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Comparison of In- and Ex-Situ Analysis of Post-Irradiation Annealing

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Irradiation-assisted stress corrosion cracking (IASCC) refers to the intergranular of austenitic iron- and nickel-base components in light water reactor (LWR) cores. IASCC is a complex phenomenon and both microstructural effects (radiation hardening) and microcompositional effects (radiation-induced segregation of major alloying elements; depletion of Cr, in particular) have been identified as potentially controlling mechanisms. While there is a reasonable understanding of radiation-induced segregation and radiation-induced microstructure, their exact roles in IASCC are unclear and identifying the controlling mechanism is a critical step in solving the problem. Post-irradiation annealing provides one means of isolating microcompositional changes from microstructural changes. Recent experimental [1,2] and simulation [3] studies have shown that dislocation loops are removed preferentially during post-irradiation annealing. Simulations also predict that dislocation loops can be removed completely while the microchemical changes remain unaffected. With this type of annealing process, the role of radiation-induced segregation (RIS) and/or microstructural changes in IASCC may be isolated.

Ex-situ anneals on bulk materials are required to obtain information on bulk properties such hardening and cracking susceptibility. In-situ annealing of thin foils is more efficient for "screening" potential annealing conditions and can eliminate statistical variations by analyzing identical areas of the same sample before and after annealing. However, artifacts caused by the annealing of thin foils may limit the application of in-situ techniques. The objective of this work is to compare the post-irradiation annealing of RIS and dislocation loops performed both in-situ and ex-situ in order to assess and quantify artifacts resulting from annealing of thin foils.

An ultra-high-purity, low-carbon 304L alloy was used in this study and the bulk composition is listed in Table 1. Irradiations to 1.0 dpa were conducted with 3.2 MeV protons ($E_d = 25$ eV) at 360° ± 10°C, resulting in a nearly uniform damage rate throughout the first 35 μm of the proton range (40 μm). Further details of the sample preparation and irradiation are given elsewhere. [4] Samples were annealed at temperatures ranging from 500°C to 650°C for either 40 minutes (in-situ) or 45 minutes (ex-situ). In-situ post-irradiation annealing was performed using a JEOL 2000FX TEM with a Gatan, single-tilt heating holder. Before, during, and after annealing, the dislocation microstructure was characterized in the same areas of each specimen with the JEOL 2000FX TEM using bright field imaging with **g** = <200>, <220>, or <111>. Microchemical analysis was performed on the same grain boundaries before and after in-situ annealing using a scanning transmission electron microscope with energy-dispersive x-ray analysis (STEM/EDS). The STEM/EDS analysis was performed in a Philips CM200/FEG at Oak Ridge National Laboratory for both in-situ and ex-situ samples. Analysis of the dislocation loop population in bulk-annealed specimens was performed using the same imaging techniques on the JEOL 2000FX and/or the Philips CM200/FEG at ORNL.

The results of post-irradiation annealing for both dislocation loops and grain boundary Cr depletion are shown in Figure 1 for both in-situ and ex-situ anneals as a function of annealing temperature. Dislocation loops are removed steadily with increasing temperature for both in- and ex-situ anneals. Annealing for 40 or 45 minutes at 600°C leaves only ~40% of the loop population while annealing at 650°C removes all dislocation loops. Depletion of Cr is removed much more slowly for both in- and ex-situ annealed specimens. Only annealing at 650°C resulted in a GB Cr depletion significantly different from the as-irradiated condition. Both in- and ex-situ anneals showed that dislocation densities are removed preferentially, similar to other experimental and theoretical studies [1-3]. Further, that the in- and ex-situ results are in good agreement indicates that there are no significant artifacts associated with the annealing of thin foils. [5]

References
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TABLE 1. Bulk composition of experimental alloy determined by electron microprobe (in at%).

Alloy	Cr	Ni	Fe	Mn	Mo	Si	C	N	P	S
HP-304L	20.9	9.0	69.0	1.1	0.01	0.02	0.028	<0.004	0.002	0.003

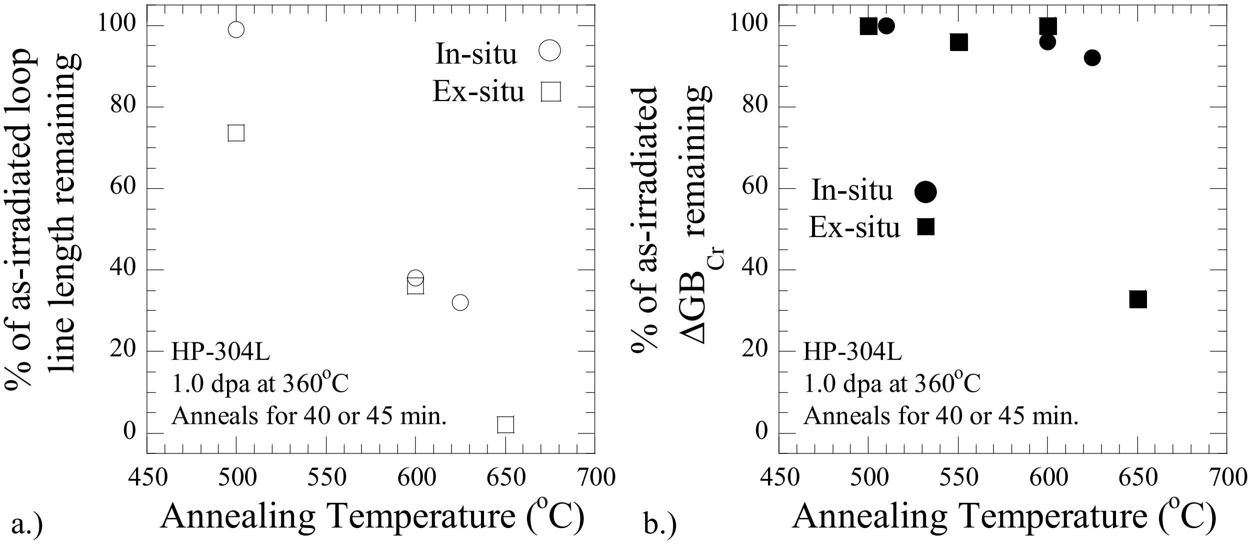

FIG. 1. Comparison of in-situ and ex-situ annealing of a.) dislocation loop line length and b.) grain boundary Cr depletion as a function of annealing temperature on HP-304L proton-irradiated to 1.0 dpa at 360°C.