Through-Focal HAADF-STEM Analysis of Dislocation Cores in a High-Entropy Alloy

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High-entropy alloys (HEAs) are a new class of multi-component alloys that exhibit surprising characteristics, [1] including very large strain hardening rates, large fracture toughness at room temperature [2], and a strong temperature dependence of yield strength at or below room temperature. These properties are closely linked to nano-twinning and dislocation-mediated plasticity, yet little experimental work has explored dislocation dissociation, stacking fault energy, or core structures in these alloys [3]. In this study, an HEA, containing 5 elements (Cr, Co, Mn, Fe, and Ni) with equiatomic composition was deformed to a 5% plastic strain at room temperature [4]. Post-mortem 3mm disks were electro-polished using a solution consisting of 21% Perchloric acid and 79% Acetic acid and analyzed using a probe-corrected Titan³ 80-300kV along a [110] zone axis. Highly planar deformation was first observed by Otto et al. [5] and was active for this study as well. This planar deformation, involving dislocation arrays on \{111\} slip systems, may imply the existence of short-range order, low stacking fault energy (SFE), and/or supplementary displacements in the wake of dislocations.

Smith et al. [6] previously demonstrated that high and medium angle annular dark field scanning transmission electron microscopy (HAADF/MAADF-STEM) could effectively be used to determine the misalignment of a dislocation through foil thickness. This misalignment created a contrast “plume” when imaged in a MAADF condition. Recently, Smith et al. revealed the presence of a broad distribution of stacking fault widths, suggesting the concept of a “local” stacking fault energy in HEAs which affect the the dislocation dissociation and may play a role in how these dislocations glide [7]. To further explore this misalignment and how it relates to the dislocation core structure, through-focal HAADF-STEM imaging was employed. Acquisition of a through-focal STEM series was shown to enable detection of the crystal rotation in association with the “Eshelby twist” around screw dislocations [8]. This technique has been employed presently to create a 3D analysis of dislocation cores in the Cantor alloy as shown in Figure 1(a) and 1(b). Changing defocus allowed different depths along a dislocation line to be imaged, allowing for a three dimensional analysis of the whole dislocation core. The field of focus (z) was calculated using [9]:

\[
\text{\(z = \frac{\lambda}{\alpha^2}\)}
\]

where \(\lambda\) was the electron wavelength (which at 300kV was .00224nm) and \(\alpha\) was the convergence angle (22mrad). Therefore, the depth of field for this study was 4.6nm. A nano-hole was drilled through the sample near the dislocation to act as a marker and reveal changes in the dislocation’s location and dissociation distance. Two different dislocation types were analyzed using this technique. One with a short contrast “plume” attached to it and another with a much longer one – the latter shown in Figure 2. A gray box with a red outline is placed over the dislocations stacking fault and represents the dislocation’s location and dissociation width. A series of different defocal images were taken for both types of dislocations and aligned using ImageJ [10]. For both dislocations, the dissociation distance changed along the dislocation line; however, the dislocation with a long plume showed a much larger variation in stacking fault width as shown in Figure 2(b), 2(c), and 2(d). These findings demonstrate the
The unique capability of through-focal HAADF imaging for probing dislocation structure information in 3D at atomic-scale. These results will be discussed in the context of the concept of a “local” SFE in this HEA, and in relationship to the unique macro-behavior exhibited by these alloys.

References:
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Figure 1: A schematic of the through-focal STEM technique at (a) 0nm defocus and (b) -15nm defocus. (c) A MAADF-STEM image showing an example of the setup for the through-focal STEM.

Figure 2: (a) A dislocation with a long (20nm) contrast “plume” attached to it that was imaged edge-on at different defocus values (b) 10nm defocus (c) -2nm defocus and (d) -10nm defocus.