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Fatigue-Induced Abnormal Grain Growth in Nanocrystalline Metals

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U.S. DEPARTMENT OF ENERGY | Office of Science

NNSA National Nuclear Security Administration

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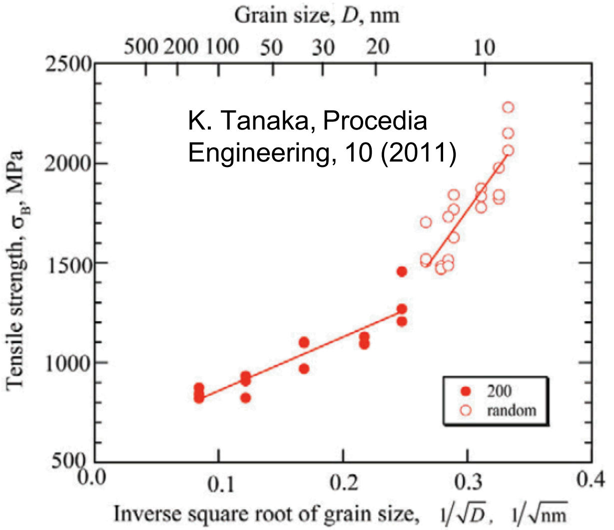
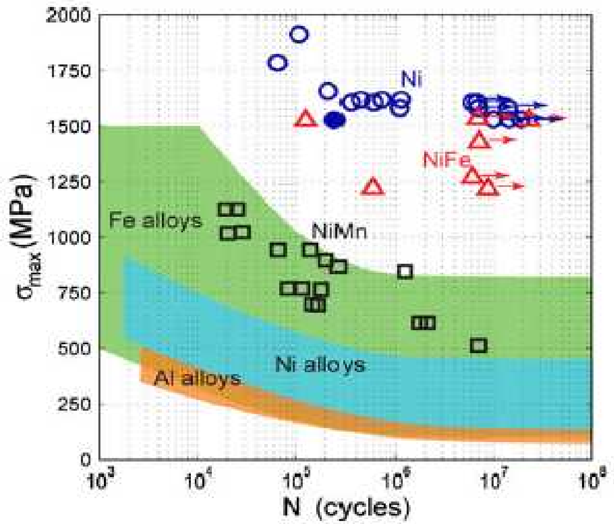
Nanocrystalline metals show a potential for many attractive properties

- **Nanocrystalline (NC)** materials are generally defined by having an average grain size of < 100nm
- When grains are at the nanoscale, unique mechanical properties and plastic responses are observed
- Main advantages to NC metals:
  - ultra-high strengths & hardness
  - high wear-resistance
  - excellent fatigue strengths

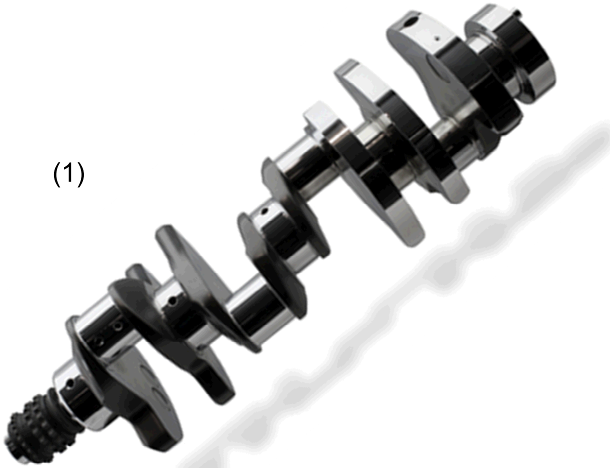




B.L. Boyce, Met. Mat. Trans. A, 42A (2011).

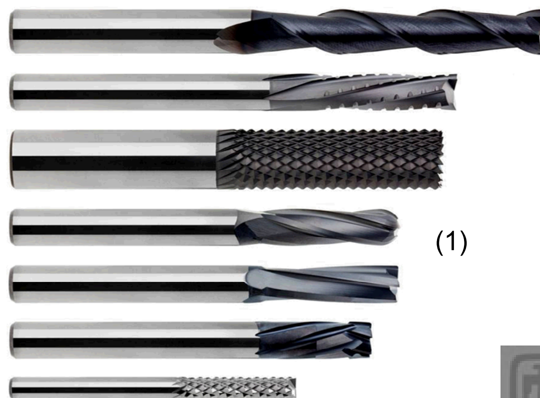
Current & potential applications for NC metals

(1)



wear resistance in complex geometries

Ultra-hard thin films



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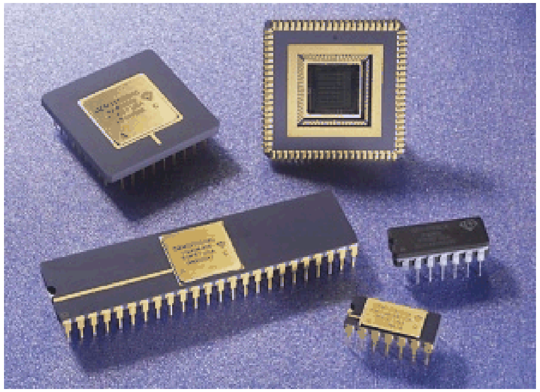


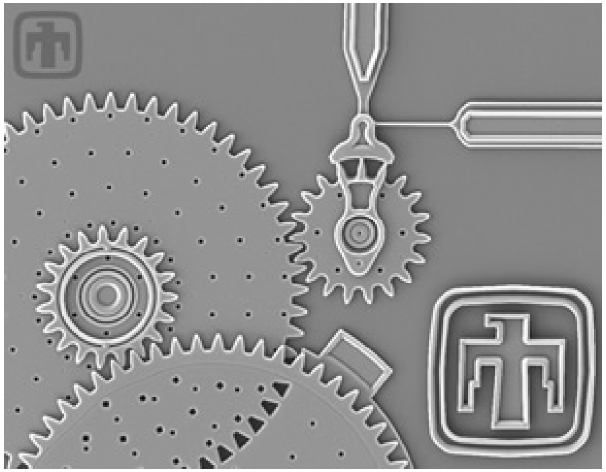
Microcircuits

(1)

Solid/dry lubricant coatings

(1) www.enduracoatings.com
(2) www.lotvacuumamerica.com
(3) www.sri.com

(2)



MEMS & Micromachines

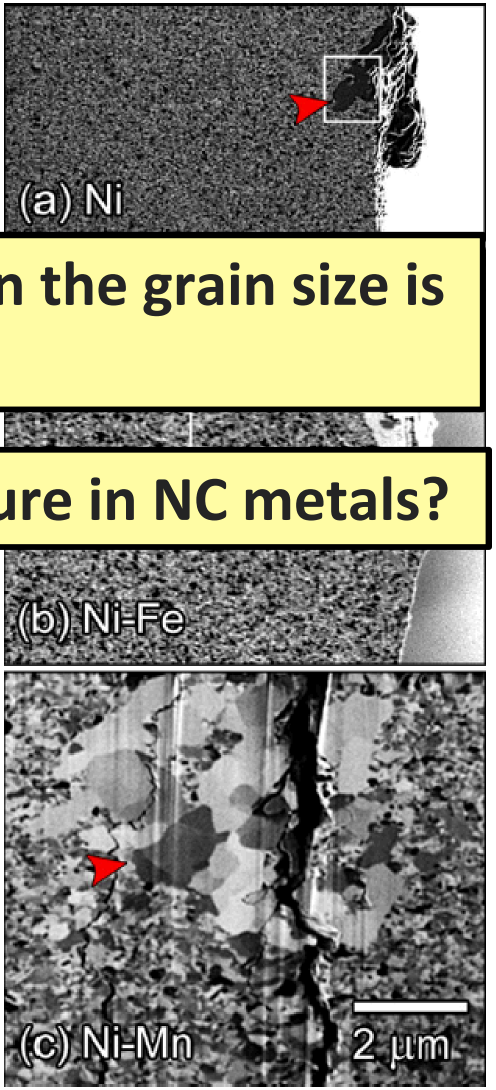
The suppression of “traditional” deformation mechanisms can lead to atypical material responses, e.g. AGG

- When the grain size is reduced to the nanoscale, traditional dislocation deformation mechanisms are suppressed
- E.g. Frank-Read source, dislocation multiplication, and

New fatigue and fracture mechanisms arise when the grain size is reduced to the nanoscale

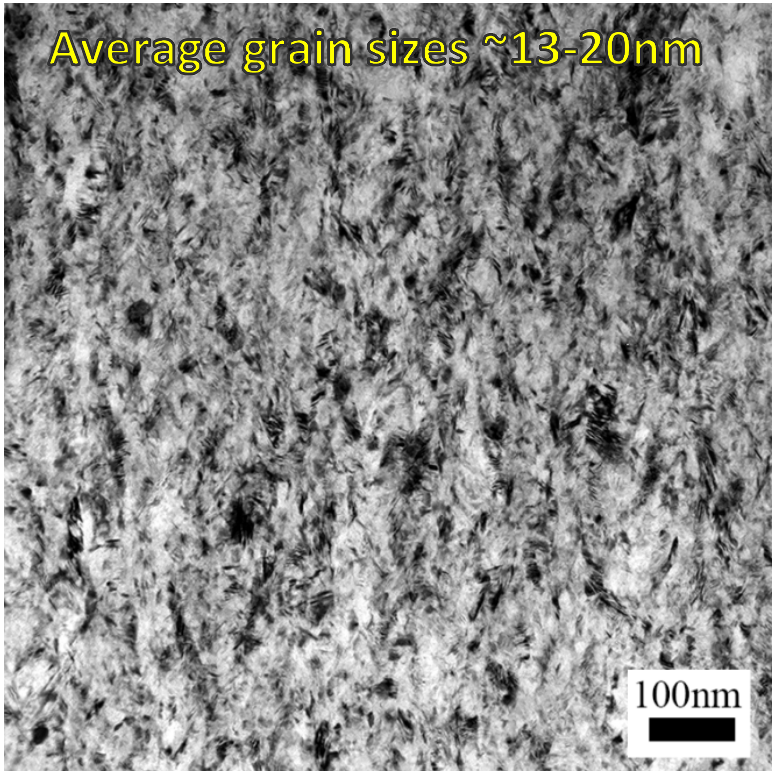
What is the role of AGG on the fatigue and fracture in NC metals?

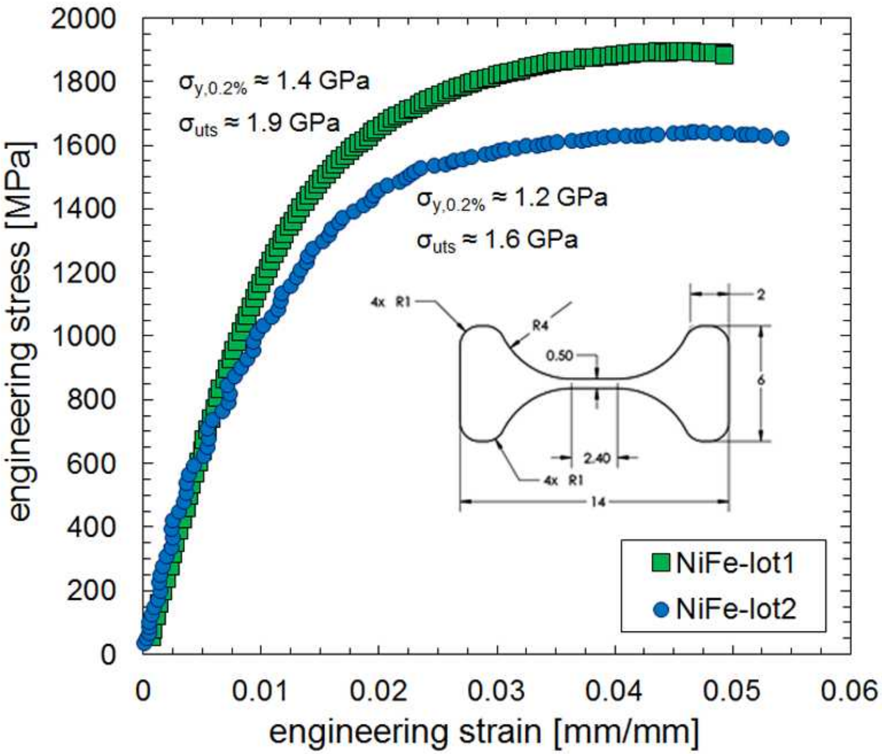
Few studies have been dedicated to the fatigue-response of NC metals – however, early reports have revealed a general propensity for abnormal grain growth (AGG), which may be tied to crack initiation and failure during cyclic loading



Examples of Ni and Ni-based alloys that underwent extensive abnormal grain growth near fatigue crack initiation sites

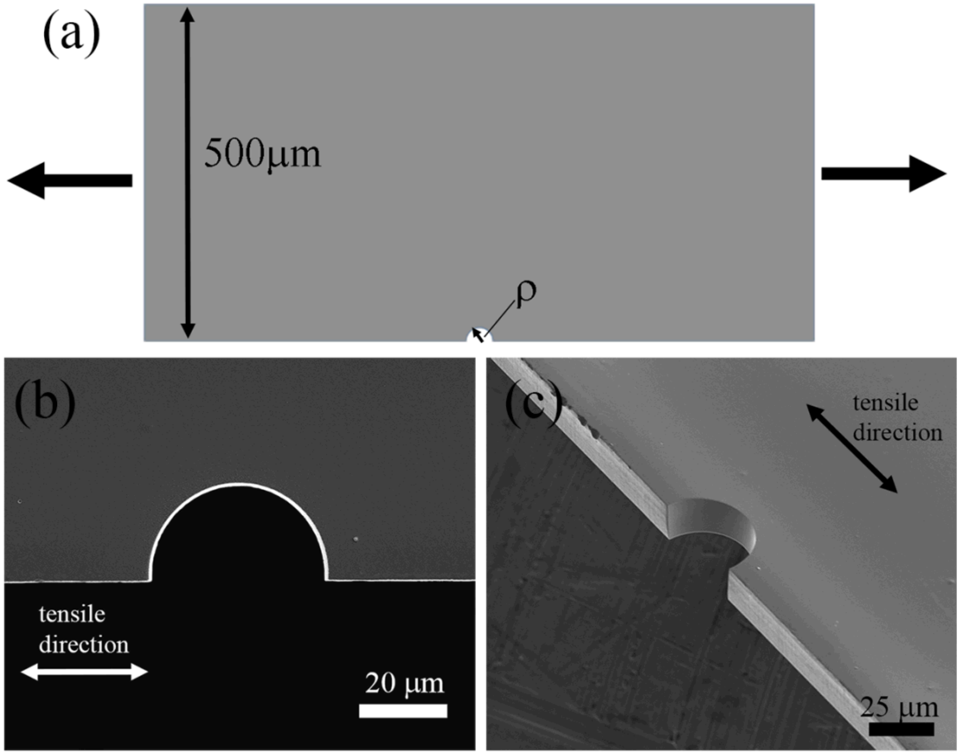
Electrodeposited NC Ni-Fe (40wt% Fe) was selected as a representative alloy to study AGG during fatigue





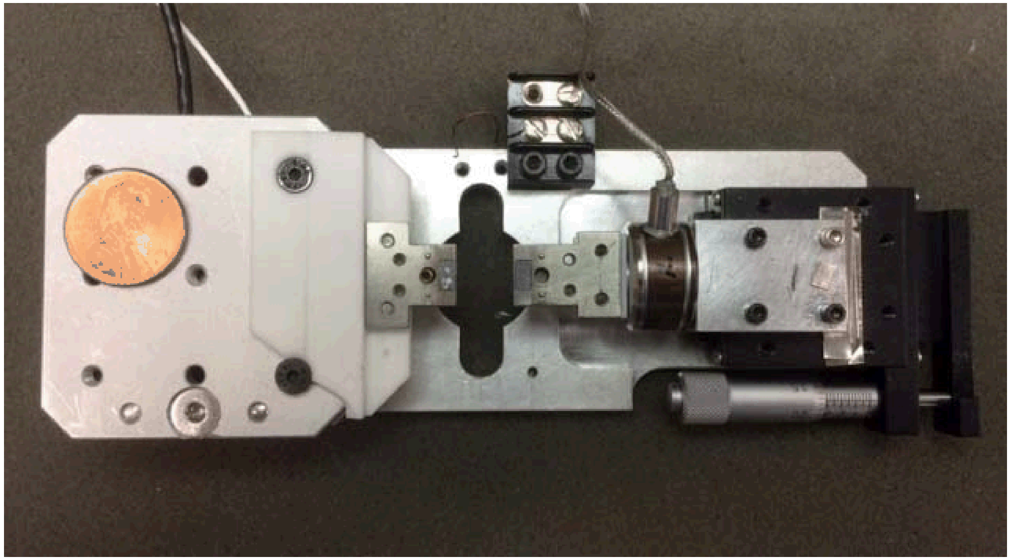
NC NiFe was selected based on previous reports of high fatigue strengths and a propensity for AGG during fatigue loading

Notched fatigue samples were synthesized to isolate fracture initiation and AGG processes



Single-edge semi-circular notches with 10 μm and 20 μm radii were synthesized using focused-ion beam milling

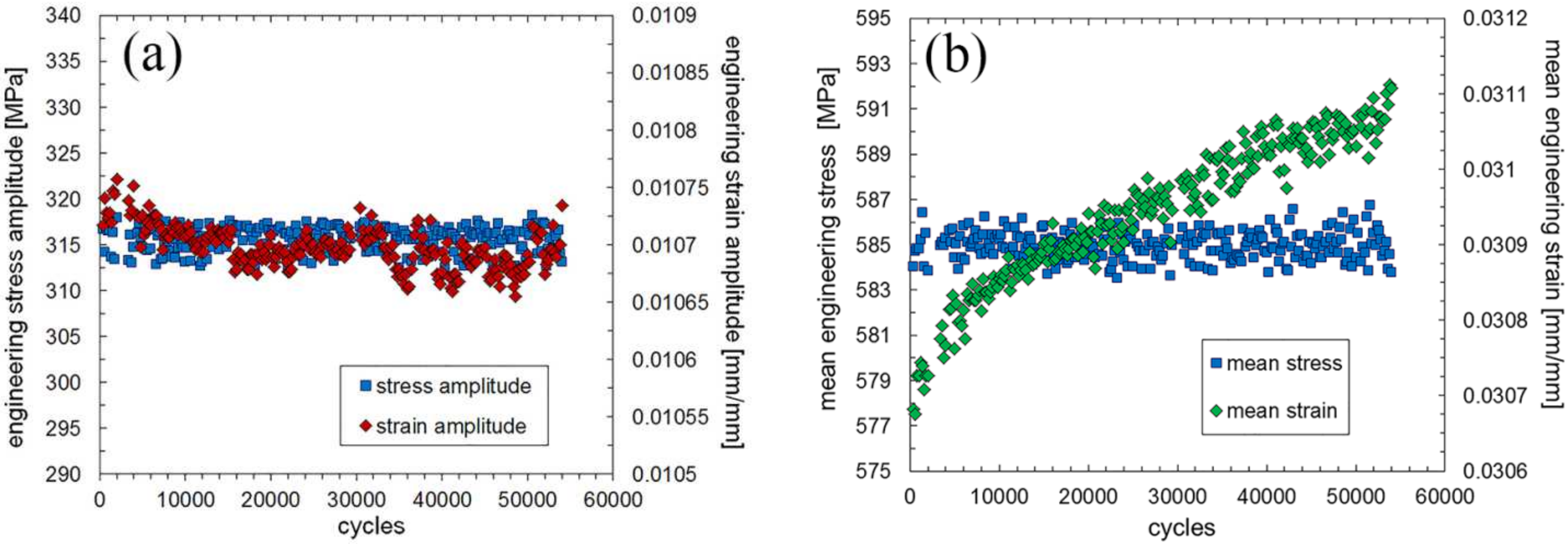
Force-controlled fatigue tests to failure were performed on the notched and unnotched specimens



- Piezo-actuated custom tester capable of force- or displacement-control
- 325 µm total displacement range
- 44.5 N load cell
- system outputs: max/min load and max/min displacement

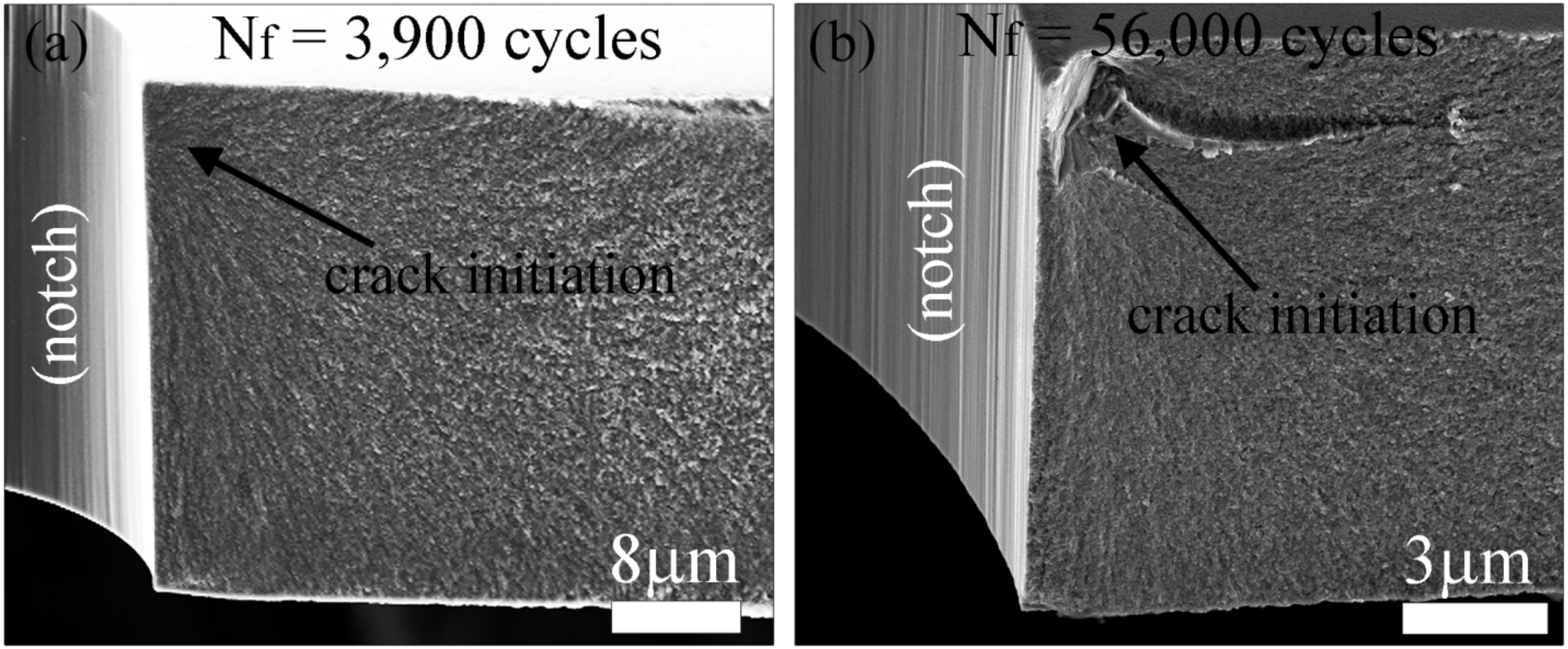
- Fatigue conditions:
  - R≈ 0.3 (f_{min}/f_{max}) [i.e. tension-tension fatigue, to avoid foil buckling]
  - freq : 4-6 Hz (sine)
  - max/min loads and displacements were recorded every 10 cycles

In all cases, a slight cyclic hardening effect was found in the first 10-20 kcycles, in addition to monotonic "ratcheting" throughout the fatigue life

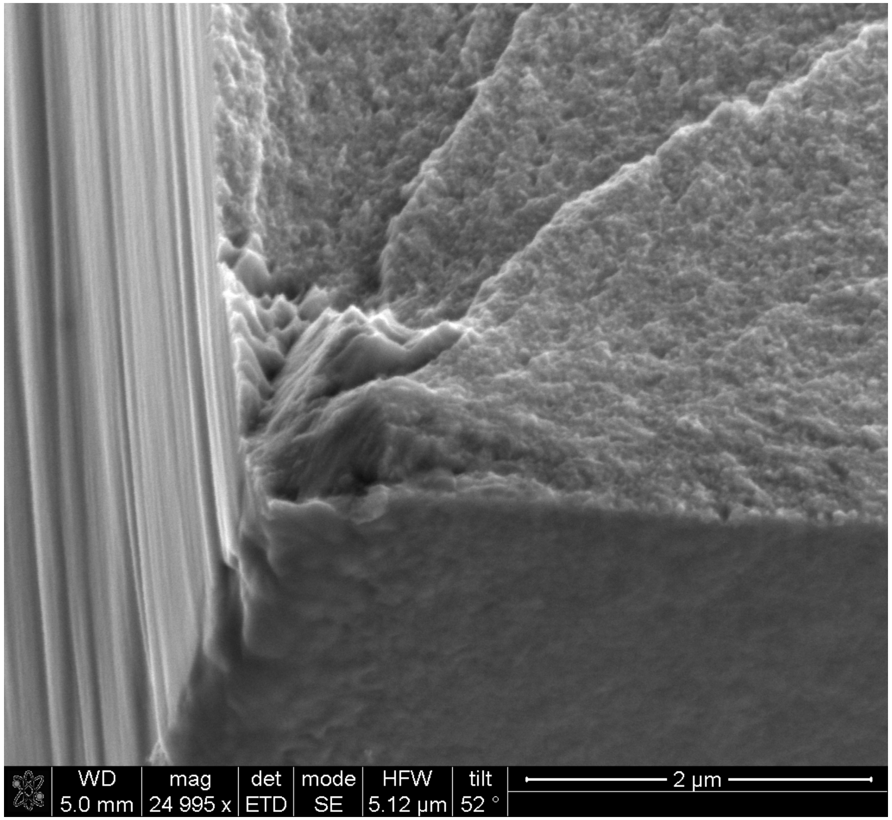


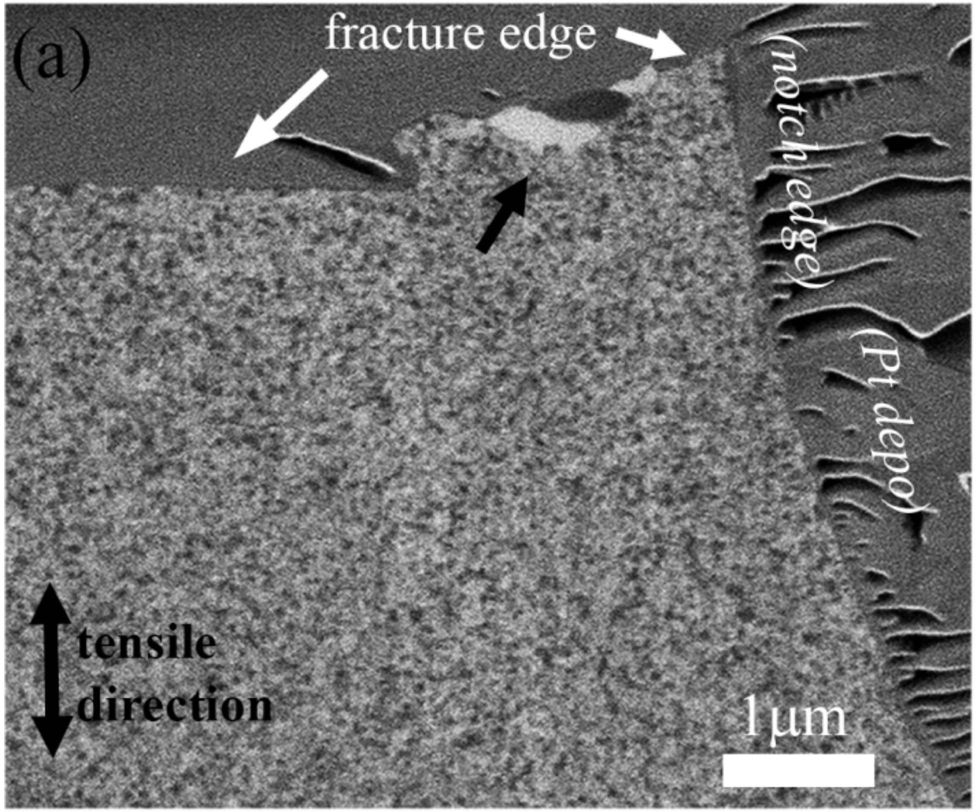
- Cyclic hardening is expected based on work-hardening effects observed during monotonic tension (possibly dislocation exhaustion)
- Monotonic accumulation of strain (separate from the cyclic hardening effects) indicates possible boundary-related activity (e.g. Coble creep mechanisms)

In samples that fractured > ~10,000, the initiation zones consisted of "blocky" protrusions, which were not observed in lower cycle fatigue samples



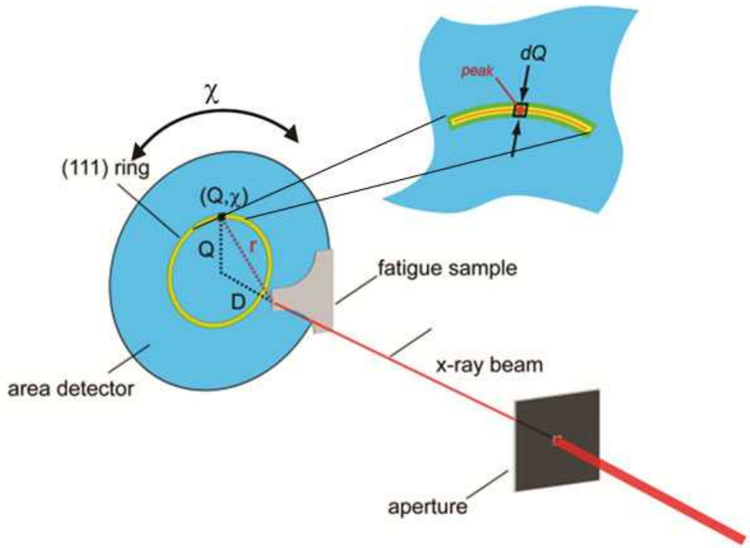
AGG regions were observed underneath the "blocky" fracture features

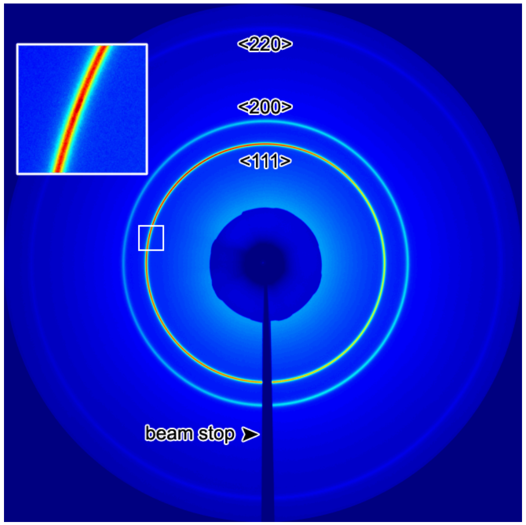




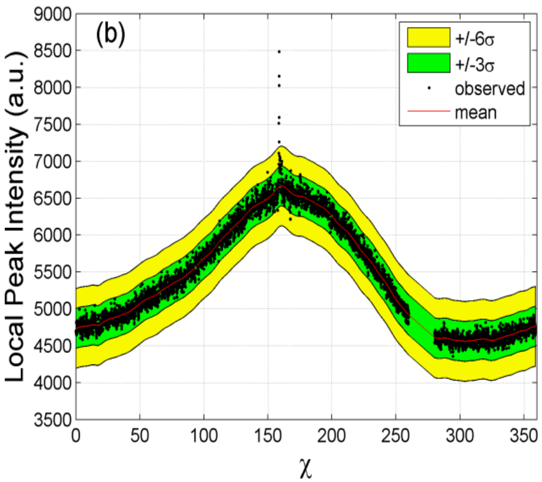
- AGG was observed under the notches with these "blocky" initiation features
- Extensive characterization of the lower cycle fatigue samples showed no signs of AGG

High energy (12.7 keV) synchrotron x-ray diffraction was used to detect regions of AGG at various stages in the fatigue life





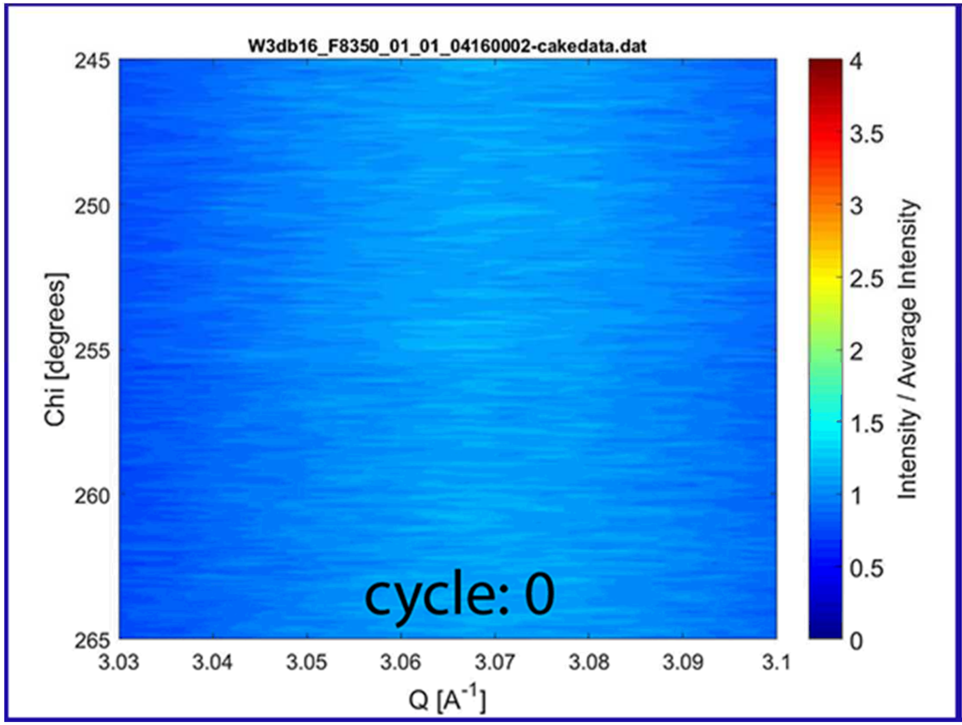
- Using this technique, we can identify clusters of abnormally large grains by identifying statistically significant intensity spikes around the diffraction rings
- Since we know that AGG occurs under the notches, we can scan these areas at different stages in the fatigue life

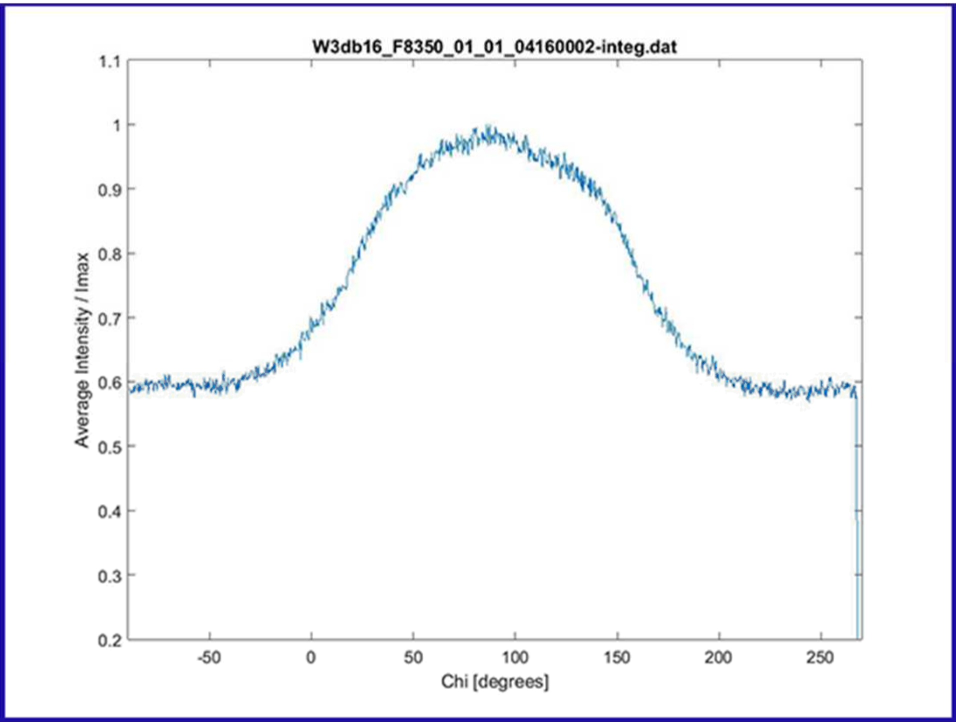


Synchrotron XRD revealed that AGG occurred well before final fracture!!

Caked data
Q= 3.03-3.1 Å^{-1} , Chi= 245-265°

Integration along Chi = -90 to 270deg
Q= 3.059-3.079Å^{-1}





AGG was first detected 30,000 cycles prior to final fracture!!

Conclusions

- Effective fatigue stress concentrations, K_f, between 1.2 and 1.6 were observed in the notched NC NiFe tested in tension-tension fatigue
- Two distinct fracture initiation profiles developed, depending on the number of cycles to failure, N_f
- When Nf > ~10,000 cycles, abnormal grain growth (AGG) was prevalent at crack initiation sites in the notched regions
- When Nf < 10,000 cycles, no AGG was observed – fracture initiation at the sample surface only, without extensive deformation
- Transmission synchrotron XRD revealed AGG occurred up to 30,000 cycles before final fracture

Acknowledgments

- A portion of the microscopy and FIB notch work was performed at the Center for Integrated Nanotechnologies (CINT)
- Synchrotron work was performed at beamline 11-3 at Stanford Synchrotron Radiation Lightsource (SSRL)

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SLAC NATIONAL ACCELERATOR LABORATORY

Thank you

Extra Slides

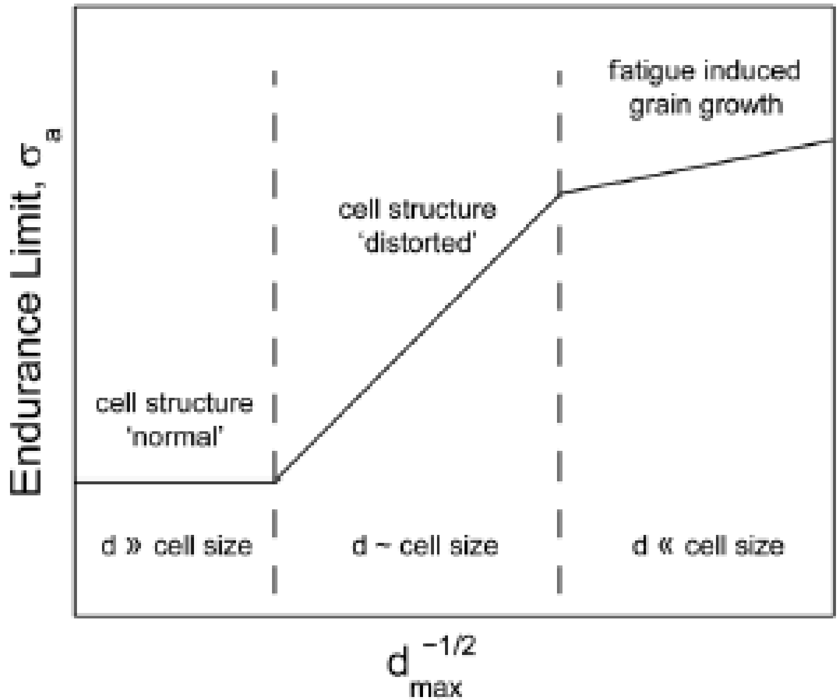
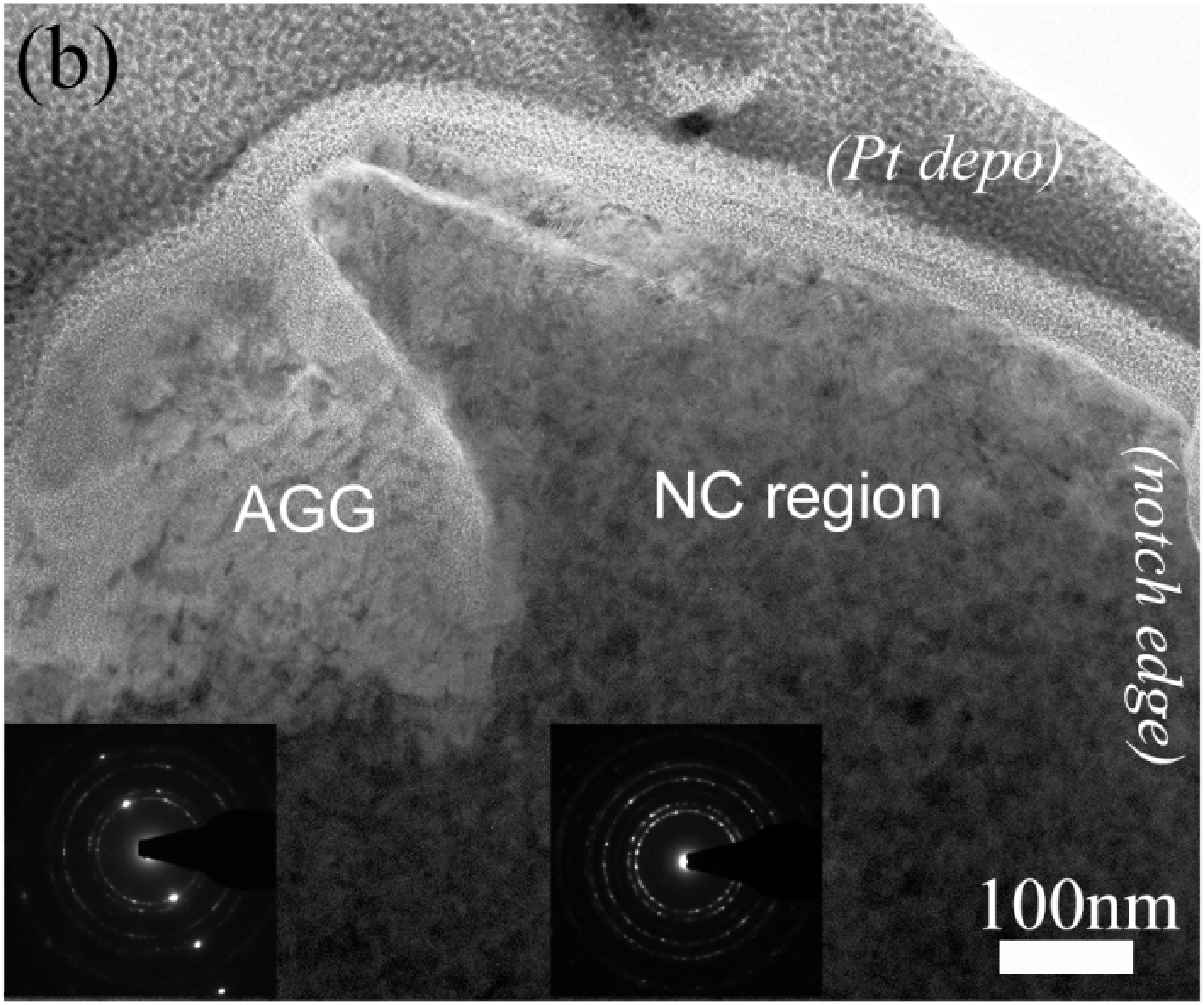
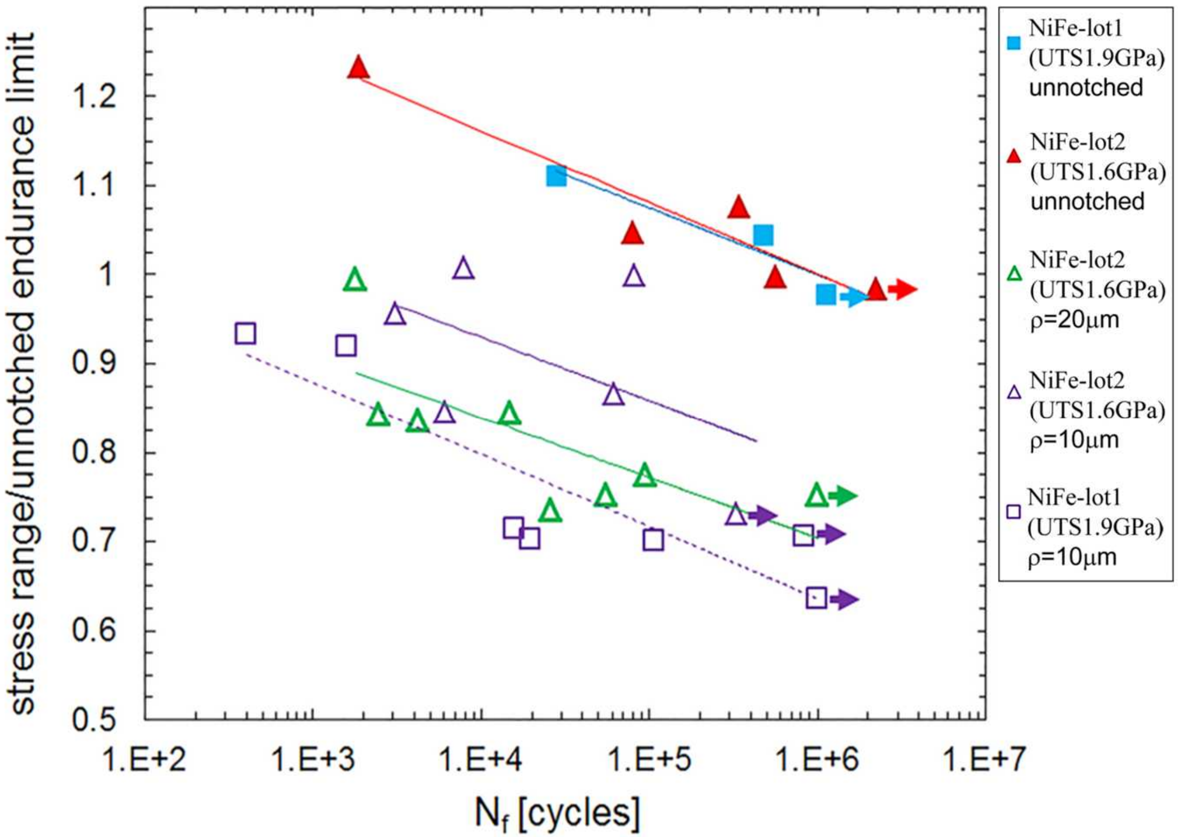


Figure 14. Schematic showing different scaling regimes for fatigue behavior with respect to the grain size.


(b)
(Pt depo)
AGG
NC region
(notch edge)
100nm

S-N curves revealed large effective fatigue concentration factor from the notches



- Overall, there is an emerging trend of decreasing stress concentration with decreasing notch radii, and decreasing strength (similar trend as for CG and UFG samples)