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Synergistic effect of helium and hydrogen for bubble swelling in reduced-activation ferritic/martensitic steel under sequential helium and hydrogen irradiation at different temperatures

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HIGHLIGHTS

- Helium/hydrogen synergistic effect can increase irradiation swelling of RAFM steel.
- Hydrogen can be trapped to the outer surface of helium bubbles.
- Too large a helium bubble can become movable.
- Point defects would become mobile and annihilate at dislocations at high temperature.
- The peak swelling temperature for RAFM steel is 450 °C.

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ABSTRACT

In order to investigate the synergistic effect of helium and hydrogen on swelling in reduced-activation ferritic/martensitic (RAFM) steel, specimens were separately irradiated by single He^+ beam and sequential He^+ and H^+ beams at different temperatures from 250 to 650 °C. Transmission electron microscope observation showed that implantation of hydrogen into the specimens pre-irradiated by helium can result in obvious enhancement of bubble size and swelling rate which can be regarded as a consequence of hydrogen being trapped by helium bubbles. But when temperature increased, Ostwald ripening mechanism would become dominant, besides, too large a bubble could become mobile and swallow many tiny bubbles on their way moving, reducing bubble number density. And these effects were most remarkable at 450 °C which was the peak bubble swelling temperature for RAMF steel. When temperature was high enough, say above 450, point defects would become mobile and annihilate at dislocations or surface. As a consequence, helium could no longer effectively diffuse and clustering in materials and bubble formation was suppressed. When temperature was above 500, helium bubbles would become unstable and decompose or migrate out of surface. Finally no bubble was observed at 650 °C.

1. Introduction

It is the good swelling resistance, mechanical property retention, microstructural stability along with technology maturity that mainly make the reduced-activation ferritic/martensitic (RAFM) steel a hopeful candidate for the first wall structural material of future fusion power reactors [1], which means the structural material must sustain the high energy (up to 14.1 MeV) neutron irradiation [2]. The abundant amount of hydrogen and helium primarily coming from nuclear transmutation reactions induced by energetic neutron irradiation [3] have been considered as a cause of irradiation embrittlement, hardening and swelling [4]. Computer simulations have shown that helium and hydrogen appear to interact synergistically to strongly promote swelling [5], but experimental data which can be provided to show the detailed

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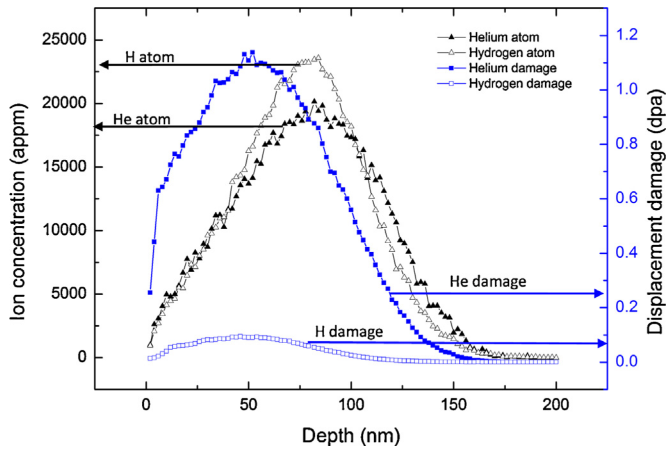

Fig. 1. Depth profile of atoms distribution and damage events for He^+ irradiation (18 keV, 1.6×10^{20} m^{-2}) and H^+ irradiation (10 keV, 8.2×10^{20} m^{-2}).

mechanism of synergistic effect on bubble swelling is insufficient and scattered, leaving many problems unsolved in a proper way. As temperature is one of the most important parameters controlling the number density and bubble size evolution in calculating model [6], systematical research on what kind of role temperature is playing in helium/hydrogen synergistic effect in RAFM steel is quite a necessity, thus this paper focuses on helium and hydrogen synergistic effect on gas bubble evolution in RAFM steel at different temperatures. In order to study the "pure" helium/hydrogen synergistic effect in bubble swelling, only He^+ beam and H^+ beam were used in the experiments in a quite wide temperature range, which is relatively rarely reported in previous studies.

2. Experimental procedure

The RAFM steel used in this experiment has the following chemical composition: 9.09%Cr, 2.34%W, 0.48%Mn, 0.25%V, 0.097%C, 0.038%Si, 0.0074%P, 0.0019%S in weight percent (namely SCRAM steel, provided by Huazhong University of Science and Technology [7]). The SCRAM steel was quenched at 980 °C for 0.5 h and tempered at 760 °C for 2 h; then quenched at 960 °C for 0.5 h and tempered at 740 °C for 2 h, and finally a fully tempered martensite lath structure was obtained. Sheets of size 20 mm × 20 mm × 5 mm were cut from bulk RAFM steel, then mechanically polished to flakes of thickness about 80 μm. Disk specimens of 3 mm diameter were punched out from these flakes and further milled to a thickness of around 30 μm. The final TEM specimens were polished by a twin-jet electro-polisher, using a $5\%HClO_4$–$95\%C_2H_5OH$ polishing solution.

Two sets of specimens were used in the experiments where specimens A, B, C, D, E were included in set-one and specimens a, b, c, d, e were included in set-two.

Specimens of set-one were irradiated by 18 keV He^+ to a dose of 1.64×10^{20} m^{-2} (with a dose rate of 2.16×10^{16} m^{-2} s^{-1}) separately at 250, 350, 450, 550, 650 °C on an ion implanter in the Accelerator Lab of Wuhan University. Specimens of set-two performed the same helium irradiation as set-one and then irradiated by 10 keV H^+ to a dose of 8.2×10^{20} m^{-2} (with a dose rate of $7.74 \times 10^{16} m^{-2}$ s^{-1}) at temperatures 250, 350, 450, 550, 650 °C corresponding to first irradiation temperature. The detailed irradiation conditions are listed in Table 1. As for the swelling rate, it is calculated by $\frac{\Delta V}{V}$ where ΔV is the bubbles volume and V is the total volume of the area we selected to count bubble number with a thickness of 100 nm (determined with the thickness fringes method). The projected depths of 18 keV He^+ and 10 keV H^+ in the SCRAM steel were both around 80 nm (Fig. 1) from the calculation of SRIM2008 using a displacement energy of 40 eV as recommended in ASTME521-89 [8]. After irradiation, these specimens were studied by a JEM-2010HT TEM.

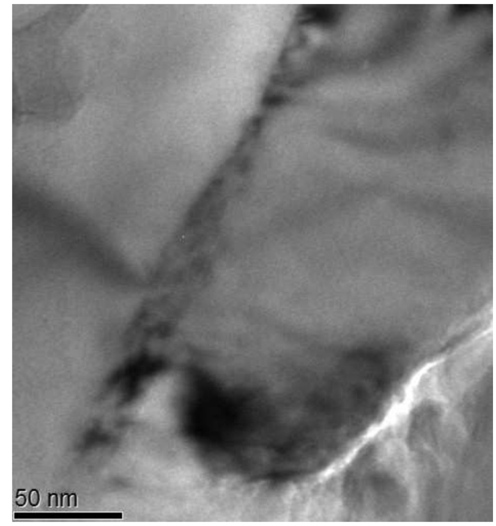

Fig. 2. TEM micrograph of the unirradiated sample.

3. 3.Results and discussion

3.1. Hydrogen helps bubble nucleation at low temperature: 250–50 °C

The unirradiated sample is shown in Fig. 2. Fig. 3(a) and (b) are of the same TEM micrograph for a specimen irradiated by sequential He^+ and H^+ beams at 350 °C in under-focused (defocused) and over-focused imaging conditions. The over-focused and defocused distance was around ±1000 nm. For the irradiated samples, a notable change induced by post-irradiation of H^+ was observed at 250 °C. As can be seen in Fig. 4 for the 250 °C irradiation conditions, no bubble was seen in set-one specimen "A" after single He^+ beam irradiation, but a few bubbles appeared in set-two specimen "a" irradiated by sequential He^+ and H^+ beams. But quite a lot bubbles appeared in 350 °C irradiation condition, for increasing temperature helps bubble nucleation [9]. And bubbles observed in the specimen irradiated by sequential He^+ and H^+ beams at 350 °C were a lot denser than the single He^+ case.

It has been mentioned that 3 keV He^+ will introduce one vacancy for each impact ion in tungsten whose threshold value for the displacement damage is 0.5 keV [10] and about nine vacancies are produced per 30 keV He^+ in tungsten [11]. It is also argued if the energy of the incident ion is higher than the threshold value for the displacement damage in metal, interstitials and vacancies with the same number are formed in the projected range in material [12]. Thus, under the 18 keV He^+ irradiation, almost the same order of vacancy number magnitude was produced compared with the concentration of helium atoms. Binding energy of one helium atom and a vacancy is as large as 4.57 eV and the binding energy of two helium atoms is 1.03 eV [13]. So under very strong attractive interaction, helium-vacancy (He-V) complex clusters [14] together with a little interstitial helium clusters [15] were formed. But at 250 °C thermal migration of He-V complexes and helium clusters were limited and bubble nucleation was suppressed. But their mobility increased with increasing temperature [14] and quite amount of bubbles appeared at 350 °C.

After 10 keV H^+ irradiation, a comparable amount of vacancies was introduced [16]. After hydrogen irradiation, the newly produced vacancies were easily trapped by He-V clusters or helium