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Obstacle strength of binary junction due to dislocation dipole formation: An in-situ transmission electron microscopy study

S.M. Hafez Haghighat [a, b, *], R. Schäublin [a, 1]

[a] Ecole Polytechnique Fédérale de Lausanne (EPFL), Centre de Recherches en Physique des Plasmas, Association Euratom-Confédération Suisse, CH 5232 Villigen PSI, Switzerland
[b] Max-Planck-Institut für Eisenforschung, Max-Planck-Str. 1, 40237 Düsseldorf, Germany

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ABSTRACT

We report the experimental observation of the ½<111> edge dislocation dipole formation and annihilation in ultra-high purity Fe using transmission electron microscopy (TEM) in-situ straining. The observation is confirmed by TEM image simulations. The edge dipole is formed by the interaction of a moving screw dislocation with an obstacle of dislocation character. It results from the glide of the two arms of the dislocation on two different glide planes, which stabilizes the dipole that is closed by a jog. The dipole is later released from the obstacle and disappears, presumably by gliding of the dipole's edge segments along their Burgers vector and freeing the mobile screw dislocation from the jog. This mechanism leads to enhanced obstacle strength of the immobile dislocation well above Orowan critical stress, promoting forest strength.

1. Introduction

Ferritic-Martensitic steels are among the main candidate materials for structural components of future fusion reactors, where they are exposed to high energy particles irradiation [1–4]. This results in the production of irradiation induced defects that degrade mechanical properties by obstacle hardening mechanism. The study of the interaction mechanism between moving dislocations and nanometric defects in the base phase, bcc-Fe, of these materials allows shedding light on the mechanisms leading to mechanical degradation.

Studies of the deformation process of bcc-Fe using in-situ transmission electron microscopy (TEM) show that at low temperatures the ½ a_0 <111> edge dislocations has higher mobility than screw dislocations leading to the microstructure consisting of screw dislocations. Screw dislocations thus soon control the deformation of the material [5–8]. A gliding screw dislocation may be stopped at an obstacle, e.g. another dislocation, and upon interaction bow out and finally escape from the obstacle [9,10].

One of the dislocation–obstacle interaction mechanisms, which are observed using molecular dynamics (MD) simulation, is the dislocation dipole formation when the dislocation is pinned at the obstacle [11–15]. MD simulations performed at temperatures between 10 and 300 K have shown that the interaction of an edge dislocation with an immobile screw dislocation, both having ½ a_0 <111> Burgers vector, leads to the formation of binary junction. As a consequence a screw dipole may form along the <111> direction before the junction destruction, when the mobile edge dislocation is free to move further. The length of the dipole increases with decreasing temperature, reaching about 10 nm at 10 K [16]. It appears from the MD simulations that the binary junction induces a comparable hardening to that of nanometric Cr precipitate [16]. Actually, the ½ a_0 <111> screw dipole formation was also observed in the interaction of an edge dislocation with a nanometric dislocation loop [13,17,18], a spherical cavity as void or He bubble [15,19], performed under constant strain rate at finite temperatures. It appears that for the annihilation of this dipole, one of the screw arms must first cross slip, and then glide towards the other arm, resulting in the release of the dislocation [12].

However, there is no experimental evidence of the formation of the ½ a_0 <111> screw dislocation dipole in bcc structures, which might be due to its nanometric size and short life time, making its observation in TEM non-trivial. The edge dislocation dipole conversely has experimentally been observed for e.g. in Fe–Si

* Corresponding author. Max-Planck-Institut für Eisenforschung, Max-Planck-Str. 1, 40237 Düsseldorf, Germany.
E-mail address: m.hafez@mpie.de (S.M. Hafez Haghighat).
[1] Present address: Laboratory of Metal Physics and Technology, Department of Materials, ETH Zurich, 8093 Zurich, Switzerland.

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crystal [9,20]. It was proposed that the edge dipole is formed by the creation of an immobile edge jog on the screw dislocation, where the glide plane of the jog differs from that of the screw dislocation [21]. At a specific jog size the two arms of the moving screw dislocation may pull an edge dislocation dipole along <112>, which later can leave a debris loop behind by a pinch off mechanism [22]. In this paper we show using TEM in-situ straining observation in ultra-high purity Fe at room temperature that another mechanism may also govern the release process of the ½ a_0 <111> screw dislocation impeded by an edge dipole resulting from the interaction with an obstacle. This enhances the dislocation obstacle strength when it is compared to that of the binary junction zipping and destruction without forming a dislocation dipole. Results are confirmed by TEM image simulation of the dislocations' contrast. The mechanism of the dipole formation and annihilation is discussed.

2. In-situ TEM straining method

In-situ straining TEM tests were carried out with ultra-high pure (UHP) Fe designed by EFDA and produced at the Ecole Nationale Superieure des Mines, Saint-Etienne, France. The impurity content is less than 20 ppm. A new technique is used to reduce the specimen size for the in-situ TEM sample, in order to minimize the beam misalignment in the TEM [10] due to the ferromagnetism of Fe. The sample is about 100 μm thick, 11.5 mm long and 2.5 mm wide. It is prepared using three separate parts, namely one necked 3 mm disk of UHP Fe and two stainless steel rectangular parts to hold it in the center. The width of the UHP Fe part is about 1 mm. It is then mechanically polished to a thickness of about 100 μm or less. The parts are then joined by spot welding. A hole is punched at each end of the sample for the gripping by screws. The composite specimen is then jet-electropolished using TENUPOL 5 unit from Struers® at about −10 °C and 20 V in a solution of 10 vol.% perchloric acid in ethanol to create in the center of the specimen a hole with an edge transparent to electrons [10].

The specimen is mounted in a straining low temperature single tilt sample holder from GATAN®. Tests were performed in a JEOL 2010, equipped with a LaB_6 gun and operated at 200 kV. Observations are conducted in bright field condition, close to a Bragg condition, and at room temperature. Stepwise straining was applied instead of continuous straining to avoid rapid deformation and rupture of the sample. Dislocation glide, if any, is recorded in between the straining steps. Acquisition is performed with the 11 Mpixels CCD Orius camera of GATAN® at full frame, providing a frame rate of about 2 images per second. The results reported here have been captured in a single grain, whose normal is close to [311].

3. Dislocation analysis

It should be noted that contrast experiment for determining dislocations' Burgers vector b is not trivial in such an in-situ straining experiment. This is due on the one hand to the sample holder single tilt character, which severely limits the accessibility to the various needed diffraction vectors g, and on the other hand to the dynamical character of the experiment, as the investigated event lasts seconds or at most minutes, which doesn't give enough time for a proper g·b analysis. Nevertheless, in order to extract a maximum of data, selected area diffraction patterns (SADP) are taken on the fly during the experiment. They are then used to infer the crystallographic directions of the observed dislocation line directions. This is done by calculating the stereographic projection corresponding to the experimental SADP of all possible directions of the type <111> and <112> in the bcc structure which are relevant to dislocation configurations, using MatLab® software. By comparing these projections to the dislocation directions in the experimental pictures, one can carefully eliminate possible directions and infer the actual dislocation directions, with a precision of ±5°. This is possible thank to the scarcity of possible directions. TEM image simulation of the dislocation contrasts were then achieved, using CUFOUR software that is based on many beam calculation [23], in order to critically analyze by image matching technique the conclusions deduced by the crystallographic direction analyses of the dislocations' character.

4. Results and discussion

Fig. 1 shows the formation and annihilation process of a dislocation dipole due to the interaction of a moving dislocation with an immobile one. More in detail, Fig. 1a shows the dislocation impinging on the immobile dislocation together with the relevant directions in the stereographic projection. Since the moving dislocation speed is high for the available time resolution of the camera, it appears blurred in this image. Dislocations in this region

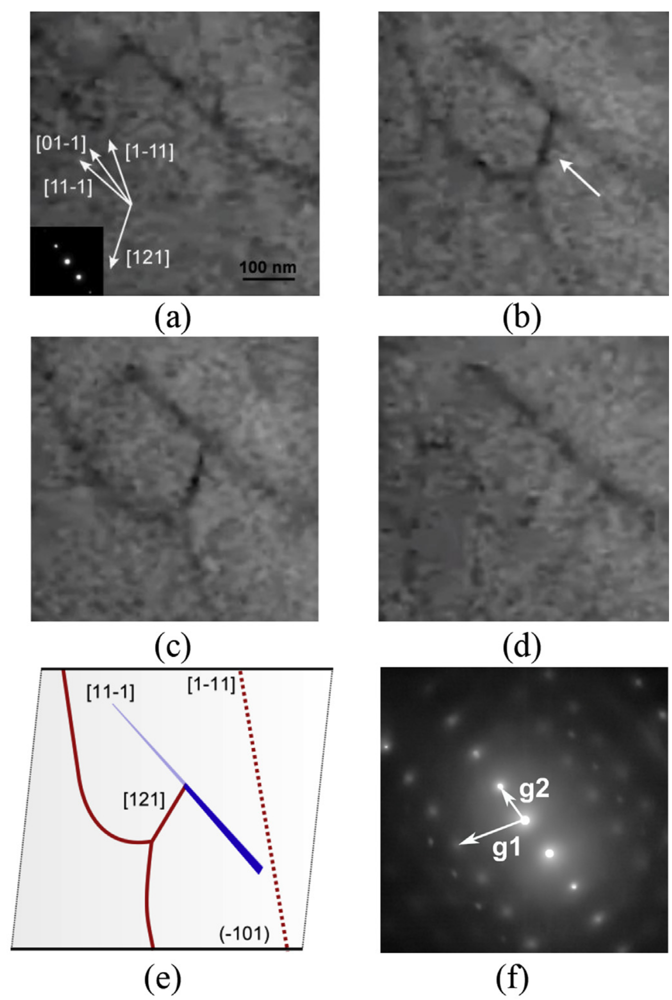

Fig. 1. TEM in-situ straining at room temperature of UHP Fe, bright field images with g = (01-1) and beam direction [311]. (a) The interaction of a moving ½[1-11] screw dislocation with an immobile screw dislocation laid along [11-1], leading to (b–c) the formation of an edge dislocation dipole along [121] (shown by arrow) and (d) the release of the moving dislocation from the immobile screw one. This interaction process takes place within about 2 s. (e) Schematic of the dislocations alignment and interaction configuration on the (−101) glide plane. (f) SADP taken during the in-situ observation with g1/g2 = 1.70 implies that the observation direction is along the [311] zone axis of bcc structure.