

Nanocrystalline alloys: Impervious to Fatigue Failure?

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April 19th , 2013



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Versailles 1842

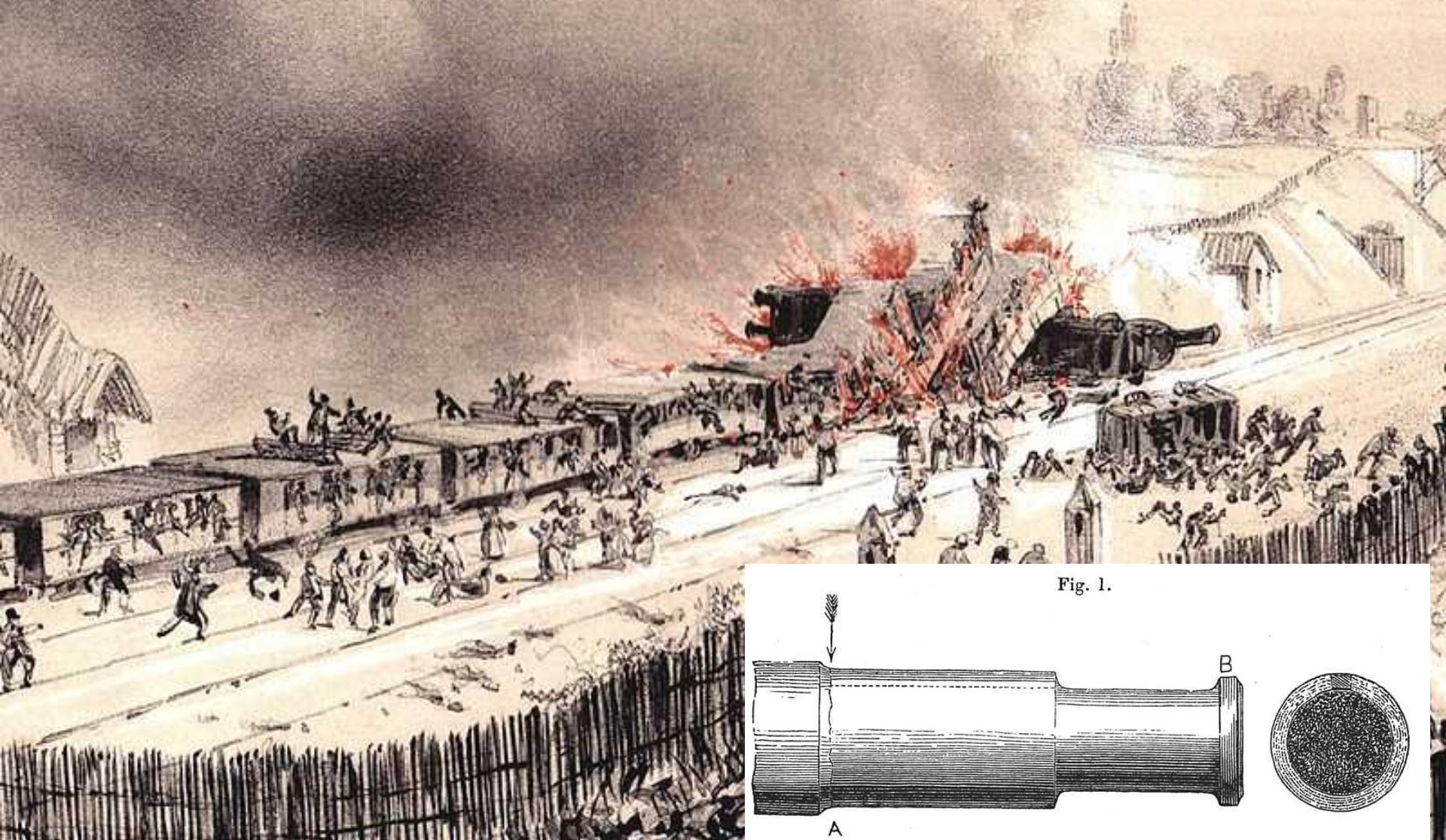
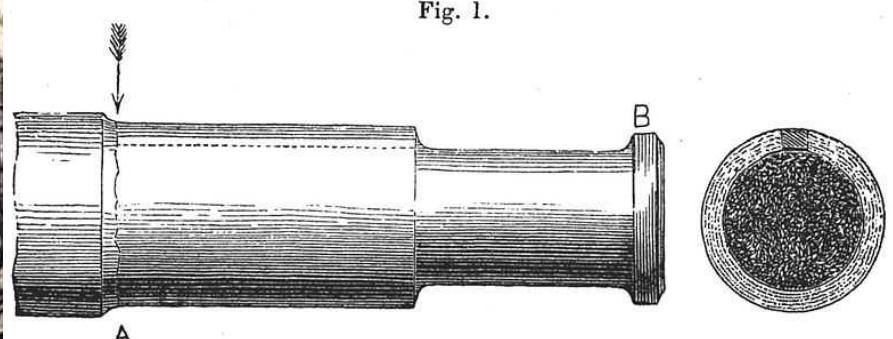


Fig. 1.



by Joseph Glynn, 1843

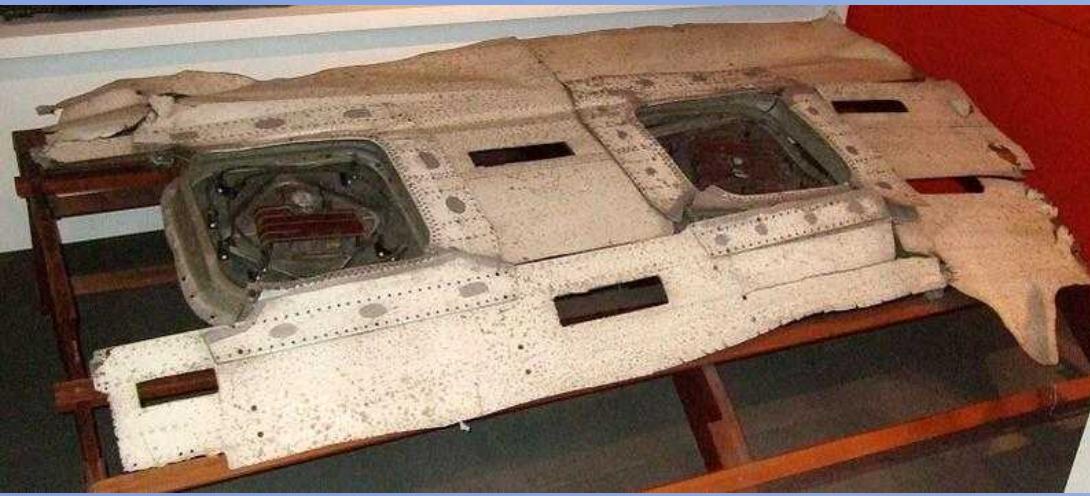


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De Havilland Comet, 1953-4





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.

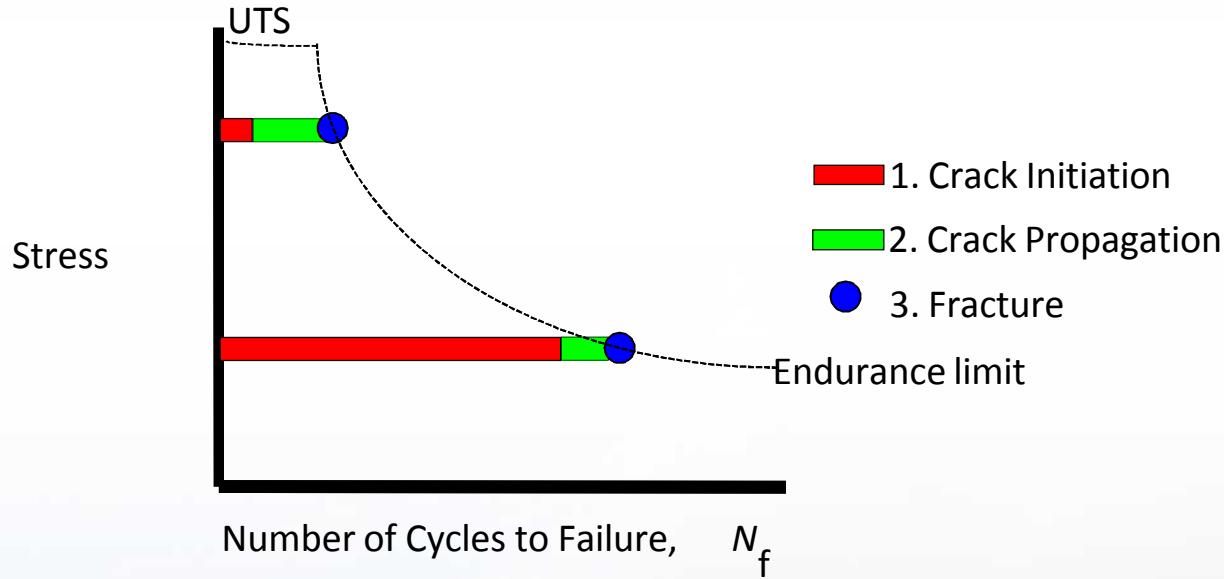
70-90% of all structural fractures result from cyclic damage accumulation (fatigue).

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



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Background Concepts: Fatigue Life

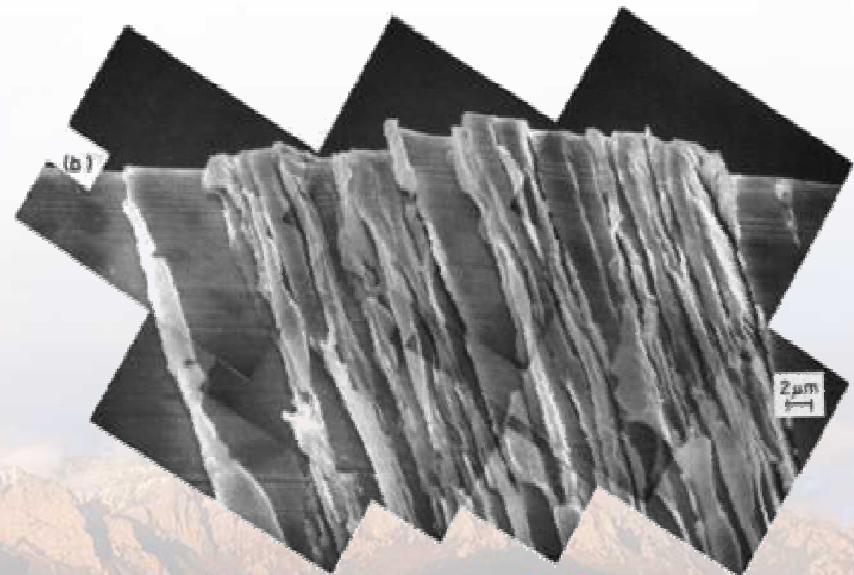
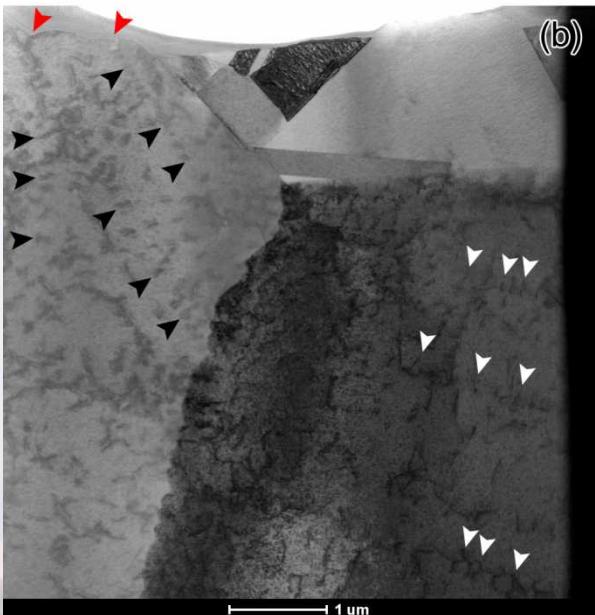
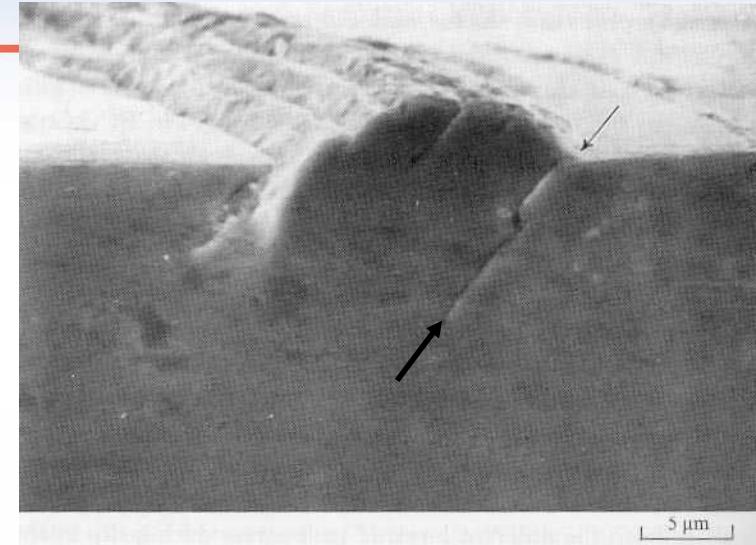
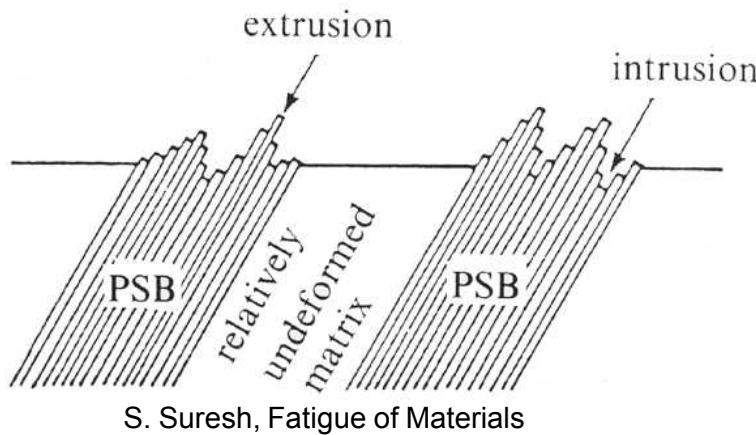


The S-N approach was originally described by Wöhler, 1860; Basquin 1910

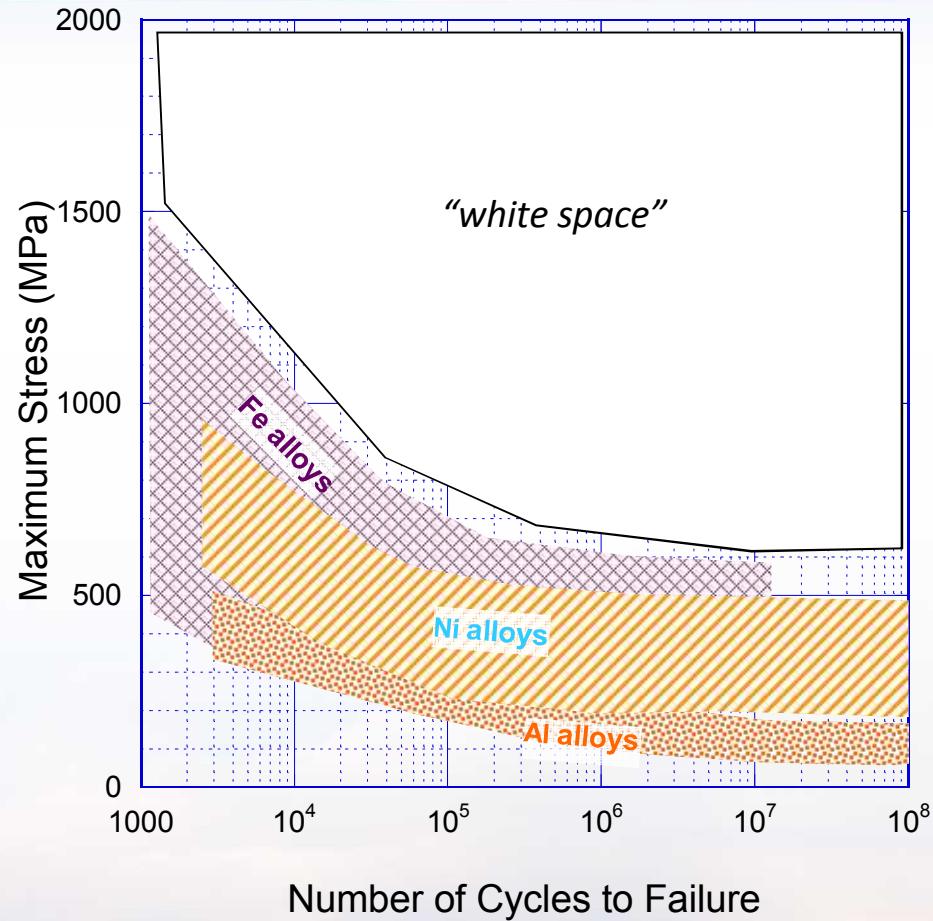


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In the absence of pre-existing defects, fatigue crack nucleation occurs by a persistent slip band process



Fatigue Life in Engineering Alloys



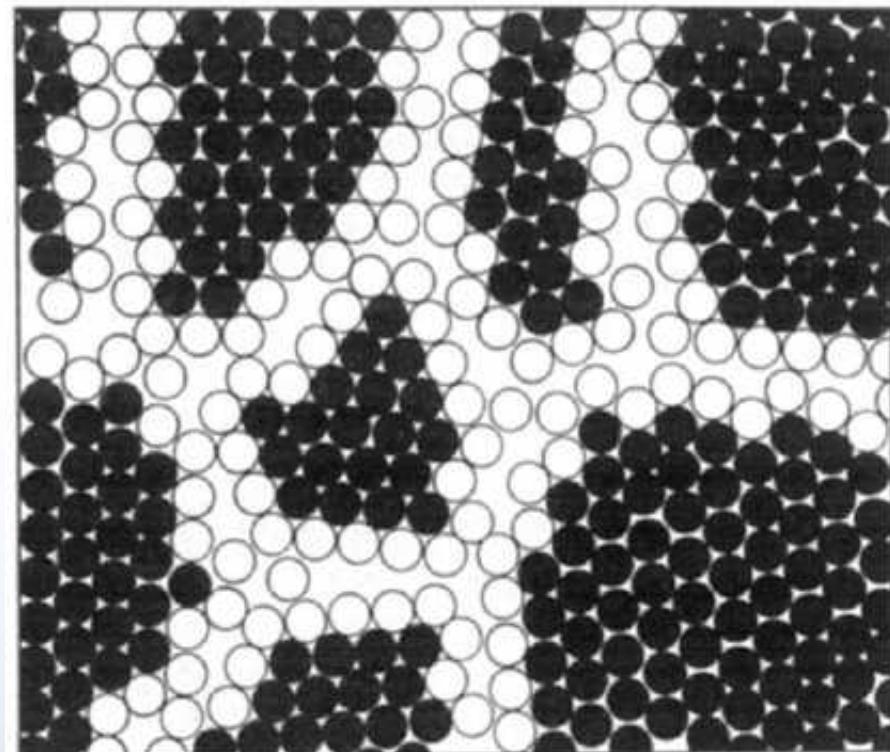
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Nanocrystalline metals possess excellent strength

1989

Gleiter

Nanocrystalline
Materials



>2000 citations

2003

Kumar,

VanSwygenhoven

Suresh

Hall-Petch Breakdown

← grain size

~ 1 μ m

100 nm

10 nm

flow stress σ_y ↑

$$\sigma_y = \sigma_0 + kd_g^{-1/2}$$

k

(grain size) $^{-1/2}$ →

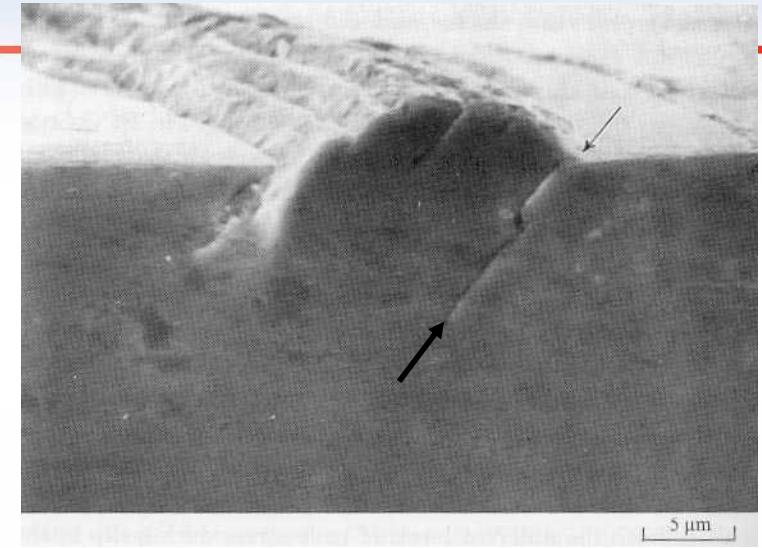
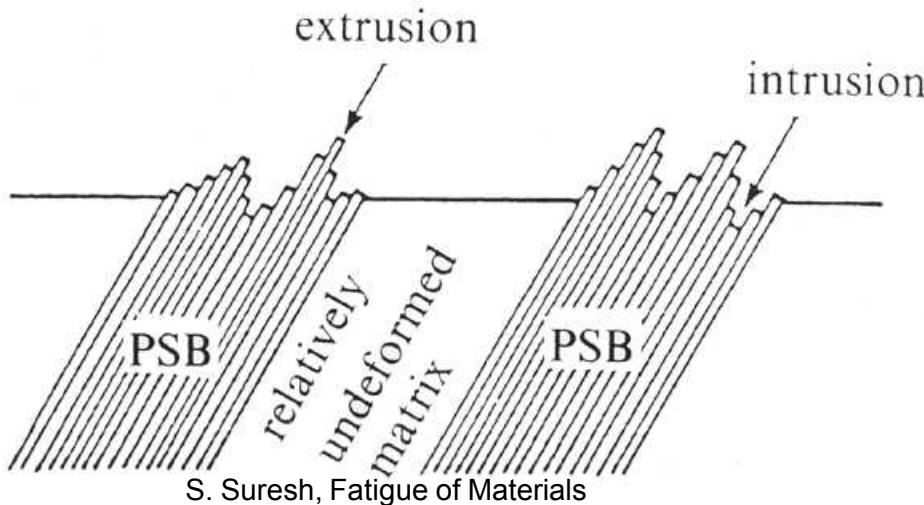
>400 citations

...what about their fatigue resistance???

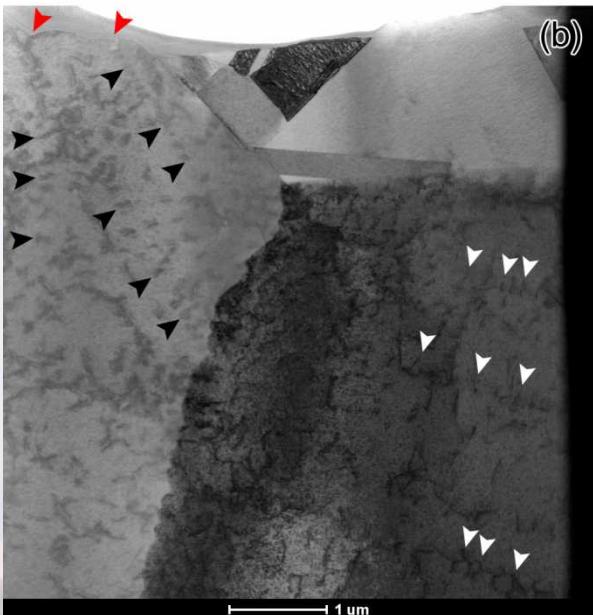


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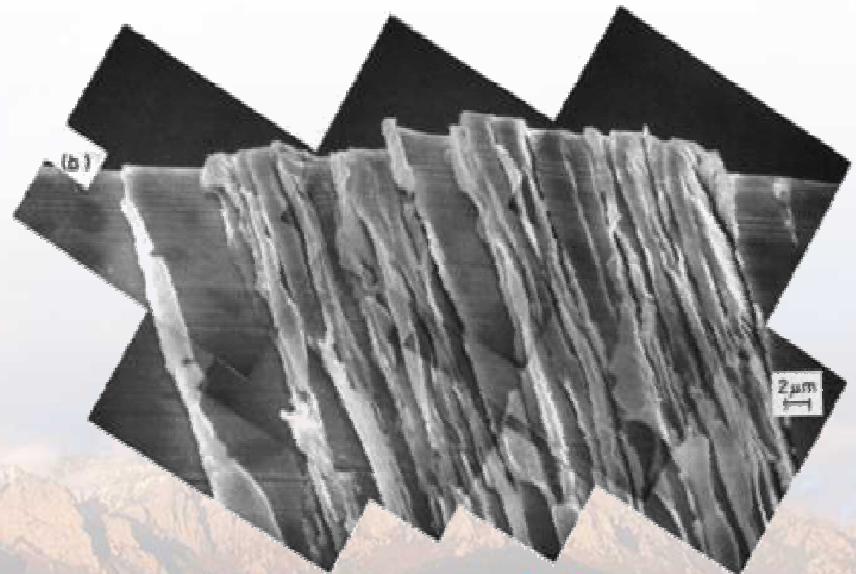
Hypothesis: the small grain size of nanocrystalline metals suppresses persistent slip crack initiation processes



Ma and Laird, 1989

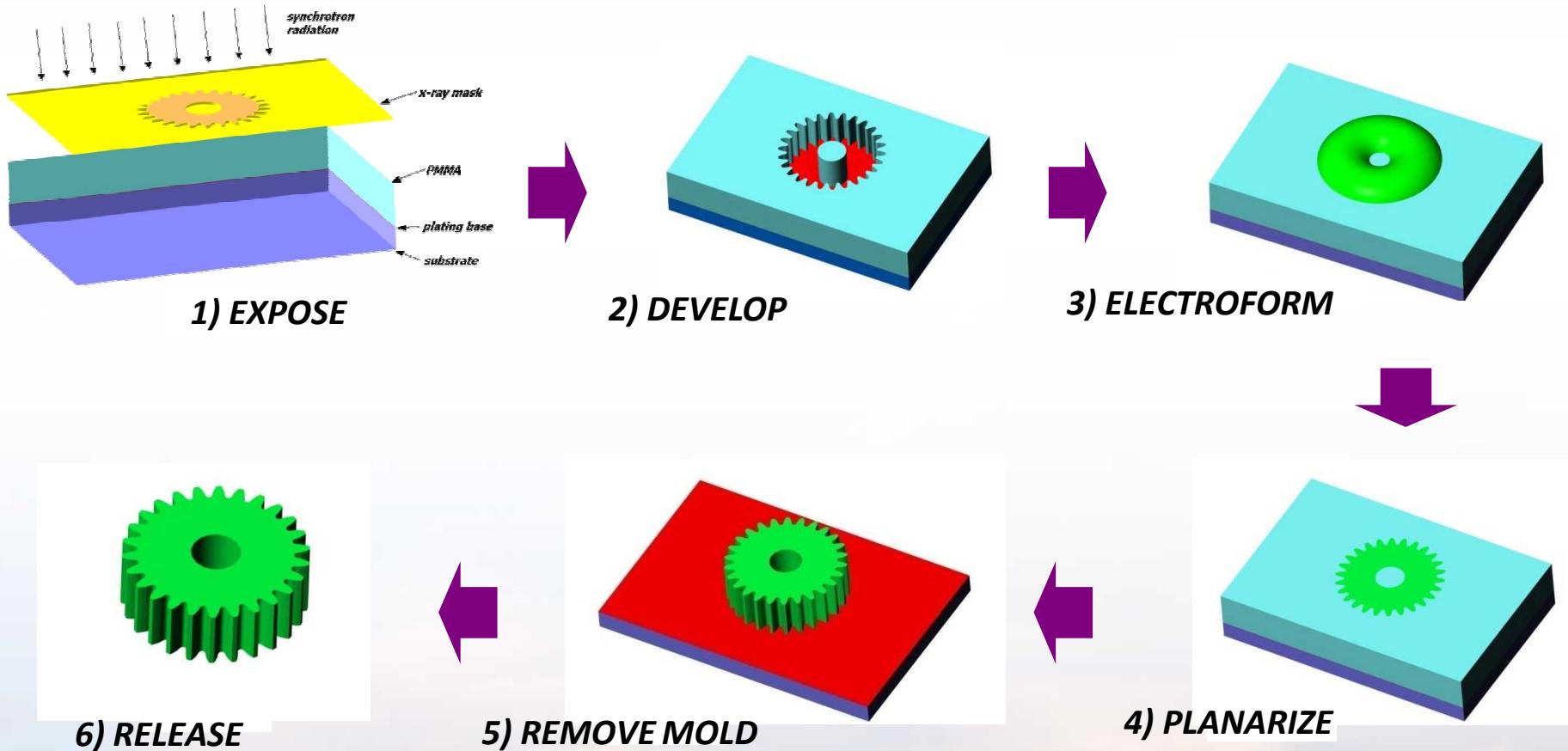


Boyce & Padilla, Metall. Mater. Trans. A, 2011



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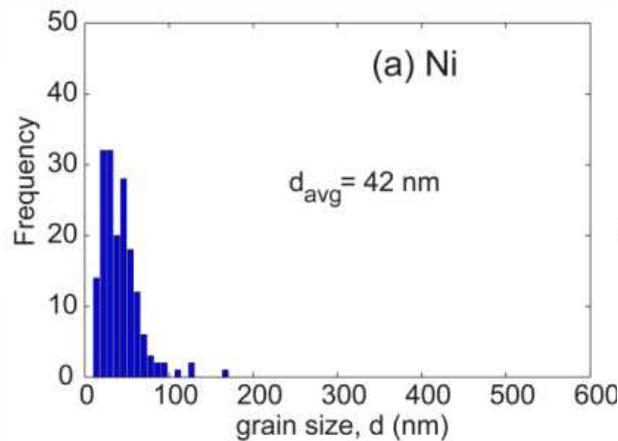
The Direct LIGA Process for Metallic Microdevices



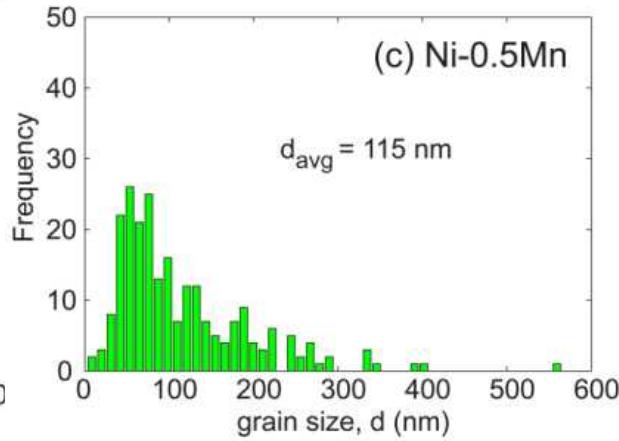
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Three Nanocrystalline Ni-Based Alloys

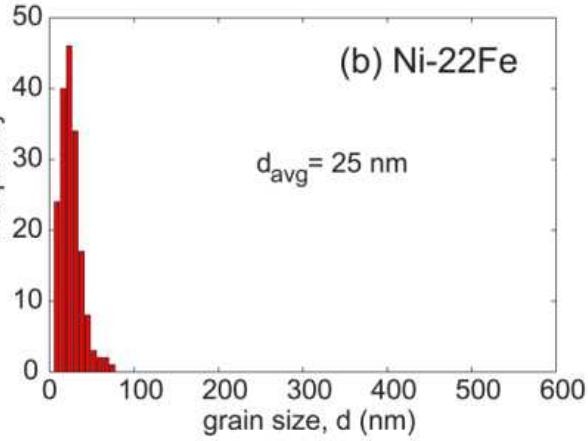
Ni



Ni-0.5Mn



Ni-22Fe



impurities

Yield ~ 1200 MPa

*$<200> + <111>$ very
weak texture*

nanoprecipitates

Yield ~ 1200 MPa

*$<220>$
texture*

high solute content

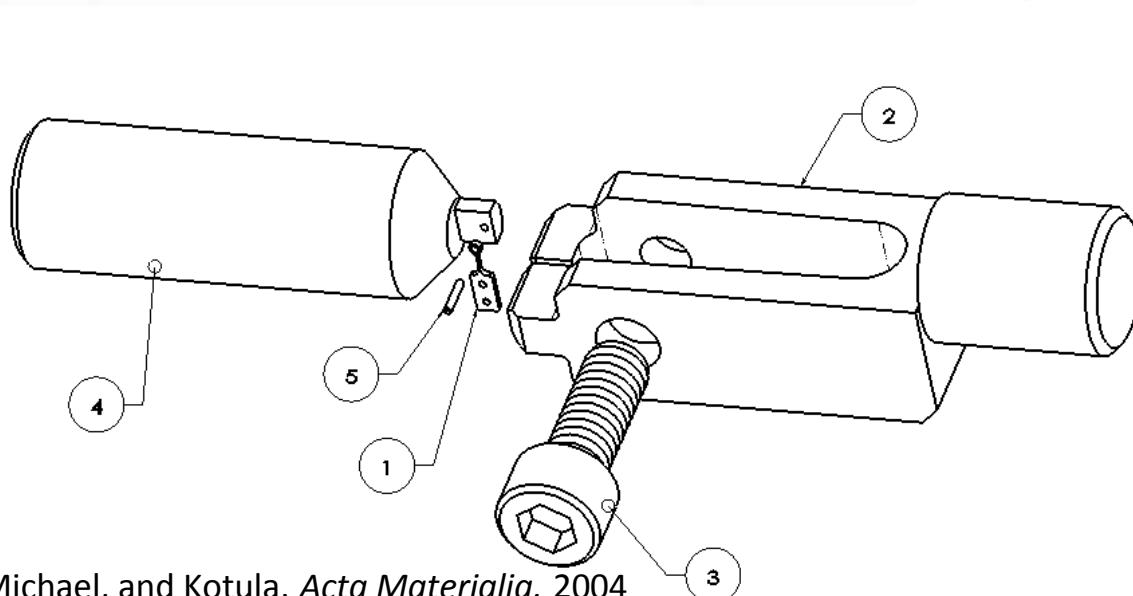
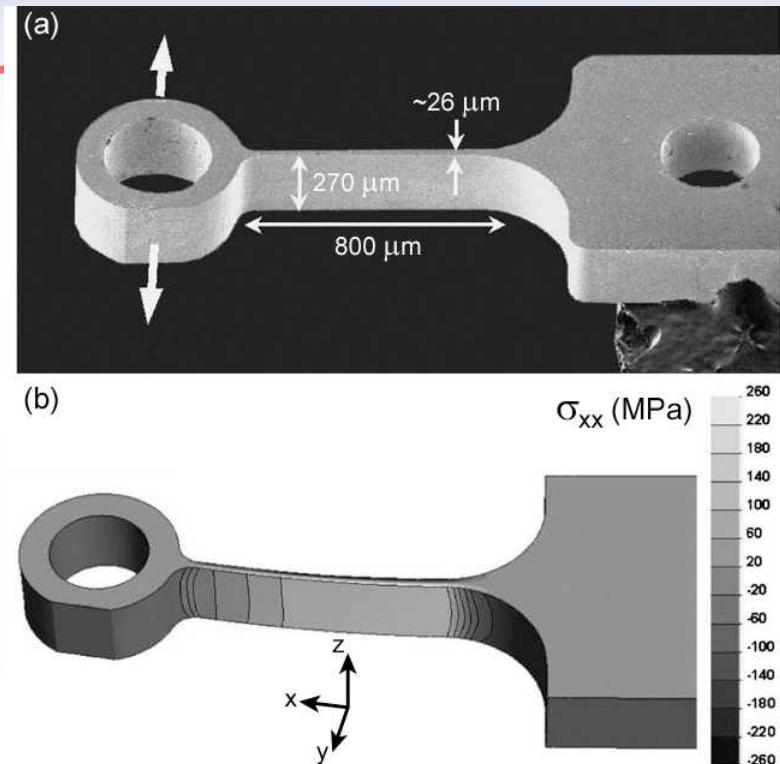
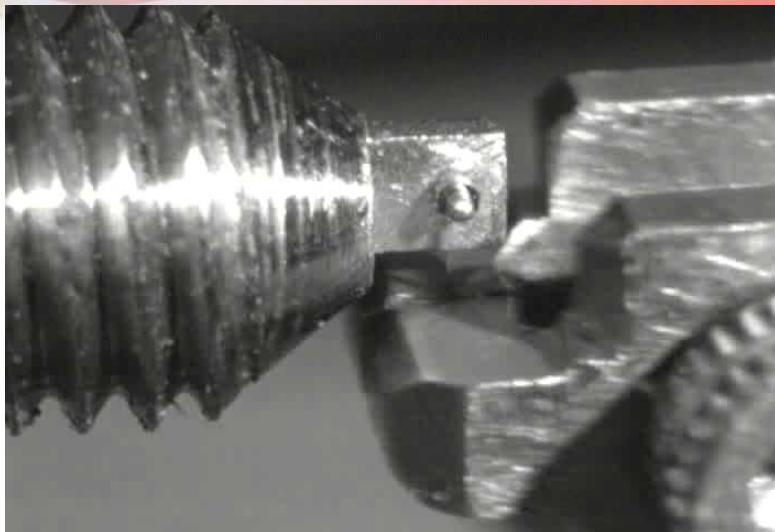
Yield ~ 1400 MPa

*$<200> + <111>$
texture*



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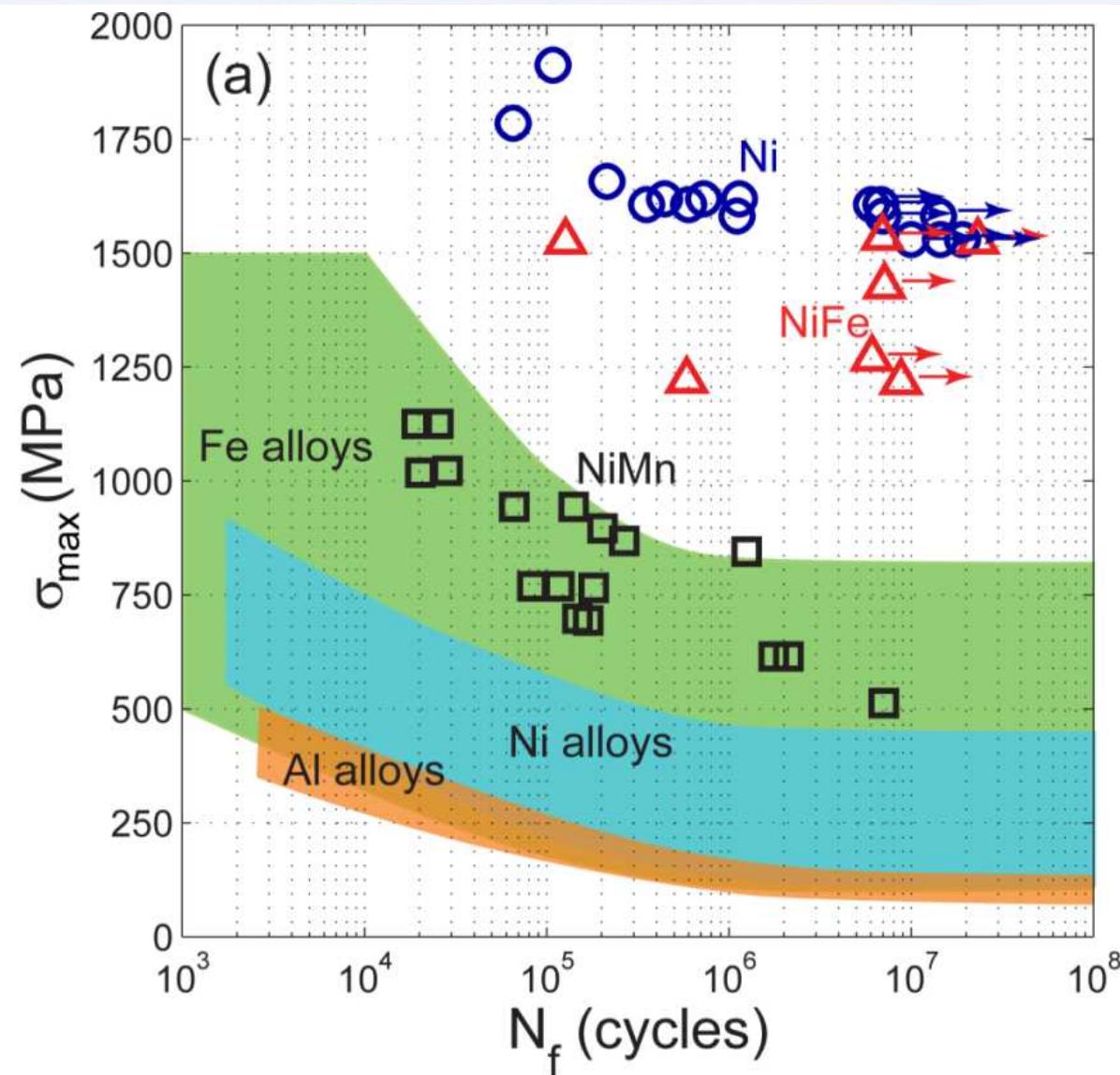
LIGA Fatigue Testing



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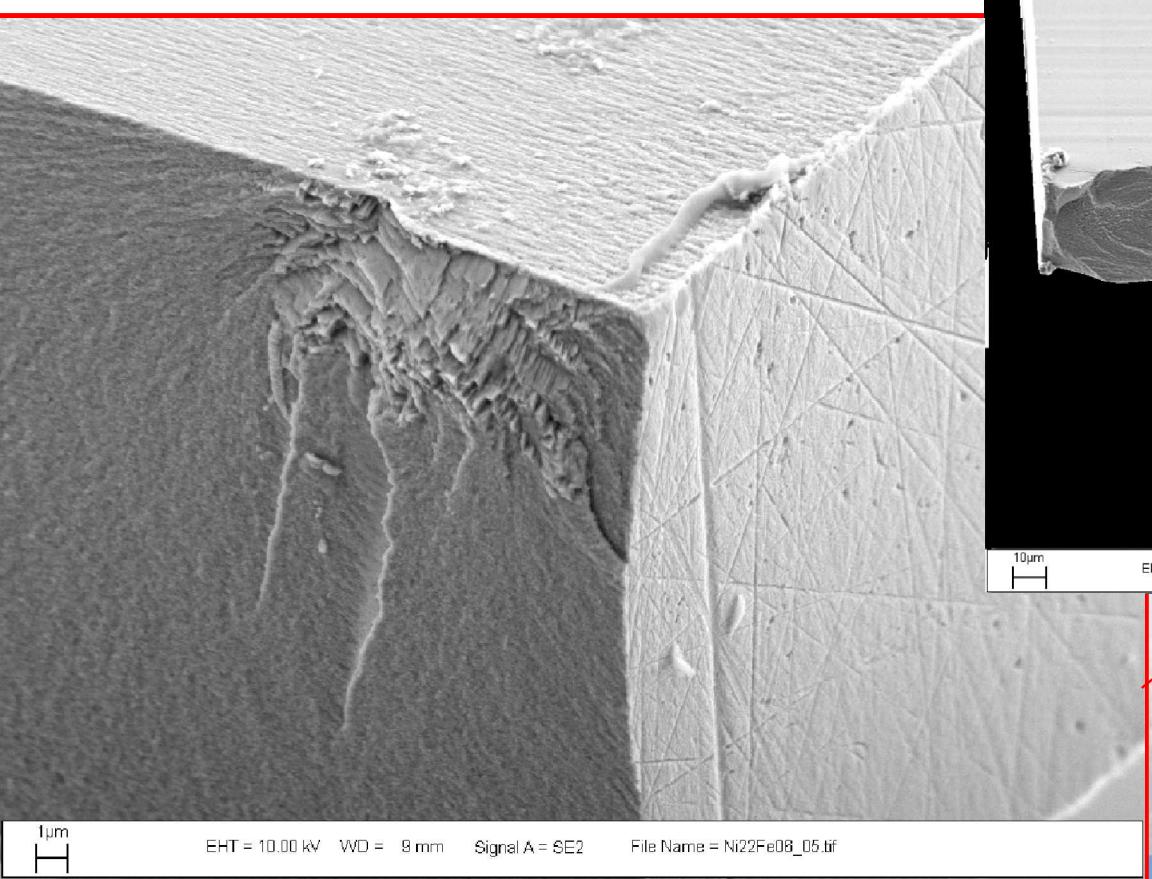
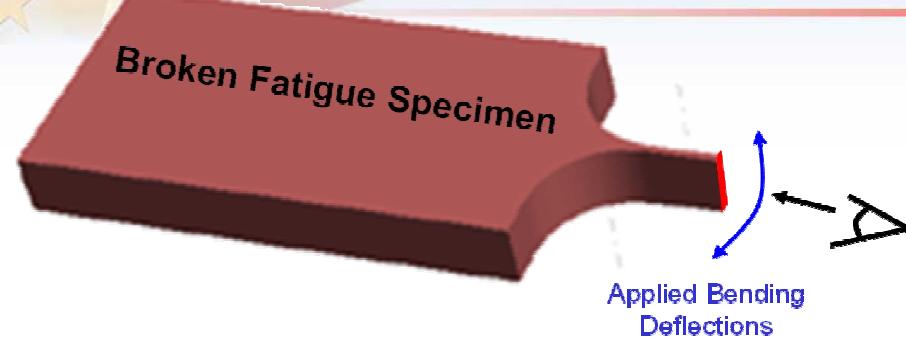
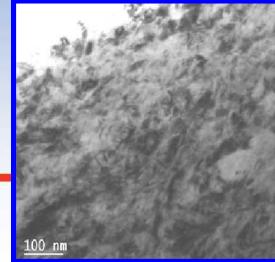


Fatigue Lifetime Results

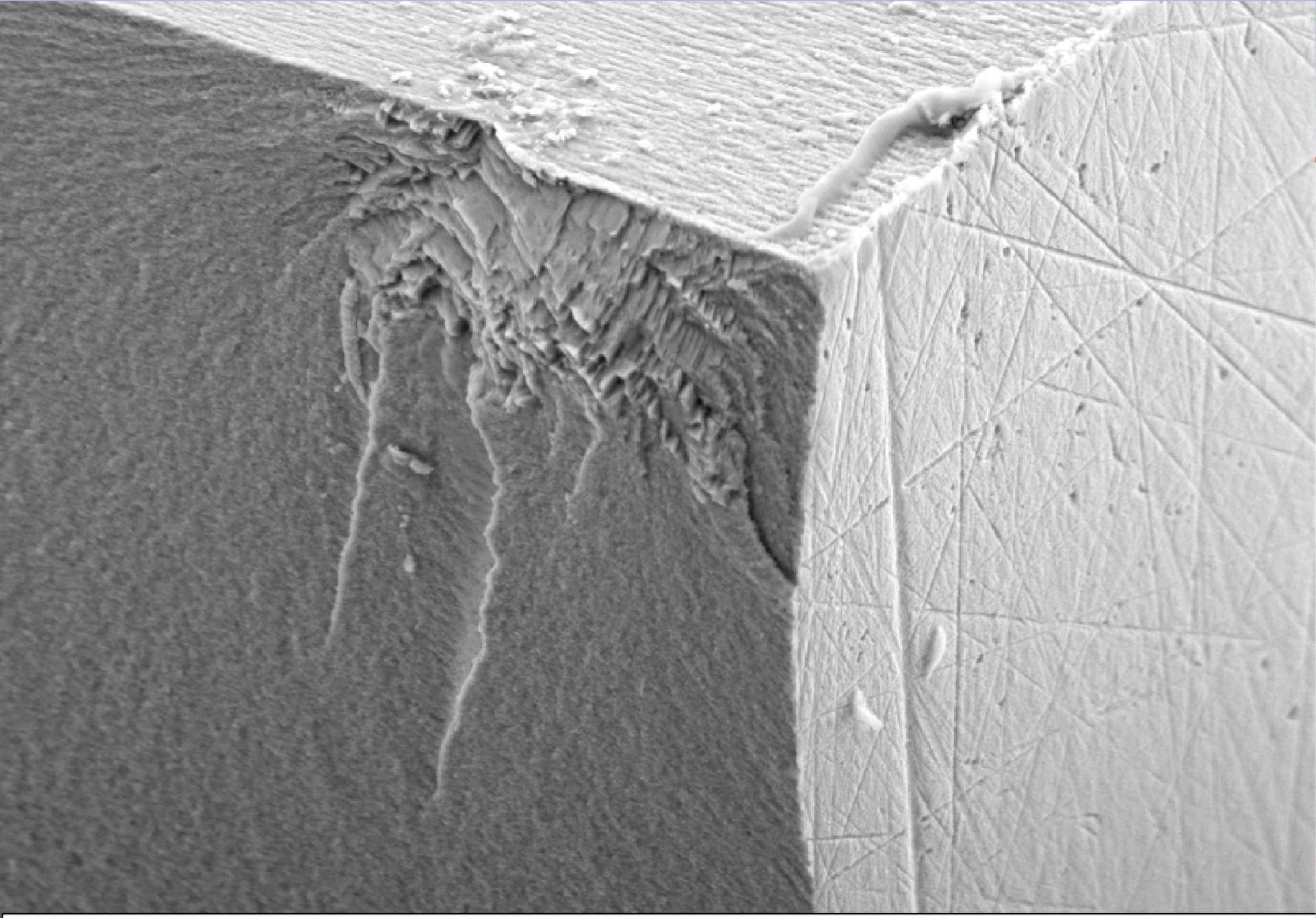


Unusual Initiation Zone In Nanocrystalline Ni-Fe Alloy

NiFe



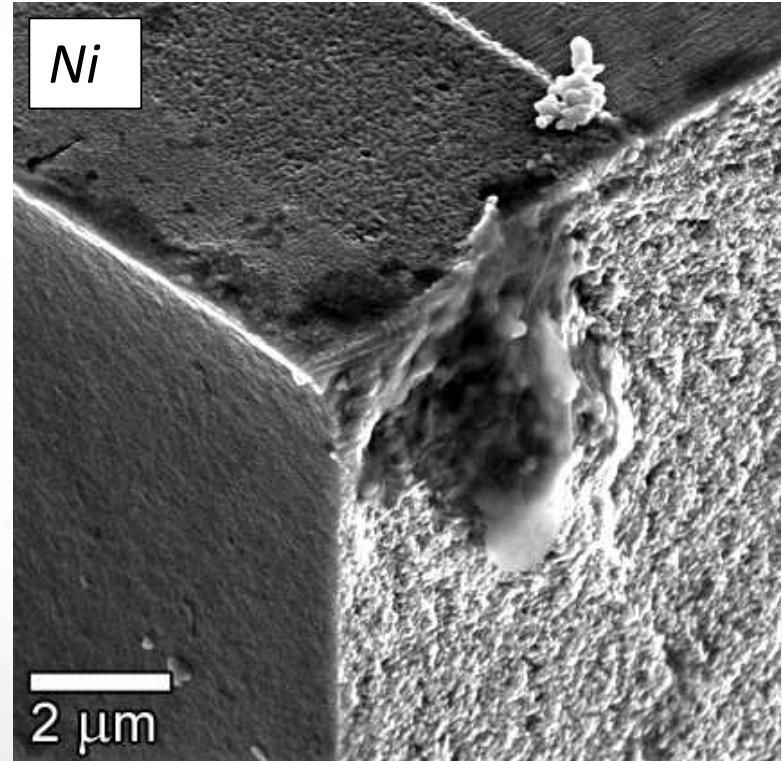
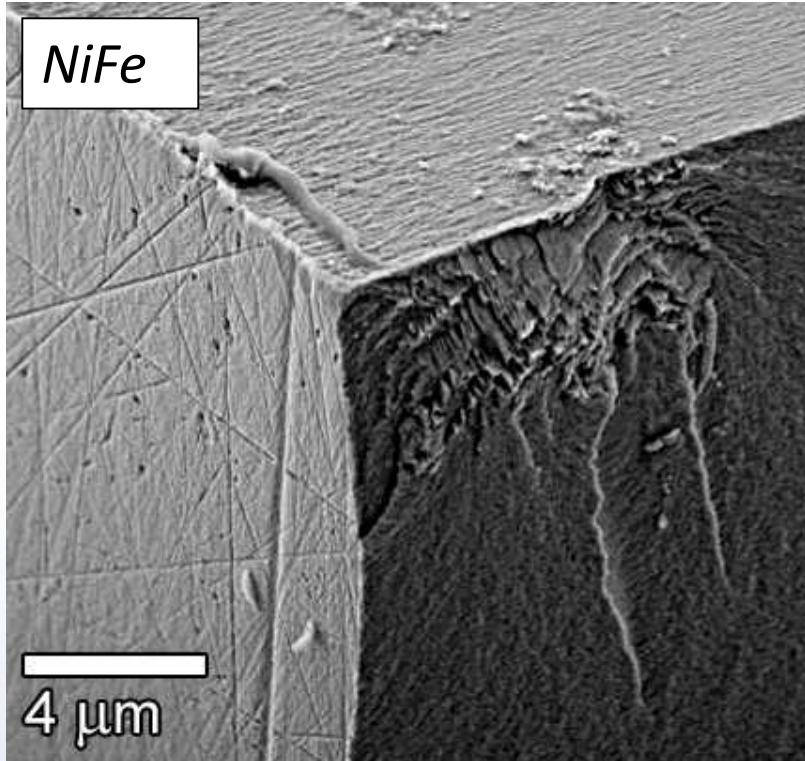
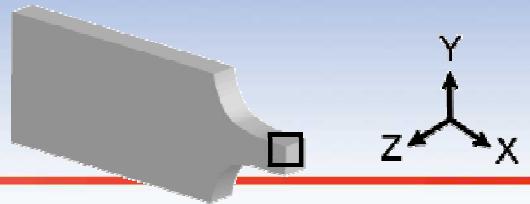
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1 μ m
H

EHT = 10.00 kV WD = 9 mm Signal A = SE2 File Name = Ni22Fe06_05.tif

Typical Fracture Surface (SEM)

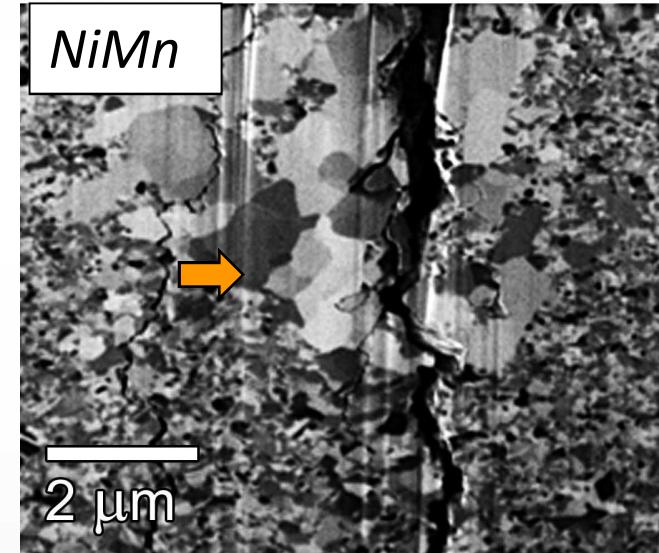
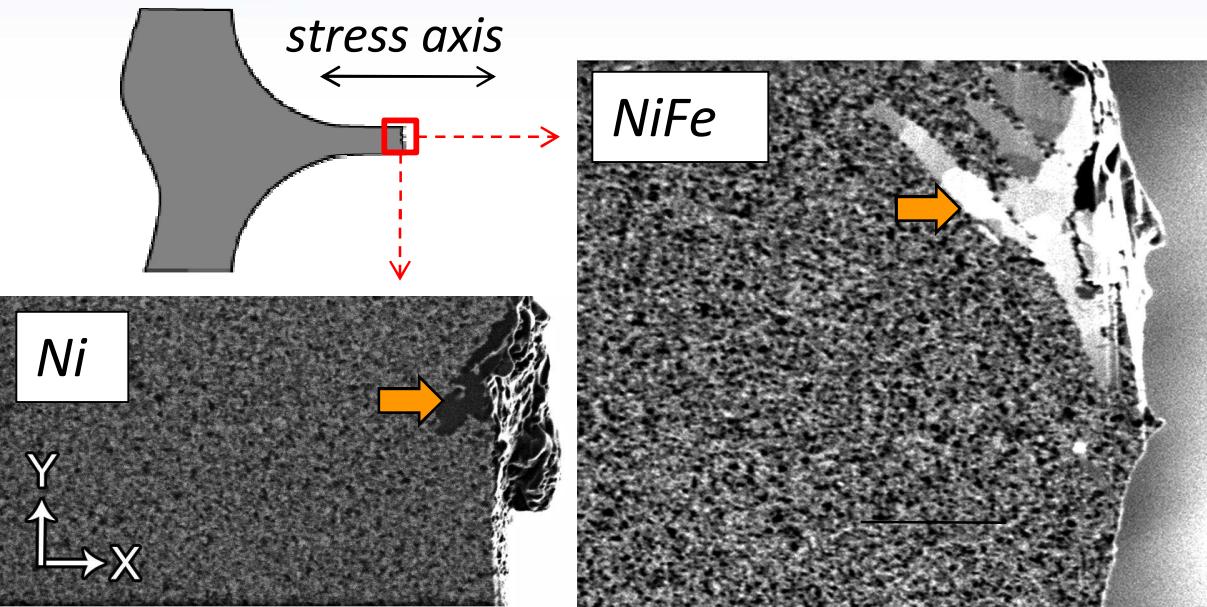


- Initiation zone marked by large blocky features
- Propagation zone morphology follows NC microstructure



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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

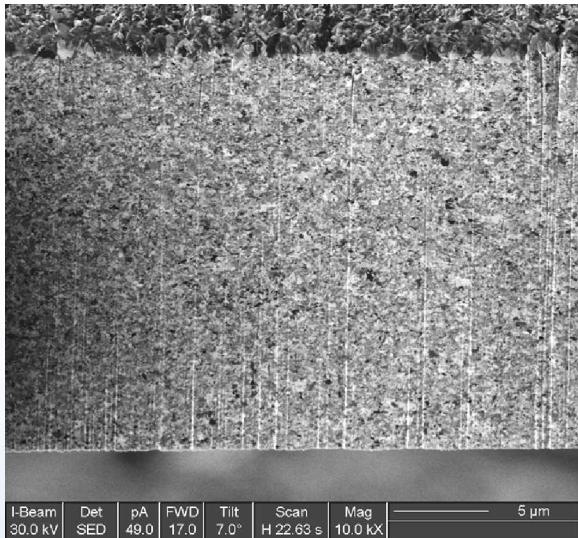


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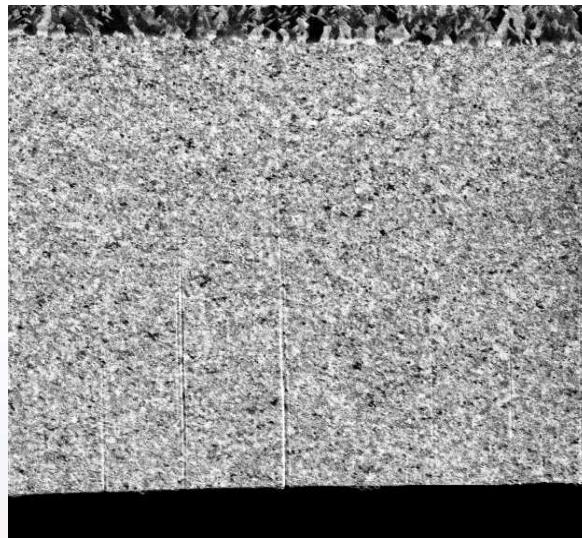


Grain growth is induced by fatigue

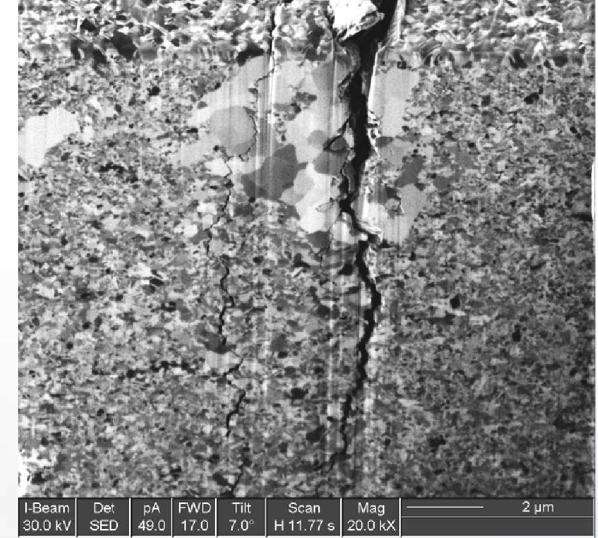
As-Deposited



After 1 Cycle

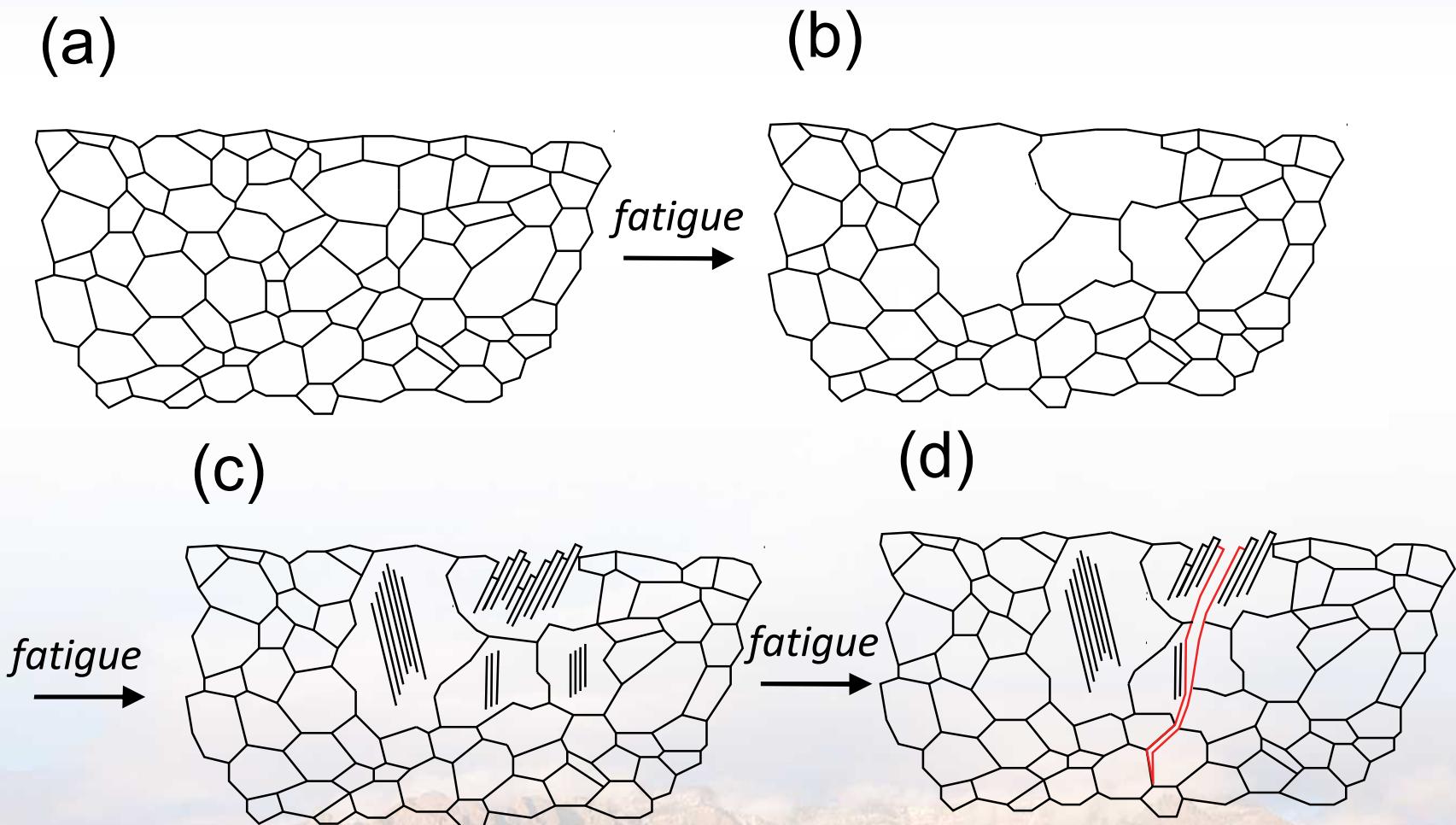


After ~100,000 Cycles





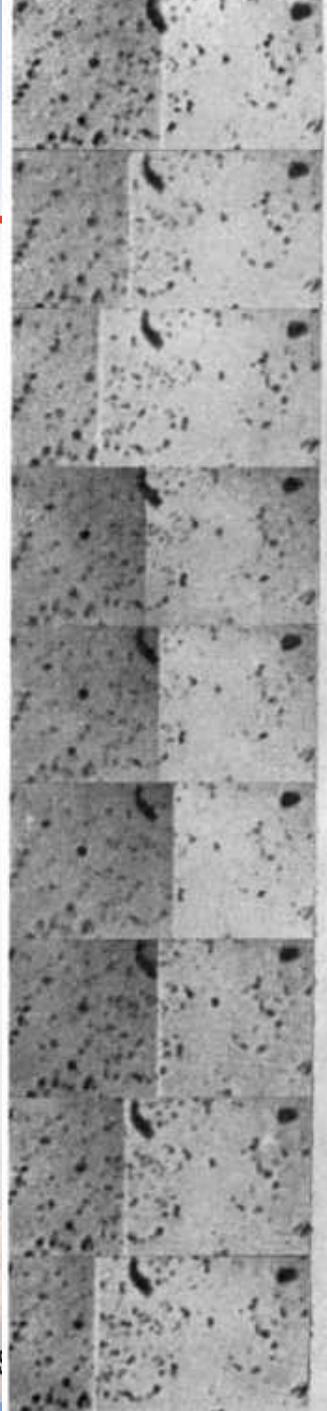
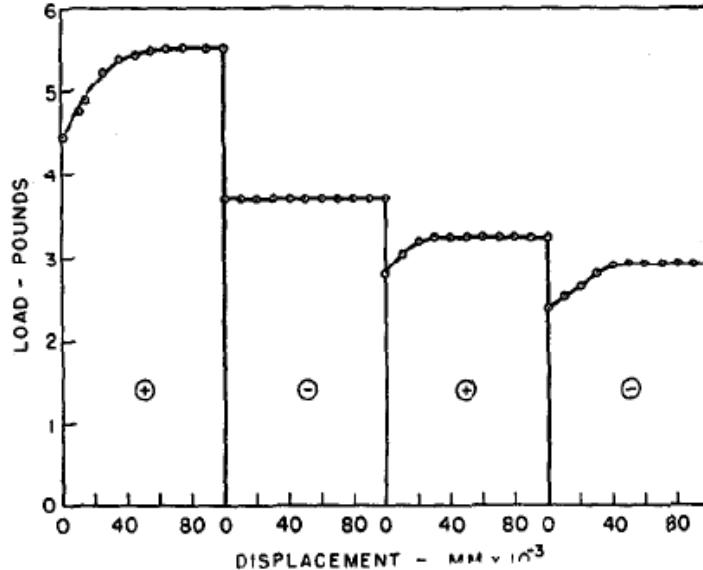
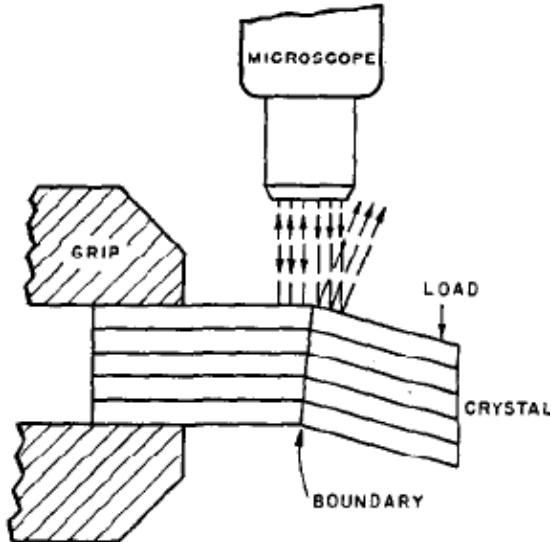
New Fatigue Process in Nanocrystalline Ni Alloys



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Mechanically-Induced Grain Boundary Motion

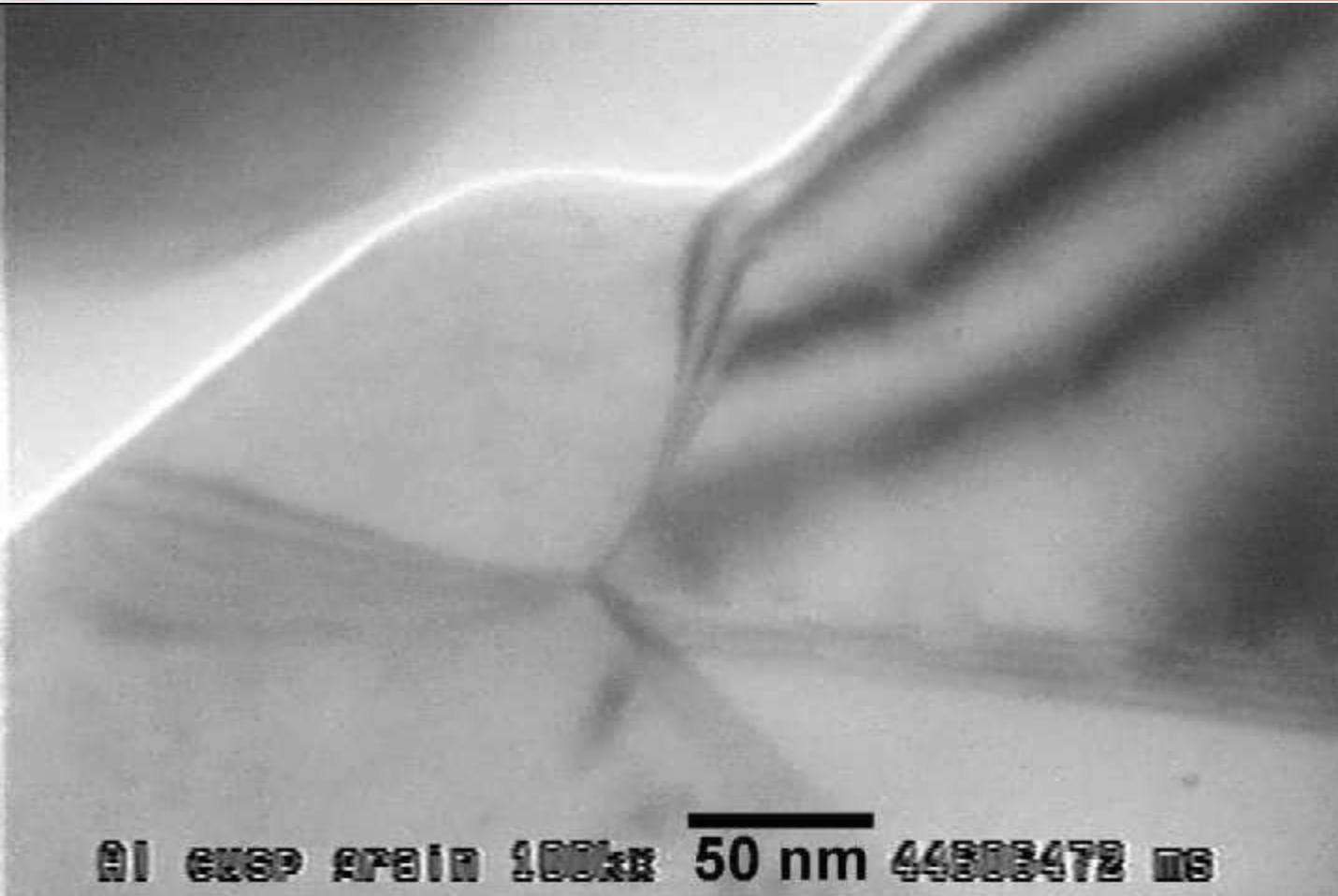
2° LAGB in 99.99% Zinc



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

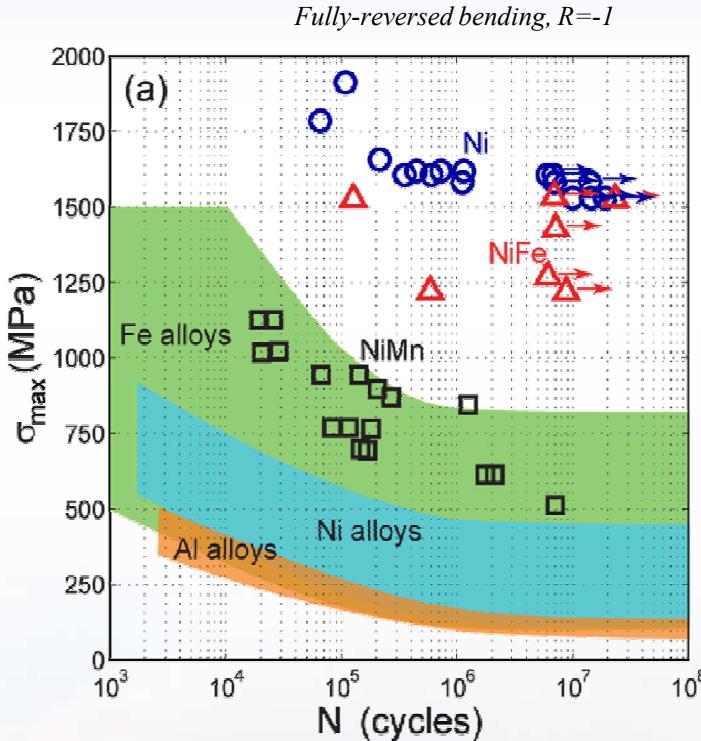
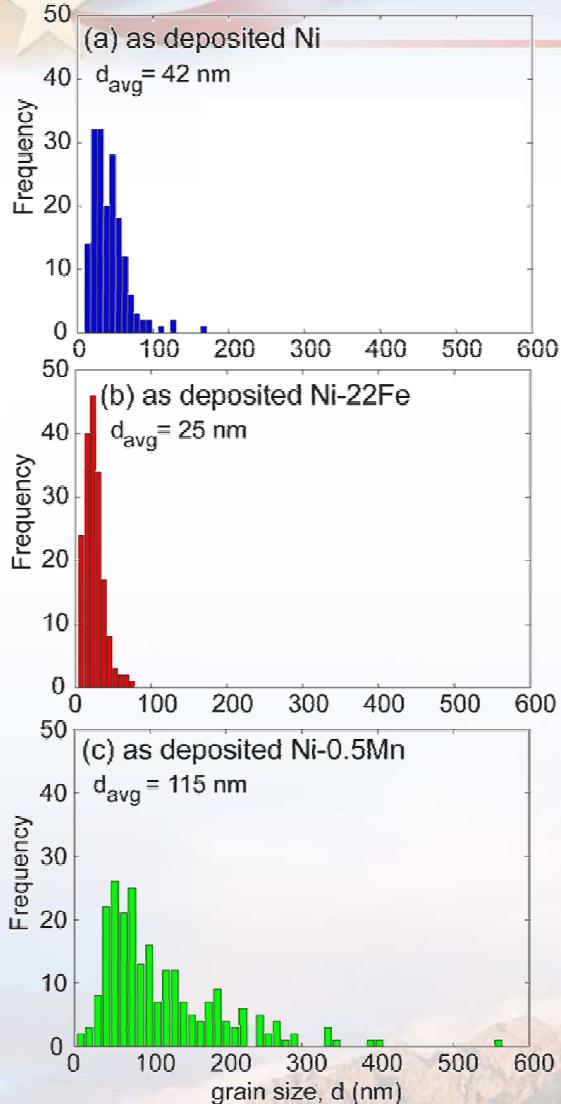
Three nanocrystalline Ni alloys exhibit exceptional fatigue resistance, apparently due to the suppression of traditional dislocation-mediated crack initiation processes.

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



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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?



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

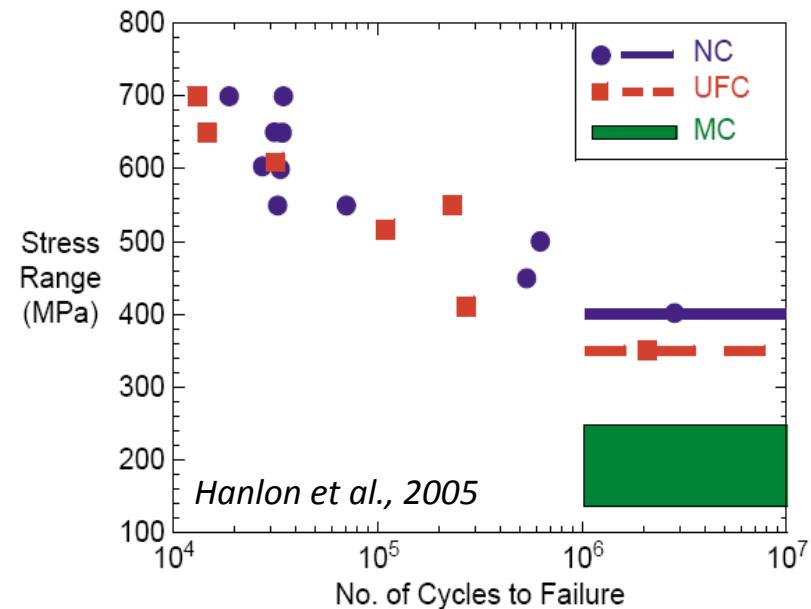
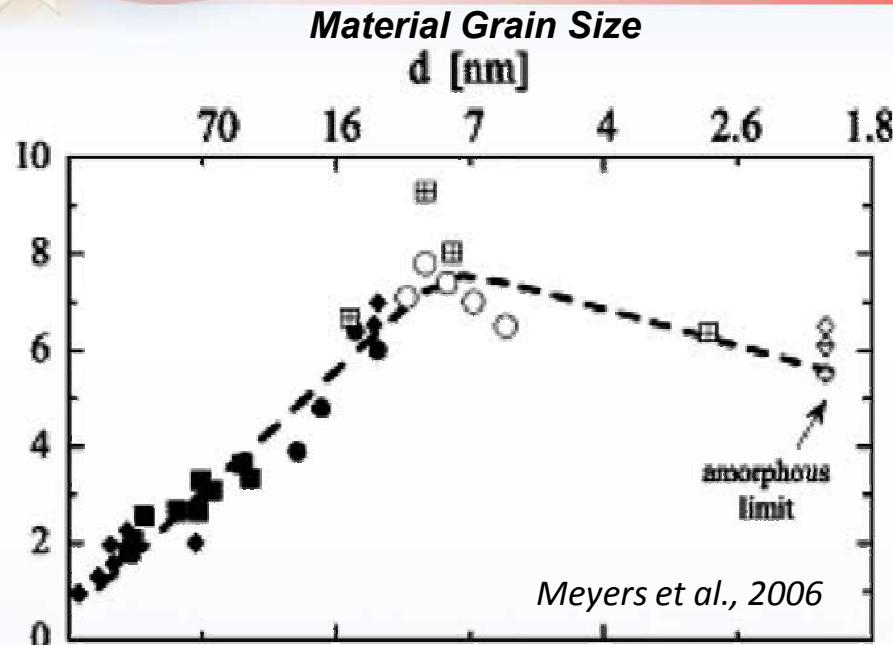


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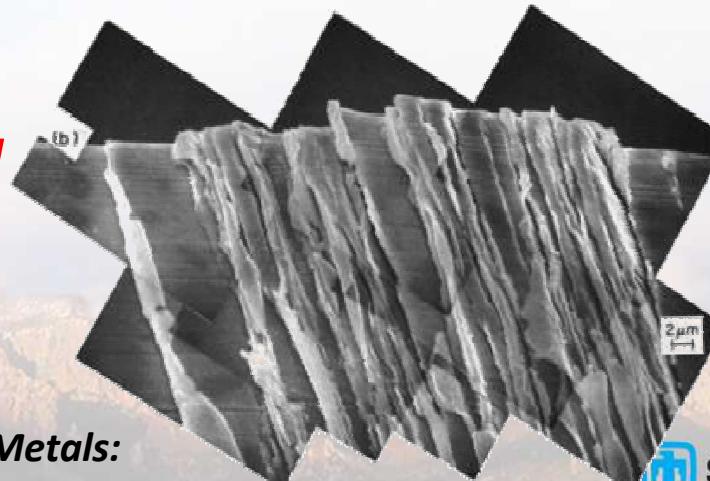
Hall-Petch Grain Size Effects



Material Hardness (or Strength)



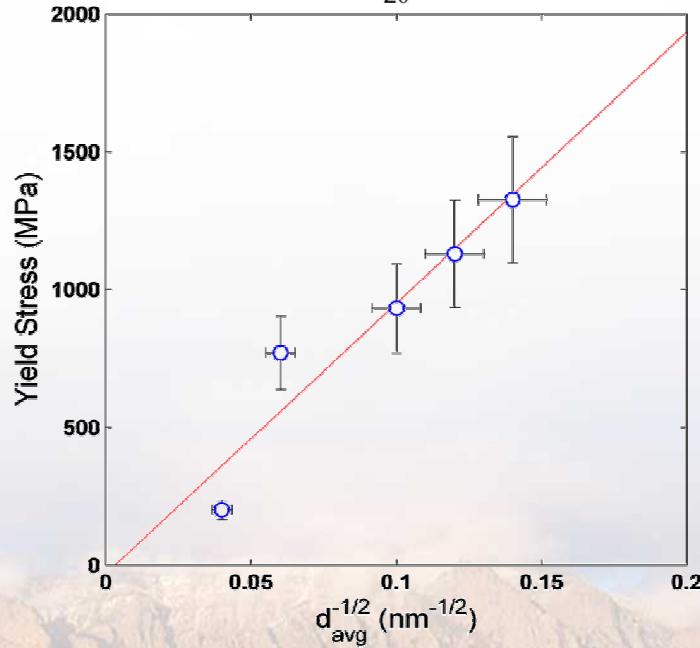
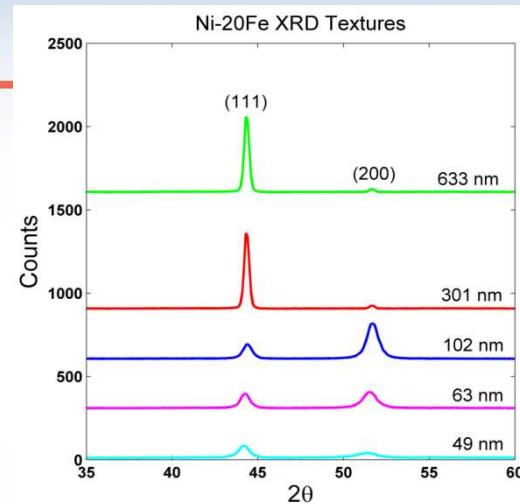
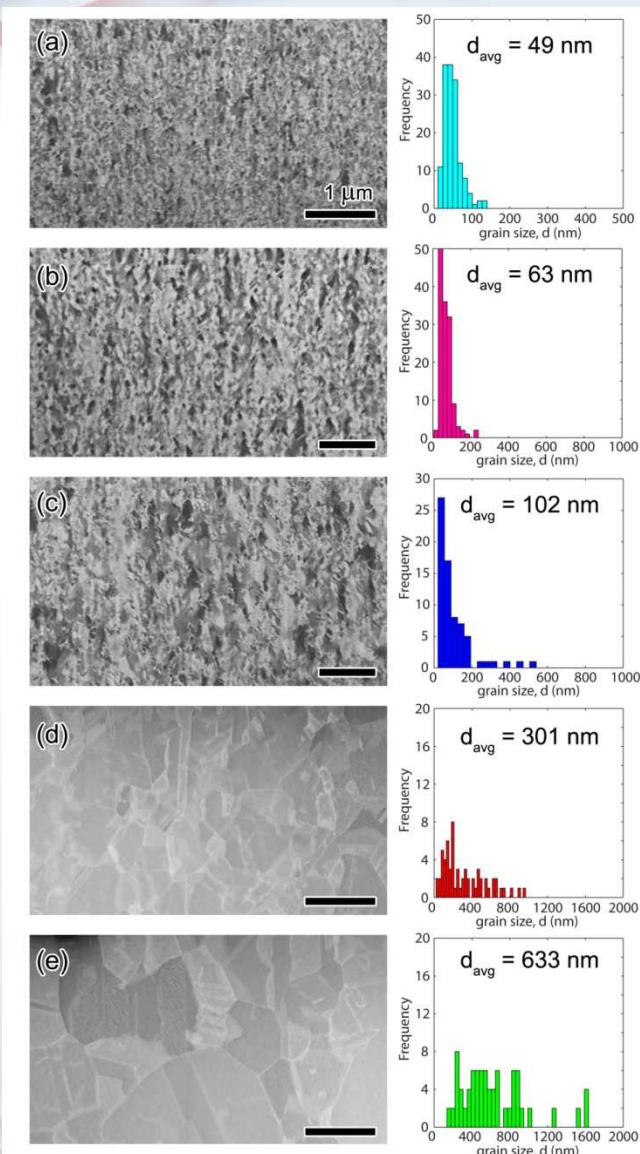
At what length scale do conventional fatigue processes (i.e. persistent slip) begin to break down?



Ma and Laird, 1989

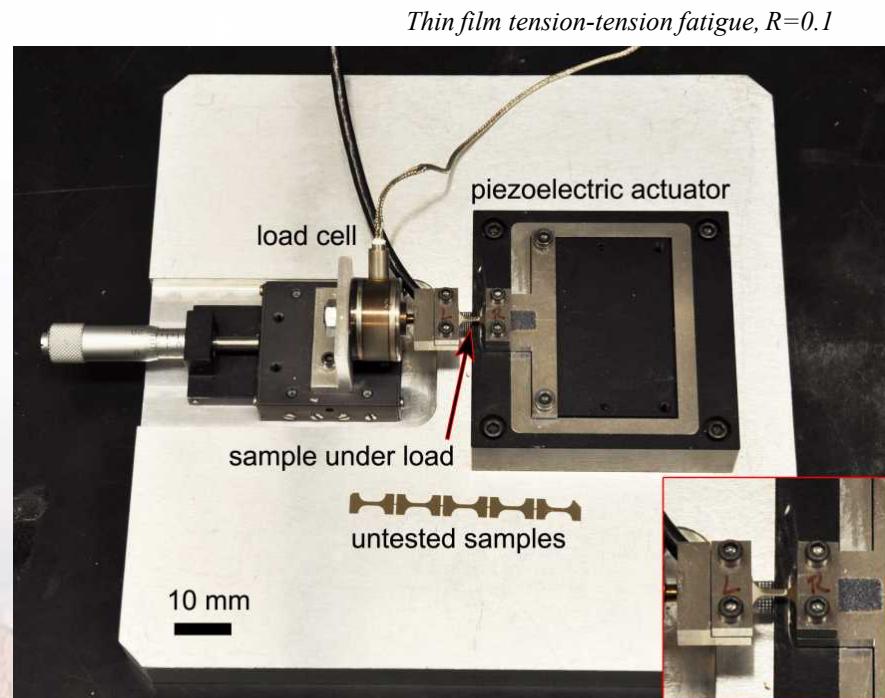
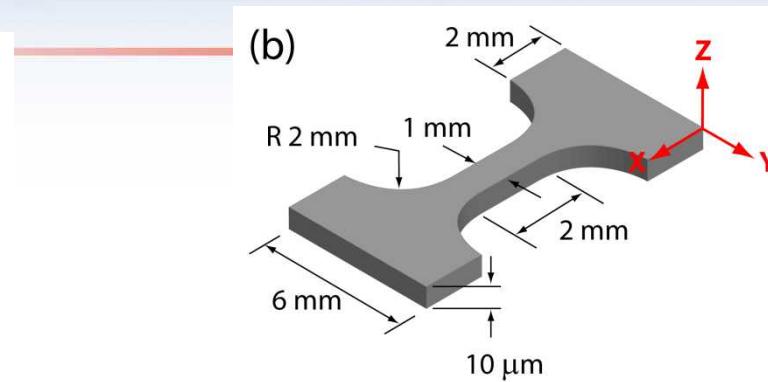
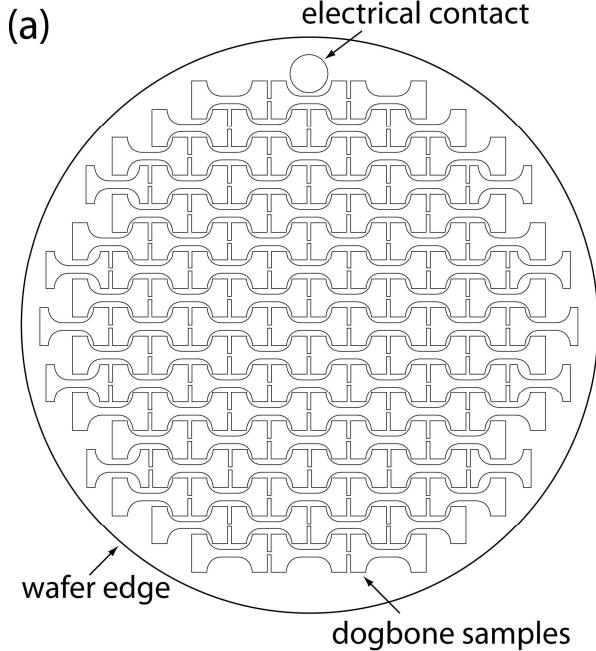


One alloy (Ni-10Fe) produced with 5 Grain Sizes



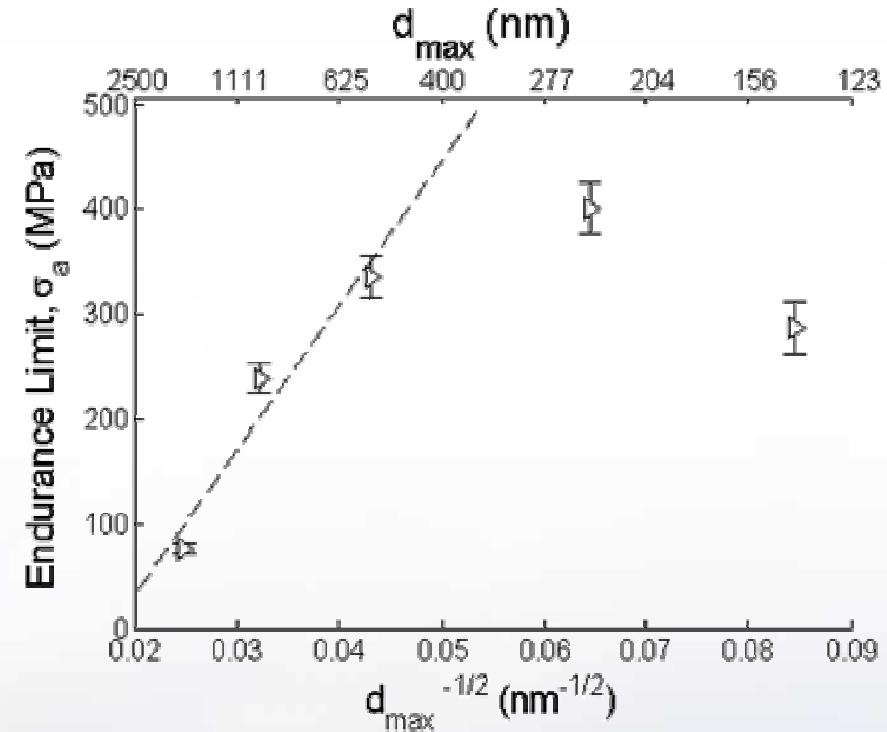
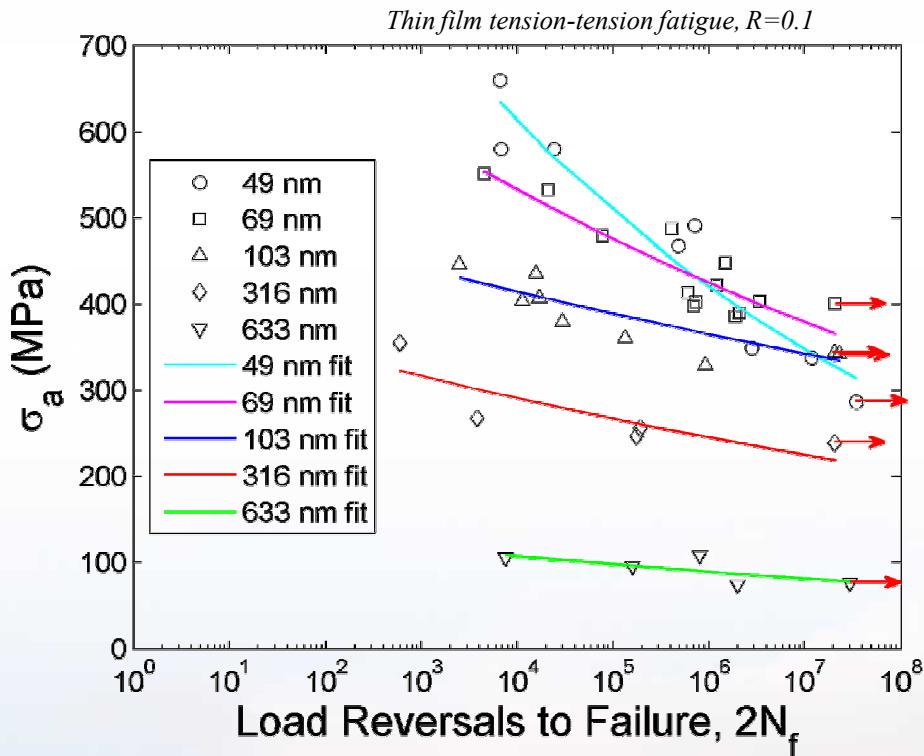
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Thin film fatigue of electrodeposited Ni-Fe (permalloy)



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

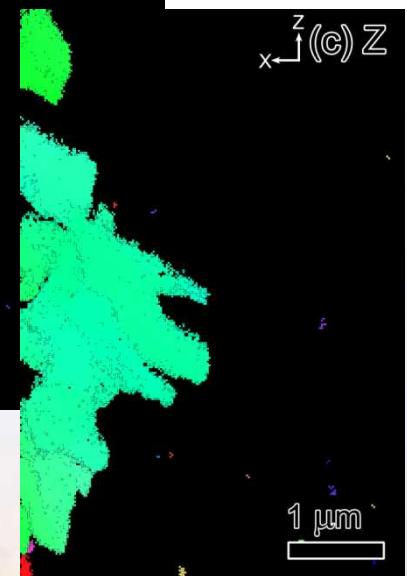
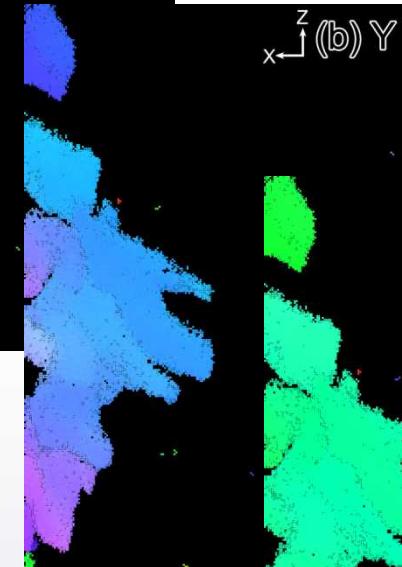
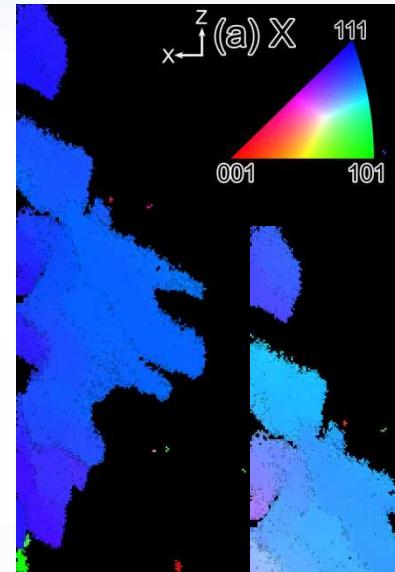
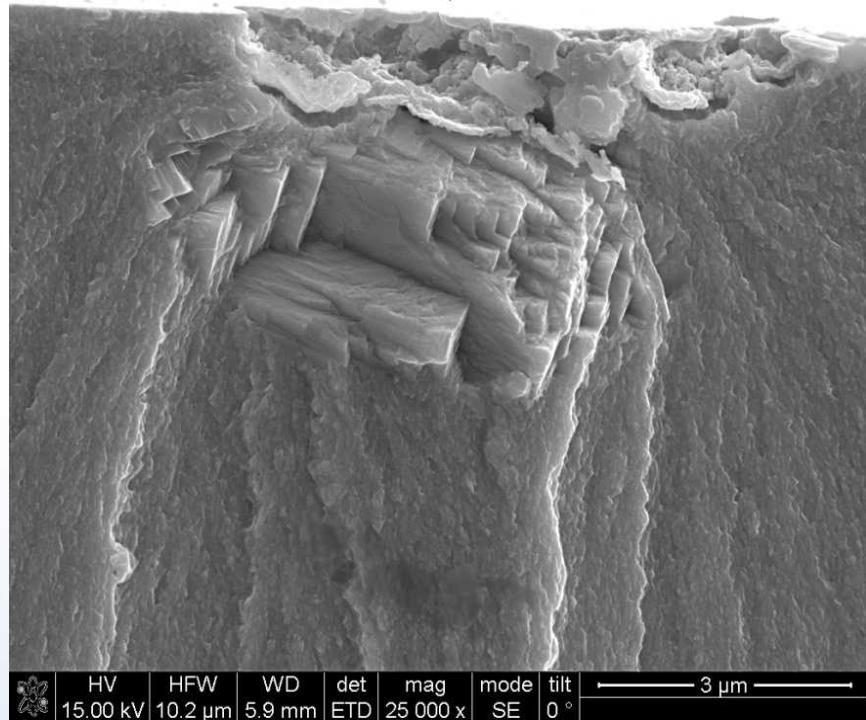


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Same morphology of highly textured, fatigue-coarsened grains form in samples with grain sizes of 100 nm or less.

(a)

FIB cut location



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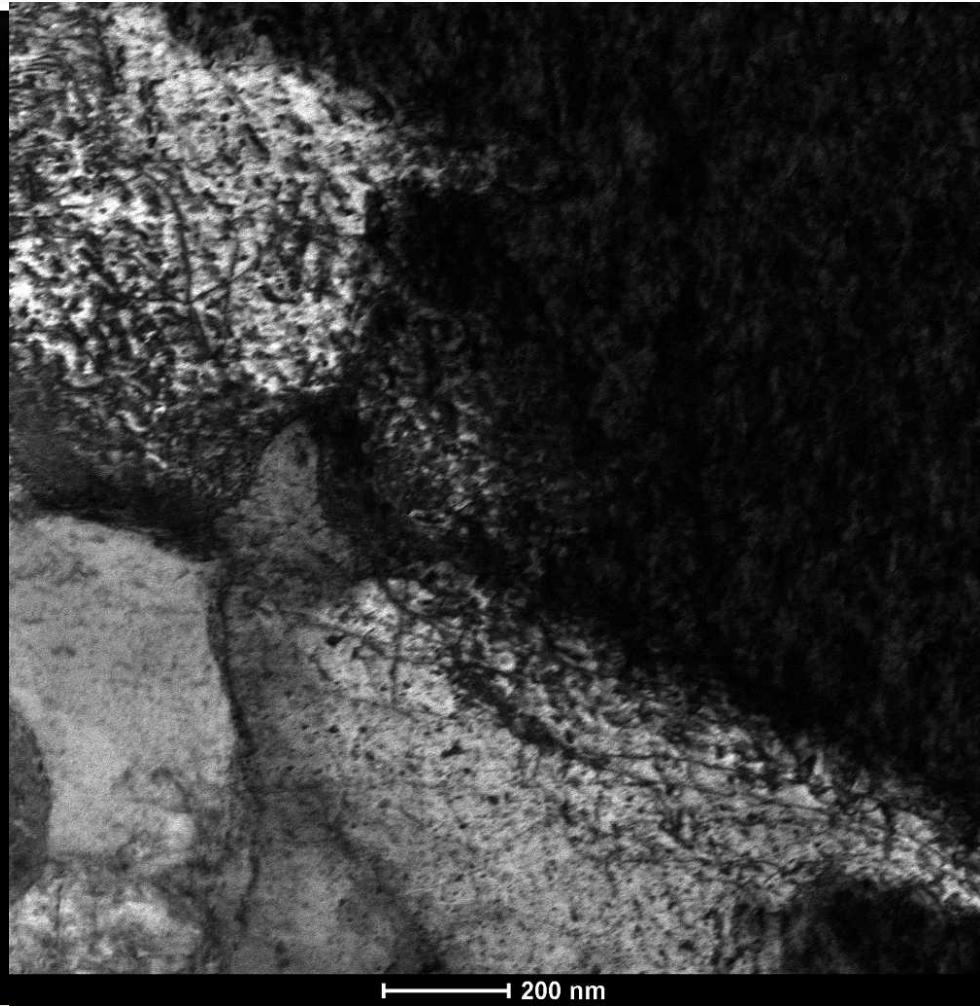
Persistent slip ladders are apparent in coarse grained metals, but have not yet been found in fatigue-coarsened grains

Coarse Grained Ni-Fe

Produced by annealing @ $650^{\circ}\text{C}/1\text{hr}$



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

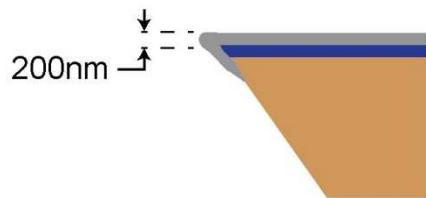
1. Start with Si wafer coated with nitride



2. Open window in nitride



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

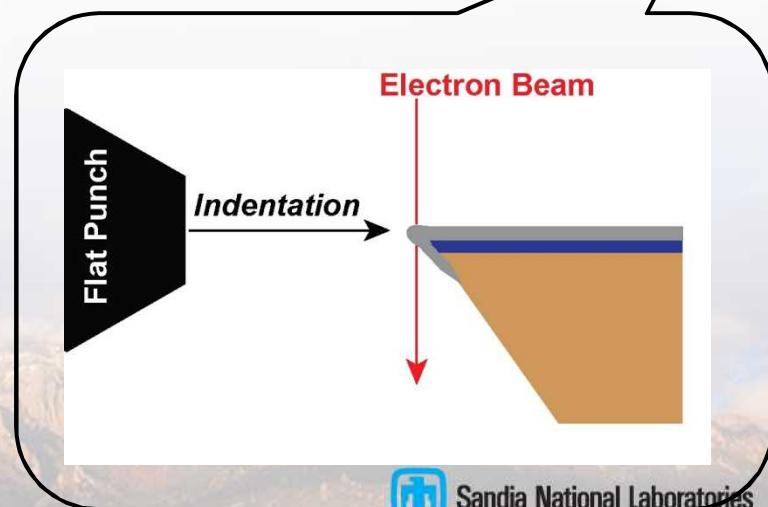


4. Pulse laser deposition of Al onto wedge



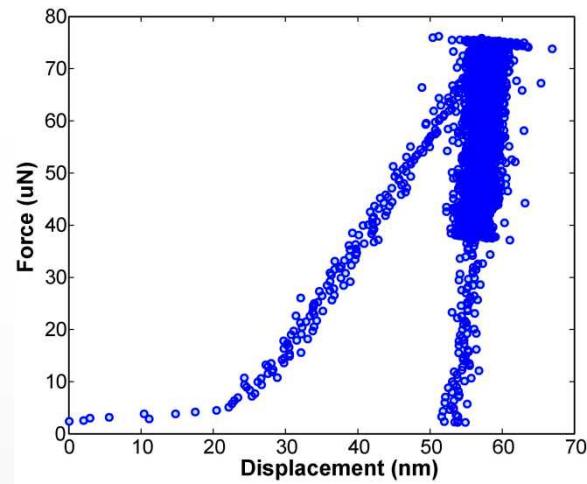
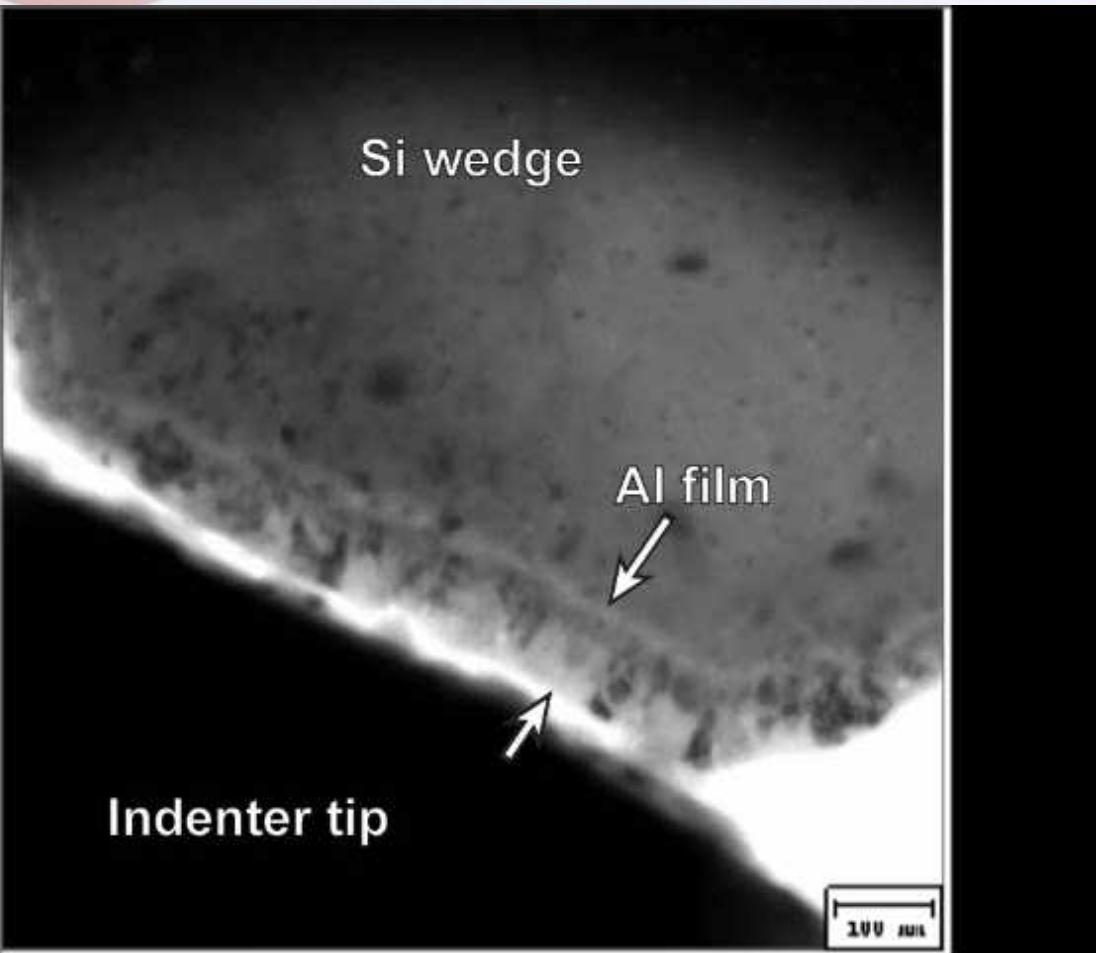
In situ TEM fatigue

- Microscope: JEOL JEM 2100 LaB_6 TEM
- Holder: Hysitron PI-95 PicoIndenter with a $1\mu\text{m}$ flat tip punch



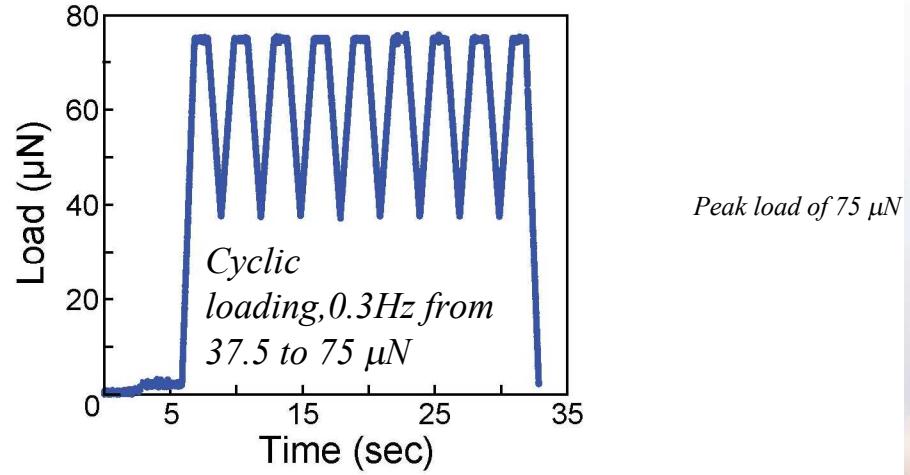
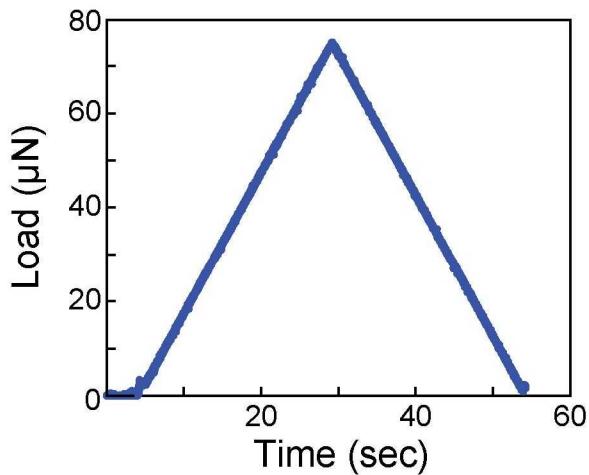
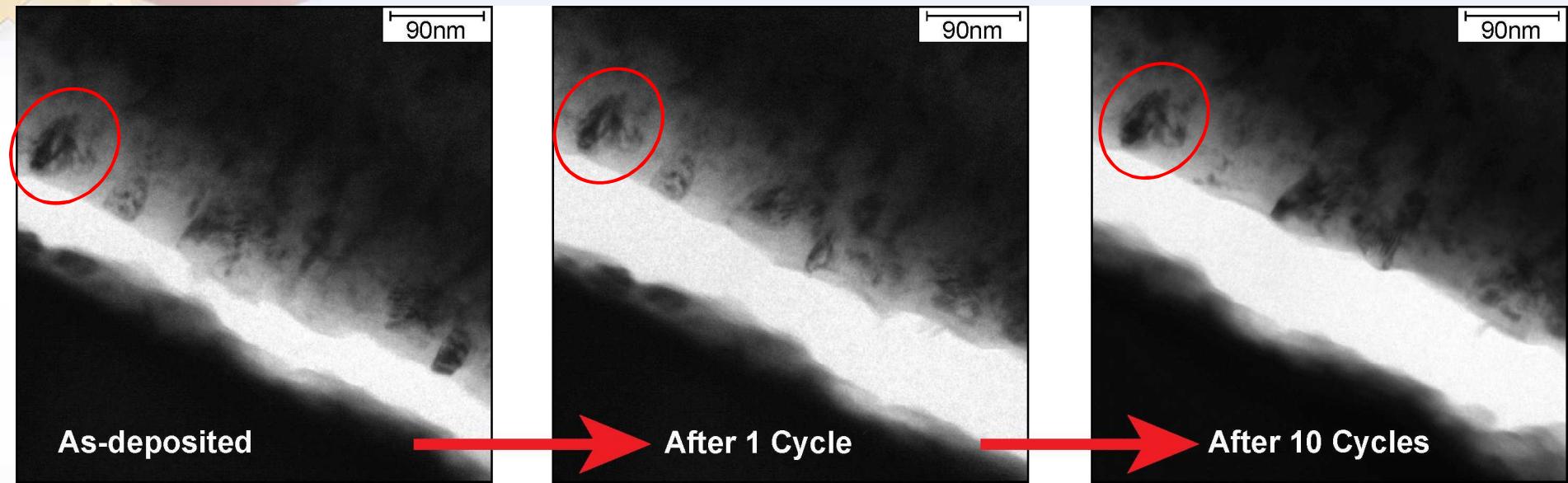
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In Situ TEM Indentation



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Some regions show no evolution

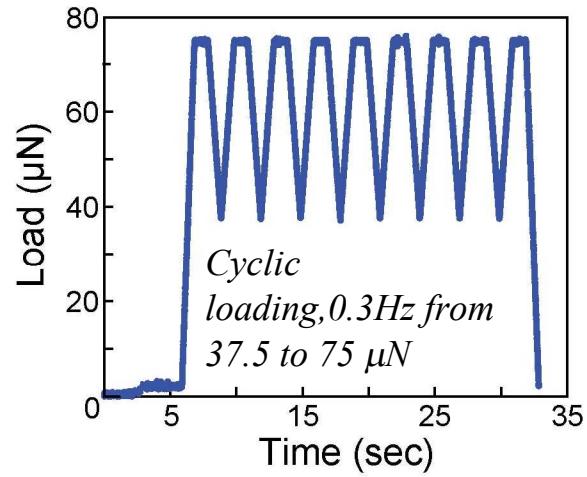
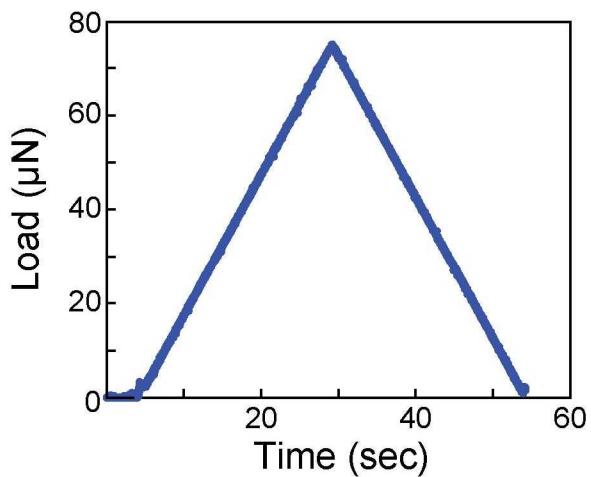
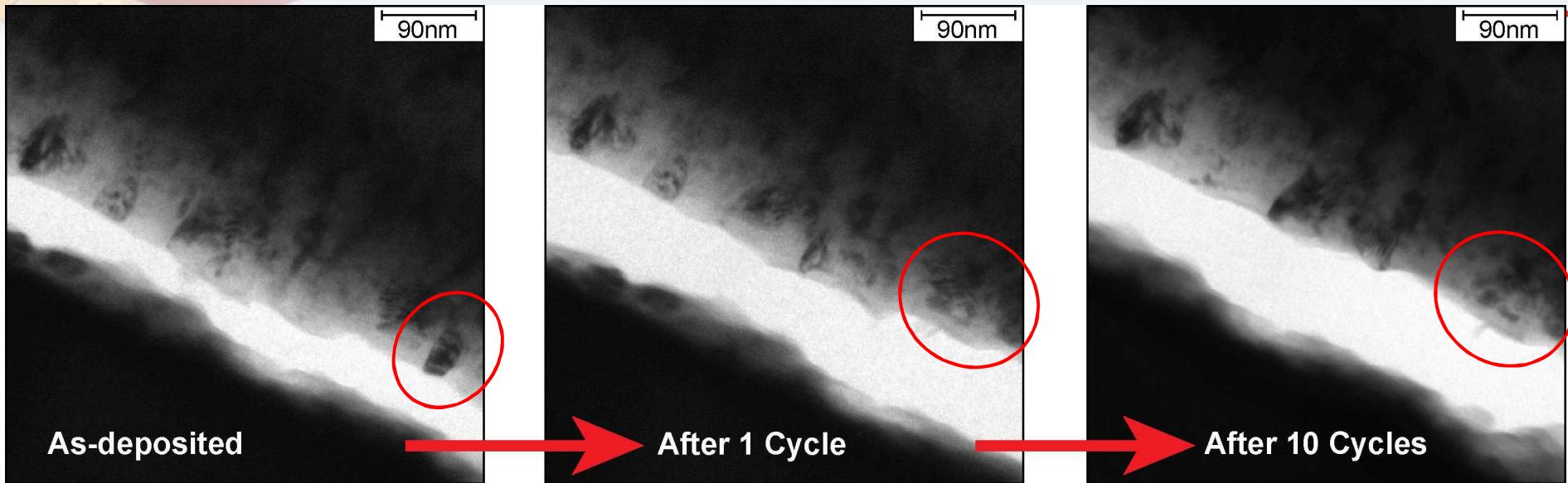


375 MPa nominal contact stress



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Some regions evolve during the 1st cycle

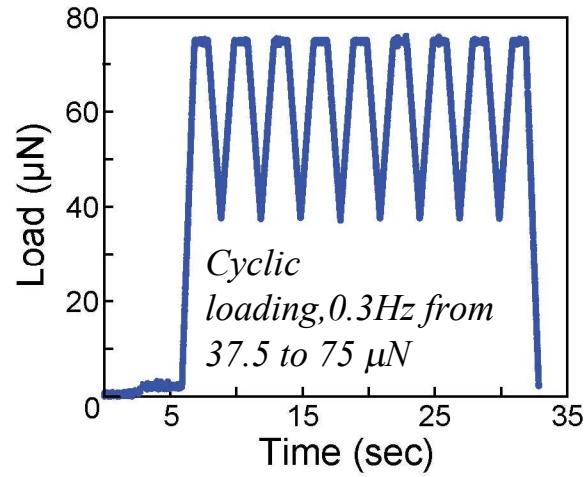
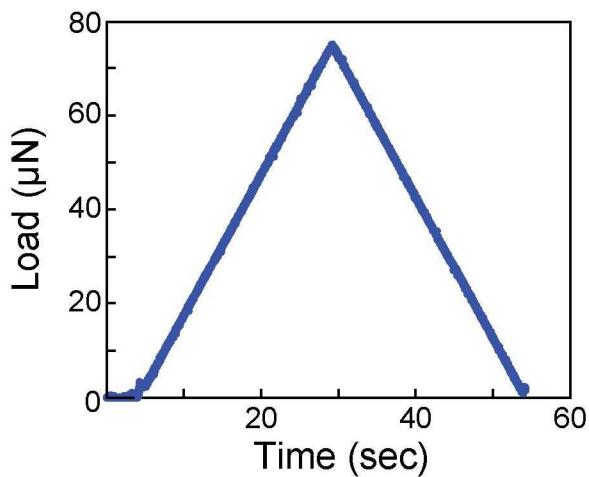
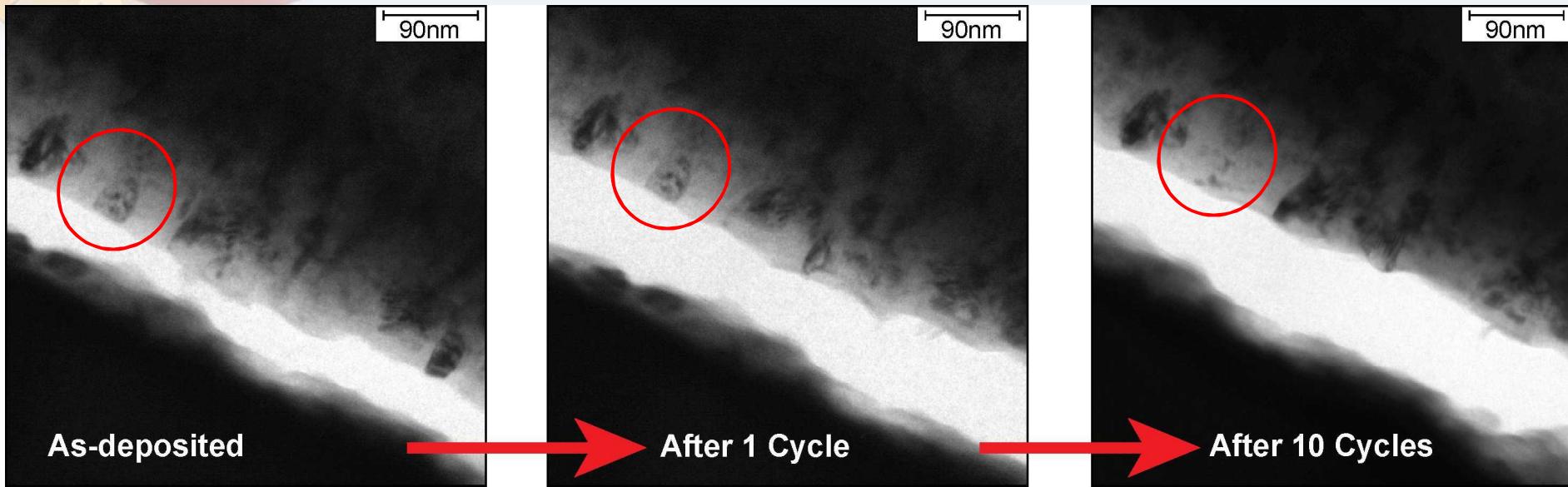


375 MPa nominal contact stress



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Some regions evolve during subsequent cycles



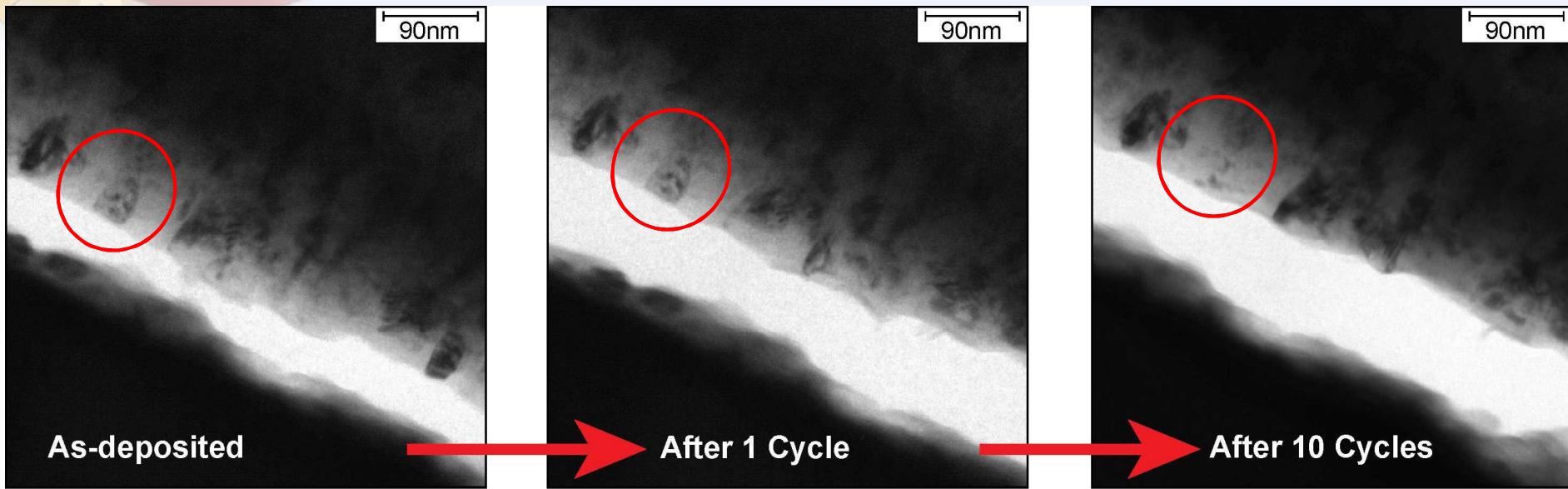
375 MPa nominal contact stress



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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.*

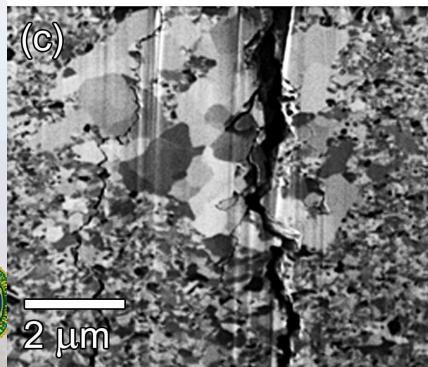
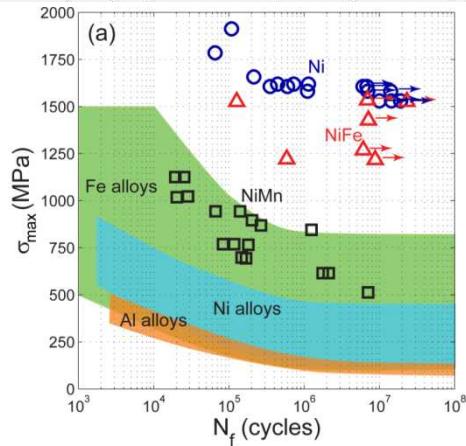


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Recap...



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.



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