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Damage behavior in an electron/helium dual-beam irradiated Fe–Cr–Mn(W,V) alloy

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Abstract

Results of dual-beam irradiations with electrons and helium ions indicated that the formation of secondary defects (dislocation loops, dislocation networks, voids) was enhanced in an irradiated Fe–Cr–Mn(W,V) alloy, and in particular, voids formed preferentially on dislocation lines at elevated temperature and that the segregation of Cr, Mn to sinks was suppressed. The reason was that helium strongly interacts with vacancies, which in turn decreases the diffusivity of vacancies and increases the density of sinks in matrix. © 1998 Elsevier Science B.V. All rights reserved.

1. Introduction

The first wall materials of fusion–fission mixed reators will have to endure long-term high flux irradiation of 14 MeV neutrons, and considerable helium will be produced by (n,α) reactions. Swelling and helium/brittle are the main restricting factors for the materials. Therefore, it is useful not only for fusion reactors but also for fast reactors and fission reactors to study the irradiation behavior when helium exists.

Because a 14 MeV neutron source which has enough intensity is still not available, simulation by electrons and ions dual-beam irradiation has advantages because of its speed and low cost, and one can observe the damage process in situ and calculate in a quantitative manner. Although there are some differences with the realistic working conditions of fusion reactors, the results were not contradictary or/to the results of neutron irradiation. The simulation method has important uses and was worldwide adopted by researchers.

This work is the first reported to study the damage behavior of dual-beam irradiated Fe–Cr–Mn alloy containing W,V, and has important theoretical and experimental understanding for the development of low-swelling low-activation austenite alloys.

2. Experimental method

In this study, an Fe–12Cr–17Mn–0.3C–0.1N–1.0Ni–2.3W–1.8V alloy was used. The alloy was prepared using high-purity raw materials, and was remelted in a vacuum induction furnace to reduce the impurities and harmful gases. O, and H contents were very low. After forging and annealing, disk specimens were punched out with 0.2 mm thickness and 3 mm diameter. After degreased and cleaning, the samples were held at 1323 K for 1 h in a sealed quartz tube at a vacuum of 6×10^{-6} Pa for solution treatment. The disk specimens for TEM observation were electro-jet polished.

Dual-beam irradiations were conducted on a high voltage electron microscopy (H-1300 keV) and a 300 keV ions accelerator. The damage rate was 2×10^{-3} dpa/s, the irradiation dose varied from 0 to 10 dpa, and irradiation temperature was from 673 to 773 K. The helium injection rate was 20 appm/dpa (corresponding to the helium production rate for fusion reactors [1]). The damage caused by helium injection was less than 1% of that caused by electron irradiation, and thus can be neglected. The thickness of irradiations area was 400 nm as measured by thickness fringes. The electron beam

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diameter was 2.4 μm and the central diameter of the irradiated area was 1 μm. Low index crystalline planes such as (1 1 1) and (1 1 0) were chosen for irradiation.

The concentration of solutes after irradiation was measured with an energy dispersive X-ray device, the variation of alloying elements was analysed across a grain boundary within both irradiated and nonirradiated zones.

3. Experimental results

3.1. Microstructural development of secondary defects

The evolution of secondary defects during dual-beam irradiation at 673 K is shown in Fig. 1. Interstitial-type loops formed from interstitial-type clusters were at high density and were distributed in the irradiated areas from the beginning of irradiation. Dislocation loops tangled with each other and formed dislocation networks as dose increased. For the dual-beam condition [2,3] dislocation loops formed earlier, and a high density of tiny voids were visible when irradiation dose reached 9 dpa, but growth rate of voids is very low. Therefore helium enhanced the nucleation of dislocation loops and voids remarkably.

The microstructural development of secondary defects at different temperatures are shown in Fig. 2. At low temperature, the density of loops was high and the formation of dislocation networks was retarded, the void dimensions were small and void density was high, and the growth rate of voids was very low. At higher temperature, however, the density of dislocation loops was low, growth rate was high, dislocation networks were formed earlier (see 723 K, 2.6 dpa), void dimensions were large and void density was low. It can also be seen that voids were mainly formed inside dislocation loops (673 K, 6.5 dpa) at low temperature, while at higher temperatures, voids formed on dislocation lines (773 K, 6.5 dpa, 7.2 dpa).

3.2. Void mean size, number density and swelling

The relationship between size, density and swelling of voids as a function of irradiation dose are shown in Figs. 3 and 4. At a given temperature, void size increased slightly with the increase in irradiation dose, but the growth rate was low. Void size increased more rapidly with the increase in irradiation dose at elevated temperatures than at low temperature. With increasing irradiation dose, the void density increased and saturated. This is concurrent with the relationship between void size and irradiation dose.

For dual-beam conditions with the increase of irradiation dose, swelling increased rapidly in the early stage, then increased more slowly. No change in void swelling was observed with continuing increase in dose. At high enough dose, swelling was slightly larger at

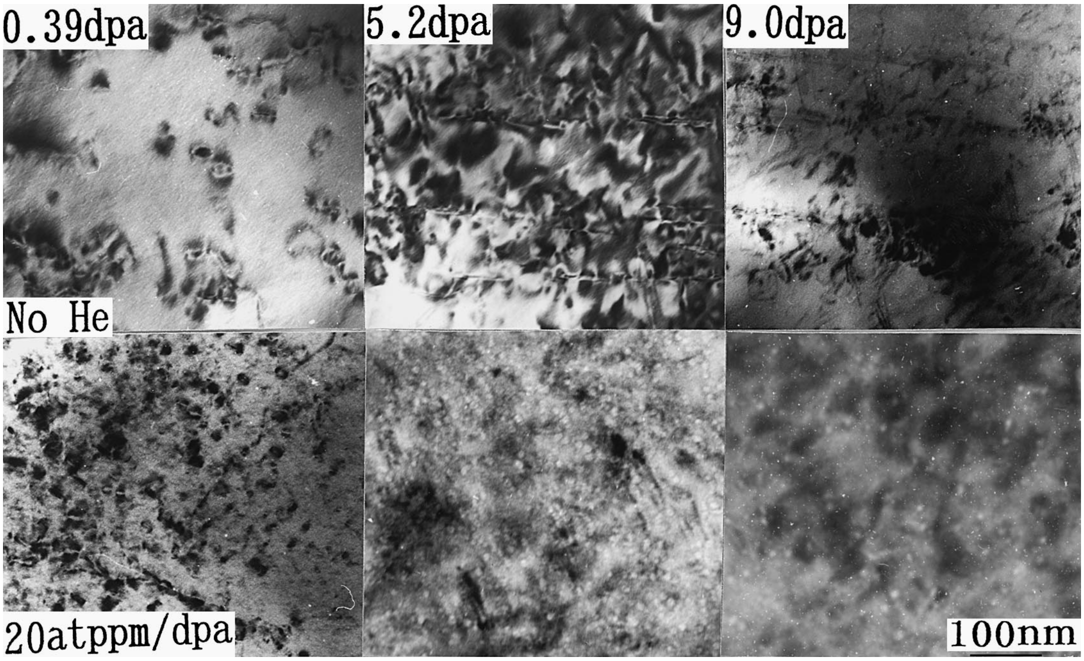

Fig. 1. Development and growth of dislocation loops and voids in alloy by dual-beam irradiation at 673 K.

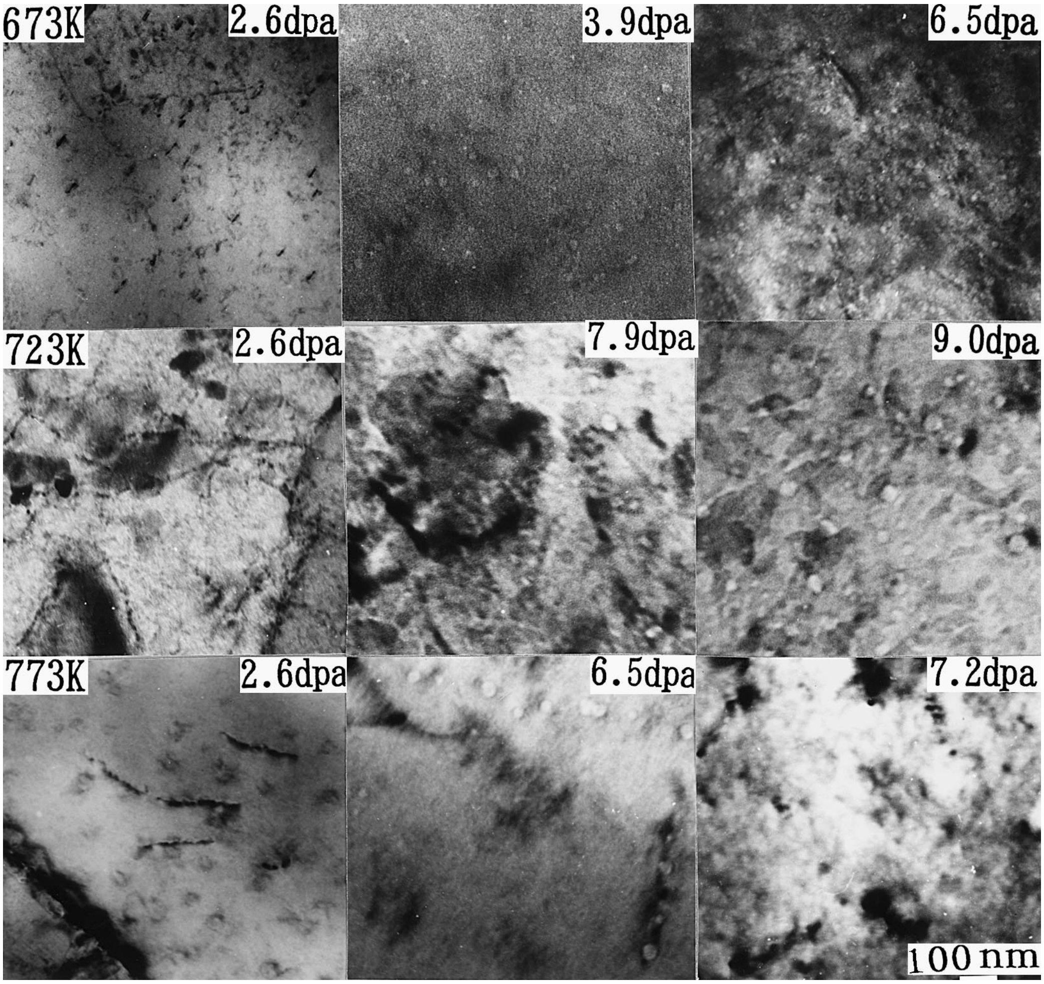

Fig. 2. Dislocation loops and void structures after dual-beam irradiation at different temperatures.

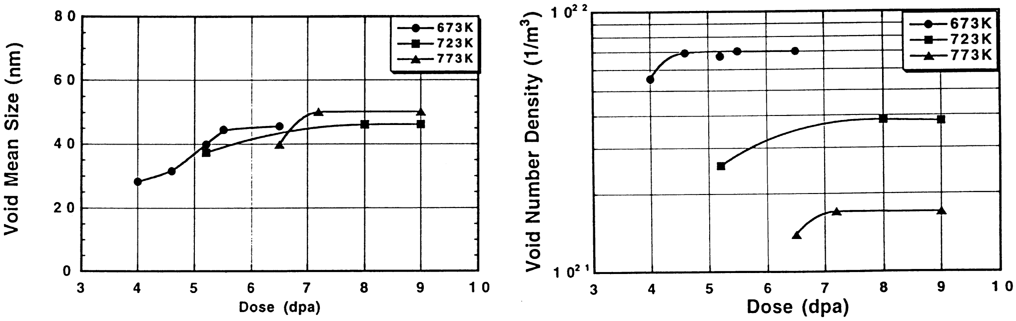

Fig. 3. Dose dependence of void mean size and void number density at different temperatures.

low temperature than that at elevated temperatures but swelling was still low at all temperatures, which demonstrates that the alloy had excellent swelling resistance.

3.3. Radiation-induced segregation

The variations of Cr, and Mn contents at grain boundaries following electron irradiation and dual-

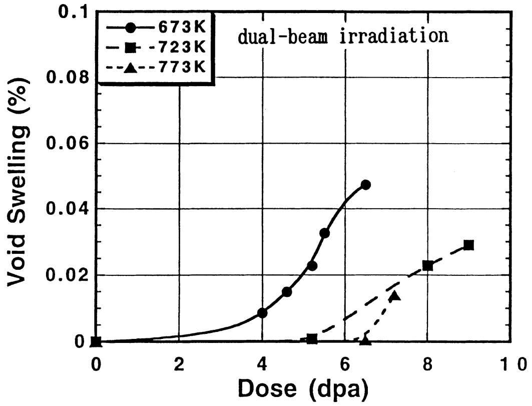

Fig. 4. Dose dependence of void swelling at different temperatures.

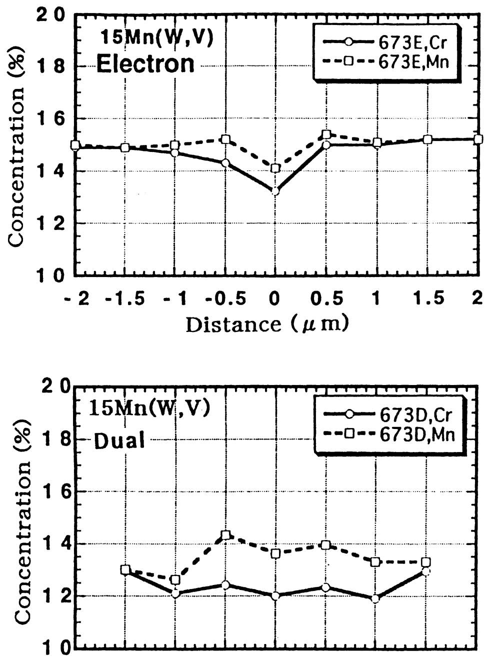

Fig. 5. Variation of Cr and Mn content at grain boundaries at 673 K.

beam irradiation at 673 K are shown in Fig. 5. It is obvious that Cr and Mn contents decreased at grain boundaries for both electron irradiations and dual-beam irradiations, but segregation was obviously lower for dual-beam condition than that for single-beam conditions. The segregation was restrainted effectively with the increase of irradiation temperature.

4. Discussion

4.1. Interaction between helium and secondary defects

Comparing electron irradiation conditions, dual-beam irradiation with helium formed a higher density of dislocation loops earlier. The presence of helium affected the behavior of point defects. On the one hand, the concentration of interstitials produced under dual-beam irradiation is 4% higher than that for electron irradiation [5], which is beneficial to the formation of dislocation loops. On the other hand, it can be seen from the time dependence of point defect density at the beginning of irradiation that the time for vacancies to flow into loops under dual-beam irradiation was very different from that under electrons irradiation after the dislocation loop density saturated [4,7]. For electron irradiation, the time was very short (<1 s). Thus the flux of vacancies recombined with interstitials before the dislocation loops could grow to a stable size, and so the formation of dislocation loop nuclei became very difficult. For dual-beam irradiation, the time varied form a few seconds to hundreds of seconds, before the dislocation loop nuclei had enough time to grow into stable loops, and the probability for the formation of loops increased. Therefore, dual-beam irradiation can enhance the formation of a high density of dislocation loops and increasing density of dislocation.

During dual-beam irradiation, because the solubility of helium in solution is very low, and activation energy for migration is very small (about 0.2 eV) [5], helium can migrate easily. In an Fe–Cr–Mn alloy, the binding energy of vacancies with helium is about 1.19 eV [6] and that for interstitial with helium is about 0.9 eV [7]. It was obvious that helium interacts more strongly with vacancies than with interstitials in austenitic steels, i.e. helium had little effect on interstitials. On the contrary, helium interacted strongly with vacancies and formed He–V combinations [8], which led to slow vacancy migration, and correspondingly decreased vacancy annihilation at sinks, and increased the concentration of vacancies in the matrix, Moreover, He–V combinations can serve as void nuclei which can increase void density. For these reasons, dual-beam irradiation can enhance the formation of voids, especially the formation of voids on dislocation lines and inside dislocation loops.

4.2. Relationship between helium and radiation-induced segregation

It can be seen from the experimental results (Fig. 5.) that both electron irradiation and dual-beam irradiation can lead to changes in alloying element distribution in the alloy. Cr and Mn contents decreased at grain boundary. Because Cr and Mn atoms are oversize atoms in relation to iron atoms, they preferentially interact with vacancies, and diffuse by a displacement mechanism. As vacancies diffuse to sinks Cr and Mn atoms diffuse away from the sinks, and as a result, Cr and Mn contents decrease at the sinks. During dual-beam irradiation, helium interacted strongly with vacancies which led to a decrease in the diffusivity of vacancies under irradiation [5], and the presence of the helium enhanced the formation of dislocation loops. Dislocation density increased, hence the sink density in the matrix increased, which decreased vacancy migration, and in turn decreased the amount of long-range diffusion of vacancies to grain boundaries. Therefore, the segregation of alloying atoms to grain boundaries during dual-beam irradiation decreased.

5. Conclusions

The damage behavior in an Fe–Cr–Mn(W,V) alloy using single-beam and dual-beam conditions showed that:

1. The formation of secondary defects (dislocation loops, dislocation networks, and voids) increased greatly during dual-beam irradiation, swelling increased a little but was still much lower than that for an Fe–Cr–Ni alloy for the same conditions.

2. Segregation of Cr and Mn to grain boundaries was decreased by dual-beam irradiation.

3. The formation of void nuclei on dislocation lines and inside dislocation loops was enhanced by dual-beam irradiation, especially for voids tending to form on dislocation lines at elevated temperatures.

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