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Irradiation-Induced Development of Nanoscale Features in Steel: Complementary 3D-APFIM and FEG-STEM Characterization

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Neutron irradiation can promote significant changes in both microstructure and properties of steels. These changes are particularly sensitive to composition, specifically the Cu, Mn, and Ni content of the low alloy steels. Irradiation causes a hardening of the steel, which may lead to a reduction in toughness of the material. The ultra-fine features that are responsible for the change in mechanical properties require analytical techniques with very high spatial and chemical resolution. Atom probe field-ion microscopy (APFIM) has been used with great success to identify and quantify these nanoscale irradiation-induced features. Diffuse solute-enriched "clusters" or "precipitates" containing significant quantities of Fe, as well as Cu, Mn, Ni, and Si, have been documented in a wide variety of low alloy steels and welds. (1-3) Conventional transmission electron microscopy has been unable to identify these chemically complex features, which are likely to exhibit the same body-centered cubic structure as the Fe matrix. In this investigation, the techniques of 3D-APFIM (using the Oxford University Energy-Compensated Optical Position Sensitive Atom Probe) and field emission gun – scanning transmission electron microscopy (FEG-STEM) energy dispersive x-ray (EDX) microanalysis (using the Lehigh University VG HB603) have been used to provide independent analyses of the irradiation-induced nanoscale structure responsible for the changes in mechanical behavior of the material.

The steel used in this investigation was a bainitic low alloy A508 Gr4N forging steel, containing 3.7 wt.% Ni – 0.3% Mn – 1.8% Cr – 0.5% Mo – 0.08% Cu – 0.05% Si – 0.2% C (bal Fe). The steel was irradiated to a dose of 68 milli-displacements per atom (mdpa) at ~250°C. Specimens for analytical electron microscopy and 3D-APFIM characterization were prepared using conventional techniques. Quantitative FEG-STEM x-ray mapping was performed using experimentally generated Cliff-Lorimer "k" factors and the Zeta factor technique (4,5). This permitted the generation of local thickness maps in addition to the composition maps.

The APFIM analyses provided direct evidence of discrete, well-defined ~2 to 4 nm "precipitates" distributed within the matrix, an example of which is shown in Figure 1. The average "precipitate" composition (at%) was ~33Ni -~15 Mn-~6 Cu-~5 Si, despite the low levels of Mn, Cu and Si within the alloy. The APFIM estimated number density was ~5 X 10^{23}/m^3. The composition (wt.%) of the matrix (precipitate-free) was ~3Ni–1.2Cr–0.2Mn– 0.4Mo–0.08Cu–0.03Si (bal Fe). Quantitative EDX maps showed the presence of very fine regions (~3 nm) enriched in Ni and Mn (Figure 2) and depleted in Fe. The number density of these local zones from the Ni-map and thickness map was estimated as ~2 X 10^{23}/m^3. This value is remarkably consistent with the ~5 X 10^{23}/m^3 number density determined by 3D-APFIM of a relatively small volume of material (~16 nm X ~16 nm X ~85 nm). Discrete spot analyses provided a measure of the matrix composition, which was generally consistent with the 3D-APFIM analysis. The maximum Ni level measured via mapping, ~8%, is consistent with the incorporation of an irradiation-induced solute-enriched "precipitate" within the analyzed volume. Thus, APFIM and FEG-STEM EDX mapping have successfully provided independent confirmation of the nanoscale features responsible for irradiation-induced hardening in these alloys.

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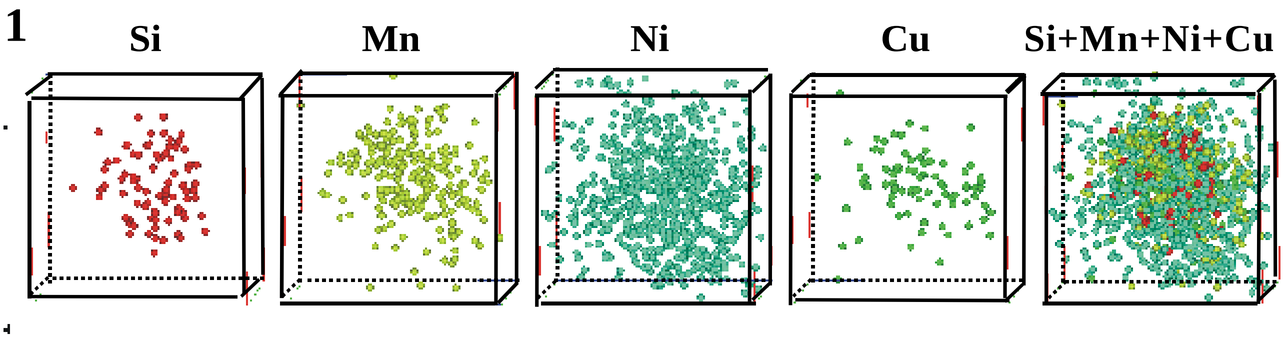

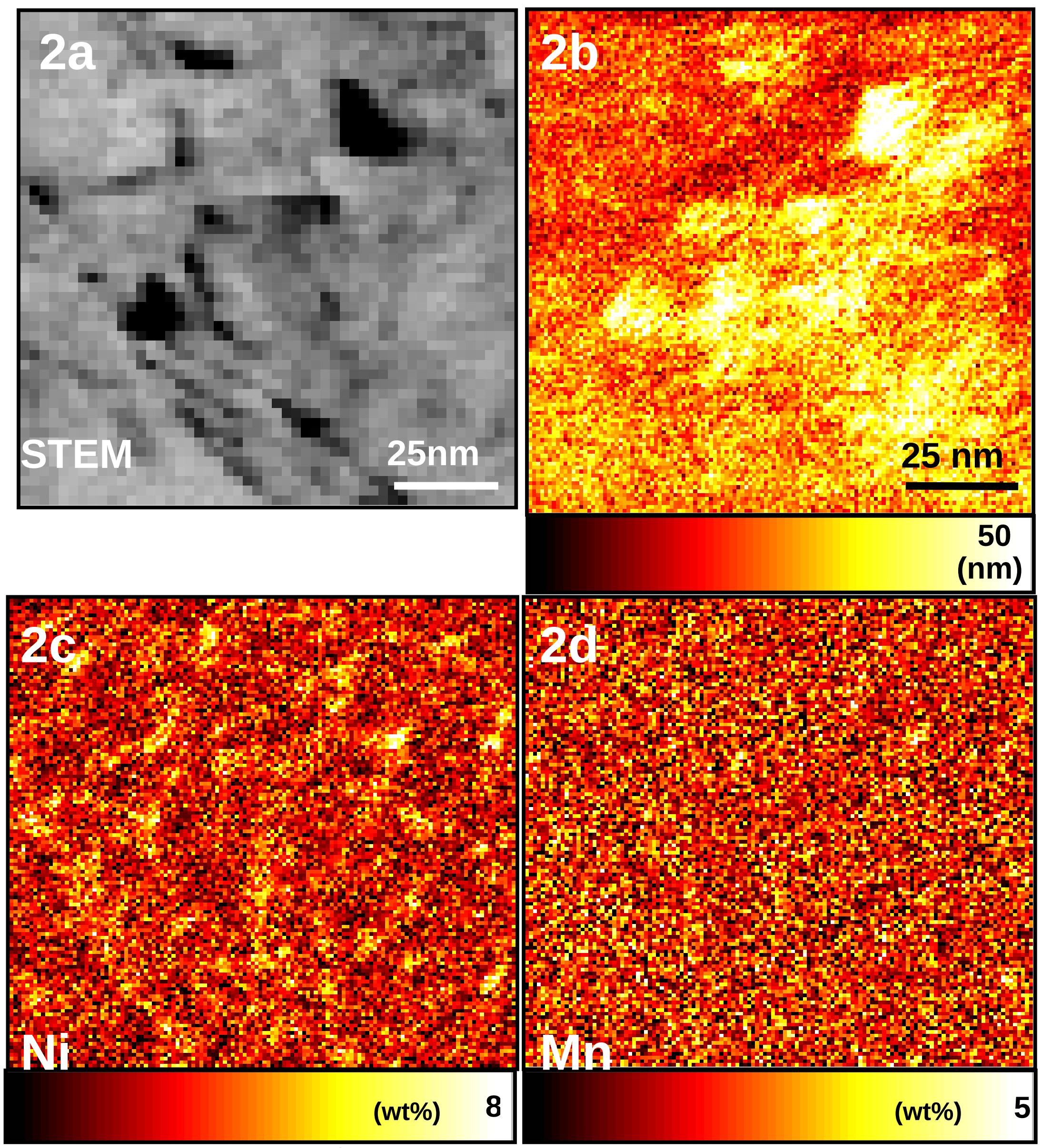

Fig. 1. 3D-APFIM reconstruction for Si, Mn, Ni, and Cu in a typical irradiation-induced solute-rich "precipitate" formed in A508 Gr4N steel. vol: 6 nm X 6 nm X 3 nm.
Fig. 2. (a) STEM image, (b)foil thickness and (c,d) quantitative x-ray maps showing the presence of ~3 to 4 nm irradiation-induced "precipitates". The nanoscale "precipitates" are not visible in the STEM image.

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