6.3 DISPLACEMENT DAMAGE AND SELF-HEALING IN HIGH-ENTROPY ALLOYS:A TEM WITH *IN SITU* ION IRRADIATION STUDY

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INTRODUCTION

Recent developments in the field of materials for future nuclear fusion reactors have led to the design of innovative metallic alloys that can sustain their mechanical and structural properties under a wide variety of extreme conditions, such as fast neutrons (E <= 14 MeV) and alpha particle bombardment (^{4}He with E up to ~ 3.5 MeV). High-Entropy Alloys (HEAs) are promising candidates for new concepts of nuclear reactors as they have mechanical properties and thermodynamic stability that is believed to be superior to conventional metallic alloys, although their radiation resistance is still a subject of intense research. The efforts to understand the behavior of HEAs under particle irradiation indicated a possible "self-healing" effect of radiation induced defects [1]. In this report, a preliminary study using Transmission Electron Microscopy (TEM) with in situ ion irradiation was performed to investigate the formation and evolution of displacement damage in the microstructure of a FeCrMnNi HEA.

SUMMARY OF EXPERIMENTS

TEM samples from the FeCrMnNi HEA were produced using the conventional Focused Ion-Beam (FIB) lift-out technique in an FEI Quanta 3D 300i. The alloy was produced in the ORNL Materials Sciences and Technology Division by plasma arc melting and the TEM lamellae were produced from the as-cast alloy. TEM with in situ ion irradiation was performed in the MIAMI-2 facility at University of Huddersfield. MIAMI-2 consists of a 350 keV ion accelerator coupled to a Hitachi H-9500 TEM that allows for the direct imaging and observation of damage as it is formed. Observations of the damaged microstructure were recorded by a Gatan Model 1095 OneView at 8 frames-per-second. SRIM2013 was used to convert MIAMI-2 fluences to displacements-per-ion (dpa) using the methodology of Stoller et al. [2]. The HEA sample was kept at 773 K in a double-tilt heating holder and irradiated using 30 keV Xe^+ ions up to a fluence of 2.6×10^{16} ions·cm^{-2} (24.8 dpa).

The image frames were extracted from the digital video recordings using the code FFmpeg [3]**,** which preserves the bit depth and high-resolution. The set of 16 frames analyzed in this report correspond to the irradiation between 296.25 and 298.25 seconds (at a fluence of approximately 1.3×10^{16} ions·cm^{-2} (12.2 dpa). MATLAB Image Processing Toolbox was used in order to extract the information of black-dots occurrence in a set of frames by taking the difference between two successive frames. The images with the differences are then summed within MATLAB to compose the accumulated damage within the set of frames analyzed.

PROGRESS AND STATUS

Figure 49 shows the first two image frames (49a-b) within the analyzed temporal interval and the combined MATLAB image (49c) with the computed difference between the two images. The images were taken in the Bright-Field TEM (BFTEM) mode. Throughout the experiment, black-dots were preferentially observed in a grain where the crystallographic direction was indexed with the software Crystalmaker [4,5] and data available in the literature. The highlighted grain is orientated so that the electron beam is approximately aligned along the [100] zone-axis of the face-centred cubic (FCC) structure.

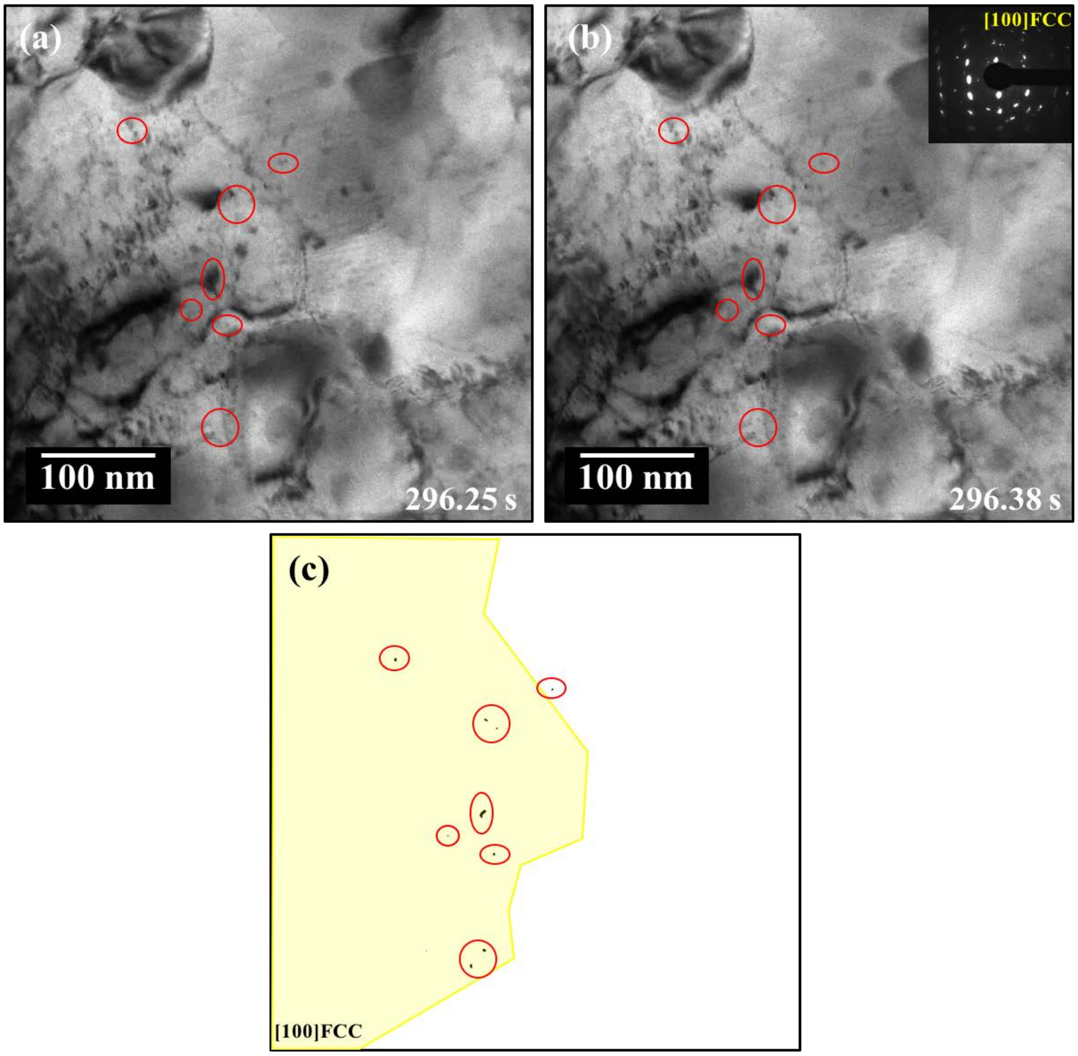

Figure 49. BFTEM images of the FeCrMnNi HEA irradiated with 30 kV Xe ions at 773 K showing (a) and (b) two adjacent frames and (c) the image computed with MATLAB and representing the difference between (a) and (b): exhibiting the black-dots that appeared between the frames.

Figure 50 exhibits (50a) the final microstructure of the 16^{th} frame and (b) the same final microstructure with the superposed information of all black-dots detected in the 16 frames analyzed. The comparison shows that the majority of the black-dots were annihilated within 2 seconds of irradiation at 773 K. Image (c) is an under focused BFTEM image that was taken at a dose of 2.6×10^{16} ions$\cdot$cm^{-2} (24 dpa) where Xe bubbles are observable. The area shown in (d) also exhibits a region of a possible secondary phase formed during irradiation. Further investigation is underway.

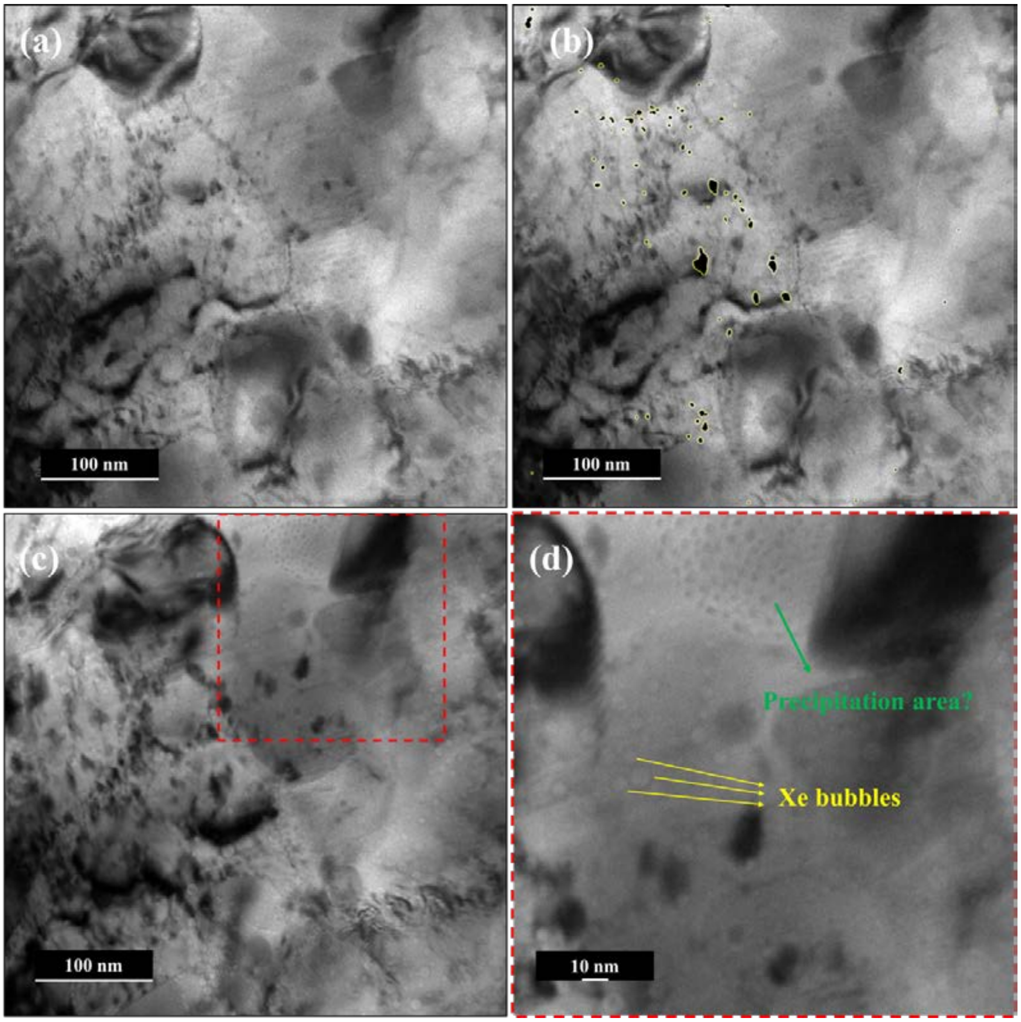

Figure 50. BFTEM images showing (a) the final microstructure of the 16th frame and (b) the same microstructure as (a) with the superposed information of the black-dots within the 16 frames analysed. The micrograph (c) is a BFTEM underfocused image with defocus degree of -1 µm of the same region at 2.6 $\times 10^{16}$ ions·cm^{-2} (24 dpa).

FURTHER EXPERIMENTS AND FUTURE PLANS

The combination of TEM with in situ ion irradiation and image processing using MATLAB described a methodology that may allow further detailed studies of the self-healing of black-dot damage in the microstructure of HEAs under exposure to energetic particles. The MATLAB routine written for this analysis needs implementation for the large data-set produced in each irradiation experiment. The atomistic nature of the black-dots is this HEA is an ongoing subject of research.

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