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Effect of pre-existing dislocations on the formation of dislocation loops: Pure magnesium under electron irradiation

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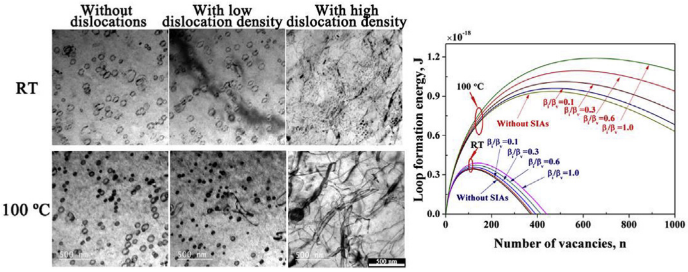
GRAPHICAL ABSTRACT



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ABSTRACT

Irradiation growth and creep are crucially dependent on the movement of dislocations under irradiation. However, despite being amongst the most commonly existing prior defects in polycrystals, the evolution of pre-existing dislocations and their impact on the formation of dislocation loops during irradiation have been relatively rarely investigated. In this study, the evolution of pre-existing dislocations and their impact on the formation of dislocation loops were investigated in magnesium by electron irradiation at different temperatures. The pre-existing dislocations have a profound influence on the loop size, density and Burgers vector of irradiation loops, and this influence is dependent on both the density of the pre-existing dislocations and irradiation temperature. Dislocation climb and glide were observed, but a significant change in pre-existing dislocation density was not seen.

1. Introduction

Understanding of the microstructure evolution during irradiation is crucially important for designing nuclear materials that are able to sustain extreme irradiation doses [1–3]. Large numbers of self-interstitial atoms (SIAs) and vacancies are created by high energy particles during irradiation [4,5] and these SIAs and vacancies aggregate to form interstitial/vacancy loops [6,7], stacking fault tetrahedrals (SFTs) [7,8], and voids [9]. Among these defects, the dislocation loops are perhaps the most frequently observed and found to be closely related to irradiation hardening [10,11], growth [5,12] and changes in corrosion resistance [13–15]. Therefore, study of the factors that might affect the nucleation and growth of dislocation loops is vital.

Previous research has mainly focused on the grain interior microstructure [16,17], and the effect of irradiation flux and fluence [7,18], and chemistry [19,20] on the formation of irradiation induced dislocation loops. Despite being amongst the most commonly existing prior defects in polycrystals, the evolution of

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pre-existing dislocations and their impact on the formation of dislocation loops during irradiation have been relatively rarely investigated. This is because 1) grain to grain dislocation density variation requires an in-situ characterization of the dislocation and dislocation loop evolution during irradiation [21], and there are only limited locations in this world that allow the in-situ observation of defect formation in high threshold energy alloys, like Fe, W and Zr; 2) the contrast from pre-existing dislocations exacerbates the difficulty in characterization of dislocation loops [22] as most dislocation loops generated by fast heavy ions are small [7,23]. Data from reactors has shown that the movement of dislocations and alteration of loop formation behavior due to those dislocations can influence irradiation creep [24–26] and growth [27] of the material. For instance, higher dislocation densities might accelerate irradiation growth rate in Zircaloys −2 and −4 alloys because of the formation <c> type vacancy loops [27,28]. There are several possibilities that contribute to the existence of dislocations in reactor materials, 1) not carrying out a full recrystallization of the prior (fabrication) deformation steps; most in-service reactor materials are manufactured by hot deformation followed by a relatively low temperature annealing, which is unlikely to remove the majority of the dislocations [29]; 2) in-reactor deformation; the load from fuel, and light or heavy water itself can cause deformation of the material and introduce dislocations.

In the present study, we choose pure magnesium with different starting dislocation densities to study the evolution of dislocations and their influence on the dislocation loop formation during electron irradiation. Mg has a low threshold energy, around 12 eV and dislocation loops can be generated under Transmission Electron Microscopy (TEM) operation voltage. Therefore, it is an ideal material for performing in-situ characterization of dislocation evolution and dislocation loop formation.

2. Experimental

2.1. Sample preparation

The material used in this study was commercial purity magnesium (99.99%). The samples were cut and ground with a series of sand papers, and then TEM samples prepared by electropolishing in a Struers Tenupol-5 electro-polisher with an electrolyte of 10% perchloric and 90% methanol at −40 °C.

2.2. In-situ electron irradiation and characterization

Regions without dislocations, with low density dislocations and with high density dislocations in TEM thin foils were selected for electron irradiation at room temperature (RT), 100 °C and 150 °C. The in-situ electron irradiation was carried out in a FEI Tecnai Osiris S/TEM operating at 200 kV and the whole irradiation process was recorded by a screen capture software. To avoid the foil orientation effect during irradiation [30], the beam direction of the incoming electrons was constantly kept close to $[11\bar{2}3]$ zone for all the irradiation experiments. For irradiation at 100 °C and 150 °C, the TEM samples were first heated up to the desired temperature. After the sample temperature was fully stabilized, the in-situ irradiation was performed. The irradiation dose in terms of displacement per atom (dpa) was calculated from the TEM screen current. The irradiation dose in terms of displacement per atom (dpa) was calculated by Ref. [4]:

$$\mathrm{dpa} = \mathrm{D}\sigma_\mathrm{d}$$

Where D is the electron fluence and σ_d is the displacement cross-section for electron radiation. σ_d can be expressed by the nuclear collision between electrons and target atoms by McKinley and Feshbach theory [31].

$$\sigma_d = (0.2495\ barn)Z^2\left(\frac{1}{\beta^4\gamma^2}\right)\left\{\left(\frac{E_m}{E_d}-1\right)-\beta^2 ln\frac{E_m}{E_d}\right.$$
$$\left.+\pi\alpha\beta\left\{2\left[\left(\frac{E_m}{E_d}\right)^{\frac{1}{2}}-1\right]-ln\frac{E_m}{E_d}\right\}\right\} \tag{1}$$

in which $E_m = 2\frac{m}{M}\ \frac{(E+2mc^2)E}{mc^2}$, and $\gamma = (1-\beta^2)^{-\frac{1}{2}}$.

In the above expression, $\mathrm{E_m}$ is the maximum energy transferred to target nucleus; $\mathrm{E_d}$ is the displacement threshold energy; Z is the atomic number; m is the mass of electron; M is the mass of target nucleus; $\alpha = \mathrm{Z}/137$ ($\mathrm{Z} < 40$); $\beta = \mathrm{v/c}$; v is the speed of the electron and c is the speed of light in vacuum.

For 200 kV TEM operating voltage and a threshold displacement energy E_d of 11 eV for the $[11\bar{2}3]$ direction in Mg [32], the relationship between irradiation dose rate and fluence is listed in Table 1. The screen current in this study is 20 nA; therefore, the irradiation dose rate is 1.95×10^{-5} dpa/s.

2.3. TEM sample thickness measurement

The foil thickness of the irradiation regions was measured by electron energy-loss spectra (EELS) using the log-ratio formula [33], $t = \lambda\ln(I_t/I_0)$, where λ is the total inelastic mean free path ($\lambda = 150$ nm for Mg), I_t and I_0 are the total and zero-loss areas under EELS. For all the irradiation conditions, the foil thicknesses were measured to be about 80 nm.

3. Results

The Burgers vector of the pre-existing dislocations were determined by the invisibility criteria. All the dislocations were <a> type with Burgers vectors of $\frac{1}{3}[1\bar{2}10]$, $\frac{1}{3}[11\bar{2}0]$ or $\frac{1}{3}[\bar{2}110]$ on the basal plane. To systematically study the effect of pre-existing dislocations on the formation of dislocation loops, experiments at three different irradiation temperatures (RT, 100 °C and 150 °C) and with different dislocation densities (no dislocations, low dislocation density (~$4.0\times\ 10^{12}\ m^{-2}$) and high dislocation density (~$2.0\times 10^{15}\ m^{-2}$)) were carried out and the loop size, density, nature and change of pre-existing dislocations were determined, respectively.

3.1. Irradiation at RT

3.1.1. General observation

Fig. 1 shows the damage microstructure with different levels of dislocation density at RT. All the micrographs were recorded under two beam dynamical dark field (DF, Fig. 1a and b) or bright field (BF, Fig. 1c) conditions using a constant diffraction vector $g = (10\bar{1}\bar{1})$ at the zone axis of $z = [11\bar{2}3]$ (see Supplementary Videos 1, 2, and 3 for regions without dislocations, with low dislocation density and high dislocation density, respectively, 64 times normal speed). In all cases, the formation and evolution of irradiation induced dislocation loops were observed.

Table 1
The relationship between TEM operating voltage, screen current, irradiation area and irradiation dose rate.

Voltage, kV	Current, A	Area, m^2	Flux, e m^{-2}s^{-1}	Dose rate, dpa/s
200	1.0×10^{-9}	3.5×10^{-12}	1.8×10^{21}	9.6×10^{-7}