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Microstructure and mechanical properties of neutron irradiated TiNi shape memory alloy

Y. Matsukawa [a,b,*], T. Suda [a], S. Ohnuki [a], C. Namba [b]

[a] *Faculty of Engineering, Hokkaido University, Sapporo N-13 W-8, 060, Japan*
[b] *National Institute for Fusion Science, Toki, 509-52, Japan*

Abstract

Microstructure, mechanical properties and transformation temperature of neutron irradiated TiNi shape memory alloy have been investigated. The doses were on the order of 10^{20} to 10^{23} n/m^2. All irradiations were performed below 423 K. Amorphization was confirmed by TEM after the irradiation of 1.2×10^{23} n/m^2. The recovery behavior of the applied strain was drastically changed after the irradiation. The DTA peak slightly shifted to lower temperature and vanished. These results indicate that amorphous phase dominates the suppression of the martensitic transformation, and causes the change in the mechanical properties.

1. Introduction

TiNi shape memory alloy is expected to be used in nuclear reactor environment [1]. Many researchers have examined the stability of its mechanical properties during neutron irradiation. Previous work has revealed that TiNi irradiated with high energy particles showed some peculiar behavior [2–4]. Differential scanning calorimetry (DSC) measurements revealed that the martensitic and its inverse transformation temperatures (Ms and Af) shifted to the lower temperatures [2,3]. An extraordinary pseudoelastic behavior was observed in TiNi irradiated at high dose [2,4]. These results implied that the shape memory effect and the pseudoelasticity were affected by high energy particle irradiation; however, the mechanism is not fully understood now. Despite TiNi being well known as an intermetallic compound in which amorphization occurs during electron irradiation [5,6], the correlation between such irradiation-induced microstructural change and the change in the mechanical property is not well understood.

In the present study, the microstructure, the mechanical properties and the transformation temperature of neutron irradiated TiNi shape memory alloy have been investigated. The primary concern is to clarify the mechanism whereby the mechanical property changes after neutron irradiation.

2. Experiments

Three kinds of TiNi alloy with different composition and transformation temperatures were used for this experiment. They were kindly supplied by Japan Stainless Steel. Minor amounts of a Ti_4Ni_2O phase were also present as an impurity and could clearly distinguished from the TiNi phase. The composition of the TiNi phase was determined by Electron Probe Micro Analyzer (EPMA) with a calibration by the sample of well-established composition. The compositions were Ti-50.0, 50.5 and 51.0 at.% Ni. The transformation temperature and the structure at room temperature before irradiation are shown in Table 1. The alloys were delivered as sheets with thickness of 0.2 mm. They were cut into discs with diameters of 3 mm and tensiles with size of 16 mm × 4 mm.

The all neutron irradiation was performed at Japan Materials Testing Reactor (JMTR). The doses were 1.4×10^{20}, 1.3×10^{22} and 1.2×10^{23} n/m^2 (>1 MeV). The irradiation was carried out in hydro-rabit capsule, where the irradiation temperature was estimated below 423 K. The alloys were kept at this temperature during the irradiation for 0.6, 3.6 and 515.4 ks, respectively.

* Corresponding author. Tel.: +81-11 706 6771; fax: +81-11 706 6772; e-mail: matsu@loam.metall.hokudai.ac.jp

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Table 1
Composition, transformation temperature and structure at room temperature before neutron irradiation of alloys

Composition	Transformation temperature (K)		Structure at RT
	Ms	Af	
Ti–50.0 at.% Ni	337	373	B19' martensite
Ti–50.5 at.% Ni	298	338	B2
Ti–50.0 at.% Ni	267	308	B2

Composition, transformation temperature and structure at room temperature before neutron irradiation of alloys. (Composition was revealed by means of EPMA).

After the irradiation, the microstructure were observed by 200 keV Transmission Electron Microscope (TEM) at room temperature. Tensile tests were performed at room temperature with strain rate of 2.8×10^{-4}/s. Differential thermal analysis (DTA) was performed in order to check the transformation temperature after the irradiation. Since the temperature range measured by DTA was only above room temperature, we measured only the inverse transformation temperature of Ti–50.0 at.% Ni. The heating rate was 10 K/s.

3. Results

3.1. Microstructure

Fig. 1 shows typical microstructures and corresponding electron diffraction patterns for each dose. In each of the three compositions, there was no obvious microstructural change after the irradiation of up to 1.3×10^{22} n/m^2. However, a halo ring appeared in the diffraction pattern of each alloys after an irradiation of 1.2×10^{23} n/m^2. This means that amorphization was induced by the neutron irradiation. Fig. 2 shows the dark field image from the halo ring. The amorphous phase was found to exist as fine particles with diameters of ~10 nm.

3.2. Mechanical property

Fig. 3 shows the stress–strain curve in which the alloys were strained up to 5% and then the applied stress was released. In Ti–50.0 and 50.5 at.% Ni samples irradiated up to 1.3×10^{22} n/m^2, a strain of approximately 2% remained after releasing the applied stress. The strain of 2% also remained in unirradiated Ti–51.0 at.% Ni, however, the strain almost completely recovered in

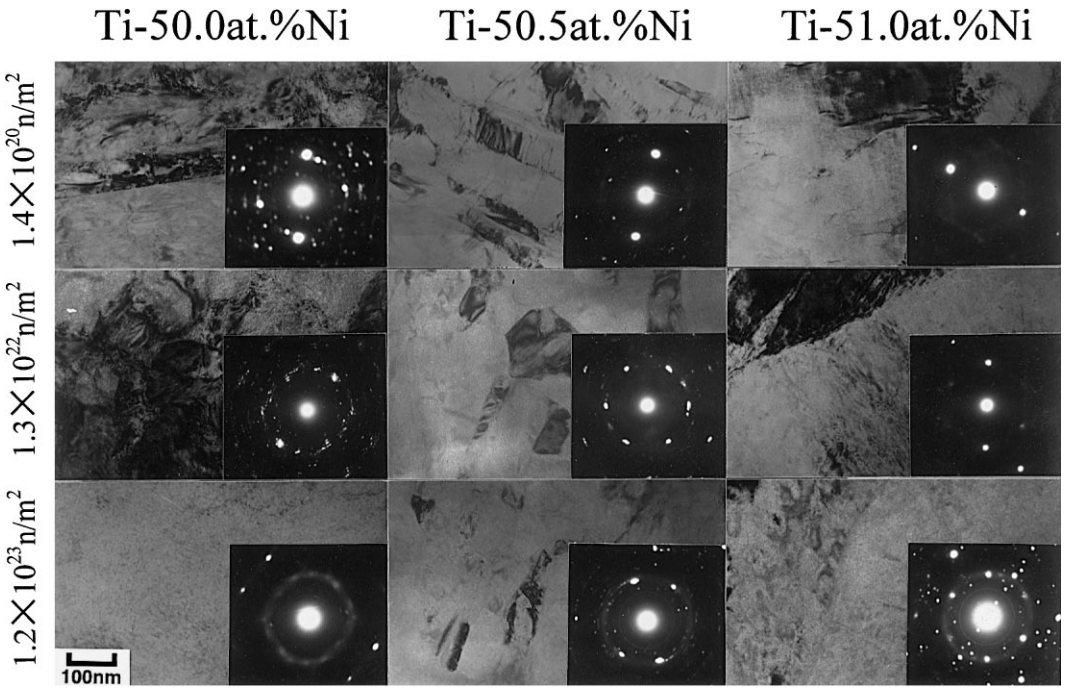

Fig. 1. Microstructure and corresponding electron diffraction pattern of neutron irradiated TiNi. Neutron doses were 1.4×10^{20}, 1.3×10^{22} and 1.2×10^{23} n/m^2. All irradiation was performed below 423 K.

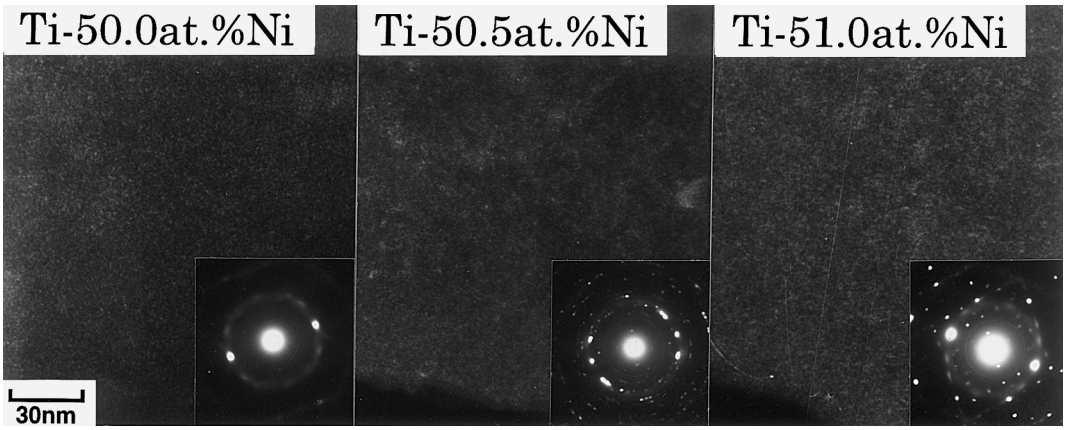

Fig. 2. Dark field image from the halo ring of the alloys irradiated 1.2×10^{23} n/m^2.

irradiated alloys. This implies that Ti–51.0 at.% Ni showed pseudoelasticity after the irradiation.

After irradiation of 1.2×10^{23} n/m^2, the strain was recovered almost completely by releasing the applied stress irrespective of the composition of the alloy. The behavior is just the pseudoelasticity from the viewpoint of strain recovery, however, the strain hysterisis was quite less than the one in conventional pseudoelasticity.

3.3. Transformation temperature

Fig. 4 shows the DTA curve of unirradiated and neutron irradiated Ti–50.0 at.% Ni. Since these are the curves generated during the heating process, the peaks on each curve depend on the inverse martensitic transformation.

The peaks slightly shifted from its initial position to lower temperatures and split after an irradiation at low dose. There was no peak at the highest dose. Comparing the peaks of the irradiation of 10^{20} and 10^{22} n/m^2, their deviation from the initial position were almost the same, and total amount of the intensity decreased with increasing dose. Therefore, the peak would vanish at the site without further shifting to the lower temperatures.

4. Discussion

4.1. Suppression of martensitic transformation

In the present study it was revealed that the DTA peak shifted to the lower temperatures side simulta-

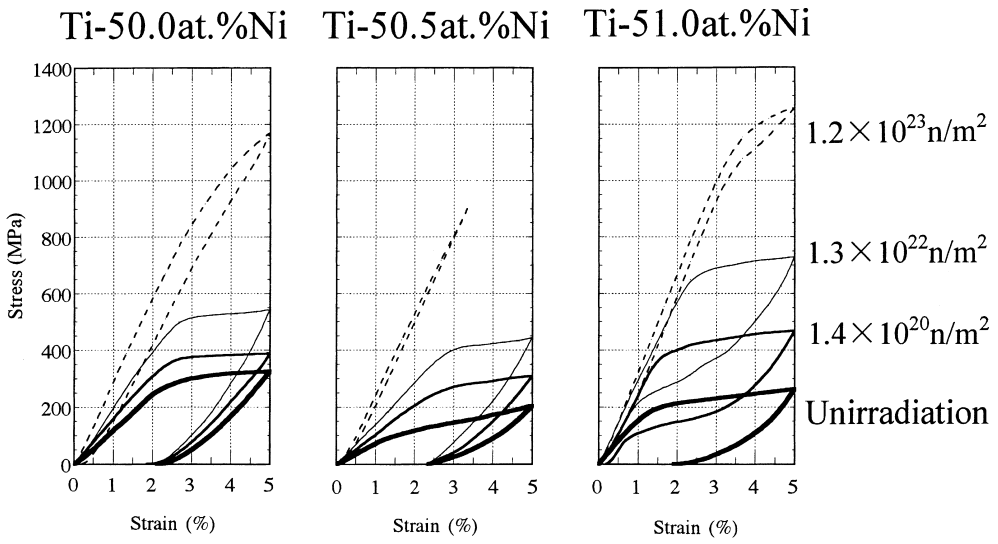

Fig. 3. Stress–strain curve of unirradiated and neutron irradiated TiNi. Neutron does were 1.4×10^{20}, 1.3×10^{22} and 1.2×10^{23} n/m^2. All irradiation was performed below 423 K.

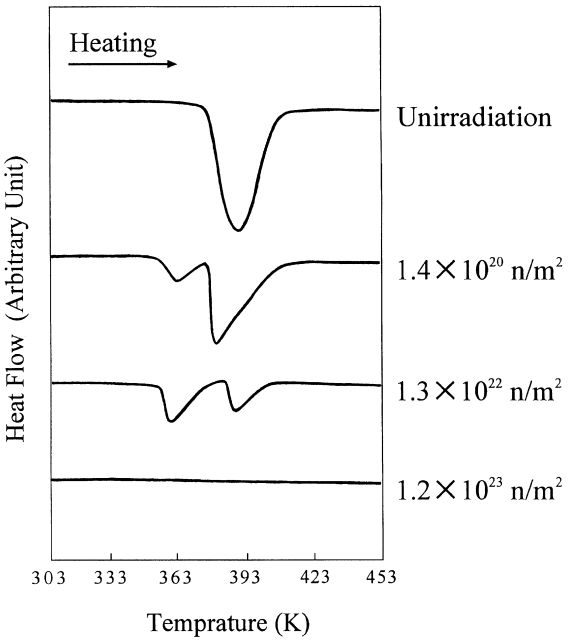

Fig. 4. DTA curve during heating of unirradiated and neutron irradiated Ti–50.0 at.% Ni. Neutron dose were 1.4×10^{20}, 1.3×10^{22} and 1.2×10^{23} n/m^2. All irradiation was performed below 423 K.

neously with decreasing intensity. Both the shift and the decrease in the intensity indicate the suppression of the martensitic transformation, however, each mechanism would be different. In general, the suppression of the martensitic transformation after neutron irradiation has been explained to result from the decrease in the chemical order [2,4]. The shift can be explained by a chemical disordering, however, there is no experimental evidence that it brings the decrease in the intensity. Therefore, the chemical disordering is not sufficient to explain the suppression of the martensitic transformation after the irradiation.

Based on the results of TEM observation, in which amorphization was induced by irradiation, the amorphization would not be irrelevant to the suppression of the martensitic transformation. On the assumption that the amorphization dominates the suppression of the martensitic transformation, both the shift and the decrease in the intensity can reasonably be understood. The amorphization is a topological disorder. Since it would make the degree of Bragg–Williams order decrease, the peak can shift to lower temperatures. The intensity of DTA peak would correspond to the amount of the martensitic transformation, therefore, the following two possible mechanisms are proposed: One is that the amount of amorphous phase directly corresponds to the decrease in the intensity. The other is that a stress-field around the amorphous area has a suppressing effect on the martensitic transformation.

Kimura et al. [2] have reported the recovery behaviors of the martensitic transformation by annealing after neutron irradiation. The DSC analysis showed that the martensitic transformation was suppressed completely after an irradiation of 10^{24} n/m^2 and it recovered after a post-annealing at 548 K. The temperature of 548 K is supposed to corresponds to the crystallization temperature of the amorphous phase in this alloy [7].

4.2. Pseudoelastic behavior

Irrespective of the composition, TiNi irradiated to the highest dose showed extraordinary pseudoelastic behavior. It was almost the same as conventional pseudoelasticity concerning the recoverable strain, but was different characterized by shape of the stress–strain curve. The stress–strain curve of the highest dose was almost linear without strain hysterisis. In the curve of Ti–51.0 at.% Ni irradiated to the highest dose, the strain hysterisis is small, and the area of the elastic deformation is larger than the one of Ti–51.0 at.% Ni irradiated to lower dose. Considering the absence of the DTA peak in TiNi irradiated at 10^{23} n/m^2, a stress-induced martensitic transformation would not occur after irradiation to high dose. Therefore, the extraordinary pseudoelastic behavior is not conjectured to be the pseudoelasticity relying on martensitic transformation but to be the expansion of recoverable elastic deformation of the B2 phase. Given that such an extent of strain can recover as elastic deformation, the amorphous phase dispersed in the B2 matrix is possibly related to the expansion of the elastic deformation.

In amorphous materials, it is well known that the extent of such a strain can recover elastically. Recently, it has been reported that thin foil amorphous TiNi shows the behavior [8]. The fact can support this hypothesis that the amorphous phase formed after neutron irradiation can influence the mechanical properties.

5. Conclusion

The microstructure, the mechanical properties and the transformation temperature of neutron irradiated TiNi were investigated. The results are summarized as follows:

(1) Amorphous phase was observed after an irradiation of 1.2×10^{23} n/m^2.

(2) The DTA peak after an irradiation of low dose decreased in the intensity with slight shifting to lower temperatures.

These results indicate that the suppression of martensitic transformation after the irradiation is associated with the irradiation-induced amorphization.

(3) Irrespective of the composition, the alloy irradiated 1.2×10^{23} n/m^2 showed extraordinary pseudoelastic behavior.

The behavior is not the pseudoelasticity relying on the martensitic transformation, but due to the expanded elastic deformation of the B2 phase. The expansion of elastic deformation is supposed to arise from the formation of the amorphous phase induced by neutron irradiation.

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