Microstructural Change and Mechanical Property of Neutron Irradiated Ti-Ni Shape Memory Alloy

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Microstructural change and mechanical property of Ti-Ni shape memory alloy after neutron irradiation have been studied. The neutron doses were from 1.4×10^{16} to $1.2 \times 10^{19} n/cm^2$, and the irradiation temperature was under 423K. A halo ring was observed after the irradiation of $1.2 \times 10^{19} n/cm^2$, which means that amorphous phase was induced by the neutron irradiation. In stress-strain curve, the critical point (σ_M) increased as the dose increased. At the highest dose, the stress-strain curve lost pseudoelasticity. These results indicate that such mechanical properties strongly depend on the amorphous formation.

KEYWORDS: shape memory alloy, neutron irradiation, amorphous

1. Introduction

Ti-Ni shape memory alloy has been trying to use for nuclear reactor applications. According to recent works[1]~[4], this alloy showed high sensitivity for high energy particles irradiation.

The martensitic and its inverse transformation temperatures, Ms and Af, shifted to lower temperature side[1,2]. The stress-strain curve changed drastically[3], which means that the shape memory effect and the pseudoelasticity were affected by the high energy particles irradiation, however, the mechanism is not fully understood.

Despite this alloy is well known as the material in which amorphization occurs during electron irradiation[4], the mechanical properties after neutron irradiation has not been discussed from the viewpoint of irradiation-induced microstructural change.

In the present study, microstructure and mechanical property after neutron irradiation have been studied. The primary concern is the relationship between the irradiation-induced amorphization and the characteristic change of mechanical property by neutron irradiation.

2. Experimental

Three kinds of Ti-Ni alloy with different compositions and transformation temperatures were used for this experiment. They were kindly supplied by Japan Stainless Steel Co. Ltd. Energy dispersive X-Ray spectroscopy (EDS) revealed the

Table. 1 Compositions, transformation temperatures and structure at room temperature of unirradiated specimens. Composition was revealed by EDS.

Composition	Ms	Af	Structure at R.T.
Ti - 51at.%Ni	337K	373K	B19' Martensite
Ti - 52at.%Ni	298K	338K	B2
Ti - 53at.%Ni	267K	308K	B2

compositions were Ti-51, 52 and 53at.%Ni. Their inhomogegeneity micro-chemical composition was within approximately 1%. The composition and transformation temperatures before irradiation are shown in Table.1.

All of the neutron irradiations were performed in hydrorabit capsules in cooling water. The doses were 1.4×10^{16}, 1.3×10^{18} and $1.2 \times 10^{19} n/cm^2$. The irradiation temperature was under 423 K. The irradiation periods were 0.6ks, 3.6ks and 515.4ks, respectively.

After the irradiation, the microstructural changes were observed by 200keV transmission electron microscope (TEM) at room temperature. Tensile tests were performed with a strain rate of $2.8 \times 10^{-4}/s$ at room temperature.

3. Results

3.1 Microstrutural Change

Fig.1 shows the microstructures and electron diffraction patterns at room temperature after irradiation. The
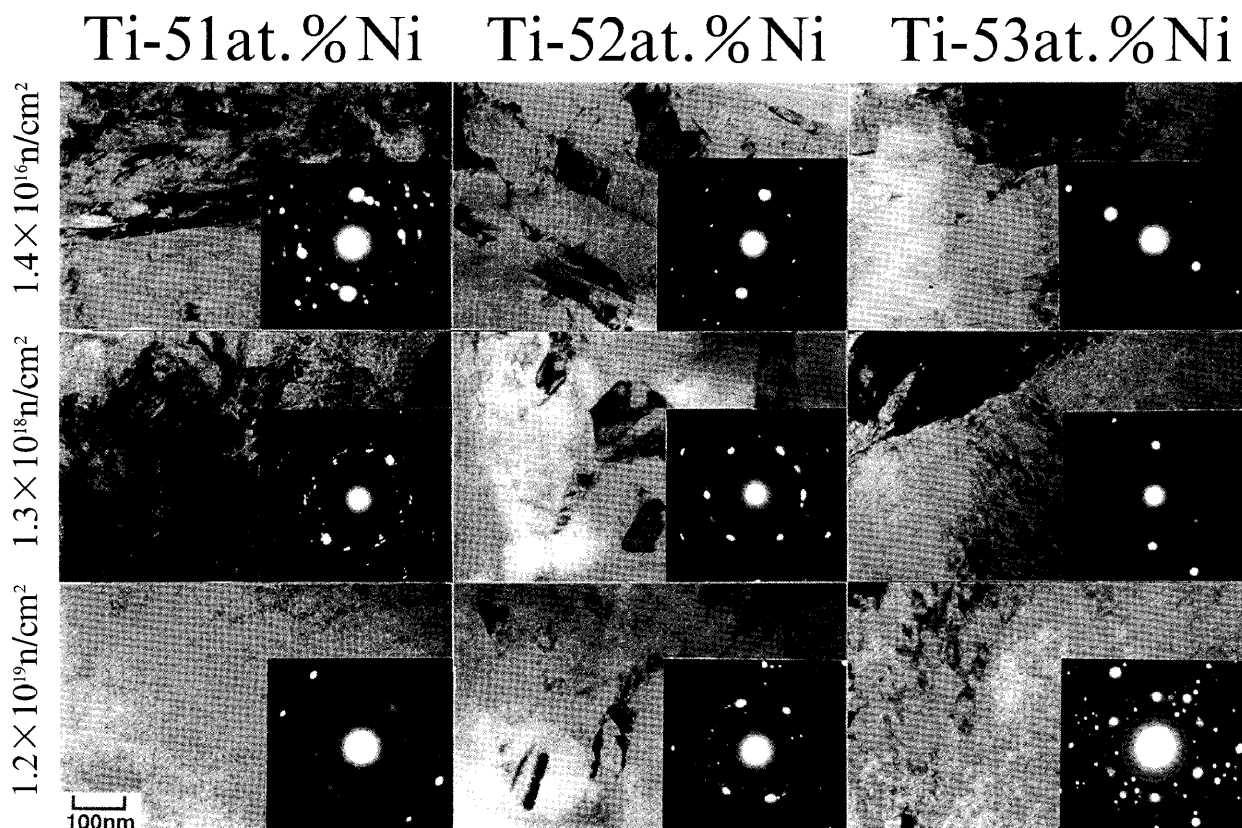
Ti-51at.%Ni   Ti-52at.%Ni   Ti-53at.%Ni

Fig.1 Microstructures and SAD patterns of neutron irradiated Ti-51at.%Ni, Ti-52at.%Ni and Ti-53at.%Ni. Neutron doses were 1.4×10^{16}, 1.3×10^{18} and 1.2×10^{19} n/cm^2. Irradiation temperature was under 423K.

specimens of Ti-51at.%Ni was martensitic phase before irradiation. There was no change in structure after the irradiation up to 1.3×10^{18}n/cm^2. However, the structure after the irradiation of 1.2×10^{19}n/cm^2 turned to B2 phase. In addition, a halo ring was developed in selected area diffraction pattern (SAD). Both of Ti-52 and 53at.%Ni were B2 phase before irradiation, and the irradiation did not produce clear changing in microstructure, however, a halo ring was observed also in each of them.

Fig.2 shows the dark field image from a halo ring. It was confirmed that the fine amorphous particles with the order of 10 Å were formed.

In Ti-53at.%Ni after the irradiation up to 1.2×10^{19}n/cm^2, extra diffraction spots excepting B2 reciprocal lattice spots and a halo ring were also observed. This suggests that another phase excepting B2 phase and amorphous phase existed in the specimen. It was not confirmed whether the phase existed before the irradiation or precipitated during the irradiation, however, the annealing experiment without neutron irradiation revealed a metastable phase precipitated as fine particles around 523K. This suggests that the phase precipitated possibly during the irradiation. The details will be discussed in another paper[6].
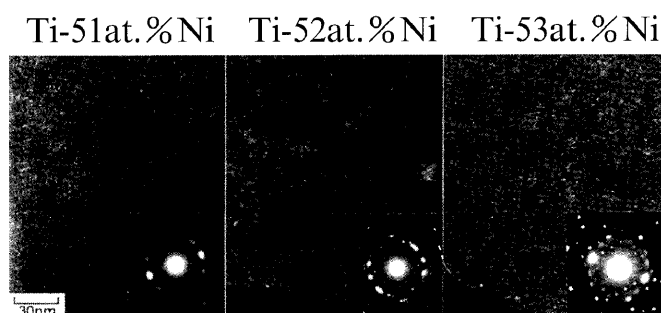
Ti-51at.%Ni   Ti-52at.%Ni   Ti-53at.%Ni

Fig.2 Dark field images from halo ring of Ti-51, 52 and 53at.%Ni irradiated to 1.2×10^{19} n/cm^2.

3.2 Mechanical Property

Fig.3 illustrates the stress-strain curve during straining up to 5% and then releasing. In Ti-51 and 52at.%Ni, unirradiated and irradiated up to 1.3×10^{18}n/cm^2, strain remained with about 2% after releasing stress. In only unirradiated Ti-53at.%Ni, the strain of approximetry 2% remained. After the irradiation up to 1.3×10^{18}n/cm^2, the stress-strain curve showed pseudoelasticity, that is, strain was recovered almost completely by releasing the applied stress.
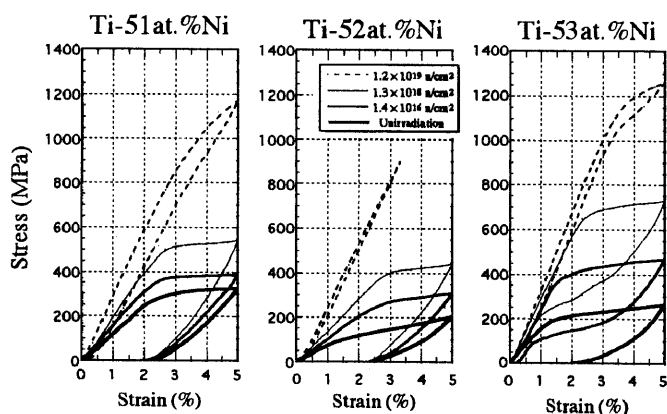
Fig.3 Stress-strain curve of unirradiated and neutron irradiated Ti-Ni. Neutron flux were 1.4×10^{16}, 1.3×10^{18} and 1.2×10^{19} n/cm^2. Irradiation temperature were under 423K.

The characteristic change in stress-strain curve after irradiation is supposed to be associated with the shifts of the martensitic and its inverse transformation temperatures[8]. In general, the character of stress-strain curve is governed by the transformation temperatures. Below Ms, the character is shape memory effect, in which strain remains when the applied stress is released, and it can recover almost completely by heating above Af point. Between Af and Md, the character is pseudoelasticy, in which strain can recover almost completely by releasing the applied stress. It was confirmed that the transformation temperatures were shifted to lower temperature side by irradiation[1)2)]. Therefore, it could reasonably be understood that the characteristic change of the stress-strain curve derived from the shift of Af to below room temperature.

After the irradiation to $1.2 \times 10^{19} n/cm^2$, the strain of each alloy was recovered almost completely by releasing the applied stress without strain hysterisis. The absense of the strain hysterisis is never explained by the shift of the transformation temperatures. Transformation temperatures have no effect on decreasing the strain hysterisis.

4. Discussion

Fig.4 are plots of the critical point of stress strain curve (σ M) and $\Delta \sigma$ M at different doses, respectively. σ M has different meaning depending on the transformation temperatures[8]. Above Md, σ M is the critical stress for the starting of plastic deformation in B2 phase. Between Af and Md, σ M is the critical stress for stress-inducing martensite. Below Af, σ M is the critical stress for reorientating martensite. Excepting the case above Md, the stability of martensite domains play an essential role for behavior of σ M. It is obvious in Fig.4 that σ M increased as the dose increased. At the same time, fine amorphous phase formed by neutron irradiation in this specimen. Therefore, it could reasonably be understood that the mechanism whereby σ M increased with the increase of dose was the pinning martensite domains by amorphous phase.

The increament of σ M showed composition dependence. From the assumption that the increase of σ M is caused by the formation of amorphous phase, the composition dependence of $\Delta \sigma$ M would correspond to the composition dependence of the irradiation-induced amorphization. In fact, the result of electron irradiation showed same tendancy in irradiation-induced amorphization[9], that is, the deviation from stoichiometric composition makes amorphization easy.

Fig.5 are plots of strain hysterisis (ε H) and $\Delta \varepsilon$ H at different doses, respectively. The ε H decreased drastically by irradiation around $10^{19} n/cm^2$. From the experimental results, the following mechanism on the decreasing in ε H can be proposed.

After the irradiation of $1.2 \times 10^{19} n/cm^2$, the σ M would be the critical stress for the starting of plastic deformation in B2 phase. It was clarified by differential scanning calorimetry (DSC) that no martensitic transformation occurred in this extent of dose[1], therefore, it is supposed that stress-inducing martensitic transformation would not occur, either. As mentioned above, 5% strain could recover by expansion of elasticity limit in B2 phase. The similar behavior has been observed in cold worked Ti-Ni[8], however, it would be different from the behavior in neutron irradiated Ti-Ni. In case of cold worked Ti-Ni, it was revealed that the initial structure before loading stress was martensitic phase[10,11]. Therefore, the recovery mechanism of a few percentage level strain would be different. The extraordinal phenomenon observed in neutron irradiated Ti-Ni is supposed to be close to the fact that a halo ring was only observed after the irradiation of $1.2 \times 10^{19} n/cm^2$, however, it need to obtain further experimental data in order to discuss the mechanism in detail.
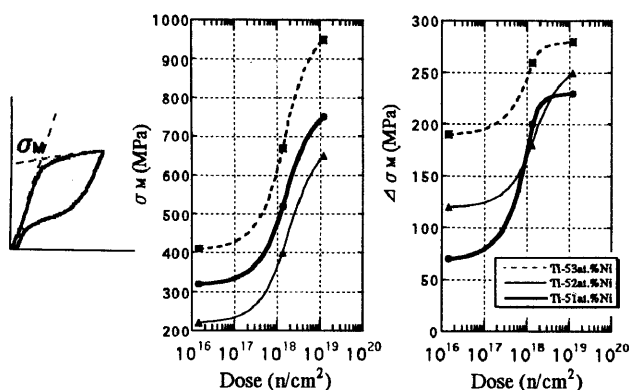
Fig.4 σ M and $\Delta \sigma$ M of neutron irradiated Ti-51, 52 and 53at.%Ni.
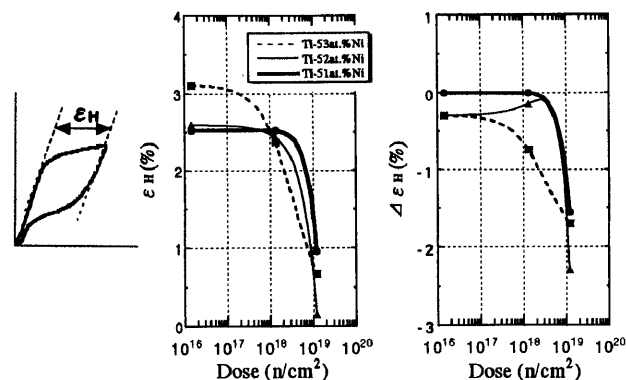

Fig.5 ε H and $\Delta \varepsilon$ H of neutron irradiated Ti-51, 52 and 53at.%Ni.

Summary

Microstructural change and mechanical property of neutron irradiated Ti-Ni were investigated. The results are summarized as follows:

(1) Fine amorphous particles were observed after the neutron irradiation of 1.2×10^{19}n/cm^2.

(2) The σ M increased as the dose increased, and $\Delta \sigma$ M showed composition dependence.

(3) The stress-strain curve was almost linear at the dose of 1.2×10^{19}n/cm^2.

These results indicate that irradiation-induced amorphization can produce the characteristic change of the mechanical properties of this alloy.

Acknowledgements

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