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Effect of solute elements in Ni alloys on blistering under He^+ and D^+ ion irradiation

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Abstract

Effects of solute atoms on microstructural evolution and blister formation have been investigated using Ni alloys under 25 keV He^+ and 20 keV D^+ irradiation at 500 °C to a dose of about 4×10^{21} ions/m^2. The specimens used were pure Ni, Ni–Si, Ni–Co, Ni–Cu, Ni–Mn and Ni–Pd alloys. The volume size factors of solute elements for the Ni alloys range from −5.8% to +63.6%. The formations of blisters were observed in the helium-irradiated specimens, but not in the deuteron-irradiated specimens. The areal number densities of blisters increased with volume size difference of solute atoms. The dependence of volume size on the areal number densities of blisters was very similar to that of the number densities of bubbles on solute atoms. The size of the blisters inversely decreased with increasing size of solute atoms. The formation of blisters was intimately related to the bubble growth, and the gas pressure model for the formation of blisters was supported by this study.

1. Introduction

Blistering was first observed on the surfaces of corundum, spinel, rutile and perdot when bombarded above threshold values with protons or helium by Primak in 1963 [1]. Since then, blistering phenomena were also observed in many materials, i.e., metals, alloys, semiconductors, ceramics and glass [2–13], but no formation of blistering was found in some kind of glass whose transmissivity was very high [14]. On the surface of the first wall blanket of fusion nuclear reactors, high energy particles produced by D–T reaction induce the blistering and sputtering. Two mechanisms for the blistering were proposed; the gas pressure model [15] and the lateral stress model [16]. In the gas pressure model, the blistering can be induced by the gas pressure in cavity of crack formed near the irradiated surface. In the lateral model, the gas pressure can serve as only a small nucleating force, while blistering results primarily from an internal lateral stresses in the surface layer. In the previous studies,

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we reported the development of dislocation loops and cavities formed in Ni alloys irradiated by He^+ ions up to 4×10^{20} ions/m^2 [17] and by D^+ ions at doses up to 4×10^{20} ions/m^2 [18]. In this study, the irradiations were performed up to a dose of 4×10^{21} ions/m^2. The purpose of this study is to examine the features of blister formation and the effect of solute atoms on the blistering process.

2. Experimental procedure

Pure Ni and binary Ni alloys containing Si, Co, Cu, Mn, Pd, Nb and Au were studied. These solute atoms of the binary Ni alloys have the different volume size factors as given in Table 1. Ni–5.0 at.%Si, Ni–9.0 at.%Si, Ni–5.0 at.%Co, Ni–4.9 at.%Cu, Ni–4.6 at.%Mn, Ni–5.4 at.%Pd, Ni–2.3 at.%Nb and Ni–1.3 at.%Au binary alloys were prepared by melting high purity metals in a high frequency induction furnace in vacuum. The ingots were annealed at 1100 °C for one week in a vacuum. After the solution treatment, the hardness of the specimens was measured using a Vickers microhardness tester. The results of hardness measurements are also given in Table 1. The hardness of Ni alloys increased with the increasing volume size factor of solute atoms. The specimens were electro-polished by the window method in a solution of 60% surfuric acid and 40% water by volume at 15 °C, and the applied voltage was 4 V. Additional polishing was done for the Ni–Pd and Ni–Au alloys in a solution of 95% methanol and 5% perchloric acid at −33 °C and applied voltage of 15 V. Ni–Mn specimens were polished again in a solution of 80% methanol and 20% perchloric acid by volume at −33 °C. The foil specimens were irradiated with 25 keV He^+ ions or 20 keV D^+ ions from an RF ion source. The helium irradiations were performed at 500 °C with fluxes of 3.2×10^{18} and 5.6×10^{18} ions/m^2 s to investigate the flux dependence on the density of bubbles and blisters. The irradiation dose was 4.1×10^{21} ions/m^2. Defect production rates and a projected range probabilities for 25 keV He^+ ions or 20 keV D^+ ions were calculated by the TRIM program.

After the irradiations, the damage structures of the specimens were investigated with a transmission electron microscope (TEM) and a scanning (transmission) electron microscope (SEM, STEM) operated at 200 kV.

3. Results

Blisters formed on the surfaces of pure Ni and Ni binary alloys implanted by He^+ at 500 °C to a dose

Table 1
Volume size factors (%) of solute atoms in Ni, Vickers hardness of pure Ni and Ni binary alloys (Hv) and the value of Vickers hardness per one atomic percent solute in the Ni alloys

Solute atoms		Si	Co	Cu	Mn	Pd	Nb	Au
Volume size factor (%)		−5.8	+1.8	+7.2	+23.2	+41.3	+51.2	+63.6
Materials	Ni	Ni–9Si	Ni–5.0Co	Ni–4.9Cu	Ni–4.6Mn	Ni–5.4Pd	Ni–2.3Nb	Ni–1.3Au
Hv (kg/mm^2)	83	122.5	86	92.5	108	117.5	138	117.5
Hv (kg/mm^2/at.%)	83	87.4	83.6	84.9	88.4	89.4	106.9	109.5

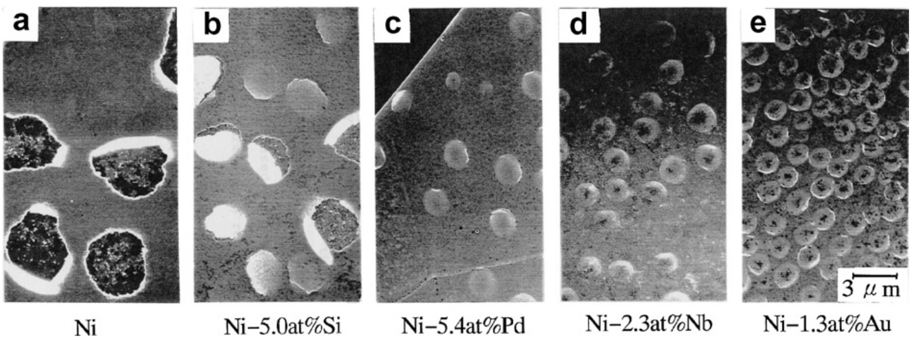

Fig. 1. Blisters formed in pure Ni, Ni–5.0 at.%Si, Ni–5.4 at.%Pd, Ni–2.3 at.%Nb and Ni–1.3 at.%Au implanted at 500 °C by 25 keV-He^+ ions to a dose of 4.1×10^{21} He^+ ions/m^2 with the flux of 3.2×10^{18} He^+ ions/m^2 s.

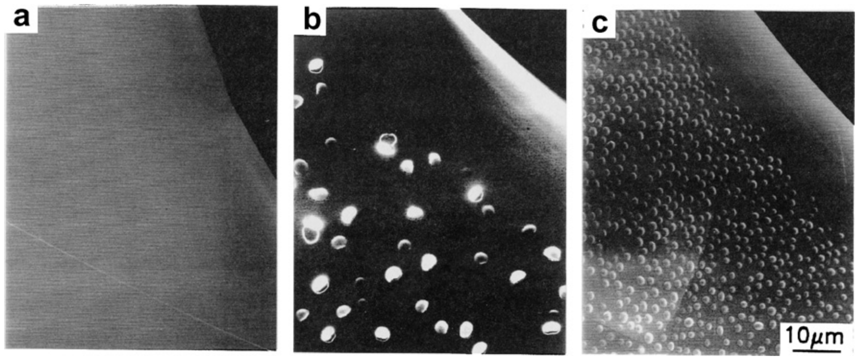

Fig. 2. Blisters formed in Ni–9.0 at.%Si, Ni–5.0 at.%Co and Ni–4.9 at.%Cu alloys implanted at 500 °C by 25 keV-He^+ ions to a dose of 4.1×10^{21} He^+ ions/m^2 with the flux of 5.6×10^{18} He^+ ions/m^2 s.

of 4.1×10^{21} ions/m^2 with the flux of 3.2×10^{18} ions/m^2 s as shown in Fig. 1. In the Ni alloys with oversized solutes such as Ni–Pd, Ni–Nb and Ni–Au, the shape of the blisters were very symmetrical. Blisters in the pure Ni and Ni–Si alloy were somewhat smaller and the shape was not circular. In pure Ni and Ni–Si alloy, many blisters had ruptured and the blister cap was lost. Fig. 2 shows the surface of the specimens implanted to a dose of 4.1×10^{21} ions/m^2 with the flux of 5.6×10^{18} ions/m^2 s. No formation of blisters was observed in Ni–Si alloy, but blisters were formed in the Ni–Co and Ni–Cu alloys. The areal number of the blisters increased with increase in the difference of the volume size between solvent and solute atoms, and the mean size of blisters decreased with the increasing volume difference as given in Fig. 3. The formation of blisters was observed in the specimens implanted with the flux of 5.6×10^{18} ions/m^2 s, but no blisters were formed in the specimens implanted with the flux of 3.2×10^{18} ions/m^2 s, with both cases to the same total dose.

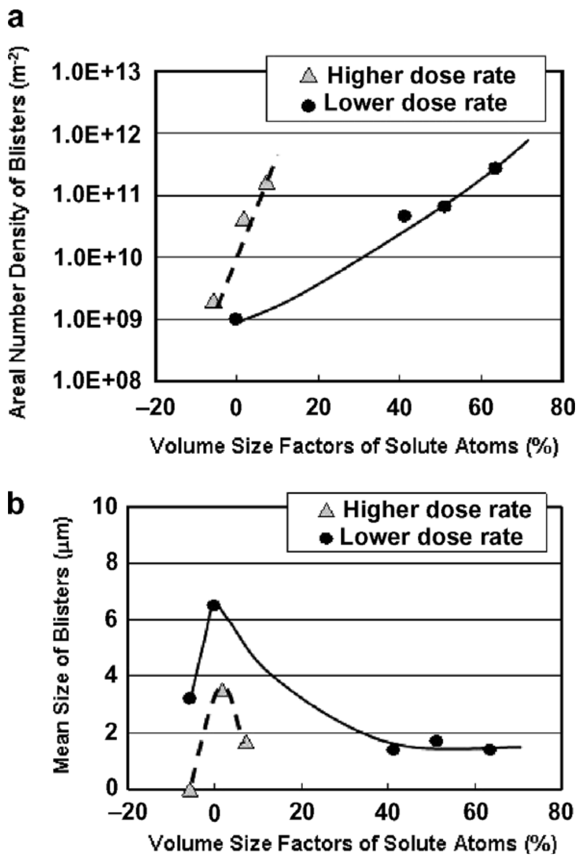

Fig. 3. Areal number density (a) and mean size (b) of blisters formed in Ni and Ni binary alloys by 25 keV He^+ ions at 500 °C to a dose of 4.1×10^{21} He^+ ions/m^2 with the flux of 3.2×10^{18} He^+ ions/m^2 s (solid line) or 5.6×10^{18} He^+ ions/m^2 s (dotted line).

In the D^+ implantation experiments, no formation of blisters was observed in any of the Ni alloys implanted at 500 °C to a dose of 4.1×10^{21} ions/m^2.

4. Discussion

In a previous study of microstructural evolution in Ni alloys irradiated with He^+ ions, the formation and growth of dislocation loops and cavities was examined [17]. The high number densities of dislocation loops, about 1.5×10^{22} m^{-3}, were formed in the specimens irradiated to a dose of 1×10^{19} ions/m^2, and the number density of the loops was almost independent of the alloys, except for Ni–Si alloy with negative volume size factor. The mean size of the loops, on the other hand, tended to increase with the volume size factor. At a dose of 4×10^{20} ions/m^2, the swelling ranged from 0.2% to 4.5%, depending on the volume size factors. The number densities of bubbles tended to increase with the absolute values of the volume size factor, and the swelling increased with the volume size factors.

As the growth of cavities continues under helium implantation, bubbles can combine with each other, and larger bubbles will result. Furthermore, as the implantation proceeds, blisters can be formed by the combination of several larger cavities. The areal number of blisters has a dependence on solute volume size, because the number densities of bubbles have a dependence of solute volume size [17]. SEM and STEM images of blisters in Ni–Cu alloy implanted at 500 °C to a dose of 4×10^{21} ions/m^2 are shown in Fig. 4(a) and (b), respectively. Fig. 4(a) shows the top surface and Fig. 4(b) is the transmission image. Blisters at the positions of combination of several large cavities in the Ni–Cu alloy were formed in the area of 15 μm from the specimen edge. The shape of specimen used in this study is a wedge, and the thickness of the specimen increases with increasing distance from the edge. As seen in Fig. 4(b), many bubbles can be seen, and larger bubbles are formed in the positions of the blisters. In the

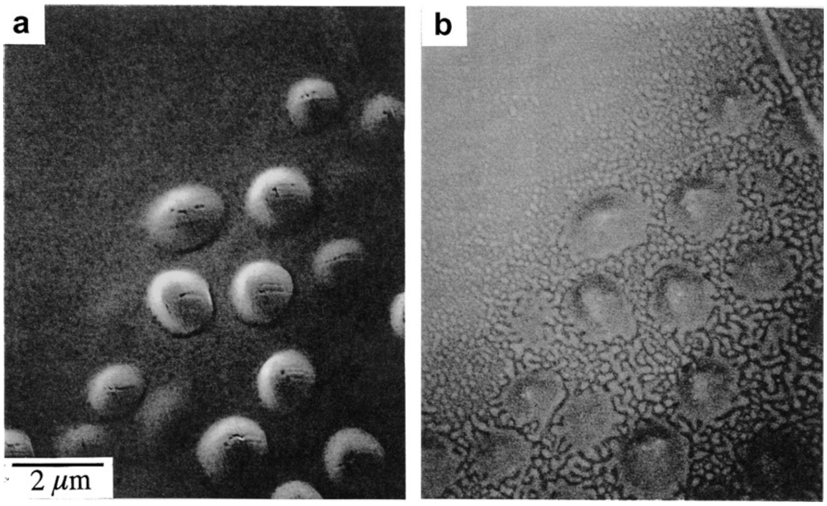

Fig. 4. SEM and STEM images of Ni–Cu alloy implanted at 500 °C to a dose of 4.1×10^{21} He^{+} ions/m^2.

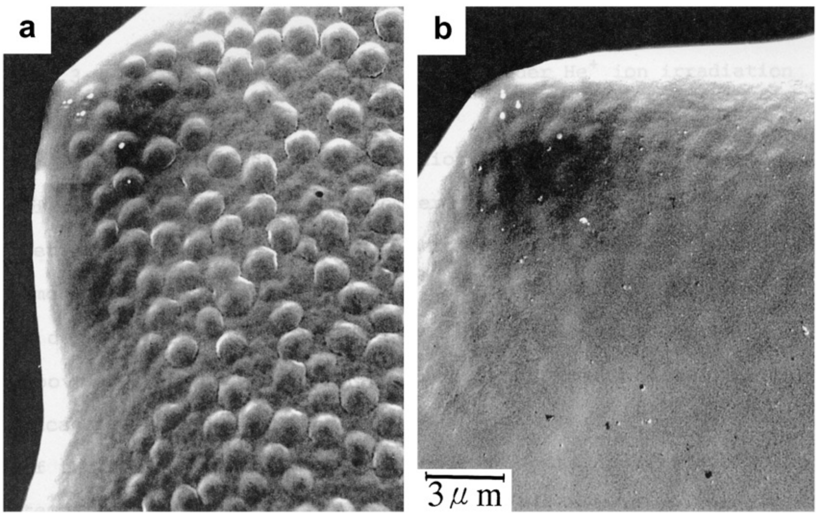

Fig. 5. SEM images of blisters taken from (a) upper surface and (b) bottom surface in Ni–Au alloy implanted at 500 °C to a dose of 4.1×10^{21} He^{+} ions/m^2.

region without blisters, larger size cavities are not observed. The thickness of the specimen in the region of blister formation is more than 600 nm and this thickness is about five times the projected range, 120 nm, of 25 keV helium ions in Ni.

Foil specimens were used to investigate the blister formation mechanism. If the blisters are formed by the lateral stresses, the bottom surface at the position of the blister should have a hollow or wrinkle in the thin region. On the other hand, if the blisters were formed by the involved gas pressures, the bottom surface should also bulge. Fig. 5 shows SEM micrographs of the top and bottom surfaces of the Ni–Au alloy irradiated at 500 °C with 25 keV He^+ ions, respectively. The blisters bulge out from both the top and bottom surfaces, and therefore, the present results of the blisters at 500 °C support the gas pressure model.

5. Summary

Effects of solute atoms on microstructural evolutions and blister formation have been investigated using several kinds of Ni alloys under 25 keV He^+ and 20 keV D^+ irradiation at 500 °C. The formation of blisters was observed at a dose of about 4×10^{21} He/m^2. The areal number densities of blisters increased with volume size difference of solute atoms, and the dependence of the areal number densities of blisters on the volume size of solute atoms was very similar to that of the number densities of bubbles. The size of the blisters decreased inversely with increasing solute volume size difference. The formation of blisters was intimately related to the bubble growth, and the gas pressure model for the formation of blisters is supported by this study. In contrast, no formation of blisters was observed in the specimens irradiated by deuterons.

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