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Microactuators of free-standing TiNiCu films

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Abstract
$Ti_{50}Ni_{47}Cu_3$ films were prepared using magnetron sputtering deposition, and their martensitic transformation and mechanical properties were characterized. Free-standing TiNiCu films showed an intrinsic two-way shape memory effect which is attributed to a composition gradient through the film thickness. Different types of TiNiCu microactuators, including microtweezers and microcages, have been successfully fabricated which employ this two-way shape memory effect for operation. Upon heating/cooling, the microtweezers showed both horizontal and vertical displacement due to combined shape memory and thermal effects. The microcage actuators could be opened/closed through substrate heating with a maximum temperature of 90 °C, or by electrical heating with a power less than 5 mW and a maximum frequency of 100–300 Hz. Issues related to the fabrication and applications, such as stability and beam bending after release, have been addressed.

(Some figures in this article are in colour only in the electronic version)

1. Introduction

Microactuators, such as microgrippers and microtweezers, have been identified as being of potential importance for biomedical applications, such as biopsy of cancerous tumours or capture and manipulation of cells for dissection. There are some basic requirements for these biological microtools, including small size, low operation temperature, low power consumption and biocompatibility. Attempts have been made by many research groups to develop microactuators for biological applications [1–3]. However, many technological challenges remain as most such devices have a high operation temperature, a complex and costly design and relatively small displacements upon actuation. TiNi-based thin film shape memory alloy (SMA) has been recognized as being promising for these applications [4–10]. The advantages of SMAs are their high power to weight ratio, large recovery stress and strain and biocompatibility. The work output per volume of thin film TiNi exceeds those of the other microactuation mechanisms. The application of TiNi films in MEMS also facilitates simplification of mechanisms with flexibility of design. Compared with TiNi thin films, the ternary TiNiCu thin films show less composition sensitivity to martensite transformation temperature and a narrower temperature hysteresis, which makes them more suitable for microactuator material [7, 9]. In this study, TiNiCu films were deposited using a sputtering method and their martensitic transformation and mechanical properties were characterized. Different types of TiNiCu microactuators have been fabricated and characterized which all employ the observed intrinsic two-way shape memory effect of free-standing TiNiCu films.

2. Film deposition and characterization

Films of $Ti_{50}Ni_{47}Cu_3$ alloy were deposited on 100 mm diameter Si(100) wafers that had previously been coated in a

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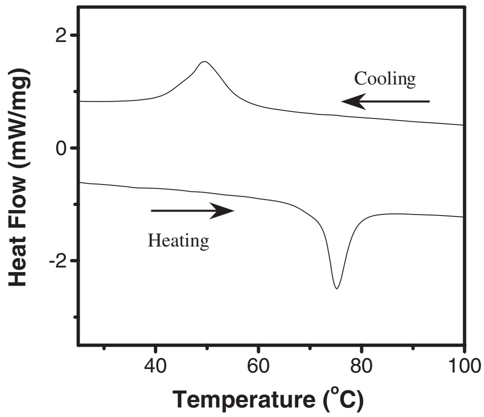

Figure 1. The result of DSC measurements of a free-standing TiNiCu film showing a single stage phase change.

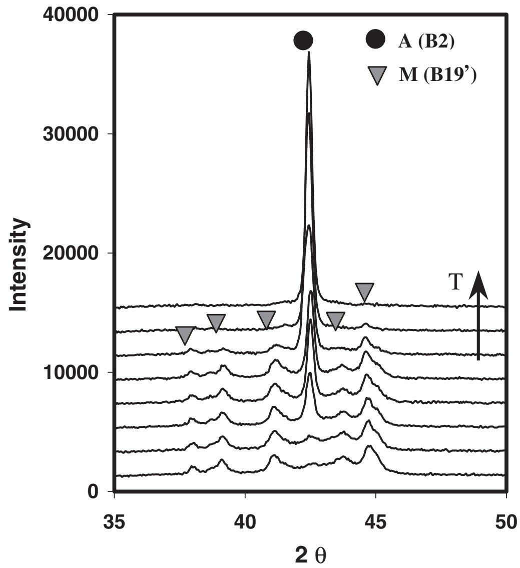

Figure 2. *In situ* XRD analysis of TiNiCu films at different temperatures showing the single stage martensitic transformation.

250 nm thick layer of silicon nitride (deposited by low pressure chemical vapour deposition). The TiNiCu films were deposited by magnetron sputtering in an argon gas environment at a pressure of 0.8 mTorr from a $Ti_{55}Ni_{45}$ target (using a 400 W rf electric field) and a 99.99% pure Cu target (using a 2 W dc electric field). The distance between the substrate and the target was 100 mm. The deposition lasted for 4 h, during which time the substrate was rotated to improve uniformity. The variation of film composition is ±0.3 at.%. The film thickness is 3.5 μm. After the deposition, the films were annealed at a temperature of 650 °C in a vacuum at 1×10^{-7} Torr. The film composition was determined using energy dispersive x-ray microanalysis (EDX).

Figure 1 shows the result of differential scanning calorimetry (DSC) measurements performed on the free-standing TiNiCu films. The heating and cooling cycles reveal a single stage transformation between martensite and austenite. The martensitic transformation start and finish temperatures are 56.8 and 38.1 °C, and the austenite transformation start and finish temperatures are 65.0 and 80.3 °C, respectively. The change in the crystalline structure of the samples was analysed using an x-ray diffraction (XRD) system at different temperatures. This analysis, shown in figure 2, confirms the existence of the martensite (monoclinic, B19') phase at room temperature. As the temperature increases, the martensite gradually changes to austenite (B2, cubic), and the phase transformation reverses upon cooling.

The shape memory effect of the TiNiCu samples was demonstrated using a wafer curvature method. The wafer curvature was measured by a Tencor FLX-2908 laser system, and the residual stress was calculated from the change in the radius of curvature before and after deposition. The residual stresses were plotted as a function of temperature, and information about the martensitic transformation was extracted from this. Figure 3 shows that there is a clear hysteresis in the stress as a function of temperature: a sharp increase in tensile stress is seen corresponding to the phase transformation from martensite to austenite during the heating cycle; during cooling, the stress relaxes significantly, corresponding to the formation of a twinning structure due to the martensitic transformation.

Figure 4 shows that the free-standing TiNiCu films possess an intrinsic two-way shape memory effect with large displacements: the film is curled up at room temperature, but becomes flat when heated and curls up once more when returned to room temperature. The free-standing film was obtained by peeling off the film from the substrate after the deposition/annealing process. For bulk TiNi alloy, a one-way shape-memory effect is normally observed—the material returns to its original shape during heating after deformation in the martensitic phase, but not in cooling. The origin of the two-way shape memory effect observed in the TiNiCu films can be attributed to the difference in sputtering yields of titanium and nickel, which produces a compositional gradient through the film thickness [11, 12]. The film layer near the substrate is normally nickel rich, and no shape memory effect is observed, but the material may possess superelasticity. As the Ti/Ni content changes through the film thickness, the material properties change from being superelastic to having a shape

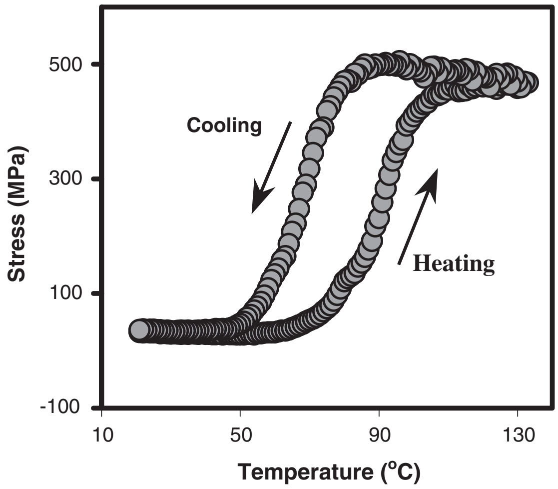

Figure 3. Stress evolution in TiNiCu samples during a heating and cooling cycle.

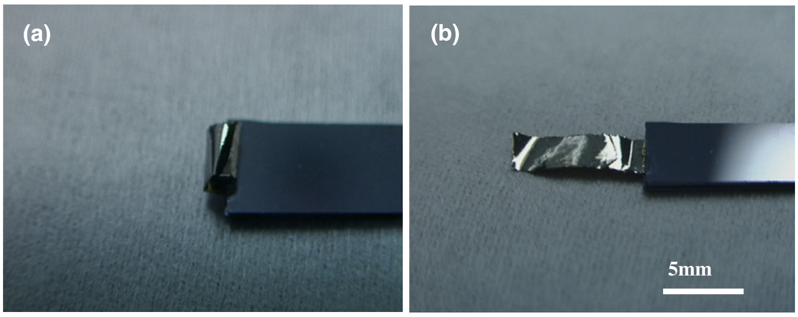

Figure 4. Two-way shape memory effect of free-standing TiNiCu films at (a) 20 °C and (b) 90 °C.

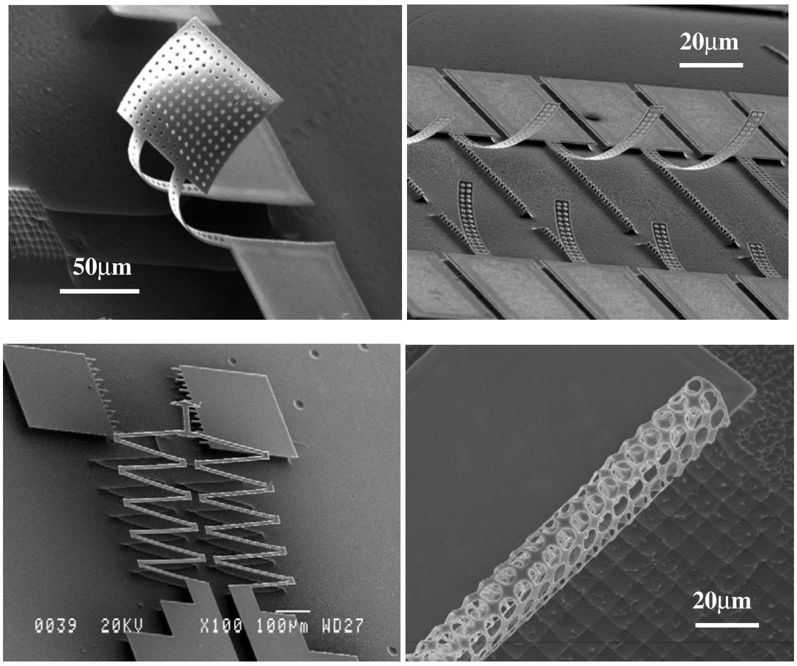

Figure 5. Free-standing TiNiCu structures: (a) cantilever mirror; (b) multi-length cantilever; (c) microspring; (d) microstent.

memory. A stress gradient is generated due to the changing microstructure and composition as a function of thickness. The bottom layer of material is under a compressive stress relative to the higher layers and so the film layer extends dramatically upon release from the substrate, causing free-standing structures to bend upward. When heated, the film layer returned to a flat position due to the shape memory effect.

The observed intrinsic two-way shape memory effect is of great practical importance, and in particular for the design of microactuators [13]. TiNi-based microactuators are normally fabricated using constrained TiNi films deposited onto silicon or other substrates, such SU-8 or polyimide [3, 4, 6]. Such structures provide a large actuation force, but the amount of deflection is quite small. Microactuators made from these free-standing TiNi films can provide large deflections from simple structures and MEMS processes. In this study, different microactuators have been fabricated based on free-standing TiNiCu films that exhibit the two-way shape memory effect, and these are reported in the following section.

3. Microactuator fabrication and characterization

Silicon substrates were used in this study that had previously been coated with 250 nm of silicon nitride. TiNiCu films were deposited using the method described in section 2. Microactuators were fabricated by photolithographically patterning 4.8 μm thick layers of AZ4562 photoresist on top of the TiNiCu films. $HF:HNO_3:H_2O$ (1:1:20) solution was employed to etch the TiNiCu films and form the microactuator patterns. The etch rate for the TiNiCu film is about 0.55 μm min^{-1} without significant etching of the Si_3N_4/Si substrate. With the remaining photoresist as the mask, the exposed nitride was removed using an rf reactive ion etch (rf-RIE) system with an SF_6 plasma (power of 100 W, 80 sccm, 100 mTorr), and the silicon substrate beneath the TiNiCu/SiN patterns was isotropically etched by SF_6 plasma until the free-standing TiNiCu structures were released. The etch rate of the Si substrate is about 0.6 μm min^{-1}. Figure 5 shows some examples of released structures which can be actuated by heating/cooling through the two-way shape memory effect.

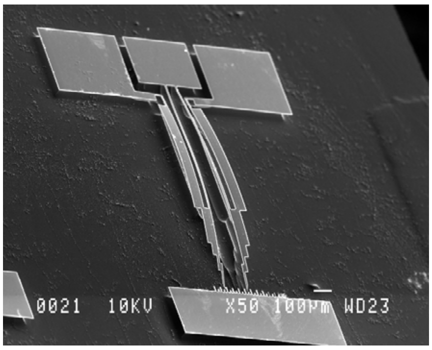

Figure 6. A fabricated free-standing TiNiCu microtweezer structure.

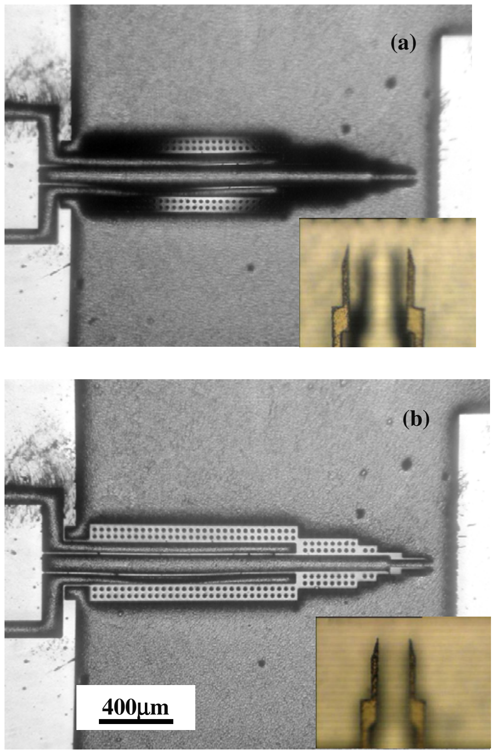

Figure 7. Optical microscopy images of the microtweezer showing its operation during heating due to the shape memory effect: (a) 20 °C; (b) 80 °C (the inset is the tip of the microtweezer).

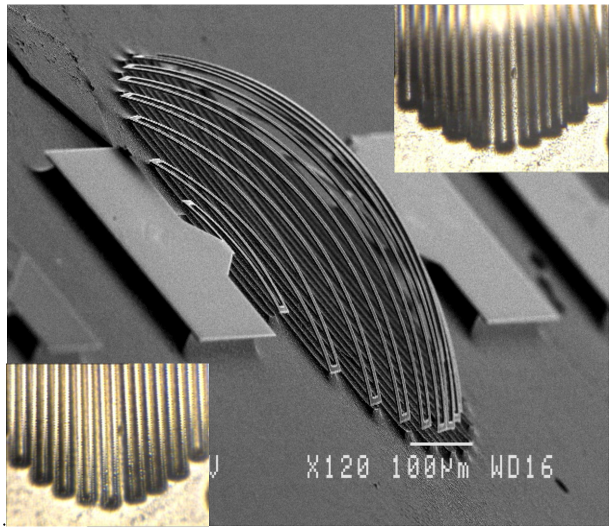

Figure 8. A downwards bending microcage structure; upper inset: microcage in bending up position at 20 °C; lower inset: microcage in flat position at 80 °C.

The actuation performance of the released microactuators was evaluated by either heating the structure on a hot plate up to a maximum temperature of 100 °C or by passing a current through the patterns and resistively heating the film in air. In the latter case, the current was provided by a Keithley 224A voltage/current power supply generating a square-wave voltage signal, and this allowed actuation as a function of frequency to be analysed. The displacement of the microgrippers was recorded on a video camera attached to a metallurgical microscope.

Figure 6 shows a released microtweezer based on TiNiCu shape memory alloy that can be actuated both horizontally and vertically with respect to the plane of the substrate, making full use of both thermal and shape memory effects. The device consists of two arms, and each arm itself is made up from two beams, one of which is wide and one of which is narrow. When a current is passed through each arm, the higher current density in the narrow beam causes it to heat up more than the wide beam, leading to a horizontal movement of up to 50 μm through a simple difference in thermal expansion between the two beams [14, 15]. With the application of TiNiCu film, vertical movement is possible due to the shape memory effect. At room temperature, the stress gradient in the TiNiCu causes the structure to bend out of the plane of the surface by up to 50 μm, and there is a tip opening up to about 40 μm (see figure 7(a)). When heated electrically, the shape memory effect causes the structure to bend down and become flat, thus producing a vertical movement of about 50 μm. At the same time, the microgripper tip closes with a displacement of about 20 μm, as shown in figure 7(b). With further electrical heating, the difference in thermal expansion between the two beams causes the opening of the microtweezer structure, which has been well documented in [14, 15]. During cooling, the movement of the microgripper and its tip reverses. Therefore, the fabricated microtweezer could perform the movements in the horizontal and vertical directions based on both shape memory and thermal effects.

Microcages provide an alternative means to the microactuator for confining small objects with the advantage that no force is applied directly to the object itself [2, 16, 17]. Microcages are therefore of particular interest for the manipulation of soft objects, such as biological cells. Figure 8 shows a fabricated microcage consisting of thin TiNiCu beams patterned to form a dome upon release from the substrate. The stress gradient in the TiNiCu thin film causes the dome to have a height of between 180 and 220 μm at room temperature. When heated electrically, the dome microcage becomes almost flat (see figure 8), returning to its original state upon cooling, forming a confining or trapping action.

A modified microcage design consisting of thin TiNiCu beams patterned into microfingers is shown in figure 9. After release from the Si substrate, the fingers of the microcage

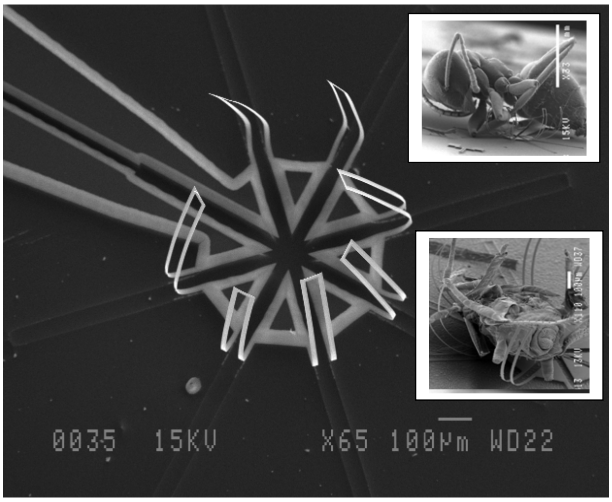

Figure 9. A TiNiCu bending up microcage structure and examples of capturing of (a) an ant and (b) an aphid.

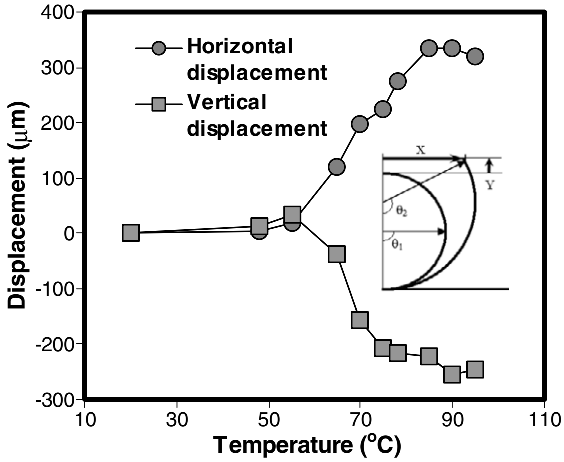

Figure 11. Vertical and horizontal displacement of the TiNiCu fingers in the microcage as a function of substrate temperature, where negative displacements denote a downwards displacement.

curl up to form a structure due to the gradient stress that can be used to confine an object at room temperature. The microfingers uncurl when heated above 55 °C and become flat at a temperature above 80 °C, as shown in the series of figures 10(a)–(e). Upon cooling, the structure returns to its original curled shape. Figure 11 plots the experimentally measured changes in the horizontal and vertical displacement of the microcage fingertips as a function of substrate temperature when heating with a hot plate. Horizontal and vertical displacements of hundreds of micrometres are

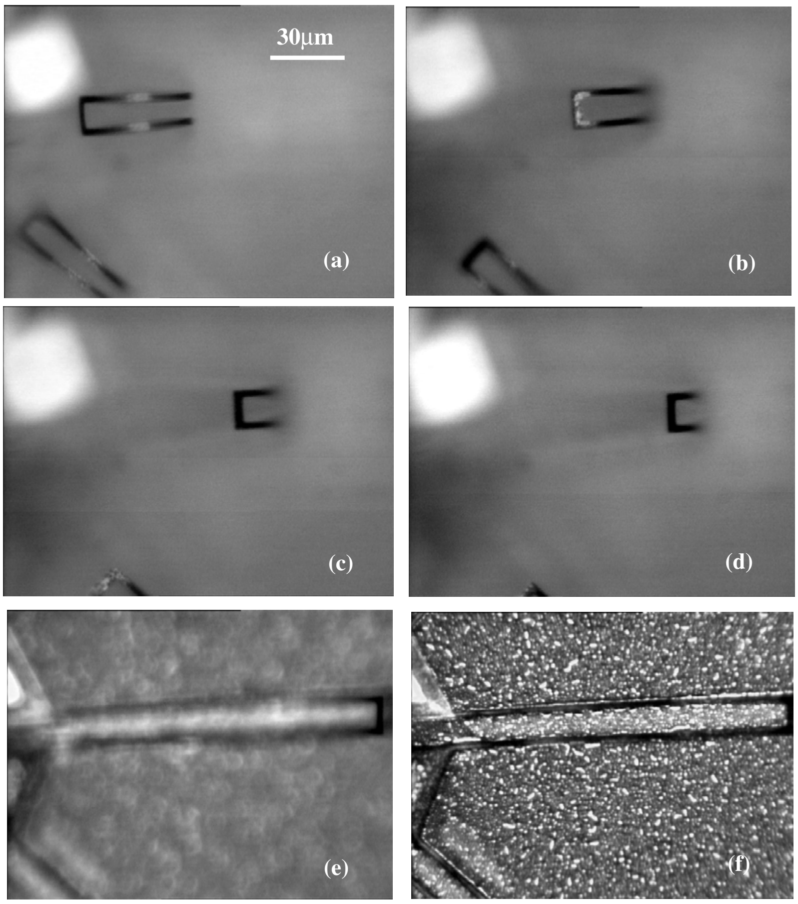

Figure 10. Optical microscopy images of microcage opening heated using a hot plate (a) 20 °C; (b) 55 °C; (c) 65 °C; (d) 70 °C; (d) 75 °C; and (d) 85 °C.

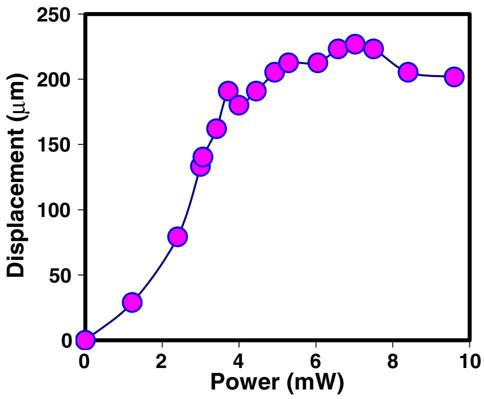

Figure 12. The horizontal displacement of the fingers of the microcage as a function of input power applied.

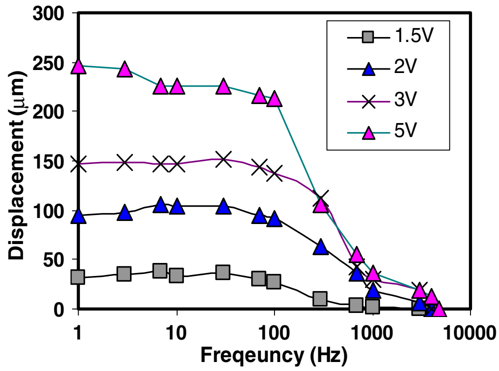

Figure 13. The horizontal displacement of the fingers of the microcage as a function of voltage and frequency of the applied actuation square wave.

achievable at temperatures less than 100 °C. It should be noted that negative values of vertical displacement indicate that the cage fingers bend downwards upon actuation. The fabricated microcage has been thermally cycled between 20 and 100 °C for more than 100 cycles, and optical microscopy observation did not reveal apparent degradation.

The microcage can be actuated by passing a current through the TiNi microfingers. Figure 12 shows the horizontal displacement of the fingers achieved for varying applied power. Significant displacements are observed for input powers between 1.5 and 5 mW as the shape memory alloy heats up through the transition temperature. Above 5 mW, the increase rate of tip displacement gradually decreases until reaching a power of 7 mW, above which the displacement slightly decreases. Excessive heating through the application of very high powers (>20 mW) resulted in visible changes in the colour of the TiNiCu—an indication of oxidation. The microcage can be used to capture microscale objects, with examples shown in figure 9, in which an ant and an aphid were captured.

Figure 13 shows the measured tip displacement produced by passing a current through the metallic layers as a function of the voltage amplitude and frequency of the square wave signal applied. For frequencies below ~100 Hz, the tip displacement increases with applied voltage. However, above ~100 Hz, the displacement decreases with increasing frequency for all applied voltages, indicating that about 10 ms is required to cool the microstructure due to the thermal capacity of the system. This places a maximum operating frequency of between 100 and 300 Hz for this microcage design. The realization of high frequency microactuators utilizing TiNi-based thin films has been a challenge. The thin film TiNi-based devices, such as microactuators and micropumps [18–21], generally have maximum operating frequencies in the range from 1 to 100 Hz. It should also be noted that the frequency is strongly dependent on the medium in which the device is placed, as well as the mass and dimension of the structure. Actuation in liquid, or thinner beams, and smaller structure dimension, may permit higher operating frequencies to be achieved due to easier thermal dissipation into the liquid.

The microcage structure has been electrically tested up to 18 000 cycles (with a fixed frequency of 20 Hz). With a low applied power less than 5 mW, and with the increase of thermal cycles up to a few thousand cycles, there are slight decreases in the horizontal displacement, as well as the original positions, but these did not change much afterwards even up to 18 000 cycles. The initial changes can be attributed to the training process, a common phenomenon for shape memory alloy. For the TiNi-based films, at the initial actuation stage, the repeated phase changes will alter the microstructure and hysteresis of the transformation and in turn lead to changes in transformation temperatures, transformation stresses and strains [5]. The recovery stress of TiNi films was found to decrease dramatically in the first tens of cycles, and became stable after hundreds of cycles [7]. However, at a power above 5 mW, the maximum displacement of the cantilever tip gradually decreases. There is a gradual shift in the room temperature position of the microfingers, and the tips of microfingers do not return to their original position after 18 000 cycles. The reason is attributed to the fatigue and degradation problem, which may be attributed to the defects in the film. With the power increased to above 8 mW, there is a significant shift of the finger tip, especially at the beginning of working cycles. This is attributed to the thermal degradation due to the excessive heating effect. It is apparent that the current or power applied to these microcages has a dramatic effect on the stability or fatigue properties. This issue should be addressed during application of TiNiCu microactuators.

Results also showed that, during the plasma releasing process, the bending mode of the TiNi beams gradually changes. During the initial release of the structure from the substrate (which may take about 25 min in 100 W SF_6 plasma), the beams bend downwards. Further release etching for another 10–15 min results in the bending up of the structure. Figure 14 shows a critical transition condition, in which longer beams bend upwards, whereas short beams remain bending downwards. The film/substrate interfacial layer could affect the bending of the free-standing TiNi structures. In this study, the TiNiCu films were deposited onto a Si_3N_4 interlayer of 250 nm thickness. During deposition and high temperature post-annealing, there is an interlayer of about 50 nm thickness between the film and substrate, where an interdiffusion layer and Ti–N bond formation can be identified [22]. After release of the cantilever structure, the Si_3N_4 layer and its interfacial diffusion layer may not easily be removed in the etching

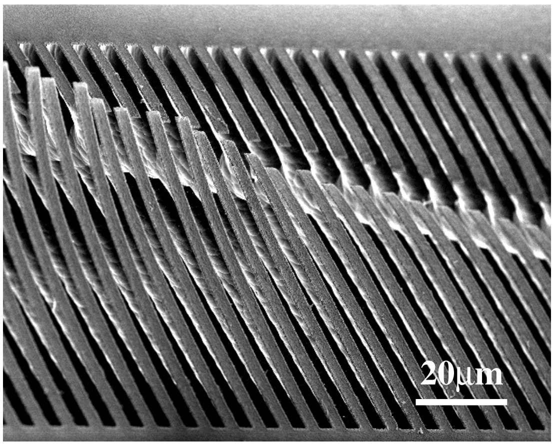

Figure 14. Released TiNiCu beams of varying lengths. The longer beams bend upward whilst the shorter beams are observed to bend downward.

process, because they are not exposed to plasma and the etch rate is relatively low. Due to the bimorph effect, the martensite layer could be extended easily, whereas the bottom layer is difficult to extend (due to silicon nitride), thus resulting in the bending down of the structure.

Further etching of the structure removes the bottom nitride and inter-diffusion layer; thus the film will bend up due to the change in the composition gradient throughout the film thickness. In the RIE dry release process, ion bombardment and thermal heating from the plasma on the material surface could induce an extrinsic gradient stress in the films, which may cause the structure to bend up gradually. Surface composition analysis using an energy dispersive x-ray analyser (EDX) on the etched surface of the TiNiCu films showed 3–5 at.% of sulphur. This is from the SF_6 plasma. Sulfur ions are implanted into the film surface, forming an intermixing layer, and this layer thickness could increase with the etching duration and power, causing the surface layer to lose the shape memory effect. Therefore, the film top layer could not be easily extended after the release etch. However, the bottom layer could have superelasticity due to the Ni-rich layer, and could be extended dramatically with the release of the film stress. This will result in the structures bending upwards.

4. Conclusions

Based on the good mechanical properties and two-way shape memory effect of free-standing TiNiCu films, different types of TiNiCu microactuators including microtweezers and microcages have been successfully fabricated. The microtweezer, which consists of a pair of heatuators, one thin hot arm and one wide cold arm, can have three stable positions due to both shape memory effect and thermal effect. Two types of microcage have been fabricated, i.e., one downward bending and one upward bending. With heating either using a hot plate or electrically, the microcages can be opened and closed, forming the functions to trap or hold microobjects. Results showed that the microcage could be actuated with a maximum temperature of 90 °C, and a low power, of less than 10 mW, with a maximum frequency of 300 Hz. The stability of movement of the free-standing structure depends much on the applied power. During plasma etching, the beam bending could be different due to the existence of an interlayer.

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