Evolution of Helium Bubbles in ODS Steel

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1. Introduction

Due to the superior thermal creep properties and the high resistance to neutron irradiation, oxide dispersion strengthened (ODS) steels are considered as potential candidate alloys for application in advanced nuclear or fusion reactor [1, 2]. The accumulation of helium after neutron irradiation has a substantial influence on the mechanical properties of materials, e.g. the intergranular embrittlement via helium bubble growth at grain-boundaries at temperatures higher than $0.33T_m$ (T_m is the melting point of metals), the void swelling, blistering of metal surface, and the rise of the ductile-brittle transition temperature (DBTT) of bcc structure metals [3]. Therefore, the understanding of the helium behavior in ODS steels is important prior to its application in the nuclear energy field. Although a substantial studies have been conducted to investigate the effects and micro-mechanism of helium in ODS steels [2, 4-8], the nucleation and growth of Helium in metals is not yet fully understood.

In this work, we used low energy helium ions to be implanted into a commercial ODS alloy (MA956[TM]) sample, and investigated the evolution of the helium bubbles with fluences at room temperature using a TEM (Transmission Electron Microscope).

2. Experiment

The sample used in this study is a 20% Cr Fe-based commercial ODS alloy (MA956). Before the samples were implanted, those with thickness of 0.5 mm were mechanically ground with SiC abrasive papers (#800-2400) and then with a diamond suspension (3 μm). The samples were subsequently electrolytically polished in a mixture solution of 5 % perchloric acid and 95 % ethanol. Helium ions with energy of 30 keV were implanted at room temperature into the samples with the fluence of 3.0×10^{14}, 3.0×10^{15}, 3.0×10^{16} and 1.0×10^{17} cm^{-2}, respectively. The helium atom concentration (at. %) and the damage (dap: displacement per atom) were 0.03 at. %/0.02 dpa, 0.28 at. %/ 0.2 dpa, 2.76 at. %/1.6 dpa and 9.25 at. %/ 5.4 dpa for the four successive fluences, respectively. Cross-sectional TEM samples were prepared by using a precision ion polishing system (Gatan model 691), and were observed by using a FE-TEM(JEOL-2100F).

3. Results

The TEM results showed that the microstructure of the MA 956 alloy specimen is consist of large grains containing nano-sized oxides particles with a number density of about 8.0×10^{14} cm^{-3}. Energy dispersive spectroscopy (EDS) measurement shows a significant enrichment of yttrium (Y) and aluminum (Al) in the oxide particles. It can be seen that there is no damage or helium bubbles existing in the ions projected range. A large amount of oxide particles with diameter around 10 nm and a few with diameter around 25 nm are observed in the matrix.

The TEM results for the samples with fluences of 3.0×10^{15}、3.0×10^{16} and 1.0×10^{17} cm^{-2} are shown in figures 1. It was found the existence of damage and helium bubbles in all these samples, and the helium bubble size increases with the increase of ions fluence. The mean diameters are 0.9, 1.8 and 3.9 nm for the samples with fluences of 3.0×10^{15}、3.0×10^{16} and 1.0×10^{17} cm^{-2}, respectively. The bubbles exist not only in the matrix and at the interface between the matrix and the oxide particles, but also in the oxide particles. The size of the bubbles in the oxide particles and at the interfaces is evidently larger than that in the matrix (Fig. 3), which indicates that these interfaces are the effective trapping sites for the helium atoms.

Due to its extremely low solubility (<<1 at.ppm) and the low migration energy via interstitial sites (about 0.15 eV) in metals, helium atoms have strong tendency to be trapped at vacancy type defects and form He_nV_m (n and m are numbers of helium atoms and vacancies in the helium bubble, respectively) clusters [3]. Once inside a vacancy or vacancy clusters, helium can be deeply trapped therein, decreasing its mobility and enhancing the aggregation of further vacancies and helium atoms around [6]. The binding energy of Helium atoms to He_nV_m clusters is independent of the cluster size but dependent on the helium to vacancy ratio n/m, which is found to increase with more helium. When the ratio is high, the pressure in the He_nV_m is so large that it can create vacancies due to their low formation energy via emission of interstitials or interstitial clusters, which decreases the n/m ratio and makes the helium clusters exist in more stable state [5, 6, 9].

In addition to the helium bubbles, some defects clusters (black dots in the micrographs) are also observed near the end region of the ion range, which probably come from the agglomerate of excessive

interstitials. Both the size and density of these defects clusters increase with the increase of fluence.

4. Conclusions

Helium ions with energy of 30 keV were used to implant into a 21Cr commercial ODS steels (MA 956) at four different fluences. The TEM was adopted to investigate the helium bubble size evolution with fluences.
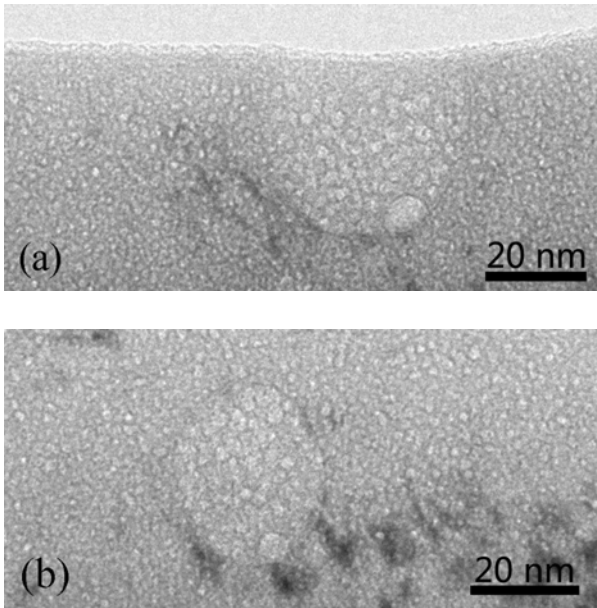

Fig. 3. Helium bubbles in the particles and at the interfaces between the particles and matrix (a) the particle close to surface, (b) the particle far from the surface.

It is found that the helium bubble size increases linearly with the increase of fluence at room temperature in the helium concentration of 0.28-9.25 at. %. The interfaces between the nano-sized oxide particles are effective trapping site for the helium atoms, which make the size of bubbles at the interface and in the oxide particles is larger than that in the matrix.

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