Deformation Defects Characterization in Short-Range Ordered CrCoNi using Fast Electron Detectors and 4D-STEM

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CrCoNi, a Medium-Entropy Alloy (MEA) with a single-phase face-centered-cubic (FCC) structure, has been extensively studied by researchers since it has shown great advantages of high strength and great ductility [1], as well as the short-range ordering (SRO) effects [2, 3]. The exceptional mechanical properties of CrCoNi have been attributed to novel deformation mechanisms such as the formation of stacking faults (SFs), nanotwins (NTs), shear bands, etc [4]. Crystalline defects in general are imaged by electron microscopy. However, short-range order effects on crystal deformation have not been fully understood.

Characterization of deformation defects is traditionally carried out using diffraction contrast imaging performed inside a TEM. More recently, weak-beam dark-field scanning transmission electron microscopy (WB-DF STEM) has been used to characterize the dissociated dislocations in CrCoNi and identify the distance between two parallel SFs. WB-DF STEM allows Shockley partial dislocations to be directly distinguished and thus a measurement of the stacking fault width between the two partials. However, both WB-DF imaging and the identification and counting of these SFs are time consuming processes, which is a major bottleneck for deformation analysis. The other challenge is that such analysis in generally limited to isolated dislocations and regions heavily deformed are difficult to analyze.

Here, we introduce a novel defect imaging method based on the cepstral analysis of electron diffuse scattering using an Electron Microscope Pixel Array Detector (EMPAD) detector. This direct electron detector has a high dynamic range and fast readout, providing large diffraction datasets for high spatial resolution characterization. By comparing regional difference cepstrums (dCp) [10] with real-space images from STEM, the local crystal defects of SFs and NTs are imaged and analyzed in deformed CrCoNi samples. By far, we have successfully obtained a large dataset of lattice defects and their corresponding diffraction and cepstral patterns. Correlation between direct electron images and cepstral patterns enable a separation between single SF and NTs. By summarizing the local variations of stacking fault width in different CrCoNi samples before and after additional thermal annealing, we could show the distribution of stacking fault energy(SFE), and the impact of SRO. The presence of SRO is further evidenced by atomic resolution STEM imaging. These inspiring results open the possibility of the auto characterization of the presence and the category of SRO and their interactions with dislocations, leading to the formation of planar defects. This talk will summarize our progress so far on the novel deformation mechanisms in CrCoNi [11].

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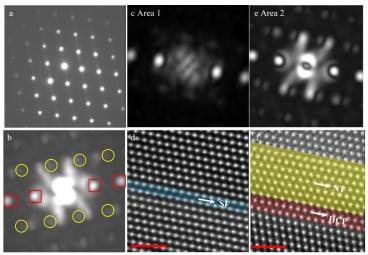


Figure 1. Cepstral transform of electron nano-diffraction pattern. (a) Experimental averaged diffraction pattern from a deformed CrCoNi sample along [110] zone axis, (b) Cepstral pattern from (a), (c) and (e) Differential Cepstral patterns from SF and NT in CrCoNi and comparison with corresponding atomic resolution HAADF-STEM images (d) and (f). The scale bar is 1 nm.

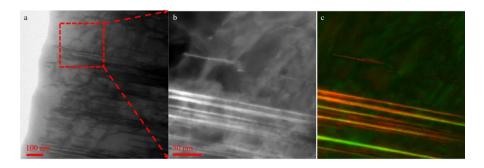


Figure 2. (a) Bright-field image for deformed CrCoNi sample along [110] zone axis, (b) Virtual dark-field image formed by the EMPAD data collected from an area in (a), (c) Color-code image from (b) to distinguish SF (red) and NT (green).

References:

- [1] Gludovatz, B. *et al.* Exceptional damage-tolerance of a medium-entropy alloy CrCoNi at cryogenic temperatures. *Nat. Commun.* 7, 10602 (2016), doi: https://doi.org/10.1038/ncomms10602
- [2] Hsiao, H.-W. *et al.* Data-driven electron-diffraction approach reveals local short-range ordering in CrCoNi with ordering effects. *Nat. Commun.* **13**, 6651 (2022), doi: https://doi.org/10.1038/s41467-022-34335-0
- [3] Zhang, R. *et al.* Short-range order and its impact on the CrCoNi medium-entropy alloy. *Nature* **581**, 283–287 (2020), doi: https://doi.org/10.1038/s41586-020-2275-z
- [4] Zhang, Z. *et al.* Dislocation mechanisms and 3D twin architectures generate exceptional strength-ductility-toughness combination in CrCoNi medium-entropy alloy. *Nat. Commun.* **8**, 14390 (2017), doi: https://doi.org/10.1038/ncomms14390
- [5] Liu, D. *et al.* Exceptional fracture toughness of CrCoNi-based medium- and high-entropy alloys at 20 kelvin. *Science* **378**, 978–983 (2022), doi: 10.1126/science.abp8070
- [6] HIRSCH, P., A, H., RB, N., DW, P. & MJ, W. ELECTRON MICROSCOPY OF THIN CRYSTALS. (1965).

- [7] Miao, J. et al. Dislocation Characterization using Weak Beam Dark Field STEM Imaging. *Microsc. Microanal.* **24**, 2202–2203 (2018), doi:10.1017/S1431927618011492
- [8] Shih, M., Miao, J., Mills, M. & Ghazisaeidi, M. Stacking fault energy in concentrated alloys. *Nat. Commun.* **12**, 3590 (2021), doi: https://doi.org/10.1038/s41467-021-23860-z
- [9] Williams, R. E. A. Microstructural and Defect Characterization of Al-Si Alloy Using PFIB and EMPAD. *Microsc. Microanal.* **25**, 920–921 (2019), doi: 10.1017/S1431927619005336
- [10] Shao, Y.-T. *et al.* Cepstral scanning transmission electron microscopy imaging of severe lattice distortions. *Ultramicroscopy* 231, 113252 (2021), doi: https://doi.org/10.1016/j.ultramic.2021.113252
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