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HIGH-CYCLE FATIGUE IN THE TEM: NANOCRYSTALLINE METALS

Brad L. Boyce[1]*, Khalid Hattar[1], Dan Bufford[1], Chris Barr[1], Zachary Milne[1], and Ryan Schoell[1]

[1]*Sandia National Laboratories, Albuquerque, NM USA*
* *Presenting Author email: blboyce@sandia.gov*

Abstract
In-situ TEM high-cycle fatigue experiments on electron transparent thin films of nanocrystalline Pt and Cu have revealed not only microstructural-sensitive crack propagation, but also unexpected microstructural-scale crack healing. Based on the experimental observations, atomistic modeling, and continuum-scale microstructural modeling, the mechanism appears to be crack flank cold welding facilitated by local compressive microstructural stresses and/or grain boundary migration. While these observations are specific to pure nanocrystalline metal thin films under a high-vacuum environment, there are potentially much broader ramifications. The existing observations can be used to help rationalize suppressed fatigue crack propagation rates in vacuum, subsurface, or under contact-inducing mixed-mode stresses; and even the precipitous decline in propagation rates near the fatigue threshold.

1. Introduction
High-cycle fatigue testing at any length scale requires specialized instrumentation capable of precision controlled forces applied over long times and/or at high frequencies. In a joint collaboration with Bruker-Hysitron, we have demonstrated the ability to perform precions force-controled high-cycle fatigue (HCF) experiments on electron transparent thin films at frequencies up to 300 Hz and force amplitudes on the order of 10 μN, under tension-tension conditions at stress ratios in the vicinity of R~0.4. Using a Si-MEMS based push-to-pull device, any electron-transparent thin film can be interrogated for nanoscale HCF response. In this study, we examine nanocrystalline metals, whose polycrystalline microstructure allows for the direct observation of microstrcutral interactions at the nanoscale, including crack arrest, crack deflection, and corresponding microstructural evolution including grain boundary migration and grain growth. Grain mapping via the precession technique, automated crystal orientation mapping (ACOM), before and after fatigue sequences allow for a more detailed interpretation of crack-microstructure interactions.

2. Results
Direct observations of in-situ high-cycle fatigue response in nanocrystalline Pt and Pt-Au have led to several observations of

a. Crack propagation: crack growth rates << 10^{-12} m/cycle were directly observed: growth rates that are generally thought to represent threshold cracking. While calculation of ΔK is imprecise due to the complexity of microstructural-scale stresses and the difficulty of accurate film thickness values, the approximate values for ΔK are in the vicinity of 1 MPa√m.
b. Crack arrest: triple junctions were observed to lead a propagating fatigue crack to arrest, requiring elevation of the stress levels to overcome the obstacle.
c. Crack deflection: cracks were found to deflect onto crystallographic planes that appear to align with the crystallographic slip system traces of the corresponding crystal orientations.
d. Crack healing: In three separate cases, deeply deflected fatigue cracks appeared to retreat by approximately one grain diameter, on the order of 10-30 nm. Subsequent propagation occurred on a completely new crack path in a different direction, further supporting the notion that the previous crack location now can support load transfer. This healing was attributed in one case to a grain boundary migration process that served to induce contact and cold welding between the crack flanks.
e. Grain boundary migration: Detailed images and crystal orientation mapping allows for determination of boundary migration, including a specific example of a viscinal $\sum 3$ twin boundary migrating. Subsequent recreation of the microstructure in a molecular dynamics simulation was able to recreated the conditions for boundary migration.

f. Grain growth: Through the use of ACOM crystal orientation mapping, specific details of grain growth ahead of the fatigue crack were directly observed. These details are comparable to post-mortem grain growth observations performed on larger-scale specimens.

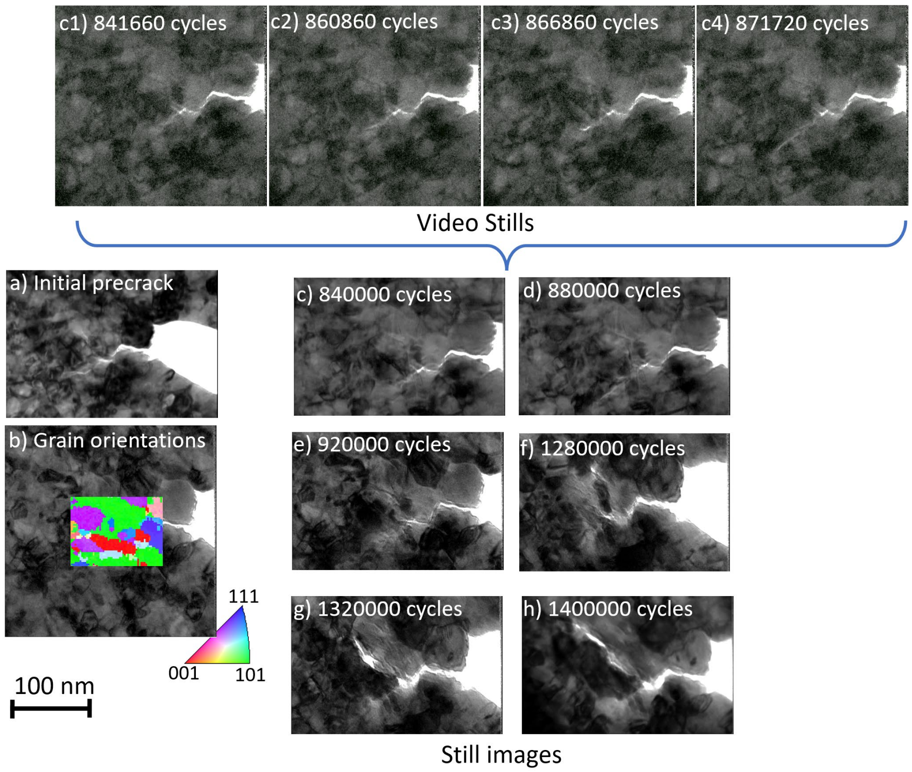

Fig.1 – An example of nanoscale crack propagation under high-cycle fatigue conditions in nanocrystalline Pt. Examples like this are used to elucidate crack propagation mechanisms at the nanoscale including the possibility of crack healing processes.

These observations have a number of important implications. The possibility that fatigue cracks have a process for partial healing runs counter to the fundamental principles of fatigue damage. In particular, the traditional logarithmic representation of crack growth in terms of da/dN vs. ΔK precludes the possibility of negative crack growth rates. A rational model for the fatigue threshold was developed on the basis of intermittent nanoscale crack healing, as an alternative to existing dislocation-based arguments for the threshold. The observations also could help explain the suppression of fatigue crack propagation in subsurface or oxygen-free environments.

3. Conclusions

In-situ TEM high-cycle fatigue observations are are revealing new details on crack propagation processes, including crack-microstructure interactions and the coupled evolution of microstructure with crack propagation. This technique could be extended to study nanoscale HCF processes in a wide range of metals and ceramics.

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