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Microstructure evolution during helium irradiation and post-irradiation annealing in a nanostructured reduced activation steel

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ABSTRACT

Severe plastic deformation, intense single-beam He-ion irradiation and post-irradiation annealing were performed on a nanostructured reduced activation ferritic/martensitic (RAFM) steel to investigate the effect of grain boundaries (GBs) on its microstructure evolution during these processes. A surface layer with a depth-dependent nanocrystalline (NC) microstructure was prepared in the RAFM steel using surface mechanical attrition treatment (SMAT). Microstructure evolution after helium (He) irradiation (24.8 dpa) at room temperature and after post-irradiation annealing was investigated using Transmission Electron Microscopy (TEM). Experimental observation shows that GBs play an important role during both the irradiation and the post-irradiation annealing process. He bubbles are preferentially trapped at GBs/interfaces during irradiation and cavities with large sizes are also preferentially trapped at GBs/interfaces during post-irradiation annealing, but void denuded zones (VDZs) near GBs could not be unambiguously observed. Compared with cavities at GBs and within larger grains, cavities with smaller size and higher density are found in smaller grains. The average size of cavities increases rapidly with the increase of time during post-irradiation annealing at 823 K. Cavities with a large size are observed just after annealing for 5 min, although many of the cavities with small sizes also exist after annealing for 240 min. The potential mechanism of cavity growth behavior during post-irradiation annealing is also discussed.

1. Introduction

Helium (He) irradiation behavior in metals has been the subject of extensive research in the past several decades [1,2]. For example, investigation by Lane et al. [3] showed that misfit dislocations at grain boundaries (GBs) are preferred nucleation sites of He bubbles in austenitic steels. Furthermore, Singh et al. [4] confirmed that dislocation intersections, both within and outside of GBs, are preferred bubble nucleation sites in aluminum. Since GBs/interfaces are known to act as potential point defect sinks, and nanocrystalline (NC) material has a large volume fraction of GBs [5], He bubbles can be effectively trapped at GBs in NC materials. Therefore, NC materials are likely to have different radiation damage tolerance from their coarse-grained counterparts [6].

Severe plastic deformation (SPD) technology, such as equal channel angular pressing (ECAP) [7], high-pressure torsion (HPT) [8] and accumulated roll bonding (ARB) [9], has been recognized as an effective way to refine microstructures of various materials. Surface mechanical attrition treatment (SMAT), sometimes called ultrasonic shot peening (USSP), is regarded as one of the most important means of SPD, and it has been employed to obtain various NC materials, including pure metals, alloys and intermetallics [10–12]. Experimental results show that large amounts of GBs/interfaces and dislocations are introduced into the matrix during SMAT [13,14], which are likely to improve the radiation resistance [6].

Due to its excellent properties [15,16], reduced activation ferrite/martensitic (RAFM) steels (Fe-Cr-W-V-Ta) have potential applications as first wall and blanket structural materials in fusion reactors, and a lot of research has been done to investigate the microstructural evolution during He ion irradiations. Studies of Ne-ions irradiated 9Cr ferrite/martensitic steel at high temperatures showed that growth of cavities at GBs, especially at GB junctions, was faster than that within the grain [17]. Microstructural

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observations of high temperature (550 °C) helium irradiated 9Cr-1Mo martensitic steel [18] also showed that voids were preferred to form on prior austenite GBs and other interfaces, such as carbide-matrix interfaces, sub-GBs, lath boundaries and dislocations inside the lath structures, while no preferential nucleation site was observed when the helium implantation was performed at room temperature (RT) [19]. In addition, a typical void distribution near a coincident site lattice boundary was observed in neutron irradiated Fe-15Cr-15Ni steel neutron-irradiated at 749 K to 18 dpa, although void denuded zones (VDZs) were formed near the random GBs [20]. Preferential He bubble formation in carbides of a tempered F82H ferritic-martensitic steel during low temperature He irradiation was observed recently, and this phenomenon was attributed to the diffusing He being trapped in the carbide due to the strong He-C bond, which can lead to the formation of He bubbles with the increase of He concentration in the carbides [21]. However, there is still a lack of research about the effect of GBs in RAFM steels on irradiation induced microstructural evolution.

In the past decade, a number of studies have been devoted to investigating the role of interface structures in the damage resistant properties of NC materials [22–24]. Studies of He ion-irradiations on NC Cu/Nb multilayers show that, in contrast to the copious bubbles contained in the He-implanted pure Cu and Nb, the Cu-Nb interfaces in the Cu/Nb multilayers are responsible for suppressing He bubble formation [22,23]. Nevertheless, when He is preferentially trapped at interfaces, there exists a critical He concentration below which bubbles were not observed [24]. Experimental results of radiation damage in He ion irradiated NC iron (Fe), which was produced by a deposition method using the magnetron sputtering technique, showed that smaller grains lead to lower density of He bubbles, and no voids were observed after He implantation [25]. However, a study about He ion irradiation in NC RAFM steel is still needed to clarify the effect of GBs on irradiation induced microstructural evolution, since the NC microstructure produced by severe plastic deformation is critically different from that produced by magnetron sputtering technique.

It is reported that voids and helium-filled bubbles have different effects on He-induced embrittlement: voids are the main reason for swelling and high temperature embrittlement, while the effect of helium-filled bubbles is comparatively small or, in some cases, even beneficial [26]. The “bubble” and “void” can be described as [27,28]: under some conditions, a helium-filled cavity with diameter less than ~ 10 nm, neither grows nor shrinks, but remains stable as a “He bubble”; while under other conditions, it grows without bounds by absorbing vacancies, and finally results in the formation of a “void”. Therefore, accurate experimental studies of microstructural evolution during ion irradiation are necessary, and this is one of the main objectives of the present work. The stability of a void is not dependent on the presence of internal pressurization from a gaseous species such as helium, and bubbles are usually defined as pressurized cavities, while cavity can be used to refer to either bubbles or voids [29]. Hence, it is necessary to study the average size evolution of cavities during irradiation and post-irradiation annealing.

In the present contribution, He bubble evolution during irradiation and post-irradiation annealing in a quenched fully martensitic NC RAFM steel is systematically studied. Section 2 deals with experimental details about materials, the SMAT process, He ion irradiation, annealing treatment and all the characterization methods. The experimental results, concerning NC structure characterization after SMAT, bubble formation and microstructural evolution under He irradiation, and void formation upon post-irradiation annealing, are presented in Section 3. In Section 4, the mechanisms of microstructural evolution during SMAT, He bubble formation during low temperature implantation, especially the effect of GBs/interfaces during irradiation and post-irradiation annealing, are discussed.

2. Experiments

2.1. Material and SMAT process

The material used in the present investigation was a RAFM steel with chemical composition (in wt%): 0.09% C, 0.49% Mn, 8.75% Cr, 0.84% Ta, 1.58% W, 0.21% V and balance Fe. Heat treatment included austenitizing at 1253 K for 45 min, followed by water quenching. It is noted that the steel, which was quenched after austenitization but not tempered, produced a fully martensitic microstructure, and the microstructure without $M_{23}C_6$ type carbides was used to study the effect of GBs on irradiation-induced microstructures evolution.

The plate sample ($\Phi50 \times 4.0$ mm in size) of the quenched steel was submitted to SMAT. The equipment and procedure of SMAT are discussed in detail in Refs. [30,31]. The ball size used for surface peening was 5 mm in diameter. The vibration frequency of the chamber was 20 kHz, and the plate was treated for 30 min. During SMAT, the strain and strain rate reduced rapidly with the increase of depth, and the strain rate at the sample surface was estimated to as high as 10^2–10^3 s^{-1} [31].

Fig. 1 shows the distinct microstructures of the surface layer and the matrix obtained from cross-sectional SEM observations. No carbides can be obviously seen both in the SMAT layer and in the matrix from the SEM results. Grains with no deformation and grain boundaries can be clearly seen in the matrix (Fig. 1a), and most of the grains are smaller than 10 μm. With increasing depth from the treated surface, gradient nano-submicron-microstructure and obvious deformation bands (Fig. 1b) are found due to the decreasing strain and strain rate. In the topmost layer regions, grains are too small to distinguish their boundaries. Microbands are clearly observed in the inner area. The thickness of the surface deformation layer in Fig. 1b is not uniform (30–46 μm), which can be attributed to the heterogeneous nature of plastic deformation within and between grains [32].

2.2. He-ion irradiation

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 300 keV were used for irradiation. The irradiation experiment was conducted at room temperature with vacuum pressure about 10^{-6} Pa. The scanned beam size was about 17×17 mm^2. The RAFM steel sample was irradiated with a fluence of 3.31×10^{17} ion/cm^2, which corresponds to the estimated displacement levels of 24.8 dpa (displacement per atom) at peak damage region. The average beam current was about 3.83×10^{12} ion/cm^2/s during the irradiation experiment. Displacement damage dose regimes for structural materials (Ferrite/Martensitic steel) in fusion energy systems can be larger than 100 dpa [33], and basic research is still needed to improve understanding of high dose irradiation-induced microstructure evolution. Hence, dose of ~25 dpa was selected in the present work to investigate microstructure evolution in NC RAFM steel under helium ion irradiation.

Fig. 2 shows the dose and deposited He concentration calculated from the stopping and range of ions in matter 2013 (SRIM-2013) software [34]. Materials with the same chemical composition in the experiment and He^{2+} ions with energy of 300 keV were used in the calculation. The peak value of He concentration can be as high as 18.20 at%, and the peak region of the deposited He in the irradiated