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CHARACTERIZATION OF A MICROPUMP ACTUATED BY TERNARY TINICu SHAPE MEMORY THIN FILMS

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ABSTRACT

Thin film SMAs have the potential to became a primary actuating mechanism for micropumps. In this study, a micropump driven by TiNiCu shape memory thin film is designed and fabricated. The micropump is composed of a TiNiCu/Si bimorph driving membrane, a pump chamber and two inlet and outlet check valves. The thickness, surface morphology and phase transformation property of TiNiCu film have been characterized by scanning electron microscope (SEM), atomic force microscopy (AFM), differential scanning calorimeters (DSC). Driving capacity of TiNiCu/Si biomorphic driving membrane has been investigated. The film surface shows a smooth and featureless morphology without any cracks, and the hysteresis width ΔT of TiNiCu film is about 10 °C. By using the recoverable force of TiNiCu thin film, the actuation diaphragm realizes reciprocating motion effectively. Experimental results show that the micropump driving by TiNiCu film has good performance, such as high working frequency, stable driving capacity, and long fatigue life time.

Keywords: micropump, TiNiCu SMA (shape memory alloys), thin film, magnetron sputtering, patterning

1. INTRODUCTION

In recent years, thin films of TiNi-based shape memory alloys (SMAs) are of increasing interest as actuating elements in silicon-based micro-electromechanical systems (MEMS) [1-3], which have the potential to become a primary actuating mechanism for micro-actuators, chemical analytics, medical fields, and biomedical applications [4-9]. TiNi-based thin films provide a large energy density, higher frequency response and long working life time at the microscale. Also they can be engineered into structures of micro-size dimensions, patterned with standard lithographic techniques and fabricated in batch, and the work output per volume of thin actuation mechanism such as bi-metallic, electrostatic, magnetic, piezoelectric, and

thermo-pneumatic, etc.. However, the martensitic transformation temperatures are very sensitive to their compositions and metallurgical factors [10]. Compared with the commonly studied binary TiNi thin films, the ternary TiNiCu thin films show less composition sensitivity to martensitic transformation temperature, a narrower temperature hysteresis (thus quick actuation response) [11, 12], stabilization of shape memory effects, superior fatigue property, etc., which makes them more suitable for micro-actuator application. Great efforts have been made to produce TiNiCu thin films using sputtering methods [13-15].

Successful implementation of micro-actuators using TiNiCu based films requires systematic studies on their transformation behaviors, especially for driving capacity of TiNiCu/Si. In this investigation, a type of micropump actuated by TiNiCu/Si composite diaphragm has been fabricated. TiNiCu thin film on silicon-based micropump has been successful prepared by magnetron co-sputtering of TiNi targets and Cu target. To realize that the micropump was actuated by shape recovery movements of TiNiCu/Si film during cooling and heating, patterning of TiNiCu thin film has fulfilled by etching. TiNiCu/Si film microstructure and phase transformation behavior have been characterized, the actuation property of TiNiCu thin film of micropumps has been analyzed. Working by recoverable force of TiNiCu thin film, the actuation diaphragm realizes reciprocating motion effectively.

2. EXPERIMENTAL

2-1. Material preparation

TiNiCu films were prepared by mix sputtering of a TiNi target (with atomic percentage of Ti 55.4% and Ni 44.6%) and a pure Cu target using magnetron co-sputtering system. The base pressure of the main chamber is 1×10^{-5} Pa, it was obtained by employing a turbo-molecular pump backed by a mechanical pump. High purity argon was used as sputtering gas. The flow rate of argon was controlled with a mass flow meter system. Polished (100)-type silicon actuator have used as the substrates. The substrate-to-target

distance was 100 mm. The RF power of the TiNi target was 450 W, and the DC power of Cu target was 8 W. Before each run, targets were pre-sputtered in a pure argon atmosphere for 15 min to clean the surface of the targets. The deposited films are $\sim 10 \ \mu m$ after sputtering 6.5 h, as shown in fig. 1. The argon pressure was changed from 0.8 Pa to 0.1 Pa in order to control Ti and Ni atomic percentage of deposited films [16]. After the deposition, the films were annealed at 873 K for 1 h for crystallization. The furnace could be cooled naturally.

Film composition is determined by energy dispersive X-ray microanalyser (EDX). The surface morphology of the films is investigated by atomic force microscopy (AFM). The martensitic transformation temperatures are measured by a differential scanning calorimeter (DSC) at a heating/cooling rate of 5 K/min over a temperature range from 233 K to 400 K.



Fig. 1 Cross-section morphology of sputtering deposited TiNiCu film

2-2. Material properties

Film composition of Ti, Ni and Cu is 50.7(at) %, 41.1(at) %, and 8.2(at) %, respectively. Fig. 2 shows AFM surface morphologies of the films deposited on the bare Si surface. There are many particles existed inside, the film surface shows a smooth



and featureless morphology without any cracks. Fig. 3 shows DSC result of TiNiCu film. From this measurement, the martensitic and austenitic start and finish transformation temperatures M_s/M_f and A_s/A_f are determined to 331K/318 K and 329 K/340 K, respectively.

The hysteresis width ΔT determined in the middle of the phase transformation is about 10 °C.



Fig. 3 DSC result for the TiNiCu film

2-3. Design and fabrication of micropump

2-3-1. The whole structure

The valve-less micropumps were fabricated using MEMS. Fig. 4 shows the Schematic diagram of the micropump with TiNiCu/Si driving diaphragm. Fig. 5 shows photograph of pump chamber structures. Its outer dimension is 12mm×10mm×1.8mm. The capacity of pump chamber is 4mm×4mm×0.9mm, and the diaphragm size is 4mm×4mm×12µm. When pulse currents heat the patterned SMA strips, an effective reciprocating driving behavior has been realized, and causes the pressure of the pump chamber to increase or decrease, driving the working fluid in or out through the check valves.



Fig. 4 Schematic diagram of the micropump with TiNiCu/Si driving diaphragm

2-3-2. Fabrication of micropump

The pump chamber and two diffuser elements were fabricated in a 500-µm-thick double–side polished silicon wafer using deep reactive ion etch step that etched the diffusers to a depth of 485-µm [11,12]. Fig. 6 shows the microstructure of diffuser element. Inlet and outlet holes were etched using an anisotropic etching solution (KOH). The TiNiCu driving film was coated with

negative photoresist layer and etched in а HF(44%)/HNO₃(54%)/H₂O₂(2%) solution at 293K. The solution can etch TiNiCu thin film well, but, Si-diaphragm was also etched at a high etch rate. It was difficult to achieve TiNiCu film patterning on silicon wafer. With water diluted solution, the etch rates of silicon wafer decreased sharply. TiNiCu can be etched with a practical rate of 120nm/min without etching Si diaphragm. The patterned driving films were successfully obtained within 85 seconds at 293K. Fig.6 shows microstructure of patterned thin film, the width of streaks was 50-µm; the distance between streaks was 50-µm too. The Au-Si eutectic bonding technology was used to bond two pump chambers with diffuser elements, the bonding was carried out at 675 K for 1 h in Ar atmosphere.



Fig. 5 Photographs of pump chamber structure



Fig. 6 Microstructure of diffuser element structure

3. TESTING RESULTS AND DISCUSSION

A laser interference micro vibrating instrument with $\pm 0.2\mu$ m measuring precision is used to measure the central displacement of the diaphragm during working. The deionized water is used as working fluid. The pump is driven by square wave current source. Fig. 7 compares the displacements of the driving diaphragm as a function of driving current in the cases of no liquid flow and with liquid flow. There is a distinct peak in the curve (without liquid flow), and no peak occurs when liquid flow is used. To obtain the same deflection of diaphragm, it needs much higher current to heat the TiNiCu strip in the case of liquid flow than that without liquid flow. This illustrates that the heat has been taken away effectively by the flowing liquid because of the good heat conduction of Si layer. Fig. 8 shows the relationship between the central displacement of the diaphragm and driving frequency. The central

displacement was almost constant at the frequency of 10-77Hz (without flow liquid) and 10-87Hz (with flow liquid), and the displacement decreases linearly as increasing the frequency. The reason is that the phase transformation rate could not catch up with the cooling and heating cycle when the driving frequency becomes too high. Therefore, the deflection of the diaphragm will decrease with increasing frequency. Fig. 9 indicates the displacement of



Fig. 7 Displacements of the driving diaphragm as a function of driving current in the cases of no liquid and with liquid flow



Fig. 8 Relationship between the central displacement of the diaphragm and driving frequency



Fig. 9 Deflection of driving diaphragm vs. driving number

the driving diaphragm as a function of driving cycle number by square wave current. The displacement decreased with increasing driving number when driving cycles number is less than 10^3 . As driving cycles number exceeded 10^3 , the displacement is almost constant. This indicates that the TiNiCu thin film has stabilization of shape memory effects, and stable working of TiNiCu/Si bimorph driving membrane can be realized. More than 2×10^6 driving cycles are achieved without decreasing the displacement of driving diaphragm, so TiNiCu/Si bimorph driving membrane has longer fatigue life.

4. CONCLUSIONS

A micropump driven by TiNiCu shape memory thin film is designed and fabricated. TiNiCu film is successfully prepared on actuator membrance of micropump by magnetron co-sputtering method. The film surface shows a smooth and featureless morphology without any cracks, M_s/M_f and A_s/A_f are 331K/318 K and 329 K/340 K, respectively. The hysteresis width ΔT is about 10 °C. The patterned driving films were successfully obtained by HF(22%) /HNO₃(27%) / H₂O₂(1%) /H₂O(50%) solutions. TiNiCu/Si bimorph driving membrane has excellent driving frequency attains to 87 Hz, and more than 2×10^6 driving cycles is realized. Experimental results show that the micropump driving by TiNiCu film has high working frequency, stable driving capacity, and long fatigue life time.

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REFERENCES

- S. Miyazaki and A. Ishida, 1999, "Fatigue life of Ti–50 at.% Ni and Ti–40Ni–10Cu (at.%) shape memory alloy wires", Mater. Sci. Eng. A 273-275, pp.658.
- [2] H.J. Zhang, and C.J. Qiu, 2006, "A TiNiCu thin film micropump made by magnetron co-sputtered method", Materials Transactions, 47, pp.532.
- [3] J. Z. Chen, and S. K. Wu, 2000, "Chemical machined thin foils of TiNi shape memory alloy", Materials Chemistry and Physics, 58 pp.162.
- [4] Y. Liu, M. Kohl, K. Okutsu and S. Miyazaki, 2004, "A TiNiPd thin film microvalve for high temperature applications", Mater. Sci. Eng. A 378, pp.205.

- [5] Yongqing Fu, Weimin Huang, Hejun Du, Xu Huang, Junping Tan, and Xiangyang Gao, 2001, "Characterization of TiNi shape-memory alloy thin films for MEMS applications", Surface and Coatings Technology, 145, pp.107.
- [6] Dong Xu, Li Wang, Guifu Ding, Yong Zhou, Aibing Yu and Bingchu Cai, 2001, "Characteristics and fabrication of NiTi/Si diaphragm micropump", Sens. Actuators A, 93 pp.87.
- [7] Eiji Makino, Takashi Mitsuya and Takayuki Shibata, 2000, "Micromachining of TiNi shape memory thin film for fabrication of micropump", Sens. Actuator A, 79, pp.251.
- [8] Kaori Kuribayashi, Koichi Tsuchiya, Zhong You, Dacian Tomus, Minoru Umemoto, Takahiro Ito, and Masahiro Sasaki, 2006, "Self-deployable *origami* stent grafts as a biomedical application of Ni-rich TiNi shape memory alloy foil", Sci. Eng. A, pp.131.
- [9] M. Kohl, D. Dittmanm, E. Quandt, B. Winzek, S. Miyazaki and D. M. Allen: Mater, 1999, "Shape memory microvalves based on thin films or rolled sheets", Sci. Eng. A 273-275, pp.273.
- [10] Po-Yen Hsu and Jyh-Ming Ting, 2002, "Growth and characteristics of TiNiCu thin films", Thin Solid Film 420-421, pp.524.
- [11] Yong Liu, and Hejun Du, 2003, "RF magnetron sputtered TiNiCu shape memory alloy thin film", Mater. Sci. Eng. A, 339, pp.10.
- [12] S. Miyazaki, T. Hashinaga and A. Ishida, 1996, "Martensitic transformations in sputter-deposited Ti-Ni-Cu shape memory alloy thin films", Thin Solid Films, 281, pp.364.
- [13] Yongqing Fu and Hejun Du, 2003, "Effects of film composition and annealing on residual stress evolution for shape memory TiNi film, Mater. Sci. Eng. A, 342, pp.236.
- [14] Yongqing Fu, Hejun Du, Sam Zhang, and YanWei Gu, 2005, "Stress and surface morphology of TiNiCu thin films: effect of annealing temperature", Surface and Coatings Technology, 198,pp.389.
- [15] Hejun Du and Yongqing Fu, 2004, "Deposition and characterization of $Ti_{1-x}(Ni,Cu)_x$ shape memory alloy thin films", Surf. Coat. Technol. 176, pp.182.
- [16] E. Stemme, G. Stemme, 1995, "A valve-less planar fluid pump with two pump chambers", Sens. Actuator A, 47, pp.549.
- [17] A. Olsson, Goran Stemme, and E. Stemme, 2000, "Numerical and experimental studies of flat-walled diffuser elements for valve-less micropumps", Sens. Actuator A, 84, pp.165.
- [18] Anders Olsson, Olle Larsson, Johan Holm, Lars Lundbladh, Ove Ohman, and Goran Stemme, 1998, "Valve-less diffuser micropumps fabricated using thermoplastic replication", Sens. Actuator A, 64, pp.63.