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Effect of annealing on Cu precipitates in H ion irradiated Fe–0.6%Cu studied by positron annihilation

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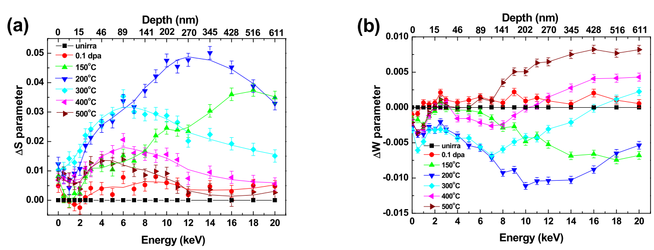
HIGHLIGHTS

- Irradiation led to the formation of the Cu–vacancy complexes.
- Vacancy clusters surrounded by ultrafine Cu precipitates formed.
- Mostly of the vacancy-like defects completely recovered by annealing at 500 °C.
- Defect-free Cu precipitates formed and coarsened at 500 °C.

GRAPHICAL ABSTRACT



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ABSTRACT

Fe–0.6%Cu alloy was irradiated with H ions to 0.1 dpa, and then annealed for 30 min from 150 °C to 500 °C. We focused the evolution of Cu precipitates in irradiated Fe–0.6%Cu alloy after the isochronal annealing from the perspective of positron annihilation. The ΔW parameters after thermal annealing (400 °C and 500 °C) were much larger than that induced by 0.1 dpa H irradiation. Annealing could promote the aggregation of the Cu-vacancy complexes, and form the Cu cluster–vacancies complexes. When the vacancy-like defects recovered around 500 °C, it meant the formation and growing of the defect-free Cu precipitates.

1. Introduction

In our previous report [1], we studied the correlation between Cu precipitates and irradiation defects in Fe–Cu alloys. The Cu precipitate was origin from the formation of the Cu-vacancy complexes, and the coarsening depended on the growth of vacancy-like defects. In Zhang et al. [2], we recently published a paper of our results concerning the effect of annealing on V_mH_n complexes in H ion irradiated Fe–0.3%Cu alloy. We mainly described the interaction between H and vacancy-like defects. However, the ultrafine Cu precipitate is an origin of the irradiation-induced embrittlement, which is considered as one of the menaces to the reactor safety [3]. It has long been known that the small bcc Cu precipitates are initially coherent with the bcc Fe matrix [4,5]. The size and composition of the small Cu precipitates in both model alloys and steels were extensively examined using atom probe [6–9], small angle neutron scattering [6,10,11] and transmission electron microscopy (TEM) [4,6,12,13]. In the present paper, we report a powerful method to examine the evolution of small Cu precipitate

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which uses the positron as a probe. As well known, positron is not only a sensitive probe for vacancy-like defects. What's more, it could be trapped by nanosize embedded particles (for instance, the Cu precipitates of ~1 nm) in materials [3,5]. Cu precipitates are easily formed in Fe–Cu alloy during thermal aging and high-energy particle irradiation. Ion beams are very efficient tools for the simulation of the radiations produced in nuclear reactors. Heavy-ion damage took the form of dislocation loops in pure Fe and A533B steel irradiated with the dose of ~1 dpa at ~300 °C [14,15]. A large number of tiny dislocation loops appeared in pure Fe irradiated with ~1.42 dpa D ion at room temperature [16]. However, once the D ion irradiation fluence decreases to 1×10^{19} D_2^+/m^2 (~0.1 dpa) at room temperature, no obvious dislocation loop formed in the Fe–9Cr alloys [17]. In our previous experiment, the Ni-base alloy C-276 was irradiated by ~120 keV argon ions at room temperature. Compared to the unirradiated sample, there was no obvious change at 0.28 dpa in TEM results. Until to the 0.8 dpa, many tiny black spots appeared in the specimen [18]. For the Cu precipitate in Fe–Cu alloy, there should be important differences between irradiation microstructural development at room temperature and at elevated temperatures. At elevated irradiation temperature of 300 °C, Xu et al. established that ultrafine Cu precipitates are nucleated before the formation of vacancy clusters [19], and such conclusions are probably not applicable to room temperature irradiations. In the present paper, we discuss the difference for Cu precipitates between induced by low dose H ion irradiation at room temperature and by thermal aging from the perspective of positron annihilation. The evolution of Cu precipitates in irradiated Fe–0.6% Cu alloy after the isochronal annealing is investigated by the Doppler broadening (DB) spectra based on a slow positron beam.

2. Experimental details

The model alloy, Fe–0.6%Cu alloy, was also melted from Fe (99.99% purity) and Cu (99.999% purity) in vacuum using a high-frequency induction furnace. The experimental methods and the preparation of the specimen were the same with the previous report [2].

The specimen was irradiated with 100 keV H ions to a dose of 0.1 dpa at room temperature, and the current density was ~2 μA/cm^2. The damage distribution and H atom deposition profiles calculated by SRIM-2008 are shown in Fig. 1, where the displacement energy was 40 eV. The damage rate is about 5.5×10^{-5} dpa/s. The 0.1 dpa irradiated specimen was annealed isochronally for 30 min in a vacuum of 10^{-5} Pa, and the annealing temperatures were at 150 °C, 200 °C, 300 °C, 400 °C and 500 °C, respectively. Positron annihilation DB spectra were performed to characterize the evolution of small Cu precipitates and the vacancy-like defects. S represents the smaller Doppler shift resulting from the annihilation of valence electrons. Thus, compared to a well-annealed sample, the increase in S for the post-irradiated one comes from the annihilation at vacancy-like defects. W comes from the annihilation with Cu 3d electrons, which is used to estimate the density of Cu atoms around positrons when they are annihilated. The presents of vacancy-like defects and Cu precipitates are represented by the relative S and W parameter, i.e., ($\Delta S = S_n - S_{not\ irradiated}$) and ($\Delta W = W_n - W_{not\ irradiated}$), respectively.

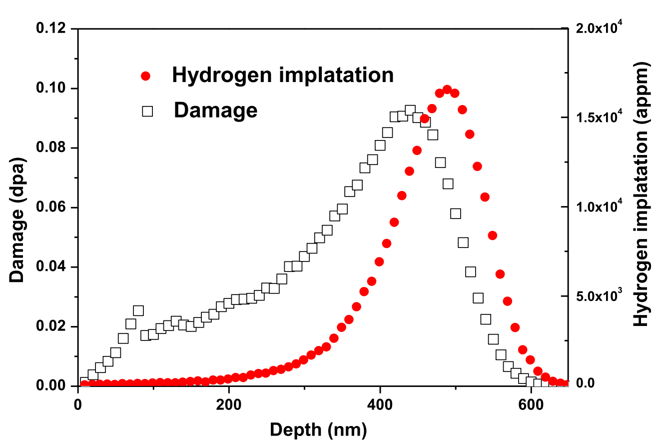

Fig. 1. SRIM calculated damage and deposition profiles for H ion irradiated Fe–0.6%Cu alloy.

3. Results and discussion

The dependence of the ΔS parameter on positron energy for Fe–0.6%Cu alloy irradiated at 0.1 dpa indicates that no expected peak formed in Fig. 2a, which is due to the vacancies were occupied by H atoms. A larger number of H atoms deposited at the damage area, and the V_mH_n complexes were considered to dominate the microstructure in Fe–0.6%Cu alloy after 0.1 dpa irradiation. D atoms were trapped by vacancies, which could decrease the S parameter in Cu ion irradiated W alloy [20]. Similarly, Troev et al. indicated that the lifetime of vacancies decreased after trapping H atoms [21]. Thus, the formation of the V_mH_n complexes would affect the annihilation of positrons with the electrons at vacancy defects.

A predicted evident ΔS peak appeared in the damage region after annealing treatment. Annealing could desorb the H atoms, and residual vacancy defects would increase the ΔS parameter. Compared to the 0.1 dpa specimen, the ΔS parameter increased after annealing at 150 °C, and it continued increasing at 200 °C (see Fig. 2a). However, the decrease of ΔS means that a small quantity of vacancy-like defects began to recovery at 300 °C. Ono et al. reported that most of D atoms were released at ~200 °C from an D ion irradiated Fe–9Cr–2W ferritic alloy [22]. Thus, most of the H atom can easily escape from vacancy-like defects at ~200 °C, but most of the vacancy-like defects still stay in the alloys. The evolution of the vacancy-like defect is not influenced by H atoms at above 200 °C. The H atom desorption indicates that the vacancies and the vacancy clusters were considered to dominate the microstructure after annealing at 200 °C. The rapidly shrinkage of vacancy clusters by dissociating their vacancies at 400 °C facilitated the ΔS decrease (see the magenta line in Fig. 2a). The ΔS parameters continued decreasing during the annealing temperature from 400 °C to 500 °C. Ishizaki et al. pointed out that the thermal decomposition of the vacancy-like defects in Fe induced by H ion irradiation finished completely between 350 °C and 450 °C [23]. For the positron energy range above ~12 keV, as shown in Fig. 2a (wine curve), the S parameter in the post-irradiated specimen after annealing at 500 °C for 0.5 h recovered nearly to the result before irradiation. This means that mostly of the vacancy-like defects were complete disappearance after annealing at 500 °C.

In addition, the dependence of the ΔS parameter on positron energy in Fig. 2a also shows that the peak in ΔS-E curve appeared and moved closer to the surface as the annealing temperature increases. The vacancy-like defects gradually recovered with the increasing annealing because of the vacancy dissolution from the vacancy-like defects. Some of the vacancies would anneal out due to occupation of interstitial atoms. However, according to the vacancy migration mechanism, partly of vacancies would migrate towards to surface region and the opposite direction.

Fig. 2b shows the ΔW-E (ΔW parameter-positron energy) curves for Fe–0.6Cu% alloy irradiated at 0.1 dpa and annealed at different temperatures. The positron is a sensitive tool for detecting the Cu