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Comparative study of He bubble formation in nanostructured reduced activation steel and its coarsen-grained counterpart

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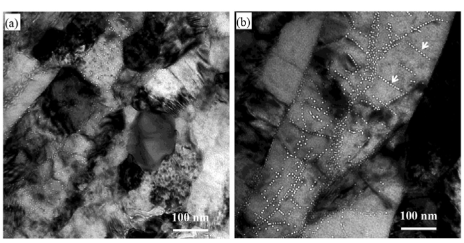
GRAPHICAL ABSTRACT

TEM morphologies of the helium bubbles after irradiation (a) in nanostructured sample (b) in coarsen-grained sample.



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ABSTRACT

High temperature (550 °C) He ions irradiation was performed on nanostructured (NS) and coarsen-grained (CG) reduced activation steel to investigate the effects of GBs/interfaces on the formation of bubbles during irradiation. Experimental results showed that He bubbles were preferentially trapped at dislocations and/or grain boundaries (GBs) for both of the samples. Void denuded zones (VDZs) were observed in the CG samples, while VDZs near GBs were unobvious in NS sample. However, both the average bubble size and the bubble density in peak damage region of the CG sample were significantly larger than that observed in the NS sample, which indicated that GBs play an important role during the irradiation, and the NS steel had better irradiation resistance than its CG counterpart.

1. Introduction

It is well known that grain boundaries (GBs) or precipitate-matrix interfaces may act as sinks for radiation-produced point defects and their clusters [1]. The number of GBs increases drastically with the decrease of grain size, and thus nanostructured (NS) materials containing large number of GBs may have different irradiation tolerance from that of its coarsen-grained (CG) counterpart [2–4]. *Singh* [5] pointed out that void nucleation and void volume swelling are grain size dependent in fine-grained steels; specifically, by decreasing the grain size under irradiation, the void

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concentration can be lowered, the void nucleation can be delayed and the volume swelling can be reduced. Theoretical analysis also showed that the sink strength of GBs increases with the decrease of grain sizes [6]. Experimental observations show that grain sizes have significant effects on radiation tolerance. For example, NS Mo is reported to have much greater radiation tolerance when the grain size is < 90 nm than their CG counterpart after He ion irradiation [7], and smaller grains lead to lower density of He bubbles in He ion irradiated NS Fe [8]. However, a NS material is found to either enhance or lower the phase-transition resistance during irradiation, leading to either greater or less radiation tolerance compared with its CG counterpart [2,9].

In the past decades, many works have been done to investigate the effects of GBs, dislocations and interfaces on the bubble formation and irradiation tolerance during irradiation [10–13]. Helium entrapment in a nanostructured oxide dispersion-strengthened (ODS) ferritic steel showed that the surfaces of Ti(N,C) precipitates-matrix interfaces, GBs and dislocations are preferential nucleation sites for He bubbles [10]. Helium bubbles with sizes larger than that in the interior of the grains were observed at some of these boundaries in He implanted copper [11]. Both experiment and simulation results showed that equal-sized nanoscale He bubbles formed at the dislocation intersection junctions in face-centered cubic materials [12]. Due to their lower thermal expansion, higher thermal conductivity, higher swelling resistance, and better liquid-metal compatibility than austenitic steels, reduced activation steels are receiving more and more attentions as potential first wall and blanket structural materials for future fusion reactors, and have been extensively investigated for the microstructure response under ion irradiation [14]. It is reported that swelling in tempered martensite is an order of magnitude less than in the ferrite phase [15]. Recently, a comparative assessment of fracture toughness behavior and irradiation study of Fe-Cr alloy showed that nanostructured ferritic alloys have much stronger radiation resistance than tempered martensitic steels, with the feature of finer helium bubbles, lower radiation-induced swelling, lower irradiation creep rate and reduced low temperature embrittlement [16]. However, there is still a lack of comparative investigation about the effect of GBs/interfaces on the bubble formation during high temperature irradiation in NS alloy and its CG counterpart.

Although some experimental results about He implantation and post-irradiation annealing of NS ferritic alloy [17,18], NS iron [8] and NS copper [19] have been reported, some contradictory conclusions or inconsistent conclusions were usually drawn due to the scattering experimental results. For example, *Han* et al. [19] pointed out that the void denuded zones (VDZs) width increased with misorientation angle, and GB sink efficiencies depended on both GB plane orientation and misorientation. However, a recent work from *El-Atwani* et al. [20] about helium bubble formation in NS iron showed that VDZs were found to be independent of grain size, grain orientation, and grain boundary misorientation angle. Hence, a direct comparison between the NS and CG RAFM steels under the same irradiation condition helps to provide insight into the dependence of bubble sizes and densities on GBs.

In the present work, high temperature (550 °C) He-ions irradiation was performed on NS and CG reduced activation steel to investigate the effects of GBs/interfaces on the formation of bubbles during irradiation. The irradiation resistance of NS reduced activation steel were also investigated by comparative study of He bubble sizes and morphologies in the NS sample and its coarsen-grained counterpart under the same irradiation condition.

2. Experiments

The material used in the present investigation was a reduced activation steel with chemical composition (in wt.%): 8.60% Cr, 0.10% C, 1.50% W, 0.55% Mn, 0.29% V, 0.09% Ta and balance Fe. Heat treatment included austenitizing at 1253 K for 45 min, followed by water quenching and then tempering at 1033 K for 90 min. Surface mechanical attrition treatment (SMAT) was applied to produce a NS layer at the surface of the steel. A plate sample (Φ50.0 × 4.0 mm in size) of the tempered steel was submitted to SMAT, with the setup illustrated in Lu's work [21]. The plate sample was treated for 30 min with a vibration frequency of 20 kHz, and the ball size was 5 mm in diameter, which is the same with our previous works [22].

Before He irradiation, the irradiation damage profile was simulated using the software package SRIM-2013 (Stopping and Range of Ions in Matter 2013) in "quick K-P mode" with a threshold energy for Fe of 40 eV, and the He concentration profile was simulated in "full Damage F-C mode" [23]. He^{2+} ions with energy of 440 keV were used in the simulation. The simulation, as shown in Fig. 1, implies the He concentration reaches a peak value of ~3.05 at.% at the depth of 800–900 nm. The peak radiation damage is about 2 dpa at the depth of ~870 nm. The range of effective irradiated region is approximately between 600 nm and 1000 nm underneath the surface.

The He ion irradiation experiment was performed on the 320 kV ECR (electron cyclone resonance) experimental platform in the National Laboratory of Heavy-ion Accelerators in Lanzhou, China. He ions with a kinetic energy of 440 keV were used for the irradiation. The irradiation experiment was conducted at 550 °C with vacuum pressure about 10^{-6} Pa. The scanned beam size was about 24 × 25 mm^2. Both the NS sample and the CG counterpart were irradiated at the same time (under the same condition) with a displacement level of about 2 dpa at the peak damage region. The average beam current was about 5 μA during the irradiation experiment, and the experiment lasted for ~120min. Cross-sectional observation of the treated samples was performed on transmission electron microscopy (TEM) of type JEOL Tecnai F20. The cross-sectional TEM samples were obtained by means of focused ion beam (FIB), which is an advanced analytical tool of sample preparation for studies of nano-scale microstructure of the surface layer.

3. Results and discussion

3.1. General observations

Cross-sectional TEM images of the samples before and after SMAT were shown in Fig. 2. Dislocations, fine precipitates as well as GBs were observed in the sample before SMAT (Fig. 2a), and the grain size was about several micrometers. As can be seen in Fig. 2b, the coarse grains have been broken down to nanostructured grains

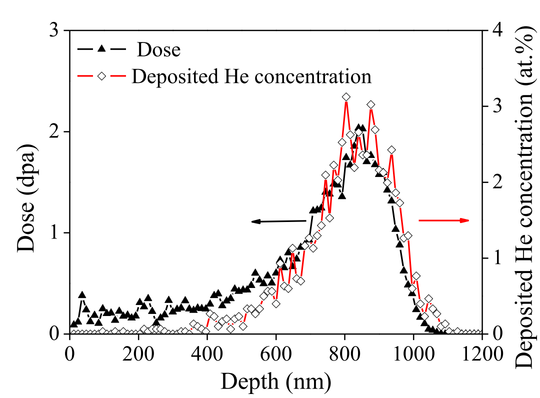

Fig. 1. Dose and deposited He concentration calculated by SRIM software.