doi:10.1017/S1431927616009521

Microsc. Microanal. 22 (Suppl 3), 2016

Combining Orientation Mapping and *In Situ* TEM to Investigate High-Cycle Fatigue and Failure

Daniel C. Bufford[1], Douglas Stauffer[2], William M. Mook[3], S.A. Syed Asif[2], Brad L. Boyce[4], and Khalid Hattar[1]

[1.] Sandia National Laboratories, Radiation-Solid Interactions, Albuquerque, NM, USA
[2.] Hysitron, Inc., Eden Prairie, MN, USA
[3.] Sandia National Laboratories, Center for Integrated Nanotechnologies, Albuquerque, NM, USA
[4.] Sandia National Laboratories, Materials Mechanics and Tribology, Albuquerque, NM, USA

Material fatigue proves to be a limiting factor in many engineering cases. Repeated cyclic loading, even at stresses well below the monotonic yield stress of the material, leads to the accumulation of microstructural damage, crack initiation, crack growth, and eventual failure. In terms of the number of loading cycles, the high cycle regime of $>10^4$ loading cycles is often of interest, although there are cases in which fatigue lifetimes may reach $>>10^7$ cycles. Cyclic loading experiments with bulk specimens can determine fatigue lifetimes, however, the crack initiation and early crack growth regimes are more difficult to examine experimentally, as these processes take place at the grain level, often inside of the specimen. These incipient processes are important to bulk material, as this is where the failure process begins. Furthermore, micro- and nanoscale devices are directly affected in this regime. Mechanical testing performed *in situ* inside of the transmission electron microscope provides the ability to see these processes in real time as they occur; however, to date no general capability for fatigue loading at frequencies above a few Hz exists. Here, the authors demonstrate a newly developed mechanical testing capability that allows controllable quantitative cyclic loading to be performed *in situ* inside of a transmission electron microscope at frequencies in the hundreds of Hz. This capability, built into a nanoindentation TEM holder, is capable of reaching 10^6 cycles within an hour. Here, observations of microstructural changes near propagating cracks in nanocrystalline Cu are presented.

Mechanical testing specimens were prepared from magnetron sputtered Cu films on single crystal, UV-ozone cleaned NaCl substrates that were heated to 65 °C for one hour. The 75 nm-thick films were deposited from a 99.997% purity target at a base pressure of 4×10^{-7} torr. Small pieces of the films were floated in deionized water and isopropanol mixture, and then collected on Hysitron Push-to-Pull devices. These devices provide a micromechanical Si test frame that converts the indentation load to stabilized tensile load. Focused ion beam milling (Ga ions, 30 keV, 7 nA rough milling; 10 keV, 30 pA final sample shaping) was employed to remove excess film, leaving a tensile specimen [1]. Mechanical loading experiments were performed at 200 Hz with mean loads of ≈100 μN, and load amplitudes of ≈50 μN. The total number of accumulated cycles typically exceeded 10^5 cycles, after which the sample failed, or testing was stopped for additional analysis. Local microstructural change was characterized by bright field TEM video during loading. Testing was periodically paused to collect additional still micrographs, as well as data collection by a precession electron diffraction-assisted automated orientation mapping technique [2]. Observable changes in contrast preceded crack nucleation. Cracks then typically grew stably for a period of time, prior to rapid unstable propagation. Localized microstructural changes near the propagating crack included larger grains oriented differently from the surrounding matrix. In Figure 1, the sample is shown prior to mechanical loading and after accumulating ~180,000 loading cycles. In the latter, a crack had nucleated and propagated stably several hundred nanometers. Grain growth was apparent near the crack. The orientation map in Figure 1c confirms the large grains at the crack tip.

The incipient nanoscale cracks observed in this work represent a portion of the fatigue lifetime that has historically been difficult to access experimentally. This work and future studies hold promise for a better understanding of this regime. Additionally, the orientation map data can readily be converted for use in phase field and crystal plasticity models, opening up exciting opportunities for synergistic studies with experiments and models on the same scale. Studying fatigue crack initiation and propagation at the nanoscale improves the understanding of the fundamental microstructural change processes, and may provide insight into more atypical phenomena like fatigue-induced grain growth [3]. This work is promising for better understanding and ultimately improving fatigue lifetimes in both bulk materials and micromechanical devices.

References:

[1] A Kobler, *et al*, Ultramicroscopy **128** (2013), p.68.
[2] EF Rauch, *et al*, Zeitschrift Für Kristallographie **225** (2010), p.103.
[3] BL Boyce and HA Padilla, Metallurgical and Materials Transactions A-Physical Metallurgy and Materials Science **42A** (2011), p.1793.

[4] Work performed by K.H., B.L.B., and D.C.B. was fully supported by the Division of Materials Science and Engineering, Office of Basic Energy Sciences, U.S. Department of Energy. Work by W.M.M. was performed, in part, at the Center for Integrated Nanotechnologies, an Office of Science User Facility operated for the U.S. Department of Energy (DOE) Office of Science under proposal #U2014A0026. Sandia National Laboratories is a multi-program laboratory managed and operated by Sandia Corporation, a wholly owned subsidiary of Lockheed Martin Corporation, for the U.S. Department of Energy's National Nuclear Security Administration under contract DE-AC04-94AL85000.

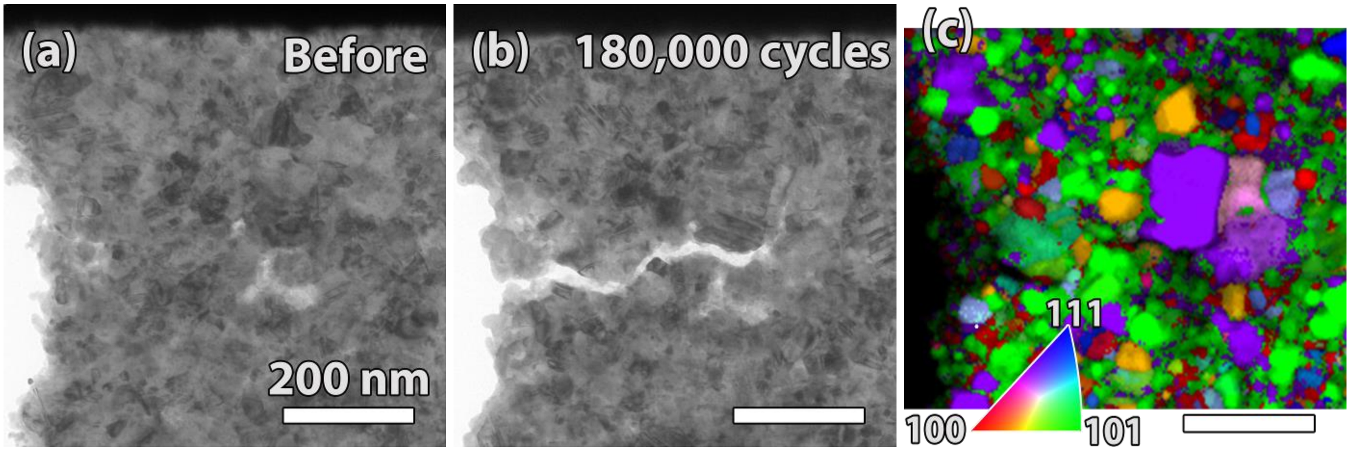

Figure 1. Nanocrystalline Cu sample (a) before mechanical testing and (b,c) after accumulating 180,000 loading cycles. The same area is presented in bright-field TEM in (a) and (b), and as an inverse pole figure orientation map in (c). The scale bar in each image is 200 nm.