Application of STEM Diffraction Contrast Imaging and Chemical Analysis for a Comprehensive Characterization of Nuclear Reactor Structural Alloys

Yuanyuan Zhu, Mychailo B. Toloczko and Danny J. Edwards,

Energy and Environmental Directorate, Nuclear Sciences Division, Pacific Northwest National Laboratory, Richland, WA 99352, USA

Neutron-induced changes in the microstructure of nuclear reactor structural materials play a pivotal role in determining the structural integrity, mechanical properties and lifetime of reactor components. Neutron irradiation, or any high-energy ion irradiation, can displace atoms, leading to cascades of defects. Over time these defects can evolve into larger features such as dislocation lines and loops, voids and bubbles that all affect bulk mechanical properties. To understand how these irradiation-induced defects govern the changes in materials behavior, it is essential to characterize these defects individually as well as collectively to understand how they form and evolve during irradiation.

Ever since the first direct observation of dislocations in conventional transmission electron microscopes (CTEM), it has matured into the characterization tool of choice for dislocations and many other microstructural features. However, recently Phillips et al [1] provided evidence that diffraction contrast imaging in STEM mode (DCI STEM) not only preserves the CTEM rules for $\mathbf{g} \cdot \mathbf{b}$ invisibility criteria, but can alleviate bend contours and allow observations in thicker region. Yet it is not clear how DCI STEM suppresses the bend contour nor how different STEM parameters affect the contrast of dislocation images. To address the bend contour question, we investigated the image formation process in the STEM mode and its reciprocal relationship with the CTEM in this work [2]. As shown in Figure 1, the bend contours in the bright field (BF) STEM image were effectively negated when the STEM collection semi-angle was increased to 3 mrad. By breaking the reciprocity, the DCI BF-STEM detector includes multiple deficiency lines, and averages out the intensity changes and led to an almost constant background contrast while preserving the strain contrast around dislocations and grain boundaries.

Using a HT-9 specimen as an example, the optimized DCI STEM image parameters were chosen to include a small convergence semi-angle that avoids STEM discs overlapping, a camera length that offers a BF-STEM collection semi-angle similar to the size of the direct transmitted disc and an annular dark field outer angle that excludes the direct disc while capturing diffraction discs up to $3\mathbf{g}$. For a dense population of defects, the orientation of the specimen is preferably a two-beam condition with small extinction distance. With optimized DCI STEM imaging parameters, the advantages of the various analytical techniques in STEM mode such as electron energy loss spectroscopy (EELS) and energy-dispersive X-ray spectroscopy (EDX), can lead to a comprehensive characterization of the defects and chemical inhomogeneity with a high special resolution. A workflow of such an attempt is schematically illustrated in Figure 2. This streamlined characterization can facilitate the comparison among nuclear alloys with different irradiation history and conditions.

References:
[3] The authors acknowledge funding from the U.S. Department of Energy NEET-3, reactor materials
Figure 1. DCI STEM dislocation imaging is advantageous over the CTEM at suppressing bend contour by breaking the rule of reciprocity.

Figure 2. The workflow of a comprehensive defect characterization in STEM mode for a BCC Ferric nuclear reactor structural alloy.