Materials Characterization 111 (2016) 1–7

Contents lists available at ScienceDirect

Materials Characterization

journal homepage: www.elsevier.com/locate/matchar

Characterization of microstructure in hydrogen ion irradiated vanadium at room temperature and the microstructural evolution during post-irradiation annealing

Jin Gao, Lijuan Cui, Farong Wan *

University of Science and Technology Beijing, Haidian District, Beijing 100083, China

ARTICLE INFO

Article history:
Received 24 September 2015
Received in revised form 5 November 2015
Accepted 6 November 2015
Available online 10 November 2015

Keywords:
Transmission electron microscopy
Burgers vector
Dislocation loops
Bubbles

ABSTRACT

The microstructure of pure vanadium after hydrogen ion irradiation at room temperature to a fluence of 1×10^{17} ions/cm^2 (and 5×10^{16} ions/cm^2) was investigated by transmission electron microscopy (TEM). Small dislocation loops (black spots) and cavities are formed after the irradiation. The nature and Burgers vector of dislocation loops formed in vanadium was characterized using $\mathbf{g}\cdot\mathbf{b}$ technique and inside–outside method. Interstitial dislocation loops with Burgers vector of 1/2<111> predominantly formed with less than 10% of 1/2<110> type. No <100> type or vacancy type dislocation loop formed. The microstructural evolution during the annealing process was also studied. Density and size of dislocation loops changed sharply when the annealing temperature was lifted up to 450 °C. When the annealing temperature was higher than 500 °C, bubble coalescence occurred with some large hydrogen bubbles formed.

1. Introduction

Vanadium-based alloys are being considered as one of the attractive candidate materials for fusion structural materials due to their excellent mechanical properties at elevated temperatures and low induced radioactivity after neutron irradiation. In addition, they generally show a good resistance for void swelling which is typical for bcc materials [1,2]. Former researches mainly focused on the swelling and mechanical properties of pure vanadium and its alloys after neutron irradiation, exploring the effect of alloying solute atoms [3,4,5,6]. Hydrogen and helium atoms are generated by nuclear transmutation in the fusion environment. These gas atoms play an important role on the evolution of the damage microstructure. Experimental data on microstructure evolution after hydrogen or helium ion irradiation in vanadium is rather limited compared with that in iron.

One of the most profound consequences of the irradiation on the microstructure of materials is the formation of dislocation loops. The nature of dislocation loop, e.g. interstitial or vacancy, as well as the Burgers vector, has a strong impact on the development of the irradiated microstructure due to its bias to interstitials and thus influences the mechanical property of the materials [7]. A general consensus on the nature of dislocation loops in iron and ferritic steels has been achieved. Both simulations [8,9,10] and experimental observations [11,12,13,14] have confirmed the existence of 1/2<111> and <100> loops. However, in other bcc materials, e.g. molybdenum and tungsten, almost predominantly interstitial dislocation loops with a Burgers vector of 1/2<111> have been reported [15].

In our present work, we report our studies on the characterization of dislocation loops in 30 keV hydrogen ion irradiated pure vanadium at room temperature, with dose of 1×10^{17} ions/cm^2 and 5×10^{16} ions/cm^2. After irradiation, the samples were annealed up to 600 °C in TEM. Detailed TEM analysis was used to characterize the nature and Burgers vector of the dislocation loops and the evolution of microstructure.

2. Material and methods

Vanadium samples with purity of 99.9% were mechanically polished on both sides to a thickness of 100 μm and then punched into 3 mm disks. The final TEM specimens were prepared by twin-jet electropolishing in Tenupol-5, using an electrolyte of 20% (volume) H_2SO_4 and 80% methanol solution close to −20 °C, and washed in ethanol.

Hydrogen ions of 30 keV were implanted to the TEM specimens at room temperature to dose of 1×10^{17} ions/cm^2 and 5×10^{16} ions/cm^2. The irradiation experiment was done by static accelerator using electron cyclotron resonance (ECR) ion source in Lanzhou, China. The measured beam current was 5 μA (equals to ion flux of $1.39 * 10^{13}$ cm^{-2} s^{-1}). Irradiation damage was calculated using the software stopping and range of ions in the metal (SRIM-2008) as shown in Fig. 1. In our typical observation region (140–180 nm), the radiation damage equals to 0.39–0.48 dpa and the helium concentration was in the range of 4 at.%–9.4 at.%. Post-irradiation analysis were done at FEI Tecnai F20 working at 200 kV. The nature and Burgers vector of dislocation loops were analyzed by standard $\mathbf{g}\cdot\mathbf{b}$ technique and the inside–

---

* Corresponding author.
E-mail address: wanfr@mater.ustb.edu.cn (F. Wan).

http://dx.doi.org/10.1016/j.matchar.2015.11.006

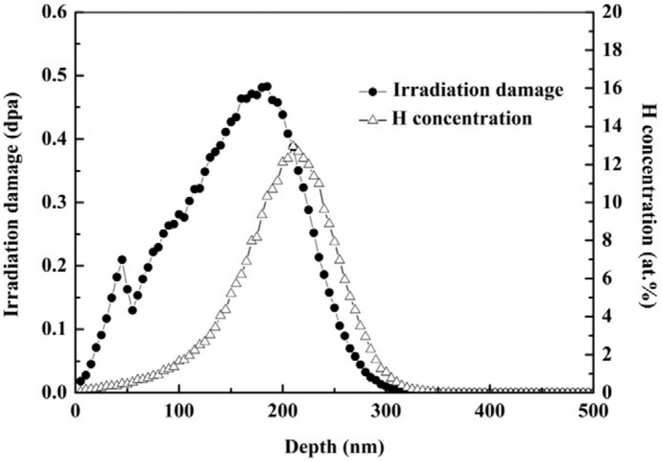

Fig. 1. SRIM calculation of irradiation damage and H concentration profile in vanadium under 30 keV hydrogen ion irradiation.

outside method. It's generally reported that thin film of pure vanadium and vanadium alloys were prone to be oxidized during high temperature annealing. Our experiments in the accelerator or sealed in quartz tube also confirm this. To minimize the oxidization during the annealing process, the post-ion irradiated specimens was heated in-situ in a JEM2010 column using heating holder.

3. Results

3.1. Irradiation damage in vanadium after hydrogen ion irradiation at room temperature

Fig. 2 shows the microstructure (a) before and (b) after hydrogen ion irradiation in pure vanadium at room temperature. After irradiation, the microstructure showed "black spot" contrast, which were dislocation loops with average size of several nanometers. The areal number density of these "black spots" was $7.5 * 10^{14}\ m^{-2}$, which underestimated the dislocation loops due to that some of them were out of contrast. Some large dislocation loops with size up to several tens of nanometers were also observed. No obvious cavities can be observed in Fig. 2. However, in Section 3.3, small bubbles also formed in the vanadium. The nature and Burgers vector of dislocation loops were characterized by TEM in Section 3.2.

3.2. Characterization of the dislocation loops formed in vanadium

3.2.1. Burgers vector of the dislocation loops formed in vanadium

In earlier studies, both <100> and 1/2<111> type dislocation loops were observed in iron and ferritic materials. Whether <100> type dislocation loops formed in other bcc materials remained a field to be resolved. Almost all of the dislocation loops (big black spots) remained visible in both Fig. 3(a) and (b) with vectors **g**(200) and **g**(020) respectively without taking the very small black spots into account. Several other spots were characterized but came to the same conclusion that no <100> type dislocation loops were present in vanadium irradiated by hydrogen ion at room temperature.

Characterization of the dislocation loops present in pure vanadium by hydrogen ion irradiation with energy of 30 keV to a fluence of 1×10^{17} ions/cm^2 at room temperature was performed, as shown systematically in Fig. 4, perceiving that all dislocation loops were of 1/2<111> type. Here, care should be taken that dislocation loops with Burgers vector of 1/2<110> [16] could also form, although the proportion is less than 10%, as evidenced by Fig. 5. Dose difference in Fig. 4 and Fig. 5 should have minor influence on the Burgers vector. As a matter of fact, compared with Fig. 4(a) and (e), dislocation loops with Burgers vector of 1/2[110] were also observed in the higher dose case. To be clear and simple, characterization of 1/2<111> and 1/2<110> were shown in Fig. 4 and Fig. 5, respectively. Contrast of dislocation loops expected here based on **g·b** criterion was listed in Table 1. Dislocation loops labeled as A, B and C were visible in Fig. 4(b) but out of contrast in Fig. 4(a). Thus, they were dislocation loops with Burgers vector of ±1/2[111]. For dislocation loops labeled as D, E and F, their Burgers vectors were characterized as ±1/2[1-11] and ±1/2[-111] respectively. The fourth type dislocation loops were not identified due to the low quality of the micrograph (g). The nature of the dislocation loops can be characterized using inside–outside contrast experiments, as shown in Section 3.2.2. For dislocation loops marked as F in Fig. 4(e), they showed the edge-on character and located in the (1–12) planes compared with Fig. 4(d). Therefore, the dislocation loops could be classified as 1/2<111>{112} interstitial loops together with analysis in Section 3.2.2.

3.2.2. Characterization of the nature of dislocation loops in vanadium after hydrogen ion irradiation at room temperature

As shown in Fig. 6, nature characterization of the dislocation loops induced in vanadium was performed. All of the four types of 1/2<111> dislocation loops were characterized here. To be simple, take

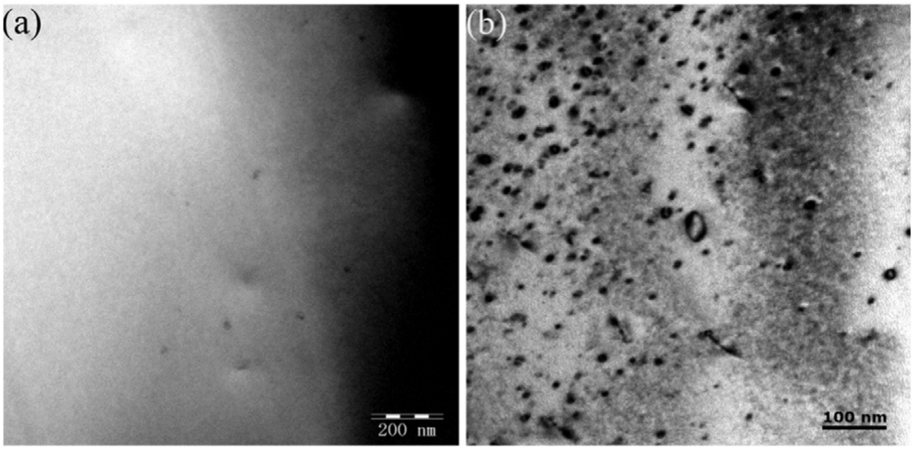

Fig. 2. TEM micrographs (a) before and (b) after hydrogen ion irradiation in pure vanadium.