Journal of Nuclear Materials 511 (2018) 304–311

Contents lists available at ScienceDirect

Journal of Nuclear Materials

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

Characterization of ordered dislocation loop raft in Fe^{3+} irradiated pure iron at 300 °C

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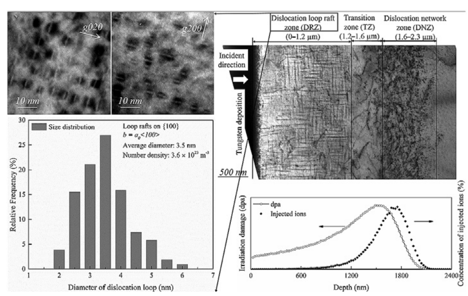
HIGHLIGHTS

- Three distinct damaged zones in ion-irradiated pure Fe.
- A well-developed dislocation loop raft was observed.
- Loop raft has a 2−D distribution on habit plane of *{100}* with $b=a_0$*<100>*
- *100>* loops had an average size of 3.5 nm with number density of 3.6×10^{23} m^{-3}.

GRAPHICAL ABSTRACT



ARTICLE INFO

Article history:
Received 23 May 2018
Received in revised form
11 September 2018
Accepted 12 September 2018
Available online 20 September 2018

Keywords:
Ion irradiation
Dislocation loop
Ordered structure
Burgers vector

ABSTRACT

Pure iron was irradiated with 6.4 MeV Fe^{3+} ions at 300 °C to a displacement damage of 30 dpa at a nominal depth of 600 nm. Distinct microstructures were observed at different depth regions by transmission electron microscopy (TEM). In the range of 0–1.2 μm from the irradiation surface, dislocation loops tended to form ordered loop rafts with their habit plane on *{100}*, while in the range of 1.6–2.3 μm, both line-shaped dislocations and dislocation loops coexisted. Dislocation loops that formed the loop raft had an average size of 3.5 nm with the number density estimated to be 3.6×10^{23} m^{-3}, considering all three variants of the Burgers vectors $b = a_0$*<100>*. However, the dislocation loops in the range of 1.6 −2.3 μm were with Burgers vectors, $b = a_0/2$*<111>*, and the number density was one order of magnitude lower.

1. Introduction

High energetic particles irradiation often results in the formation of ordered damage structures such as void lattice, loop string and loop raft in metallic materials. Ordered dislocation loop raft structure has been observed in heavy ion or neutron irradiated BCC pure metals or alloys [1–7].

The formation of loop raft in tungsten (W) was reported extensively in the literature. Zhang et al. reported that dislocation loop rafts were observed in Fe^{3+} irradiated pure W at temperatures of 300 °C, 500 °C and 700 °C up to a peak damage of 4.3 dpa [1]. Hasenhuetl et al. also investigated the damage structures in pure W single crystals after Fe^{3+} ion-irradiation at 300 °C, and observed a partially ordered networks of dislocation loop rafts. As for the crystal orientation dependence, dislocation loop rafts were observed in the W single crystals with the surface orientations of < *100>* and < *011>* at the dose of 1 and 2 dpa, while the loop rafts were only observed in the W single crystal with the surface

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https://doi.org/10.1016/j.jnucmat.2018.09.020
0022-3115/

orientation of < *011*> in the case of 0.1 dpa [8]. Yi et al. reported that after the irradiations at temperatures higher than 500 °C, an ordered loop raft structure was observed in self-ion irradiated pure W but not in W–Re or W–Ta alloys, although they suggested that such loop structures appeared at higher doses [9]. Unlike most of the other studies, however, loop rafts were also observed recently in W with coarse grains irradiated with 3 MeV Cu^+ at a low dpa rate at room temperature (RT) [10]. Studies on the formation of loop raft in pure iron (Fe) or iron based alloys are quite limited but similar microstructures with pure W still can be found in the literature. M. Hernández-Mayoral et al. investigated the microstructure of heavy-ion irradiated ultra-high-purity Fe and Fe–Cr model alloys in thin foils at 300 °C. At lower doses <1 dpa, spatial randomly distributed dislocation loops with the Burgers vector of $b = a_0$<100> were formed. However, at a higher dose of 1.3 dpa, strings of 4–10 small loops have developed. When the dose further increased to 6.5 dpa, chains of resolved dislocation loops with $b = a_0/2<111>$ are visible, indicating the dose dependence of string formation. When pure Fe was irradiated at room temperature (RT), similar dislocation loop strings with the Burgers vector $b = a_0/2<111>$ were observed as those at 300 °C [11,12] and in Fe–5%Cr alloy irradiated with 2 MeV Fe^+ ions up to 100 dpa at 500 °C [13]. Under neutron irradiations, loop raft structure was observed in pure Fe irradiated to 0.79 dpa at 50–70 °C [14]. The threshold dose for the raft formation was determined to be ~0.3 to 0.4 dpa and the raft formation was well developed at 0.79 dpa. The examination of dislocation loops indicated that most of the loops were with $b = a_0/2<111>$ clustered along *{111}* habit planes. To conclude, the formation of loop rafts heavily depended on material, irradiation temperature and dose. As for molybdenum the loop raft was only observed after irradiations at intermediate temperatures from 400 °C to 600 °C [3], while in iron and tungsten the loop raft was formed both at low temperatures and high temperatures, RT to 800 °C [1,7,8,10–14].

However, in the previous studies no detailed examination of the loop rafts was performed so far. In this study, we characterized the ordered dislocation loop rafts formed in the ion-irradiated pure Fe with respect to the Burgers vector of the loops and habit plane of the raft, and finally the possible formation mechanism was discussed.

2. Experimental

The material used in this study was pure Fe (99.99 wt%) which was cold rolled to a thin sheet and punched out to the geometries of $2 \times 3 \times 0.3$ mm^3. Then the specimen was normalized at 1050 °C for 0.5 h followed by furnace cooling. Specimen surface was mechanically polished with SiC papers from # 800 to # 4000, and buff-polished with diamond pastes of diameters from 3 μm to 0.25 μm. Finally, the specimen was electrolytically polished in an electrolyte of 5 vol% of perchloric acid with methanol at − 50 °C with an applied voltage of 12 V.

The specimen was irradiated with 6.4 MeV Fe^{3+} at 300 °C with a tandetron accelerator, DuET, in Kyoto University. The irradiation temperature was controlled within an error of ±10 °C by a heater attached to the specimen holder and measured by a thermocouple on the specimen holder as well as an infrared pyrometer [15]. The vacuum was at a level of 10^{-6} Pa at 300 °C. The beam was raster-scanned with frequency of 1000 Hz to horizontal-direction and 300 Hz to vertical-direction. The average beam current was 6.5 nA during the irradiation. The corresponding displacement damage and damage rate at a nominal depth of 600 nm was 30 dpa and 4.4×10^{-4} dpa/s, respectively. Fig. 1 shows the calculated dose and concentration of the injected Fe ions as a function of depth using the Monte Carlo simulation code SRIM, where the dose calculation was done using the Kinchin-Pease option and the displacement energy of 40 eV for Fe atoms.

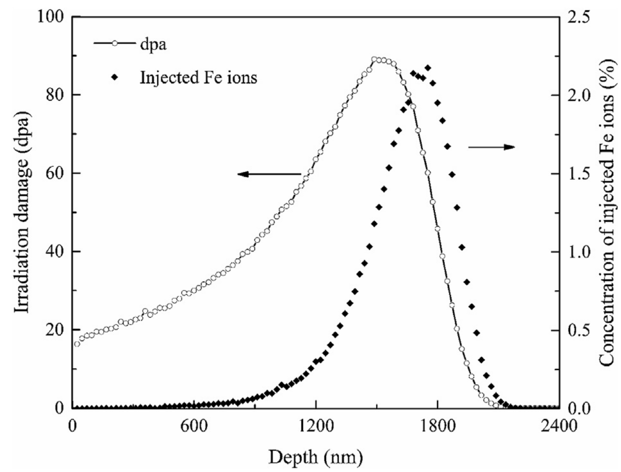

Fig. 1. Profiles of displacement damage and concentration of the injected iron ions as a function of depth calculated with SRIM code.

TEM specimens were prepared on a focused ion beam (FIB) system followed by flash electro-polishing to further remove the artifacts introduced by FIB. Then the microstructure of irradiation damage was investigated on a JEM 2010. The Burgers vector of dislocation loops was characterized based on a combination of crystallographic information and $g \cdot b = 0$ invisibility criterion. Both the size distribution and number density of dislocation loops were obtained with the thickness of the specimen measured by convergent beam electron diffraction (CBED) method.

3. Results

Fig. 2 shows the whole cross sectional view of the damaged area

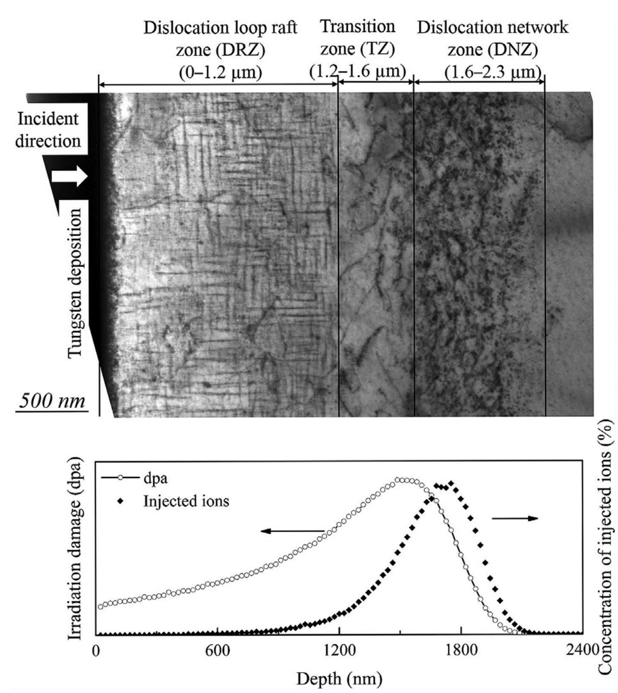

Fig. 2. Overall view of the damaged region in 6.4 MeV Fe^{3+} irradiated pure Fe up to 30 dpa at 300 °C. Three regions were observed: dislocation loop raft zone, transition zone and dislocation network zone.