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Early Stages of Solute Clustering in Irradiated 1 Ni – 1.3 Mn Welds

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Radiation damage of low alloy steels and welds remains an important issue for the use of these materials in neutron irradiation environments. Research on irradiation damage in low alloy steels and welds has 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.

Energy-Compensated Position-Sensitive Atom Probe (ECOPoSAP) and Local Electrode Atom Probe (LEAP) results have been obtained to assess early stages of irradiation-induced clustering in a low Cu (0.03wt.%) high Ni (~1wt.%) weld. The welds were irradiated at a very high dose rate. Table 1 contains the composition and irradiation doses for the welds. Supporting microstructural information was obtained by small angle neutron scattering (SANS) and positron annihilation (PALA). 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 work was performed using two instruments: an energy-compensated position-sensitive atom probe (ECOPoSAP) at Oxford University (U.K.) and a Local Electrode Atom Probe (LEAP) at a U.S. laboratory.

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. The effect is clearly shown in Figure 1 in which a high number density of clusters is shown near the surface of the reconstructed volume, but relatively few within the bulk of the analysis volume. 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.

ECOPoSAP and LEAP 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 $\sim 5 \times 10^{22} m^{-3}$. Their mean diameter was estimated to be 1.3nm and their mean composition 66Fe-1Cu-13Mn-17Ni-2Si (at.%). Characterization of the low Cu weld irradiated to 95 mdpa was performed using the LEAP. Analysis of the LEAP data (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 $\sim 2.5 \times 10^{23} m^{-3}$. The average composition of the clusters detected was 47Fe-2Cu-12Mn-26Ni-14Si (at.%).

The atom probe results show that the irradiation-induced microstructure in these welds can be described as a population of a low number density of solute clusters (maximum solute separation of 0.4nm) or a higher number density of 'more-diffuse' or dilute clusters (maximum solute separation distance of 0.5nm). The number densities and sizes of clusters observed by SANS are consistent with the latter 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 steel 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.

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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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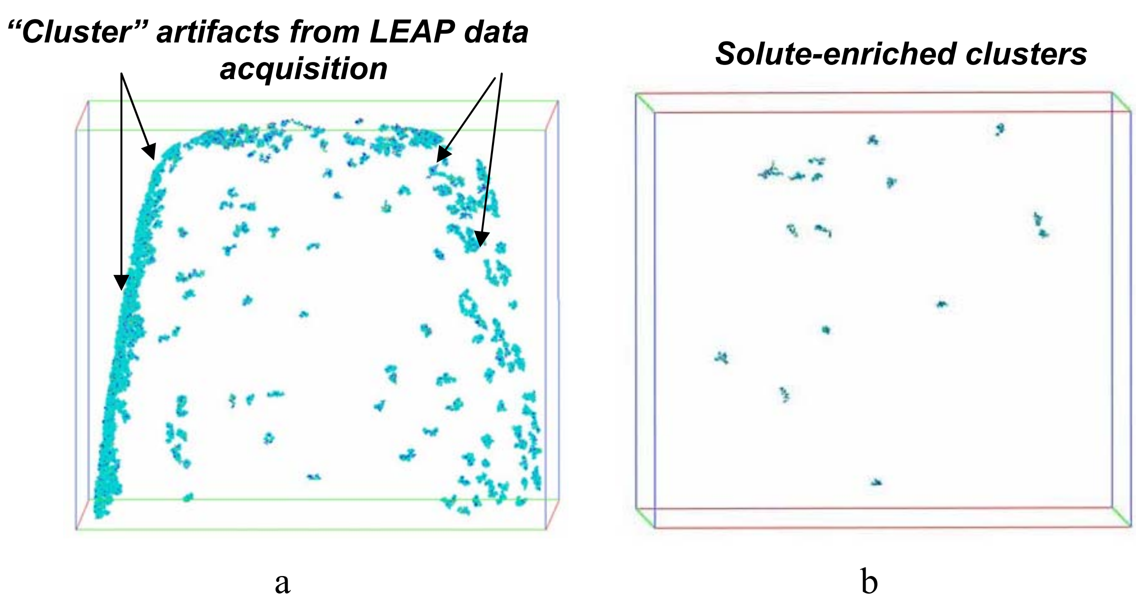

Figure 1. (a) Reconstructed LEAP dataset showing the high concentration of solute clusters located near the surface of the analyzed volume; (b) true clusters detected within the LEAP dataset after removal of the outer 3 nm of the analyzed volume.

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