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TEM *in situ* cube-corner indentation analysis using ViBe motion detection algorithm

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ABSTRACT

Transmission electron microscopic (TEM) *in situ* mechanical testing is a promising method for understanding plasticity in shallow ion irradiated layers and other volume-limited materials. One of the simplest TEM *in situ* experiments is cube-corner indentation of a lamella, but the subsequent analysis and interpretation of the experiment is challenging, especially in engineering materials with complex microstructures. In this work, we: (a) develop MicroViBE, a motion detection and background subtraction-based post-processing approach, and (b) demonstrate the ability of MicroViBe, in combination with post-mortem TEM imaging, to carry out an unbiased qualitative interpretation of TEM indentation videos. We focus this work around a Fe-9%Cr oxide dispersion strengthened (ODS) alloy, irradiated with Fe^{2+} ions to 3 dpa at 500 °C. MicroViBe identifies changes in Laue contrast that are induced by the indentation; these changes accumulate throughout the mechanical loading to generate a “heatmap” of features in the original TEM video that change the most during the loading. Dislocation loops with **b** = ½ <111> identified by post-mortem scanning TEM (STEM) imaging correspond to hotspots on the heatmap, whereas positions of dislocation loops with **b** = <100> do not correspond to hotspots. Further, MicroViBe enables consistent, objective quantitative approximation of the **b** = ½ <111> dislocation loop number density.

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1. Introduction

In situ transmission electron microscopy (TEM) mechanical testing techniques inherently lend themselves to qualitative observations that can provide an improved understanding of the microstructural mechanisms of plasticity, especially in shallow ion irradiation layers, thin films, and other volume-limited materials. Ion irradiation is increasingly used as an accelerated, low-cost method to emulate neutron irradiation effects in metals and alloys. However, a major difference between neutron and ion irradiation is that the damage from ion irradiation occurs in only a shallow, few-μm thick region at the surface of the material. Whereas this surface ion irradiated layer has little impact on the mechanical response when not isolated from the unirradiated bulk [1], this layer can be separated from the bulk by creating an *in situ* TEM-scale specimen contained entirely within the irradiated region. This enables one to assess the mechanical performance of the irradiated region alone, without influence from the bulk. As such, an ion irradiated material is uniquely suited for *in situ* TEM mechanical testing and subsequent microstructural analysis.

Amongst the most exciting implications of TEM *in situ* mechanical testing techniques is the ability to conduct mechanical tests on the same length scale as crystal plasticity models. So where there was previously a gap between the modeling and experimental scales, TEM *in situ* mechanical tests can potentially provide direct validation of model-predicted plastic phenomena [2–5]. While one of the simplest TEM *in situ* mechanical testing experiments to conduct is that of indentation on a thin (i.e. electron-transparent) foil, the subsequent analysis and interpretation of the experiment is difficult and presents numerous quantitative and qualitative challenges [5–11]. Quantitatively, the geometry gives rise to a complex stress distribution that is difficult to precisely represent in a finite element-type mesh. But qualitatively, video interpretation is further challenged by several

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factors, including microstructural complexity, non-optimal imaging conditions, drift and vibrations, and human error.

First, discerning individual slip events or dislocation movements becomes increasingly difficult as the material's compositional and microstructural complexity increases. Materials with simple microstructures, such as pure Al, Cu, Mg, or Zn, allow for relatively easy observation of dislocation motion and obstacle interaction. Individual dislocation nucleation and motion has been clearly observed [6,9,12–20] along with grain boundary coarsening and migration [6–8,17,21–27]. Twin boundary formation can also be observed [28–33]. But as material complexity increases, including by way of irradiation, the ability to observe distinct events becomes more difficult and more susceptible to interpretation. There are only limited studies in which individual dislocation motion is seen during *in situ* TEM straining of microstructurally complex materials [34–36].

Next, because most TEM *in situ* mechanical testing holders do not allow for double-tilting (both α and β tilt), the experimenter cannot easily guarantee proper crystallographic orientation with respect to the electron beam so as to set up ideal imaging conditions for observation of dislocation motion. In such a case, one can only conduct detailed microstructural characterization of the sample pre- or post-mortem. Even with this pre or post imaging step, it can be difficult to compare the indentation video to the still microstructure images collected at different tilt angles.

Additionally, once the indenter tip contacts the specimen, mechanical vibrations of the motor driving the tip are transferred to the specimen. This can result in an appearance of "shaking" in the TEM video recorded during the test, which makes it impossible to observe changes from one frame to the next.

Finally, identification of fine features such as dislocation loops or lines in microstructurally complex alloys is subject to interpretation [37]. So even if one is able to see or count features in TEM indentation videos, two different researchers will likely arrive at different results. While some of this subjectivity can be eliminated by using computer-based software to help identify or count microstructural features, these types of software are also less effective for interpreting the images from microstructurally complex materials, such as ion irradiated engineering alloys currently used or under consideration for nuclear reactor structural and cladding components.

In this work, we study an Fe^{2+} irradiated model Fe-9%Cr oxide dispersion strengthened (ODS) alloy because of its high microstructural complexity. We carry out cube-corner indentation of thin lamellae of this material. Given the microstructural and geometric complexities of these experiments, there is a critical need for a consistent, non-subjective approach to more definitively discern plasticity in the microstructure. The objective of this work, then, is to utilize a motion detection and background subtraction-based post-processing approach to carry out an unbiased qualitative interpretation of TEM indentation videos. Given that the background subtraction algorithms used in the macroscale are reasonably mature [38,39], evaluating them as tools for analyzing TEM scale videos is well motivated.

The image processing algorithm developed in this work, hereafter referred to as "MicroViBe", uses as its first step the widely accepted ViBe [40] algorithm for foreground detection, due to its reliability and computational performance [41]. The MicroViBe algorithm will generate a "heatmap" of the indentation region, which will identify areas having the highest degree of foreground motion. This heatmap, in combination with post-mortem TEM images, will subsequently be used to qualitatively assess the relative strengths of microstructural features.

2. Methods

This work follows four major tasks, and each is discussed in the following sub-sections. First, we irradiate a model Fe-9%Cr ODS alloy using Fe^{2+} ions at 500 °C. Second, we conduct the *in situ* TEM cube-corner indentation on electron-transparent lamellae taken from within only the shallow damage layer. Third, we apply the MicroViBe motion detection algorithm to the TEM resolution video acquired from the indentation test. Finally, the indented lamellae are imaged by scanning TEM (STEM) along the ideal zone axes for dislocation loop imaging, and we correlate STEM results with hotspots from the MicroViBe algorithm. Automatic TEM phase-orientation mapping (NanoMEGAS ASTAR) is also conducted on the indented lamellae to determine the grain orientations in the original indentation video as compared to the on-zone axis STEM images.

2.1. Material and irradiation

A tempered martensite Fe-9%Cr ODS alloy is selected for this study because of its complex, but well-characterized microstructure [42]. A rod of the alloy (composition in Table 1) is obtained from Lot M16 from the Japan Nuclear Cycle Development Institute (now the Japan Atomic Energy Agency). The rod was processed by mechanically alloying ferritic steel with Y_2O_3 powders, then hot extruding at 1150 °C; its final heat treatment involved 1 h, 1050 °C anneal with air cooling, then an 800 °C tempering with air cooling. Further detail regarding the mechanical alloying and fabrication are available in Ref. [43].

A specimen from the rod is prepared for bulk ion irradiation by electrical discharge machining into 1.5 mm $\times$ 1.5 mm $\times$ 16 mm bars. Each bar is mechanically polished through 4000 grit SiC paper, followed by electropolishing for 20 s in a 10% perchloric acid +90% methanol solution maintained between −30 °C and −40 °C, with a 35 V applied potential between the specimen (anode) and platinum mesh cathode. The specimen is subsequently irradiated with 5.0 MeV Fe^{2+} ions to a dose of 3 displacements per atom (dpa) at 500 °C using a 1.7 MV General Ionex Tandetron accelerator at the Michigan Ion Beam Laboratory. The beam is rastered at 255 Hz. A combination of resistance heating and air cooling are used to maintain the irradiation temperature at 500 ± 10 °C at high vacuum pressures below 1.3×10^{-5} Pa (10^{-7} torr). Beam current is recorded throughout the duration of the experiment to ensure accurate dose accumulation. The irradiation dose rate is ~10^{-4} dpa/s.

The displacement damage profile for 5.0 MeV Fe^{2+} ions normally incident on Fe-9%Cr is calculated using the Stopping and Range of Ions in Matter (SRIM) 2013 program in "Quick Calculation" (Kinchin-Pease) mode [44] and displacements are obtained from the vacancy.txt file. The displacement energy of the Fe and Cr species used was 40 eV as specified by ASTM E 521-96 [45]. The damage profile (Fig. 1) exhibits a steep gradient between the surface and the damage peak, which is located approximately 1.2 μm from the surface. It is necessary to avoid both the surface (where there is a depletion of defects) and the peak damage (where the ions come to rest, changing the chemical composition and interstitial-to-vacancy ratio of the material) [37]. As such, the samples are milled from a region 300–700 nm deep.

2.2. In situ TEM lamella indentation

Indentation lamellae are prepared by focused ion beam (FIB) milling at the Center for Advanced Energy Studies (CAES) using a