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Effect of aging treatment on irradiation-induced segregation of high Mn-Cr steel

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Abstract: To investigate the effect of aging treatment on irradiation-induced segregation of high Mn-Cr steel, specimens for electron-beam irradiation were prepared from the high Mn-Cr austenitic steel which was solution treated at 1 373 K for 1 h and aging treated at 573 K for 1 000 h, respectively. The electron-beam irradiation was performed at 573 K up to doses of 5.4 dpa in a 1 250 kV HVEM and irradiation-induced segregation analyses were carried out by an EDX in a 200 kV FE-TEM. The results show that void formation is not observed in both solution treated and aging treated ones. The amount of Cr segregation at the grain boundary decreases in the aged one; however, that of Mn is not changed in solution treated one.

Key words: high Mn-Cr steel; HVEM; electron-beam irradiation; irradiation-induced segregation; aging

1 Introduction

Structure materials for nuclear and/or fusion reactors such as SUS 304 and SUS 316 steels used for nuclear reactor materials are of good properties in un-radiated condition, but properties of these materials are reduced with the change of composition by the segregation of solute atoms under long-term neutron radiation conditions[1−2].

Since SUS 304 and SUS 316 steels contain 9%−12%Ni, the attenuation of radiation requires a very long time. Therefore, Mn is proposed as a replacement for Ni due to its rapid decay of radioactivity to 1/10 of Ni level. So, many researches were performed to the high Mn-Cr austenitic steels because of their potential use for structure materials of nuclear and/or fusion reactors from the point of view of the reduced activation[3−6]. Among them, the reduced radioactivated 12%Cr−15%Mn austenitic steels with high content of nitrogen have good properties of high temperature strength and high temperature phase stability[4].

In this work, the effect of aging treatment on the irradiation-induced segregation of reduced activation 12%Cr-15%Mn austenitic steel was investigated by using electron-beam irradiation techniques to simulate the reactor damage processes.

2 Experimental

High Mn-Cr steel used had the following chemical composition (mass fraction, %): C 0.10, N 0.18, Si <0.10, Mn 15.10, P <0.003, S 0.005, Cr 11.85, Ni 0.84, W 2.15, V 0.46 and Ti 0.10. Specimens for electron-beam irradiation were prepared from the high Mn-Cr austenitic steel which was hot rolled and solution treated at 1 373 K for 1 h and aging treated at 573 K for 1 000 h, respectively. Disk specimens with a diameter of 3 mm for TEM observation were punched out and electrochemically-jet polished.

The electron-beam irradiation was carried out with a damage rate of about 0.5×10^{-3} dpa/s at 573 K up to doses of 5.4 dpa in a 1 250 kV HVEM. The specimen thickness of irradiation area was selected to be about 400 nm to avoid the surface effect[7]. Irradiation-induced segregation analyses were carried out by an energy dispersed X-ray (EDX) analyzer in a 200 kV FE-TEM with a beam diameter of about 0.5 nm.

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3 Results and discussion

3.1 Microstructure changes by irradiation

Figures 1 and 2 show the microstructure changes observed for the solution treated and aging treated high Mn-Cr steel during electron irradiation at 573 K with HVEM, respectively. Figs.1(a) and 2(a) show the microstructures before irradiation, and Figs.1(b), 1(c), 1(d) and 2(b), 2(c), 2(d) show irradiated microstructures after up to doses of 1.8, 3.6 and 5.4 dpa, respectively. Voids are not formed even after an irradiation dose of 5.4 dpa in both solution treated and aging treated specimens. So it can be considered that the irradiation damage resistance of high Mn-Cr steel is superior to that of SUS304 steel[8]. Many strained regions are observed after up to doses of 3.6 and 5.4 dpa at the aging treated specimens, as shown in Figs.2(c) and (d). And the area of strained region formed by dislocation loop increases with an irradiation dose of 5.4 dpa, as shown in Fig.2(d).

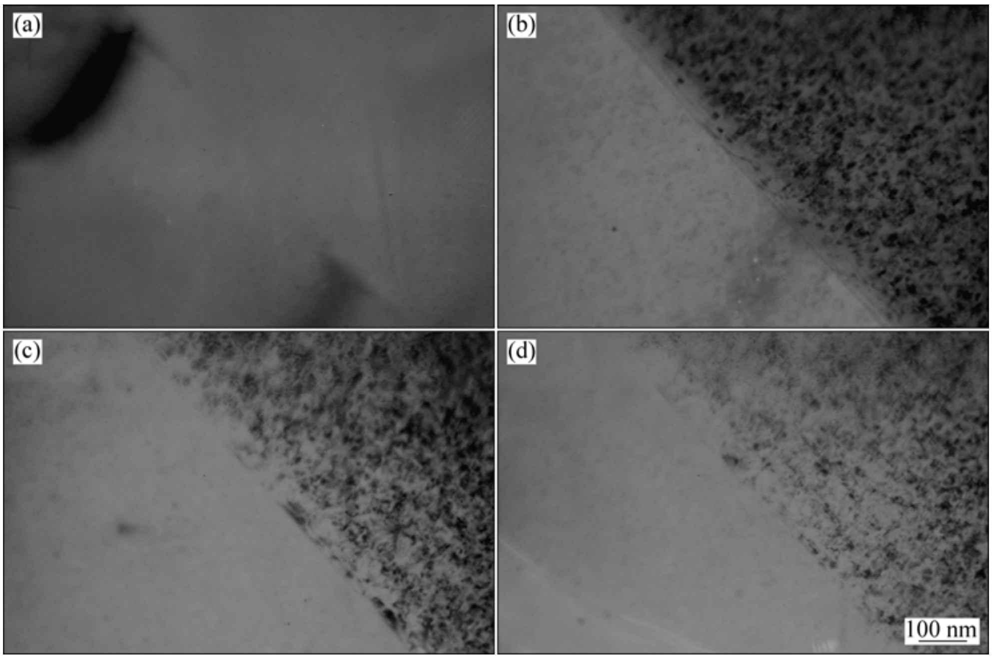

Fig.1 Microstructure changes of solution treated high Mn-Cr steel after electron-beam irradiation doses up to 0 dpa (a), 1.8 dpa (b), 3.6 dpa (c) and 5.4 dpa (d) at 573 K

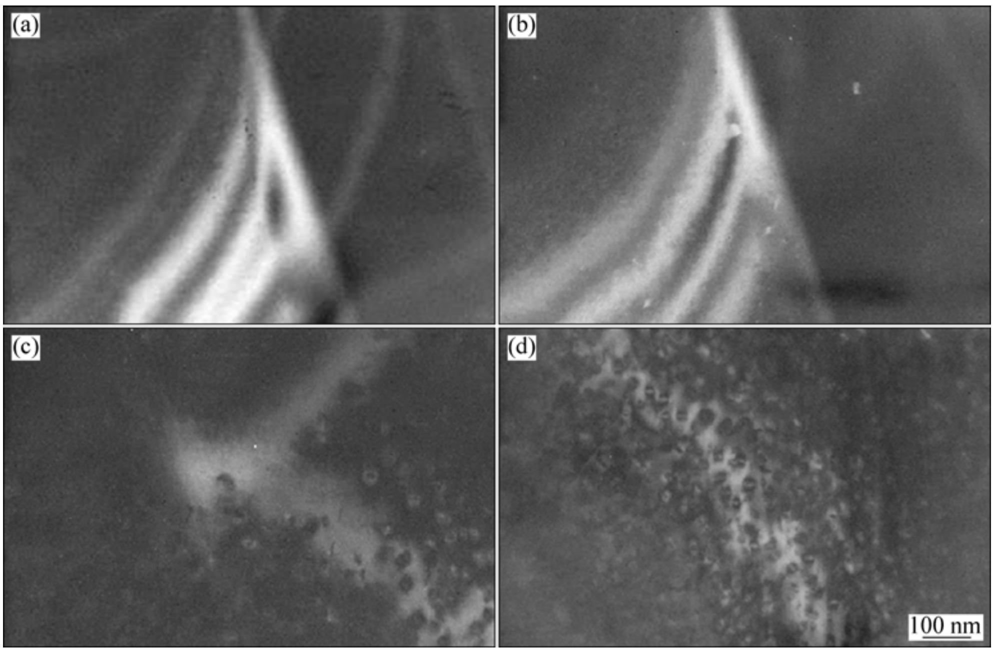

Fig.2 Microstructure changes of high Mn-Cr steel aging treated after electron-beam irradiation doses up to 0 dpa (a), 1.8 dpa (b), 3.6 dpa (c) and 5.4 dpa (d) at 573 K

3.2 Irradiation-induced grain boundary segregation

Figures 3 and 4 show compositional concentration profiles of Cr and Mn near a grain boundary after electron irradiation up to dose of 5.4 dpa at 573 K for the solution treated and aging treated high Mn-Cr steels, respectively. Large amount of point defects and the secondary defects are introduced continuously under irradiation conditions. In this case, the diffusion of solute atoms takes place by the interaction of solutes with the fluxes of point defects to defect sinks so that non-equilibrium segregation with the enrichment or depletion of solute atoms occurred near sink sites such as grain boundaries and voids[9].

The phenomena of non-equilibrium segregation, such as concentration changes of Cr and Mn, are also observed at grain boundaries by electron irradiation. The enrichment of Cr and the depletion of Mn at grain boundaries are produced by electron irradiation at 573 K, as shown in Figs.3 and 4.

According to Ref.[10], fine precipitates are formed at grain boundaries and within grains in the early irradiation stage, then the size and the number of precipitates increase gradually with irradiation dose in high Mn steel electron-irradiated to a dose of 10 dpa at 723 K. And these precipitates affect the grain boundary segregation. Therefore, it is considered that the changes of precipitation behavior affect the concentration distributions of near grain boundaries with aging treatment at 573 K for 1 000 h.

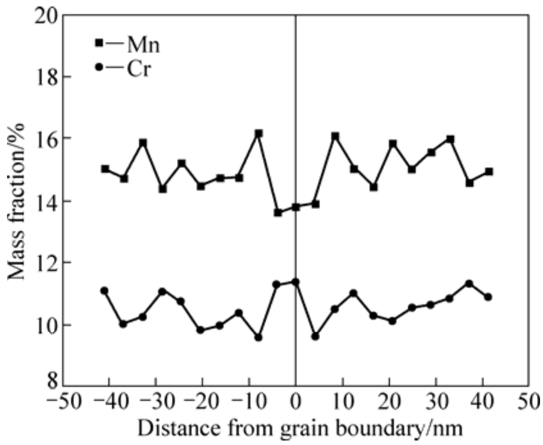

Fig.3 Comparison of concentration profile near grain boundary after electron-beam irradiation dose up to 5.4 dpa at 573 K in solution treated high Mn-Cr steel

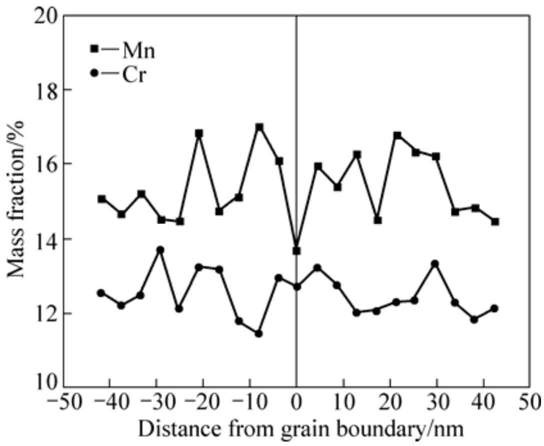

Fig.4 Comparison of concentration profile near grain boundary after electron-beam irradiation dose up to 5.4 dpa in high Mn-Cr steel aging treated at 573 K for 1 000 h

Fig.5 shows the changes of irradiation-induced segregation near a grain boundary with electron-beam irradiation temperature by trimming the results from Figs.3 and 4.

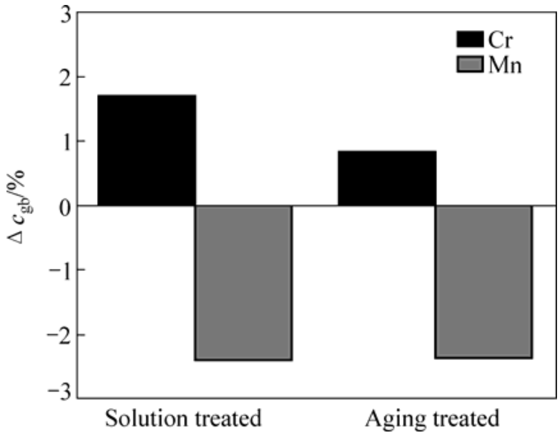

Fig.5 Comparison of irradiation induced segregation near grain boundary with electron-beam irradiation dose up to 5.4 dpa at 573 K in solution treated and aging treated high Mn-Cr steel

The change of grain boundary segregation (Δc_{gb}, mass fraction) was calculated from the difference between that at the grain boundary and concentration at the nearest position from the grain boundary. The Cr concentration of solution treated one near a grain boundary shows about 1.73%Cr concentration difference (namely, enrichment of Cr); however, the concentration difference of aging treated one shows about 0.85%Cr after electron-beam irradiation at 573 K. From this, it can be understood that the enrichment of Cr at the grain boundary is reduced and then concentration difference near the grain boundary decreases when aging treated at 573 K for 1 000 h.

Mn concentrations near a grain boundary in both solution treated and aging treated ones show −2.37%Mn and −2.34%Mn concentration difference (namely, depletion of Mn) after electron-beam irradiation at 573 K, respectively. That is, the amount of Mn depletion at a grain boundary is not affected by aging treatment in electron-beam irradiation at 573 K. It may be possible that the region of unstable austenite phase will be formed near grain boundary by decreasing the Mn content near grain boundary with aging treatment at 573 K for 1 000 h.

4 Conclusions

1) Void formation is not observed in both solution treated and aging treated ones after electron-beam irradiation at 573 K.

2) The area of strained region formed by dislocation loop is observed and the number of strained region increases with the increase of irradiation dose in aging treated one.

3) The amount of Cr segregation at the grain boundary decreases in aging treated one at 573 K for 1 000 h; however, that of Mn is not changed in solution treated one.

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