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Fatigue-induced Abnormal Grain Growth in Nanocrystalline Metals: an *in-situ* Synchrotron XRD Approach

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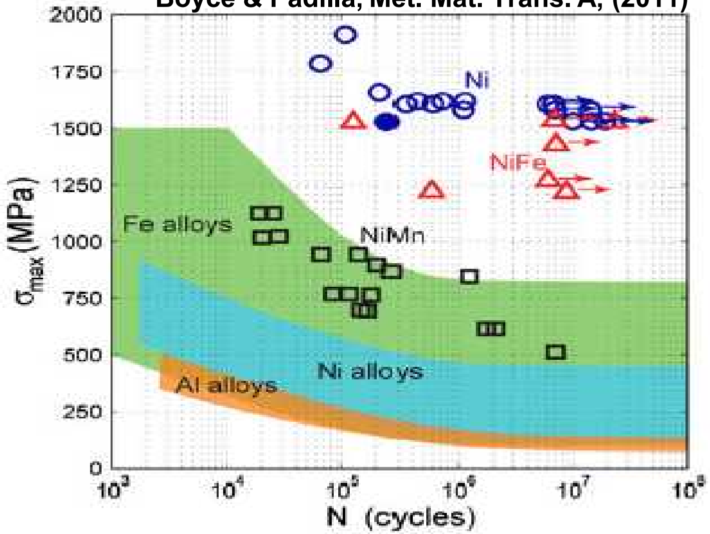
Nanocrystalline metals show unique mechanical behaviors, including a general propensity for mechanical grain growth

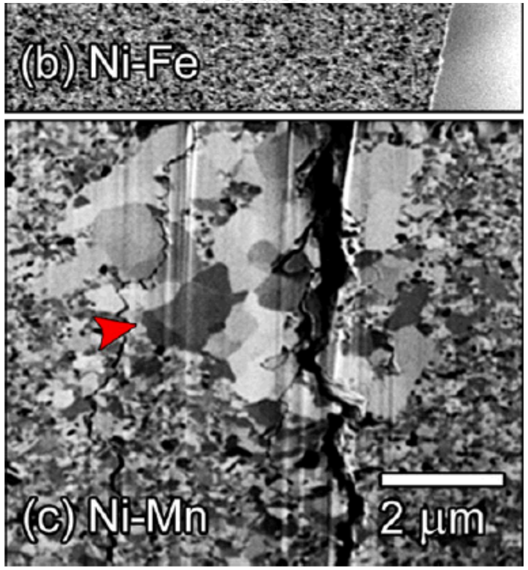
- Typical properties of NC metals include:
  - Ultra-high strength & hardness
  - High wear-resistance
  - **Ultra-high fatigue strengths**
- When grains are at the nanoscale, unique mechanical properties and plastic responses arise, including **mechanically-induced grain growth**
- In fatigue, ***abnormal* grain growth** has been suspected to lead to **crack initiation**

- Post-fracture techniques leave us with a “chicken and egg” problem (fracture or AGG?)
- Additionally, are these abnormally large grains pre-existing (grown during deposition) or cyclically-induced?

Boyce & Padilla, Met. Mat. Trans. A, (2011)

Boyce & Padilla, Met. Mat. Trans. A, (2011)





Post-fracture analysis can only tell us so much – to understand the role of abnormal grain growth on the fatigue deformation and crack initiation, non-destructive *in-situ* techniques are required.

Transmission synchrotron x-ray diffraction can provide non-destructive means to detect AGG in a NC matrix

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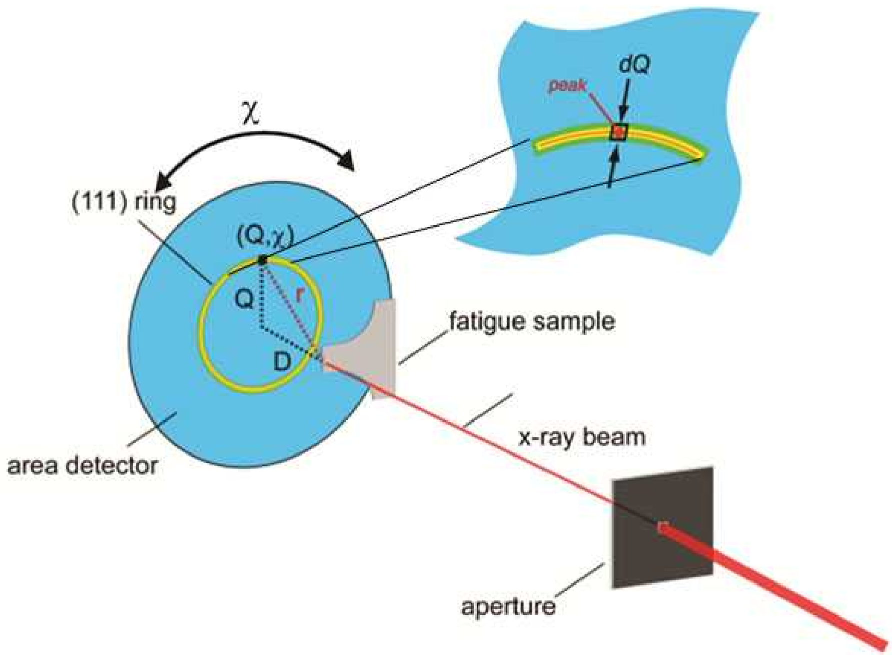
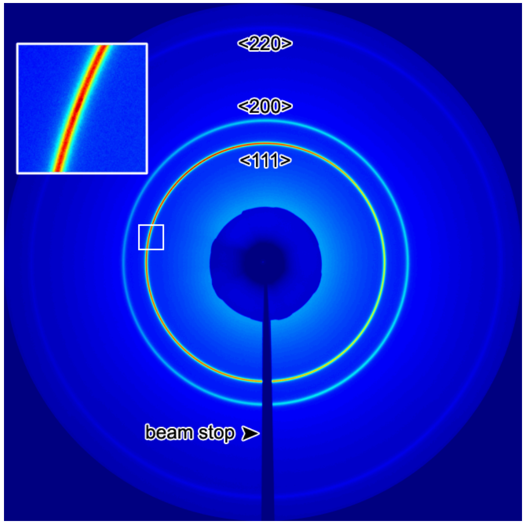
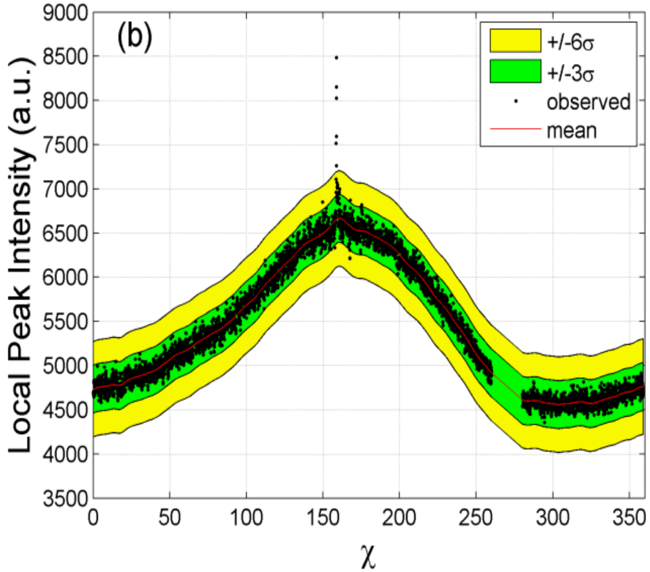
CrossMark

Detecting rare, abnormally large grains by x-ray diffraction

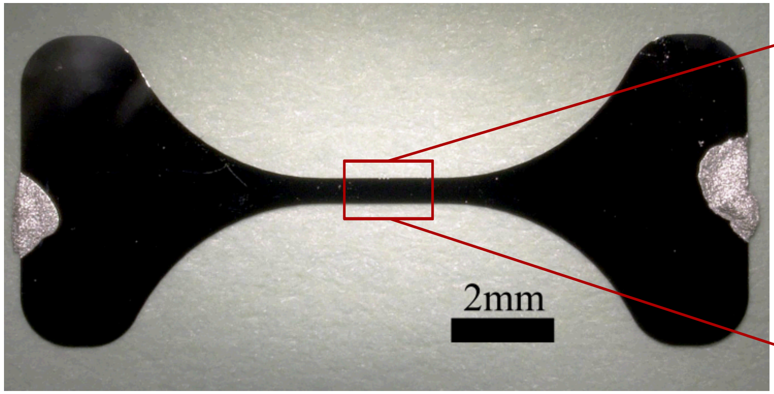
B. L. Boyce[1] · T. A. Furnish[1] · H. A. Padilla II[1] · D. Van Campen[2] · A. Mehta[2]

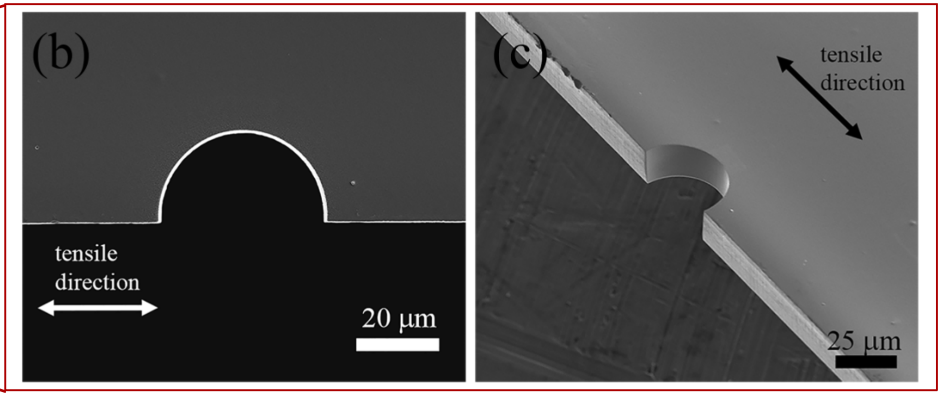






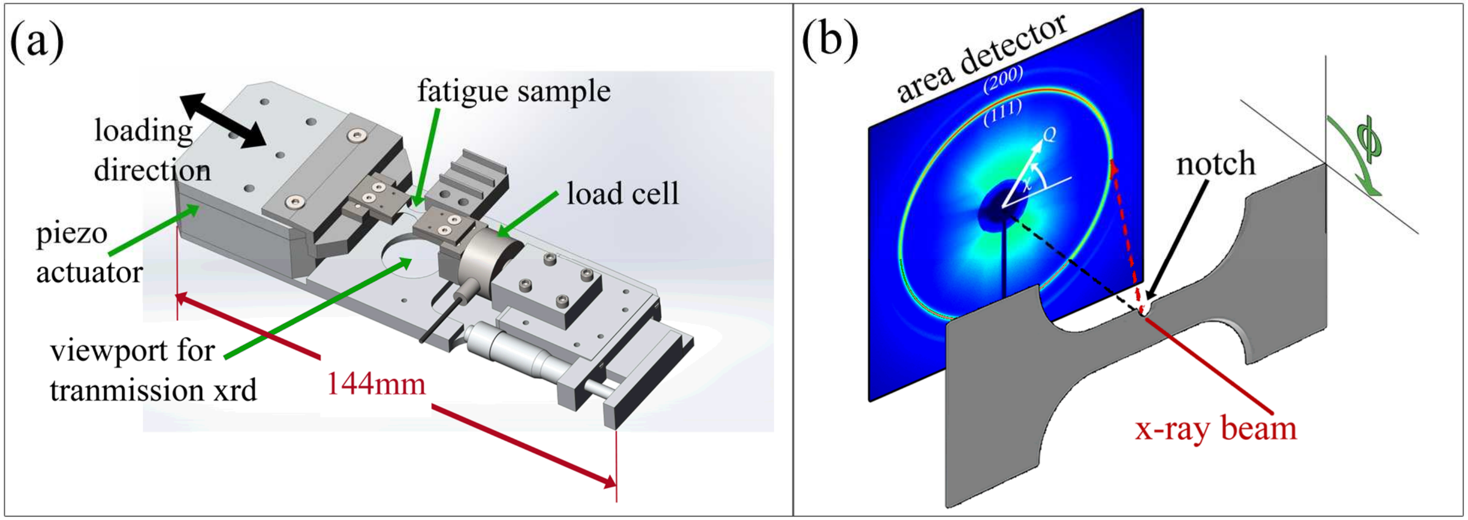
X-ray beam was centered on a FIB-milled semi-circular notch (used to prescribe crack initiation to a known location)



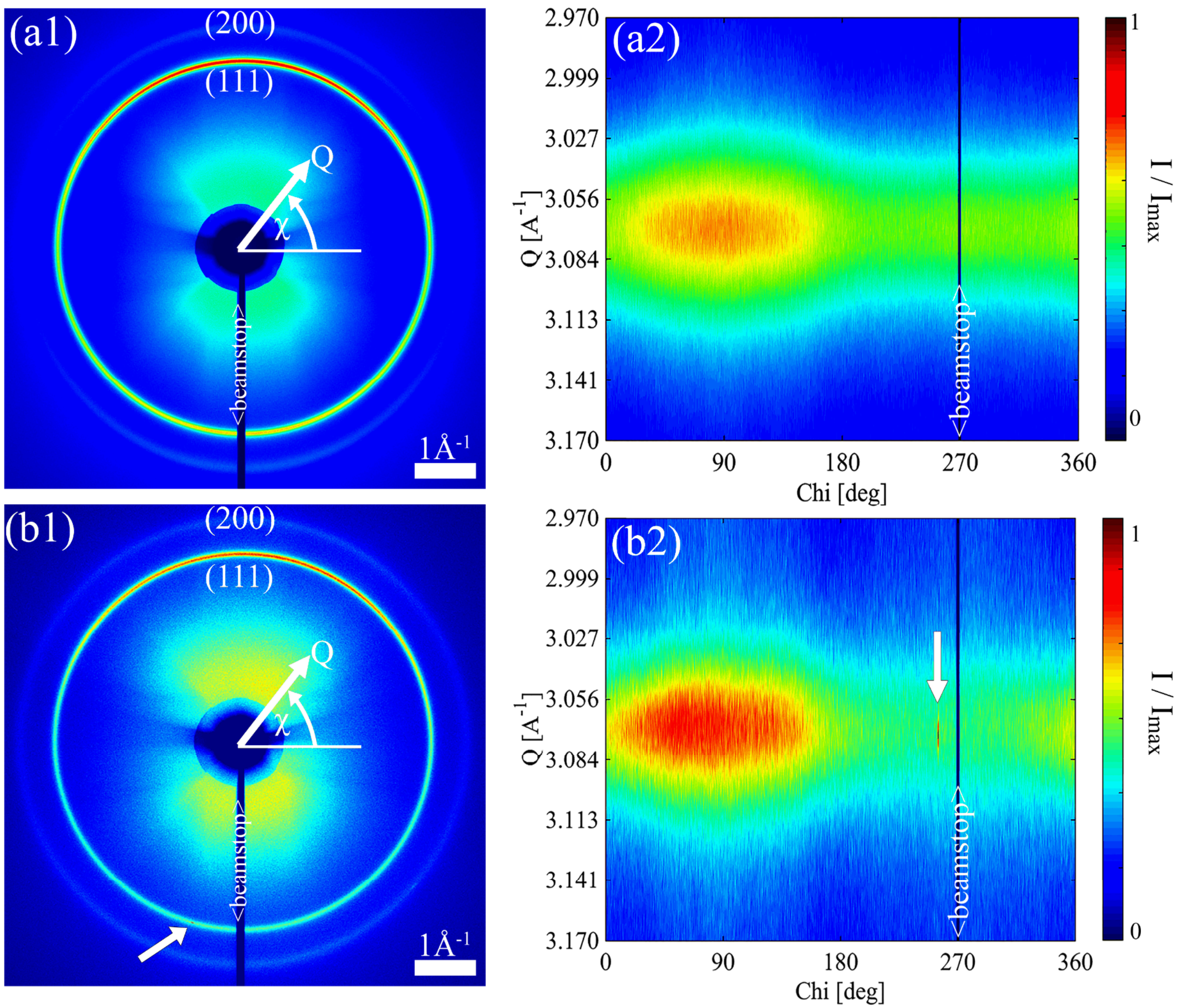


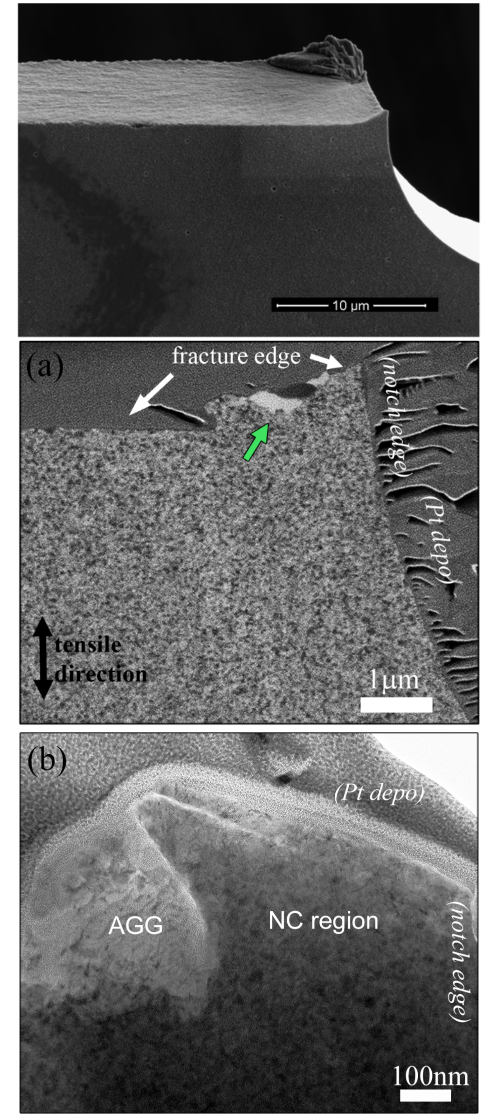
Notched electrodeposited NC Ni-Fe tensile specimens. Gage: 0.5 x 2.4 mm, 10-20 µm thick. Notch radius: 10 µm. Grain size: ~16 nm.

(a) Piezo-actuated in-situ micro-fatigue tester; (b) transmission xrd setup used in this study.

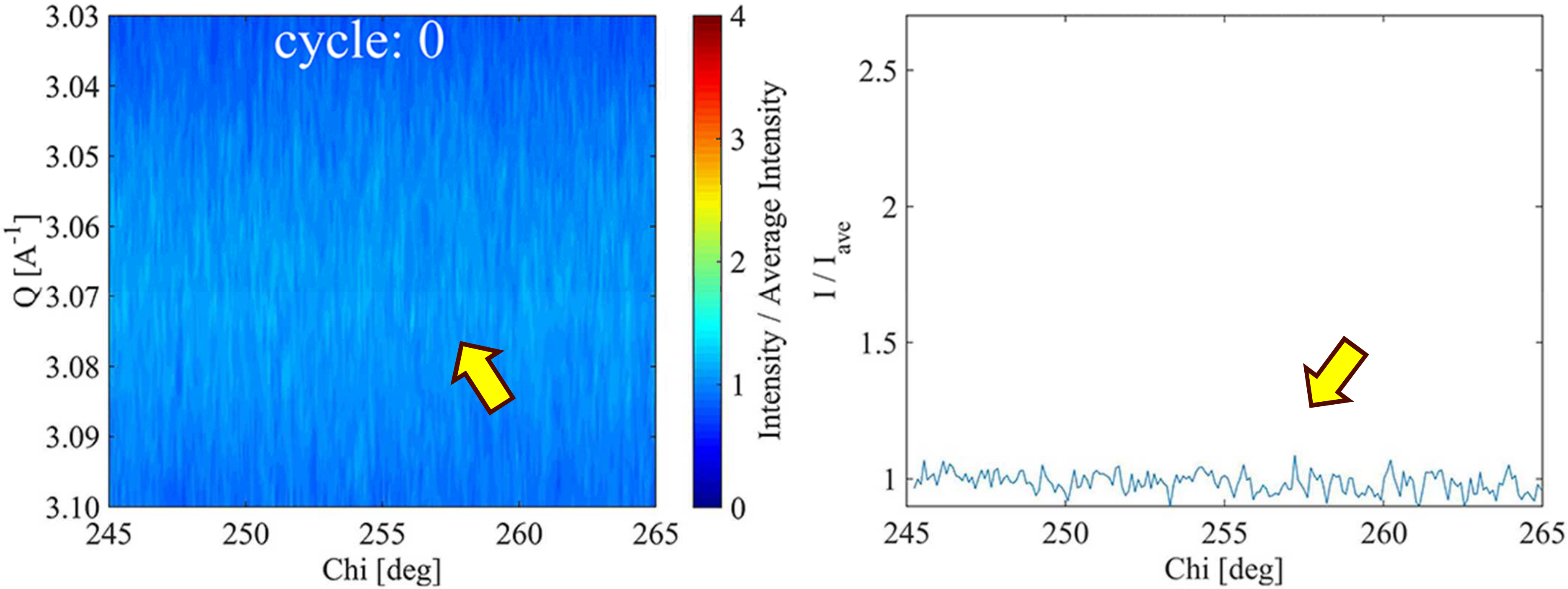


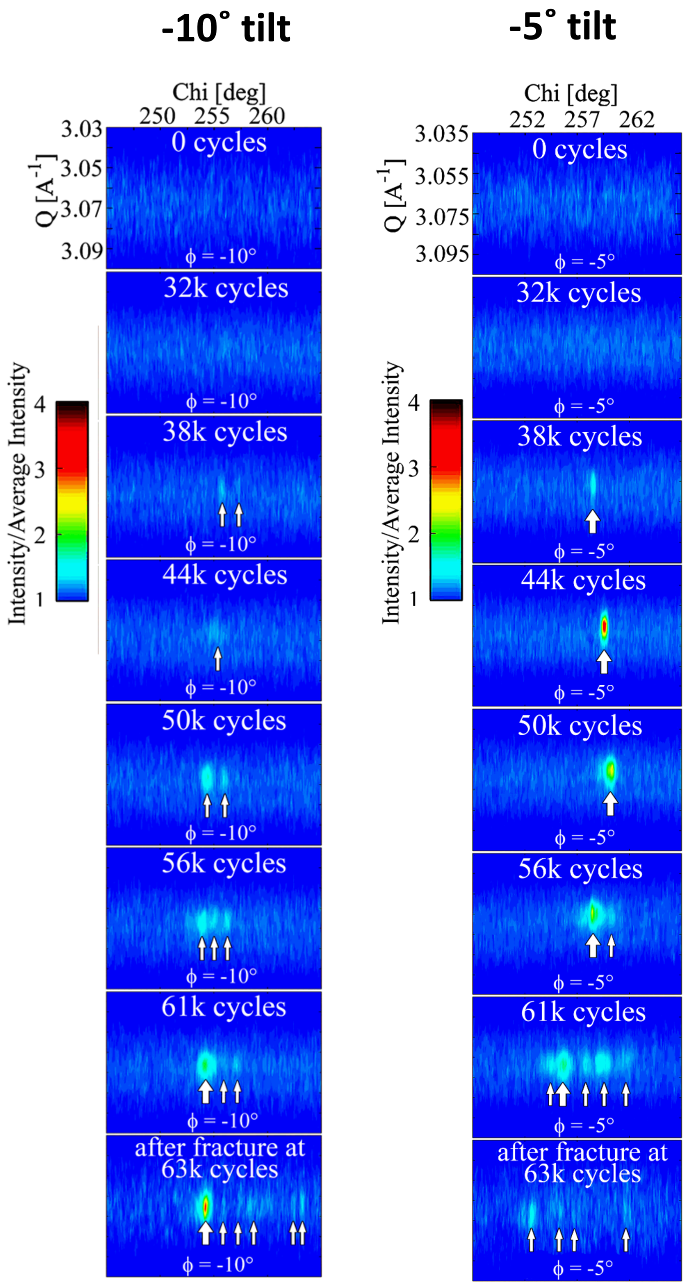
Post-fracture analysis revealed AGG in the notched region and anomalous intensity peaks in the diffraction data!





In-situ scans… showed the same anomalous peak present after 38,000 cycles – *25,000 cycles before final fracture*!





- We first detect the anomalous intensity spike at 38k cycles
- The intensity is actually split into two (or more) intensity peaks
  - implies some slight misorientation (~2deg) within the single grain, suggesting collective dislocation activity
- The predominant peak intensity continues to increase, while many other peaks appear (further dislocation accumulation/ grain rotation)
- If only taking this data, it appears that the grain gradually grows – however, if we do a side-by-side comparison with a different tilt angle, the intensity is maximized at first, then gradually decreases – this implies out of grain rotation
- It is likely that the abnormal grain growth occurs relatively quickly, reaches a critical size that can support dislocation activity, then cyclically deforms, leading to crack initiation
- This points to a multi-mode deformation, leading to more traditional crack initiation

In conclusion, this in-situ fatigue study corroborates our initial hypothesis that abnormal grain growth is prevalent during high cycle fatigue in NC metals, and that the subsequent deformation of these abnormally large grains is likely the precursor for crack initiation.

This in-situ approach provides us with a valuable tool to understand the role of abnormal grain growth on the deformation behaviors of NC metals.

Acknowledgments

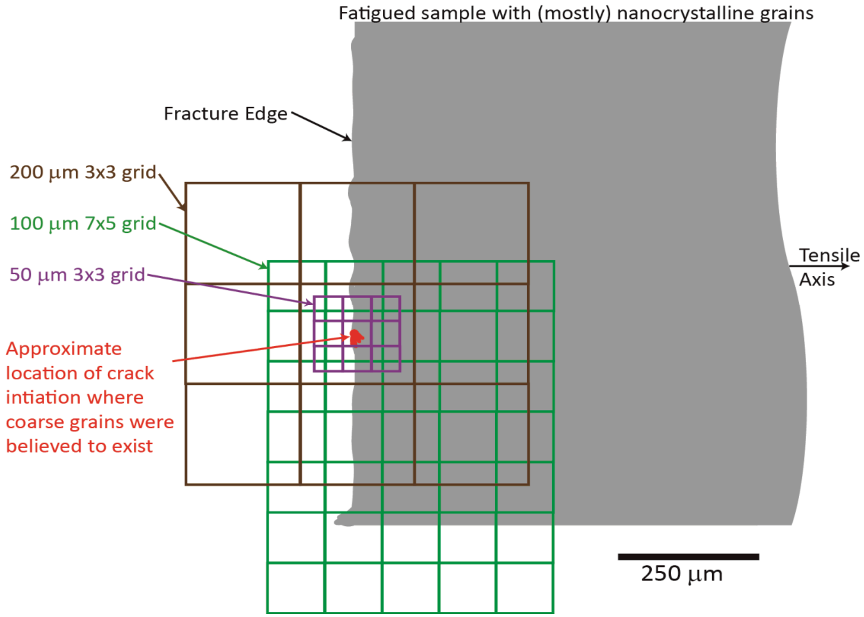
- This work was funded by the U.S. DOE, Office of Basic Energy Sciences, Division of Materials Science and Engineering
- A portion of the microscopy and FIB work was performed at the Center for Integrated Nanotechnologies (CINT)
- Synchrotron work was performed at beamline 11-3 at Stanford Synchrotron Radiation Lightsource (SSRL)

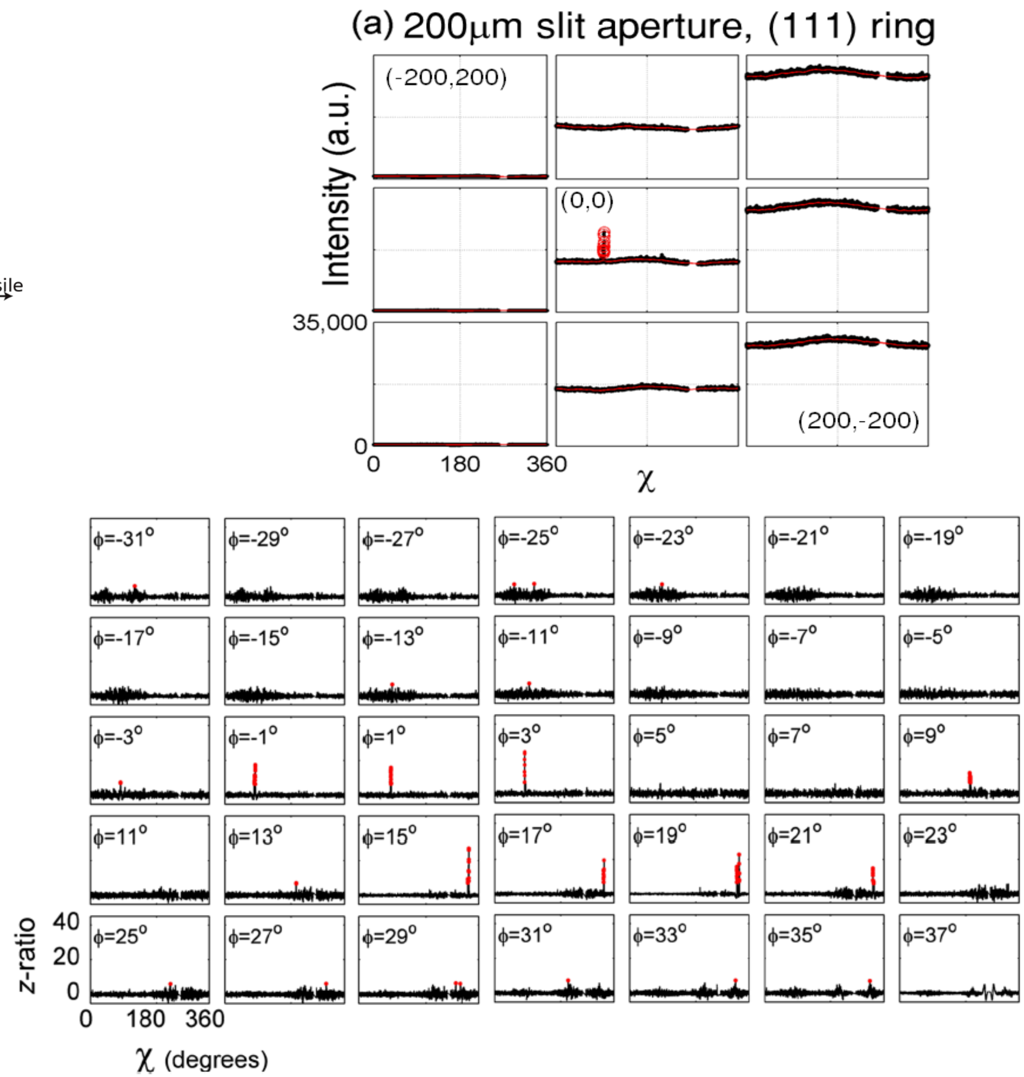
Extra Slides

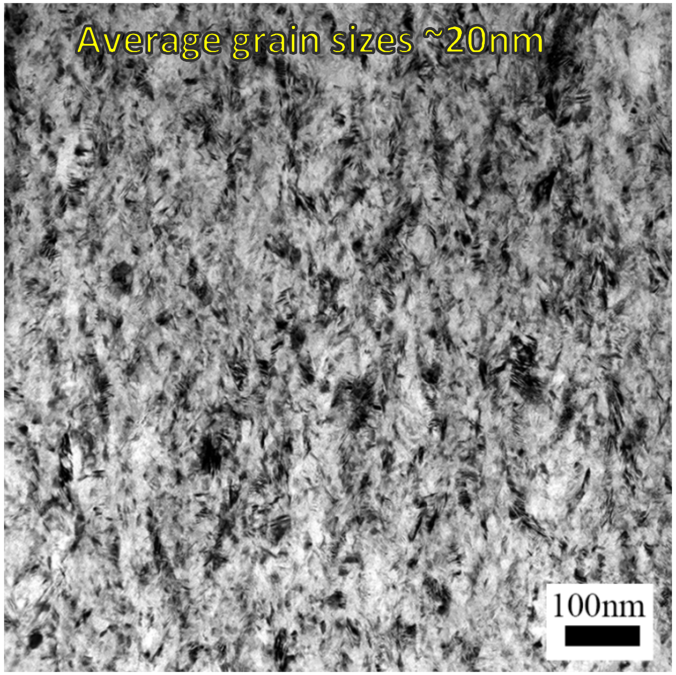
Supplementary information

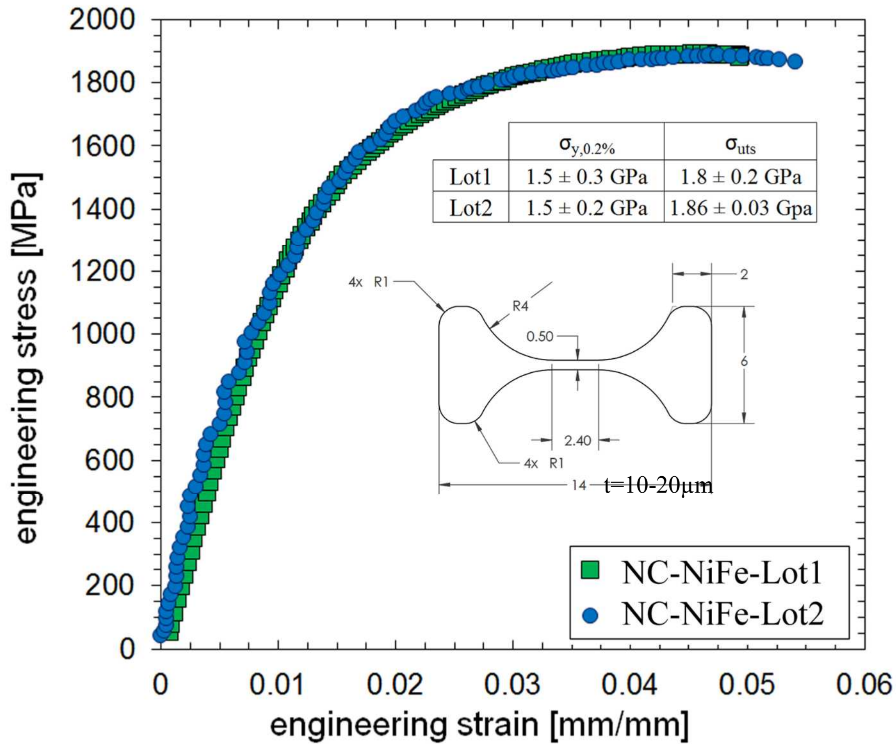
- …evidence "suggest that the NC matrix undergoes AGG during cyclic loading, allowing dislocation activity to persist over length scales necessary to initiate a fatigue crack by traditional fatigue mechanisms." [Boyce and Padilla] also, "grain growth process that appears to be a necessary precursor to crack initiation". "insufficient space for the collective dislocation interactive mechanisms found in CG metals such as pileups and subgrain formation." "fatigue mechanisms may be influenced more by the evolved grain structure than by the initial structure". Ni-Fe shows the <112> pole aligned with the stress axis, which is the well-known orientation that any fcc single crystal rotates toward during uniaxial deformation to take full advantage of available slip systems. In NC metals, neighboring grains under an external stress can rotate to a common orientation, eliminating the HAGB allowing for collective dislocation motion

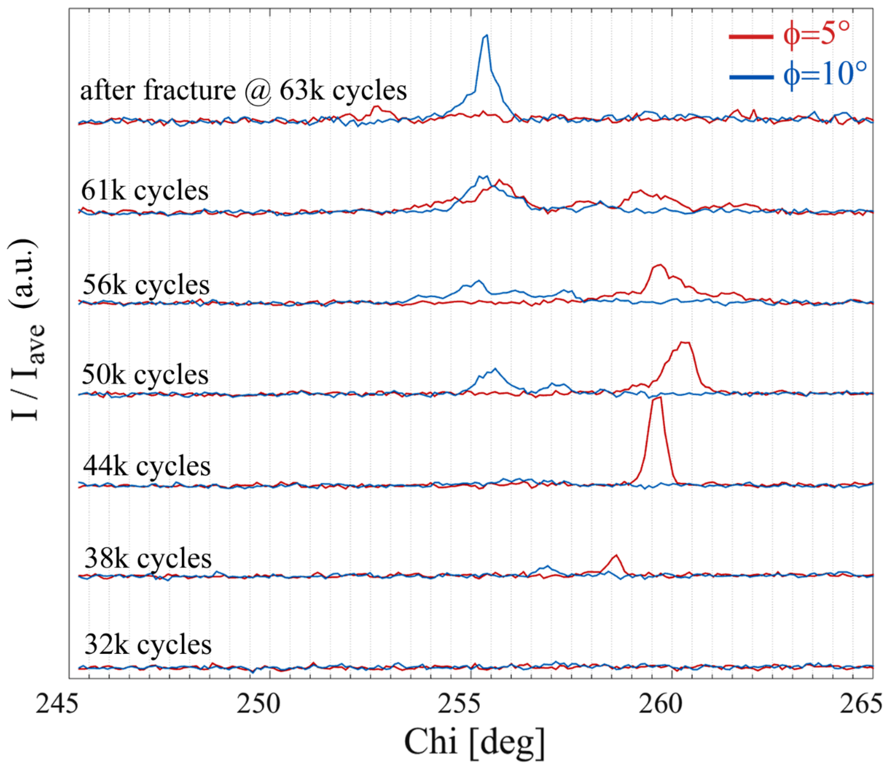
This x-ray technique identified the AGG region, despite only occupying < 0.0001% of the sampled volume!



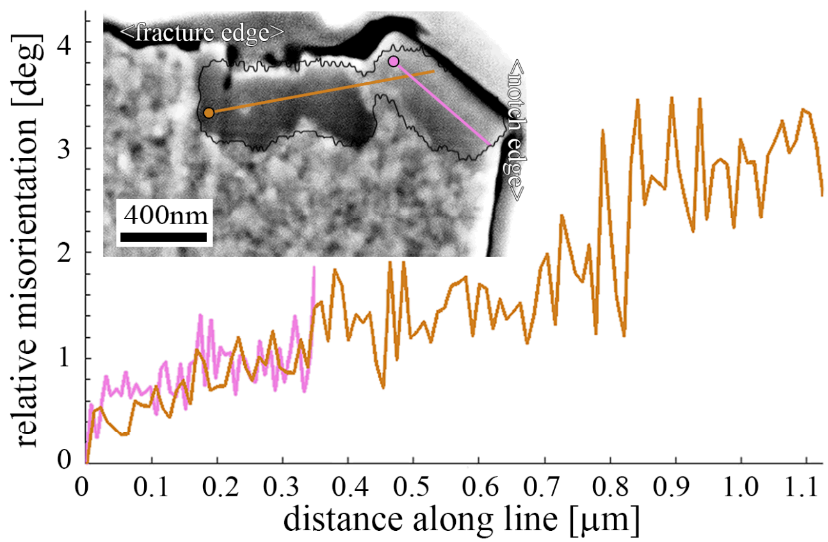


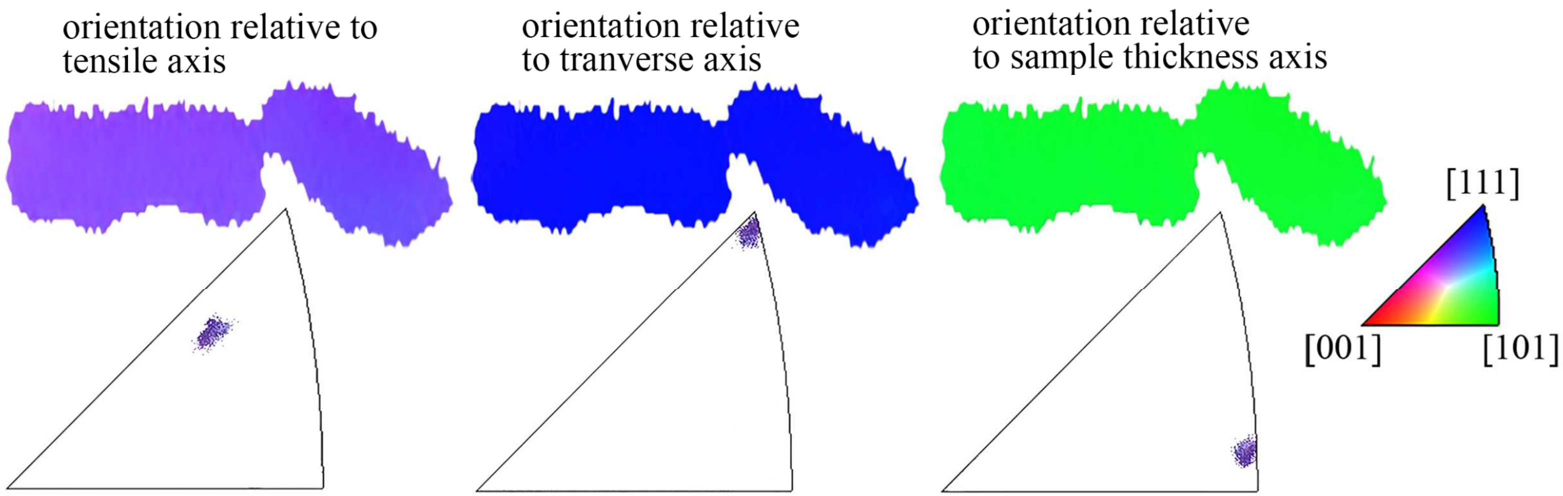

Average grain sizes ~20nm
100nm


engineering stress [MPa]
engineering strain [mm/mm]
0
200
400
600
800
1000
1200
1400
1600
1800
2000
0
0.01
0.02
0.03
0.04
0.05
0.06
σy,0.2%
σuts
Lot1
1.5 ± 0.3 GPa
1.8 ± 0.2 GPa
Lot2
1.5 ± 0.2 GPa
1.86 ± 0.03 Gpa
4x R1
R4
0.50
2
6
2.40
4x R1
14
t=10-20μm
NC-NiFe-Lot1
NC-NiFe-Lot2

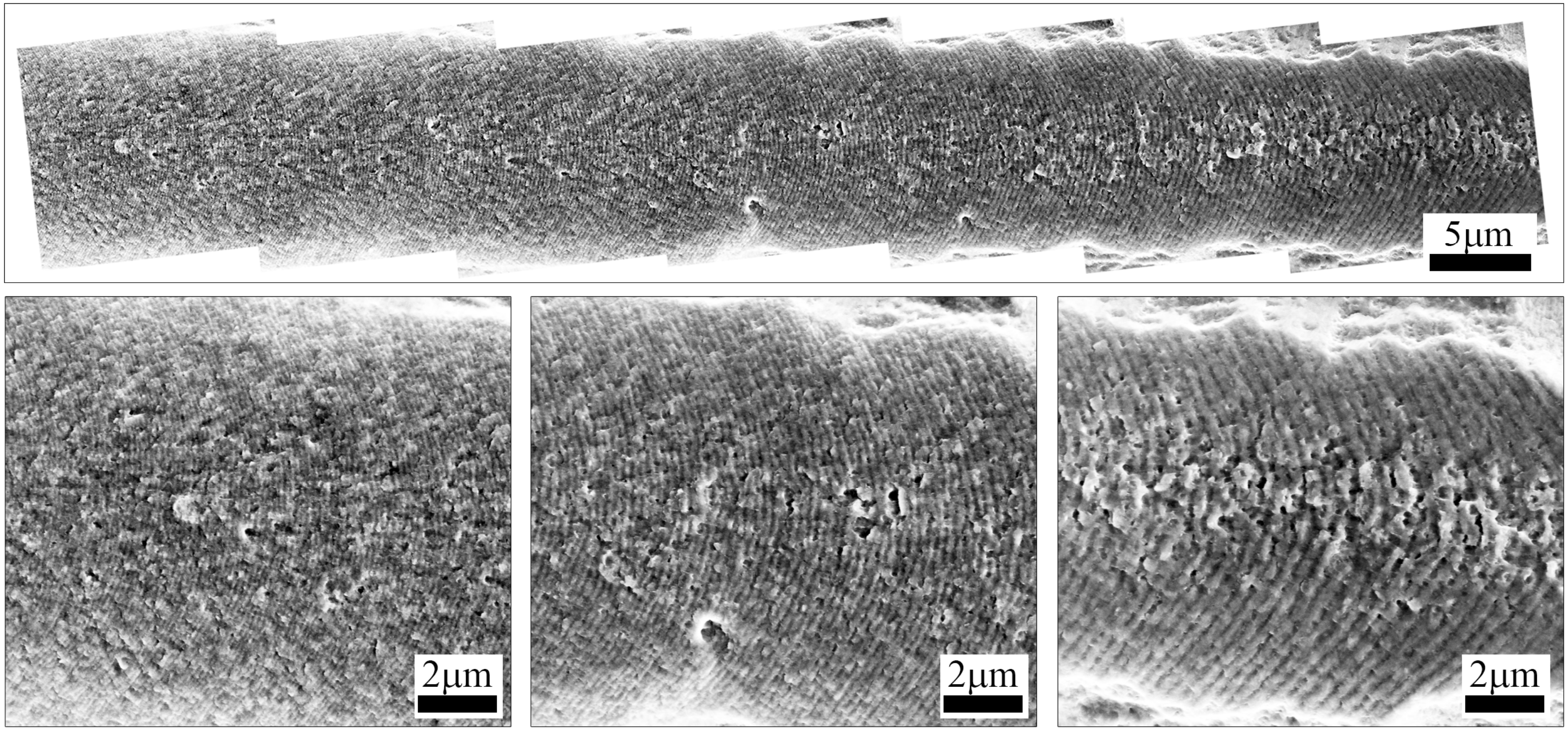


ϕ=5°
ϕ=10°
after fracture @ 63k cycles
61k cycles
56k cycles
50k cycles
44k cycles
38k cycles
32k cycles
I / I_ave (a.u.)
245
250
255
260
265
Chi [deg]

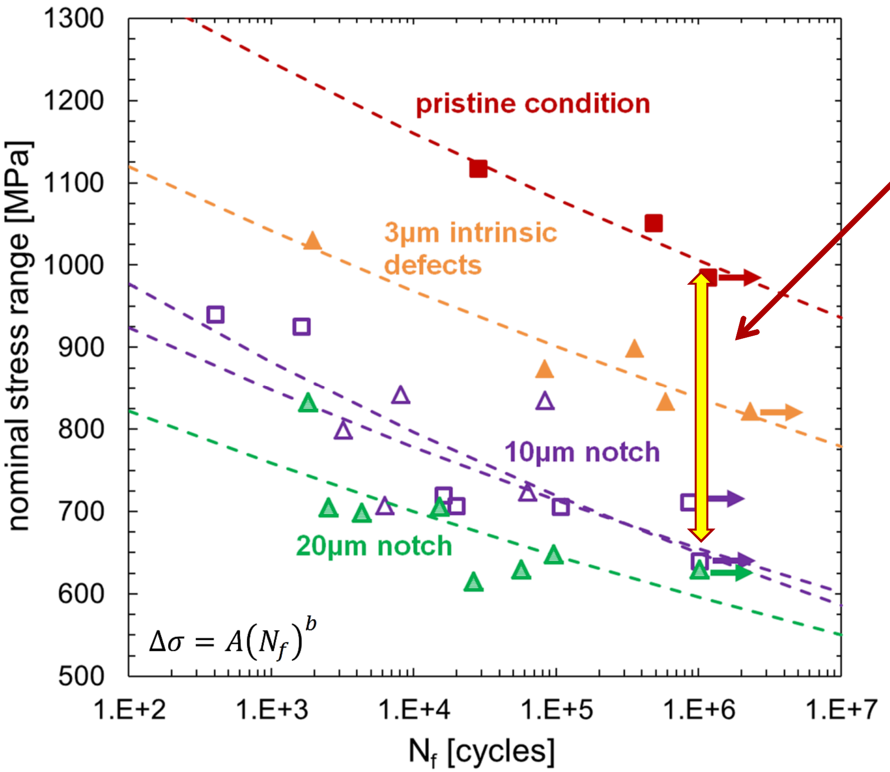

<fracture edge>
<notch edge>
400nm
relative misorientation [deg]
0
1
2
3
4
0
0.1
0.2
0.3
0.4
0.5
0.6
0.7
0.8
0.9
1.0
1.1
distance along line [μm]


orientation relative to tensile axis
orientation relative to tranverse axis
orientation relative to sample thickness axis
[111]
[001]
[101]

Fatigue striations in fractured edge



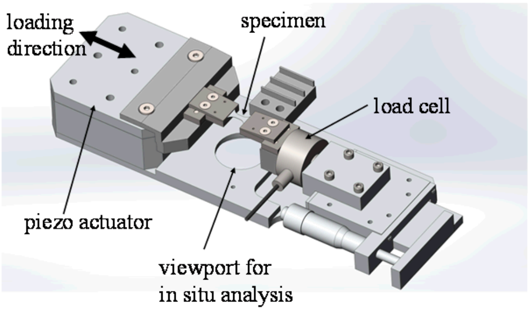
Notched fatigue tests revealed dramatic fatigue stress concentrations and high notch sensitivity!



$$K_f = \frac{\Delta\sigma_{unnotched,lim}}{\Delta\sigma_{notched,lim}}$$

$$q = \frac{(K_f - 1)}{(K_t - 1)}$$

q = 0 (totally notch insensitive)
q = 1 (fully notch sensitive, $K_f = K_t$)

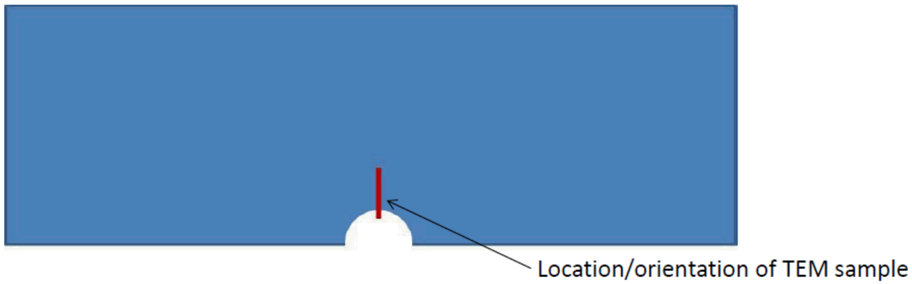


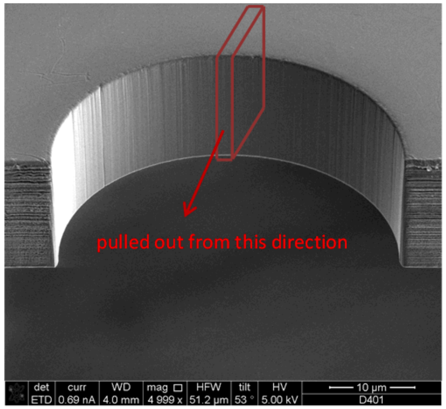
- Custom built piezo actuated *in situ* fatigue tester for thin foil testing
- Constant-load, 4-6 Hz (sine) used in this study

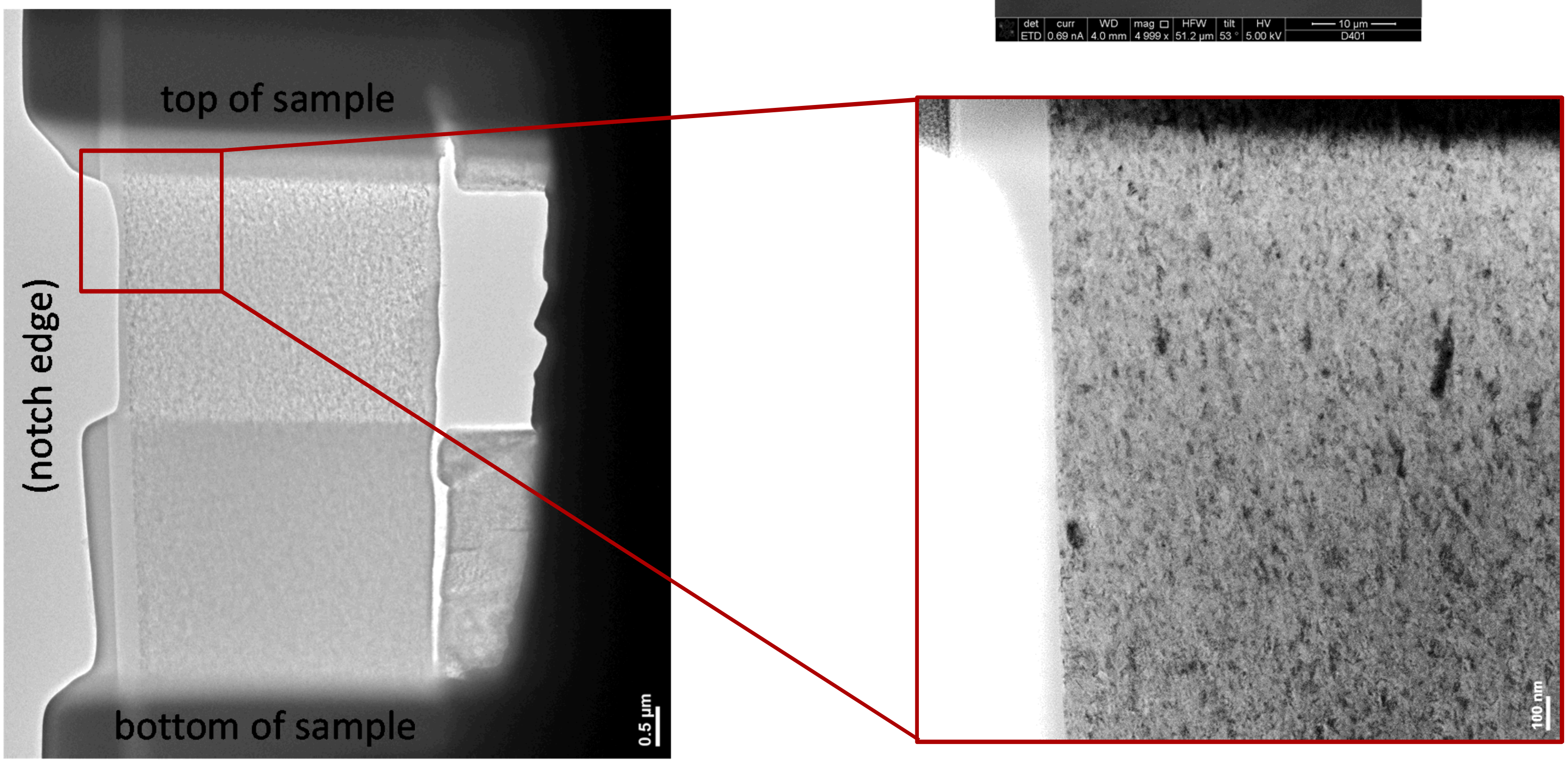
- Fatigue stress concentrations:
  - 1.2 (3µm intrinsic defects)
  - 1.5 (10µm notch)
  - 1.7 (20µm notch)
- Notch sensitivity factors, q:
  - 0.28 (10µm notch)
  - 0.37 (20µm notch)

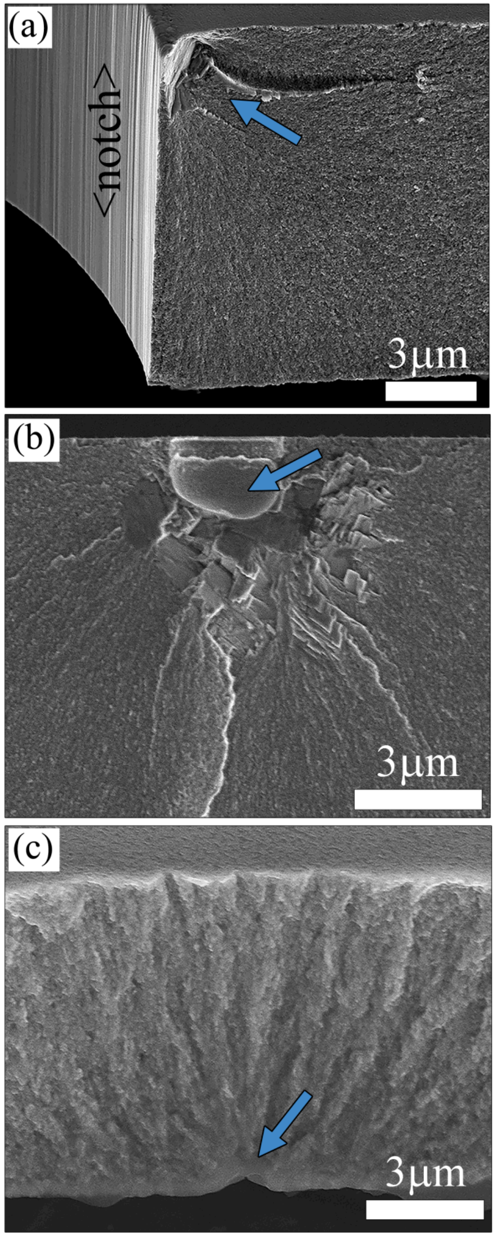
*Furnish, Sharon, Arrington, Pillars, Clark, and Boyce *submitted*

No obvious FIB-induced abnormal grain growth under notches

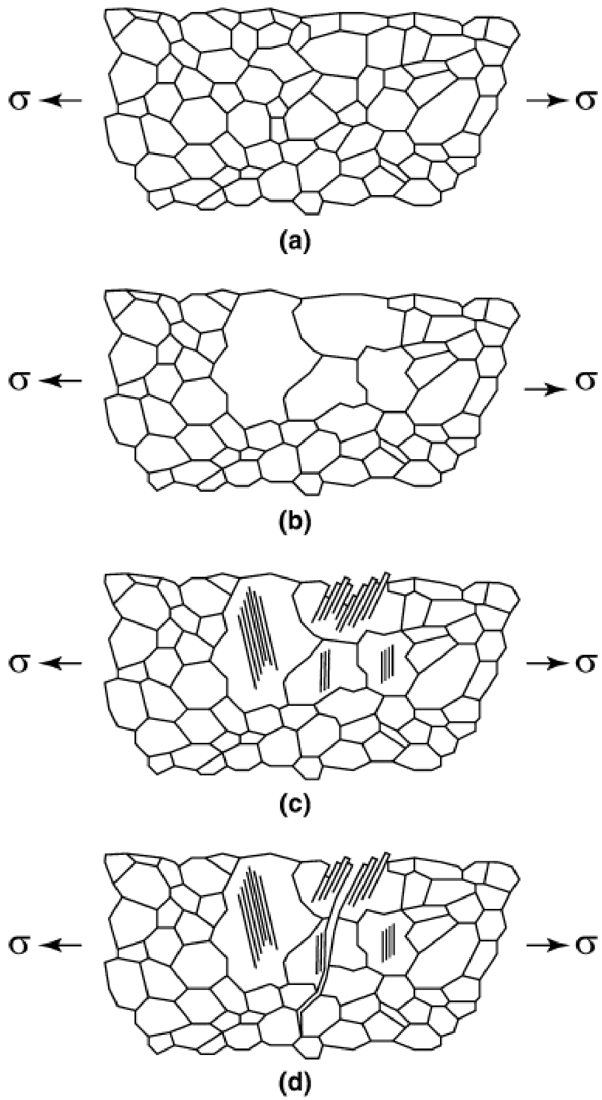







(a)
<notch>
3μm
(b)
3μm
(c)
3μm

Proposed mechanisms for crack initiation in NC metals



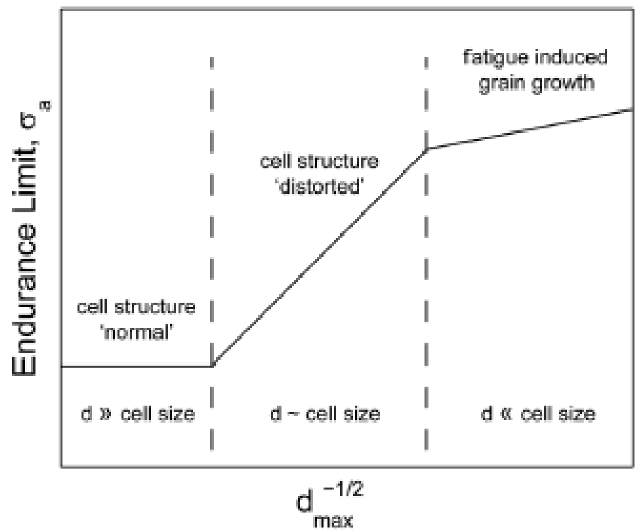
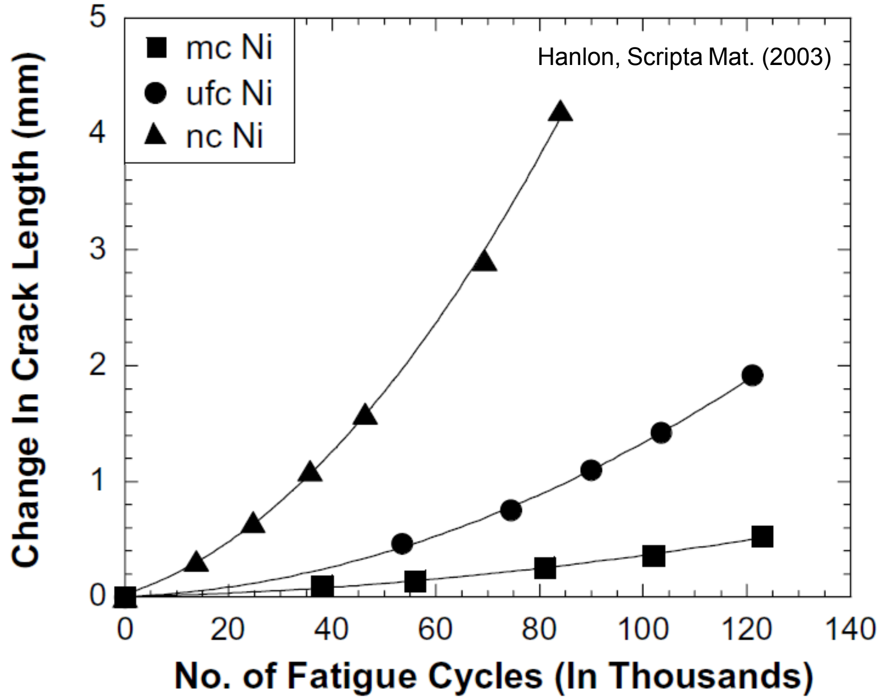
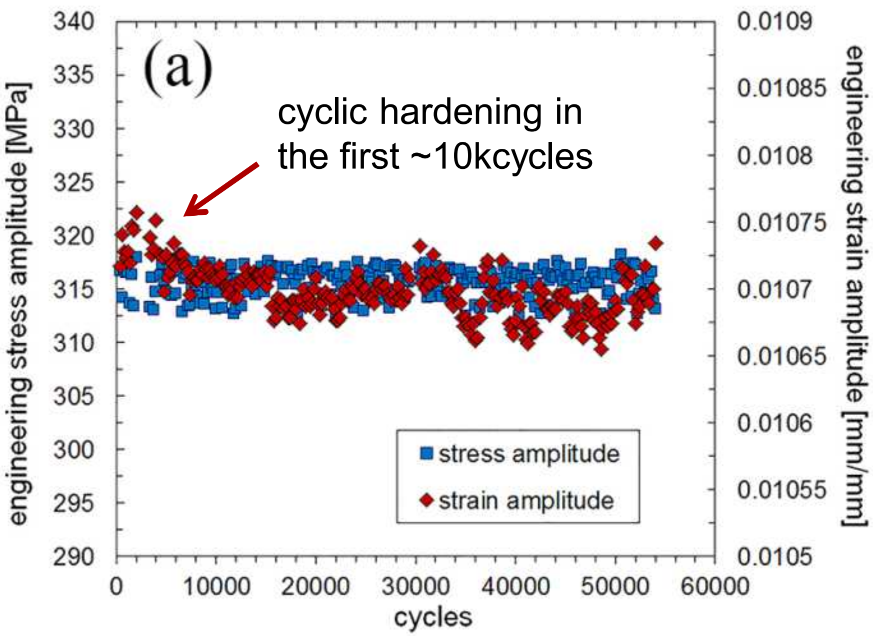


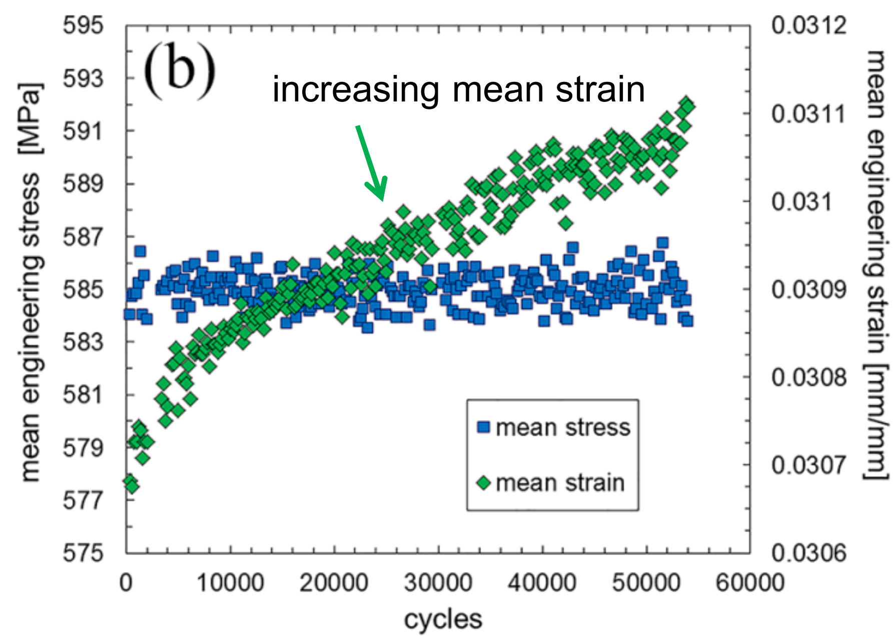
Figure 14. Schematic showing different scaling regimes for fatigue behavior with respect to the grain size.

Fracture toughness of NC metals has been shown to be much lower than for ufg and CG counterparts



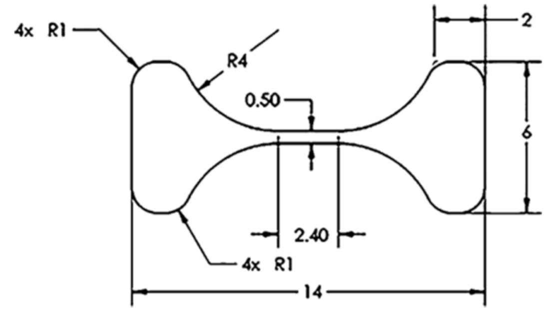
Some cyclic hardening was observed, in addition to monotonic "ratcheting" throughout the fatigue life

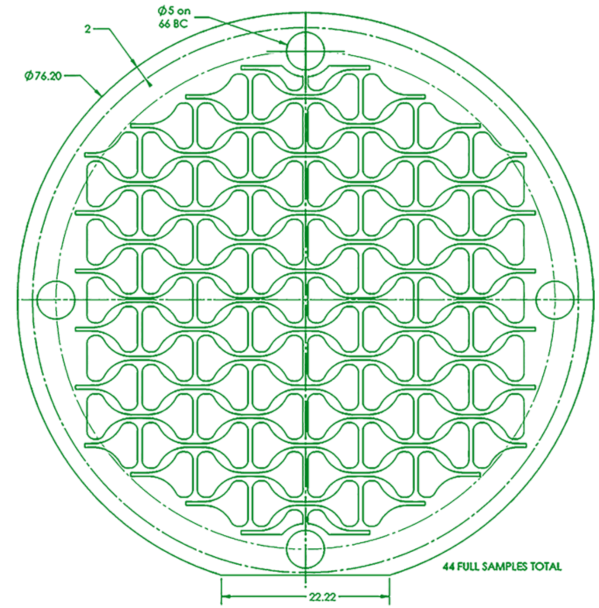




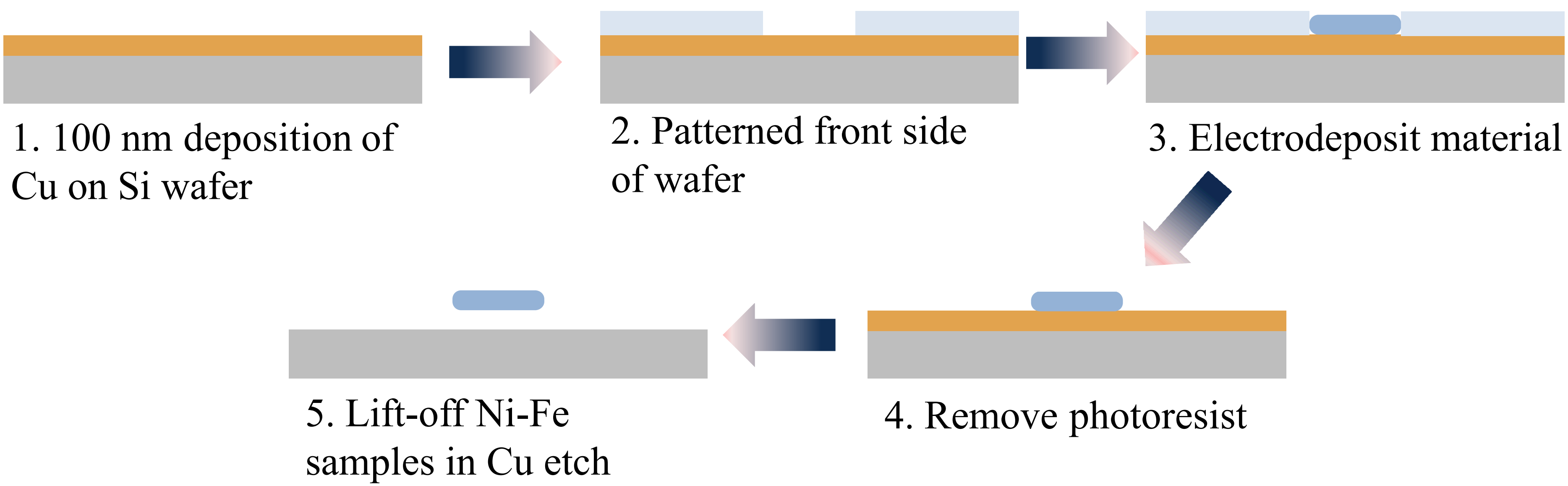
- Cyclic hardening is expected based on work-hardening effects observed during monotonic tension (likely some dislocation activity)
- Monotonic accumulation of strain (separate from the cyclic hardening effects) indicates possible boundary-related activity (e.g. Coble creep mechanisms)

Sample Preparation

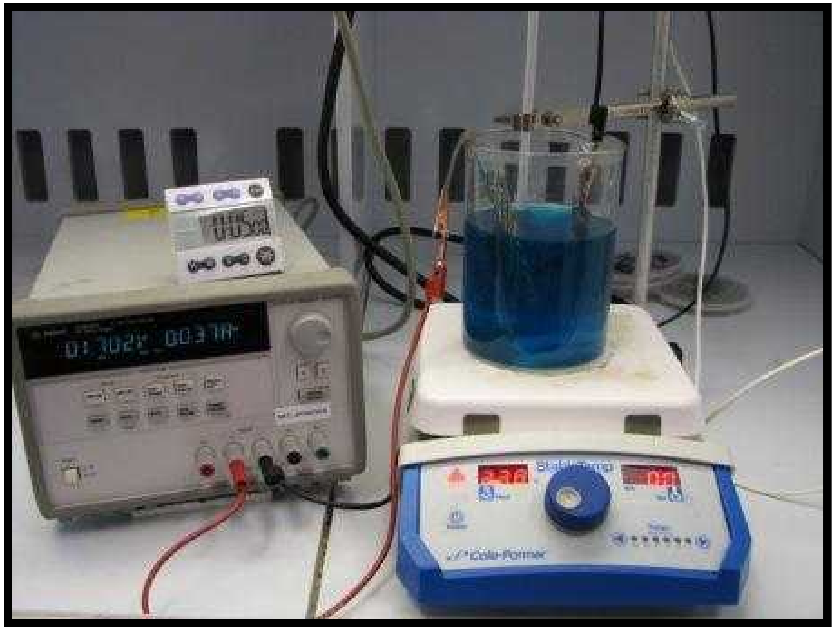




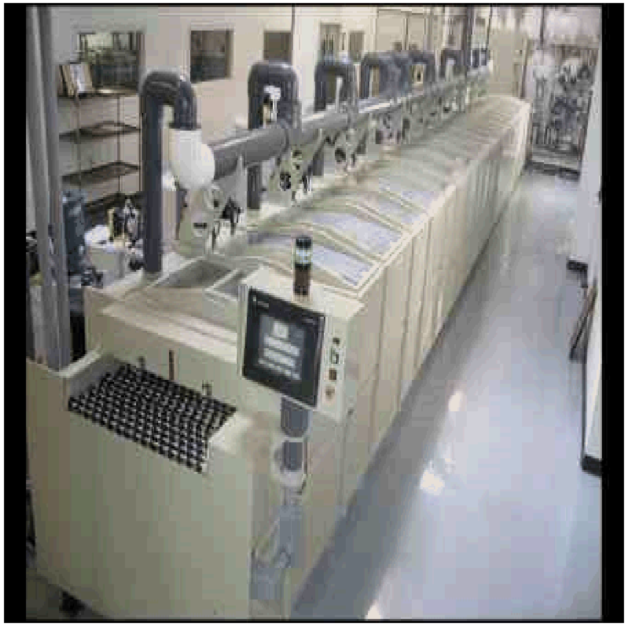
Nickel/iron chemistry make-up	
$NiSO_4$ Nickel Sulfate	112 g/L
$FeSO_4$ Iron sulfate	5 g/L
$Na_3C_6H_5O_7$ Sodium Citrate	75 g/L
KSO_4 Potassium Sulfate	1.5 g/L
$C_7H_5NO_3S$ Saccharin	1 g/L



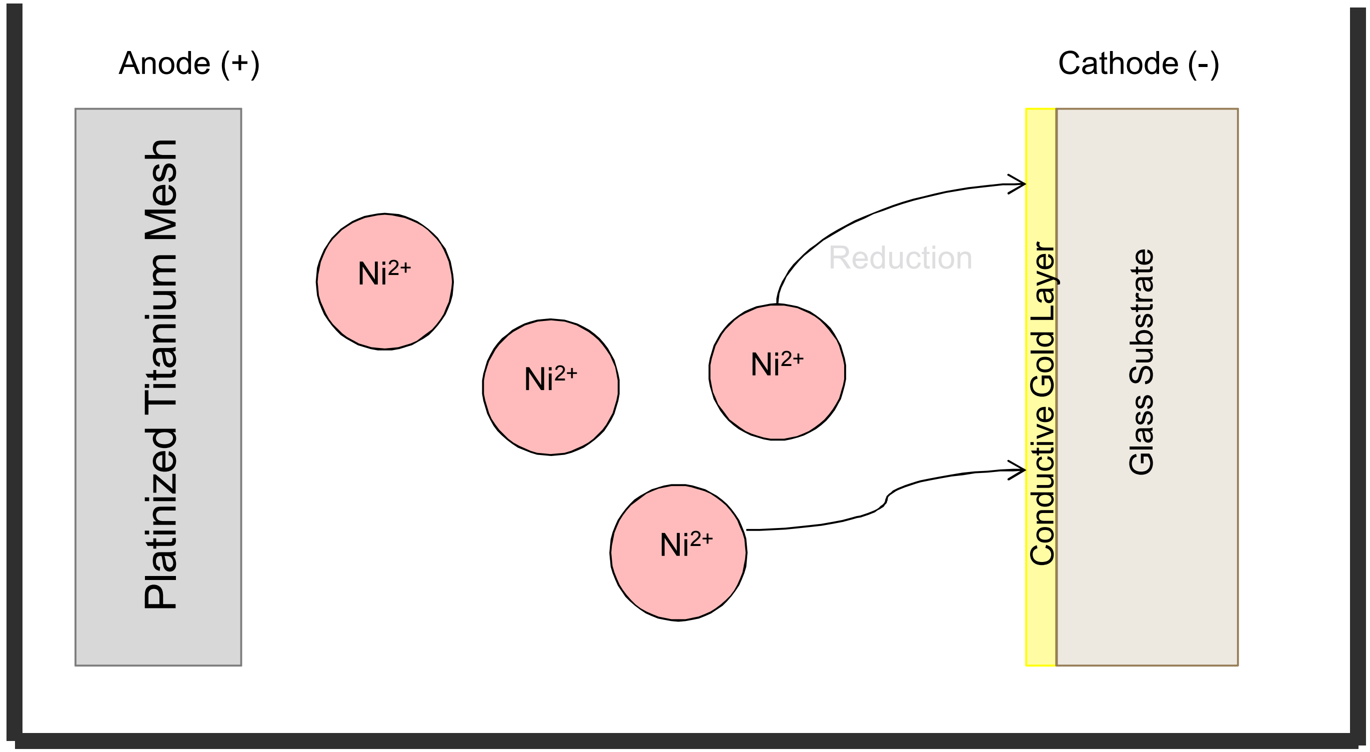
Bench Top to Industrial Plating



Bench Top Plating



Industrial Plating Bench


Anode (+)
Cathode (-)
Platinized Titanium Mesh
Ni2+
Ni2+
Ni2+
Ni2+
Reduction
Conductive Gold Layer
Glass Substrate

efficiency tradeoff…

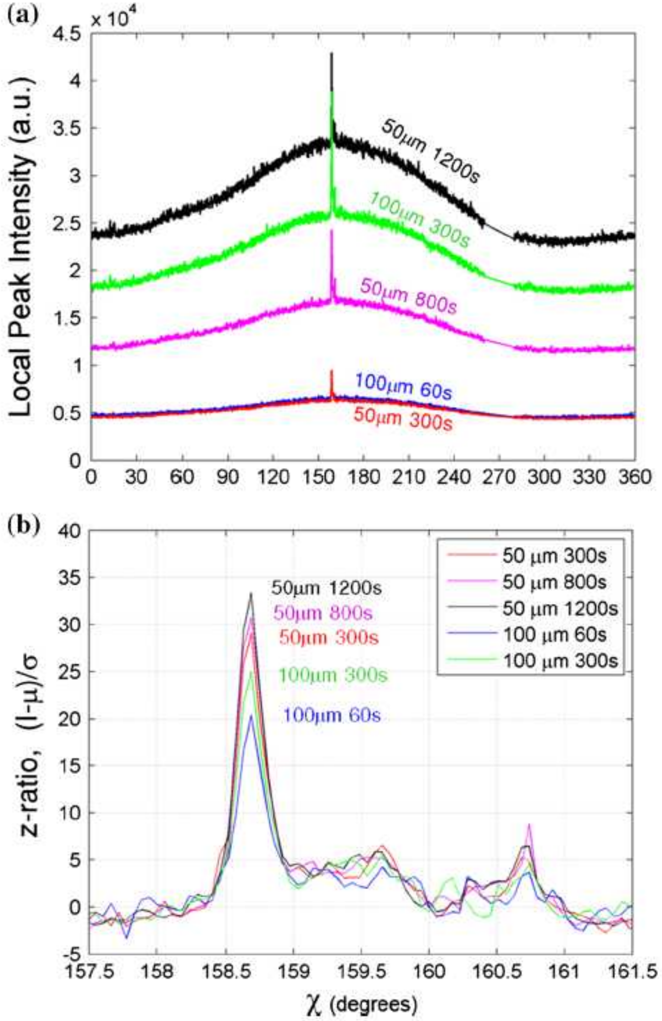
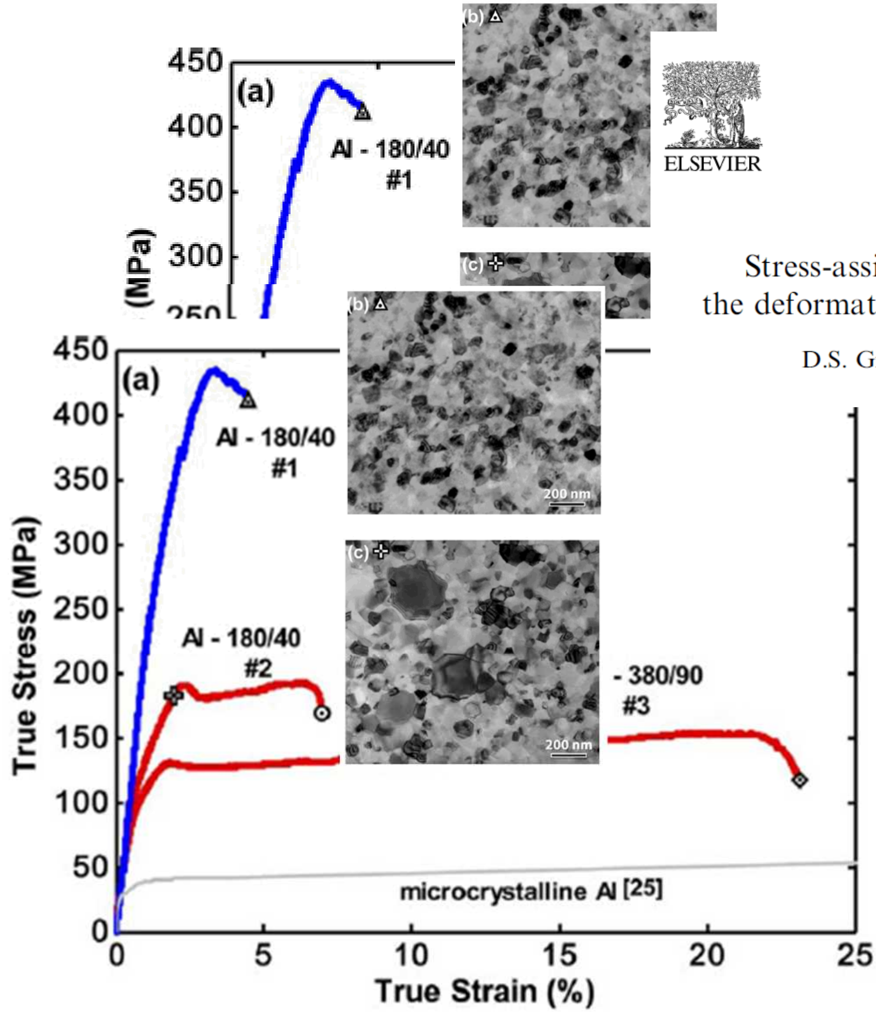


Table 1 *Q* data for the (111), (200), and (220) peaks

Peak	Q (Å^{-1})	Q range analyzed (Å^{-1})
(111)	3.08	2.7–3.3
(200)	3.56	3.3–3.9
(220)	5.03	4.7–5.3

Grain growth leading to enhanced ductility



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Stress-assisted discontinuous grain growth and its effect on the deformation behavior of nanocrystalline aluminum thin films

D.S. Gianola [a], S. Van Petegem [b], M. Legros [c], S. Brandstetter [b], H. Van Swygenhoven [b], K.J. Hemker [a,*]