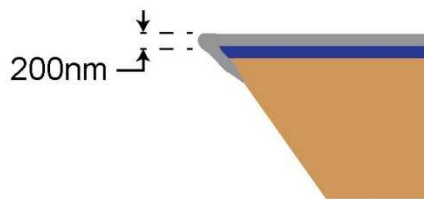
Sample Fabrication

001 Si Wafer

1. Start with Si wafer coated with nitride

2. Open window in nitride

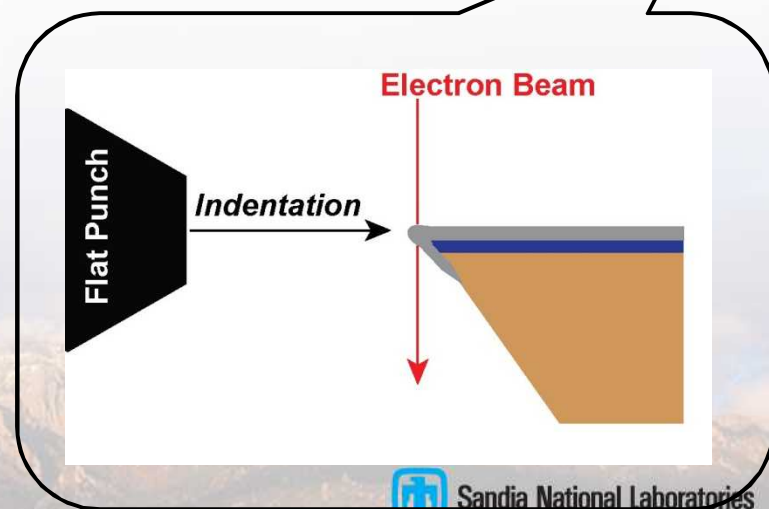
3. Anisotropic wet etch (TMAH) exposes 111 plane to create a wedge



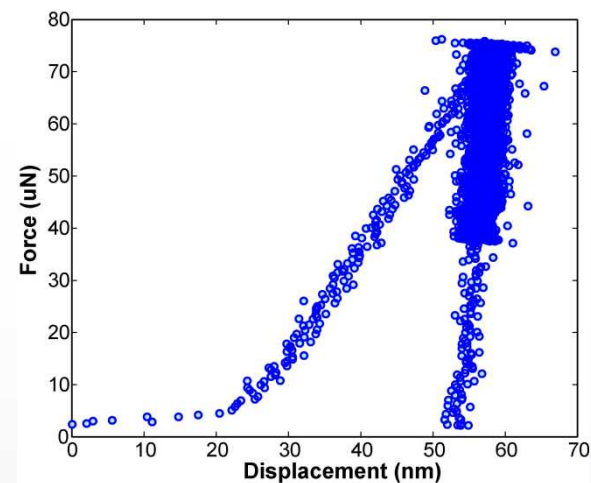
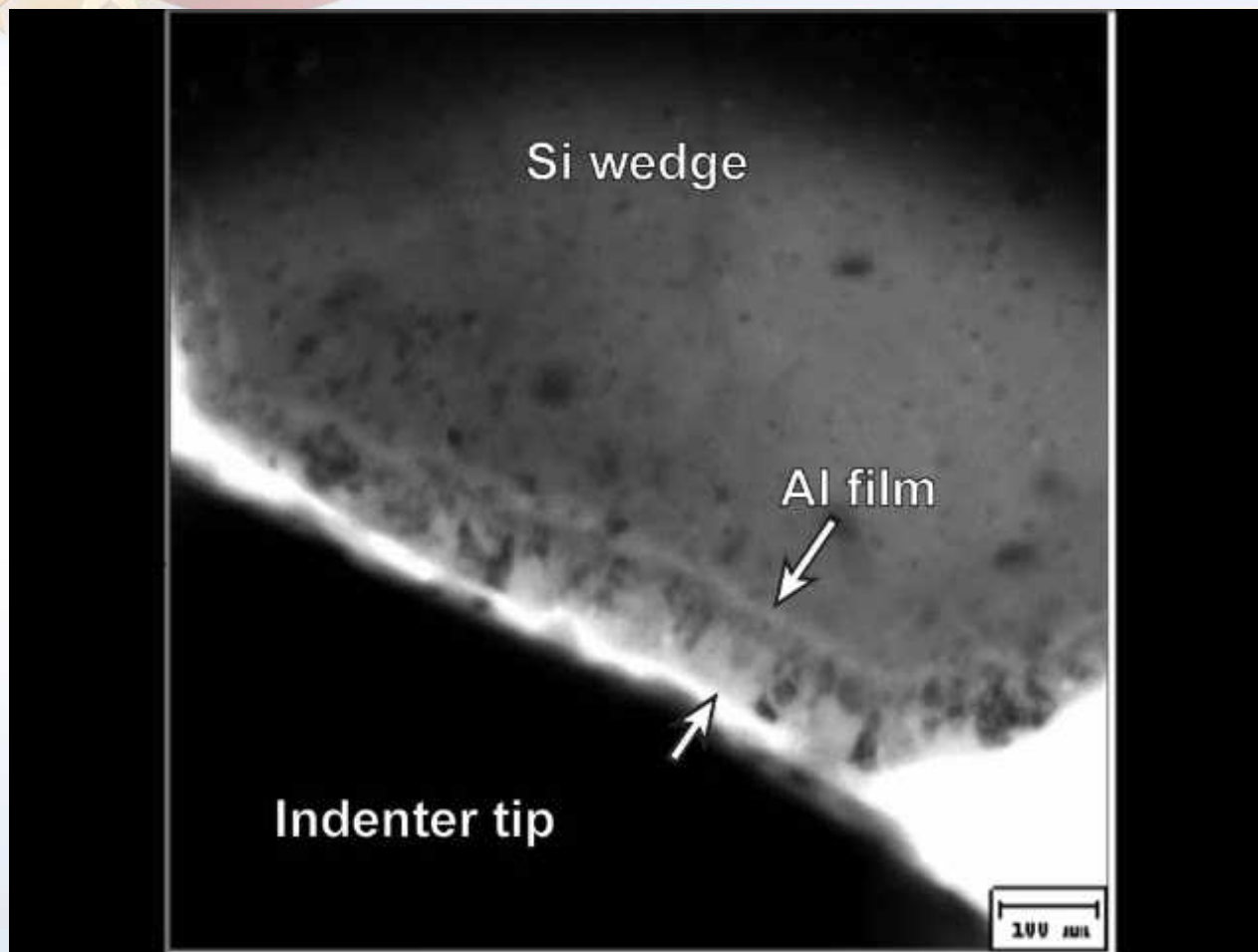
4. Pulse laser deposition of Al onto wedge

In situ TEM fatigue

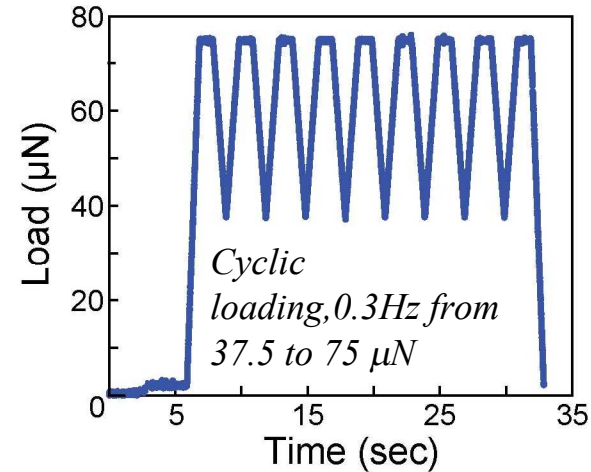
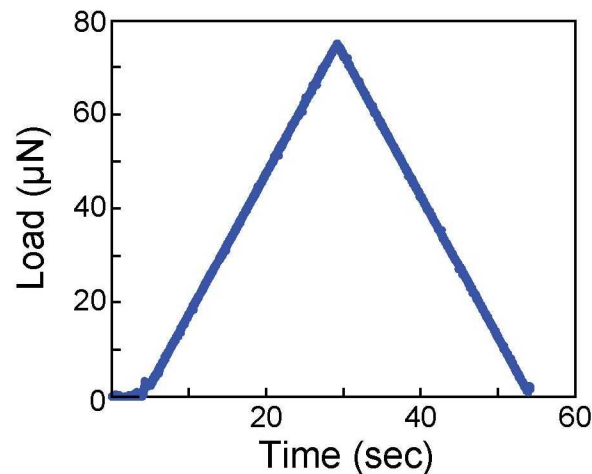
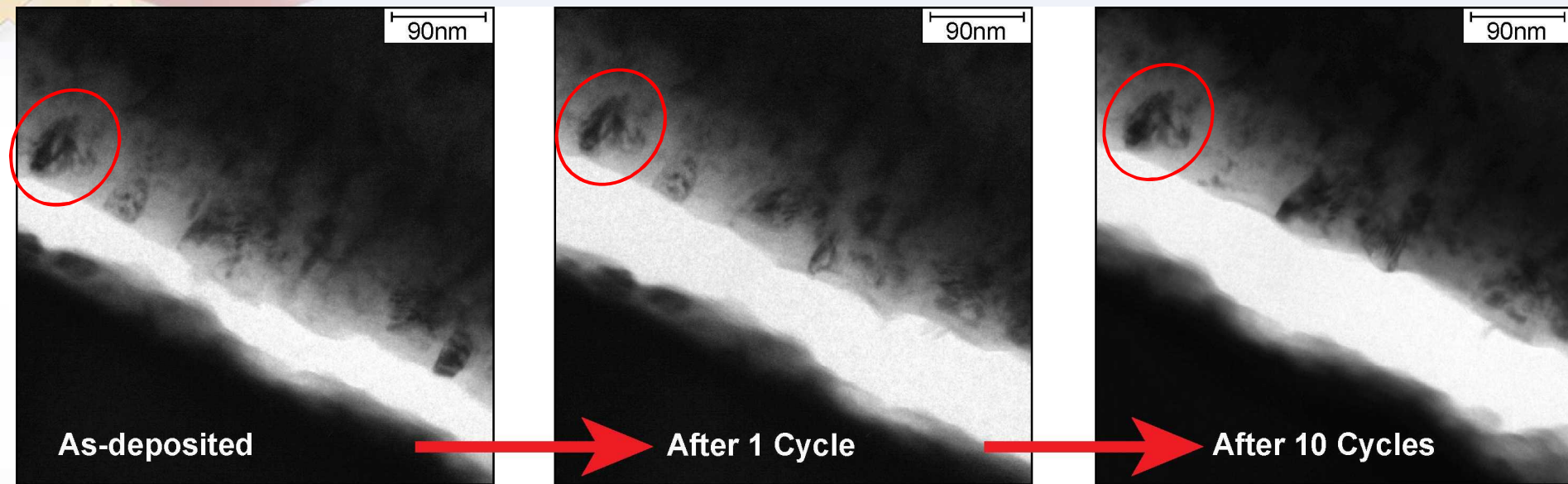
- Microscope: JEOL JEM 2100 LaB₆ TEM
- Holder: Hysitron PI-95 PicoIndenter with a 1 μm flat tip punch



In Situ TEM Indentation



Some regions show no evolution



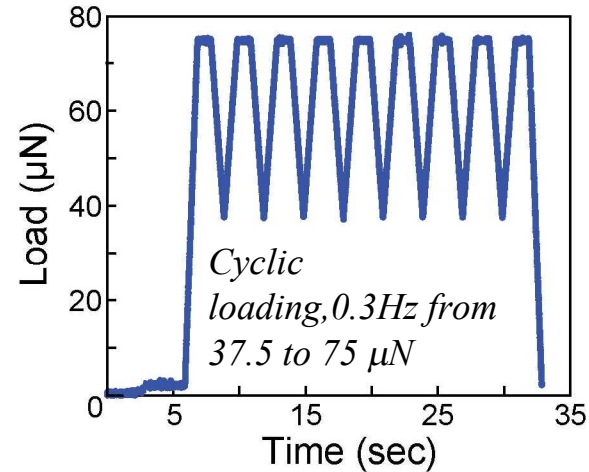
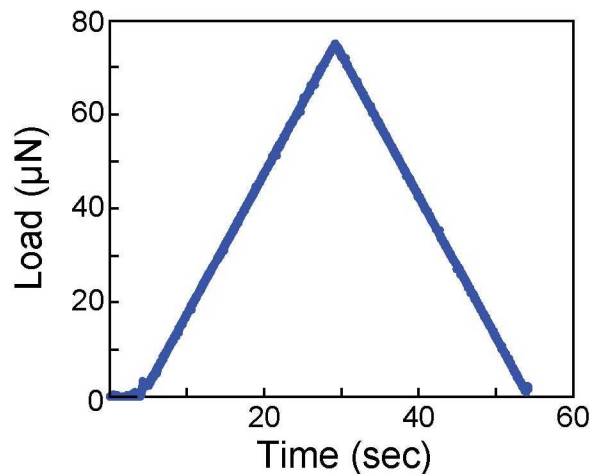
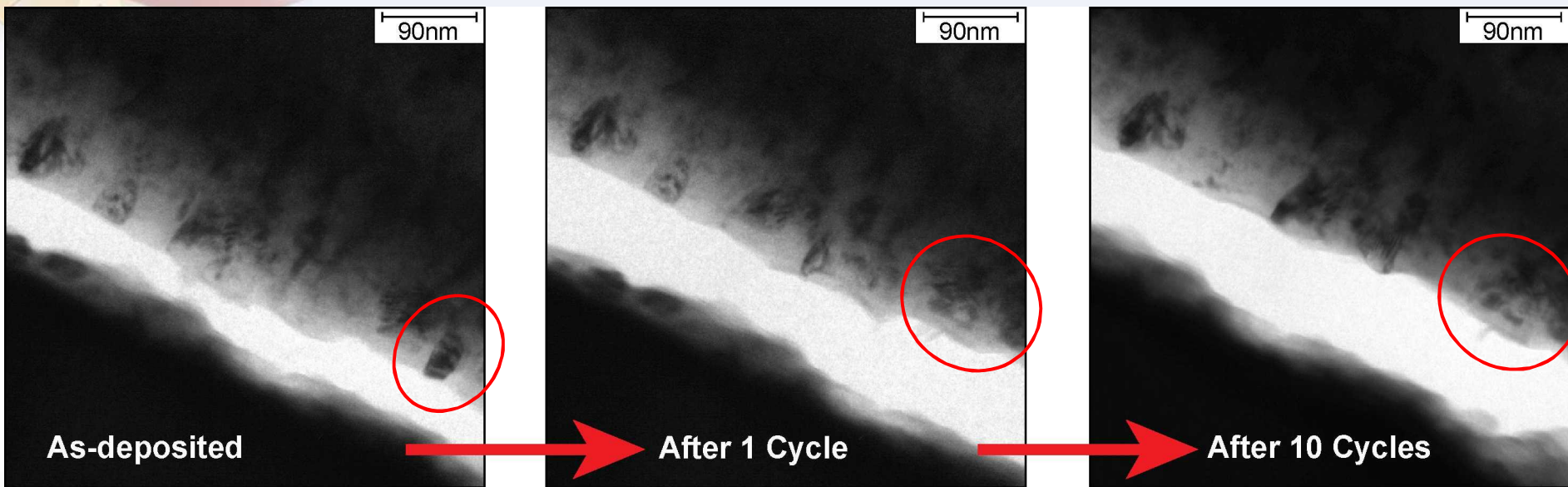
Peak load of 75 μN

375 MPa nominal contact stress



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Some regions evolve during the 1st cycle

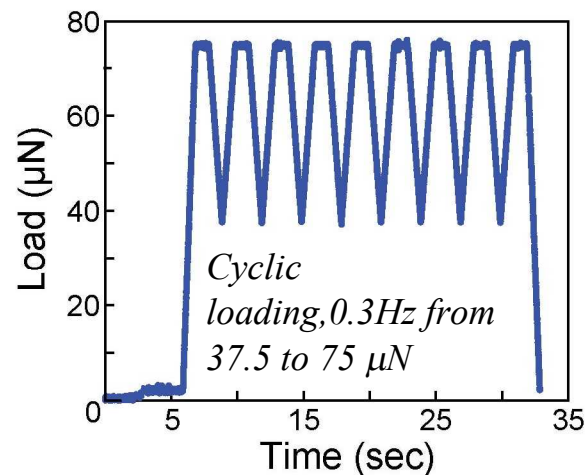
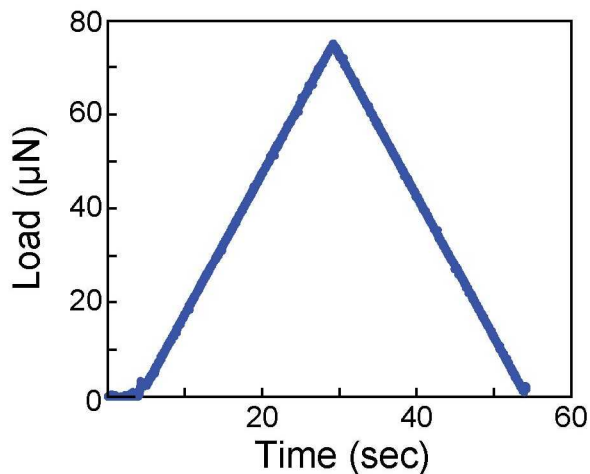
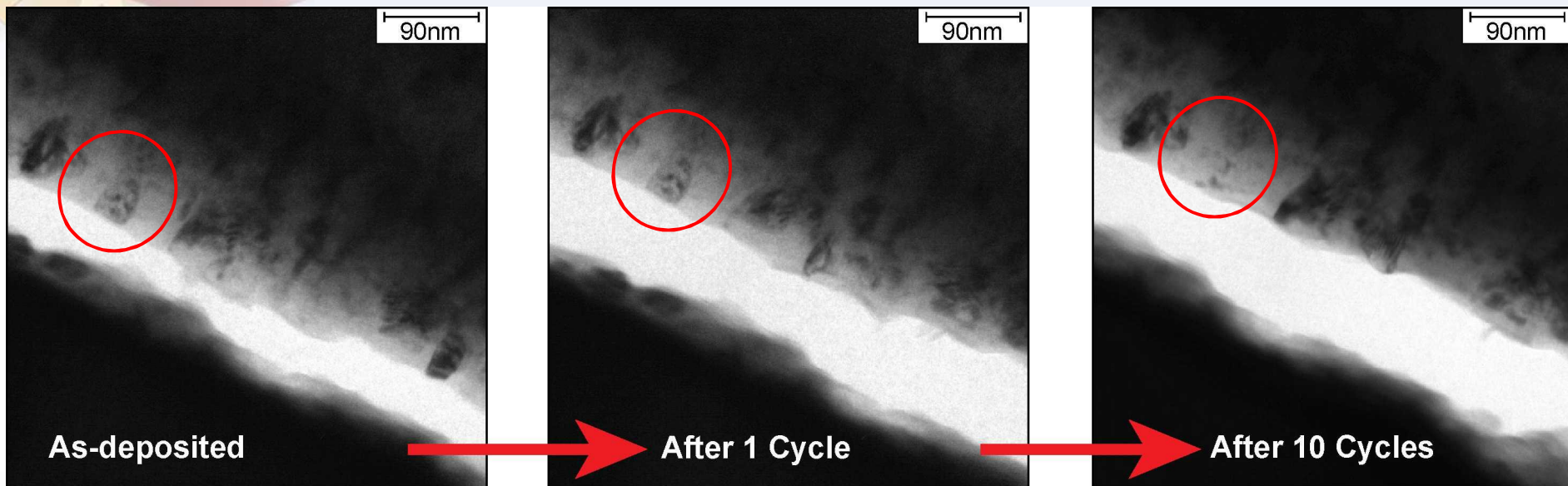


375 MPa nominal contact stress



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Some regions evolve during subsequent cycles

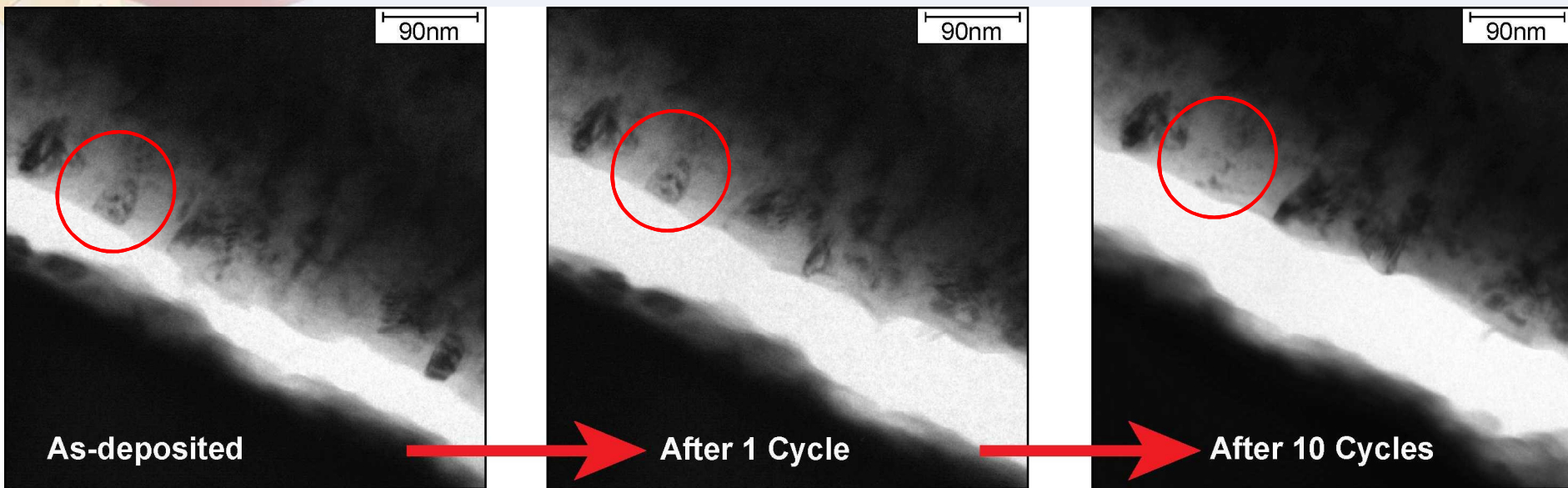


375 MPa nominal contact stress



Sandia National Laboratories

Implications of this Observation

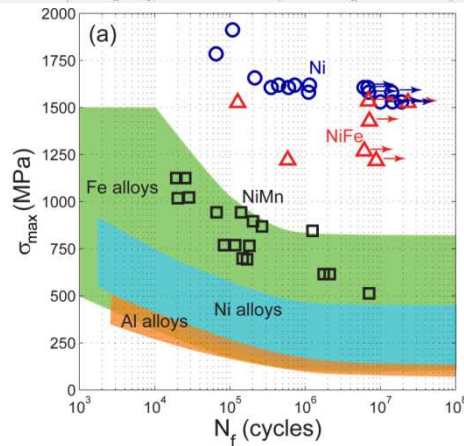


1. *The first direct imaging observation of grain evolution under cyclic loading*
2. *Grain boundaries are perpendicular to loading axis, suggesting the possibility That the boundary migration mechanism may not be shear driven*
3. *This grain evolution is not dynamic recrystallization.*

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.

