Journal of Nuclear Materials 386–388 (2009) 161–164

Contents lists available at ScienceDirect

Journal of Nuclear Materials

journal homepage: www.elsevier.com/locate/jnucmat

TEM characterisation of heavy-ion irradiation damage in FeCr alloys

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A B S T R A C T

Heavy-ion damage in a series of FeCr binary alloys and pure Fe has been investigated by TEM. Specimens were irradiated in bulk with 1.5 MeV Fe^+ ions at an irradiation temperature of 300 °C. A TEM specimen preparation technique has been developed to access the peak of the buried damage region. The damage took the form of dislocation loops with sizes ranging up to a few tens of nanometres. Loops with Burgers vectors of type **b** = <1 0 0> and b = ½<1 1 1> were both present in all the specimens, but the proportion of <1 0 0> loops was higher in FeCr alloys than in pure Fe. In Fe–8%Cr the loop number density was determined to be $(1.6 \pm 0.2) \times 1021$ m-2. Nature determinations showed that <1 0 0> loops in Fe–11Cr and ½<1 1 1> loops in Fe–8Cr were of interstitial type.

1. Introduction

Fusion power is one of the most promising major power sources for the future due to its potentially unlimited availability and environmental advantages. However, structural materials in fusion reactors will be exposed to irradiation by fast neutrons of energy of up to 14 MeV. Such radiation will cause lifetime radiation damage in the material and a whole range of deleterious effects such as activation, embrittlement, void-swelling and creep are likely to occur. Such effects will seriously degrade the mechanical properties and service lifetime of reactor components. It is doubtful if any existing material can withstand the conditions in a fusion reactor economically. It is necessary, therefore, to develop new structural materials if commercial fusion reactors are to be realised.

Ferritic–martensitic steels such as Eurofer 97 are the furthest in development of the three major classes of candidate structural materials for fusion applications [1]. Such steels, which are based on Fe–9%Cr, have the advantages of low activation, good swelling resistance and adequate mechanical properties [2,3]. Model FeCr alloys, as the basis of these steels, are now the subject of several multi-scale modelling programs in the UK, Europe and the United States. However the development of irradiation damage in these alloys has not been studied very extensively. It is known that alloys containing 9%Cr have the lowest increase in DBTT under irradiation, and the Cr content in FeCr alloys is known to have a pronounced effect on void-swelling [1,4,5] The origin of these effects is not understood. In this paper we report our early results on a study of heavy-ion irradiation damage in FeCr binaries up to 11%Cr using TEM.

2. Experimental methods

Bulk Fe–Cr specimens (of thickness about 100 μm and with Cr contents 0%, 5%, 8% and 11%) were irradiated with 1.5 MeV Fe^+ ions at the surrey ion beam centre. The irradiation was carried out at 300 °C to a peak dose of 1dpa. The damage profile calculated by SRIM 2003 is shown in Fig. 1.

Specimens for TEM were prepared using the method described fully by Yao et al. [6]. The peak of the buried damage region at a depth of about 400 nm was accessed by controlled removal of a thin top surface region of the irradiated specimens by timed electropolishing, followed by back-thinning.

Specimens were examined using a Philips CM20 Transmission Electron Microscope. Standard diffraction contrast methods were used to image point-defect clusters and to determine their morphology and nature (see Jenkins and Kirk [7] for details).

3. Results

Weak-beam dark-field (WBDF) micrographs of the microstructures of the Fe and Fe–8%Cr materials are shown in Fig. 2 as examples. The main feature seen in all specimens was a moderately high density of resolvable dislocation loops, qualitatively similar to the microstructures seen in neutron-irradiated Fe–Cr alloys [8]. Typically the loops had diameters about 5–20 nm in all materials. The local foil thickness in a Fe–8%Cr specimen was measured using a convergent-beam method [9] and thereby the number density of loops was measured to be $(1.6 \pm 0.2) \times 10^{21}$ loops/m^3. Similar loop number densities were seen in other materials, but quantitative measurements have not yet been made. There was no evidence for the presence of other defects such as voids.

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doi:10.1016/j.jnucmat.2008.12.078

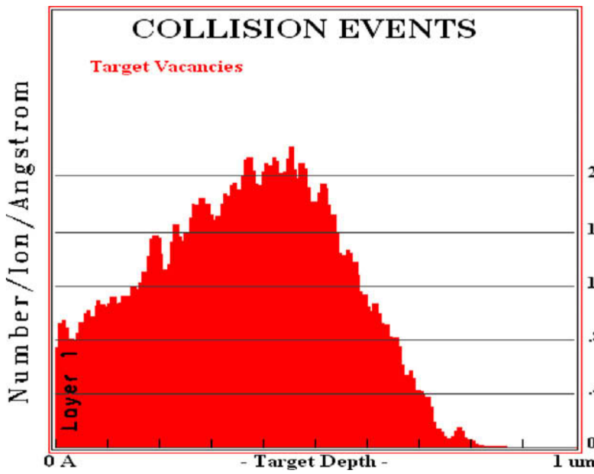

Fig. 1. SRIM 2003 calculation of the damage profile produced by 1.5 MeV Fe^+ ion irradiation in Fe.

3.1. Burgers vector determinations

The Burgers vectors of the loops were determined using the standard **g** · **b** = 0 invisibility criterion. Examples of Burgers vector analyses of loops in Fe–8%Cr and pure Fe are illustrated in Fig. 3 in the upper and lower set of panels respectively. In both cases, the foil normal orientation is [001], Both kinematical bright-field (KBF) and WBDF micrographs of the same area were taken using the four different **g** vectors available at this pole (**g** = 200, 020, 1-10 and 110), and with both ±**g**, but only a selection of KBF micrographs are shown here. Some of the loops are ringed. By following the contrast of individual loops from one micrograph to the next, it can be seen that loops exhibited systematic contrast absences. Where a loop is not visible, its position is marked by a broken circle. This enabled us to determine the Burgers vectors of the loops according to the |**g** · **b**| (See Table 1) shown. For example the ringed loop **A** shown in Fig. 3 is absent only on the **g** = 020 micrograph. Its Burgers vector is therefore **b** = ±[100]. The Burgers vectors of other loops can be found in a similar manner (see figure caption). Loop A and others with the same Burgers vector or with **b** = ±[010] show characteristic 'parallel double line' contrast at this foil orientation (see for example loop **A** in the **g** = 200 micrograph in Fig. 3). This is consistent with these loops lying on the edge-on (100) or (010) planes. These loops are therefore of pure edge character.

Burgers vector analyses were carried out for a large number (∼150) loops in each material, including all visible loops within the area of analysis. The results therefore should be representative of the total loop populations. It was found that in pure Fe, the great majority (92%) had Burgers vector of type ½<111>, and <100> loops were present only in small sizes (∼5 nm). In contrast, in Fe–Cr alloys the proportion of loops of each type was similar and larger <100> loops were present in comparable numbers to ½<111> loops. The proportion of ½<111> loops was 30% for Fe–5%Cr, 46% for Fe–8%Cr and 37% for Fe–11%Cr.

3.2. Loop nature determination

The nature of the loops was determined in some cases by inside–outside contrast experiments, such as that illustrated in Fig. 4. The success of such experiments for small loops depends highly on the quality of the specimen and available information about the loops. It is usually necessary to determine the specific loop Burgers vector and habit plane or sense of inclination. This is often difficult or impossible in ferritic specimens. In the example shown in Fig. 4 it was possible to carry out a tilting experiment to distinguish between two possible ½<111> variants and to determine the loop habit plane. So far unequivocal experiments have been carried out in just the case illustrated (½<111> loops in Fe–8%Cr and <100> loops in Fe–11%Cr). In both cases loops were found to be of interstitial nature.

4. Discussion

It is widely known that both ½<111> and <100> loops are found in Fe and FeCr as a result of radiation damage, e.g. [8,10–12]. However, previous work by Gelles [10,11] found that the fraction of ½<111> loops *increased* with increasing Cr content after neutron irradiation at 400–450 °C to 15 dpa. Porollo et al. [8] also reported that in Fe, Fe–2%Cr and Fe–6%Cr neutron-irradiated at 400 °C to 5–7 dpa, dislocation loops with **b** = <100> were predominant and that the proportion of ½<111> loops increased with

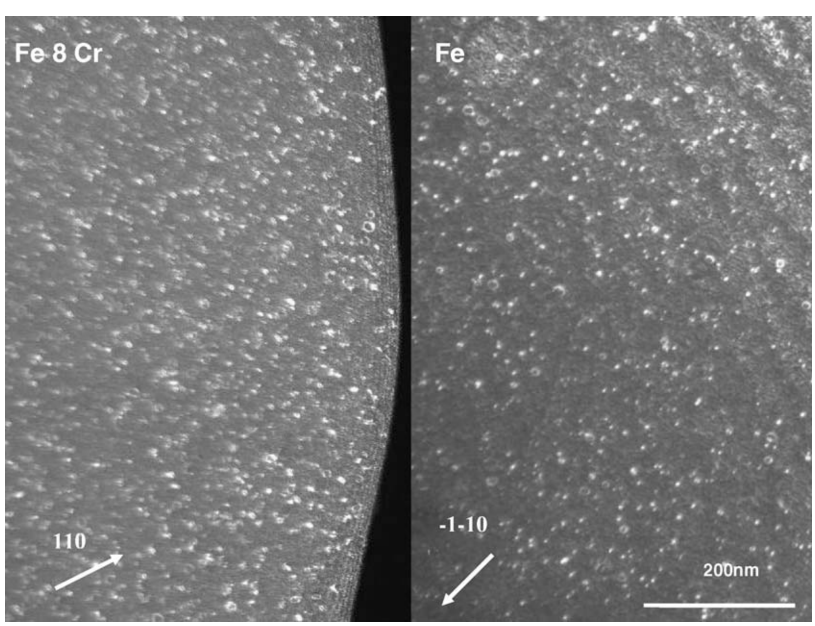

Fig. 2. Weak-beam dark-field micrographs of Fe–8%Cr and pure Fe.

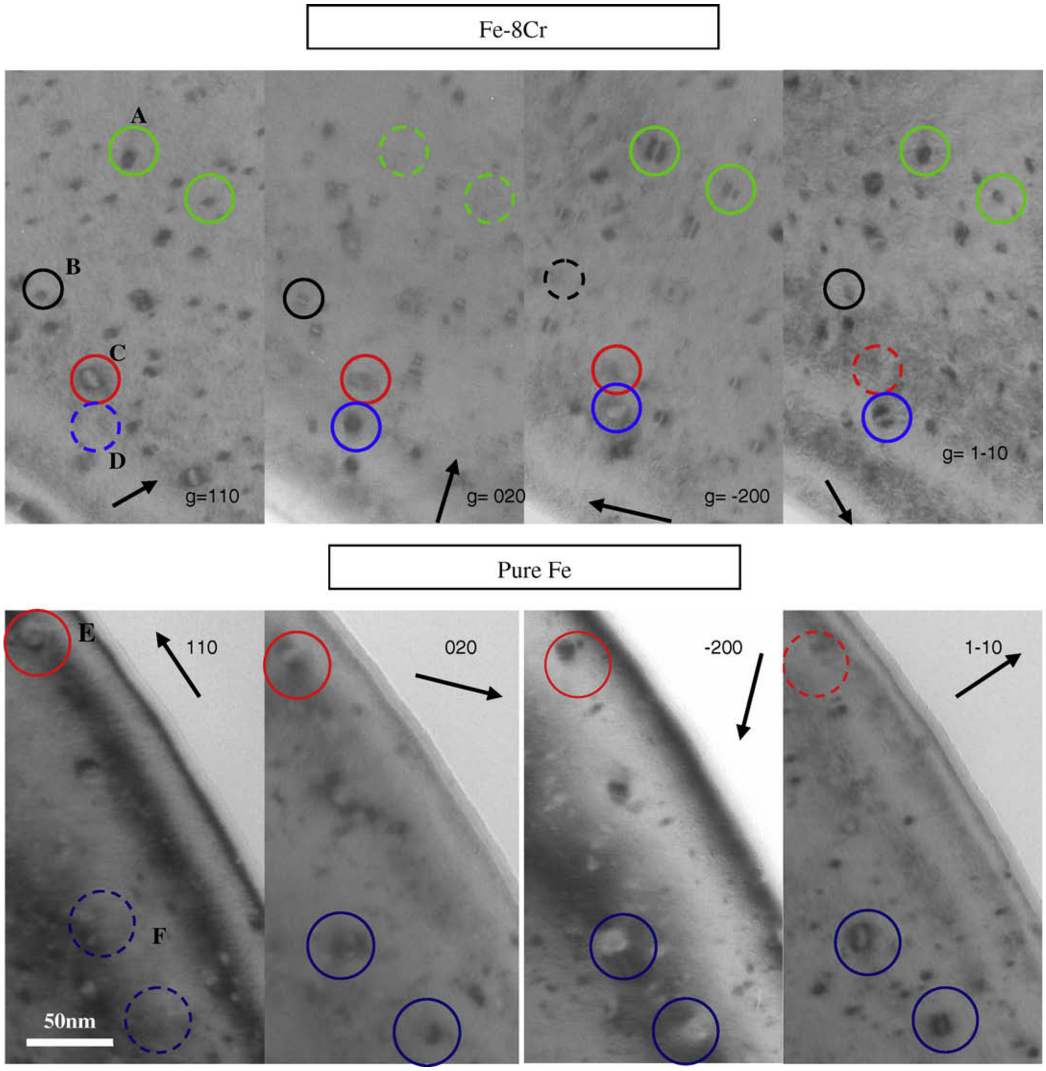

Fig. 3. Burgers vector determination in Fe–8%Cr (upper panel) and pure Fe (lower panel). *Fe–8%Cr:* loop **A**: **b** = [1 0 0]; loop **B**: **b** = [0 1 0]; loop **C**: **b** = ½ [1 1 1] or **b** = ½[1 1 $\bar{1}$]; loop **D**: **b** = ½[$\bar{1}$ 1 1] or **b** = ½[1 $\bar{1}$ 1]. *Pure Fe:* loop **E**: **b** = ½ [1 1 1] or **b** = ½[1 1 $\bar{1}$]; loop **F**: **b** = ½[$\bar{1}$ 1 1] or **b** = ½[1 $\bar{1}$ 1].

higher Cr concentration. Robertson et al. [12] found that in Fe, after neutron irradiation at reactor ambient temperature to low doses where loops were mostly associated with line dislocations, most of the smaller loops had Burgers vector **b** = ½<1 1 1> whilst most of the larger loops had **b** = <1 0 0>. Early experiments by Masters [13] found that the interstitial loops after high dose 150 keV

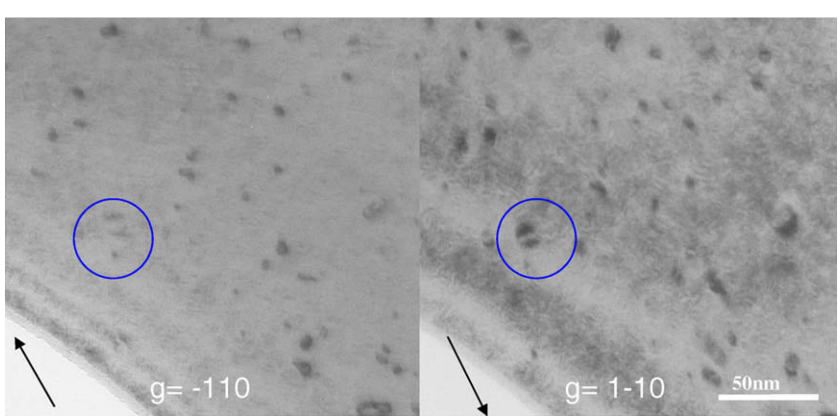

Fig. 4. Inside–outside contrast shown by a loop in Fe–8%Cr. Left: outside contrast; right: inside contrast. The circled loop was shown to have possible Burgers vectors **b** = ½ [$\bar{1}$ 1 1] or **b** = ½ [1 $\bar{1}$ 1] from **g** · **b** analysis. The sense of inclination of the loop was determined by tilting the specimen 20 about the axis [$\bar{1}$ 1 0]. With the assumption that the loop is pure edge, the habit plane of the loop was found to be ($\bar{1}$ 1 1) and the Burgers vector **b** = ½ [$\bar{1}$ 1 1].This information, together with the inside–outside contrast, is sufficient to determine the nature of the loop as interstitial

Table 1
|**g** · **b**| Table for the [001] foil orientation.

g/b	½ [111]	½ [$\bar{1}$11]	½ [1$\bar{1}$1]	½ [11$\bar{1}$]	[100]	[010]	[001]
110	1	0	0	1	1	1	0
1-10	0	1	1	0	1	1	0
200	1	1	1	1	2	0	0
020	1	1	1	1	0	2	0

self-ion irradiation all had **b** = <100>. These results seem contrary to the trends found in our experiments. Further experiments are in progress to investigate this.

5. Conclusions

Experiments have been carried out to characterise heavy-ion radiation damage in pure Fe and Fe–Cr alloys. Damage was in the form of dislocation loops with sizes of tens of nanometres. Loops with both ½<111> and <100> Burgers vectors were present in all materials. In pure Fe, the loops were predominately (92%) of ½<111> type, whereas in Fe–5%Cr, Fe–8%Cr and Fe–11%Cr, the proportion of ½<111> loops was 30%, 46% and 37%, respectively. All loops analysed to date have been found to be interstitial.

Acknowledgement

We are grateful to the UKAEA, Culham Science Centre for funding this work.

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