Understanding Formation of Irradiation-Induced Defects through 4D-STEM, Electron Tomography, and WBDF-STEM

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A major challenge in advancing nuclear materials for next-generation fission and proposed fusion reactors is to comprehensively understand the formation of irradiation-induced defects [1]. It is essential to correlate the evolution of irradiation-induced defects and the degradation of mechanical properties, as they collectively dictate the material's lifespan and ensure nuclear safety [2]. Scanning transmission electron microscopy (STEM) based techniques have emerged as indispensable tools for irradiation-induced defect characterization [3-4], offering high spatial resolution imaging and chemical analysis, such as electron energy loss spectroscopy (EELS) and energy dispersive X-ray spectroscopy (EDXS). These techniques have been effectively used to obtain an atomic-scale view of the defect structure [5]. Recent advances in electron microscopy, particularly in 4D-STEM [6], offer detailed insight into microstructural evolution by capturing full 2D diffraction patterns at every pixel position. Using high-speed direct electron detectors, this technology generates a four-dimensional dataset, overcoming the limitations of traditional STEM imaging.

In this presentation, we discuss the potential for combining 4D-STEM, weak-beam dark-field (WBDF) STEM, electron tomography, and EDXS for defect analysis, specifically focusing on irradiation-induced dislocation loops in proton-irradiated Fe-5Cr model alloys. This approach offers the advantage of obtaining the 3D distribution of dislocation loops in atomic scale, as well as identifying their type ($\langle 100 \rangle$ or $\frac{1}{2}\langle 111 \rangle$) and nature (interstitial or vacancy). Through 4D-STEM strain mapping and STEM-EDX elemental mapping, we can assess the local strain field and strain interactions between loops, while also detecting chemical composition changes near the loops within the same area of interest. 4D-STEM and atomic-resolution STEM revealed that when loops are small (diameter <5 nm), their centers may not precisely overlap with the "black-dot" features (Fig. 1). In the case of an $\langle 100 \rangle$ edge-on loop (Fig. 1b), its center may lie between a pair of black-dots, which represent high-strain areas at the two ends of the loop. It was observed that $\langle 100 \rangle$ loop strings are composed of ½$\langle 111 \rangle$ and $\langle 100 \rangle$ loops arrayed along $\langle 100 \rangle$ directions (Fig. 2). STEM-EDX and analysis revealed Cr enrichment associated with the dislocation loops (Fig. 2f), supporting prior observations that Cr impedes dislocation loop motion, resulting in a more sluggish dislocation loop evolution process in Fe-Cr alloys than in pure Fe [7]. STEM and EDX tomography of the loops and Cr-enriched features will also be presented [8]. The experimental results from these advanced techniques validate simulation models [9], enhancing the understanding of irradiation effects on material properties, crucial for materials development and selection in nuclear applications or other extreme environments [10-11].

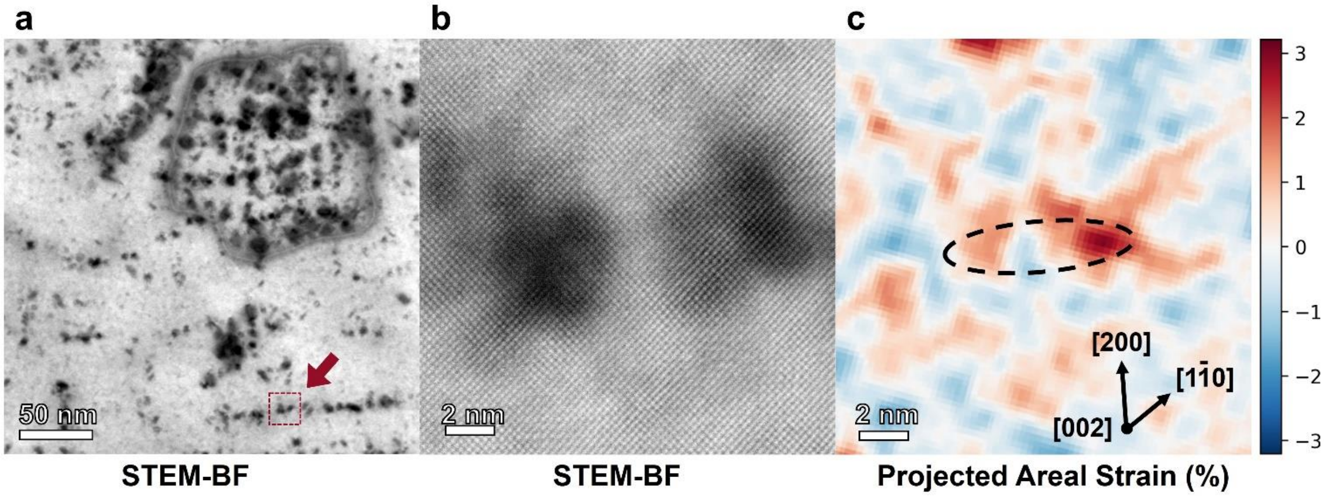

Fig. 1. Dislocation loop strings observed in Fe-5Cr irradiated with protons to ~5 dpa at 250°C: (a) STEM bright-field (BF) image taken along the [002] zone axis; (b) High-resolution STEM-BF image of the red-square-marked region from Fig. 1a; (c) Areal strain perpendicular to the beam direction quantified using the 4D-STEM-EWPC method.

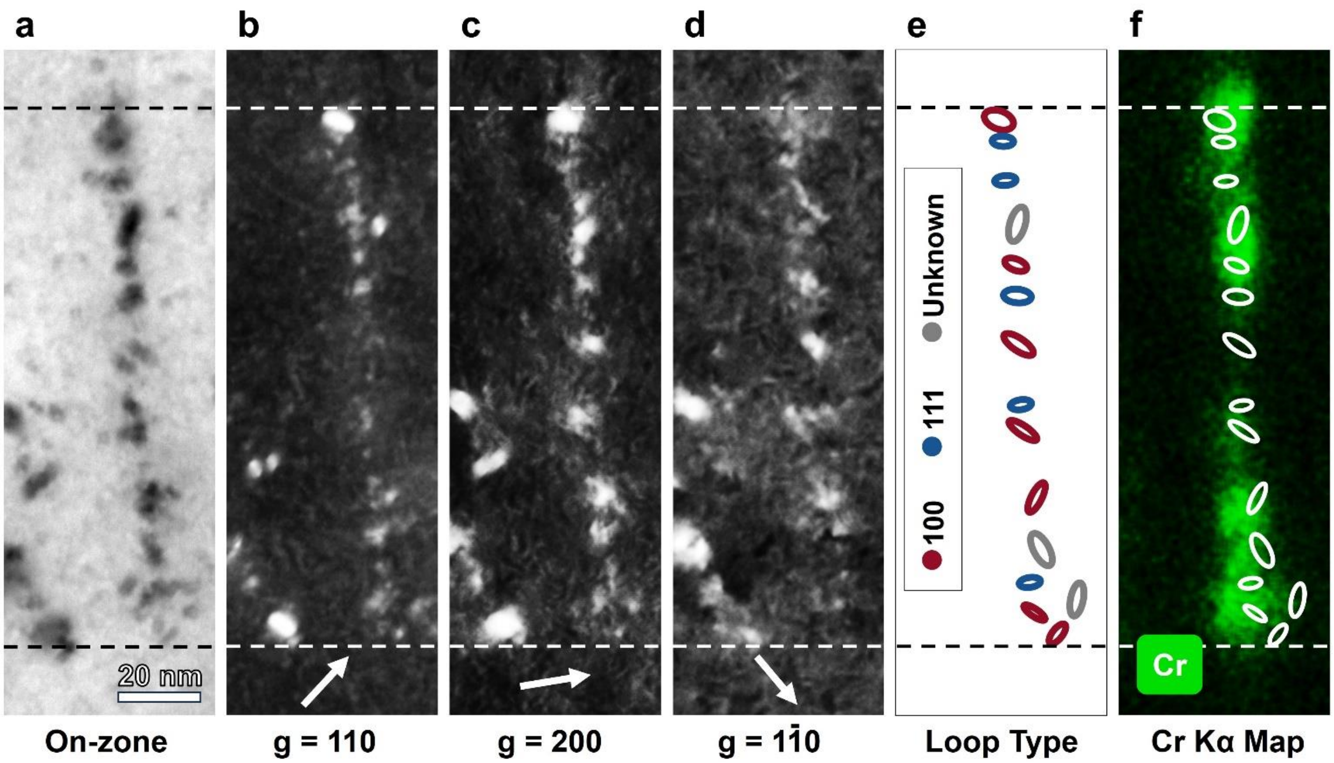

Fig. 2. WBDF-STEM and STEM-EDX analysis: (a) STEM-BF image along the [002] zone axis, (b-d) WBDF-STEM images along various g-vectors near the [002] zone axis, (e) Identification of dislocation loop types through comprehensive g-dot-b analysis, (f) elemental map for Cr Kα (green) extracted from the STEM-EDX spectrum image dataset.

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