



Fatigue Behavior in Three LIGA Electrodeposited Ni-based Alloys

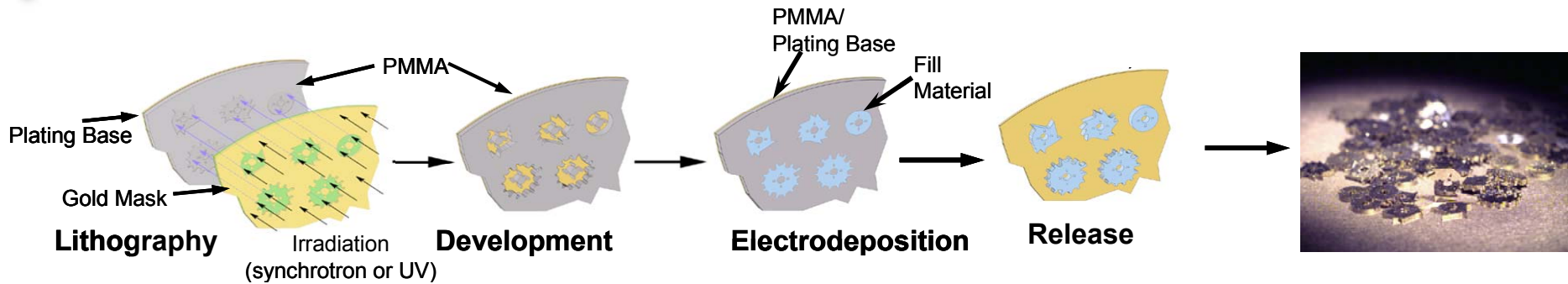
Brad L. Boyce, Joseph R. Michael, Paul G. Kotula

Sandia National Laboratories
Albuquerque NM and Livermore CA

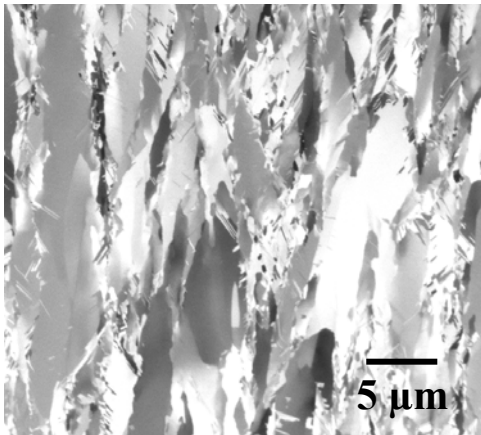
Sandia is a multiprogram laboratory operated by Sandia Corporation, a Lockheed Martin Company, for the United States Department of Energy under contract DE-AC04-94AL85000.



Three Ni-based LIGA MEMS Alloys

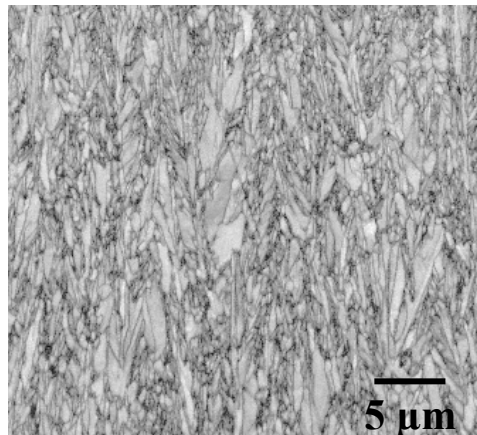


Ni (*sulfamate bath*)



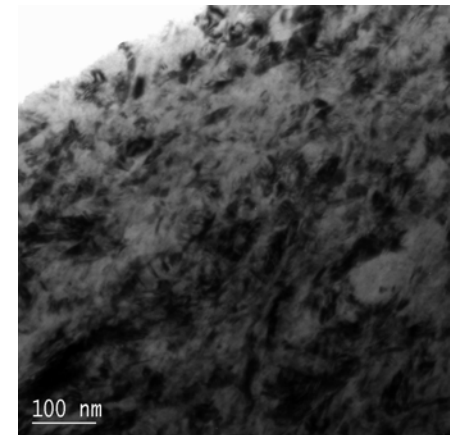
Grain Size ~ 3000 nm
Ultimate Strength ~ 500 MPa

Ni-Mn



Grain Size ~ 100 nm
Ultimate Strength ~ 1500 MPa

Ni-Fe



Grain Size < 50 nm
Ultimate Strength ~ 2000 MPa

Fatigue Testing of Metallic (LIGA) MEMS

Applied Loads:

~15 mN

Deflections:

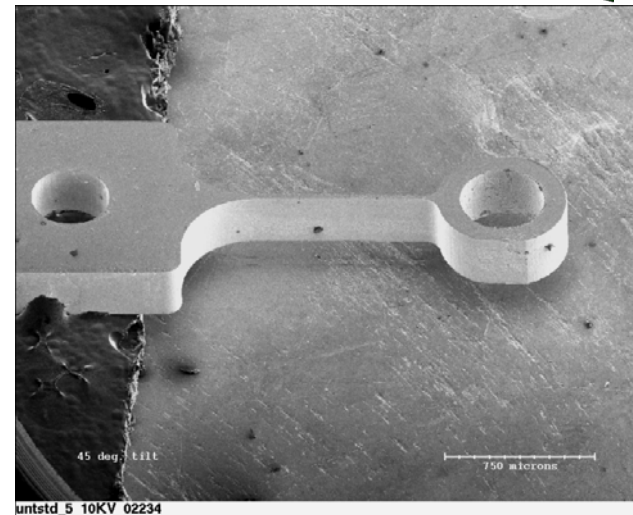
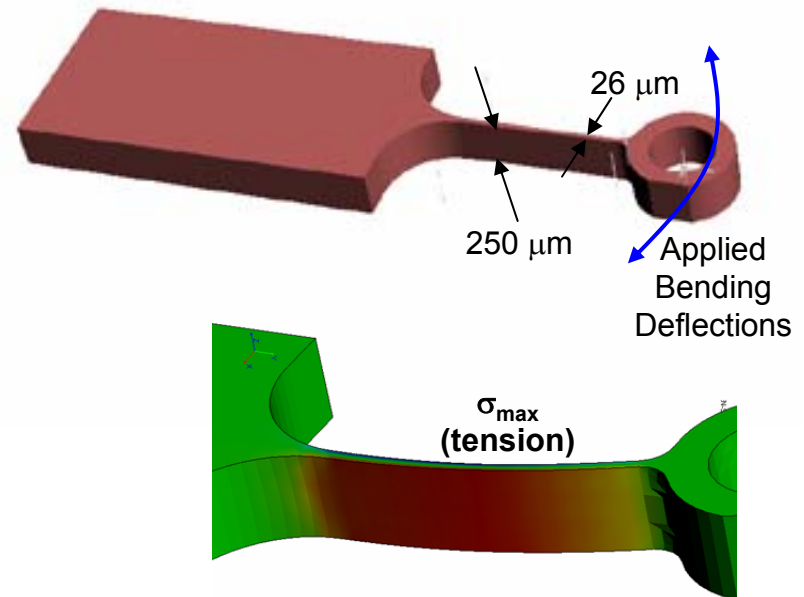
~100 μm

Test Frequency

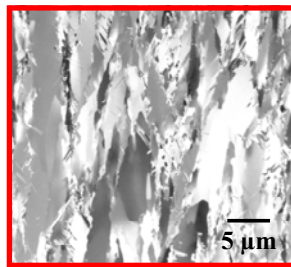
10-20 Hz

Room Temp.

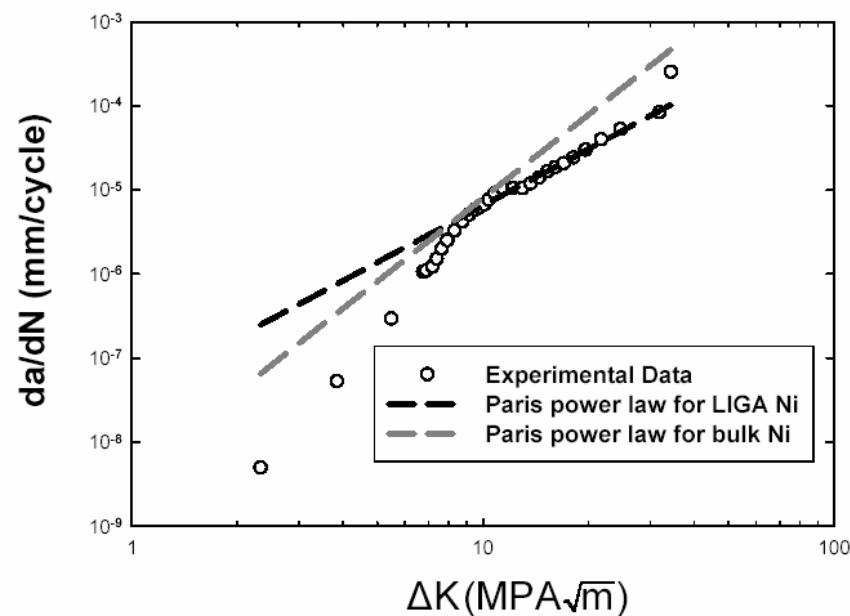
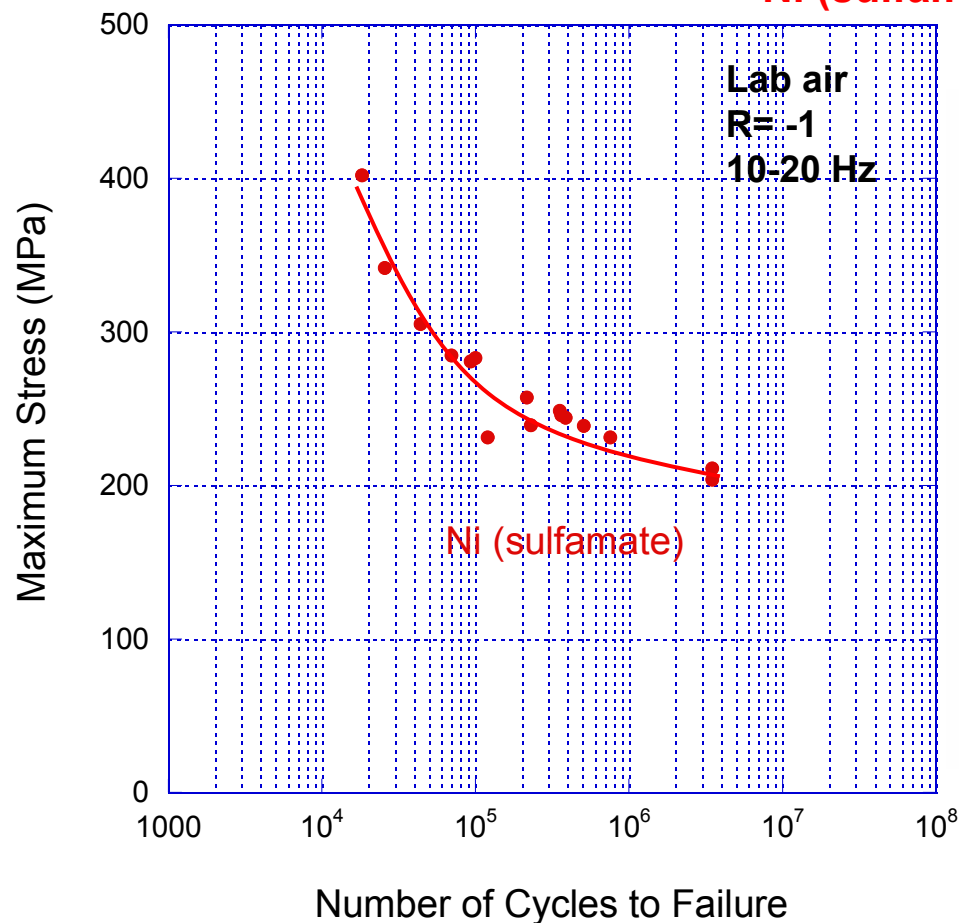
Lab Air



High Cycle S-N Results On LIGA Ni

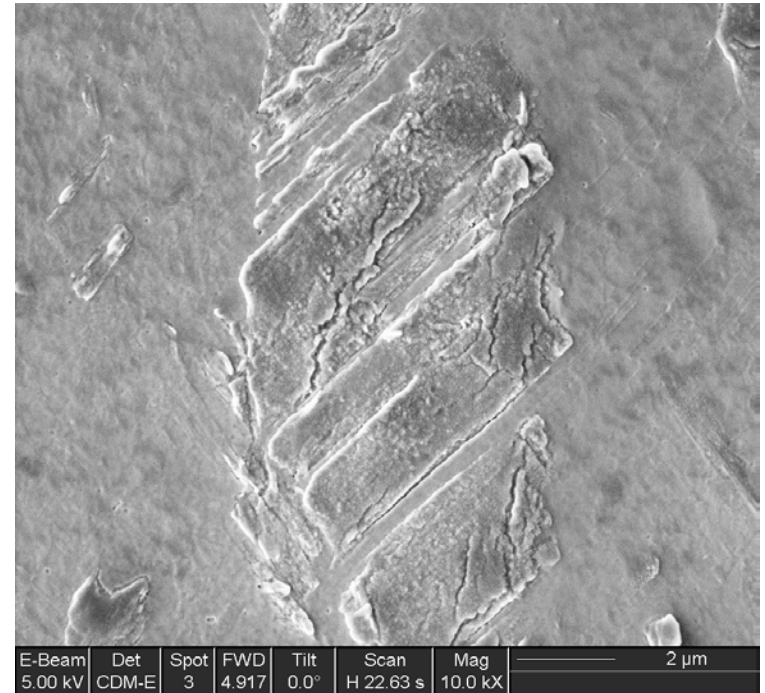
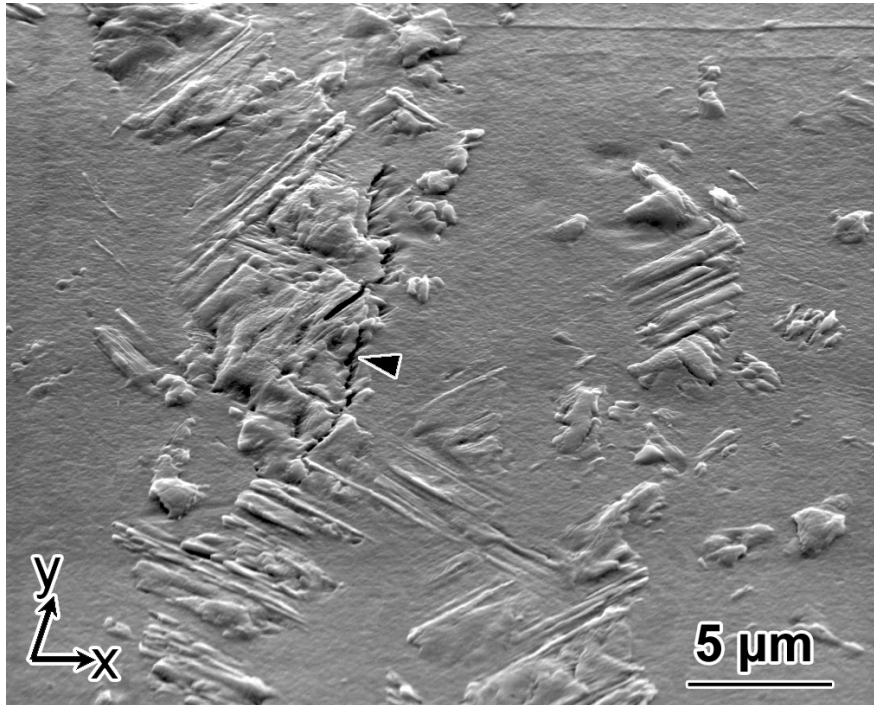


Ni (sulfamate)

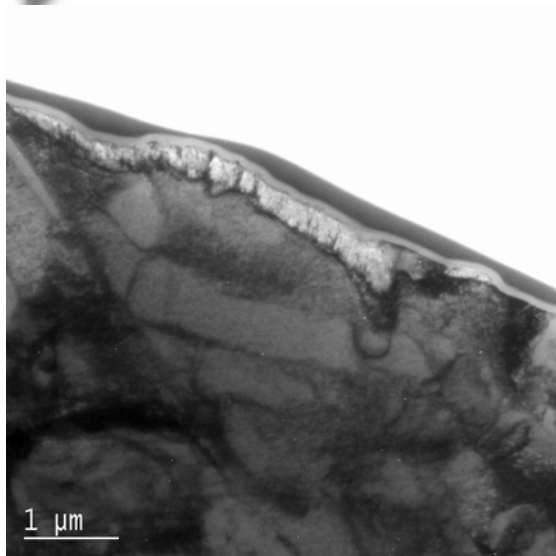


Y. Yang, S. Allameh, J. Lou, B. Imasogie,
B.L. Boyce and W.O. Soboyejo, Metall. Trans. A.,
In Review

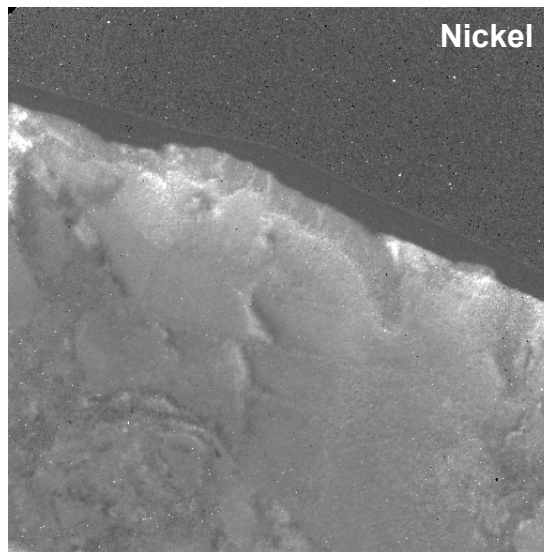
Cracks Are Found in Persistent Slip Bands On the Surface



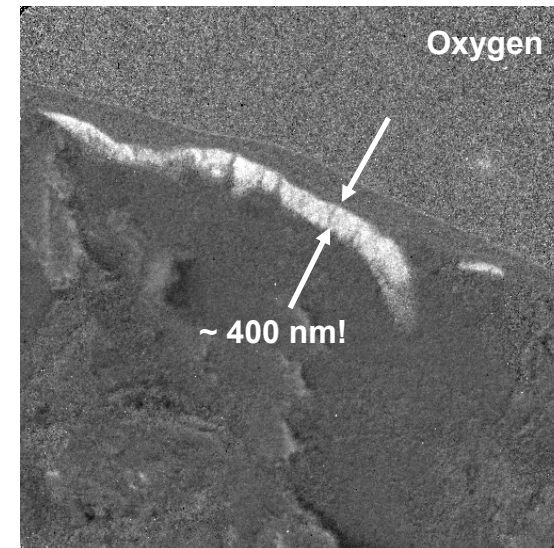
These Surface Layers Are Covered in A Thick Oxide!



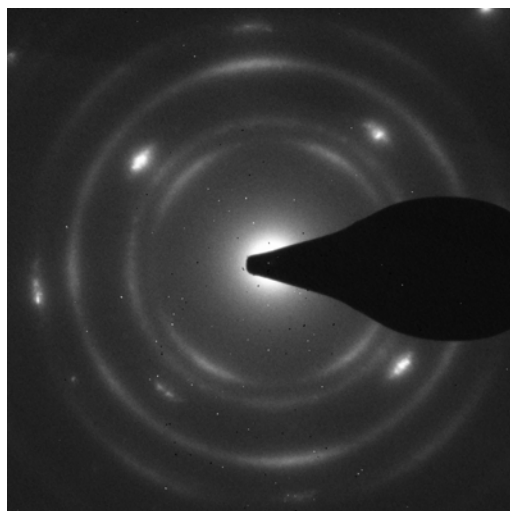
Bright Field Image



**White intensity scales
with intensity of nickel
peaks in EELS spectra**



**White intensity scales
with intensity of
oxygen peaks in EELS
spectra**



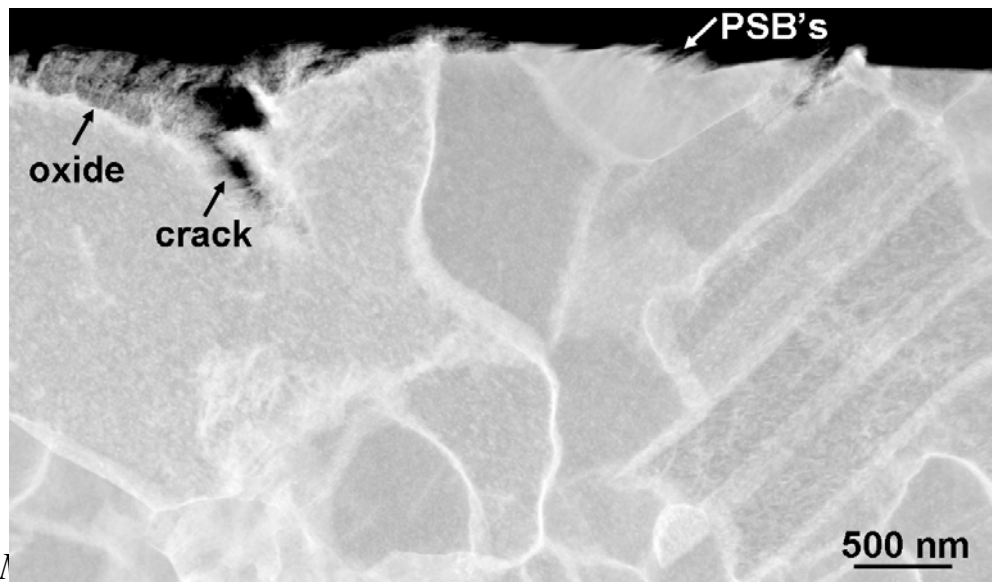
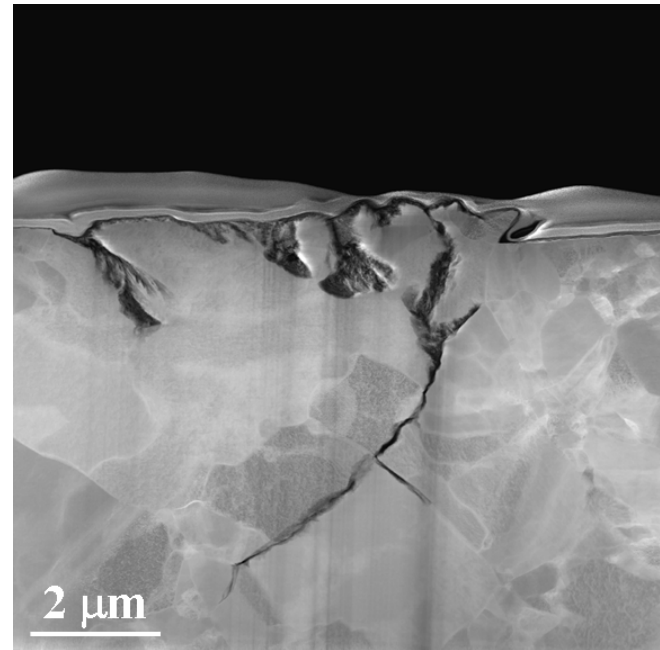
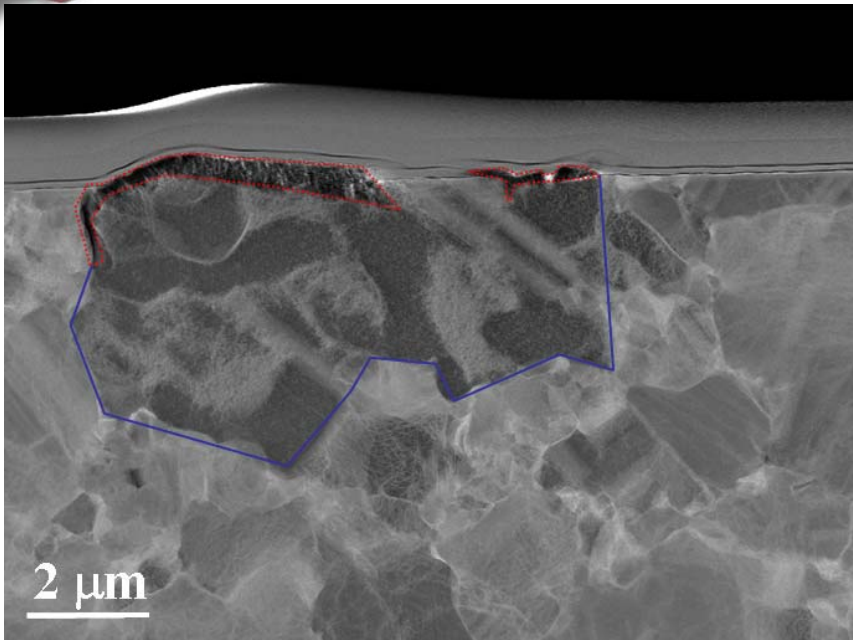
Selected Area Diffraction
Dept. 1824 Microsystems Materials

Boyce, Kotula, Michael, *Acta Materialia*, 2004



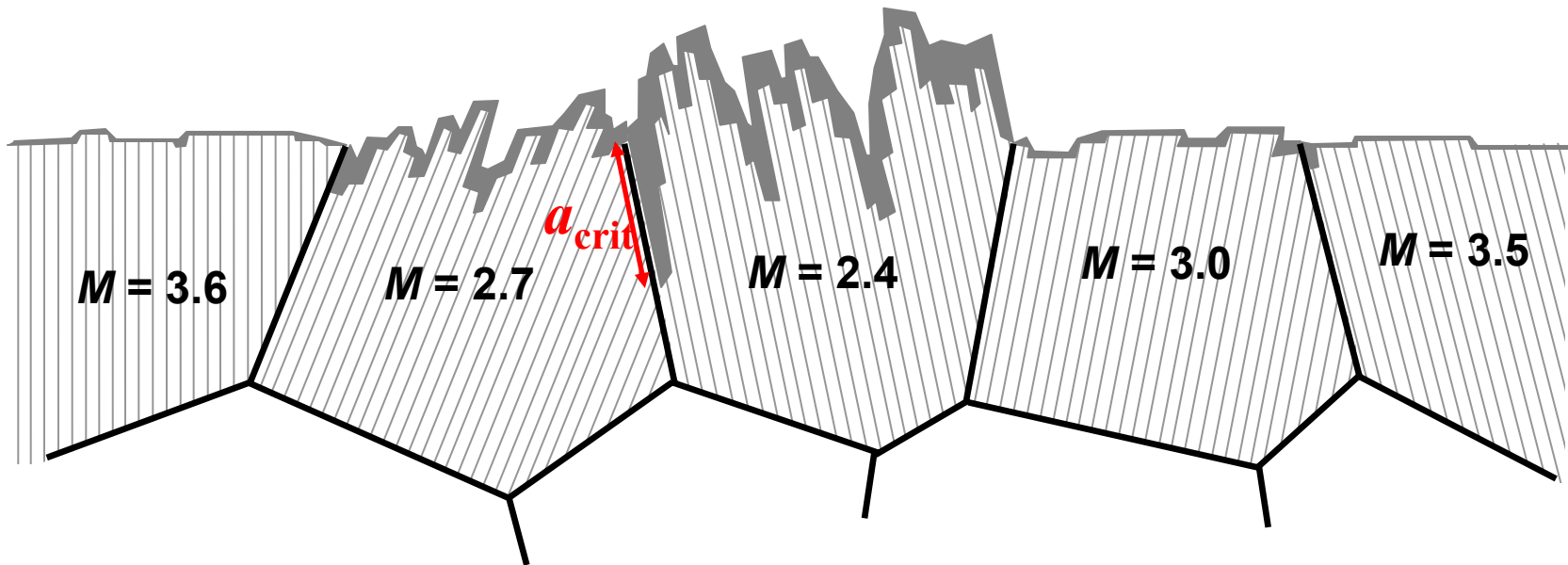
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Cross-sectional TEM of PSBs and Oxide Microcracks

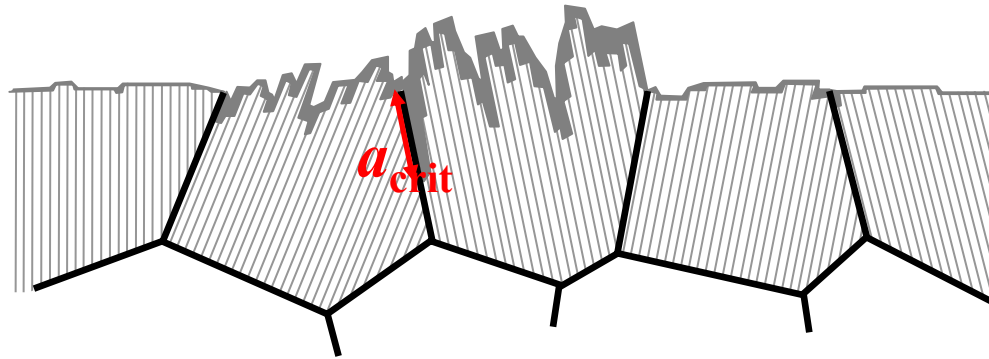


INITIATION: A Transition from Oxide Growth to Fatigue Crack Propagation

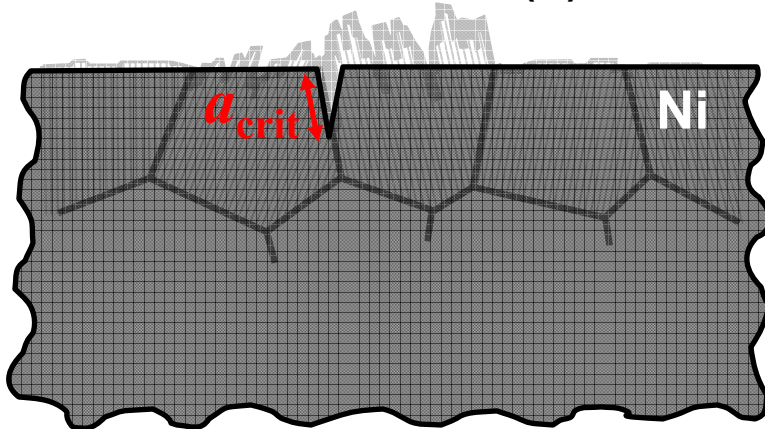
Hypothesis: The fatigue cycles serve to cyclically rupture the repassivated film. Once an oxide of sufficient thickness is grown, the imbedded flaws are large enough to exceed the fatigue threshold, allowing a crack to propagate into the matrix



Two Bounding Solutions for “PSB Oxide Initiation”



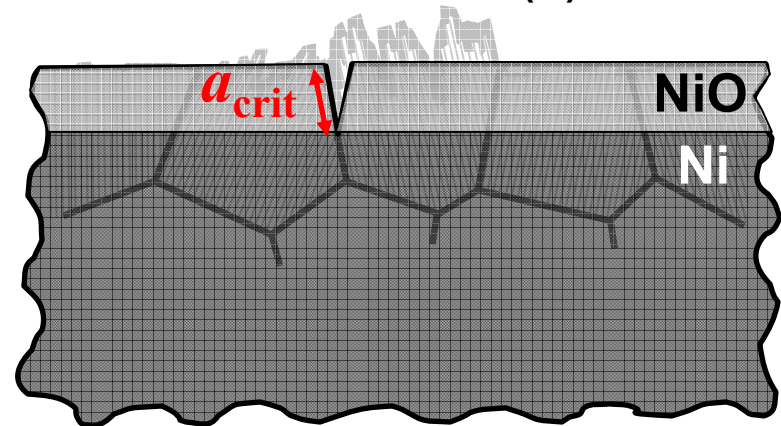
APPROXIMATION (A):



A crack in a homogeneous monolith



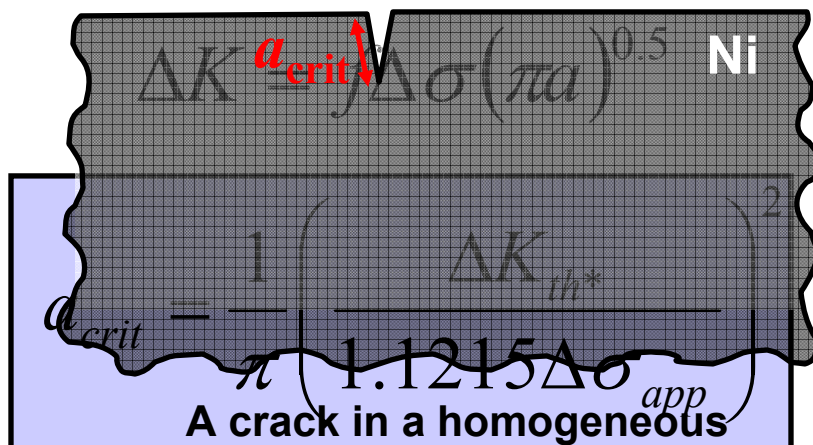
APPROXIMATION (B):



A fully-cracked film penetrating into the substrate

Two Bounding Solutions for “PSB Oxide Initiation”

APPROXIMATION (A):

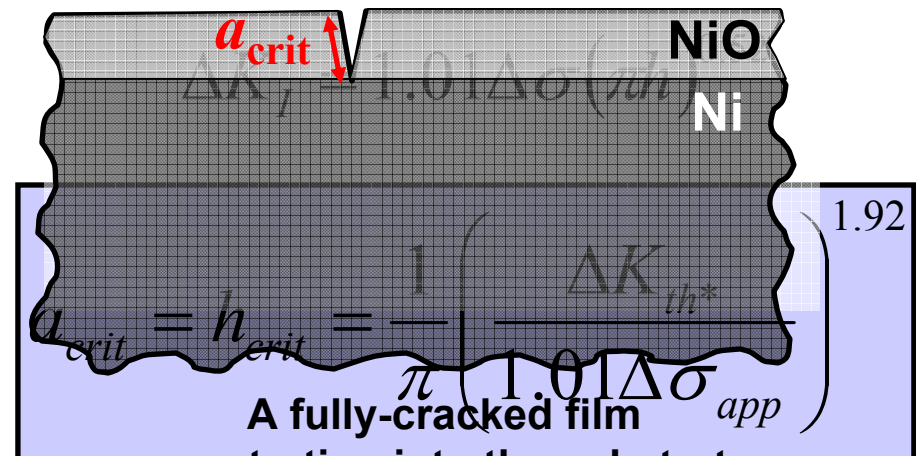


$$\Delta K = \Delta \sigma (\pi a)^{0.5}$$

$$a_{crit} = \frac{1}{\pi} \left(\frac{\Delta K_{th}^*}{1.1215 \Delta \sigma_{app}} \right)^2$$

A crack in a homogeneous monolith

APPROXIMATION (B):



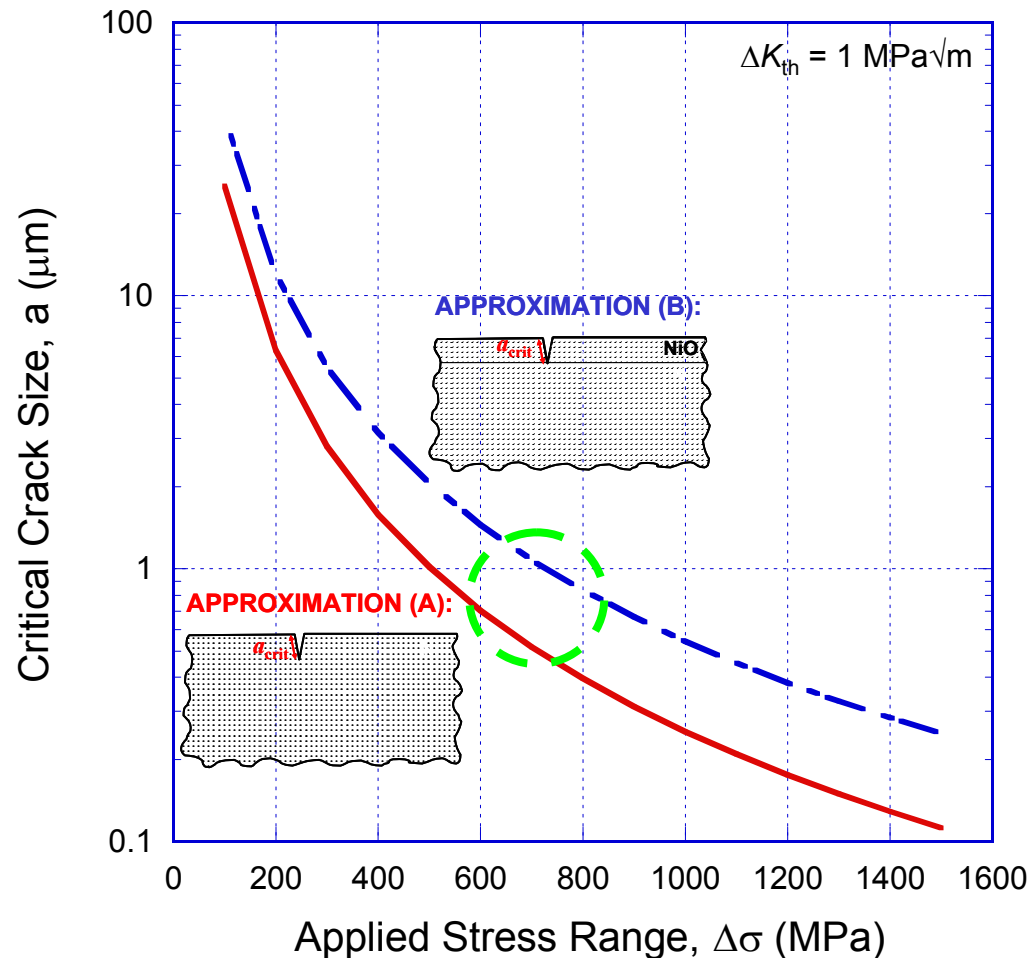
$$\Delta K_I = 1.01 \Delta \sigma (\pi h)^{0.5}$$

$$a_{crit} = h_{crit} = \frac{1}{\pi} \left(\frac{\Delta K_{th}^*}{1.01 \Delta \sigma_{app}} \right)^{1.92}$$

A fully-cracked film penetrating into the substrate

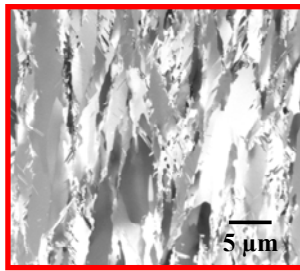
Ye, Suo, and Evans; Hutchinson and Suo (1992); Bleuth (1991)

Critical Oxide Thickness Depends on Applied Stress

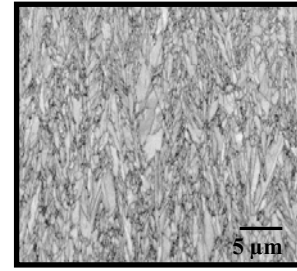


These estimates predict a critical oxide thickness in the vicinity of 600-1200 nm, depending on the applied stress levels.

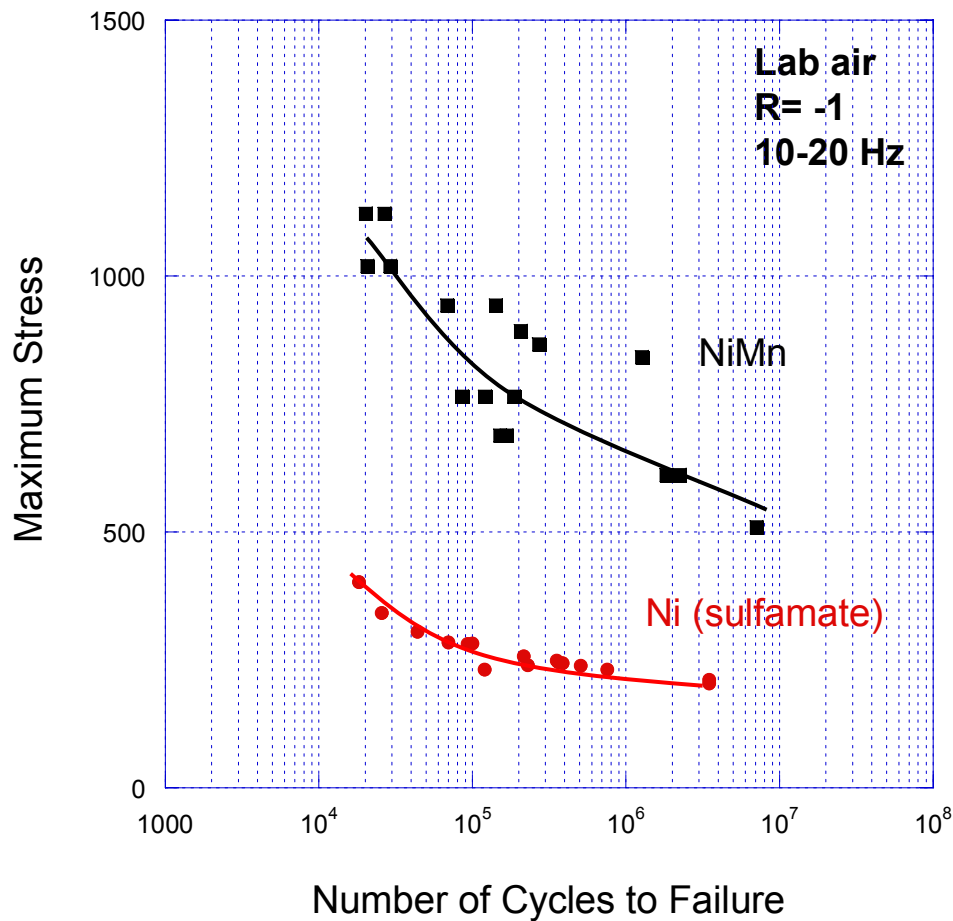
Fatigue Behavior: Two Ni Alloys



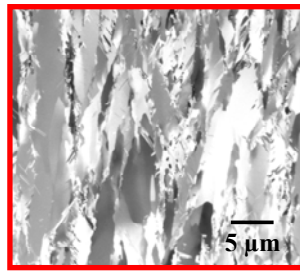
Ni



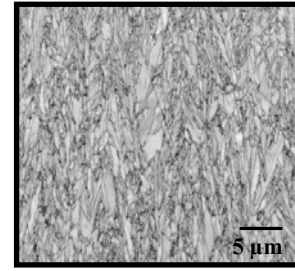
NiMn



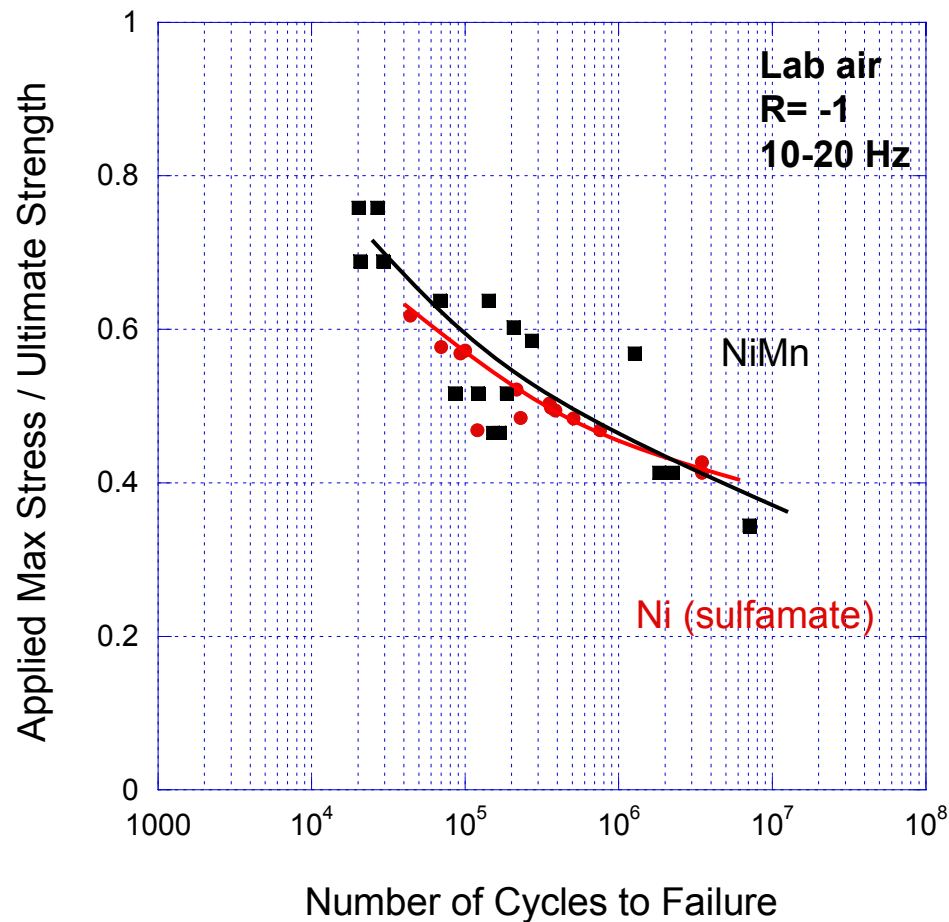
Fatigue: Normalized S-N Results



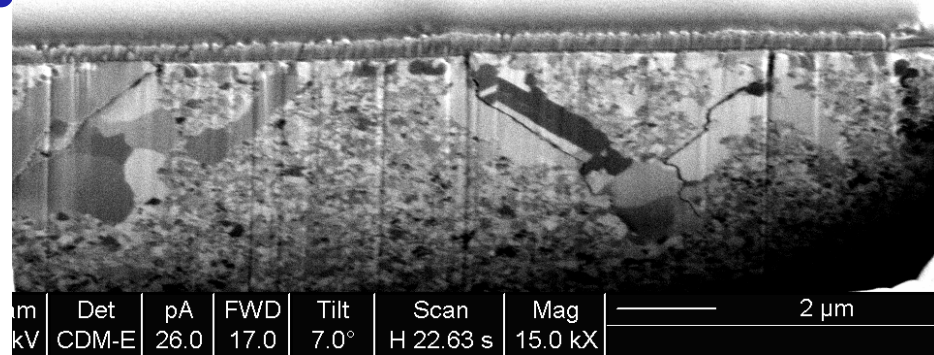
Ni



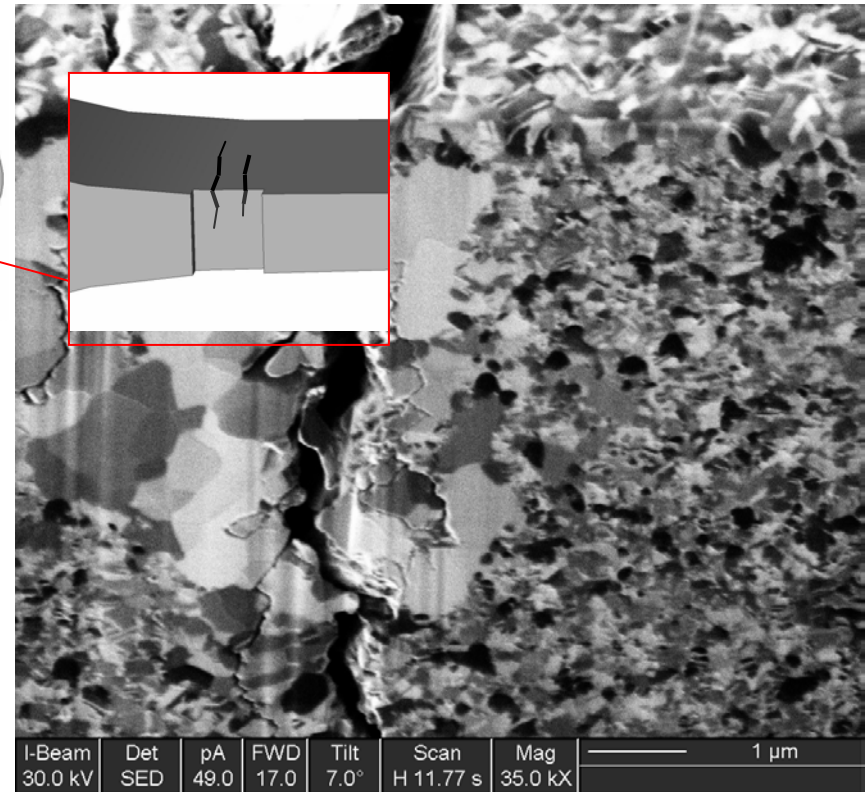
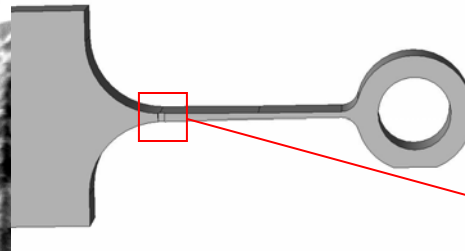
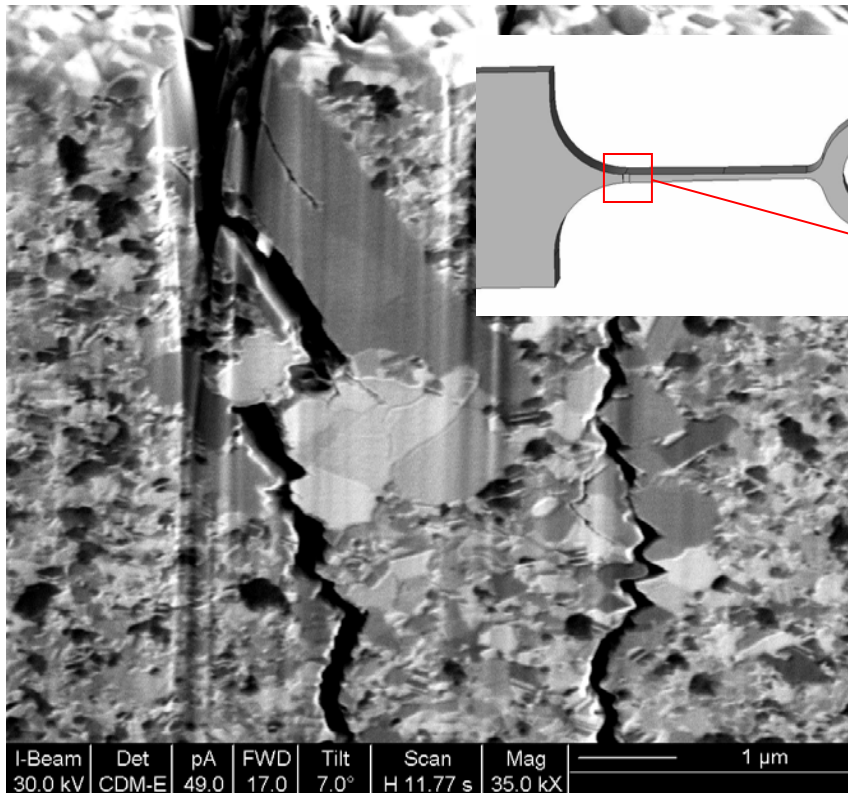
NiMn



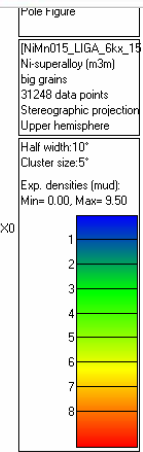
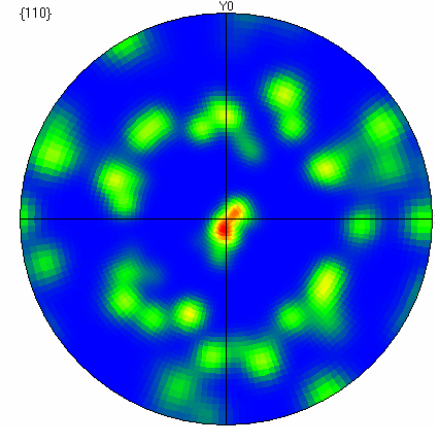
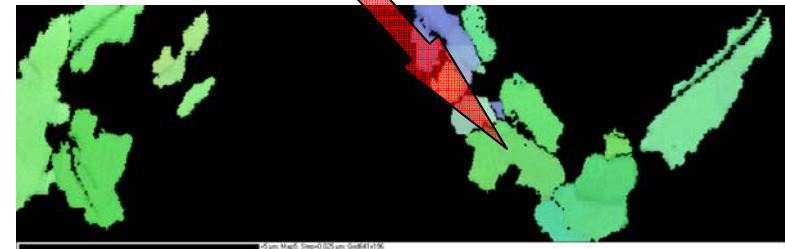
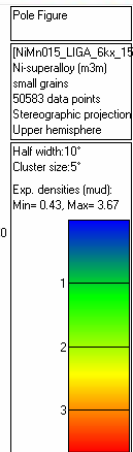
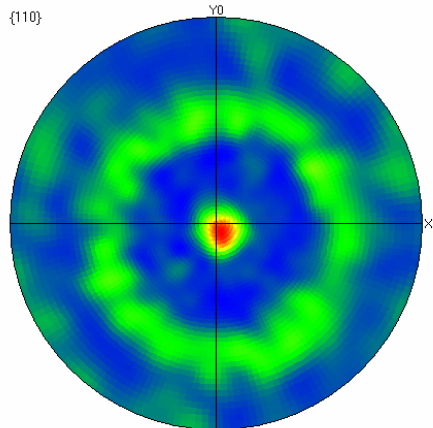
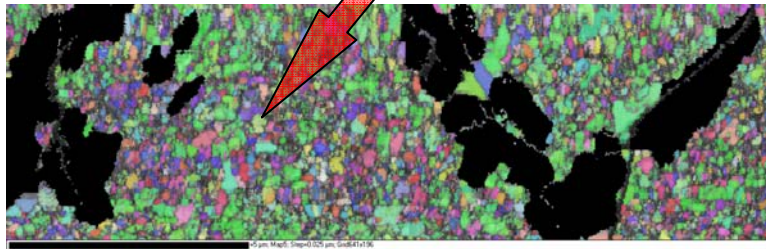
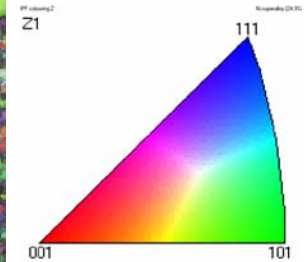
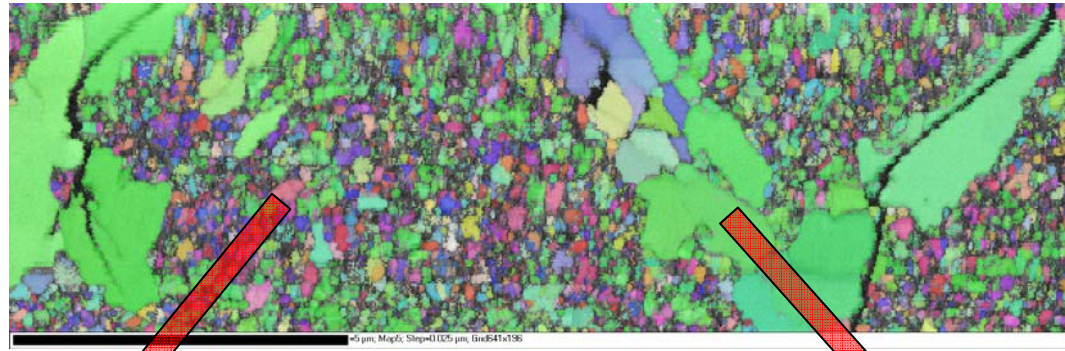
Fractography after Failure



Subsurface microstructure revealed by FIB excavation and Ion Channeling Contrast Imaging



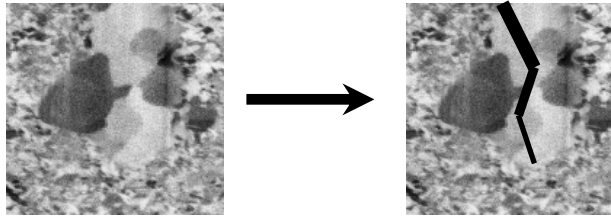
Large Grains Retain (110) Deposition Texture



Three Scenarios for Coarsening+Initiation

1

Deposition causes coarse grains.
Initiation prefers coarse regions.



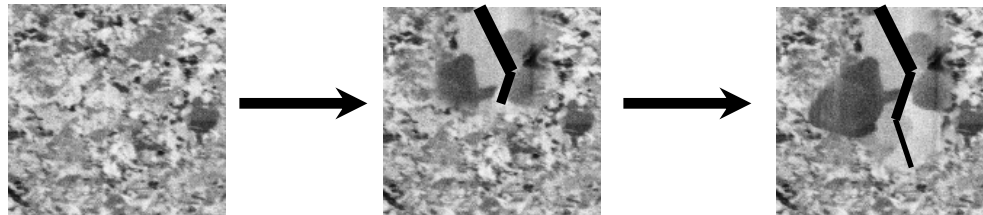
2

Fatigue stresses cause coarse grains.
Subsequent initiation prefers coarse regions.



3

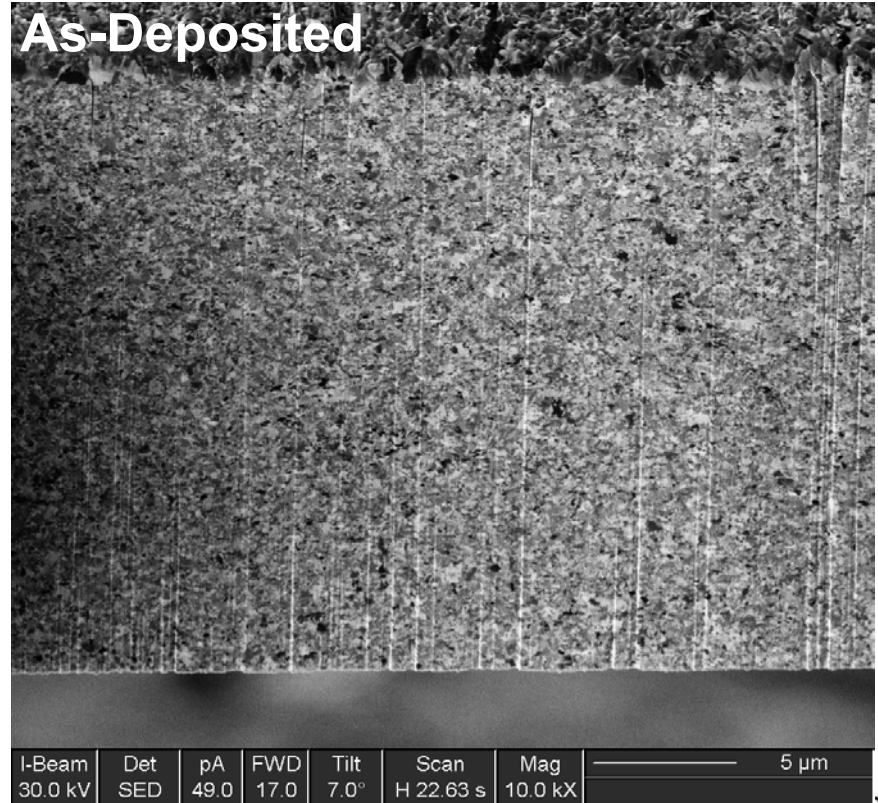
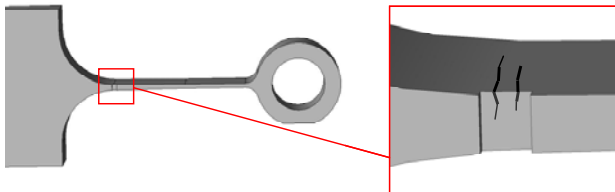
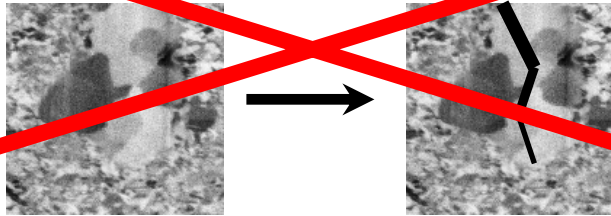
Cracks initiate first.
Crack tip stress fields cause coarsening.



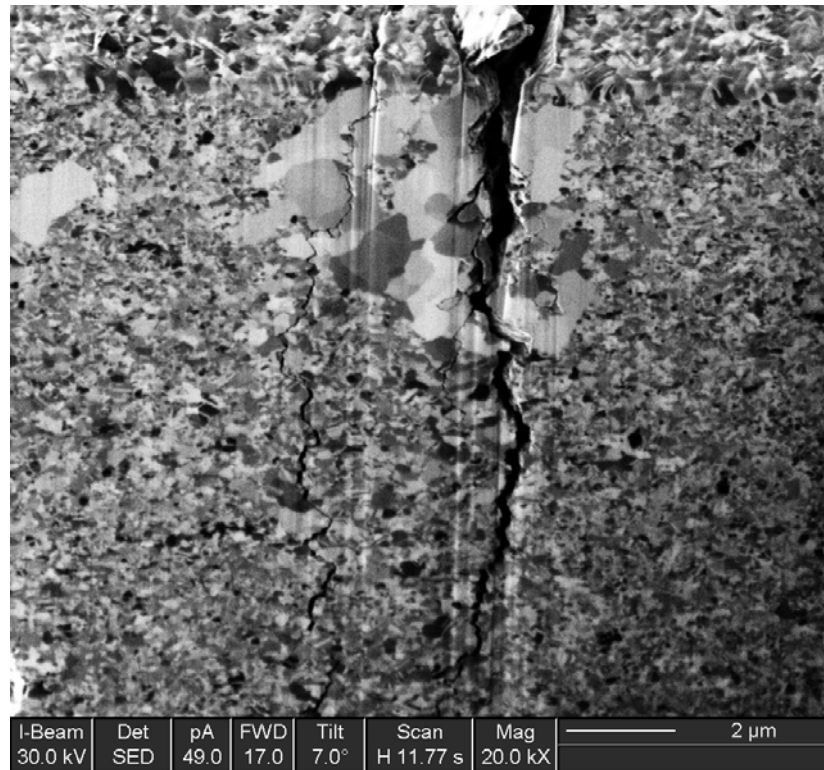
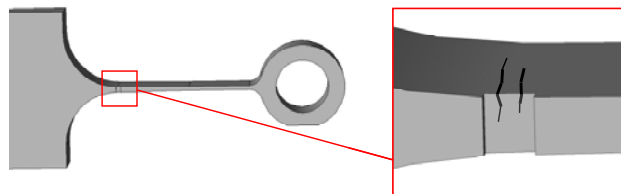
Three Scenarios for Coarsening+Initiation

1

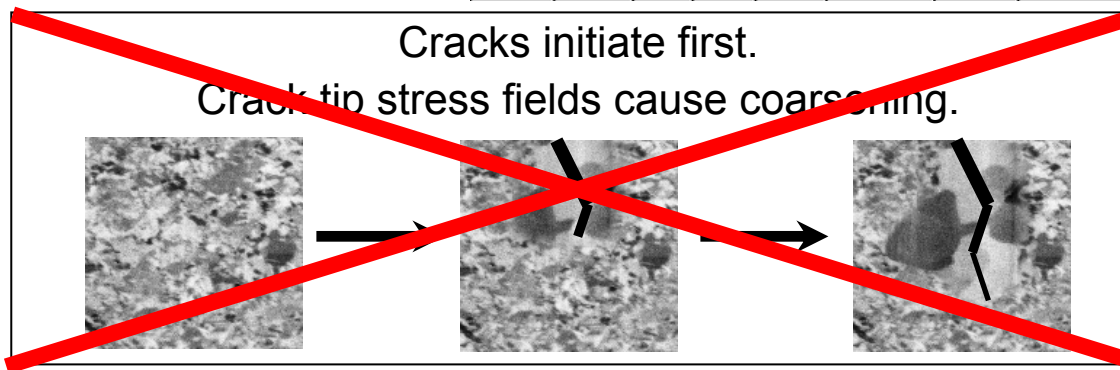
Deposition causes coarse grains.
Initiation prefers coarse regions.



Three Scenarios for Coarsening+Initiation



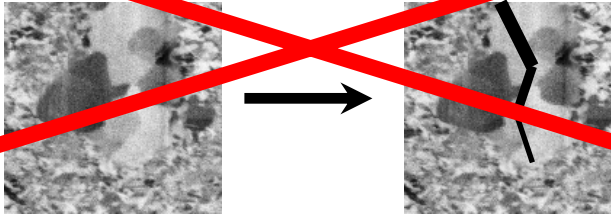
3



Three Scenarios for Coarsening+Initiation

1

Deposition causes coarse grains.
Initiation prefers coarse regions.



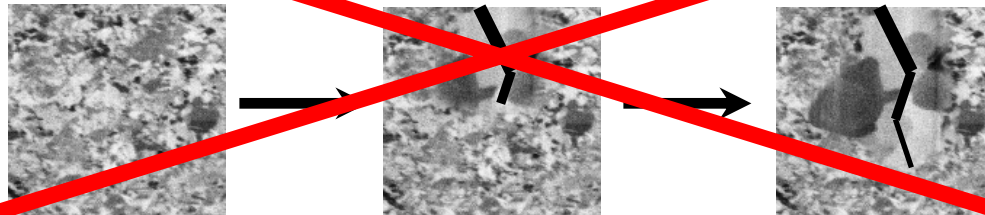
2

Fatigue stresses cause coarse grains.
Subsequent initiation prefers coarse regions.



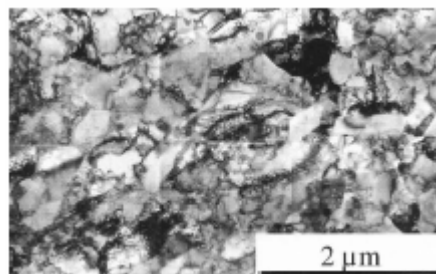
3

Cracks initiate first.
Crack tip stress fields cause coarsening.

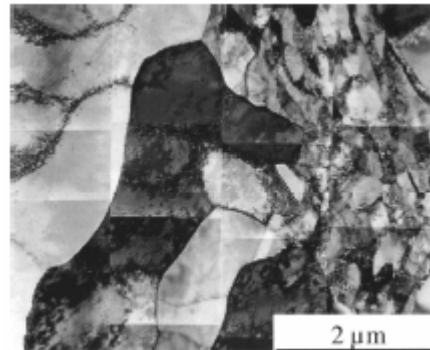


Other Examples of Mechanical Coarsening

High Cycle Fatigue of UltraFine Grained Copper (99.99%)

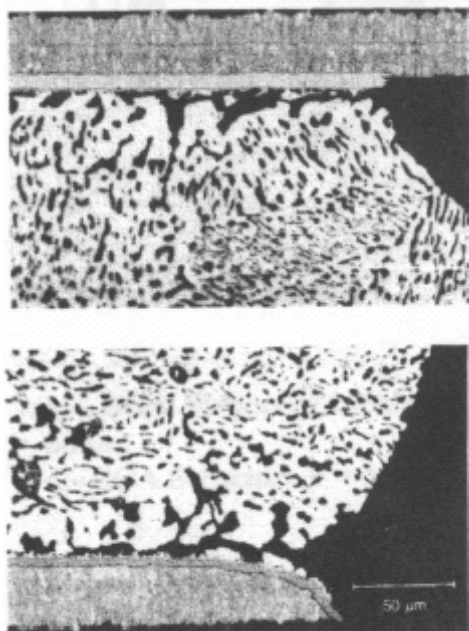


fatigue



Höppel, Zhou, Mughrabi, and Valiev, 2002

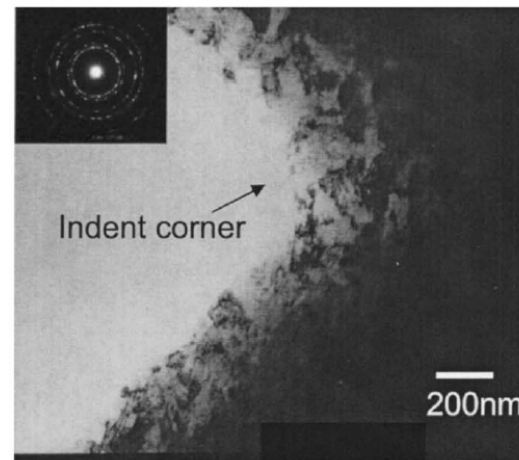
Thermomechanical Fatigue of Solder Alloys



Muller and Dreyer, *Int J Solids Struct*, 2000

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Monotonic Indentation of Nanocrystalline Copper (99.999%)

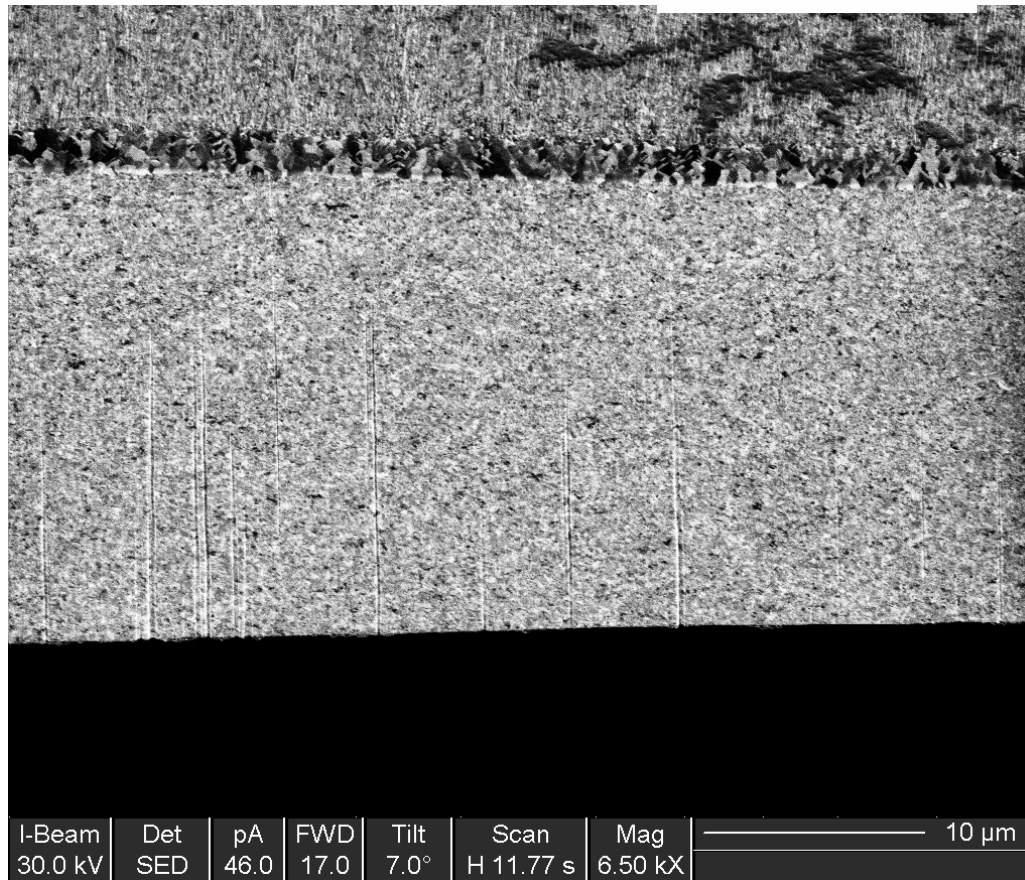
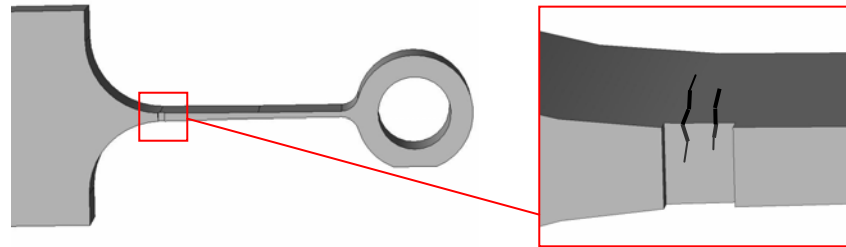


Zhang, Weertman, and Eastman, 2003

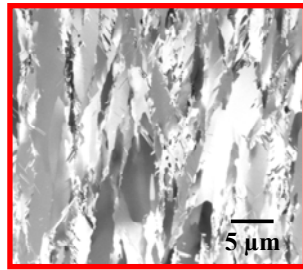


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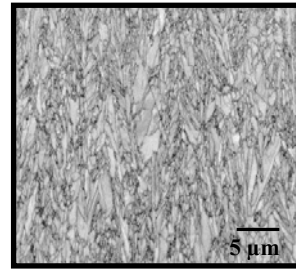
After a Single Fatigue Cycle, No Coarsening Is Found



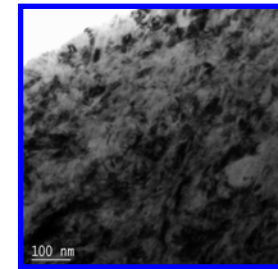
Fatigue Comparison: All 3 Alloys



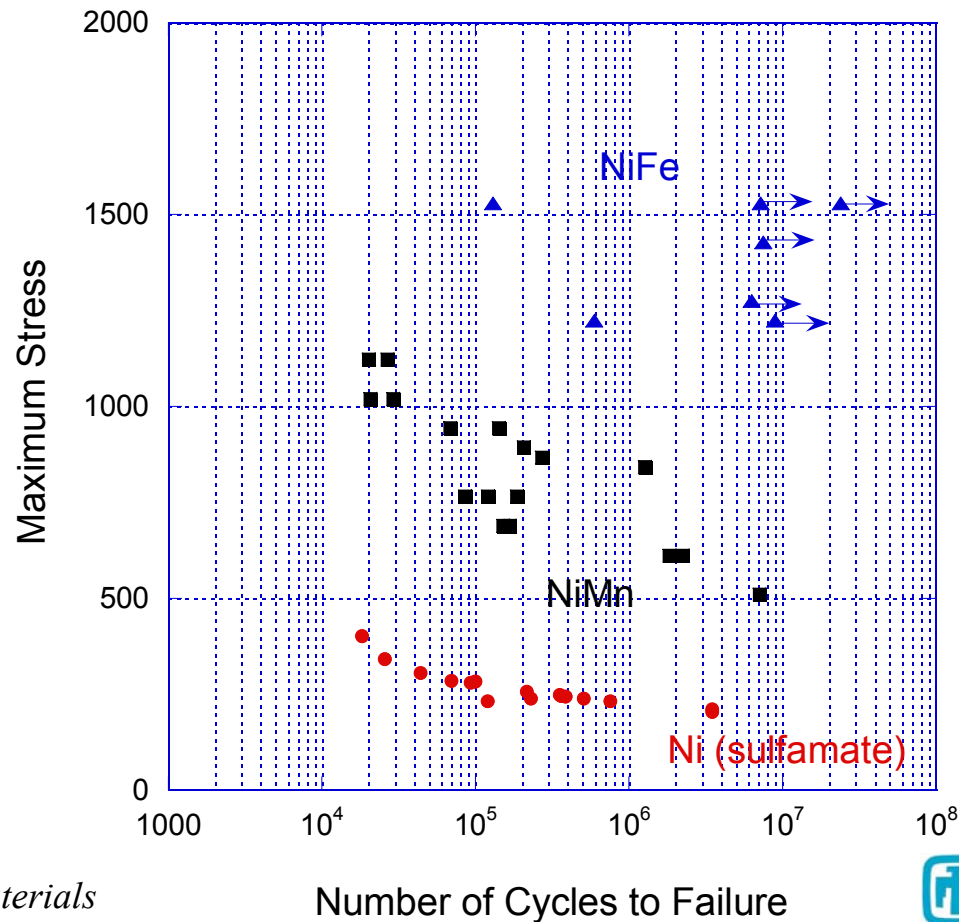
Ni



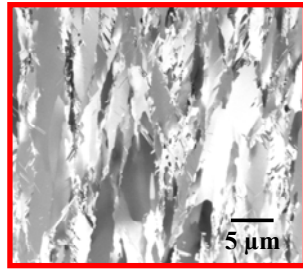
NiMn



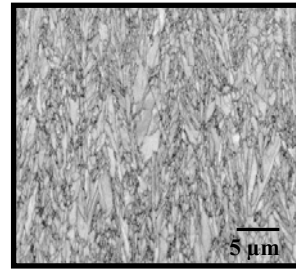
NiFe



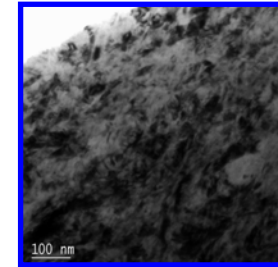
Fatigue Comparison: All 3 Alloys



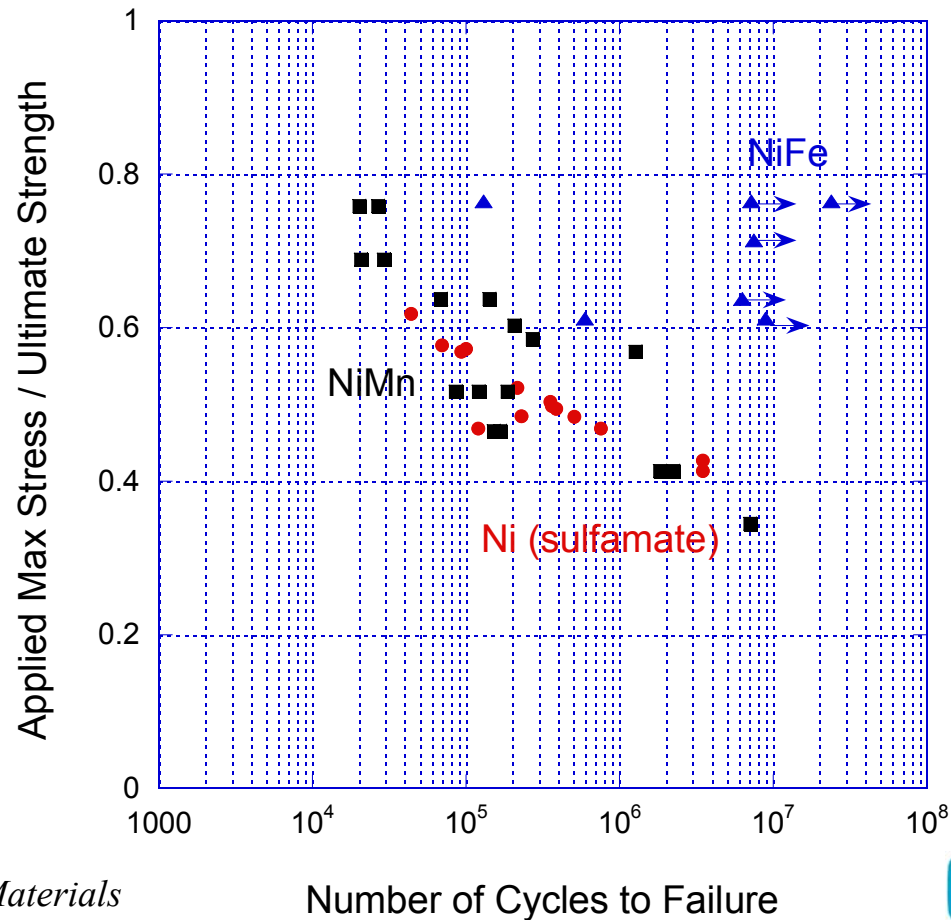
Ni



NiMn



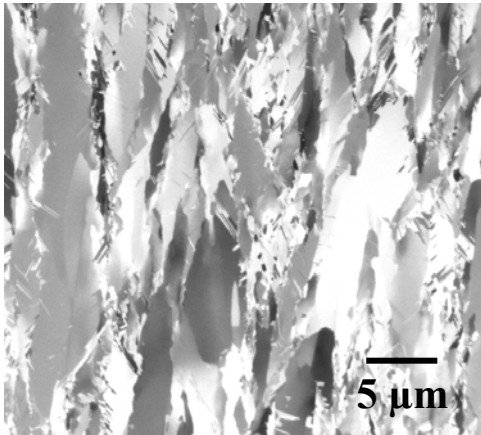
NiFe



Summary: Fatigue Behavior in Three LIGA Electrodeposited Ni-based Alloys

Ni (sulfamate bath)

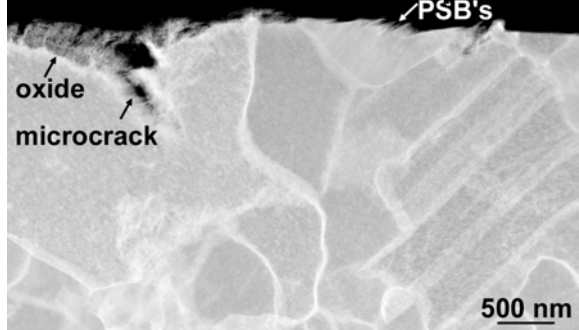
Grain Size ~ 3000 nm



“Normal S-N Behavior”:

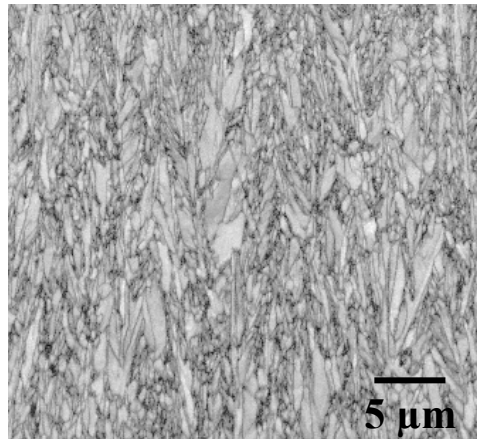
$$\sigma_{\text{fat}} / \sigma_{\text{uts}} = 0.35$$

Oxidation of Persistent Slip Bands



Ni-Mn

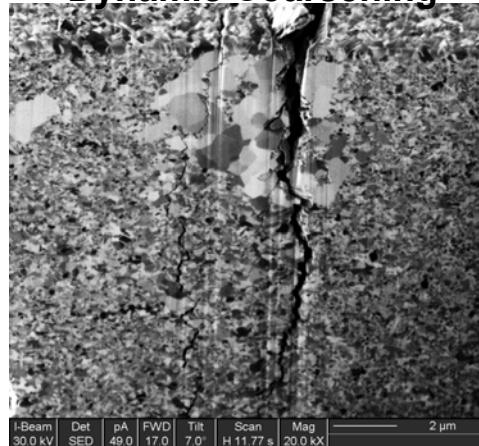
Grain Size ~ 100 nm



“Normal S-N Behavior”:

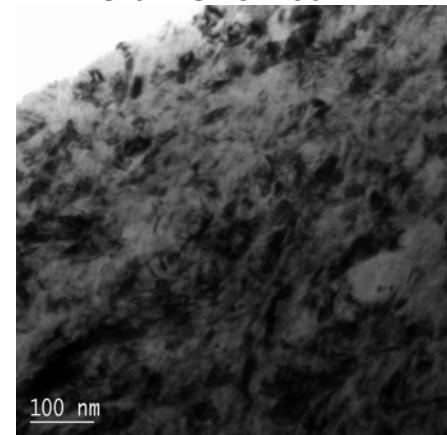
$$\sigma_{\text{fat}} / \sigma_{\text{uts}} = 0.35$$

Dynamic Coarsening



Ni-Fe

Grain Size < 50 nm



“Enhanced S-N Behavior”:

$$\sigma_{\text{fat}} / \sigma_{\text{uts}} \gg 0.35$$



(Boyce, Michael and Kotula, *Acta Mater.*, 2004)

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