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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 Titan[3] 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]:

$$z = \frac{\lambda}{\alpha^2} \tag{1}$$

where λ was the electron wavelength (which at 300kV was .00224nm) and α 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 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:
[1] J.-W. Yeh et al. Adv. Eng. Mater. **6** (2004) 299–303.
[2] B. Gludovatz et al. Science. **345** (2014) 1153–1158.
[3] Z. Zhang et al. Nat. Commun. **6** (2015) 10143.
[4] B. Cantor et al. Mater. Sci. Eng. A. 375-377 (2004) 213–218.
[5] F. Otto et al. Acta Mater. **61** (2013) 5743–5755.
[6] T.M. Smith et al. Microsc. Microanal. **21** (2015) 2205–2206.
[7] T.M. Smith et al. Acta Mater. Pending Submission (2016).
[8] J.E. Allen et al. Nat. Nanotechnol. **3** (2008) 168–173.
[9] P.D. Nellist et al. Microsc. Microanal. **14** (2008) 82–88.
[10] C.A. Schneider et al. Nat. Methods. **9** (2012) 671–675.
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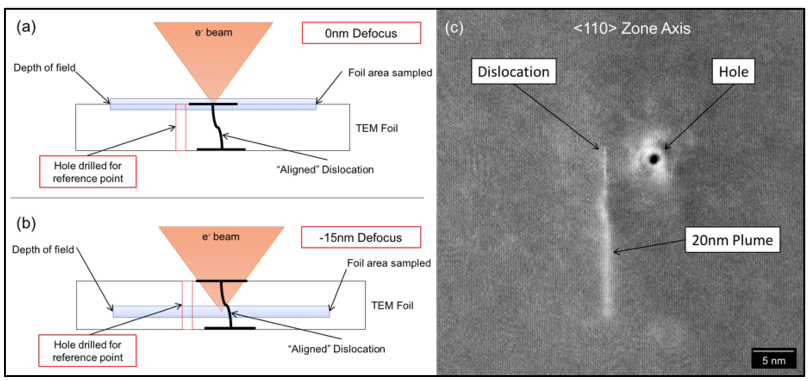

Figure 1: A schematic of the through-focal STEM technique at (a) 0nm defocus and (b) -15nm defocus. (c) A MAADF-STEM image showing an example of the setup for the through-focal STEM.

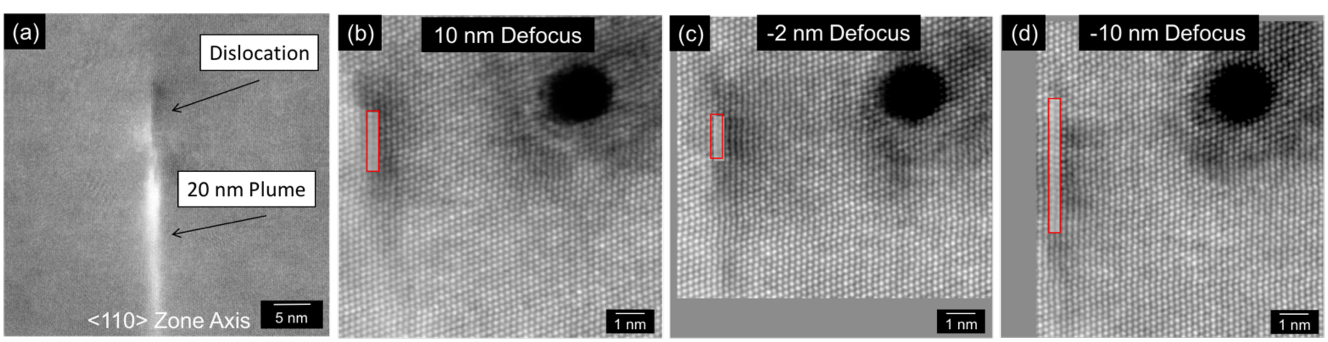

Figure 2: (a) A dislocation with a long (20nm) contrast “plume” attached to it that was imaged edge-on at different defocus values (b) 10nm defocus (c) -2nm defocus and (d) -10nm defocus.

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