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SHAPE MEMORY THIN FILM OF TITANIUM-NICKEL FOR MICROACTUATOR FORMED BY SPUTTERING

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ABSTRACT

Thin films of Ti-Ni alloy were formed by sputtering under various Ar gas pressures and r. f. powers to investigate the optimum sputtering conditions and to demonstrate their shape memory effect. The composition and structure of the films were examined by electron microprobe analysis and scanning electron microscope. These films were annealed in order to crystallize them. The mechanical property of the annealed films was evaluated by a conventional bending test. The transformation tmeperatures were determined by differential scanning calorimetry. The shape memory behaviour was examined quantiatatively by changing in sample temperature under various constant loads. It was found that the Ar gas pressure had a critical effect on the mechanical property of the thin film,s although the r.f. power also affected it. The films formed at a high Ar gas pressure were too brittle to be bent successfully. However, the films formed at a low Ar gas pressur could be bent and their shape memory behavior was found to be comparable with that of bulk Ti-Ni alloys.

INTRODUCTION

Micromachines such as micromanipulators and fluid microvalves are expected to be used in the near future in various fieleds such as biotechnology, medicine and the semiconductor industry. In order to produce such a micromachine, the development of an effective microactuator is essential. The driving force of a microactuator can be obtained using various physical phenomena. Among them, the shape memory effect of Ti-Ni alloys seems to have attractive characteristics for a microactuator. They demonstrate large transformation strain and stress by heating and cooling and can also produce complicated motion by themselves. Therefore thin films of Ti-Ni are a promising candidate for a microactuator. Some research and development work[1-4] has been carried out on Ti-Ni thin films. However, the relationship between the formation and the mechanical properties of the films has not been reported. The objectives of this study are to investigate the optimum sputtering conditions and to demonstrate the shape memory behaviour of thin Ti-Ni films.

EXPERIMENTAL PROCEDURE

Thin films of Ti-Ni were prepared by sputtering. The films were deposited on glasses in a 6 inch r.f. magnetron sputtering apparatus using a Ti-50.0at%Ni target. The sputtering condition are given in Table 1. The substrate-to-target distance was 82mm. The substrate temperature was kept at 523K except in one case, where a further increase in the substrate temperature was observed, probably because of an increase in the target temperature. The Ar gas pressure and r.f. power were varied from 0.67 to 13.3Pa and from 200 to 600W respectively to investigate the optimum sputtering conditions. The deposition rate was in the range of 1.0 to 3.5μmh^{-1} and the final thickness was from 3.5 to 8.1μm.

The compositions of the films were determined by electron microprobe analysis using a standard sample of Ti-51.0at%Ni. The structure of the films was examined by observing a fracture cross-section of each film with a scanning electron microscope.

The thin films were removed from the glasses after the sputtering and then heat treated for 30 min at 973K to produce crystallization of the Ti-Ni, homogenization of composition and relaxation of the stress induced during film deposition. The crystallization of the film was confirmed by X-ray diffraction. Subsequently, some of the annealed specimens were aged for 1h at 773K to produce a distribution of fine Ti_3Ni_4 particles. These precipitates were confirmed by transmission electron microscopy.

The mechanical properties of the annealed films were evaluated qualitatively by a conventional bending test at room temperature.

The shape memory behaviour was examined for the films which exhibited good mechanical behaviour in the above bending test. The martensitic transformation temperature were measured by differential scanning calorimetry (DSC). Both heating and cooling rates were 10K min^{-1}. The shape memory effect was measured quantitatively by changing the specimen temperature under a various constant load of up to 300MPa. This test involved loading at 373K, cooling to 173K at a rate of -10Kmin^{-1}. The specimen size was 1mm × 5mm × 5-15μm..

RESULT AND DISCUSSION

Table 1 shows the composition of the films formed by sputtering, which were analysed with an electron microprobe. They are slightly different from the composition of the target, Ti-50.0at%Ni.

A conventional bending test was carried out for Ti-Ni thin films after annealing for

Table 1. Sputtering conditions and film compositions

Run	Ar gas pressure (Pa)	R.f. power (W)	Substrate temperature (K)	Coating rate (μm^{-1})	Coating duration (min)	Film thickness (μm)	Film composition (at.%)	
							Ti	Ni
1	13.3	400	523	1.5	180	4.5	50.2	49.8
2	6.7	600	573	3.2	150	8.1	50.5	49.5
3	6.7	400	523	2.5	84	3.5	50.6	49.4
4	6.7	200	523	1.0	300	5	51.6	48.4
5	0.67	400	523	3.5	110	6.4	48.6	51.4

30 min at 973K. The result revealed that only the film prepared at 0.67Pa could be bent without breaking, while the other films fractured. Therefore the Ar gas pressure seems to have a critical effect on the mechanical properties of the thin films. The effect of the r.f. power was also observed, although it was small compared with that of the Ar gas pressure. Of the films fractured, the film prepared at an r.f. power of 600W fractured into tiny pieces.

Figure 1 shows the structure of the as-deposited thin films. X-ray measurements revealed these films were amorphous. The film prepared at a low Ar gas pressure exhibits a featureless structure, while the films prepared at a high Ar gas pressure exhibit a columnar structure. This columnar structure suggests that the films are porous. This struc ture seems to be caused by the restricted mobility of deposited atoms on surface of the growing film. A high Ar gas pressure is likely to decrease the energy of the sputtered atoms by collision., resulting in a decrease in their surface diffusion[5]. Furthermore, under a high Ar atoms adsorbed on the film sruface can interfere with the surface diffusion of sputtered atoms[6].

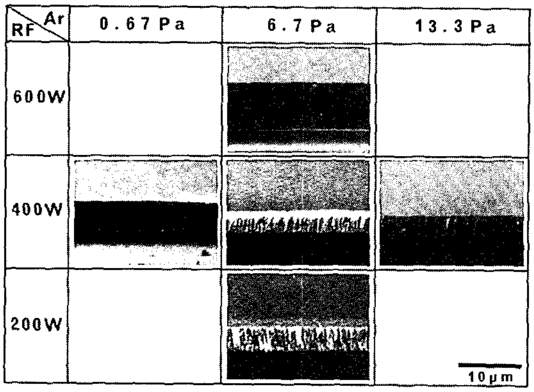

Fig. 1. Structure of Ti-Ni films formed under various sputtering conditions.

Of the films prepared at a high Ar gas pressure, the structure of the film prepared at an r.f. power of 600W seems to be less porous than the other films. This result corresponds with the result of the bending test. However, in order to discuss the actual of the r.f. power on the structure, further work is necessary since the substrate temperature was slightly high in this case compared with the other cases, as shown in Table 1.

Figure 2 shows the structure of the thin film prepared at a high Ar gas pressure and annealed for 30min at 973K. It seems to be slightly densified compared with the as sputtered structure, but columnar structure still remains. Therefore the columnar structure formed during sputtering is likely to be the reason for the brittleness of the films.

Figure 3(a) and 3(b) show the DSC results of thin films of Ti-50.4at.%Ni and Ti-51.4at.%Ni respectively. The thin film of Ti-50.4at.%Ni was prepared by placing Ti plates on the Ti-50.0at.%Ni target. The film of Ti-50.5at.%Ni was prepared by placing Ti plates on the Ti-50.0at.%Ni target. The film of Ti-

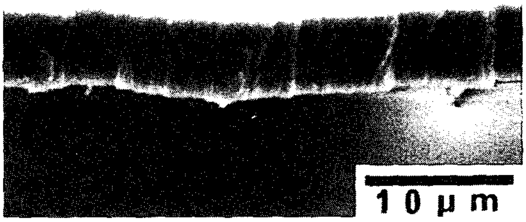

Fig. 2. Structure of TiNi films, formed at an Ar gas pressure of 13.3Pa, which were subject to annealing at 973K for 30 min.

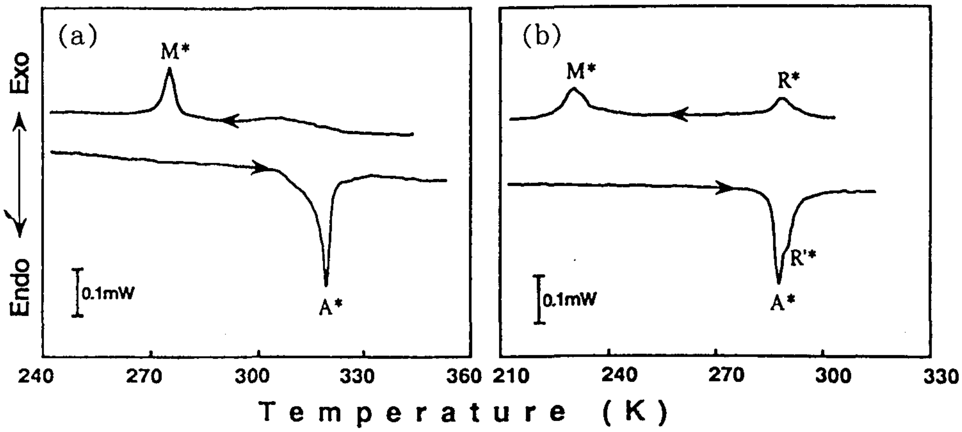

Fig. 3. DSC curves for thin films of (a) Ti-50.4at. %Ni after annealing at 973K for 30 min and (b) Ti-51.4at. %Ni after a solution treatment at 973K for 30min followed by aging at 773K for 1h.

51.4at.%Ni was aged for 1h at 773K after annealing at 973K to produce a dispersion of fine Ti_3Ni_4 particles in a matrix of Ti-Ni.

In Fig. 3(a), two peaks can be observed. One is located at 275K on cooling and the other at 320K on heating. The formed(M*) is related to martensitic transformation from the parent phase to the martensite. The latter (A*) is related to the reversion of the martensite to the parent phase.

In Fig. 3(b) the two peaks are located at lower temperatures than those in Fig.3(a), The location being at 230K (M*) and 288K (A*). Furthermore, in addition to these two peaks related to the martensitic transformation, two other small peaks can be detected at higher temperatures than the martensite-related peaks. One (R*) is located at 289K and related to the R phase transformation. The other (R*) is located at 290K and related to the reverse R phase transformation.

Figures 4(a)-4(c) show the results of the thermal cycle tests for the thin film of Ti-50.4 at%Ni. The strains associated with both the martensitic and the revers martensitic transformations were observed. The transformation strain increased with increasing load and reached 3.5% under a constant stress of 303MPa. The plastic strain was 0.3% for this case. Both the Martensitic transformation start temperature M_s and the reverse martensitic transformation start temperature A_s increased with increasing applied stress. However, the formed was more sensitive to stress than the latter, resulting in a decrease in the thermohysteresis under a high stress.

Figure 4(d)-4(f) show the results of the thermal cycle tests for the thin film of Ti-51.4at.%Ni. The strain related to the R phase transformation was detected in addition to that related to martensite tranformation. These strain was 0.15 for a constant stress of 313MPa and thus reduced compared with that of the 50.4at.%Ni thin film. This improvement can be atttributed to precipitation strengthening with fine Ti_3 Ni_4 particles formed by aging at 773K. As with the film of Ti-50.4at.%Ni, the martensitic transformation stasrt temperature M_s, the reverse martensitic transformation start temperature A_s and the phase transformation temperature R_s all depended on the applied stress. Since

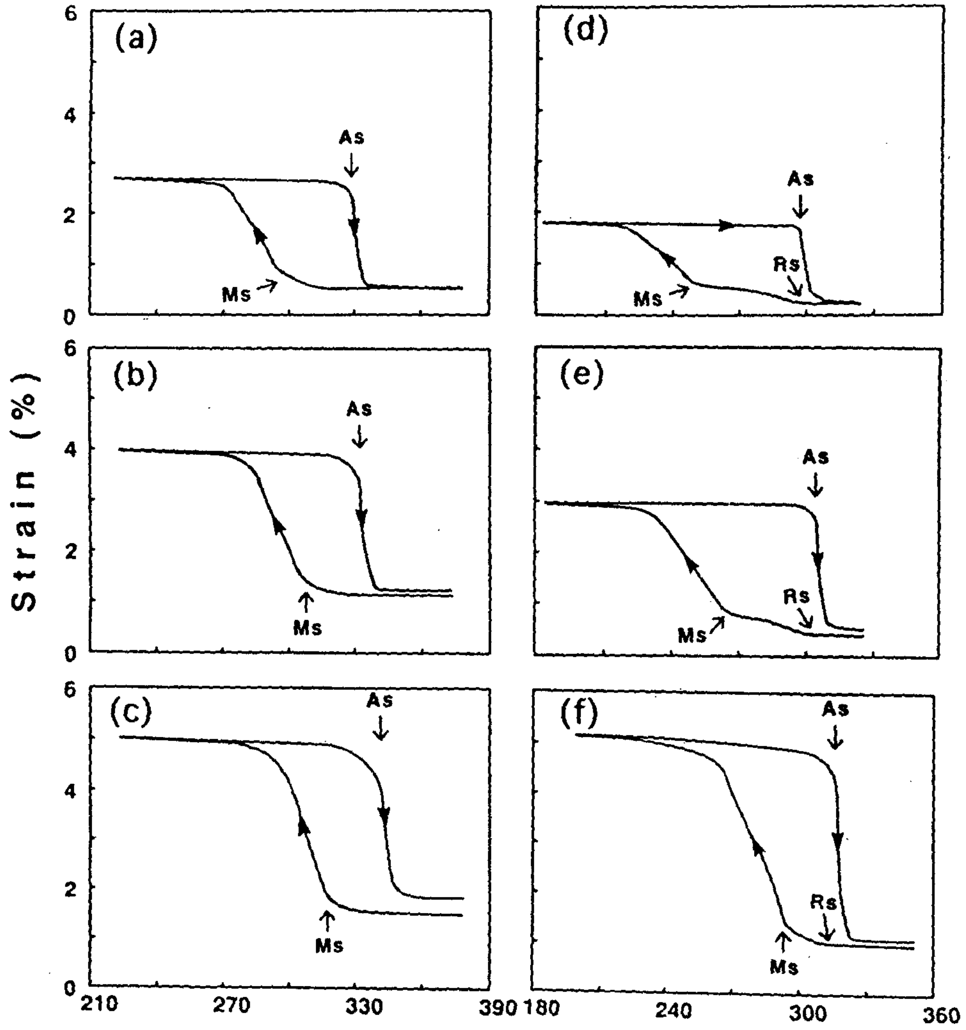

Fig. 4. Strain vs. temperature curves at constant loads of (a) 121MPa, (b) 182MPa and (c) 303MPa for the Ti-50.4at. %Ni thin film and at constant loads of (d) 104MPa, (e) 174MPa and (f) 313MPa for the Ti-51.4at.%Ni thin film.

the martensitic transformation start temperature was more sensitive to stress than the other temperatures, the thermophysteresis decreased and R phase transformation was not clear under a higher load. The shape memory behaviour of these films was almost comparable of bulk Ti-Ni alloy.

CONCLUSION

Thin films of Ti-Ni alloy were formed by sputtering under various Ar gas pressures and r.f. powers, and their mechanical properties and shape memory behaviour were examined. The following conclusions were obtained.

It was found that the Ar gas pressure had a critical effect on the mechanical properties of thin films of Ti-Ni, although the r.f. power slightly affected it. The film formed at high Ar gas pressure exhibited poor mechanical properties owing to the porous structure formed during sputtering. However, the films prepared at low Ar gas pressure exhibited a good shape memory behaviour comparable with that of bulk Ti-Ni alloy.

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