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Single- and dual-beam in situ irradiations of high-purity iron in a transmission electron microscope: Effects of heavy ion irradiation and helium injection

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Abstract

In order to study the effects of 14 MeV neutron irradiation on materials used in the first walls of future fusion reactors, high-purity iron was ion-irradiated with and without helium in the JANNuS facility. Thin foils of high-purity iron were dual-beam irradiated in situ in a transmission electron microscope using 1 MeV Fe^+ and 15 keV He^+ ions. Several important results regarding dislocation loops and helium bubbles were obtained. For example, it was demonstrated that dislocation loops with $a_0 \langle 010 \rangle$ type Burgers vectors are glissile and can move and eliminate at the surface of the thin foil at 500 °C. A comparison of irradiations with and without helium showed that helium atoms reduce the mobility of dislocation loops in pure iron irradiated at 500 °C. Also, we demonstrated that the heterogeneous formation of bubbles inside dislocation loops found previously is also present for helium implantation rates of ~80 atomic parts per million (appm) He/displacements per atom (dpa).

Keywords: Helium; In situ transmission electron microscopy; Iron; Ion irradiation; Dislocation loop

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1. Introduction

The first walls in future fusion reactors will have to withstand high temperatures and 14 MeV neutron irradiation [1]. In the exposed materials, these high-energy neutrons will simultaneously cause displacement cascades and transmutation reactions. The transmutation reactions will produce large quantities of helium, and studies have shown that helium may have an effect on the mechanical properties and swelling of metals [2–4].

Several theoretical and experimental studies [5–11] have recently been conducted in order to understand the effect of helium on the microstructural evolution of irradiated high-purity iron. Iron is of particular interest, since it is the basic material for all ferritic–martensitic steels, which will probably be used as structural materials in the first walls of future fusion reactors. A thorough understanding of the evolution of iron under irradiation with simultaneous production of helium will allow us to comprehend the mechanisms at play in more complex steels.

For decades, researchers have been simulating the effect of 14 MeV neutrons on materials by ion irradiation experiments [12]. This is practical, since high doses can be reached quicker than in a reactor, but also necessary, since 14 MeV neutrons are absent from fission reactor neutron spectra [13,14] and the International Fusion Materials Irradiation Facility (IFMIF) [15] is not yet built. Typically, self-ion irradiations are used to simulate the displacement cascades and helium ions are directly implanted to simulate the products of transmutation reactions. Many studies [16–19] have shown that pre-implantation of helium ions

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before the self-ion irradiation does not produce the same microstructure as simultaneous ion irradiation (also called co-implantation or dual-beam irradiation). Therefore it is necessary to build complex irradiation facilities with two or three coupled ion accelerators, in order to correctly simulate irradiation of materials in a fusion reactor. At JANNuS Orsay (CSNSM), two ion accelerators are connected to an in situ transmission electron microscope (TEM), in order to characterize the evolution of materials under dual-beam irradiations. We report here our recent experimental results obtained at the JANNuS Orsay facility, which has allowed us to demonstrate some very interesting effects of helium on the microstructural evolution of pure iron under dual-beam (Fe^+ and He^+ ions) irradiation. We have also demonstrated important features of the microstructural evolution of pure iron under heavy-ion irradiation without helium, a subject that has also been of great interest in the past years [20–24].

2. Experimental procedure

The high-purity iron used for these experiments was supplied by the European Fusion Development Agreement (EFDA) and was fabricated at the Ecole des Mines de Saint-Etienne. The impurity concentrations are given in Table 1. The average grain size in the as-received material was ~180 μm, and the dislocation density was $\sim 10^{12}\ m^{-2}$. Slices 1 mm thick were cut from the as-received bars. These slices were then polished mechanically to ~90 μm. Discs with a diameter of 3 mm were then punched out from the slices. Finally, TEM samples were electropolished in a Tenupol 5 with a 5% perchloric acid–95% methanol electrolyte at −40 °C in order to produce thin foils suitable for TEM investigations.

The irradiations and observations were performed in a FEI TECNAI G^2 20 Twin, at JANNuS Orsay [25]. All irradiations were performed at 500 °C. For the helium implantation, 15 keV He^+ ions were used, and the damage cascades were created with 1 MeV Fe^+ ions. The He^+ beam struck the sample surface with an incidence of 45°, and the Fe^+ beam struck the sample surface with an incidence of 61°. We performed three irradiations to 0.81 displacements per atom (dpa) with a damage rate of $3.0 \times 10^{-4}\ dpa\ s^{-1}$, each with a different helium implantation rate. The irradiation conditions for these irradiations are given in Table 2. During the single-beam irradiation, the microstructure was filmed using a GATAN ES500W Erlangshen CCD camera. The size of the captured image was 640 × 480 pixels, and an image is saved every 30 ms.

Table 1
Impurity content in the high-purity iron specimens.

Element	Wt. ppm
C	4
S	2
O	4
N	1
P	<5
Cr	<2

During dual-beam irradiations, it was not possible to continuously film the evolution of the microstructure, because the 15 keV He^+ beam would be deviated if the objective lens was turned on. One of our goals was to discover the effect of helium on the mobility of dislocation loops in pure iron. In order to do this, we performed a second batch of irradiations. These irradiations contained two steps: first we realized a dual-beam irradiation to 0.26 dpa with the objective lens of the microscope turned off, then we turned off the He^+ beam and turned on the objective lens as well as the electron beam, and filmed the microstructure under single-beam irradiation for 3 min and 30 s, up to 0.32 dpa. This allowed us to characterize the mobility of dislocation loops in a quasi-dual-beam condition. The irradiation conditions are indicated in Table 3.

All damage levels and helium implantation rates were calculated using stopping and range of ions in matter (SRIM) [26] (displacement energy of 40 eV) and the indicated values correspond to the average calculated between 0 and 160 nm, since this is the typical thickness of the analyzed zones. An example is given in Fig. 1.

During and after irradiation, we observed and analyzed dislocation loops and three-dimensional (3-D) objects in bright field conditions. The use of the weak-beam dark field technique was not advantageous due to the quality of the surface of specimens. It was not determined whether the 3-D objects were bubbles or cavities (i.e. we did not attempt to determine the helium content inside them), but for clarity we shall call them bubbles. TEM foil thicknesses were determined by counting the thickness fringes at the Bragg condition.

3. Experimental results

3.1. Single-beam irradiation of pure iron

During in situ irradiations, some phenomena are only detectable by direct observation of the microstructure: nucleations of point defect clusters, movements of dislocation loops and dislocation lines, interactions between dislocation loops and dislocation lines, etc. These are the kinetic effects that will be presented in the first section. Then the microstructural phenomena that do not necessitate continuous observation will be presented.

3.1.1. Kinetic effects

For an irradiation of iron at 500 °C with a damage rate of $3.0 \times 10^{-3}\ dpa\ s^{-1}$, the first point defect clusters appear at a dose close to 0.04 dpa. These point defect clusters rapidly become resolvable interstitial dislocation loops. Many of the dislocation loops become mobile. The movements are intermittent, and eventually end by eliminations at the surface. In most cases, first a very slow movement of the loop is observed, then the loop glides faster and faster, and finally it disappears. Fig. 2 shows the glide of a loop

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