Article ID: 1003 - 6326(2000)03 - 0375 - 03

Characteristics of shape memory and superelasticity for TiNi thin films①

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Abstract: TiNi shape memory alloy thin films were deposited by using a RF magnetron sputtering apparatus. The transformation and shape memory characteristics of the thin films have been investigated by using DSC and tensile tests. After aging, perfect shape memory effect and superelasticity were achieved in TiNi thin films.

Key words: TiNi alloy; shape memory effect; superelasticity; thin film **Document code: A**

1 INTRODUCTION

The development of micromachines or micro-robots has been a priority in such fields as medicine, biochemistry, semiconductor. However, in order to drive such micromachines, it is necessary to develop microactuators. Among the several types of high performance materials proposed for fabricating such microactuators, TiNi shape memory alloy thin films exhibit significant advantages including large deformation and recovery forces. Therefore, an important demand for the TiNi thin films has been generated in the field of micromachines, and some efforts have been made to fabricate TiNi thin films using the sputter-deposition technique[1~4]. However, such films are so brittle that they will fracture by applied stress, hence it is difficult to investigate the shape memory characteristics by mechanical testing.

The purpose of the present paper is to clarify the characteristics of shape memory and superelasticity including the transformation temperatures, the strain associated with both R-phase and martensitic transformations, the stress vs transformation temperature relationship and so on by observing deformation behavior in aged thin films during cooling and heating under various constant stresses.

2 EXPERIMENTAL

TiNi thin films were prepared by a RF magnetron sputtering method by using a Ti-50.0Ni target of 101.6 mm diameter. The films were deposited on glass substrates. Films with different alloy compositions were prepared by putting a number of small Ti plates put on the TiNi target sequentially, respectively. The Ni-contents of the films were determined to be 49.8%, 50.3%, 51.2% and 51.9% (mole fraction), respectively, by electron microprobe analysis. After removed from the substrates, the TiNi films were cut into two shapes of specimen, i.e., 3 mm × 3 mm for DSC (differential scanning calorimetry) and 1 mm × 5 mm (gauge portion) for mechanical tests then annealed at 973 K for 3.6 ks followed by aging at 773 K for 3.6 or 36 ks. The Ti-49.8Ni thin film used in this experiment was not age-treated, because it did not show aging effect. Annealing and aging were followed by quenching into water. Transformation temperatures were determined by Shimadzu DSC-50 with a heating and cooling rate of 10 K/min. Shape memory behavior was characterized by measuring the strain induced in the film during cooling and heating under a variety of constant applied stresses between 5 and 470 MPa, and superelasticity behavior was characterized by measuring stress-strain relationships at different temperatures with Shimadzu Autograph DSS-10T-S.

3 RESULTS AND DISCUSSION

Fig.1 shows the martensitic transformation and its reverse transformation observed by DSC in a Ti-49.8Ni film. The peak temperatures for the forward and reverse transformations (M^* and A^*) are 332 K and 361 K, respectively. These temperatures are almost the same as those of similar composition bulk specimens[5,6] produced by an electron beam melting method or a radio frequency vacuum induction melting method, indicating that the contamination effect was negligible in the film. Other types of transformation processes were also observed in films of different compositions as shown in Figs.2 and 3. Fig.2 shows that an age-treated Ti-51.9Ni film reveals two stage transformation B_2-R-M, i.e., the B_2 (parent phase)-R transformation for the first stage and the R-M (martensite) transformation for the second stage

① **Received date:** May 4, 1999; **accepted date:** Aug.13, 1999

on cooling, but the reverse transformation presents only the martensite to the parent B_2 phase upon heating. Fig.3 shows that an age-treated Ti-51.2Ni film reveals two-stage transformations both on cooling and heating. These three types of transformation behavior are the same as those observed in bulk specimens[7].

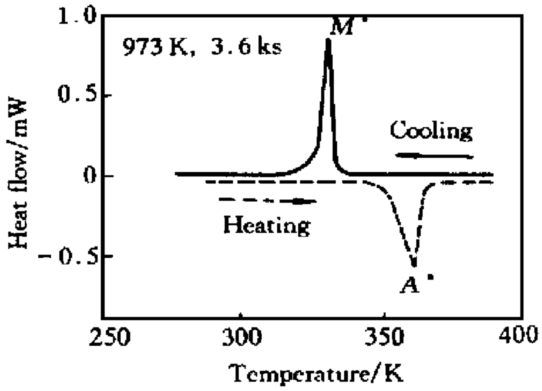

Fig.1 DSC curve of Ti-49.8Ni film

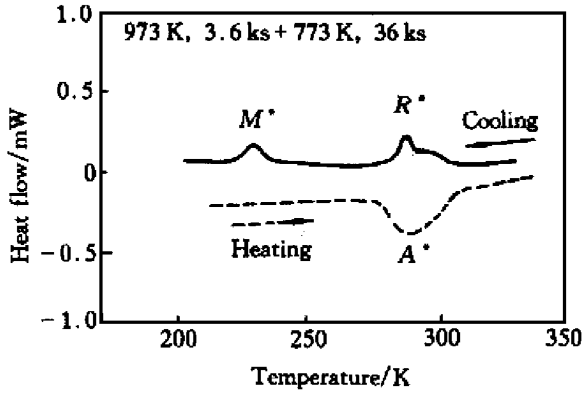

Fig.2 DSC curve of Ti-1.9Ni film

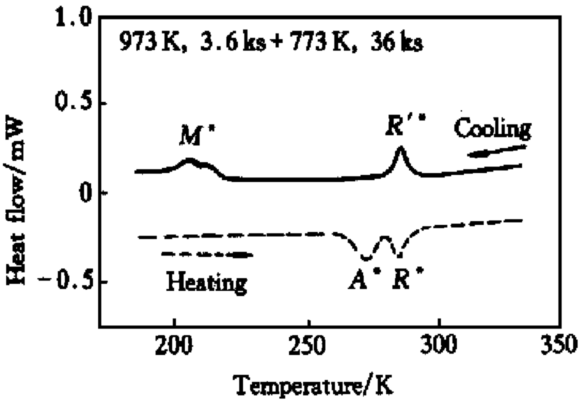

Fig.3 DSC curve of Ti-51.2Ni film

The above data show that the TiNi thin films formed by sputtering reveal the thermoelastic R-phase and the martensitic transformations. Hence it is expected that the thin films will show shape memory effect[8].

Shape memory behavior observed in the Ti-51.2Ni film is shown in Fig.4. Three strain-temperature curves are shown in the figure; these were measured upon a cooling and heating cycle under different constant stresses, i.e., 150, 320 and 470 MPa, respectively. The curve measured under 150 MPa shows that on cooling a first shape change appears at R_s due to the R-phase transformation and on further cooling a second shape change occurs at M_s due to the martensitic transformation. Upon heating, the original specimen shape was recovered due to a two-stage transformation occurring at A_s for the first stage and at R'_s for the second stage. The first shape change on cooling is not sensitive to applied stress, while the second shape change increases with increasing applied stress. The second-stage shape change on cooling is characterized by a large strain and a large thermal hysteresis (A_s - M_s). The shape change associated with the R-phase transformation is characterized by a small strain and a small temperature hysteresis (R'_s - R_s). Therefore, quick response is expected for the movement of an actuator using such R-phase transformation.

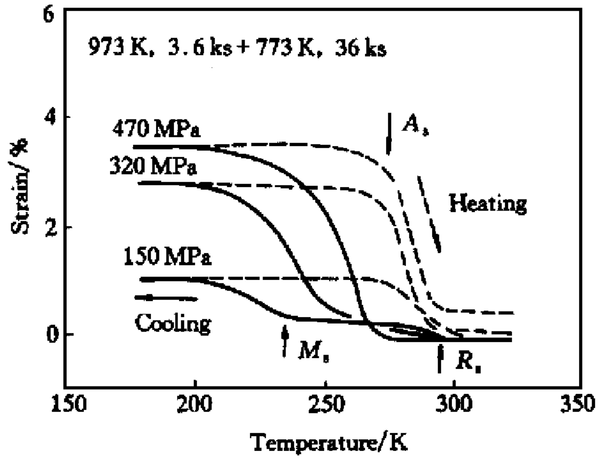

Fig.4 Strain vs temperature curves of Ti-51.2Ni film

Relationships between the transformation temperatures (R_s, M_s and A_s) and applied stress in TiNi thin films are shown in Fig.5. They show linear relationships. The slope for the reverse martensitic transformation (A_s) is steeper than those for the forward martensitic transformation (M_s), causing the temperature hysteresis to become narrower in higher stress region. The slope for the R-phase transformation is steeper than that for the martensitic transformation. Therefore, the effect of applied stress on the R_s is weaker.

Three types of transformation strains i.e., strain (ε_R) due to the B_2-R transformation, strain (ε_M) due to the R-martensite transformation and strain (ε_A) due to the martensite-B_2 reverse transformation, are plotted as a function of stress in Fig.6. They increase with increasing stress. However, the increasing rate of ε_R is very small when compared with the other two strains. Besides, the strain ε_R stops increasing above 150 MPa, implying that

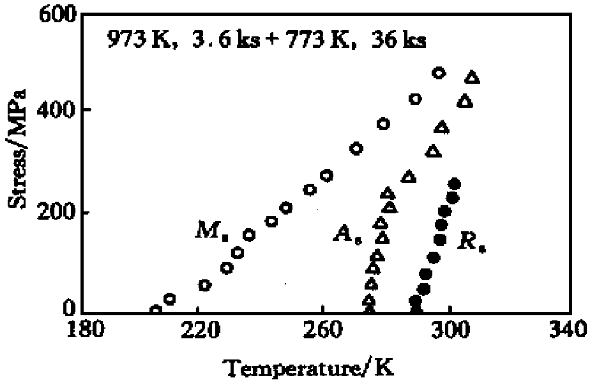

Fig.5 Stress vs transformation temperature of Ti-51.2Ni film

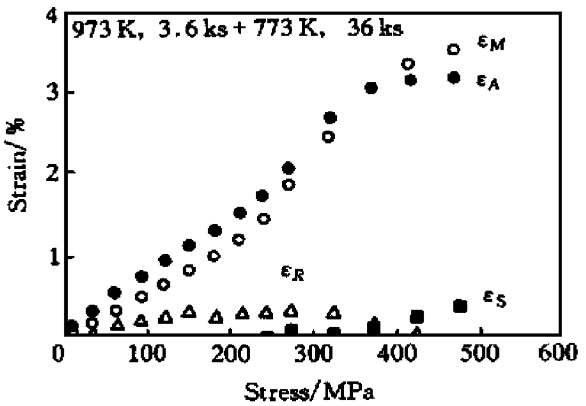

Fig.6 Various types of strains vs stress in Ti-51.2Ni film

preferentially oriented R-phase variants will be achieved under a low stress. The strains ε_M and ε_A increase rapidly with increasing stress. Therefore, they become about ten times larger than ε_R in a higher stress region. The strain ε_A is larger than the strain ε_M under a critical stress, where the two lines for the strains intersect each other, because the total strain $\varepsilon_R + \varepsilon_M$ induced upon cooling recovers by a single stage reverse transformation inducing ε_A upon heating. At the higher stress region, the film induces a plastic strain ε_s, resulting in suppression of part of the shape recovery upon heating. Therefore, ε_A becomes smaller than ε_M in the higher stress region.

Fig.7 shows the stress-strain curves of a Ti-50.3Ni film at different temperatures; they are the same as the deformation properties of bulk TiNi alloys[9,10]. Curves (a) and (b) show the stress-strain curves obtained below A_s, so that the shape change remains after unloading. The residual strain disappears upon heating to above A_f, revealing the perfect shape memory effect. Curve (c) is obtained by deforming the film at a temperature between A_s and A_f so that it shows a partial shape recovery upon unloading and another shape recovery on heating. Finally, curve(d) shows a perfect superelasticity is accompanied by a stress hysteresis, it is necessary to apply a stress high enough to observe such superelasticity. In this case, the maximum stress applied to the film is higher than 600 MPa. This is a good evidence for the slablity of the sputter-deposited TiNi thin films.

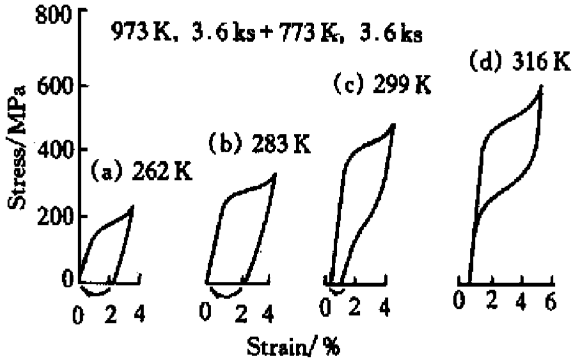

Fig.7 Stress-strain curves of Ti-50.3Ni film

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(Edited by ZHU Zhong-guo)