Advanced Analytical TEM Characterisation of Irradiation-Induced Nano-Scale Features in Low-Alloy Steel

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The Reactor Pressure Vessel (RPV) of a nuclear reactor is part of the primary pressure boundary for the reactor, and is irreplaceable. As operation periods are extended to 60-80 years it is important that the fracture toughness of the RPV does not significantly deteriorate. Atom probe field-ion microscopy provided the first identification of the ultrafine solute-enriched clusters in neutron-irradiated reactor pressure vessel steel welds [1]. Those Cu-containing welds exhibited enhanced embrittlement and were found to contain Cu-Ni-Mn-(Si)-enriched clusters that were not detectable by transmission electron microscopy. The nature of these solute-enriched clusters varied depending on the composition of the steel/weld. Solute-enriched clusters cause hardening and degradation in fracture toughness. Thus, the detection and analysis of nano-scale solute enriched features is crucial for the understanding of the behaviour of irradiated materials, and for developing predictive models for long-term performance.

A508 Gr 4N Steel is a low Mn-high Ni martensitic/bainitic forging steel, Figure 1. The higher Ni content gives the steel much greater hardenability and significantly lower ductile-to-brittle transition temperature than current generation RPV steels (A508 Gr 3). Previous work on neutron-irradiated (18 mdpa) A508 Gr4N steel by Burke *et al* has shown that no stable solute-related hardening features formed in very low Mn (0.02 wt%) steels[2][3], thereby demonstrating that Mn is an essential element in the development of the solute-enriched clusters (and the associated hardening).

Although it had only been possible to detect and analyse these solute-enriched features with Atom-Probe analysis, advances in analytical TEM including the use of multiple silicon drift detectors (SDDs) have now enabled such nanoscale features to be directly analysed via EDX spectrum imaging and analysis. In this research, we have used the FEI Talos F200 with X-FEG and Super X (4 SDDs) to directly detect nm-scale irradiation-induced solute clustering and segregation in low alloy forging steel.

A508 Gr4N steel has been irradiated with 2 MeV protons to 3 different temperatures in order to study the effects of irradiation temperature on this material. A wide variety of microstructures and resultant changes in the mechanical properties have been produced over the range of irradiation-temperatures investigated, Figures 2-3. The detection and quantification of solute-enriched clusters as seen in a 2-dimensional STEM-EDX elemental image extracted from the STEM-EDX spectrum image dataset of a 3-dimensional volume will be discussed, including determination of the amount of Fe present in these irradiation-induced coherent features.

References

[1] M.G. Burke and S.S. Brenner, J. de Physique, 34:C2 (1986) 239-243.

[2] M.G. Burke, R.J. Stofanak, J.M. Hyde, C.A. English, and W.L. Server, in *Eff. Radiat. Mater. ASTM STP 1447* (ASTM Intl, 2002) 194-207.

[3] M.G. Burke, *et al.*, in *10th Int. Symp. Environ. Degrad. Mater. Nucl. Power Syst. React.*, (NACE, 2002) CD.

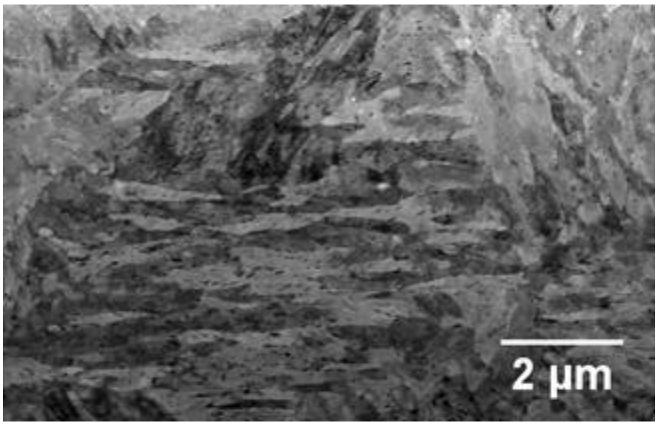

Figure 1: STEM-Bright Field (BF) image of A508 Gr 4N steel, showing Martensitic and Bainitic laths and prior austenite grain boundary.

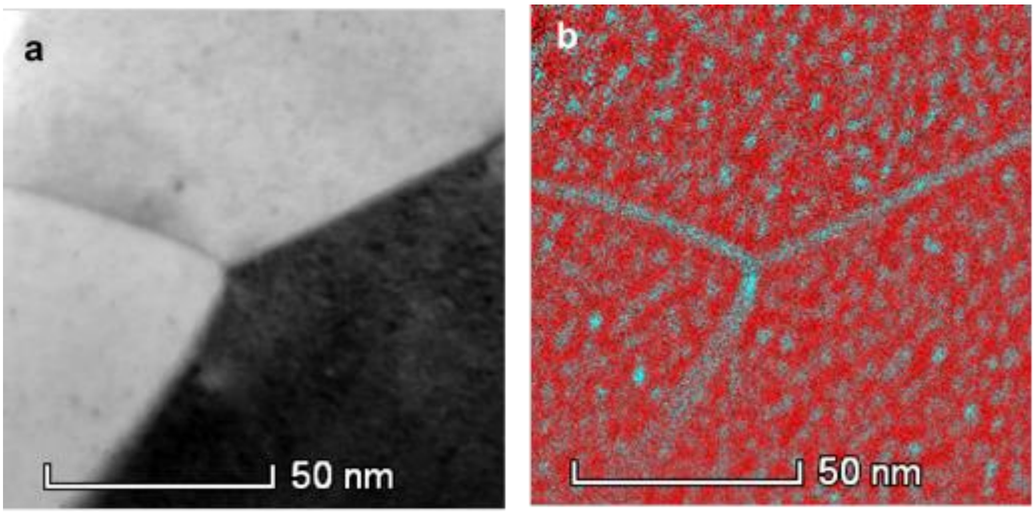

Figure 2: (a) Bright-field (BF) image and (b) combined Fe Kα and Ni K EDX map of A508 Gr 4N steel after proton irradiation at 270 °C to 0.6 dpa. Note the presence of nanoscale Ni-enriched solute clusters throughout the matrix as well as Ni enrichments at grain boundaries.

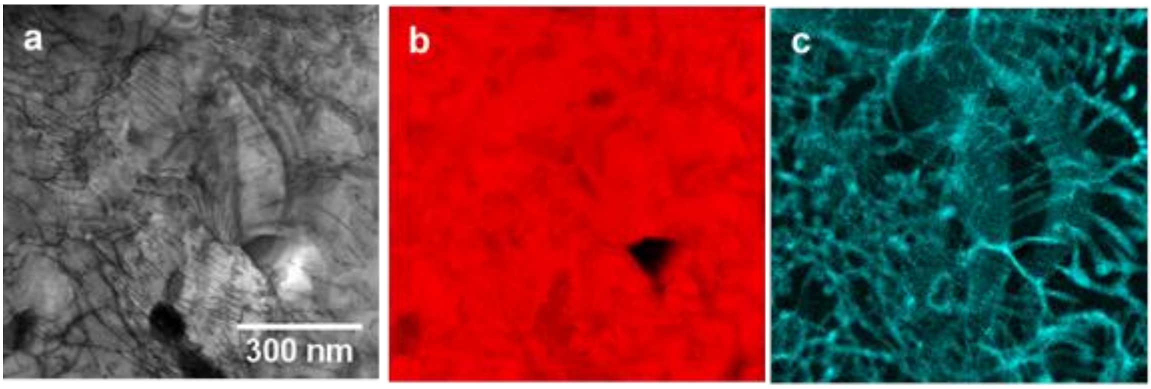

Figure 3: (a) BF-STEM image and corresponding (b) Fe and (c) Ni maps obtained from A508 Gr4N steel proton-irradiated to 0.8 dpa at 450 °C. Significant Ni segregation to pre-existing sinks including dislocations, grain boundaries and carbide-matrix interfaces occurred resulting in pronounced depletions of Ni in the matrix (~1 wt% Ni remaining in the matrix).