



Effects of residual strain on deformation processes of neutron-irradiated Ti–Ni and Ti–Pd shape memory alloys

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Abstract

Ti–Ni and Ti–Pd shape memory alloys (SMA) reveals good workability and shape memory properties and these SMAs seem to be one of hopeful functional materials in a severe irradiation field. The effects of irradiation induced residual strain on the deformation processes of Ti–Ni and Ti–Pd SMAs after neutron irradiation with fluences ($E > 1$ MeV) up to $3.9 \times 10^{24} \text{ m}^{-2}$ at Japan Materials Testing Reactor (JMTR) were investigated by the remote controlled X-ray diffraction measurement. Residual strains of Ti–Ni SMAs took place over damage of 0.1 dpa and the strains were not completely removed by post-irradiation annealing at 473, 523 and 573 K. On the other hand, residual strains of Ti–Pd SMAs scarcely occurred after irradiation, and furthermore, the Ti–Pd SMA seems to be an irradiation-resistant material. This may be explained by a difference between the irradiation response of a parent phase and that of a martensitic phase to neutron irradiation. Deformation processes of SMAs are associated with stress fields generated by irradiation in a parent phase with B2 type ordered structure or in a martensitic phase with 2H and 9R type close-packed structures.

1. Introduction

TiPd based alloys exhibit the shape memory effect even at high temperature range up to 810 K in the study of the martensitic transformation of the TiNi–TiPd pseudobinary alloys [1]. However, detailed information on high temperature mechanical properties is not clear by tensile tests. In the previous report, internal structures of the martensitic phases in TiPd–Fe and TiPd–Cr alloys were clarified by microstructure observation using TEM and the 2H, 9R and incommensurate phases were found to exist when the content of the third element (Cr or Fe) was larger than a certain amount [2]. It is also confirmed that the shape memory effect exists even at high temperatures by high

temperature compression tests [3]. To develop the high temperature shape memory alloys using TiPd based alloys, detailed mechanical data are useful on deformation behavior at high temperatures. The effect of third element addition on the deformation behavior at high temperatures is quite an important item to establish how to use these promising alloys in a high temperature environment.

Simple and quick replacement of coupled components in fission and fusion reactors under irradiation has been identified as important for improving operational efficiencies. For this purpose, high temperature shape memory alloys have been considered to be desirable. Few studies have been carried out to investigate systematically neutron-irradiation-associated phase transformation that Ti–Pd shape memory alloys may reveal. The purpose of this study is to investigate the relationships between deformation behavior and internal stress of neutron-irradiated Ti–Pd high temperature shape memory alloys.

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2. Experimental procedures

Ingots were prepared by arc melting under argon atmosphere. The nominal composition of the alloys used is $\text{Ti}_{50}\text{Pd}_{50-x}\text{Cr}_x$ ($x = 0, 1, 2, 3, 4$ at.% Fe) and $\text{Ti}_{50}\text{Pd}_{50-x}\text{Fe}_x$ ($x = 0, 2, 3, 5, 6, 7$ at.% Fe). Specimens for DSC measurement and for tensile test were cut by spark-cut from the sheets with a thickness of 1.0 mm, and then, were sealed off in argon-filled quartz capsules and were homogenized at 1373 K for 600 s followed by water quenching without breaking capsules. Transformation temperatures of specimens before irradiation were determined by the DSC measurement.

Neutron irradiation for TiPd–Cr alloys was carried out with a fast neutron fluence ($E > 1$ MeV) of $3.9 \times 10^{24} \text{ m}^{-2}$ at a temperature of 490 K in Japan Materials Testing Reactor (JMTR) of Japan Atomic Energy Research Institute (JAERI). After irradiation, high temperature tensile tests were carried out in a temperature range between ($A_s - 50$ K) and ($A_s + 50$ K) by using a remote controlled tensile machine. Specimens were heated by a remote controlled electric furnace with argon flow system. Tensile stress was loaded until a strain of 2% was attained and then, was unloaded and heated up to a temperature of ($A_f + 100$ K). Residual strain was determined after heating. The X-ray diffractometry were carried out by using the remote controlled X-ray diffraction machine with operating voltage at 40 kV and 40 mA. $\text{Ti}_{50}\text{Ni}_{50}$ specimens irradiated with $3.0 \times 10^{24} \text{ m}^{-2}$ at 323 K in JMTR were also prepared as a reference specimen for the X-ray diffractometry.

3. Results and discussion

3.1. Deformation behavior of TiPd containing chromium or iron

Transformation temperatures of unirradiated alloys are shown in Fig. 1, which were determined by DSC measurement. Transformation temperatures decreased with increasing chromium or iron content. M_s temperatures were linearly changed from 806 to 580 K by chromium addition.

Fig. 2 shows the stress–strain curves of the TiPd–3Cr alloy at temperatures between 626 and 726 K. When the chromium content was increased to 3 at.%, the positive temperature dependence of yield stress was confirmed at temperatures above A_s ($A_s = 677$ K). This temperature dependence is due to formation of stress-induced martensites. A slight amount of the residual strain around 0.7% was observed as shown in the figure. A small amount of residual strain was reported in the solution-treated TiNi alloys [4]. In the present study, specimens were solution-treated at 1373 K. This residual strain can be explained by the stabilization of the martensitic phase. This suggests that some kinds of thermomechanical treatments for TiPd

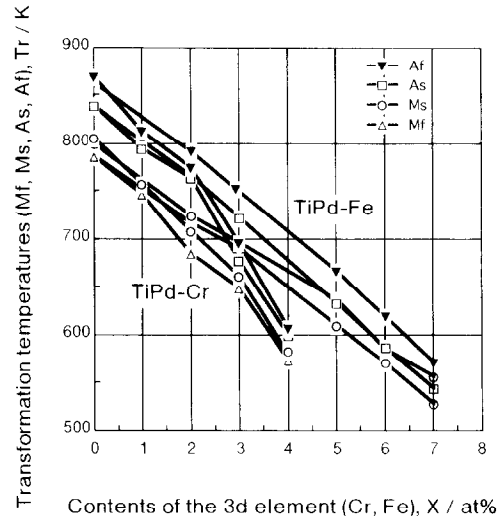


Fig. 1. Transformation temperature versus content of the third element (chromium or iron) for TiPd based alloys.

based alloy are effective to improve the shape memory capabilities. No pseudoelasticity was observed in tensile tests. In the preliminary study, it was considered that this was due to the artifact of the extensometer system of measurement of the strain at high temperature used. Thick specimens with a thickness of 6.0 mm were used in compression tests and that a sort of retardation effect of pseudoelasticity was observed [3]. It was supposed that TiPd based alloys have a pseudoelastic effect at temperatures above A_f . In the present study, recoverable strain due to pseudoelasticity seems to exist, and may be counterbalanced out by the plastic strain which were caused by slip deformation at high temperatures. These characteristic features of TiPd–Cr or TiPd–Fe alloys between the critical stress of stress-induced martensites and the test temperature are summarized in Fig. 3a.

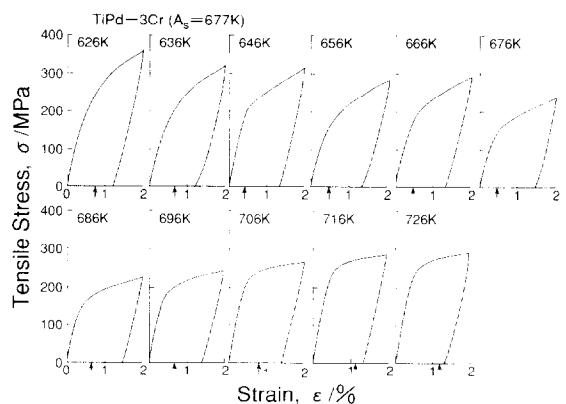


Fig. 2. Stress–strain curves of TiPd–3Cr alloys at temperatures between 626 and 726 K.

Recoverable strains as a function of testing temperature for the TiPd–Cr or TiPd–Fe alloy were shown in Fig. 3b. From the point of view of the residual strain, recoverable strain depends on the amount of deformation. In the previous paper, the residual strain over 2% for TiPd based alloy did not recover completely when the specimen was heated up to A_f [5]. This may be due to the stabilization of martensities caused by heavy deformation in the thick specimens. In the present study, the relative recoverable strain was determined as the ratio of amount of recovered strain divided by the amount of applied initial strain (2%) that were measured after unloading and heating. Relative recoverable strains increased with increasing chromium or iron content. At temperatures below A_s , the amount of shape recovery due to shape memory effect depends on content of addition. For higher chromium content alloys, the relative recoverable strain has a maximum of 90%. And then, recoverable strain decreased suddenly at temperatures around A_s . When the temperature was increased over A_s , the higher chromium content alloy (TiPd–3Cr and TiPd–4Cr) behaved pseudoelastically. The recoverable strain due to pseudoelasticity began to fall down in a case that the temperature difference of $(T - A_s)$ was attained at 40 K. The above results suggest that the TiPd–Cr or TiPd–Fe alloys reveal good properties of shape recovery although the additional thermo-mechanical treatment such as aging and rolling would be needed for improving the shape recovery due to shape memory effect and pseudoelasticity. It is concluded that TiPd based alloy has enough possibility as high temperature shape memory alloy if we choose a proper chromium content and a thermo-mechanical treatment.

Fig. 4 shows the stress–strain curves of TiPd–3Cr alloys deformed at temperatures between 626 and 726 K

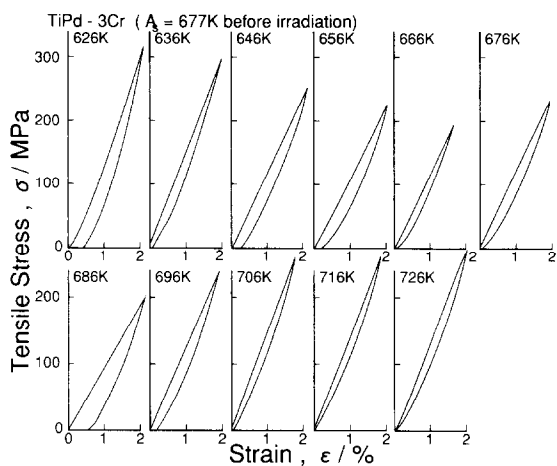


Fig. 3. 0.2% offset stress and relative recoverable strain as a function of test temperature for TiPd based alloys containing chromium or iron.

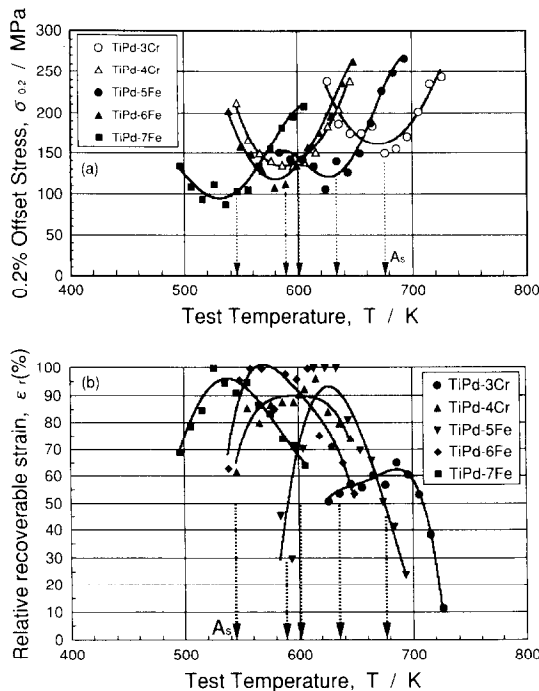


Fig. 4. Stress–strain curves of TiPd–3Cr alloys at temperatures between 626 and 726 K after neutron irradiation.

after irradiation with fast neutron fluences of $3.9 \times 10^{24} \text{ m}^{-2}$. The irradiation temperature was 490 K. Stress–strain curves had a minimum at 666 K and showed the pseudoelasticity induced by irradiation. The positive temperature dependence of tensile stress was pronounced at temperatures above A_s ($A_s = 677 \text{ K}$ before irradiation). This temperature dependence is due to the formation of stress-induced martensites. The hysteresis of the stress–strain curves showed very narrow area. Irradiated alloys behaved pseudoelastically at each temperature and revealed the irradiation-induced pseudoelasticity similar to that of an irradiated TiNi alloy [6]. It is confirmed that by neutron irradiation at 490 K, specimens have pseudoelastic effect on deformation behavior, and thus, had quite a few influences on martensitic transformation. This can be explained by the irradiated state at which disordering caused by irradiation were balanced out ordering facilitated by vacancy migration [7].

Fig. 5 showed the Young’s modulus versus temperature curves for TiPd–Cr alloys irradiated by neutrons with fluences of $3.9 \times 10^{24} \text{ m}^{-2}$. The positive temperature dependence of Young’s modulus was observed at temperatures above A_s in the case of higher chromium content alloys. This is due to the lattice instability of the parent phase. Before and after irradiation, the lattice instability was observed at temperatures above A_s , and it indicates

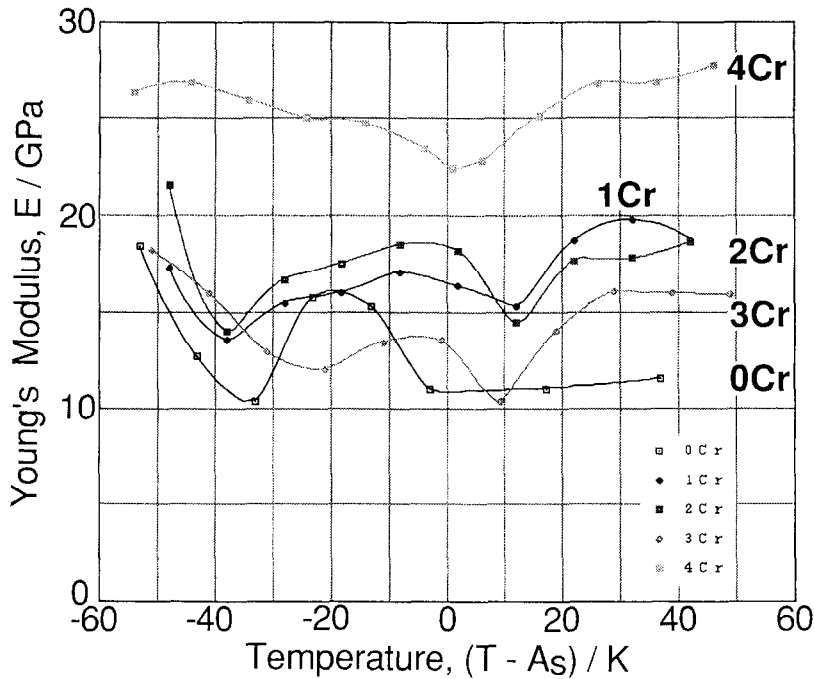


Fig. 5. Young's modulus versus test temperature curves for TiPd–Cr alloys irradiated by neutrons with fluences of $3.9 \times 10^{24} \text{ m}^{-2}$.

that lattice instability due to martensitic transformation exists even after irradiation. In these irradiation conditions, an irradiation temperature of 490 K is somewhat lower than a temperature around 520 K at which restoration phenomena of damaged state into normal state can take place in the B2 phase. The displacement of 10^{-1} dpa is larger than that of 10^{-2} dpa which corresponds to the threshold value for restoration phenomena [8]. These irradiation conditions may not correspond to the threshold value for the restoration. As a result, the deformation process of TiPd–Cr alloys after irradiation reveals unique behavior characterized by the irradiation-induced pseudoelasticity.

3.2. X-ray diffractometry of TiNi and TiPd–Cr

Fig. 6 shows the X-ray diffraction pattern of TiNi specimens as a function of neutron fluence. In the TiNi specimens, phase change took place over $1 \times 10^{22} \text{ m}^{-2}$ ($\sim 10^{-2}$ dpa) of a high temperature phase (parent phase) to a low temperature phase (martensitic phase). This means that the transformation temperature slightly increased and furthermore decreased over $1 \times 10^{23} \text{ m}^{-2}$ ($\sim 10^{-1}$ dpa). The diffraction pattern shows the intense peak around 42° which corresponds to the main peak of the parent phase. The strong broadening of peak was observed at fluences of $3 \times 10^{24} \text{ m}^{-2}$ as shown in Fig. 6a. This indicates the existence of residual strain after irradiation. These strains did not disappear after post-annealings at 573 K (Fig. 6b).

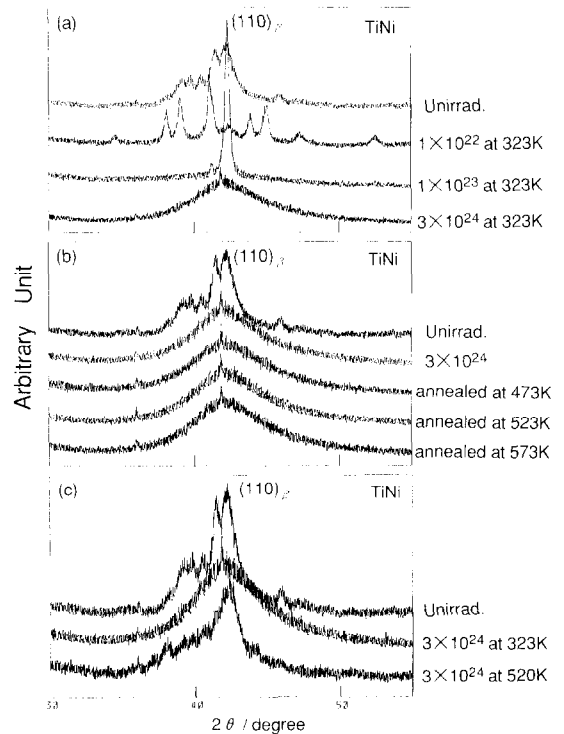


Fig. 6. X-ray diffraction patterns of TiNi alloys irradiated by neutrons with fluences of $3 \times 10^{24} \text{ m}^{-2}$ as functions of neutron fluence, irradiation temperature and annealing temperature.

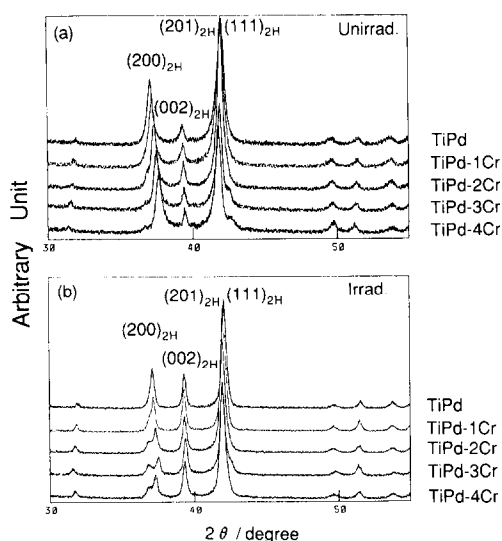


Fig. 7. X-ray diffraction patterns before and after irradiation of TiPd–Cr alloys irradiated by neutrons with fluences of $3.9 \times 10^{24} \text{ m}^{-2}$.

From the view point of restoration phenomena [8] in TiNi specimens, 520 K irradiation may be effective rather than post-annealings (Fig. 6c). Fig. 7 reveals the diffraction patterns of TiPd–Cr alloys irradiated with fluences of $3.9 \times 10^{24} \text{ m}^{-2}$ at 490 K. No broadening of peaks was observed in respective content alloy. Therefore, it is confirmed that the TiPd–Cr alloys seem to be new material with irradiation-resistance and without residual strain due to neutron irradiation.

4. Conclusions

Tensile tests at high temperatures were performed for irradiated TiPd– x Cr ($x = 0, 1, 2, 3, 4$ at% Cr) alloys by

neutrons with fluences of $3.9 \times 10^{24} \text{ m}^{-2}$ at temperatures between 546 and 886 K. X-ray diffractometry was carried out for irradiated TiNi and TiPd–Cr alloys. The obtained results are as follows:

(1) From Young's modulus versus temperature curves, TiPd–3Cr and TiPd–4Cr alloys show the irradiation-induced pseudoelasticity even at enough high temperatures above 600 K. This is caused by the presence of back stress at defects.

(2) From the X-ray diffractometry, TiNi alloys shows the strong broadening caused by the existence of residual strain. On the other hand, no broadening was observed in TiPd–Cr alloys after irradiation.

(3) TiPd–Cr alloys can be used as high temperature shape memory alloys with irradiation resistance against neutron irradiation if an appropriate irradiation condition is selected.

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