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The Characterization of TiNi Shape-Memory Actuated Microvalves

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ABSTRACT

Co-sputtering has been used to fabricate equiatomic thin films of TiNi, a shape memory alloy, which form the basis of microactuators with many applications in MEMS. The stress evolution of TiNi films will be described. The performance of the TiNi actuators has been characterized, with regards to actuation force, recoverable strain, time response, and fatigue resistance. The performance of microvalves using these actuators will also be presented.

INTRODUCTION

Shape Memory Alloys (SMAs) are a promising material for MEMS microfluidic applications, such as micropumps and microvalves. The physical basis of the shape memory effect is the reversible martensitic transformation. In TiNi, the material transforms reversibly on heating from the low temperature ductile martensite phase with the B19' structure to the stiffer, high temperature austenite phase with the B2 structure. The high recoverable strain and high actuation work density of SMAs are responsible for the significant interest in device applications [1]. The high recoverable strain allows large strokes, while the high actuation work density generates large output force per unit volume for microfluidic devices.

Among the many alloy systems known to produce SMAs, TiNi is the most widely studied. Bulk SMAs based on equiatomic TiNi have been known for over 35 years [2], and in thin film form appropriate for MEMS for about a decade [3]. It is well known that equiatomic TiNi has the highest transformation temperatures. Departures from exact TiNi stoichiometry in either direction cause a decrease in the transformation temperatures. TiNi films appropriate for MEMS applications have been batch fabricated successfully by our group [4]. Good thickness and compositional uniformity across a large area were observed. The TiNi films also have high recoverable stresses and reproducible transformation temperatures. Since the performance of microactuators is strongly related to the recoverable stress, the stress evolution of TiNi films during fabrication and thermal cycling is important and will be described here.

It has been previously demonstrated that SMAs display good fatigue resistances; lifetimes of a few million cycles are expected at 3% strain (in the martensite phase) [1]. However, most of the previous fatigue studies focused on TiNi in bulk form. In this paper, the fatigue of co-sputtered TiNi in thin film form and the performance of TiNi actuated MEMS microvalves will be described.

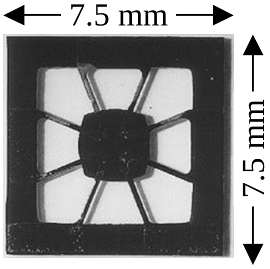

Figure 1. *TiNi microactuator*

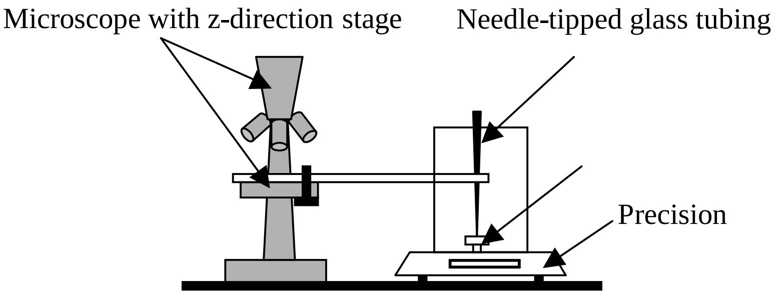

Figure 2. *Setup of fatigue test*

EXPERIMENTAL

Deposition procedure

The sputtering system used is a commercial Denton Vacuum Inc. Discovery 18 apparatus, which has a 460 mm diameter chamber equipped with two convergent DC-magnetron sputtering guns. Both sputtering guns are tilted by 60° from a horizontal substrate and are situated at a nominal 100 mm from the substrate. The substrate holder, used to suspend the 100 mm silicon wafer, can be rotated and heated by backside high intensity lamps. The sputtering gas is argon; sputtering can be carried out at pressures from 2.6 mtorr to 10 mtorr (0.3 to 1.3 Pa). A RF source for sputter-cleaning substrates prior to deposition is also present in the system.

TiNi films are deposited on 100 mm diameter and nominally 500 µm thick Si (100) wafers. The base pressure is about 5×10^{-7} torr (7×10^{-5} Pa). Before sputtering, the chamber is back-filled with Ar gas. Then the substrate is cleaned by RF sputtering for 10 seconds at 100 W, and is heated to 230^{o}C, with a 30 minute ramp-up time. During sputtering, the Ar pressure and substrate temperature are kept at 3.5 mtorr (0.46 Pa) and 230^{o}C, respectively. The TiNi films are co-sputtered at a deposition rate of ~1.0 µm/hr, using 75 mm diameter TiNi alloy and elemental Ti targets. Both targets can be controlled independently, which allows for tuning of deposition rate and film composition. The as-deposited TiNi films are amorphous. Shortly after sputtering, *in situ* annealing is performed at 420°C for 15 minutes to crystallize the amorphous as-deposited films; Ar is then evacuated from the deposition chamber and the wafer cools in vacuum.

Fabrication of TiNi microactuators

The patterned TiNi microactuator shown in Fig. 1 was fabricated using standard micromachining techniques. A TiNi path connects an outer Si frame with a central Si island. The reason to use patterned TiNi films, as opposed to continuous films, is to reduce the thermal mass, while retaining high actuation force. This actuator can be used as the moving part in microvalves or micropumps. In the microvalve described in [5], a microfabricated silicon spring was used to bias the TiNi microactuator such that in the martensite phase, the TiNi was deformed, and the Si island covered the valve orifice. When the TiNi transformed via Joule heating, the TiNi film returned to the original flat shape of the austenite phase, lifting the island off the orifice and opening the microvalve.

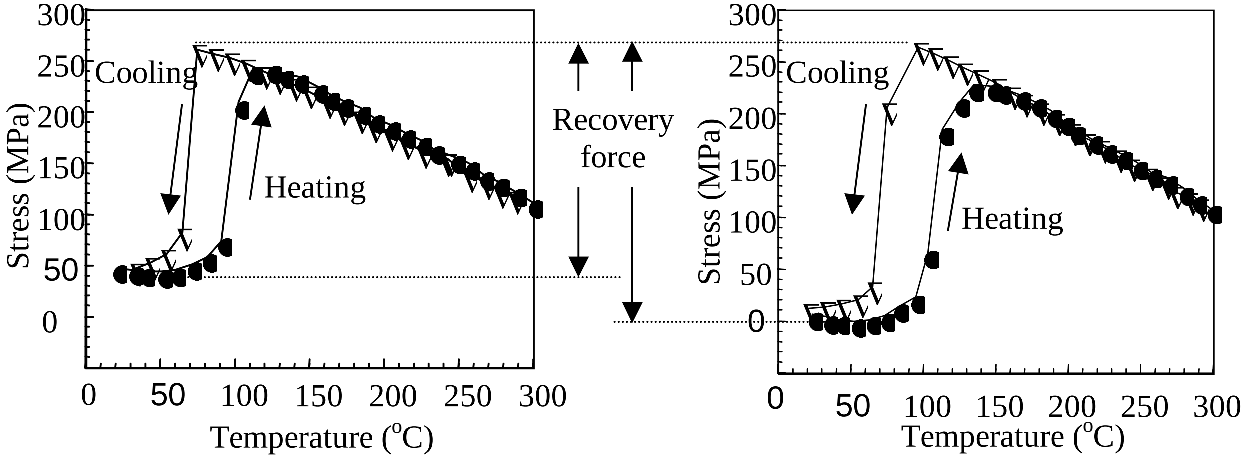

***Figure 3**. Stress evolution in TiNi films deposited on a (a) unheated substrate and (b) heated substrate*

Fatigue testing

Two kinds of fatigue tests, employing either fixed strain or fixed load, were performed. For the fixed strain tests, the setup shown in Fig. 2 was employed. The needle-tipped glass tubing attached to the z-direction stage of the microscope was used to apply fixed deflections on the Si island of the microactuator and the corresponding load was read by the precision balance. For the fixed load tests, a weight was epoxied to the Si island while the actuator sat on the z-direction stage, and the deflection of the island was measured by alternately focussing the microscope on the Si island and the Si frame. The loads and deflections of the Si island were measured after different thermal cycles, as well as for the different TiNi phases. Thermal cycling was accomplished via Joule heating by passing a current through the TiNi path of the actuator. Knowing the geometry of the microactuator, the corresponding stresses and strains can be determined from the loads and the deflections, respectively.

RESULTS AND DISCUSSION

Heated and unheated substrates

TiNi films were deposited on unheated ("room temperature" (RT)) or heated substrates. As noted already, for both substrate temperatures, the as-deposited films are amorphous, and must be annealed to crystallize the films to the B2 austenite structure. The room temperature films required an *in situ* anneal of 450°C (RT/450 film) for 25-30 minutes, whereas the 230°C films could be crystallized completely by a 420°C (230/400 film) 15 minute anneal. The XRD patterns [4] taken at room temperature indicate that the RT/450 films are partially transformed while the 230/420 films are fully transformed. The partial transformation of the RT/450 films also causes the recoverable strain and stress to be lower than that of the 230/400 films, as shown in Fig 3. Therefore, the deposition process involving heating to 230°C and annealing at 420°C is preferred.

Stress evolution of TiNi films

Development of residual stress is an intrinsic phenomenon in thin film deposition experiments, and strongly influences the mechanical properties and stability of thin films. It is well known that tensile or compressive stresses can be generated in sputtered films, depending on the deposition conditions. The details of stresses generated in TiNi films due to varying deposition conditions are described in more detail elsewhere [4].

A schematic stress evolution curve for TiNi films is shown in Fig. 4. The as-deposited stresses in the amorphous films vary with the sputter gas pressure. Ar pressures of 2.6, 3.5, 7, and 9 mtorr result in deposition stresses of 620, 380, 100, -50 MPa, respectively ("-" indicates compressive stress). We have found that 3.5 mtorr is an appropriate pressure. 2 µm thick films deposited at 2.6 mtorr cracked and delaminated. Films deposited at 7 and 9 mtorr had lower deposition stresses; however, microcracks were observed after annealing. The causes of the microcracks are not clear; they may be due to low film density or stress relief during annealing. The films deposited at 3.5 mtorr shows a suitable martensitic structure at RT, and no cracking.

As seen in Fig. 4, the thermal expansion mismatch between TiNi and Si causes the intrinsic tensile stress initially to decrease on heating until crystallization occurs, when the stress begins

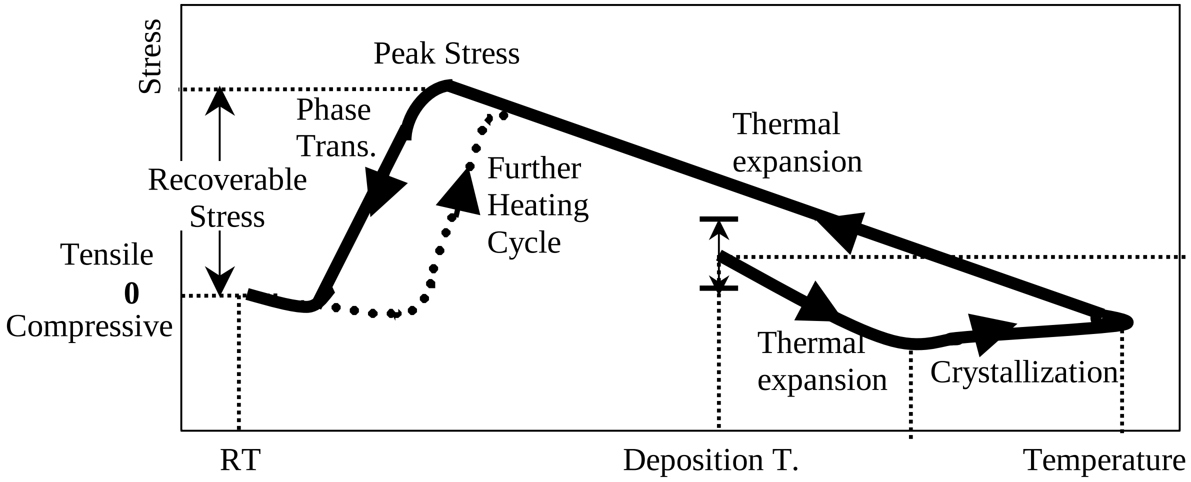

***Figure 4**. Schematic drawing showing stress evolution of TiNi SMA film.*

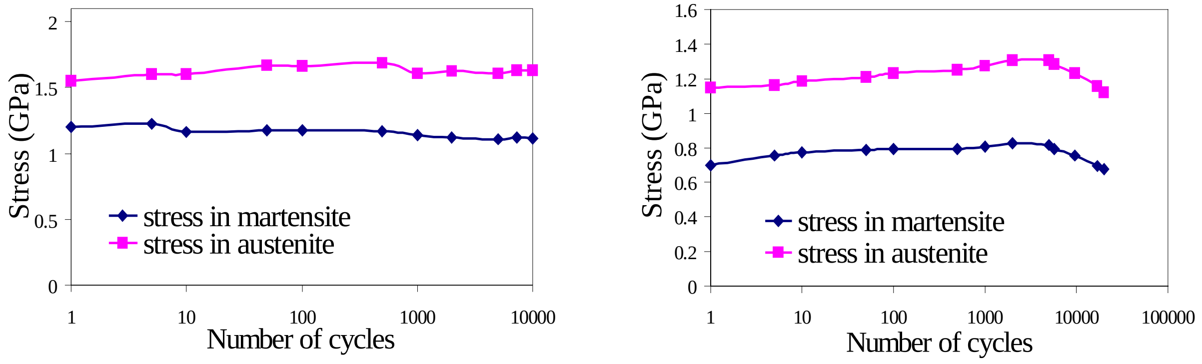

***Figure 5**. Stress as function of thermal cycles (log scale) at a fixed strain of (a) 0.6 % and (b) 1.2 %.*

to increase. This stress increase is caused by the onset of crystallization of the amorphous TiNi film, and is due to the small density difference between amorphous and crystalline TiNi. The increase in stress on subsequent cooling is again due to the thermal expansion mismatch until the phase transformation occurs. The residual stress in TiNi films deposited on 230°C substrates and at an Ar pressure of 3.5 mtorr, and annealed to 420°C, is very small because of the self-accommodation of martensite variants, an essential feature of SMAs. The hysteresis of the phase transformation is repeatable if further heating and cooling cycles are applied, as seen in Fig. 3. The crystalline TiNi films have A_s (austenite start temperature) of ~100°C and M_s (martensite start temperature) of ~80°C, which reproduces the highest transformation temperatures observed in this system.

Fatigue testing at fixed strain and fixed load

Lifetime and fatigue are important issues for moving actuators. At least one million cycles would be required for many commercial uses. In order to determine the effects of thermal cycling on TiNi microactuators, fixed strain and fixed load thermal cycling tests have been performed using the experimental setup shown in Fig. 2. The phase transformations in films were induced using Joule heating.

Fig. 5 shows the preliminary results of thermal cycling at fixed strains of 0.6 % and 1.2 %, respectively. At a fixed strain of 0.6 %, the forces for the austenite and martensite phases are stable up to 10000 cycles. The results for the 1.2 % fixed strain test show that the forces increase during the first 5000 cycles and decrease after 5000 cycles. The increase in force before 5000 thermal cycles may be due to work hardening caused by dislocations.

Fig. 6 shows the results of thermal cycling at a fixed load of 0.19 N, which corresponds to initial stresses of 0.64 GPa and 1.03 GPa in the martensite and austenite phases, respectively. (The difference in initial stress is caused by the change in geometry of the actuator. In the austenite phase, the actuator is flatter, and therefore the fixed downward load creates greater stresses along the legs of the actuator.) The measured strains initially decrease, and after about 1000 cycles the strains become stable. This is probably due to the "training" effect whereby the martensite variants within individual TiNi grain develop a preferred orientation on transformation from austenite.

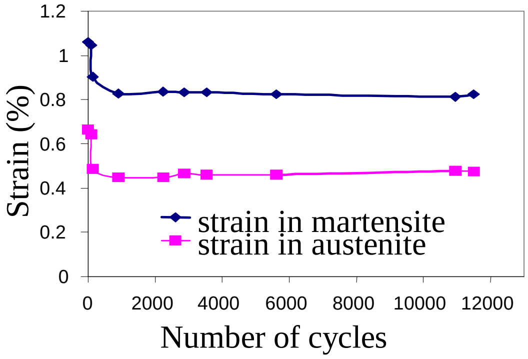

Figure 6. Strain as function of thermal cycles a fixed load of 0.19 N.

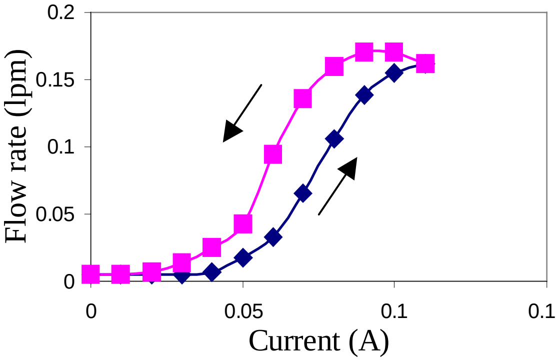

Figure 7. The flow rate of a assembled microvalve as function of electric current.

Fatigue tests for one million cycles

To investigate the lifetimes of microvalves, two additional fatigue tests have been performed without interruption for one million cycles. In the first test, a 1.2% strain was applied during the entire test using the z-axis motion on the stage of an optical microscope, as shown in Fig. 2. Stress-strain data acquired after one million cycles of actuation showed that the degradation of recoverable stress was about 3%. In the second test, an assembled microvalve was used. After one million cycles, the decrease in recoverable stress was less than 5%. Both tests show that the degradation of recoverable stress is low enough to be acceptable for most applications, and device lifetimes of more than one million cycles are expected.

Performance of microvalve

Patterned TiNi microactuators have been used as part of a MEMS microvalve device. Under the operating pressure of 240 kPa, the operating frequency of the microvalve was greater than 4 Hz with a flow rate of air of 0.17 liter per minute, as shown in Fig. 7. No leakage was observed in the closed state, using a flow meter with a minimum resolution of 0.005 lpm. The hysteresis in the flow rate is due to the phase transformation hysteresis seen in Fig. 3. More details about microrobotic applications of this microvalve are described elsewhere [5].

CONCLUSIONS

Equiatomic TiNi thin film SMAs were fabricated using co-sputtering, and the stress evolution of TiNi films was studied. Fatigue tests were performed to evaluate TiNi thin film actuators. Stable deflection after 1000 cycles were observed under fixed load, and a lifetime over one million cycles was demonstrated for an assembled microvalve. Under an operating pressure of 240 kPa, an operating frequency of about 4 Hz with a flow rate of 0.17 liter per minute was achieved.

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