# TEM Characterization of Radiation-Induced Segregation at Irradiation-Induced Dislocation loops in Al0.3CoCrFeNi and CoCrFeMnNi High Entropy Alloys

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# TEM Characterization of Radiation-Induced Segregation at Irradiation-Induced Dislocation loops in $Al_{0.3}CoCrFeNi$ and CoCrFeMnNi High Entropy Alloys

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Alloys under irradiation at elevated temperatures (0.3-0.5 $T_m$) undergo radiation-induced segregation (RIS) at defect sinks such as grain boundaries and dislocation loops [1]. RIS modifies local composition at these locations and can lead to detrimental consequences. In addition, when the local solution concentration is driven above the solubility limit, precipitation can occur, and the material properties can, as a result, change significantly. For example, in irradiated austenitic stainless steels, RIS causes Cr depletion at grain boundaries that can lead to irradiation-assisted stress-corrosion cracking [2-3].

It is well established that oversized solutes are depleted and undersized solutes are enriched at defect sinks due to the preferential exchange of oversized elements with vacancy flux (Inverse-Kirkendall Effect) and the preferential association of undersized elements with interstitial flux (interstitial binding) [1]. RIS at irradiation-induced dislocation loops is traditionally studied with analytical transmission/scanning electron microscopy (TEM/STEM/AEM). Analysis of defects created by the irradiation are conducted with the region of interest and/or dislocation loops tilted so that they are edge-on to the optical axis and x-ray electron dispersive spectroscopy (XEDS) line scans are performed across the loops. This approach provides one-dimensional compositional profiles vertical to the defect habit plane. In addition to line-scan on edge-on dislocation loops, hyperspectral imaging on tilted or flat-on dislocation loops can provide in-plane 2-dimentional elemental distributions to answer questions such as if the segregation occurs at dislocation lines (ring form), or on the loop plane (disc form) [4].

Recently, atom probe tomography (APT) has been successfully employed to investigate the RIS at dislocation loops in high entropy alloys and austenitic stainless steels [5-8]. APT has advantages of high mass resolution, high spatial resolution, and the ability to reconstruct elemental distribution in 3D. However, APT is insensitive to lattice defect and it cannot identify the precise location of the loop. Therefore, it is unable to reveal the subtle spatial correlation between the dislocation loops and the elemental distributions. In contrast to APT, TEM/STEM/AEM measurements can not only locate and characterize the crystallography of the lattice defect but also determine concurrently the local elemental distribution. In the past, elemental mapping of loops not in edge-on orientation has been challenging due to the low count rate of characteristic x-ray and the insufficient signal-to-noise ratio. The enhanced capabilities of today's new instruments provide unprecedented XEDS proficiency and facilitates elemental hyperspectral imaging on inclined and flat-on irradiation-induced dislocation loops, providing precise information correlating the dislocation loop and elemental segregation for the first time.

Two single-phase high entropy alloys, $Al_{0.3}CoCrFeNi$ and CoCrFeMnNi, were prepared by arc-melting at National Tsing Hua University. The fabrication details, pre-irradiation characterization and TEM specimen preparation has been reported in a previous work [9]. The two materials were irradiated with 1 MeV krypton ions at 500°C with a flux of $1.3 \times 10^{12}$ ions/cm$^2$/s to a fluence of $6.3 \times 10^{14}$ ions/cm$^2$ (1 dpa). The in-situ TEM study of their loop evolution under irradiation has been reported in Ref. [10], and the post-irradiation APT characterization of the RIS at loops in the CoCrFeMnNi specimen can be found in Ref. [5]. High spatial resolution elemental analyses of the irradiated $Al_{0.3}CoCrFeNi$ and CoCrFeMnNi were carried out in the Argonne National Laboratory Analytical PicoProbe instrument (the prototype of the ThermoFisher Spectra UltraX) at room temperature at 300 kV in probe corrected STEM mode [11]. Before XEDS measurement, the specimens were plasma cleaned to reduce carbon contamination during the prolonged mapping [12].

XEDS measurements determined that the dislocation loops in $Al_{0.3}CoCrFeNi$ are depleted in Al and enriched in Co and in addition, the formation of Ni/Al rich nanoparticles, presumably $L1_2$ ordered phase, were also observed. On the other hand, dislocation loops in CoCrFeMnNi were depleted in Mn and enriched in Co and Ni. An example of which is presented in Figure 1 where XEDS line profiles across an inclined perfect dislocation loop and an edge-on faulted dislocation loop in CoCrFeMnNi mapped at <110> zone axis is illustrated. While both loop orientations indicate consistent segregations, the complex elemental distribution can now be revealed from the inclined loop orientation for the first time, thanks to the excellent XEDS proficiency [13].

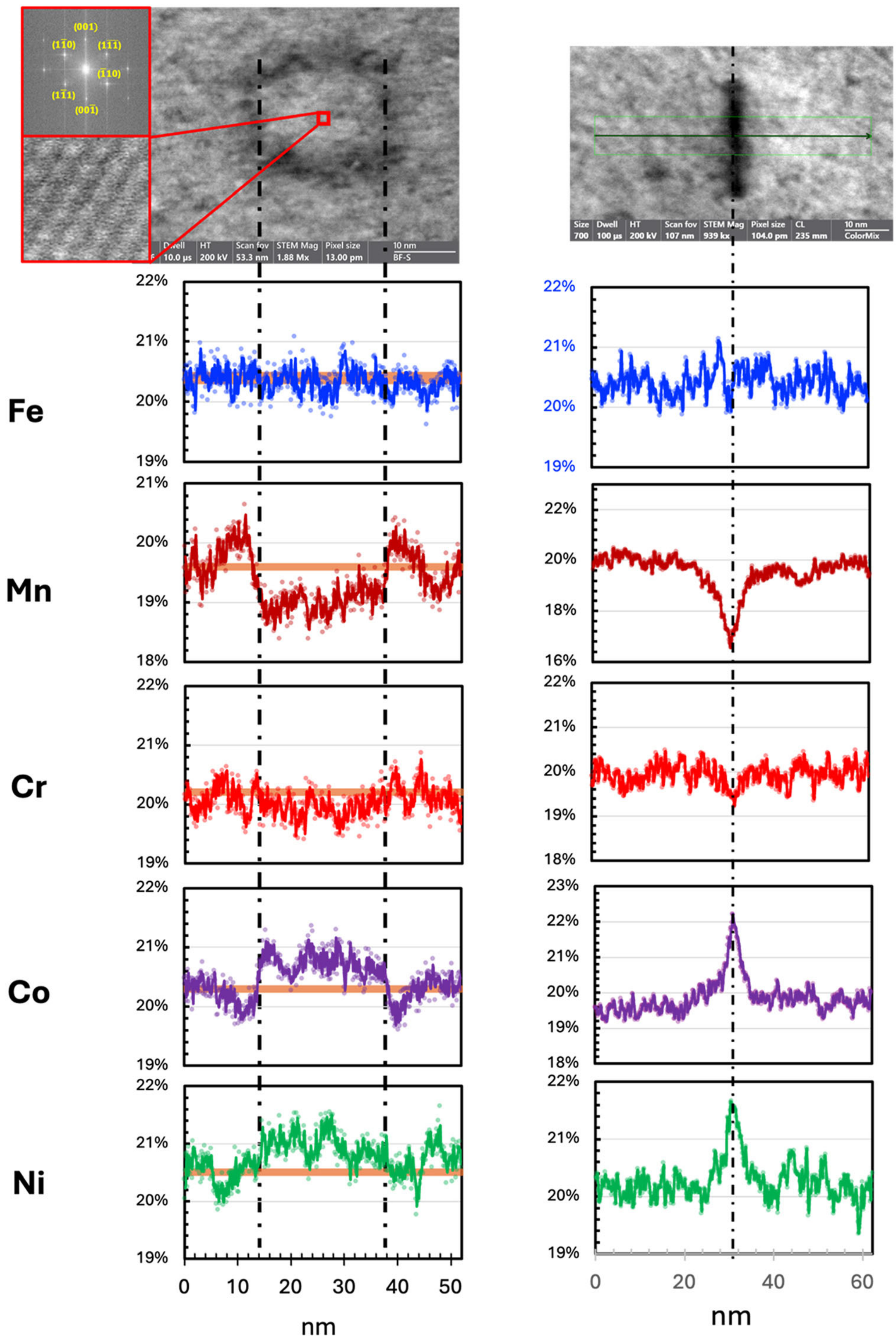


**Figure 1.** XEDS analysis showing the composition profiles across (left) an inclined perfect loop and (right) an edge-on faulted loop.



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