Transactions of the Materials Research Society of Japan 29[7] 2985-2988 (2004)

Joule Heat Induced Shape Memory Behavior of Ti-Ni Shape Memory Alloy Thin Films

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The Joule heat induced shape memory behavior of Ti-Ni thin film has been investigated using a specially designed tensile tester. Specimens were 5 μm-thick Ti-47~52at%Ni films deposited onto Cu foils by dc magnetron sputtering. These films were annealed at 600°C for 60 min for crystallization and memorization into a flat shape after dissolving the Cu substrates in a dilute HNO_3 solution.
Ti-52at%Ni film showed the superelasticity, whereas Ti-47~50at%Ni films showed the shape memory effect at RT. These results agreed with the transformation temperatures of the films measured by DSC. In order to study the effect of total input energy given by an electrical current pulse on the shape memory behavior, recovery strains of the films at a constant stress were measured with changing the duration of current pulse. The Ti-46.8~49.4at%Ni films showed a perfect shape recovery under a constant stress by applying the total input energy of less than 0.1 J. The extended test of cyclic shape memory effect revealed that the Ti-48.4at%Ni film showed more than 1.6 million times cyclic shape recovery of ~0.15%.
Key words: Ti-Ni alloy, thin films, magnetron sputtering, Joule heat, shape memory behavior

1. INTRODUCTION

Ti-Ni shape memory alloy thin films have attracted much attention for microactuators of micromachines because of their large deformation and strong recovery force [1-3]. There are many published works on their microstructures [4], mechanical properties [5], quantitative evaluation of shape memory effects [6], stability of shape memory effect against thermal cycles [7], and so on. It can be concluded from these investigations that the shape memory characteristics and the mechanical properties of sputter deposited Ti-Ni alloy thin films are comparable or even superior to those of bulk specimens.

When we actuate Ti-Ni shape memory alloy films in an air environment, the transformation temperature must be higher than room temperature (RT). Transformation temperatures of Ti-rich Ti-Ni films are known to be higher than those of Ni-rich films, and can be higher than RT by choosing the annealing condition appropriately [8, 9]. The Ti-Ni shape memory alloy actuator is usually designed to be driven by Joule heating. However, the Joule heat induced shape memory behavior of Ti-Ni thin film has not been studied sufficiently. The primary purpose of the present work is to get some insight into Joule-heat-induced shape memory behavior of Ti-Ni shape memory alloy thin films.

2. EXPERIMENTAL PROCEDURES

2.1. Deposition of Ti-Ni shape memory thin films

Ti-Ni shape memory alloy thin films were deposited onto thin Cu foils (50 μm in thickness) by dc magnetron sputtering. Ultimate pressure of the apparatus used was less than 1.0×10^{-4} Pa. Pure argon gas was used as sputtering gas and its pressure was kept constant at 0.2 Pa. The target used was Ti-50at%Ni alloy disk (99.9%, $\phi 50 \times 3$ mm). The target-substrate distance was 50 mm. In order to control the composition of the deposited films, several pure Ti chips ($3 \times 3 \times 1$ mm) were placed on the erosion area of the alloy target. This allows us to control the film composition between 47 and 56at% of Ni. Applied dc power was 120 W and the film thickness was controlled to be ~5 μm by deposition time. The substrate temperature was maintained at 250°C during deposition.

The XRD measurements revealed that the as-deposited films are in amorphous state regardless of the film composition. In order to crystallize and to memorize a flat shape, the deposited films were heat treated at 600°C for 60 min after dissolving the Cu substrates by a dilute HNO_3 solution. These heat treatments were carried out in evacuated quartz capsules, followed by water quenching.

The chemical composition of deposited films was determined by means of energy dispersive X-ray analysis (EDX: Horiba, EMAX-2770) using a conventional standardless ZAF method. Martensitic and reverse transformation temperatures of the films were measured by means of differential scanning calorimetry (DSC: SEIKO Instruments, DSC6100) with a scanning rate of 5 °C/min.

2.2. Tensile test

Tensile test of annealed Ti-Ni films were carried out by using a specially developed tensile testing apparatus. This apparatus is a flat bed type, and the applied stress and the elongation of the specimen were measured by a load cell and a differential transformer, respectively. Constant force is applied by a weight and the specimen deformed can recover its strain without any change of applied force. The specimen size was $2^W \times 4^L$ mm in its gauge portion. The grips of specimens are electrically isolated to the body of the testing machine, and electrically connected to a dc pulse power supply. This setup allows us to raise the specimen temperature by Joule heating.

3. RESULTS AND DISCUSSION

3.1. Transformation temperatures and stress-strain curves

We have determined the transformation temperature of Ti-Ni films, with various Ni contents after annealing at 600°C for 60min, by differential scanning calorimetry. These Ti-Ni films exhibit three different types of DSC curves and typical examples are depicted in Fig. 1, where (a) 46.2, (b) 48.4 and (c) 50.6at%Ni. As describe below these types depend on Ni content. In Fig. 1, the solid lines indicate the transformation occurring during cooling, while the broken lines the reverse transformation during heating. These curves were obtained at the 3rd or 4th measurement cycle to ensure transformations. The DSC curve of the Ti-46.2at%Ni film shows two transformation peaks in both cooling and heating processes, as seen in Fig. 1(a). The first peak during cooling corresponds to a B2-to-R transformation and the second one to a R-to-B19' transformation. The film containing less than 48at%Ni revealed this type of transformation. In (b), the DSC curve of the Ti-48.4at%Ni film shows a two-step B2-to-R-to-B19' transformation in the cooling process and a direct transformation from B19' to B2 in heating process. These transformation behaviors are found in a range of Ni content between 48.4 and 49.3at%. When the Ni content in films was 49.4~49.7at%, the films showed the two-step transformation in both cooling and heating processes. On the other hand, the DSC curve (c) of the Ti-50.6at%Ni film shows one peak in both cooling and heating processes within the temperature range measured. The occurrence of the single peak is believed to correspond to B2-to-R and R-to-B2 transformations because of its small hysteresis

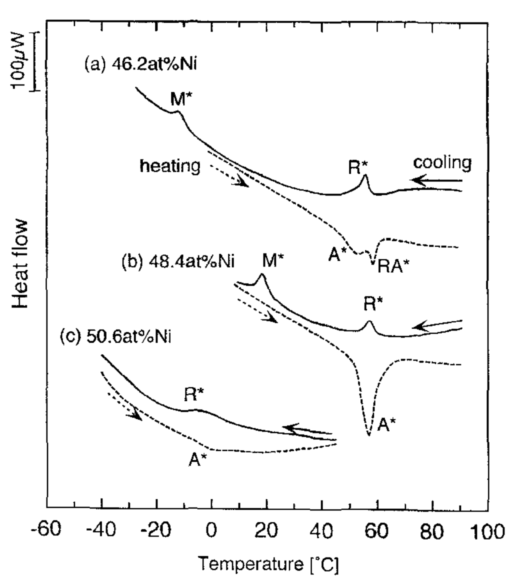

Fig. 1 Typical DSC curves of Ti-Ni films with various Ni contents annealed at 600°C for 60min followed by water quenching.

between cooling and heating process. M_s temperature of this film should be lower than -50°C. Ni-rich film fabricated in this work showed this type of DSC curve.

We have measured the transformation temperatures of R_s, M_s and A_f from DSC measurements such as shown above and they are plotted against the Ni content in Fig. 2. R_s temperature of films containing less than 49.5at%Ni is nearly constant at around 55°C and it decreases rapidly with increasing Ni content from 49.5 to 50at%Ni. When the Ni content is more than 50.5at%, R_s temperature becomes constant, being around 0°C. The change of A_f temperature with Ni content is similar to that of R_s temperature. As seen in Fig. 2, M_s temperature of films containing less than 49.5at%Ni are slightly lower than RT, whereas M_s temperature of films containing more than 50.5at%Ni were found to be much lower than -50°C. Similar behavior of transformation temperatures with Ni content has also been reported by Gyobu et al. [8]. They explained the behavior as a phenomenon related to the circumstance of precipitates in the grain. In the present work, films containing less than 49.5at%Ni can be expected to show shape memory effect at above RT. On the other hand, films containing more than 50at%Ni are expected to show superelasticity.

Figure 3 shows some stress-strain curves of Ti-Ni films with various Ni contents, deformed at RT. Because the change in the shape of stress-strain curves was observed for the first 2~3 loading-unloading cycle, the curves obtained at the 4th loading are depicted in this figure except for that of the Ti-46.2at%Ni film. As seen, the Ti-46.2at%Ni film has broken during the first loading after small apparent plastic deformation. Ti-Ni films containing between 46.8 and 49.4at%Ni show perfect shape memory effect. The apparent plastic strain seen in the specimen after unloading has been perfectly recovered by Joule heat, as indicated by dotted arrows. The recoverable strain reaches ~2.5% for Ti-49.4at%Ni film. On the other hand, the Ni-rich (Ti-52.0at%Ni) film shows superelasticity. These stress-strain characteristics at RT are consistent with the results of the DSC measurement shown in Fig. 2.

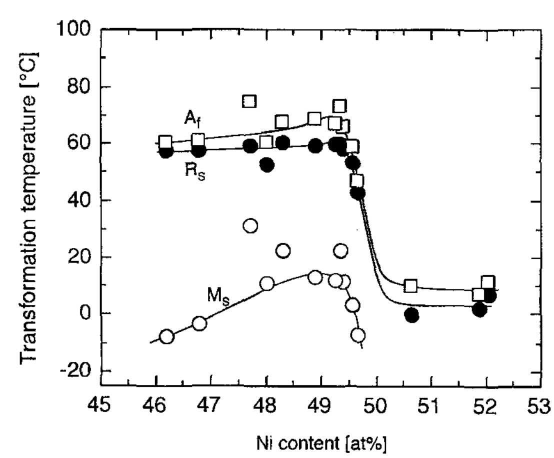

Fig. 2 The transformation temperatures of Ti-Ni films annealed at 600°C × 60 min -> WQ as a function of Ni content in the films.

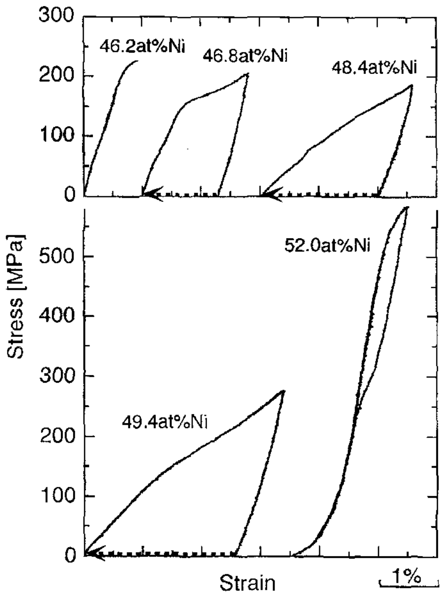

Fig. 3 Typical stress-strain curves of Ti-Ni films with various Ni contents. Dotted arrows indicate the recovery of the strain by Joule heat.

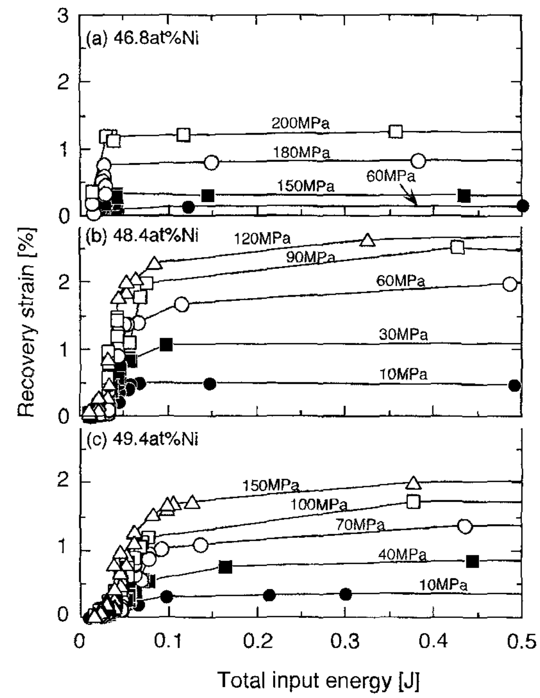

Fig. 4 The recovery strain under various applied stresses as a function of a total input energy.

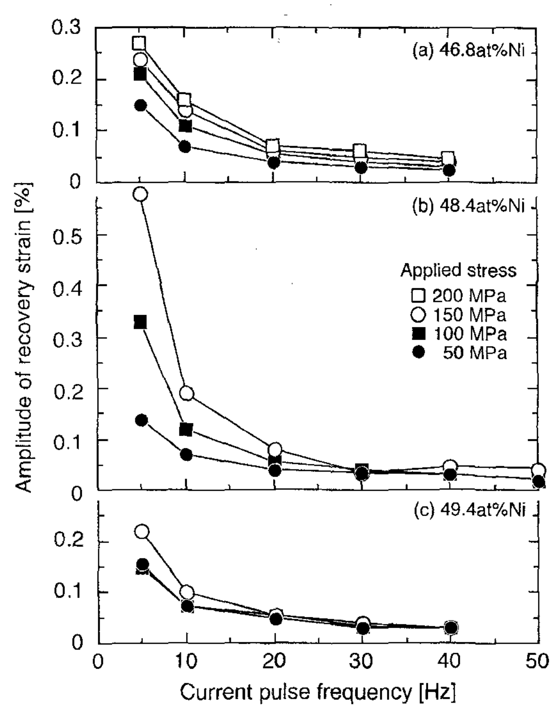

Fig. 5 The amplitude of the recovery strain found in the Ti-Ni films as a function of current pulse frequency.

3.2. Joule-heat-induced shape memory effect

The Joule-heat-induced shape memory behavior of Ti-Ni films has been investigated using a specially made tensile tester. Firstly, we have measured the recovery strain under a constant tensile stress when a single pulse current was applied. Figure 4 shows the recovery strain under various applied stress as a function of a total input energy. For convenience, the total input energy was calculated by the product of the applied current (i), voltage (V) and pulse duration (t). Figure 4(a), (b) and (c) show the results taken from Ti-Ni films containing 46.8, 48.4 and 49.4at%Ni, respectively. The recovery strain increases rapidly with increasing total input energy, and it reaches a prestrain depending on an applied stress at the total input energy of less than 0.1 J. No plastic strain could be detected in all specimens tested. The recovery strain (or prestrain) of the Ti-46.8at%Ni film [Fig. 4(a)] is found to be small as compared with that for the other films. On the other hand, the films of 48.4 and 49.4at%Ni [Fig. 4(b), (c)] show relatively large recovery strain (or prestrain) even if the applied stress is small. These results agree with the crystal phases present in these films at RT, which was clarified by DSC measurements, Fig. 2. That is, the Ti-46.8at%Ni film showed lower M_S temperature than RT, and the relatively large stress is necessary to induce a martensitic transformation. It is found that a small strain of ~0.3% was recovered for the film [Fig. 4(a)] at an applied stress less than 150MPa, and such strain may be attributed to the reorientation of R-phase. On the other hand, Ti-Ni films of 48.4 and 49.4at%Ni have M_S temperature higher than RT, so that the reorientation of martensite can be easily induced from a small stress level.

We have tried to actuate these films by cyclically applying Joule heat under a constant stress. The amplitude of the recovery strain measured in the Ti-Ni films under various applied stresses is summarized against current pulse frequency in Fig. 5, where (a), (b) and (c) show the results of the Ti-Ni films containing 46.8, 48.4 and 49.4at%Ni, respectively. As seen, in all cases the amplitude of recovery

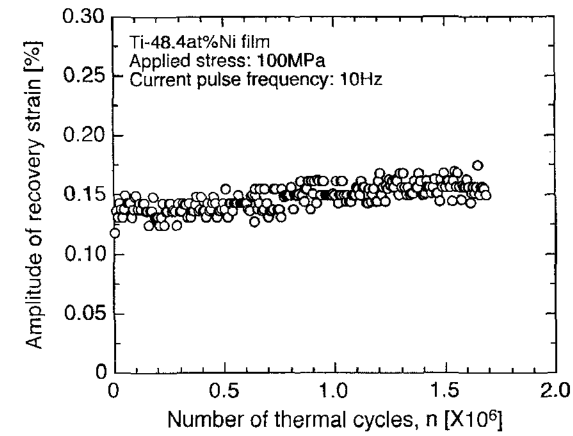

Fig. 6 The amplitude of recovery strain of Ti-48.4at%Ni film as a function of the number of thermal cycles.

strain decreases with increasing current pulse frequency and/or decreasing applied stress. It is noted that a strain oscillation occurs in sync with the current pulse up to 50Hz in Ti-48.4at%Ni film. This agrees with the frequency limit found in the diaphragm type actuator [10]. A maximum recovery-strain amplitude of 0.58% is obtained in this work for Ti-48.4at%Ni film when the applied stress and current pulse frequency are 150MPa and 5 Hz, respectively. It should be noted however that this amplitude is much smaller than recovery strain when a single pulse current was applied [see Fig. 4]. This may be attributed to a cooling efficiency of the film. From the results described above, it can be concluded that the Ti-48.4at%Ni film is the best candidate to be used as a micro actuator among Ti-Ni films deposited in the present work because of its large recoverable strain and force. In order to understand more the effect of repetition on shape memory behavior of the Ti-48.4at%Ni film, the cyclic test was further performed under an applied stress of 100MPa and a pulse frequency of 10 Hz. The recovery strain amplitude obtained is depicted in Fig. 6 as a function of the number of thermal cycles. Although the strain amplitude fluctuates slightly between 0.13 and 0.17%, there is essentially no degradation up to the number of cycles (1.6 million times) employed in the present experiment. After termination of this cyclic experiment, the stress-strain curve was measured and it was found that the curve was almost the same as that obtained before the cyclic test. This is strong indicative for Ti-Ni films containing 48.4at%Ni can be used for practical applications.

4. CONCLUSION

Ti-Ni films with various Ni contents were fabricated by means of dc magnetron sputtering using a composite target. The shape memory characteristics of Ti-Ni films annealed at 600°C for 60 min followed by water quenching were investigated using a custom-made tensile testing machine with a Joule heating unit. The results obtained are summarized below.

(1) R phase transformation temperature of Ti-rich Ti-Ni films was nearly constant and around 60°C. The temperature decreased rapidly with increasing Ni content from 49.5 to 50at%. On the other hand, it was also constant and around 0°C for Ni-rich Ti-Ni films containing Ni above 50.5at%.
(2) Ti-Ni films containing 46.8~49.4at%Ni showed shape memory effect at around RT. The films showed perfect shape recovery under a constant stress of up to 200 MPa by applying the total input Joule energy of less than 0.1 J.
(3) Ti-50.6at%Ni film showed superelasticity at around RT.
(4) Strain oscillation in sync with the current pulse was observed up to 50Hz in Ti-48.4at%Ni film. Cyclic shape memory effect was investigated for Ti-48.4at%Ni film under a constant stress of 100 MPa and a perfect thermal-cyclic shape recovery of ~0.15% was attained more than 1.6 million times.

ACKNOWLEDGEMENT

The authors are very grateful to Prof. S. Miyazaki, Univ. Tsukuba, for his valuable comments, and to Prof. K. Inoue, Univ. Washington, for his continuous encouragement. We also thank Mr. Y. Okamoto for his experimental assistance.

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(Received October 10, 2003; Accepted March 20, 2004)