1.3 MICROSTRUCTURAL CHARACTERIZATION OF A Fe+ IRRADIATED Fe-6%Cr ALLOY — Y. Wu, P. Wells, G. R. Odette, T. Yamamoto (University of California Santa Barbara), S. Roberts and C. Hardie (University of Oxford), D. Bhattacharyya (Australian Nuclear Science and Technology Organization), M. Bachhav and E. A. Marquis (University of Michigan)

OBJECTIVES

The objective of this work is to carry out correlative atom probe tomography (APT) and transmission electron microscopy (TEM) studies on the effects of Fe-ion and neutron irradiation on microstructural evolution in a simple binary Fe-6Cr alloy.

SUMMARY

Both ion-beam and neutron irradiations of candidate structural alloys produce large populations of interstitial dislocation loops that lead to hardening and other effects on mechanical properties. Here we report on the number density, size distribution and character of the loops produced by high energy Fe-ions in a Fe-6Cr binary alloy; and we further compare these results to the corresponding microstructures produced by neutron irradiation. While the two types of irradiations produce qualitatively similar features, there are possibly some notable differences. In particular, it appears that only Si segregates to the loops in the Fe-ion irradiations, while both Si and Cr are associated with the loops in the case of neutron irradiations.

BACKGROUND

Simple Fe-Cr simple binary "model" alloy variants of the complex tempered martensitic and ODS structural alloys that are leading candidates for use in future fission and fusion reactors have been extensively studied [1-4]. At less than 9%Cr, the primary effect of both ion and neutron irradiations at ≈ 300°C is to produce a large population of segregated dislocation loops, often along with impurity atom segregation and solute clustering. For example, Bachhav et al. found two kinds of dislocation loops in APT studies of a Fe-6%Cr alloy neutron irradiated to ≈ 1.8 dpa at ≈ 290°C [1]: small loops with Si and Cr segregated along the dislocation lines and larger loops with Cr segregated along the dislocation lines and Si inside the loops. Here we report on both APT and TEM studies on the same alloy Fe-ion irradiated to ≈ 1.7 dpa at ≈ 290°C.

PROGRESS AND STATUS

Experimental Procedure

The Fe-6 wt.%Cr alloy was heat treated under an argon atmosphere at 950°C for 15 minutes followed by air cooling and then annealed at 750°C followed by air cooling. Irradiation was conducted at the Ion Beam Center in the Rossendorf Institute in Dresden, Germany (Hardies and Roberts). The implantation was designed with multiple ion charges, energies and beam currents to maintain an approximately constant level of dpa damage and dose rate with depth into the sample. The implantation temperature was measured and controlled at a nominal 290°C by thermocouples mechanically clamped to the base plate. The Fe-ion beam produced an average dose of 1.7 dpa in ≈ 2 μm deep layer.

The surface of the Fe+-irradiated samples were prepared by polishing for scanning electron microscopy (SEM) and electron back–scattered diffraction (EBSD) observations. Atom probe tomography (APT) specimens were fabricated by focused ion beam (FIB) micromachining with a FEI Helios tool using standard lift-out procedures. The technique for preparing TEM specimen can be found in Ref. [5].

The APT specimens were run at 55K in voltage mode with a pulse fraction of 20% and a pulse repetition rate of 200 kHz, with an ion detection rate of 0.003-0.005/pulse. Data were reconstructed and analyzed using the Cameca Visualization and Analysis Software (IVAS 3.6.6). The average evaporation field (33V/nm), and the detector efficiency (0.36) were used in the reconstruction of the analysis. The APT

atom maps contain a number of solute segregated dislocation loops. The loops were also characterized using weak-beam transmission electron microscopy (TEM) [6].

Results and Discussion

The measured APT compositions of the Fe+ irradiated Fe-6%Cr alloy for three different runs are in good agreement with the nominal Cr content (≈ 6 at.%). Various trace impurities were also observed, most notably ≈ 0.04 at.% Si.

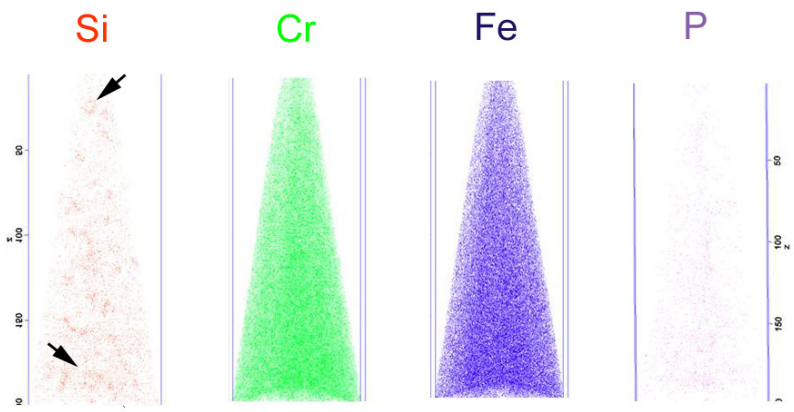


Figure 1. APT element maps, showing the distribution of Si, Cr, Fe and P, respectively.

Visual inspection of the atom maps in Figure 1 show that Si is segregated at the loops, but Cr is not. Note there are some indications of Cr clustering, but may be an artifact, since these atoms are concentrated along low index crystallographic zone lines and towards poles, and are not clearly associated with loops. Likewise, identification of apparent P clusters that emerge in low iso-concentration threshold images are not considered to be fully reliably identified features, although some segregation of this element to loops is observed, along with impurity C. These Fe-ion irradiation results differ from those for the same Fe-6%Cr alloy neutron irradiated to 1.8 dpa at 290°C, where both Si and Cr segregate to dislocation loops [1].

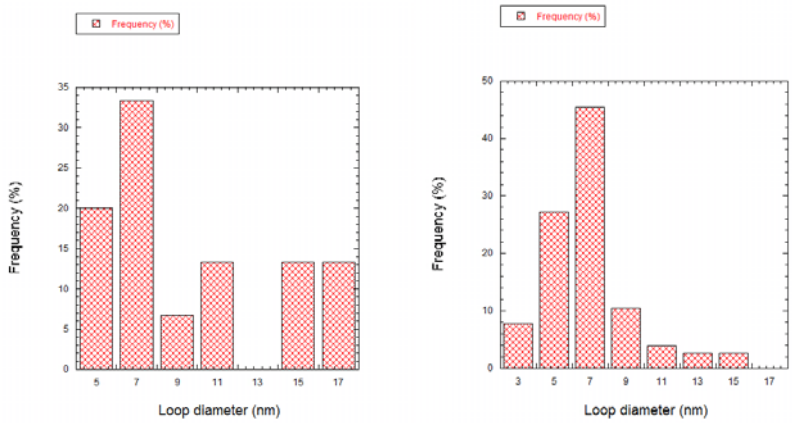
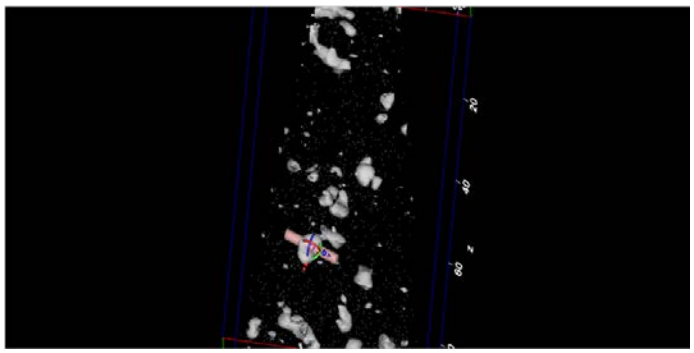


Figure 2. The size distribution of loops measured by APT a) and TEM b).

The dislocation loops found in the APT characterization are ≈ 7-10 nm in diameter with a number density of ≈ $1.4\times10^{22}/m^3$. A histogram of the loop sizes is shown in Figure 2a. There are also some smaller features in the iso-surface with Si threshold of ≈ 0.41% images, that we suspect to be small highly segregated loops. The concentrate profile shown in Figure 3 demonstrates that the dominant segregating species is Si along with smaller amounts of P and C. A comparison of the observed blue line (Cr/20) while the horizontal dashed blue line, which is the nominal Cr concentration, divided by 20 may indicate a slight enrichment. Examination of six composition profiles suggests that any average enrichment of Cr between the Si peaks is less than a factor of ≈ 1.2.

a)



b)

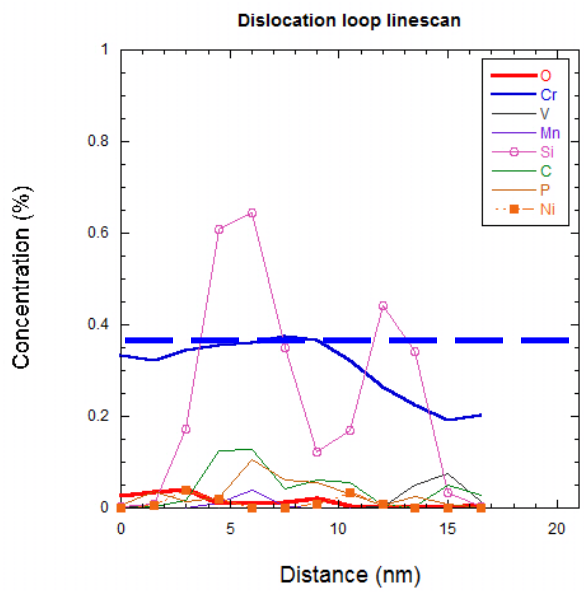


Figure 3. a) Isosurface of Si with the region of interest is getting through one of the dislocation loops; b) APT composition profiles across the highlighted dislocation loop (Cr concentration are divided by 20).

An example of a weak beam bright field TEM images, shown in Figure 4, reveals both <100>{200} and ½<111>{111}-type loops, but the latter are more frequent. The loops have an average diameter of ≈ 6.4 nm. A population of smaller features and black spot type damage are also observed, but details are difficult to resolve in TEM. The corresponding histogram of TEM loop sizes is shown in Figure 2b. While on average the loops are smaller, the TEM results are broadly consistent with the APT data.

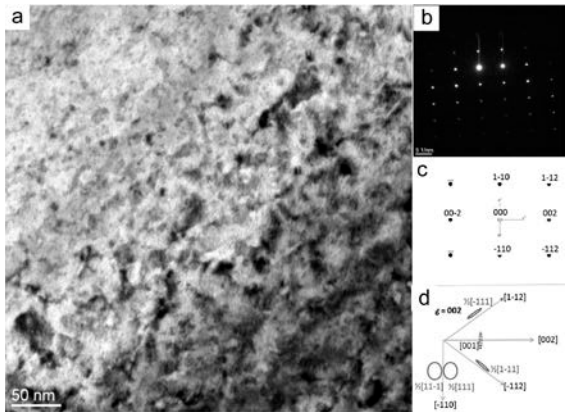


Figure 4. a) Weak-beam bright field image showing dislocation loops near the [110] zone axis using g002; b) The corresponding selected area diffraction (SED); c) The simulated diffraction pattern of bcc-Fe under [110] zone axis; d) A dislocation-loop map showing that the projected dislocation-loops are excited by g002.

Summary

The TEM and APT characterization data on dislocation loops in the ion irradiated Fe-6Cr alloy are summarized in Table 1 and compared to the corresponding neutron data.

Table 1. Comparison of the dislocation loops in ion irradiated and neutron irradiated samples

	Ion		Neutron		Neutron (limited)	
	TEM	APT	APT		TEM	
Loop types	Mostly ½<111>{111}		Mostly ½<111>{111}		Small segregated loops* + larger loops?	
Segregation		Si on loop dislocations lower C & P	Cr at loop periphery Si inside	Si&C on loop dislocations lower C & P	Small segregated loops?	Larger segregated loops
Diameter (nm)	6.4	8	8-10	18-20	2.5	5.4
# density(/m^3)	1.3x10^{22}	1.4x10^{22}	2.9x10^{22}		4.3x10^{22}	1.3x10^{21}

* Seen as solute clusters?

References

[1] M. Bachhav, L. Yao, G. R. Odette, E. A. Marquis, J. Nucl. Mat. 453 (2014) 334.
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[5] Y. Wu, T. Yamamoto, N. Cunningham, G. R. Odette, S. Kondo, and A. Kimura, Fusion Reactor Materials Program June 30, 2013 DOE/ER-0313/54 – Volume 54, PP 42-50.
[6] B. Yao, D. J. Edwards, R. J. Kurtz, J. Nucl. Mat. 434 (2013) 402-410.