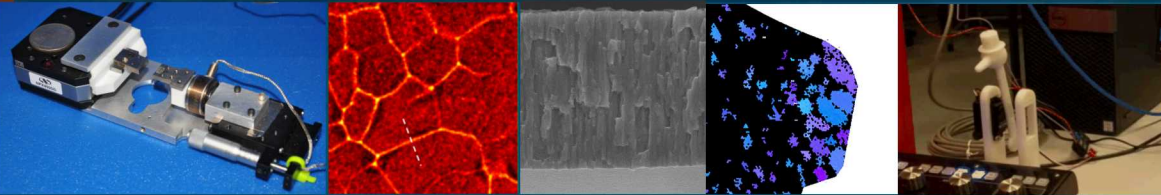
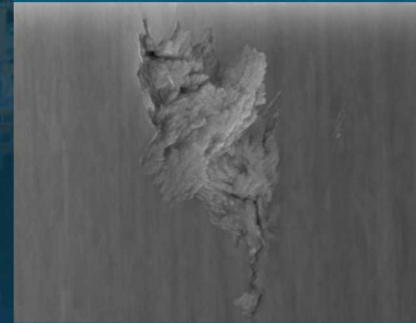


Watching High-cycle Fatigue in Nanocrystalline Pt and Pt-Au



TMS 2020:

Fatigue in Materials:

Fundamentals, Multiscale

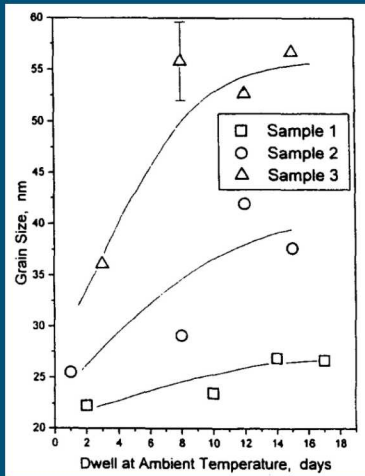
Characterizations and

Computational Modeling

PRESENTED BY

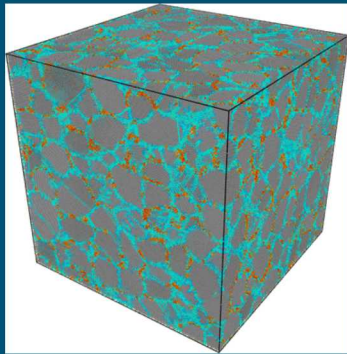
Nathan M. Heckman, Christopher M. Barr, Khalid Hattar,
David P. Adams, Timothy A. Furnish, Brad L. Boyce

2 Motivation – solute segregated nanocrystalline alloys



Gertsman, V.Y. and Birringer, R., *Scr. Metall. Mater.* 1994

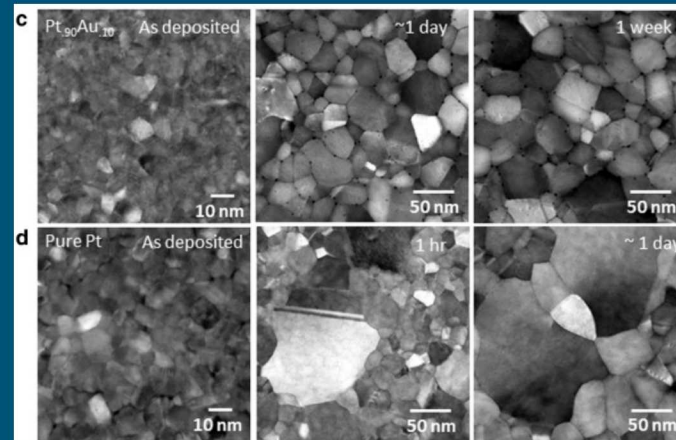
Room temperature grain growth in pure nanocrystalline Cu



FCC Pt Non-FCC Pt Au

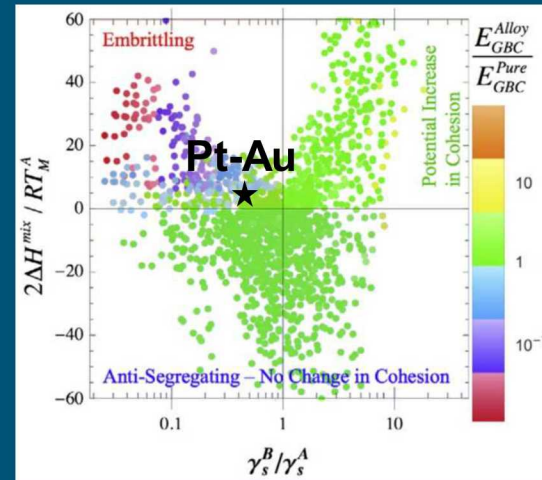
O'Brien, C.J. et al., *J. Mater. Sci.* 2018

Stabilization through grain boundary segregation



Lu, P. et al. *Materialia*, 2019

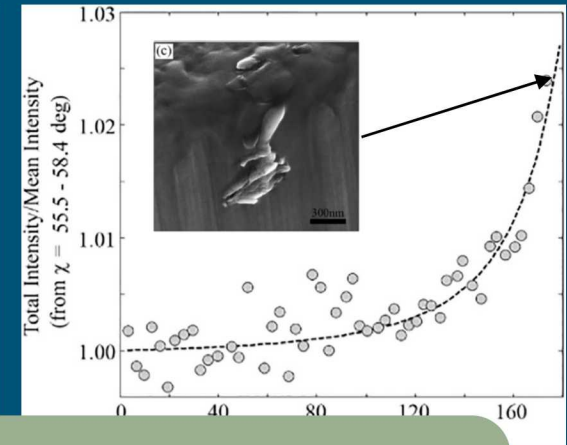
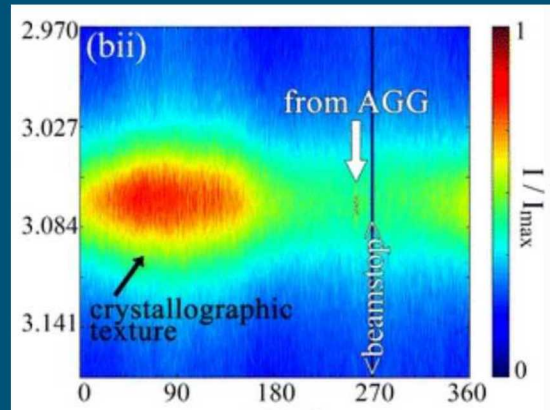
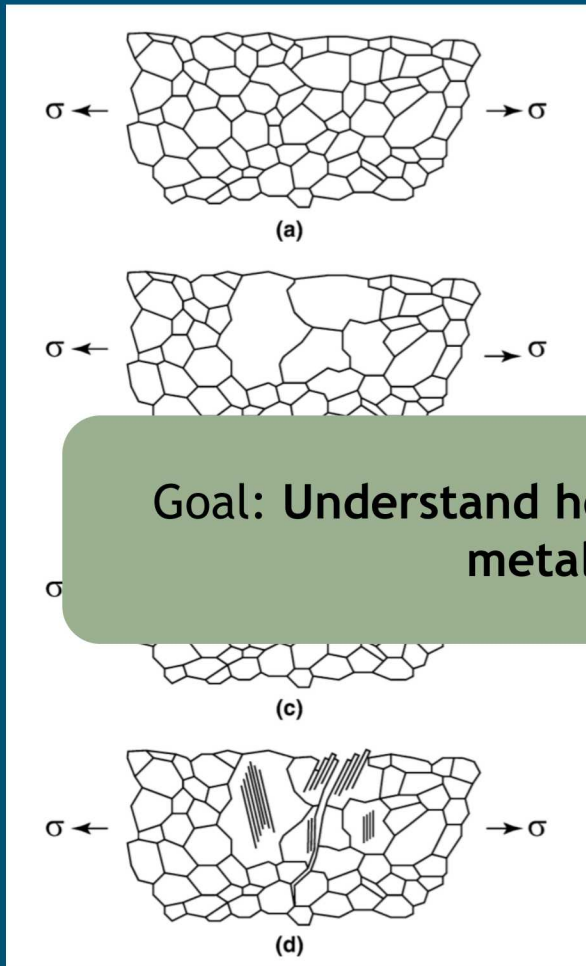
Improved thermal stability



Gibson, M.A., Schuh, C.A., *Acta Materialia*, 2019

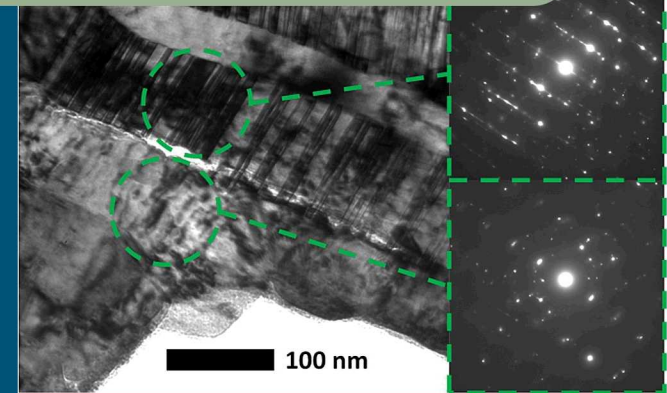
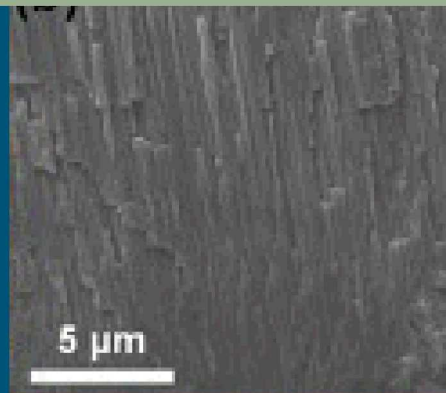
At the cost of grain boundary embrittlement

3 Background – fatigue in nanocrystalline metals



Goal: Understand how complex solute segregated nanocrystalline metals behaves under fatigue loading

Padilla, H., Boyce, B. *Exp. Mech* 2010



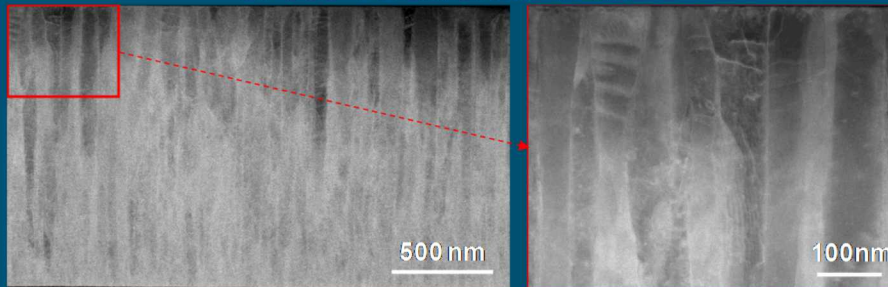
Heckman, NM et al., *Acta Mat.* 2017.

Abnormal grain growth typically observed in simple nanocrystalline FCC metal

Complex nanocrystalline metals (nanotwinned CuAl) can show other fatigue modes

Material System: Nanocrystalline Pt and Pt-Au

Cross-sectional TEM



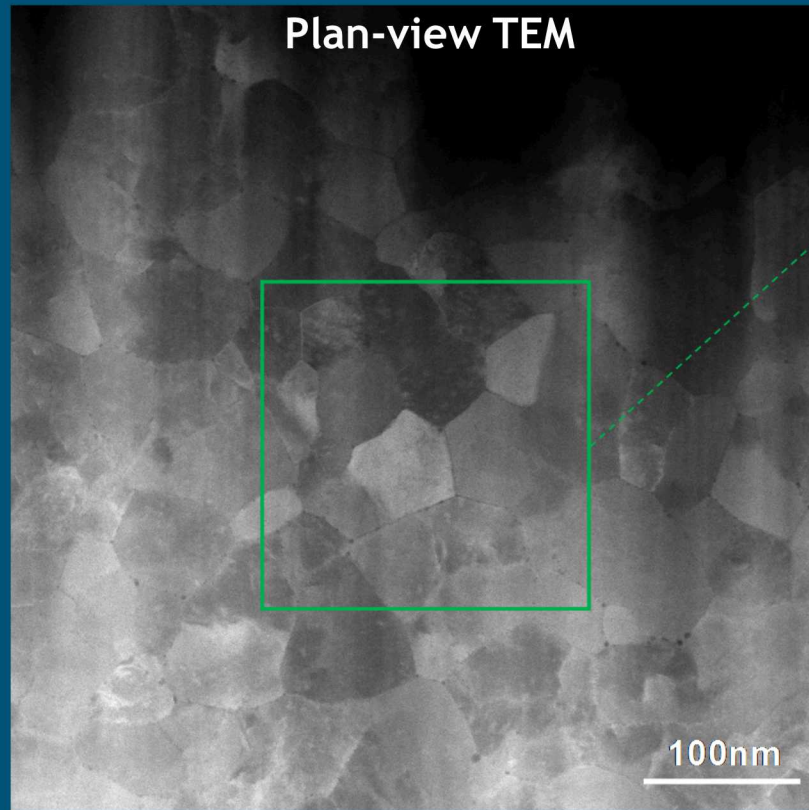
5 μm thick magnetron sputtered films

- Pure Pt, Pt-10at%Au (Pt-10Au)

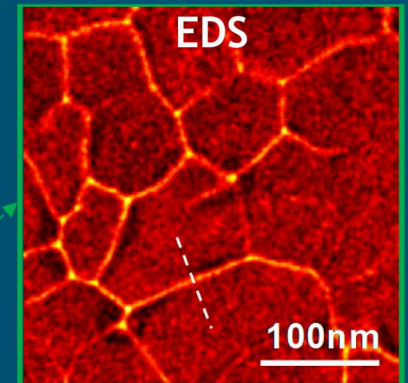
~40 nm mean grain width

Au segregation to boundary observed in as-sputtered films

Plan-view TEM

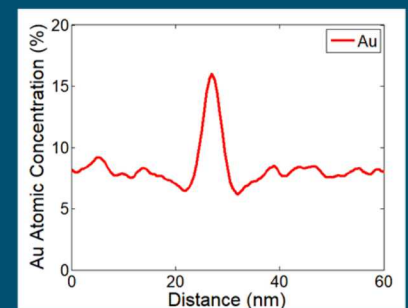


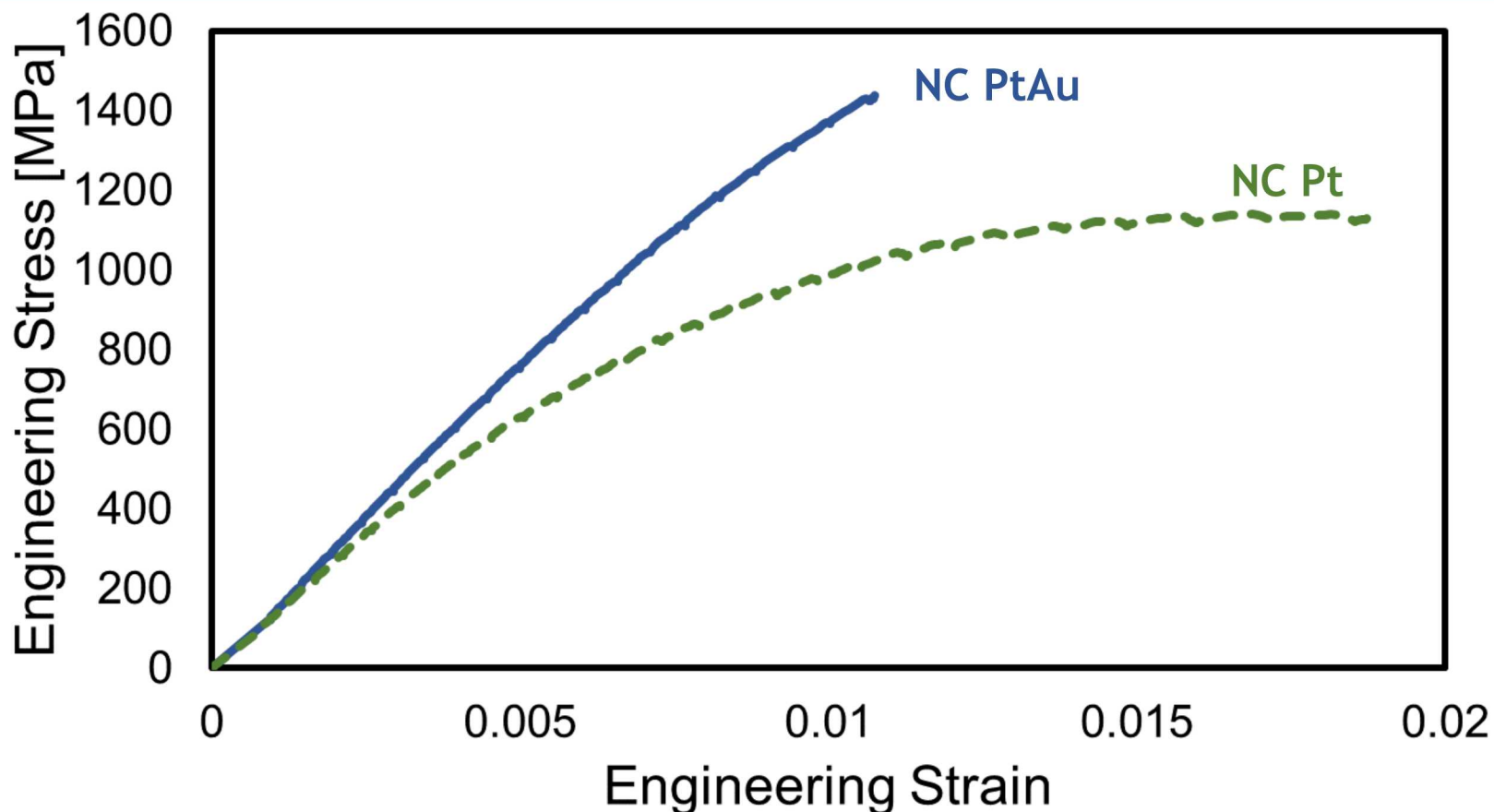
EDS



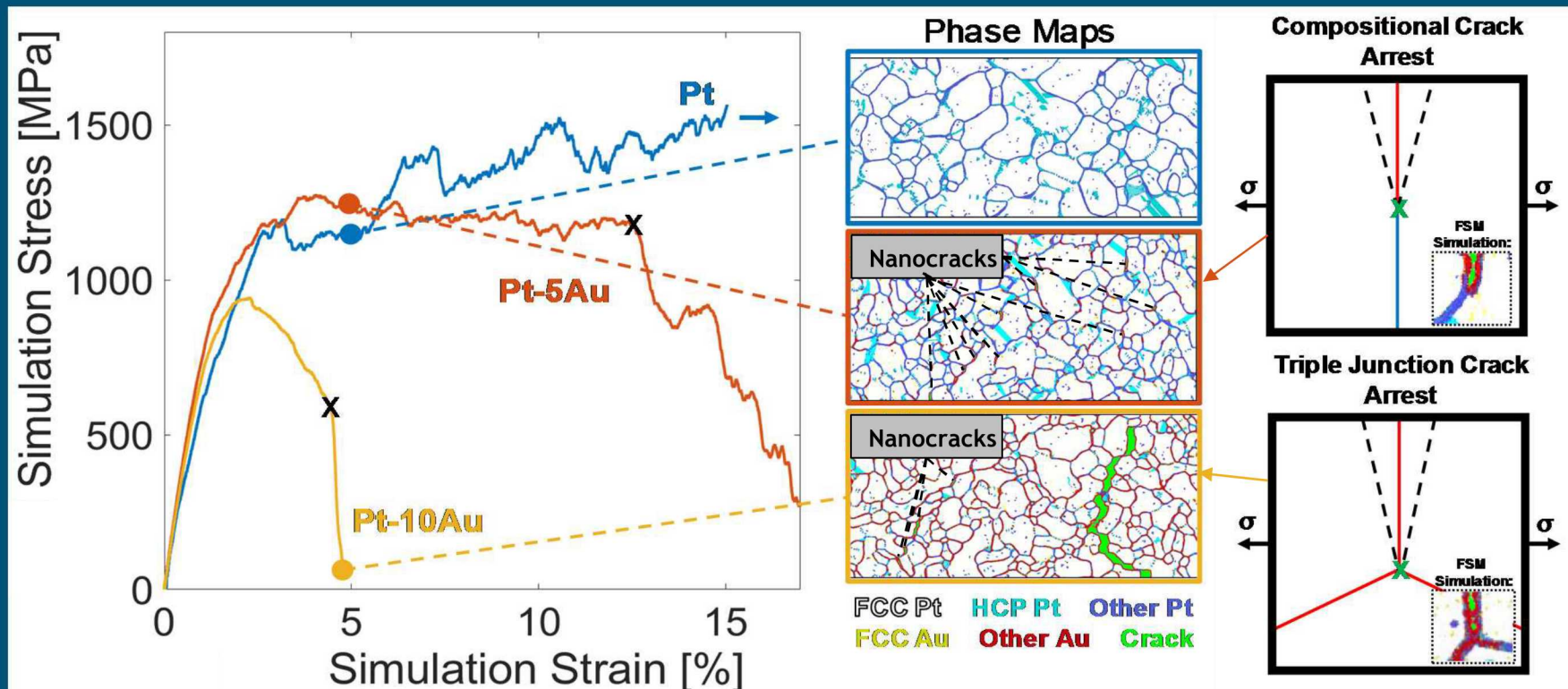
Au Atomic Concentration

0.05 0.1 0.15 0.2 0.25 0.3





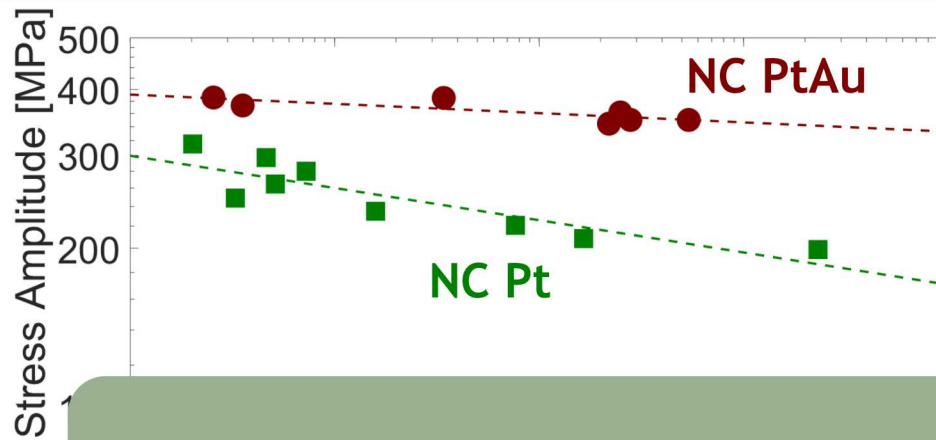
NC PtAu system shows improved strength, however reduced ductility compared to NC Pt



NC PtAu has the potential to maintain some toughness through the formation of nanocrack networks that form due to various crack arrest mechanisms

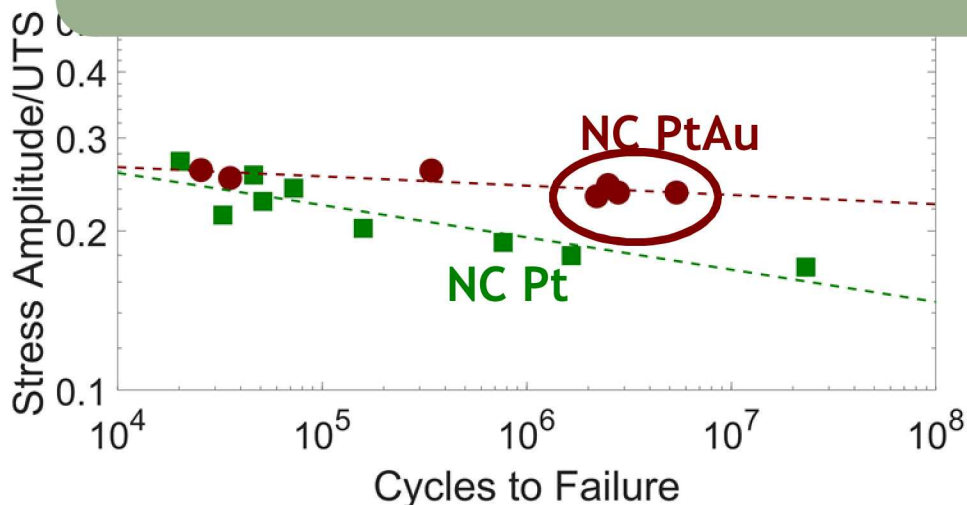
The experimental PtAu is most similar to Pt-10Au in the simulations, which shows the lowest toughness

7 NC Pt and PtAu – Unnotched Wöhler (S-N) Curves



High fatigue strengths in NC PtAu are at least partially due to high tensile strength of material

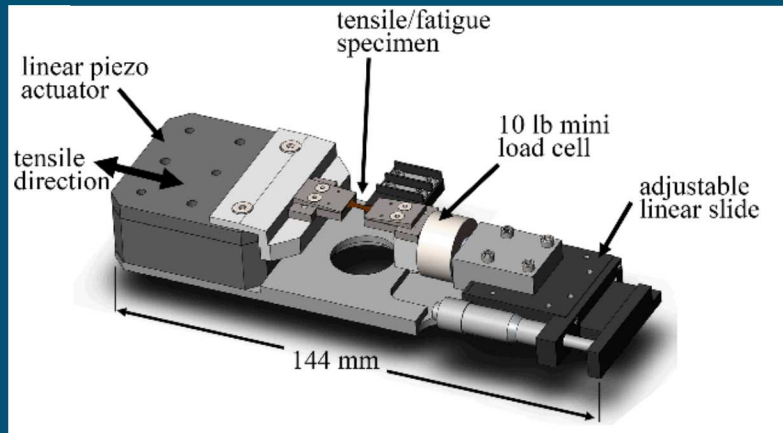
Can we explain these differences in fatigue properties by understanding the cyclic deformation mechanisms of each system?



NC PtAu shows relatively high fatigue strengths at higher fatigue lifetimes, with relatively small Basquine slope

Method: Automated In-situ SEM Fatigue Tests

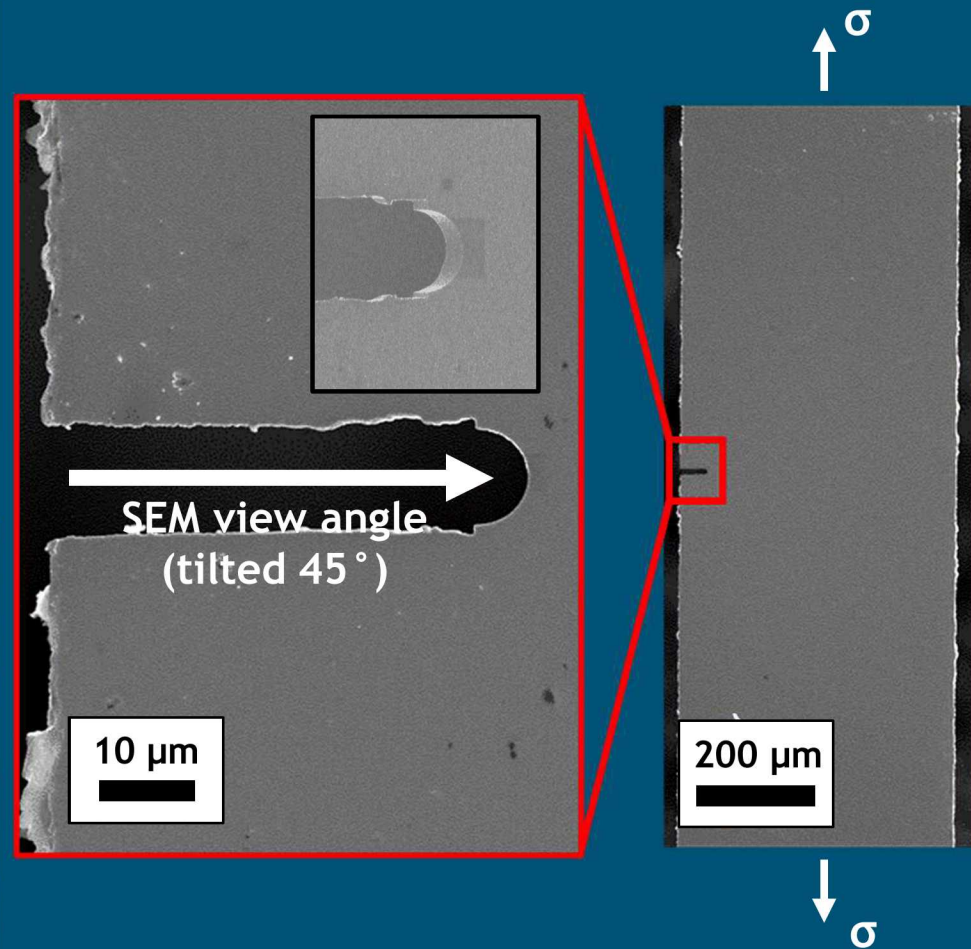
Custom fatigue load frame



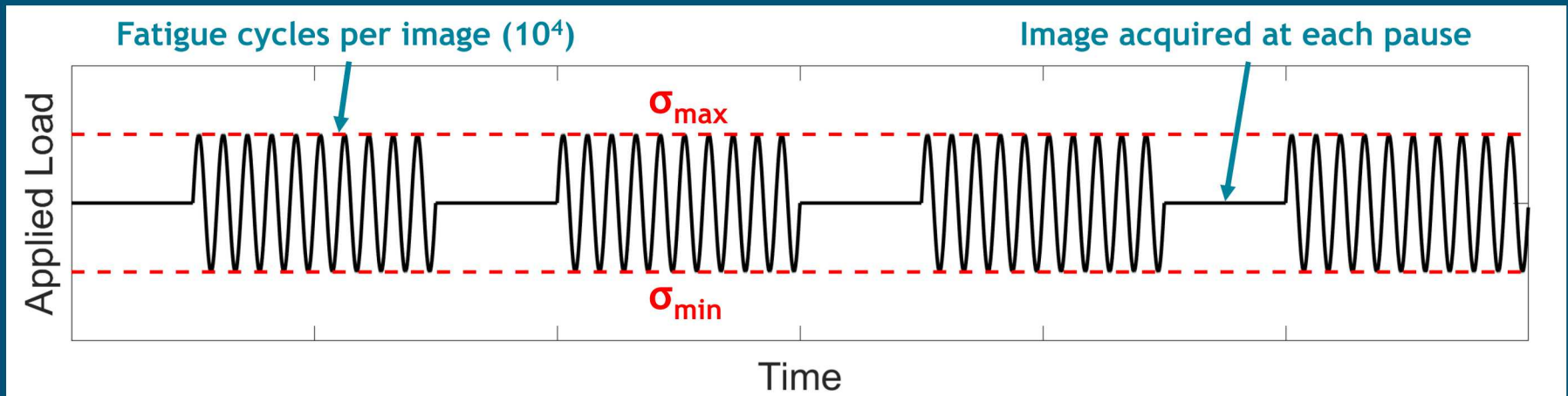
I³SEM at Sandia National Labs*



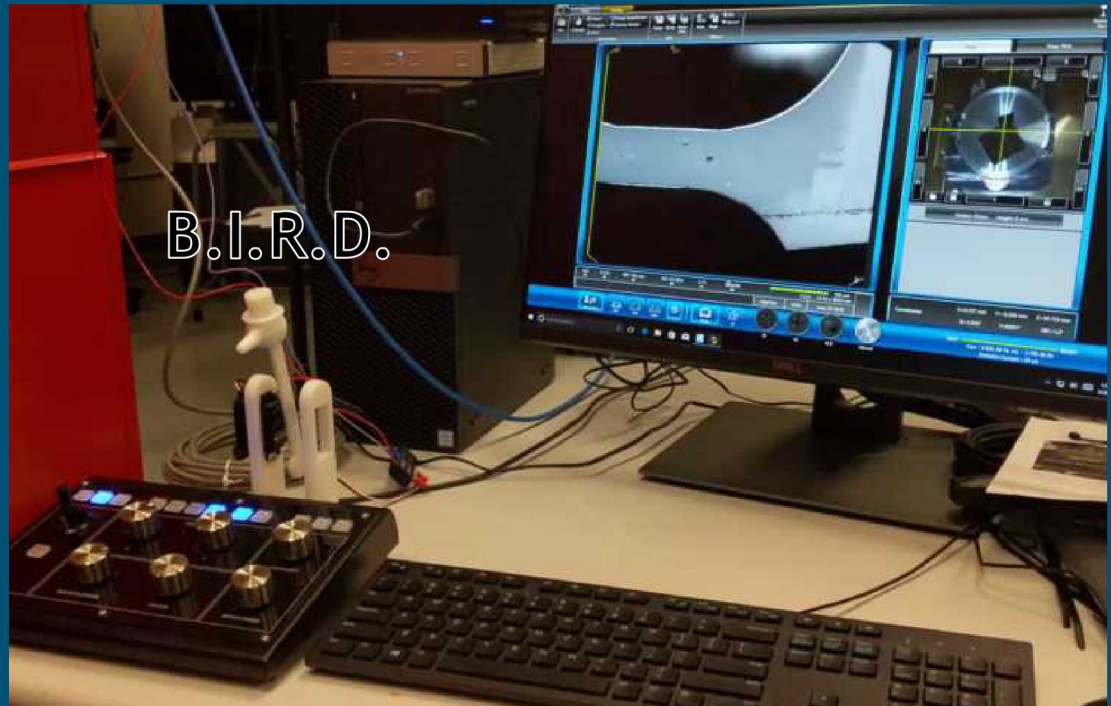
Sample preparation



9 Method: Automated In-situ SEM Fatigue Tests

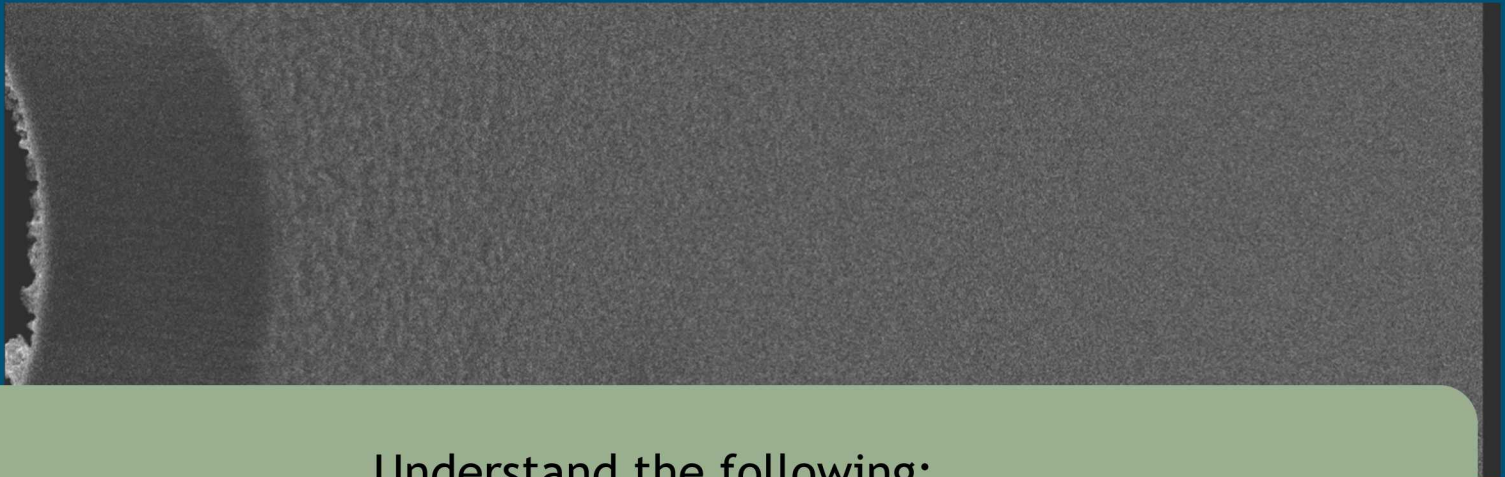


- Automation of entire process with Basic Image Recording Device (B.I.R.D.)
- All fatigue tests performed at 30 Hz, $\sigma_{\min}/\sigma_{\max}=0.3$
- 3-5 tests in 10^6 - 10^7 cycle regime (varying load) for both Pt and Pt-Au
- Longest test performed: 2.7×10^7 cycles over 15 days



Watching High-cycle Fatigue in Nanocrystalline Pt and Pt-Au

NC PtAu
 5.7×10^6 cycles
to failure



Understand the following:
(1) How the crack initiates (2) How the crack propagates

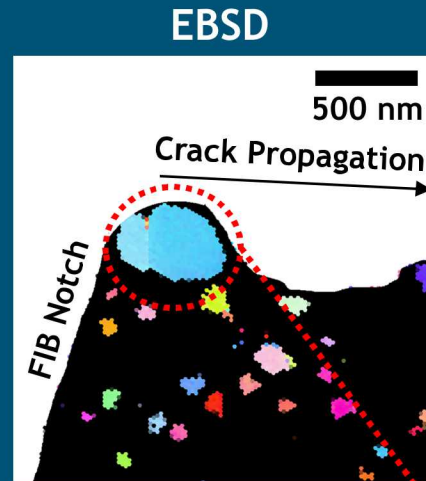
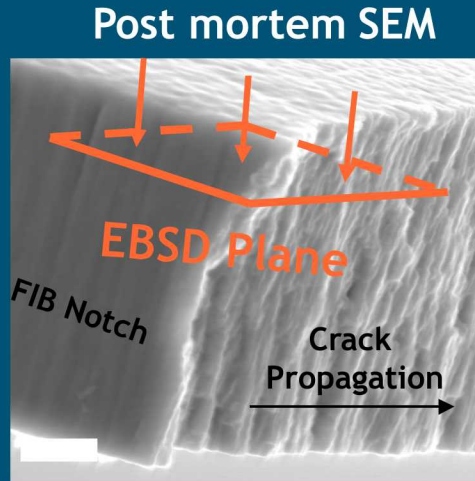
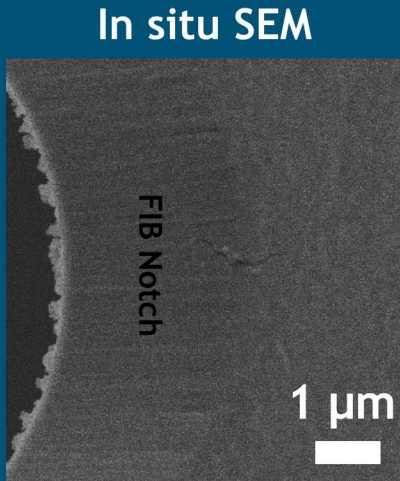
NC Pt
 4.0×10^6 cycles
to failure



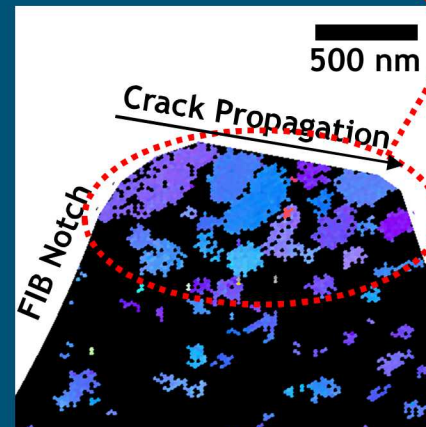
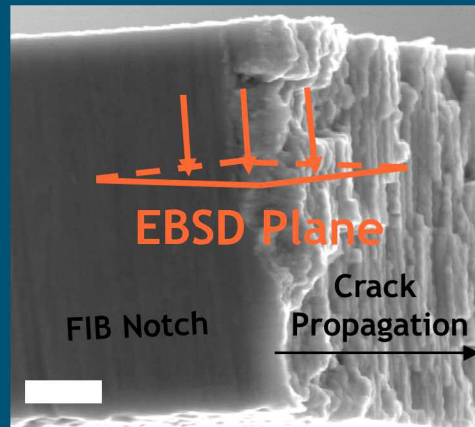
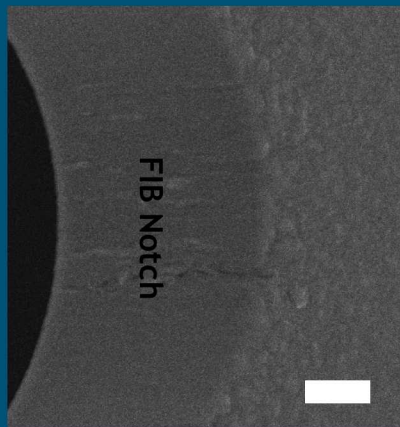
5 μm 

10,000 \times speed \rightarrow 3×10^5 fatigue cycles per second

NC PtAu
 2.8×10^6 cycles
 to failure

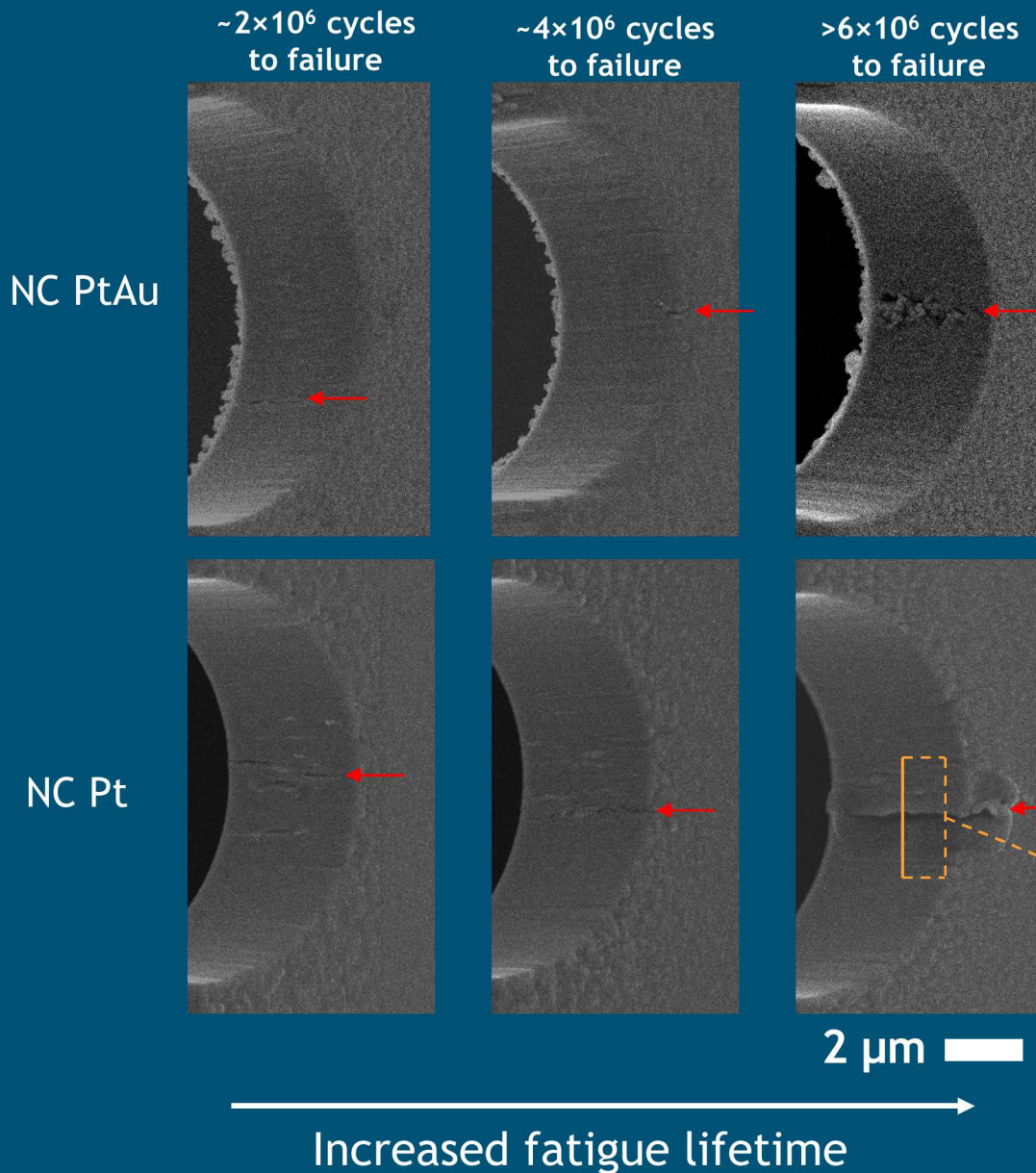


NC Pt
 4.0×10^6 cycles
 to failure

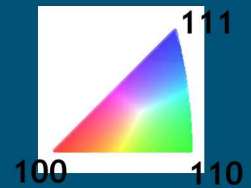
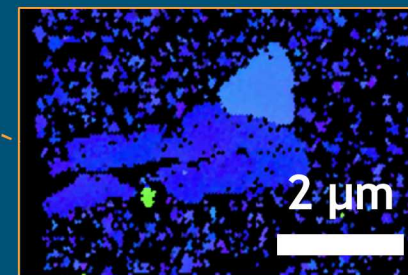


Enlarged grains

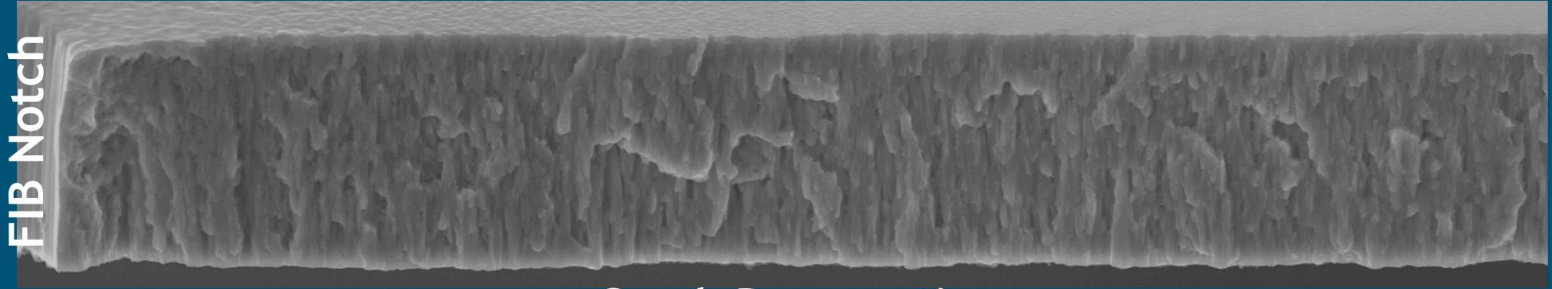
In both NC Pt and PtAu, the crack initiates through abnormal grain growth



Larger grain growth required prior to crack initiation at lower fatigue stresses/higher fatigue lifetimes

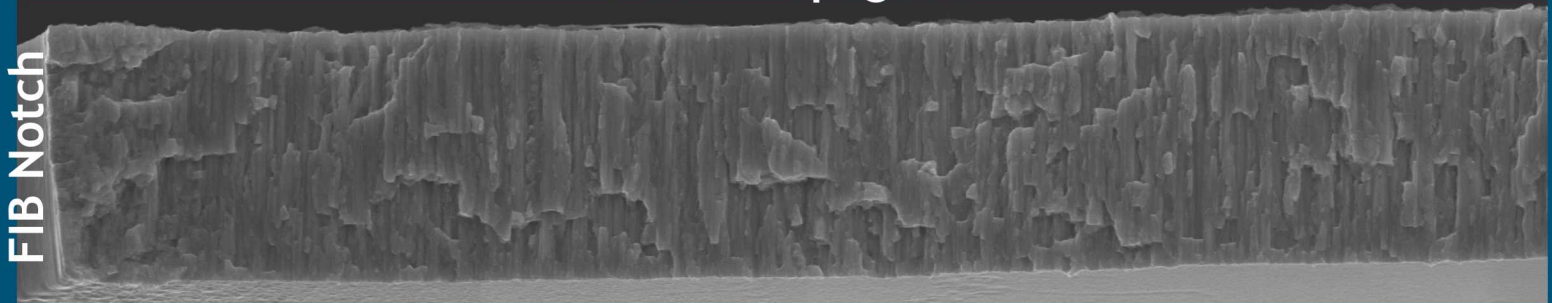


NC PtAu
 2.8×10^6 cycles
 to failure



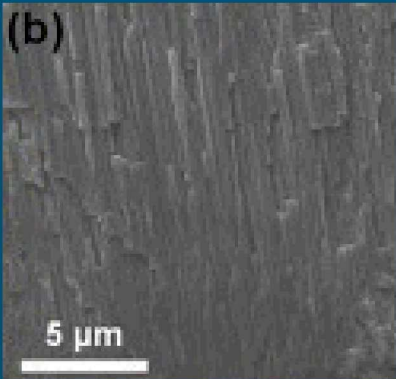
Crack Propagation

NC Pt
 4.0×10^6 cycles
 to failure



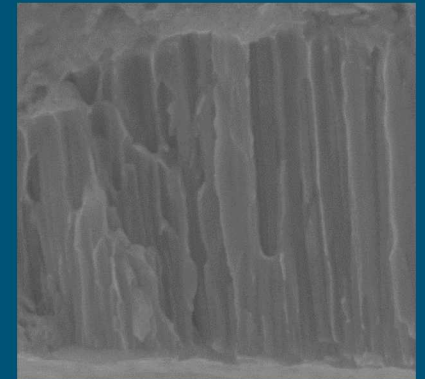
2 μm

Fatigue: Nanotwinned Cu-Al

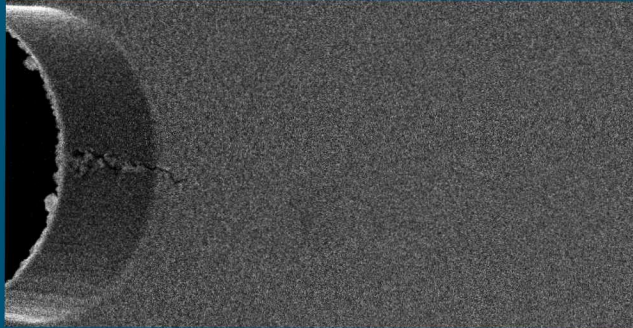


Fracture surface morphologies in both systems consistent with intergranular fracture observed in previous studies

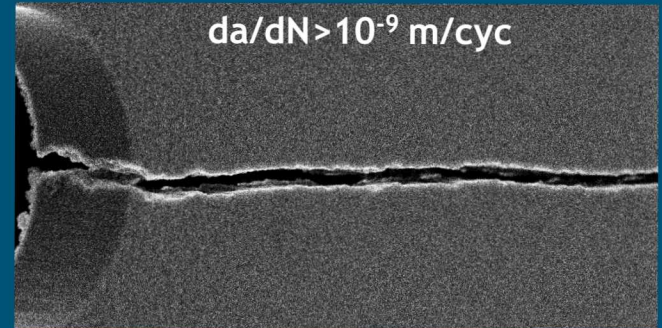
Tension: Nanocrystalline PtAu



NC PtAu
 5.0×10^6 cycles
to failure

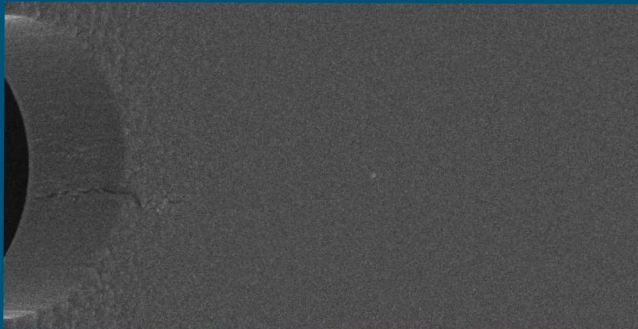


10^4 cycles
 $\Delta K \sim 3.8 \text{ MPa } \sqrt{\text{m}}$

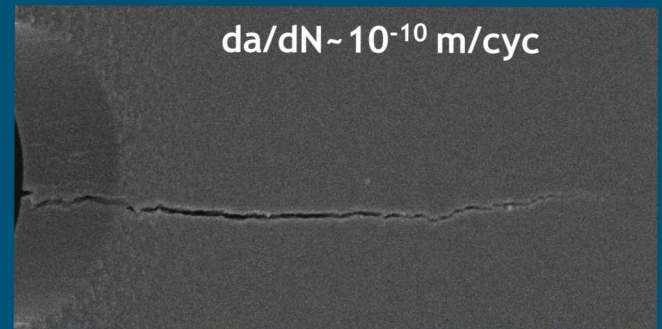


5 μm 

NC Pt
 4.0×10^6 cycles
to failure

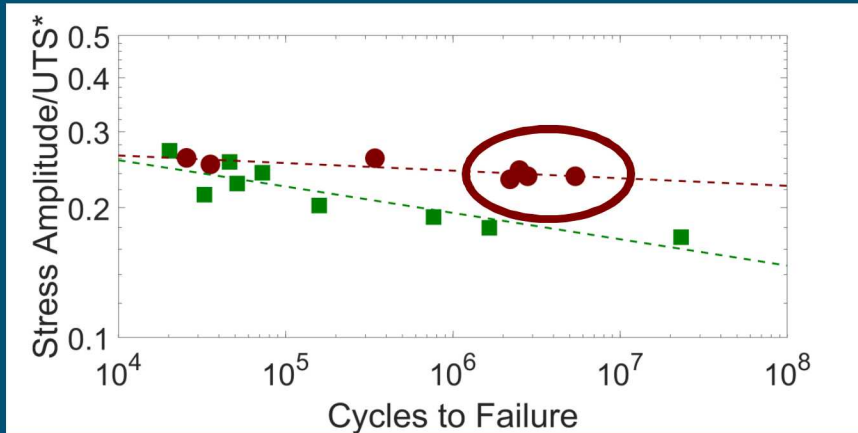


10^5 cycles
 $\Delta K \sim 3.0 \text{ MPa } \sqrt{\text{m}}$

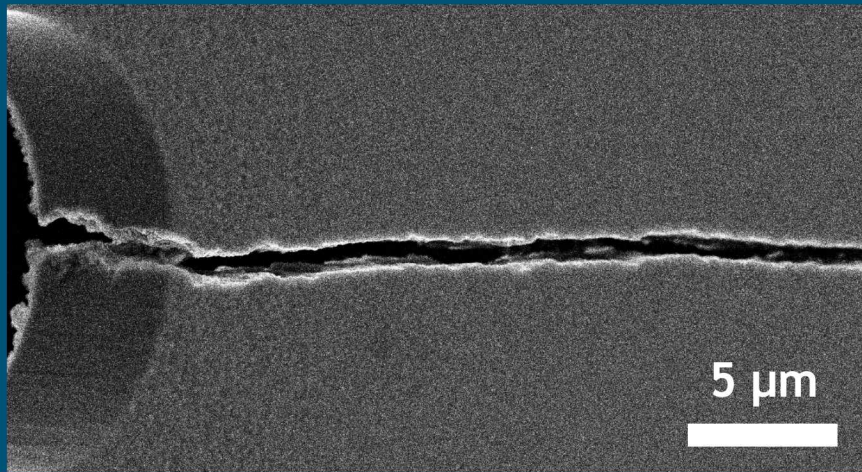


In the high cycle fatigue regime, stable crack propagation was much faster in the PtAu system

Conclusion: **Cyclic** vs **Monotonic** Deformation



High fatigue strength in PtAu,
especially at high lifetimes



Higher crack growth rates in PtAu

Larger grain growth required for crack
initiation at higher regimes



Higher resistance to grain growth in
monotonic loading in NC PtAu

Fracture surfaces consistent with
intergranular fracture



Grain boundary embrittlement in NC
PtAu due to Au segregation

Future work will further investigate the nanoscale deformation mechanisms
through TEM

Thank you!



Relevant talk: Watching cracks heal in TEM in nanocrystalline Pt

4:00 PM Invited

Fatigue-crack Healing in Pure Nanocrystalline Pt Enabled by Boundary Evolution: Christopher Barr¹; Ta Duong²; Daniel Bufford¹; Nathan Heckman¹; Michael Demkowicz²; Khalid Hattar¹; *Brad Boyce*¹;

¹Sandia National Laboratories; ²Texas A&M University