Irradiation-Induced Solute Clustering in 1 Ni – 1.3 Mn Welds

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Abstract - Advanced characterization techniques including atom probe microanalysis, small angle neutron scattering (SANS), and positron annihilation lineshape analysis (PALA) have provided significant insight into the development of the microstructural features responsible for irradiation hardening and embrittlement in low alloy steel welds.

Radiation damage of low alloy steels and welds remains an important issue for the use of these materials in neutron irradiation environments. Studies of irradiation damage in low alloy steels and welds have demonstrated that this damage can be classified as a combination of matrix damage resulting from radiation-produced point defect clusters and the irradiation-enhanced formation of 1-2nm diameter clusters containing solutes such as Cu, Mn, Ni and Si. [1-4] Despite the ultrafine size of these features, they can exert a deleterious effect on materials properties. In this study, the Energy-Compensated Position-Sensitive Atom Probe (ECOPoSAP) and Local Electrode Atom Probe (LEAP) have been employed to characterize the early stages of solute clustering in low Cu (0.03wt.%) high Ni (~1wt.%) welds, which had been neutron-irradiated at a very high dose rate. The composition and irradiation doses for the welds are shown in Table 1. Additional analyses using SANS and PALA provided complementary information concerning cluster sizes and number densities, as well as data on the presence of "open volume" defects (vacancies). The purpose was to obtain mechanistic insight into the role of solute elements such as Cu, Mn, Ni and Si in irradiation damage by characterizing the irradiation-induced changes in microstructure of the welds. The atom probe data were acquired using the ECOPoSAP at Oxford University (U.K.) and a U.S. laboratory LEAP.

Reanalysis of the LEAP data showed that the density of atoms near the rim of the reconstructed volume was significantly greater than in the bulk of the material. This is a particular problem when attempting to characterize the early stages of solute clustering using a maximum separation method since the increase in atom density will result in a reduction of the mean separation between atoms and, therefore, an increased chance for detecting solute clusters. Consequently, all atoms within 2 to 3nm of the surface of the reconstructed volumes had to be excluded before further analysis of any of the LEAP data was performed. This resulted in a significant reduction in the size of the original LEAP datasets by approximately 35%, and a reduction in the number of clusters by approximately a factor of 10.

Atom probe analysis of the low Cu weld irradiated to 15 mdpa revealed the presence of a few Cu, Mn, Ni, Si enriched clusters, corresponding to a number density of ~$5 \times 10^{22} m^{-3}$. Their mean diameter was estimated to be 1.3nm and their mean composition 66Fe-1Cu-13Mn-17Ni-2Si (at.%). Analysis of the LEAP data from the 95 mdpa weld (after removal of the outer 3 nm of the analysis volume) showed that the mean diameter of the clusters was ~1.4nm with a number density of ~$2.5 \times 10^{23} m^{-3}$. The average composition of the clusters detected was 47Fe-2Cu-12Mn-26Ni-14Si (at.%).

The number densities and sizes of clusters observed by SANS are consistent with the interpretation of atom probe data. Analysis of the irradiated materials showed increasing clustering of Cu, Mn, Ni and Si with dose. In the low Cu weld, the results showed that initially the irradiation damage results in clustering of Mn, Ni and Si, but at very high doses, at very high dose rates, redistribution of Si is significantly more advanced than that for Mn or Ni.

Table 1. Weld composition (wt.%) and irradiation doses.

	Dose (mdpa)	C	Ni	Mn	Cr	Mo	Si	Cu	P	Fe
Low Cu weld	95	0.086	1.0	1.3	0.07	0.53	0.15	0.03	0.008	bal.
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