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Damage behavior in helium-irradiated reduced-activation martensitic steels at elevated temperatures

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ABSTRACT

Dislocation loops induced by helium irradiation at elevated temperatures in reduced-activation martensitic steels were investigated using transmission electron microscopy. Steels were irradiated with 100 keV helium ions to 0.8 dpa between 300 K and 723 K. At irradiation temperatures $T_{irr} \leqslant 573$ K, small defects with both Burger vectors $\mathbf{b} = 1/2\langle 111 \rangle$ and $\mathbf{b} = \langle 100 \rangle$ were observed, while at $T_{irr} \geqslant 623$ K, the microstructure was dominated by large convoluted interstitial dislocation loops with $\mathbf{b} = \langle 100 \rangle$. Only small cavities were found in the steels irradiated at 723 K.

1. Introduction

Reduced-activation ferritic/martensitic (RAFM) steels are candidate structural materials in the future fusion reactor because of their excellent mechanical properties, microstructural stability and swelling resistance [1–3]. However, the helium atoms generated in structure materials exposed to 14 MeV neutrons generated by the fusion reaction can cause significant hardening at temperatures below about 673 K that may result in embrittlement [4,5]. The embrittlement is attributed to the formation of voids, precipitates, and dislocation loops under irradiation that dislocations and thereby impede their glide during deformation.

Experimental evidence suggests that the observed defects in irradiated Fe and ferritic/martensitic steels are $1/2\langle 111 \rangle$ and $\langle 100 \rangle$ type dislocation loops, and there exists a transition irradiation temperature above which only $\langle 100 \rangle$ type loops appear [6,7]. In the 1960s, Eyre and Bartlett found only $1/2\langle 111 \rangle$ type interstitial loops in pure Fe neutron-irradiated at 333 K and annealed above 573 K [8]. At nearly the same time, Master observed exclusively $\langle 100 \rangle$ interstitial loops in pure Fe under 150 keV Fe^{+} irradiations at a temperature of 823 K [9,10]. Later, Little et al. found 98% of loops were $\langle 100 \rangle$ type in FV448 martensitic steel after neutron irradiation at 653 K, 693 K and 733 K [11]. Jenkins and Yao et al. found that the fraction of $\langle 100 \rangle$ loops increased with the increasing irradiation temperature in Fe when irradiated at elevated temperature by heavy ions and concluded that the transition temperature of loop structures in Fe was 765 K [7].

This paper presents a transmission electron microscopy (TEM) study of dislocation loops induced by helium irradiation at temperatures between 300 K and 723 K. The purpose of this work is to investigate the temperature dependence of loop formation.

2. Experimental

The reduced activation martensitic steel (namely SCRAM steel) supplied by Huazhong University of Science and Technology has a composition of 9.24 Cr, 2.29 W, 0.49 Mn, 0.25 V, 0.25 Si, 0.088 C and 0.0059 P in wt%, and Fe for balance [12]. The samples had twice quenching and tempering processes, including quenching at 1253 K for 0.5 h, tempering at 1033 K for 2 h, and then quenching at 1233 K for 0.5 h, tempering at 1013 K for 2 h. Plates were first cut into 0.5 mm thick sheets and then thinned to about 0.1 mm. Standard TEM disk specimens were punched out and thin foils were prepared using a MTPA-5 twin-jet electro-polishing machine (produced by Shanghai Jiaotong University, China), using 5% perchloric acid and 95% ethanol polishing solution at −30 °C.

TEM thin foils were irradiated with 100 keV helium ions using the Accelerator Laboratory of Wuhan University. The sample temperature was maintained at 300 K, 573 K, 623 K and 723 K during irradiation, and was monitored with a thermocouple within an error of ± 5 °C. The irradiation dose was 0.8 dpa calculated with SRIM with a threshold displacement energy input parameter of $E_d = 40$ eV, as recommended in ASTME521-89 [13]. The

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investigation of the microstructure of the specimens was performed with an EM-2010HT TEM operated at 200 kV, which is below the threshold for atomic displacement damage in iron. The image conditions were kinematical bright-filed (KBF) and weak beam dark-field (WBDF). Foil thickness was commonly estimated by counting the number of thickness fringes from the edge of the thin foil.

3. Results

3.1. Loops

KBF micrographs of specimens before irradiation and after irradiation at 300 K using $g = 1\bar{1}0$ are shown in Fig. 1. The line dislocation pileups were observed as shown in Fig. 1(a) and the density of the dislocations was estimated to be $\sim 10^{14}\ \mathrm{m}^{-2}$. No dislocation loops were found in the unirradiated steels. Only few small dislocation loops or radiation-induced defects appeared in the steels irradiated at 300 K, which were marked with dark arrows as shown in Fig. 1(b).

Many small dislocation loops could be observed in the steels irradiated at 573 K. KBF micrographs of these small dislocation loops were taken using four different **g** vectors near the pole [001] ($g = \bar{1}10, \bar{2}00, \bar{1}\bar{1}0$ and $0\bar{2}0$) as shown in Fig. 2. The Burgers vector of dislocation loops were determined using $\mathbf{g} \cdot \mathbf{b} = 0$ invisibility criterion. Table 1 gives $|\mathbf{g} \cdot \mathbf{b}|$ values for a [001] foil normal for loops of the types expected in specimens. Some of the loops are ringed and marked with a letter for easier identification. Loop A is only absent on the $g = \bar{1}10$ micrograph and therefore its Burgers vector is $\mathbf{b} = \pm 1/2[111]$ or $\pm 1/2[11\bar{1}]$. The Burgers vector of loop B, C and loop D are $\mathbf{b} = \pm 1/2[\bar{1}11]$ or $\pm 1/2[1\bar{1}1]$, $\mathbf{b} = \pm[100]$ and $\mathbf{b} = \pm[010]$, respectively, determined using the same method as loop A. Therefore both $1/2\langle 111\rangle$ and $\langle 100\rangle$ type dislocation loops are determined in the steel irradiated at 573 K.

KBF images of specimens irradiated at 623 K and 723 K were shown in Fig. 3. Surprising, mutually perpendicular lines observed in both conditions were determined to be dislocation loop by tilting the specimens. The size of these dislocation loops irradiated at 623 K is significantly larger than that at 573 K. These loops were determined to have $\langle 100\rangle$ Burgers vector and with interstitial nature in the specimen irradiated at 623 K [14].

The Burgers vector analyses of dislocation loops in the specimen irradiated at 723 K were shown Fig. 4 using $g = \bar{1}10, \bar{2}00, \bar{1}\bar{1}0$ and 020 near the [001] zone axis. Loops were marked with letters for easy identification. Take loop E for example, it is only absent on the $\mathbf{g} = 020$ micrograph, thus its Burgers vector is $\mathbf{b} = \pm[100]$. Burgers vector of loop F is $\mathbf{b} = \pm[010]$, using the same method as loop E. No $1/2\langle 111\rangle$ loops were detected in the sample irradiated at 723 K.

3.2. Cavities

No bubbles could be observed when irradiated at temperature $T_{irr} \leqslant 623$ K, and some small bubbles were found in the specimen

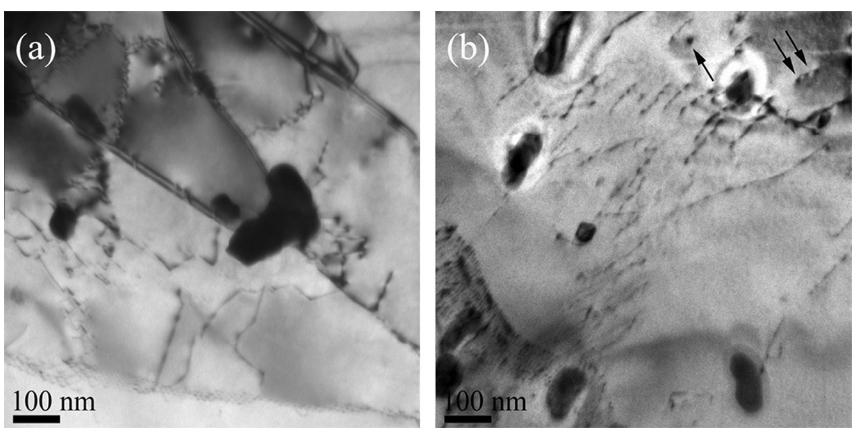

Fig. 1. KBF micrographs of specimens (a) before irradiation and (b) after irradiation at 300 K.

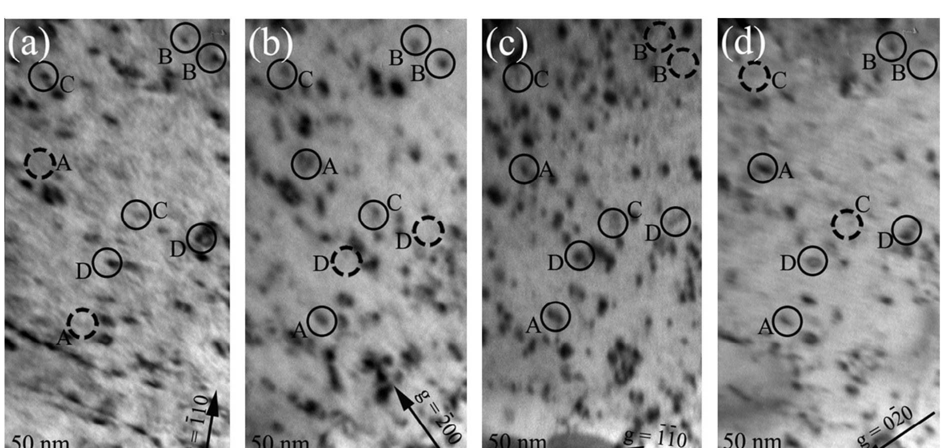

Fig. 2. BF images of dislocation loops in specimens irradiated at 573 K using (a) $g = \bar{1}10$, (b) $g = \bar{2}00$, (c) $g = \bar{1}\bar{1}0$ and (d) $g = 0\bar{2}0$ near pole [100].