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In-situ dislocation imaging during deformation in high entropy alloys

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Mechanical testing performed inside an in-situ transmission electron microscopy (TEM) has attracted considerable interests recently due to its advantage to observe defect behaviors directly and to measure mechanical response [1, 2]. Among all different experiments, the most successful ones were done using nanopillars. However, the imaging of defects is challenging and largely affected by the imaging conditions and sample geometry of nanopillars. Here, we explore the best imaging conditions of dislocations in in-situ TEM compression testing of high entropy alloys (HEAs). This material is selected for study due to its excellent thermomechanical properties, including high strength and hardness, high temperature stability and high fracture resistance at cryogenic temperatures. However, its deformation mechanism still remains unclear. By directly imaging dislocation dynamics during deformation, the deformation mechanism of HEAs can be revealed.

Single crystalline $Al_{0.1}CoCrFeNi$ HEA nanopillars were studied by in-situ compression testing. To prepare single crystal nanopillar, we first used electron back-scattered diffraction (EBSD) (JEOL 7000F Analytical SEM, JEOL) to identify the orientation of grains. The EBSD result is shown in Fig. 1(a). After EBSD characterization, three grains A, B, and C were identified. The grain C was selected for the fabrication of pillars with focused-ion-beam (FIB) technique (Helios Nanolab 600i, FEI). Figure 1(b) and (c) show a representative TEM bright field image of a 512nm HEA pillar and its diffraction pattern. In-situ TEM compression testing was performed using a Hysitron PI95 picoindenter TEM holder (Hysitron) equipped with a 2 μm cube flat punch indenter in a JEOL 2010 LaB6 TEM operating at 200 KeV. The compression testings were all conducted in the displacement-control mode with a strain rate of 1×10^{-3} /s. The videos were captured with a charge-coupled device (CCD) camera (Gatan Orius, Gatan) running at 10 frames per second. Among all the pillars tested, pillars with diameter ~500 nm give the best stability during compression.

Figure 2 shows an in-situ TEM compression testing performed on a 512 nm HEA pillar. The still images extracted from videorecording in Fig. 2(b-c) display clear and sharp dislocation contrast (marked by the arrows in Fig. 2(b-c)). Threading dislocations followed each other and propagated toward the bottom of the pillar from point (b) to (c). The compression axis of the HEA pillar is (5.7,4,7). The slip system with the highest Schmid factor is (1-11)[011] with a Schmid factor of 0.4. Thus, the Burgers vector of the observed dislocations is ±1/2[011]. This identification is further verified by the ex-situ experiment. A TEM lamella was prepared by low energy Ga^+ milling from a deformed HEA pillar with diameter of 705 nm (see Fig. 3). To characterize the nature of the dislocations, post-mortem $\mathbf{g} \cdot \mathbf{b}$ analysis of the lamella sample was performed by orientating the sample under different two beam conditions. By using (0-22) and (-3-11) reflections to form dark field imaging (Fig. 3(b-c)), the dislocation contrast disappeared. This indicates that the observed dislocations are pure dislocations with Burgers vector ±1/2(011), which agrees with the identification based on the slip system observed in-situ.

In conclusion, in-situ TEM compression testing was carried out to study the deformation mechanism of $Al_{0.1}CoCrFeNi$ HEAs. Sharp dislocation contrast was achieved with pillars of diameter ~500 nm. The nature of the observed dislocation is determined by Schmid's law in in-situ experiments and further verified by ex-situ characterization.

References:
[1] J Kacher *et al*, MEMS and Nanotechnology **5**(2016), p. 9.
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[3] The work is supported by DOE BES (Grant No. DEFG02-01ER45923).

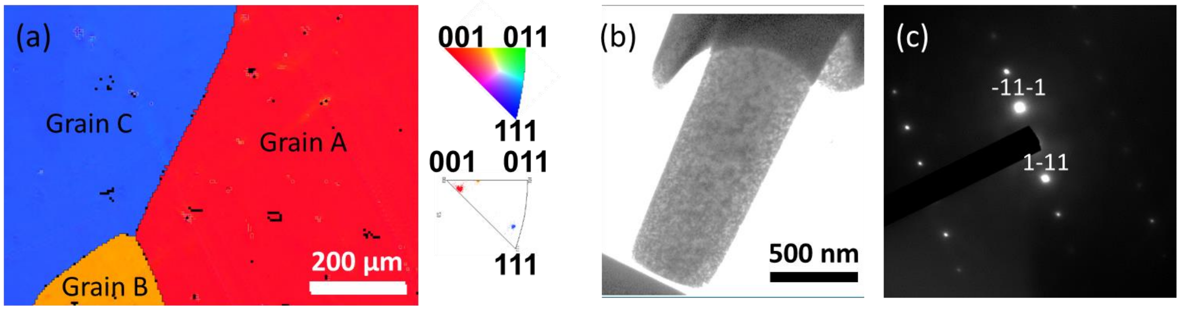

Figure 1. (a) EBSD results of Al0.1CoCrFeNi HEA. (b) and (c) show the TEM bright field image of a 512 nm HEA pillar and its diffraction pattern.

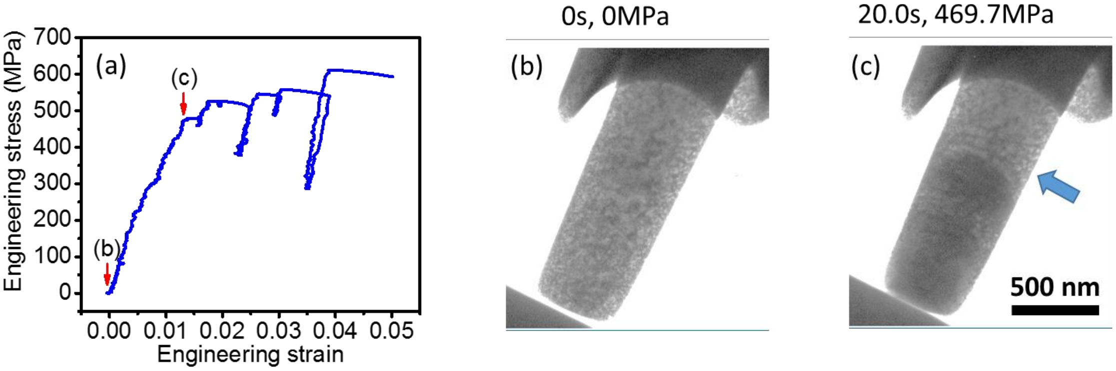

Figure 2. (a) the measured stress-strain curve with still images (b) and (c) extracted from real-time video recording at each marked position in stress-strain curve.

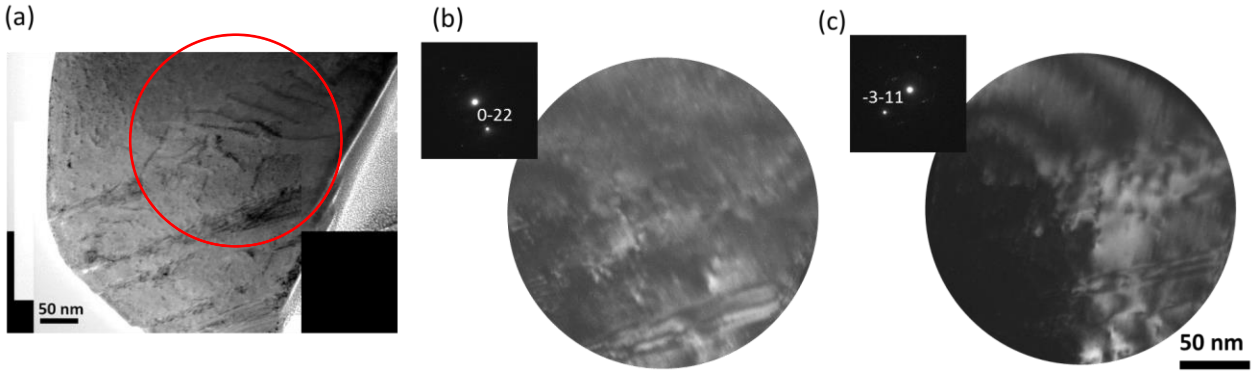

Figure 3. (a) A montage TEM bright field image of a deformed 705 nm diameter HEA pillar after low energy Ga^+ ion milling at 5 KeV. (b) and (c) show the $g \cdot b$ analysis results. Dislocation contrast disappears when using (0-22) and (-3-11) reflections to form the dark field imaging. The dark field imaging region is marked by the red circle in (a).