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7.3 PRELIMINARY ASSESSMENT OF THE IRRADIATION BEHAVIOUR OF THE FeCrMnNi HIGH-ENTROPY ALLOY FOR NUCLEAR APPLICATIONS

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OBJECTIVE

In the search for new nuclear materials with improved radiation tolerance and behavior, the high-entropy alloys (HEAs) have arisen as new candidates for structural components in nuclear reactors due to their suspected superior stability under irradiation. The metallurgical definition of HEAs is any alloy with multiple elements, five or more all in equiatomic compositions. The basic principle is the high mixing entropy of its solid solution lowers the Gibbs free energy giving a strong enhancement of the microstructural stability at low and high temperatures.

The objective of this project is to assess the irradiation behaviour of the FeCrMnNi HEA system in order to investigate whether the high entropy effect is responsible for a microstructure with better radiation resistance compared to conventional alloys. In this work transmission electron microscopy (TEM) with in-situ ion irradiation has been used at the MIAMI-1 facility at the University of Huddersfield, UK: a 100 kV ion accelerator coupled with a JEOL JEM-2000FX TEM. This methodology allows the evolution of the HEA microstructure to be studied on the nanoscale during the ion irradiation.

SUMMARY OF EXPERIMENTS

The preliminary aim of this project was focused on the characterization of the HEA sample as-cast at ORNL and the production of TEM samples using the Focused Ion Beam (FIB) technique. In addition, some preliminary in-situ TEM irradiation experiments were performed using two different ion beams at 298 K: 30 keV Xe^+ and 6 keV He^+.

Energy dispersive X-ray spectroscopy (EDX) was used to determine the composition of the alloy. The quantitative analysis from the EDX measurements in the as-received alloy showed that the composition in weight percent (%-wt.) was: 25.08-Fe, 20.48-Cr, 27.34-Ni and 20.89-Mn. The balance (6.21 %-wt.) was due to the impurities of C, Si and O. TEM diffraction patterns taken of this HEA revealed it to be a single phase solid solution with a BCC structure.

PROGRESS AND STATUS

To investigate the response of the alloy to low energy ion implantation, a first experiment was carried out at MIAMI-1 using a 6 keV He^+ beam at 298 K. Figure 49 shows bubble nucleation occurring at a fluence of 6.4×10^{16} ions.cm^{-2} (~4 dpa) and bubble size was observed to increase slightly with ion fluence. Details of bubble populations are shown in Figure 50, with migration to twin interfaces was observed during the irradiation (see Figure 50a). After 1.3×10^{17} ions.cm^{-2} (~9 dpa) at 298 K, the temperature was increased to 673 K where bubble coalescence was observed for those bubbles situated on twin interfaces. Figure 50b is the microstructure after irradiation showing He bubbles throughout the alloy at 673 K. The diffraction pattern remained unchanged (inset in Figure 49d) after the irradiation and the annealing.

Another experiment using 30 keV Xe^+ ions showed that after 9.3×10^{16} ions.cm^{-2} (~65 dpa) the BCC structure had changed as can be seen looking at the inset diffraction patterns in Figure 51 and it is attributed directly to the effects of the irradiation. Bending or curling were not observed during the experiment. Bubble nucleation was observed to occur at around 4.7×10^{16} ions.cm^{-2} (~32 dpa) of irradiation at 298 K. Compared to the 6 keV He+ irradiation, the accumulation of bubbles in the twin interfaces was observed, however, it was not significant when compared to the 6 keV He^+ irradiation.

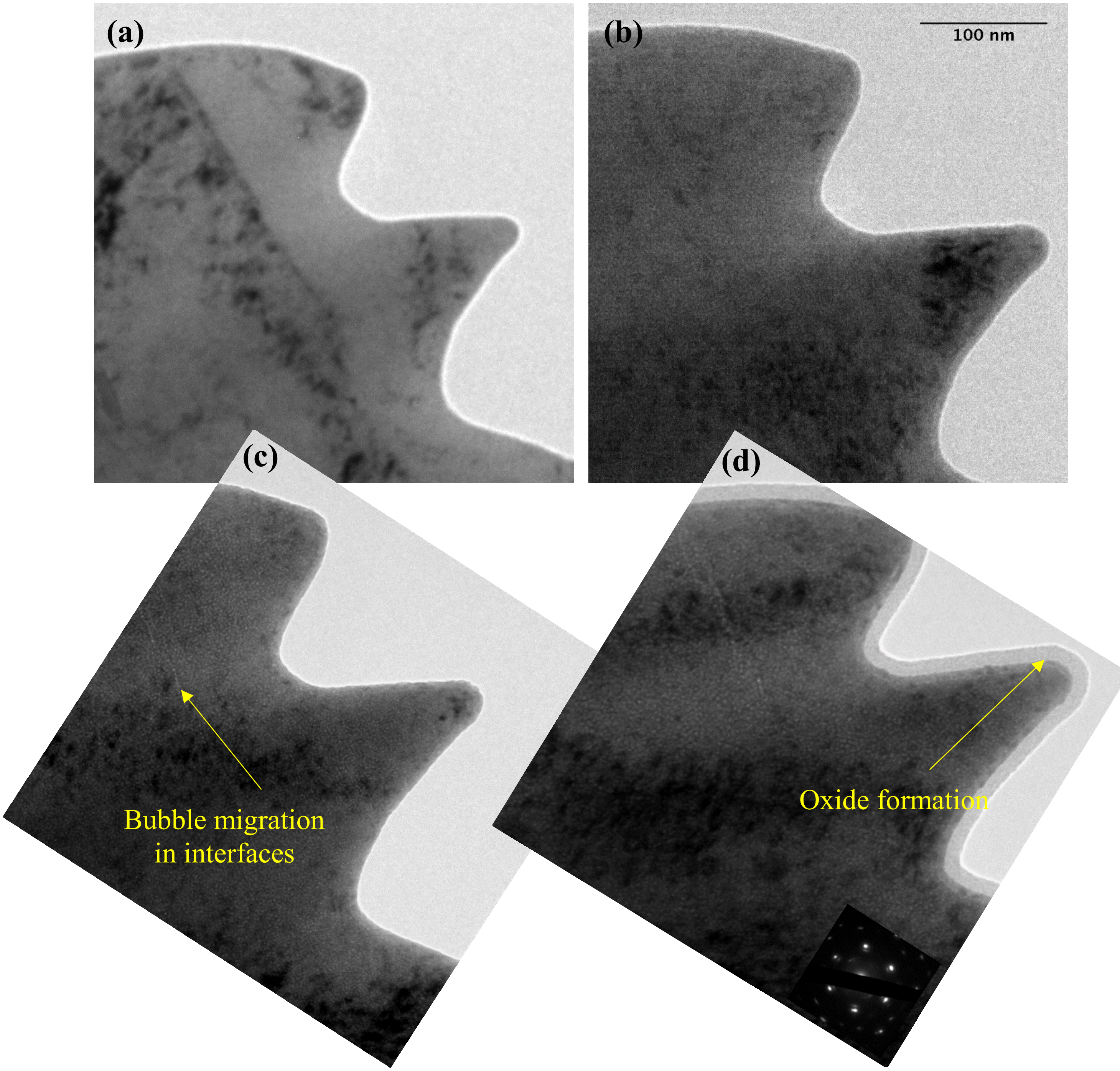

Figure 49. **6 keV He+ in-situ TEM irradiation showing (a) bright-field (BF) TEM image of the unirradiated sample, (b) BF of the microstructure at 298 K after 6.4×10^{16} ions.cm^{-2} (~4 dpa), (c) BF image at 298 K after 1.3×10^{17} ions.cm^{-2} (~9 dpa) and (d) BF image at 673 K after annealing. Note all images taken in underfocus condition of -32 (24 nm).**

An interesting feature of this experiment was the notable degree of sputtering: Figure 51a shows a very sharp needle-like region in which the shape changed to a more rounded geometry (Figure 51c) as the fluence increased suggesting that the Xe ions were responsible for the removal of noticeable amounts of material from the sample. As reported before in the scientific literature, this sputtering might be due to the thermal spike from the individual impacts causing a localised melting so that flow processes driven by surface tension then changes the shape of the specimen.

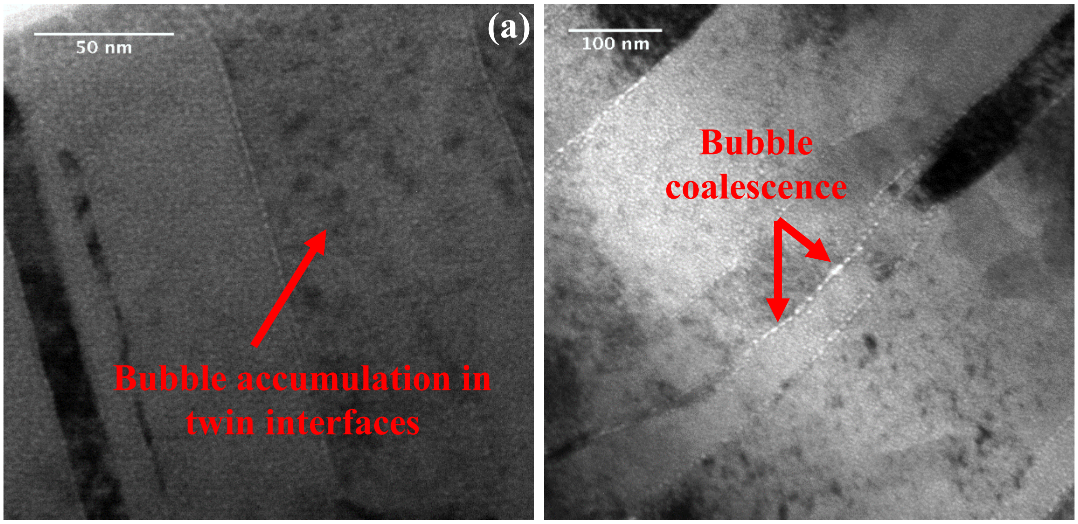

Figure 50. **BFTEM images showing (a) massive bubble accumulation at 298 K and 6.4×10^{16} ions.cm^{-2} of 6 keV He+ irradiation and (b) the final microstructure at 673 K with fluence of 1.3×10^{17} ions.cm^{-2}.**

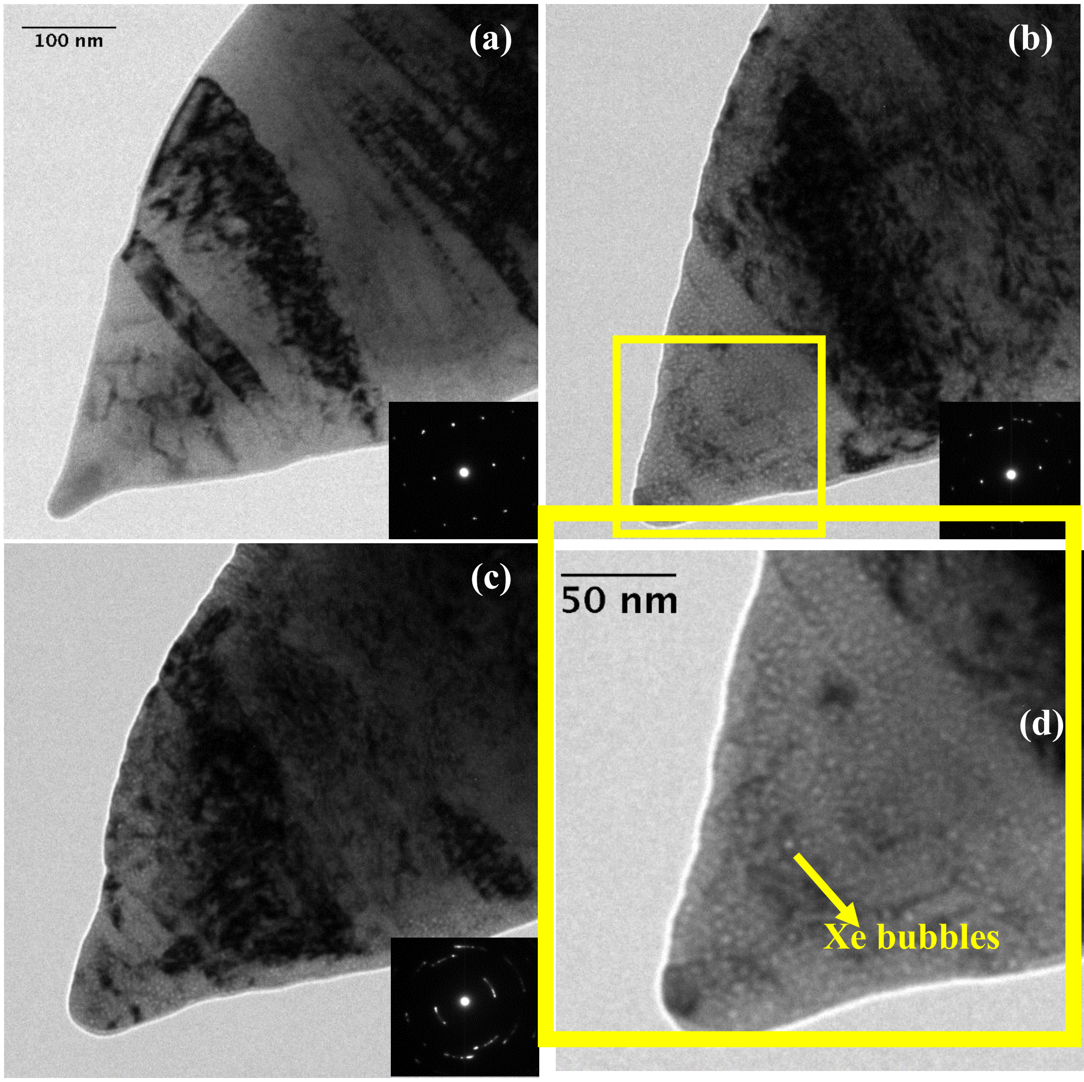

Figure 51. **30 keV Xe+ in-situ TEM irradiation at 298 K of the FeCrMnNi HEA system showing (a) the unirradiated microstructure, (b) BFTEM image after 4.7×10^{16} ions.cm^{-2} (~32 dpa) of irradiation and (c) BFTEM image after 9.3×10^{16} ions.cm^{-2} (~65 dpa). Figure (d) shows in detail Xe bubbles in the microstructure.**

FUTURE PLANS AND FURTHER WORK

Some preliminary experiments have been performed in order to investigate the effects of low energy He (or higher energy Xe implantation) on the microstructure of the FeCrMnNi alloy at 298 K. Bubble nucleation and coalescence were observed using 6 keV He^+ ions and changes in the microstructure were observed with 30 keV Xe^+ irradiation suggesting that the microstructural stability provided by the high entropy effect is sensitive to the energy and the mass of the ion. The observation of bubble accumulation in the twin interfaces may diminish the applicability of this alloy in the nuclear field as inert gas bubbles play key roles in embrittlement, swelling and degradation of mechanical properties in nuclear materials, but these phenomena should be studied further. For future work, new irradiations will be carried out at higher temperatures as well as comparative studies with similar non-high-entropy alloys-to better evaluate the influence of the high entropy effect upon irradiation.