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Towards Optimized Characterization of Dislocation Loops in Irradiated FCC Alloys Using On-Zone STEM Techniques

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Face-centered-cubic (FCC) based materials have long been used for core materials in existing light-water reactors, and are proposed to be used in various next-Gen nuclear reactor systems. The potential extensive adoption of FCC-based materials, such as austenitic stainless steels, nickel-based superalloys and high-entropy alloys, is due to their acceptable radiation resistance, high-temperature corrosion resistance, ductility and creep resistance. The analysis of dislocation loops in these FCC materials under irradiation – of ½⟨110⟩{110} perfect and ⅓⟨111⟩{111} faulted types – is of particular interest because dislocation loops are known to contribute to the mechanical property degradation through hardening and embrittlement. Yet, there has not been a way to effectively characterize both types of dislocation loops of all variants in irradiated FCC materials using transmission electron microscopy (TEM) based techniques. The traditional Rel-Rod conventional TEM (CTEM) dark-field imaging has been extensively used for imaging two variants of faulted loops (out of four in total) that appear edge-on at [011] zone axis. However, this technique fails to image the other two faulted loop variants, nor any of the six perfect loop variants. All these overlooked features contribute significantly to irradiation hardening. Another traditionally used method, called two-beam condition imaging in both CTEM and scanning TEM (STEM) modes requires extremely careful experimental design and implementation. Series of sample tilting experiments are time-consuming, and the ambiguity between dislocation loops being visible or invisible usually causes confusion and inaccuracy for Burgers vector analysis.

In this study [1], using an irradiated model $Ni_{40}Fe_{40}Cr_{20}$ alloy, we developed an on-zone [001] STEM bright-field (STEM-BF) based methodology to efficiently and effectively characterize all variants of perfect and faulted dislocation loops in FCC-based material systems. As shown in Figure 1, all six perfect loop variants are either edge-on or inclined in the specimen. On the other hand, all four variants of faulted loops are inclined and exhibit interior shadow contrast, so that they are easily distinguished from perfect loops. Several key advantages are taken collectively to achieve the results: (i) the development of predicted dislocation loop morphology based on their habit planes and projections, (ii) strong diffraction contrast obtained at on-zone STEM imaging conditions, (iii) the complete removal of FIB-induced surface damages using flash-polishing [2–5], and (iv) the removal of bend contours or thickness fringes [6]. With a single snapshot at this imaging condition, dislocation loop structures are captured with high visual clarity, with Burgers vector analysis extremely easy. Multiple factors affecting this methodology together with its comparison with traditional imaging methods are discussed in detail [1].

The on-zone STEM-BF based technique has been proved to work well in Ni-based FCC alloys [1,7,8], Fe-based BCC alloys [9,10], and ceramic nuclear fuel materials [11] in recent years. This developed methodology is expected to greatly contribute to the current and future high-throughput characterization efforts, thus should be extensively adopted by the nuclear material research field [12].

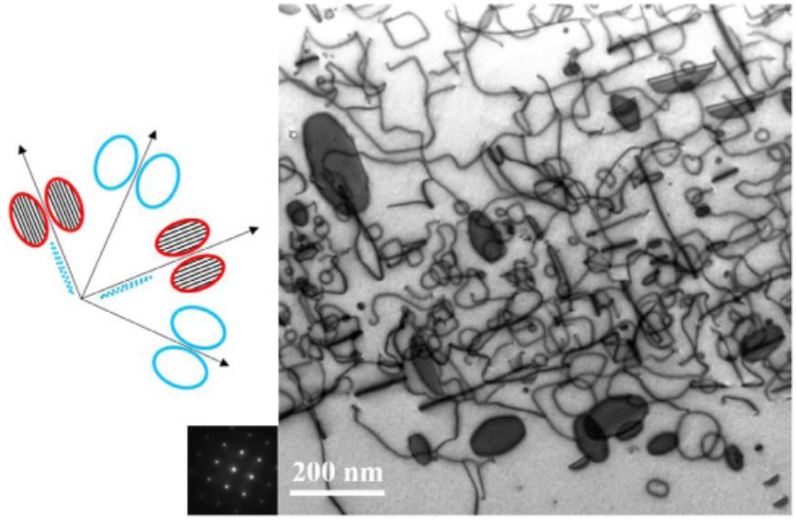

Figure 1. An example of the effective flash-polishing Cross-sectional on-zone [001] STEM-BF image of an irradiated model $Ni_{40}Fe_{40}Cr_{20}$ alloy with correctly oriented simulated morphology map and experimental diffraction pattern. Note there are in total six and four variants of perfect and faulted dislocation loops highlighted in blue and red, respectively, in the morphology map on the left. Adopted from [1].

References:

[1] P. Xiu et al., J. Nucl. Mater. **544** (2021) 152658. https://doi.org/10.1016/j.jnucmat.2020.152658.
[2] C. Lu et al., Acta Mater. **127** (2017). https://doi.org/10.1016/j.actamat.2017.01.019.
[3] Y. Zhang et al., Acta Mater. **181** (2019) 519–529. https://doi.org/10.1016/j.actamat.2019.10.013.
[4] T. Yang et al., Acta Mater. **151** (2018) 159–168. https://doi.org/10.1016/j.actamat.2018.03.054.
[5] C. Lu et al., Nat. Commun. **7** (2016) 13564. https://doi.org/10.1038/ncomms13564.
[6] Y. Zhu, C. Ophus, M.B. Toloczko, D.J. Edwards, Ultramicroscopy. **193** (2018) 12–23. https://doi.org/10.1016/j.ultramic.2018.06.001.
[7] Y. Zhang et al., Acta Mater. **181** (2019) 519–529. https://doi.org/10.1016/j.actamat.2019.10.013.
[8] P. Xiu et al., J. Nucl. Mater. **538** (2020) 152247. https://doi.org/10.1016/j.jnucmat.2020.152247.
[9] C.M. Parish, K.G. Field, A.G. Certain, J.P. Wharry, J. Mater. Res. **30** (2015) 1275–1289. https://doi.org/10.1557/jmr.2015.32.
[10] B. Yao, D.J.J. Edwards, R.J.J. Kurtz, J. Nucl. Mater. **434** (2013) 402–410. https://doi.org/10.1016/j.jnucmat.2012.12.002.
[11] P. Xiu et al., J. Nucl. Mater. **557** (2021) 153244. https://doi.org/10.1016/j.jnucmat.2021.153244.

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