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Convoluted dislocation loops induced by helium irradiation in reduced-activation martensitic steel and their impact on mechanical properties

Fengfeng Luo [a], Z. Yao [b], Liping Guo [a,*], Jinping Suo [c], Yongming Wen [a]

[a] Key Laboratory of Artificial Micro- and Nano-structures of Ministry of Education, Hubei Nuclear Solid Physics Key Laboratory, School of Physics and Technology, Wuhan University, Wuhan 430072, China
[b] Department of Mechanical and Materials Engineering, Queen's University, Kingston, ON, Canada K7L 3N6
[c] State Key Laboratory of Mould Technology, Institute of Materials Science and Engineering, Huazhong University of Science and Technology, Wuhan 430074, China

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ABSTRACT

Helium irradiation induced dislocation loops in reduced-activation martensitic steels were investigated using transmission electron microscopy. The specimens were irradiated with 100 keV helium ions to 0.8 dpa at 350 °C. Unexpectedly, very large dislocation loops were found, significantly larger than that induced by other types of irradiations under the same dose. Moreover, the large loops were convoluted and formed interesting flower-like shape. The large loops were determined as interstitial type. Loops with the Burgers vectors of $\mathbf{b}=\langle 100\rangle$ were only observed. Furthermore, irradiation induced hardening caused by these large loops was observed using the nano-indentation technique.

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1. Introduction

Reduced-activation ferritic/martensitic (RAFM) steels are proposed as main candidate structural materials for fusion reactors because of their excellent mechanical properties, microstructural stability and swelling resistance [1–3]. Under fusion conditions, considerable helium and hydrogen are supposed to be induced through transmutation reactions in addition to severe displacement damage [4]. The synergistic effect of those impurities (He and H) and displacement damages will cause significant irradiation hardening at temperatures below 400 °C that may result in embrittlement of materials [5]. So far most of the studies concerning the effect of helium on microstructure in RAFMs were focused on helium bubbles and swelling [6–8], whilst few were reported to investigate the dislocation structures induced from energetic helium particles. It is not clearly understood how helium affects irradiation structures other than causing atomic displacement or forming bubbles.

In this work, dislocation loops induced by helium ion irradiation were our primary care and they were studied with transmission electron microscopy (TEM). Surprisingly, very large loops of up to 440 nm were observed, much larger than that induced with other irradiations such as neutron, proton and heavy ion irradiations at the same dose. Moreover, dislocation loops with very interesting shapes were also observed. The dislocation loops were characterized with advanced TEM techniques.

2. Experimental

The reduced-activation martensitic steel used in the present study has a composition of Fe–9.24% Cr, 2.29% W, 0.49% Mn, 0.25% V, 0.25% Si, 0.088% C and 0.0059% P in weight percent (namely SCRAM, supplied by the Huazhong University of Science and Technology [9]). The steel was subjected to two heat treatments: firstly quenching at 980 °C for 0.5 h and tempering at 760 °C for 2 h; secondly quenching at 960 °C for 0.5 h and tempering at 740 °C for 2 h. After the treatments, specimens had a final fully tempered martensite lath structure. Plates were first cut into 0.5 mm thick sheets and then mechanically polished down to about 0.1 mm. Standard TEM disk specimens were punched and milled to a thickness of 40–50 μm with silicon carbide (SiC) paper of grades 800–2500. Finally, thin foils were polished by a MTPA-5 twin-jet electro-polishing machine (produced by Shanghai

* Corresponding author. Tel.: +86 27 6875 2481x2223; fax: +86 27 6875 2569.
E-mail address: guolp@whu.edu.cn (L. Guo).

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Jiaotong University, China), using polishing solution of 5% perchloric acid and 95% ethanol. Bulk materials were fabricated into flake-like specimens of 20 mm × 5 mm × 1 mm sizes for nano-indentation measurement. Surface of the specimens were initially mechanically polished with SiC paper of grades 800–4000, then polished with 0.5 μm alumina powder. To remove the polishing damage, specimens were finally electropolished with 8% perchloric acid, 10% butoxyethanol, 60% ethanol, and 22% distilled water (vol%) at 273 K at a voltage of 40 V for 30 s. The thickness of the electropolished specimens is about 0.9 mm.

TEM specimens were irradiated with 100 keV helium ions at 350 °C to a fluence of 5×10^{16} ions cm^{-2} using an ion implanter in the Accelerator Laboratory of Wuhan University. The temperature was monitored with a thermocouple during the irradiation, within an error of ±5 °C. The equivalent damage dose at the observed area was estimated to be 0.8 dpa according to the SRIM calculation with displacement energy of 40 eV as recommended in ASTME521-89 [10]. The investigation of the microstructure of the specimens was performed with a Philips CM20 TEM operated at 200 kV, below the threshold for knock-on damage in Fe. The often used image conditions were kinematical bright-field (KBF) and weak beam dark-field (WBDF), but only KBF images are shown here. Foil thickness at the observed area was fixed at ~100 nm, which is estimated by counting the number of thickness fringes from the edge of the thin foil. In terms of Li's model and experimental results of the in situ TEM irradiation damage study [11], only 7–10 nm large denuded zones are formed near top and bottom surfaces of the TEM thin foil due to surface loop loss effects, thus irradiation induced microstructures observed in the present study represent important characteristics from bulk irradiation.

Flake-like specimens were irradiated with 250 keV helium ions at 350 °C to 0.8 dpa. After irradiation and thermal aging, nano-indentation was performed using an MTS Nano Indenter XP instrument at the State Key Laboratory for Advanced Metals and Materials, University of Science and Technology, Beijing. A Berkovich diamond indenter was used with the continuous stiffness measurement (CSM) method. The indentation depth is up to 1500 nm for all specimens.

3. Results

The unirradiated steel exhibits a typical martensitic lath structure and no dislocation loop could be observed. Fig. 1(a) presents a KBF micrograph of the microstructures of specimen after irradiation using **g**=01−1 in a foil oriented near [100]. Unambiguously, these mutually perpendicular lines were confirmed to be dislocation loops by tilting the foil from pole [100]→[111], whilst maintaining roughly the same diffraction condition with **g**=01–1, as shown in Fig. 1. Interestingly, prior to the tilting, sharp fringes were seen inside the loops, which looked like stacking fault fringes. However, when tilting to near pole [111] convoluted structure of loops appeared, which looked entirely different from stacking fault fringes, as shown in Fig. 1(f).

Dislocation loops could be found in most grains with the size ranging from several nanometers to several hundred nanometers. The distribution of these loops in different areas is shown in Fig. 2. These loops could be sorted into two sizes: the large ones (over 50 nm) and the small ones (less than 50 nm). We examined dislocation loops in twenty grains of irradiated specimen. Among them, eleven grains' foil was oriented near ⟨110⟩ within 10°. Large loops were only observed in six grains and small loops were mainly observed in other five grains. Since both large loops and small loops were observed under the same foil orientation, no obvious correlation between the sizes of these loops and the foil orientation was found.

For large loops, near the pole [100] they look sharp and form fairly regular arrays as shown in Fig. 2(a), whereas near the pole [111] they have irregular shape with slightly serrated borders and look like flowers as shown in Fig. 2(b). The largest size of loops observed in the present study is as large as 440 nm. The mean size and density of large loops are 138 nm and 1.0×10^{21} loops m^{-3}, respectively. For small loops as shown in Fig. 2(c), the mean diameter and density of the small loops are 26 nm and 1.67×10^{22} loops m^{-3}, respectively. Table 1 summarizes the size of dislocation loops under different irradiations in pure Fe and RAFM steels [12–19]. The sizes of these dislocation loops mainly depend on the radiation type, temperature and dose. It is obvious that the loops induced by helium irradiation in the present study are significantly larger than that induced by other types of irradiations (such as neutron, proton and heavy ions) under the same dose.

The contrast of dislocation loops is expected to vanish or become faint under weak beam or kinematical diffraction conditions while **g**·**b**=0. Fig. 3 showed the Burgers vector analyses of both large and small dislocation loops. Both kinematical KBF and WBDF micrographs of the same area were taken using four different **g** vectors close to the pole [100] (**g**=011, 01−1, 0−20 and 002) for large loops and three different **g** vectors close to the pole [111] (**g**=01−1, 10−1, and 1−10) for small loops, with both ±**g**, but only a selection of KBF images are shown here. Some loops are marked with a letter to be easily identified. For large loops, loop A is only absent on the **g**=002 micrograph and its Burgers vector is therefore **b**=±[010]. The Burgers vector of loop B is **b**=±[001], using the same method as loop A. A third set of loops with **b**=±[100] which lie in the foil plane is invisible because of **g**·**b**=0. For small loops, the Burgers vectors of loops D, E and F are determined using the same method to be **b**=±[100], ±[010] and ±[001] respectively. Both large loops and small loops have the same Burgers vector of ⟨100⟩ in this irradiated specimen.

The nature of these loops was determined by the inside and outside contrast changes (the method is well explained by Jenkins and Kirk [20]), as shown in Fig. 4. These micrographs were acquired by first tilting the foil ~20° away from the pole [100] along a 01–1 Kikuchi band. And KBF micrographs were then taken using **g**=±01−1 with deviation parameter $s_g > 0$, keeping the magnitude $|s_g|$ constant. Loop A shows outside contrast in Fig. 4(a), **g**=01–1 and s_g to be positive. Outside contrast implies (**g**·**b**) $s_g > 0$, therefore the Burgers vectors of loop A is **b**=010 rather than **b**=0−10. The sense of inclination of loop A at the imaging orientation was confirmed by tilting along the 01−1 Kikuchi band relative to this pole. The direction of **b** at the imaging orientation could be determined, thus **b**·**z**<0. Loop A is therefore of interstitial nature. Loop B is also found to be of interstitial type using the same method as loop A.

Typical nano-hardness curves of the thermal aging and irradiated specimens obtained by the nano-indentation test are shown in Fig. 5. Since the nano-hardness in the indented specimens reflects the hardness value at the five times indentation depth [21], the hardness values are taken at indentation depth of 120 nm which reflects the hardness at about 600 nm depth corresponding the peak damage zone. From the nano-hardness curves, the irradiation induced hardening is found to be ~280 MPa at the depth of 120 nm.

4. Discussion

We strive to give an explanation on the unexpected large size and interesting shape of dislocation loops induced by helium

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