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Electron irradiation effects on Ti–Ni shape memory alloys

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Abstract

Effects of 1 MeV electron irradiation were investigated for Ti–Ni alloys of three different compositions; Ti–49.9, 50.2 and 50.7 at.% Ni, by using an ultra-high voltage electron microscope. Obvious amorphization by the irradiation at room temperature was observed for the Ti–49.9 at.% Ni alloy in the as-received state, while in the Ti–50.2 at.% Ni alloy, the amorphization proceeded only a small extent for much higher doses. No amorphization could be observed for both specimens irradiated at temperatures higher than 343 K. The Ti–50.7 at.% Ni alloy did not amorphize even after the irradiation of 8 dpa at room temperature. Higher nickel concentrations and higher irradiation temperatures suggested the retardation of the amorphization. Prior to the amorphization, the disordering takes place and brings about the shape memory characteristics loss, as well. The disordering process always takes place even if the amophization does not occur. Deformation of the alloys enhances the amorphization induced by the electron irradiation. No defect clusters could be seen in the present experiment.

1. Introduction

Ti–Ni shape memory alloys can generate a very large force by the phase transition at temperatures below 400 K, so that these alloys are expected as high performance functional materials for actuators. Applications for driving machine components from a remote place or connecting detachable machine elements with strong force can be realized in a limited space, so that these alloys are expected as useful functional component materials for a fusion reactor [1]. In such a case, the irradiation effects on the shape memory characteristics of these alloys are very important. Many research works on shape memory characteristics of neutron irradiated Ti–Ni alloys have been reported [2–4], however, not so many reports on the structural changes have not been published so far.

The amorphization of Ti–Ni was reported on electron irradiation experiments [5] and neutron irradiation experiments [6]. The disordering process of this alloy system by irradiation with high energy particles is also important. Because the shape memory characteristics originates from the martensitic transformation which is only allowed in the ordered phase. The disordering process of the intermetallic compounds by the electron irradiation was reported by in situ observation by Mori and Fujita [7] and Luzzi et al. [8]. In the present work, the electron irradiation effects of Ti–Ni alloys with three different alloy compositions: Ti–49.9, 50.2 and 50.7 at.% Ni alloys, were investigated.

2. Experimental procedures

2.1. Specimens

Ti–Ni alloys of three different compositions were prepared for the electron irradiation experiment. The as-received specimens were thin sheets of 0.1 mm thickness finished by cold rolling by a reduction of 30%. In addition to the as-received specimens, two sets of specimens were prepared: (1) after solution treated the as-received specimens from 1273 K, they were annealed at 673 K for 3.6 ks and quenched into ice water, and (2) after quenching into ice water, quenched into liquid nitrogen in order to investigate the irradiation effects on the sub-zero quenched specimens.

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2.2. Electrical resistance measurement

In order to examine the thermal behavior of the specimens, electrical resistance–temperature curves were measured for a heat cycle from 77 to 473 K.

2.3. Electron irradiation

The specimens were irradiated at a constant dose rate of 5×10^{23} e/m^2 s with an ultra-high voltage electron microscope operated at 1 MV. The change in the microstructures and the diffraction patterns were traced for the irradiation at 300, 343 and 373 K.

3. Results and discussion

3.1. Electrical resistance measurement

1. *As-received specimens*: The electrical resistance–temperature curves shown in Fig. 1(a) revealed the phase transition in the Ti–49.9% and Ti–50.2% Ni alloys, while the transition is not clear in the 50.7 at.% Ni alloy. In the figure, M_s is the starting temperature of the martensite transformation, A_s and A_f are the starting and finishing temperatures of the phase transition from martensite to B2 phase, respectively.

2. *Specimens homogenized, annealed and quenched into ice water*: On heating up the specimens, the inverse transformation from martensite to B2 phase takes place at about 320 K, and on cooling down the specimen, the martensitic transition temperature is below 300 K as shown in Fig. 1(b).

3. *Specimens homogenized, annealed and quenched into liquid nitrogen*: The shapes of the electrical resistance–temperature curves for three specimens are similar each other as shown in Fig. 1(c).

3.2. Irradiation effects of the as-received specimens

1. *Ti–49.9% Ni*: Before the irradiation, band like structure of the martensitic phase can be seen as in Fig. 2(a). Diffused Debye rings show lattice distortion due to the final cold rolling of 30%. During the irradiation at 300 K, an inmost spotty Debye ring gradually changed its contrast to diffused one as seen in Fig. 2 from (a) and (d). Spots from ordered phase very close to the central spot seen in (a) disappeared after irradiation for 80 s, and irradiation for 390 s the ring became a halo of uniform intensity as shown in (d), and the outer rings and the fine structure of the central portion of the structure image of (d) also disappeared. These changes of the diffraction patterns and structure image contrast show the amorphization. The irradiation temperature, 300 K, is much below A_f point. If the observable amorphization starts after irradiation for 100 s and is completed after 400 s, the irradiation doses correspond to 0.2 and 0.8 dpa, respectively.

After the specimen amorphized, in order to trace the crystallization process, the amorphized specimen was continuously heated up from 300 to 673 K in the microscope and structure changes of amorphized portion were observed at several ten minutes intervals. During heating the specimen, area of the observation was put out of the electron beam. In Fig. 3(a), taken just after discontinuing irradiation at 300 K, diffraction spots from b.c.c. with strong intensity are already seen and the intensity of the halo ring became weak. This shows rapid crystallization by stopping the irradiation. At 423 K, the intensity of diffraction spots increased. At 673 K, the halo ring almost disappeared and fine grained structure became more clear as seen in (b).

No amorphization can be observed for the specimens even after the irradiation of 600 s at 373 K.

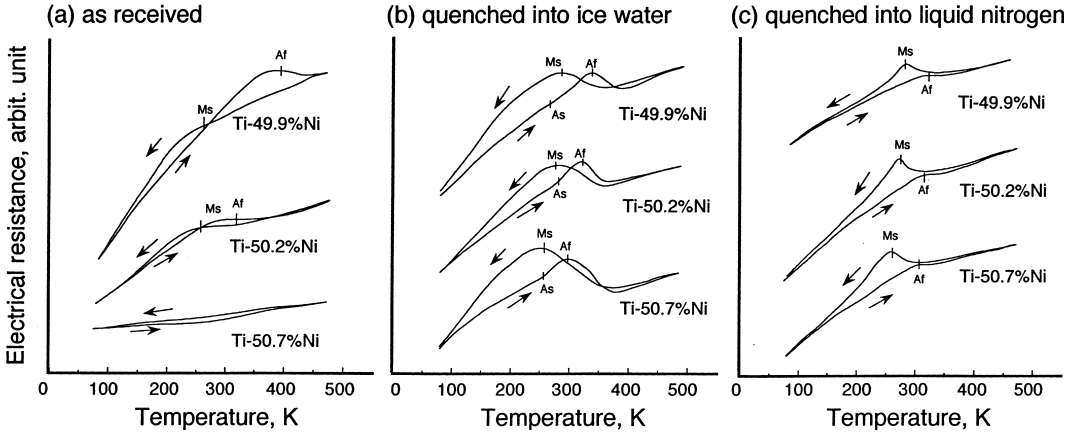

Fig. 1. Electrical resistance-temperature curves for specimens, as-received (a), solution treated from 1273 K and annealed at 673 K for 3.6 ks and quenched into ice water (b), and quenched into liquid nitrogen after quenched into ice water (c).

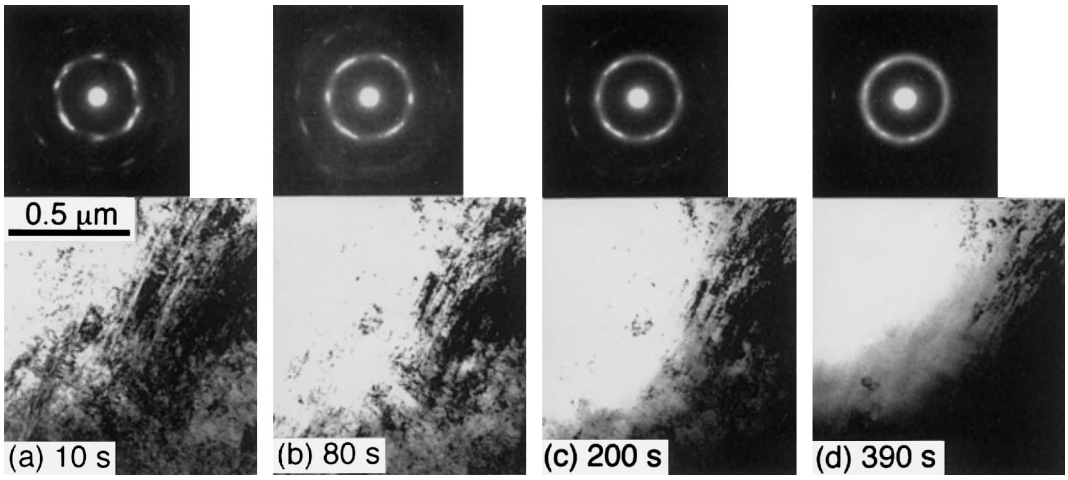

Fig. 2. Amorphization process of the as-received Ti–49.9% Ni observed for the irradiation at 300 K.

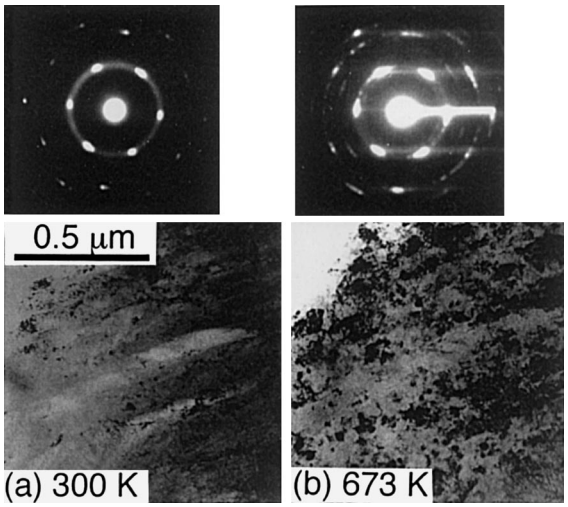

Fig. 3. Crystallization process by heating up the as-received Ti–49.9% Ni after amorphization.

2. *Ti–50.2% Ni*: The irradiation temperature, 300 K, is close to A_f point. At the beginning of the irradiation the microstructure is composed of high density dislocations as shown in Fig. 4(b-1). After irradiation for 330 s, the diffraction pattern shows the amorphization and a part of the fine structure disappeared as seen in (b-2).

3. *Ti–50.7% Ni*: Even after the irradiation for 2.2 ks at 300 K, the diffraction pattern and the structure image did not change (although spots from the ordered phase do not appear on a (1 1 1) pattern) as seen in Fig. 4(c-2).

Defect clusters such as dislocation loops could not be observed for all the as-received specimens because of the heavily deformed condition. The defect cluster formation is unlikely in the as-received specimens, because vacancies and interstitials produced by the irradiation may be absorbed by high density dislocations introduced by cold rolling. For all the specimens, the irradiation at much higher temperatures: 343 and 373 K, only the disordering was observed.

3.3. Specimens homogenized, annealed and quenched into ice water

1. *Ti–49.9% Ni*: At the irradiation temperature, 300 K (between A_s and A_f points), the martensitic phase and B2 phase coexist. The initial sharp diffraction spots in Fig. 4(d-1) became a little diffused ones after irradiation for 200 s. A halo ring appeared after the irradiation for 3.5 ks, but any change in the microstructure could not be observed as seen in (d-2).

2. *Ti–50.2% Ni*: Typical disordering process can be clearly observed in Fig. 4(e-1) and (e-2). After irradiation at 300 K for 250 s, many diffraction spots from the ordered phase observed at the beginning of the irradiation as seen in (e-1) decreased their number to form b.c.c. pattern, but after 490 s, arrays of diffused spots of weak contrast still remained as seen in (e-2). This suggests that the ordered phase still exists. After a prolonged irradiation at 300 K for 3.6 ks, amorphization could be observed.

3. *Ti–50.7% Ni*: After irradiation at 300 K for 3.5 ks, halo rings from the amorphization could be observed as shown in Fig. 4(f-2). The contrast of the irradiated portion (at the central portion of the micrograph (f-2)) changed to be bright and fine structures disappeared.

3.4. Specimens homogenized, annealed and quenched into liquid nitrogen

1. *Ti–49.9% Ni*: Diffraction spots from the ordered phase disappeared by the irradiation at 300 K for 1.5 ks,

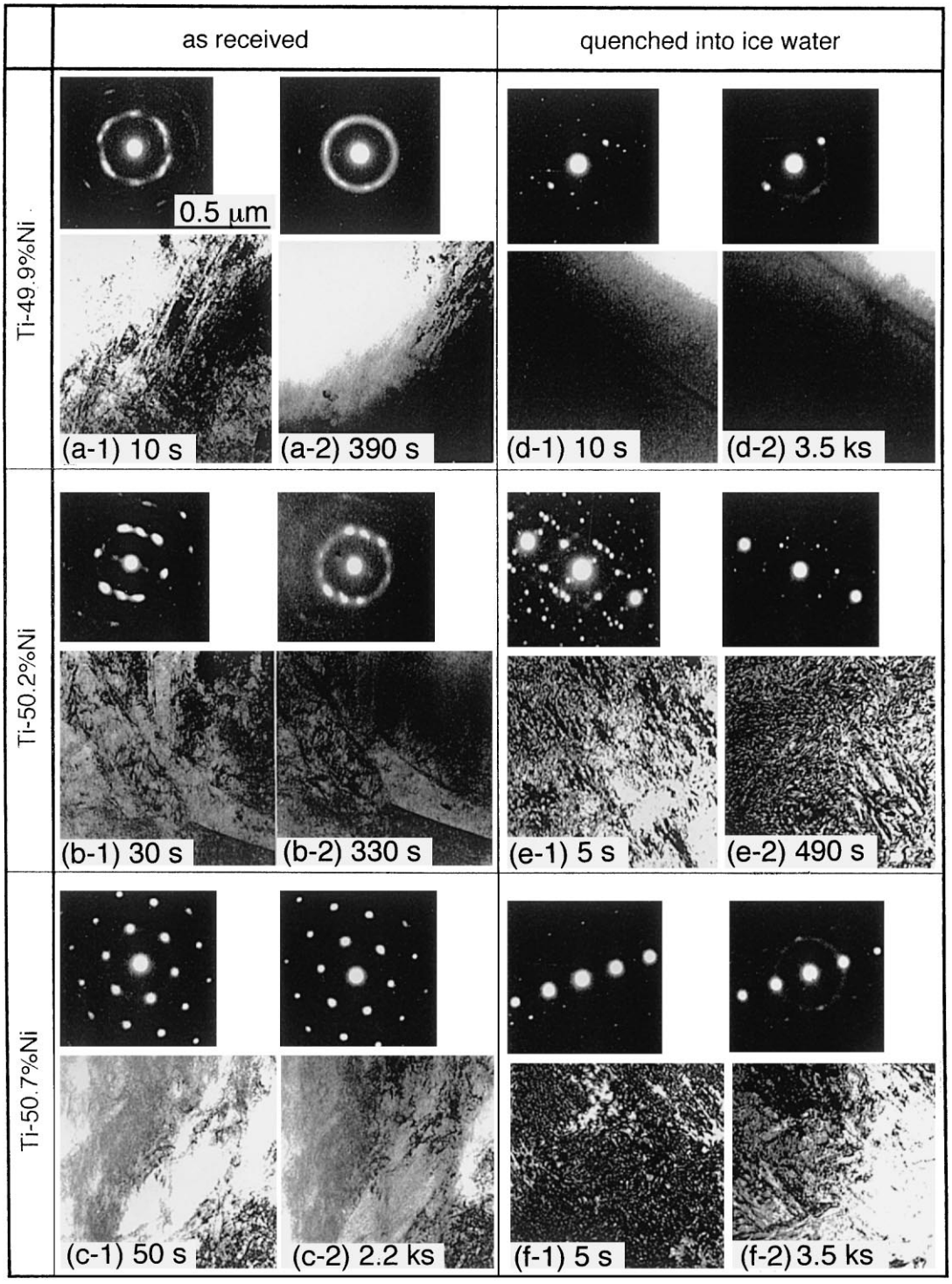

Fig. 4. Changes in microstructures and diffraction patterns by the electron irradiation at 300 K. The as-received specimens, from (a-1) to (c-2) and specimens after solution treated and quenched into ice water, from (d-1) to (f-2). The irradiation times are indicated at the bottom of each photograph.

and a halo ring can be observed after irradiation of 3.6 ks.

2. *Ti–50.2% Ni*: Diffraction spots from the ordered phase became diffused ones and changed to a halo ring after the irradiation at 300 K for 1.8 ks. However, strong diffraction spots from crystalline phase still remained after irradiation for 1.8 ks.

3. *Ti–50.7% Ni*: Spots from the ordered phase disappeared after irradiation at 300 K for 400 s. No amorphization can be observed even after irradiation for 2 ks.

For all the specimens which were quenched into liquid nitrogen, the amorphization was observed for doses higher than several dpa. In the electrical resistance–temperature curves for these specimens as shown in Fig. 1(c), the inverse transformation of martensite are not clear. These curves show that the inverse transformation almost finished below 300 K, so that alloy phase at the irradiation temperature (300 K) may be already transformed to B2 phase. It can be considered that in this crystal structure the amorphization takes place at much higher doses than that for the amorphization in the martensitic phase. The thermal history of the specimens is also sensitive to the amorphization as well as the alloy compositions.

4. Conclusion

The amorphization of the Ti–Ni alloys strongly depends on the alloy composition and its heat history. On electron irradiation at temperatures below A_f point, viz., Ti–Ni alloys in the martensitic phase easily amorphized. Alloys with nickel-poor composition from Ti–Ni stoichiometry, and severely deformed martensitic phase may have less resistance against the amorphization. Prior to the amorphization, the disordering takes place at an earlier stage of the irradiation.

By discontinuing the irradiation, amorphous phase rapidly recovers to the disordered crystalline phase even at room temperature. The recovery of the shape memory characteristic is difficult unless the alloys are heated at temperatures above 1373 K (order–disorder transition temperature of Ti–Ni) for obtaining the ordered phase. Even if the amorphization does not take place, the disordering process is unavoidable.

For the shape memory characteristics, the disordering process may be important for irradiations with high dose rate such as the electron irradiation, while in the irradiations with high energy particles such as neutrons which produce cascade damages, the formation of defect clusters and the amorphization may be more important.

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