PARAMETRIC STUDY OF AN ANNEALING PROCESS FOR A FABRICATION OF BIMORPH NITINOL-COPPER CANTILEVER USING QUARTZ TUBE FURNACE WITH ARGON OVERFLOW

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Abstract

The scope of this work was to study an annealing process in a quartz tube furnace with argon overflow for a fabrication of bimorph micro cantilevers. The bimorph micro cantilever consists of two layers of different materials, i.e. nitinol and copper. The effects of the annealing process were investigated under various temperatures, i.e. 500, 600 and 650^{o}C and pre-stressing conditions, i.e. a straight and curve mold. With X-ray diffraction and Energy dispersive spectroscopy techniques, the results showed that, among all annealing temperature conditions, the bimorph micro cantilever annealed at 650^{o}C exhibited more relatively oriented micro structures. In addition, during the annealing, the composition ratio of nickel and titanium was modified in a manner that nickel content reduced from the as deposited nickel-titanium layer. Regarding the effects on a deflection of bimorph micro cantilever, it was found that the annealing temperature had significant effect while that of pre-stressing conditions was relatively less. From the experiments, when heating the fabricated cantilevers at 125^{o}C, the 22.5-mm long bimorph micro cantilever annealed at 650^{o}C with curve mold exhibited the largest deflection that was around 3.6 mm.

Keywords: NiTi, bimorph, quartz tube furnace, annealing temperature, pre-stressing

Introduction

Since a decade ago, shape memory alloy, especially nitinol (NiTi), has been drawn much attention in a field of Micro-Electrol-Mechanical systems (MEMS) because this kind of material has an ability to recover large transformation when it is heated and cooled. Among various shape memory alloy materials, NiTi is relatively suitable for micro sensor and actuator such as low operating temperature, large deflection and high actuating force. As a result, its applications are found in several micro systems such as actuator (Shin et al. 2005; Fu et al. 2001), sensor (Chan et al. 2008), pump (Sassa et al. 2012; Makino et al. 2001) and gripper (Takeuchi and Shimoyama 2000; Gill et al. 2001). The fabrication of NiTi thin film for these devices is consisted of two main processes that are sputtering deposition and post-sputtering annealing (Fu et al. 2004, etc.). Especially, the post-sputtering annealing is considered to be an important process to customize the device's performance. The post-sputtering annealing is a process to form a crystalline structure of NiTi, and typically is performed at high temperature. The post-sputtering annealing can be performed by several methods such as heating in high vacuum furnace (Chung and Chan 2011; Fu et al. 2008), local heating by high power laser (Wang et al. 2005) and heating in quartz tube furnace with inert gas overflowing (Sassa et al. 2012). Temperature and time duration for annealing are different in each past work. Chung and Chan (2012) annealed a NiTi thin film micro cantilever with 5 μm thick in a vacuum furnace at pressure of $2x10^{-6}$ mbar and temperature of $700^{o}C$ for 30 minutes. Fu et al. (2008) annealed the DLC/NiTi microcage made from a NiTi film with 800 nm thick at $480^{o}C$ for 30 minutes in a high vacuum condition. Makino et al. (2001) annealed a NiTi thin film with 6 μm thick at $500^{o}C$ for 1 hour in vacuum.

In this paper, a bimorph micro cantilever was developed. It consists of two materials that are NiTi and copper. With this combination, the cantilever bends due to mismatch of strain between the two materials when the cantilever's structure is heated or cooled. In this study, the annealing process was extensively studied. During the annealing at high temperature, a diffusion of material into another material and oxidation of material itself might be happened. Therefore, the effects of different temperature for annealing process were investigated under two pre-stressing conditions in a quartz tube furnace with argon overflow. Characterization of these Ni-Ti thin films at different annealing temperature conditions was performed by X-ray diffraction (XRD) and Energy dispersive spectroscopy (EDS) techniques. At the end, the deflection of fabricated micro cantilevers that were annealed with different pre-stressing conditions was examined.

Methodologies and Results

Fabrication Process

The bimorph micro cantilever was fabricated by depositing Ni-Ti thin film on a freestanding copper structure following the work of Wongweerayoot et al. (2011). The substrate used was a stainless steel plate that was polished until its surface roughness smaller than 100 nm. Then, copper film with a thickness of around 30.0±5.6 μm was constructed by electroplating technique at a rate of 30 μm/hour inside photoresist mold on the stainless steel plate. The shape and size of copper structure are shown in Figs. 1a and b. After removing the photoresist, the copper film was naturally detached from the stainless steel substrate. After that, the Ni-Ti thin film was then deposited by DC magnetron sputtering on each freestanding copper structure. The distance between copper structure and Ni-Ti target (Ni to Ti=1.0) during sputtering was around 6 mm. The base pressure in the deposition was set at $1x10^{-5}$ mbar, and the DC power was controlled at current of 0.2 A with argon pressure of $3x10^{-3}$ mbar. At these conditions, the Ni-Ti film was deposited at a rate of 1.16 μm/hour until its thickness was around 5.58±1.3 μm.

Annealing Conditions

After that, the bimorph micro cantilever was annealed in the quartz tube furnace whose schematic setup is shown in Figure 2. Before the annealing process could be started, air inside the quartz tube was removed by vacuum pump. After turning off the vacuum pump, argon gas was circulated in order to force oxygen out from the quartz tube. This process was repeated three times before the annealing process starts.

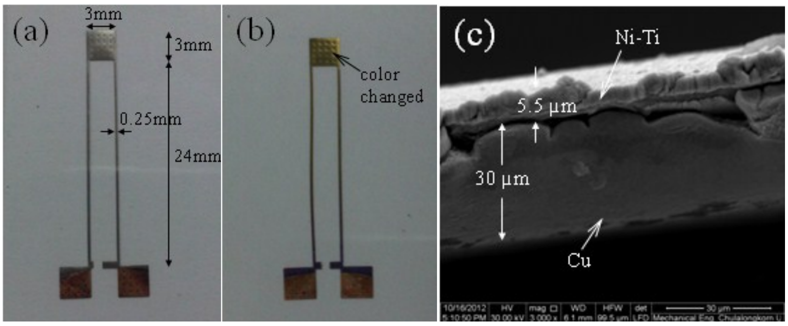

Figure 1 Configuration of bimorph micro cantilever; a) cantilever before annealing, b) cantilever after annealing, c) Scanning electron micrograph (SEM) of cross-section of bimorph micro cantilever

To prevent oxidation, argon gas over flow was employed during the annealing process. The heating was controlled at a heating rate of 10°C/minute. The various annealing temperature, i.e. 500, 600 and 650°C, was held constant for 30 minutes. After holding for 30 minutes, the quartz tube furnace was turned off resulting of a natural cooling rate of 3°C/minute.

Figure 3 shows XRD results of Ni-Ti thin film and copper structures at various annealing temperatures. For the structures without annealing and with annealing at 500 °C, the XRD results exhibited no peak of NiTi (110) at 2θ of 42.5°. It was implied that Ni-Ti thin films in both cases were not oriented and in an amorphous form. On the other hand, for the structures with annealing at 600 and 650°C, the XRD results exhibited austenite phase of NiTi (110), and the peak became larger at higher annealing temperature. It was implied that the as deposited Ni-Ti films were oriented after annealing. Moreover, the crystalline structure of Ni-Ti film annealed at 650°C was better oriented than that annealed at 600°C. However, at this high annealing temperature, the alloy structure between nickel and copper (NiCu) occurred as the small peak at 2θ of 44° was observed. Besides, in the Ni-Ti thin film, Ni_3Ti was probably occurred during the annealing process as the peak at 2θ of 43° became higher than other conditions (Zhang et al. 2007; Sanjabi and Barber 2010).

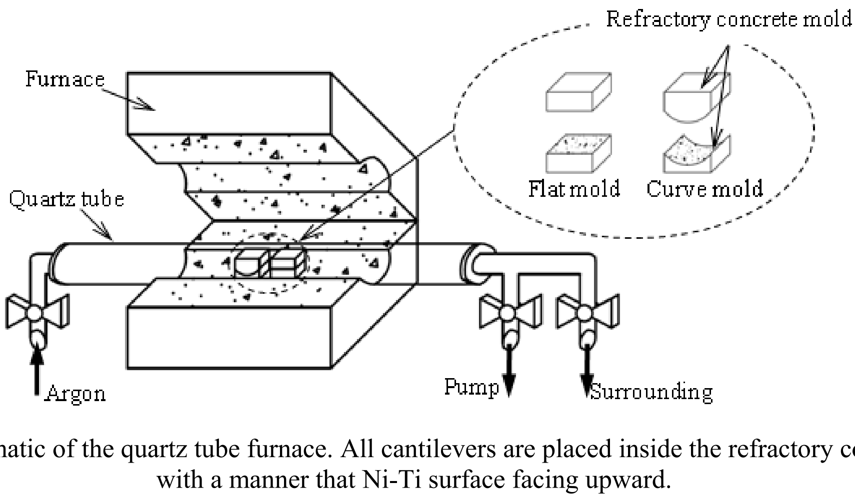

Figure 2 Schematic of the quartz tube furnace. All cantilevers are placed inside the refractory concrete molds with a manner that Ni-Ti surface facing upward.

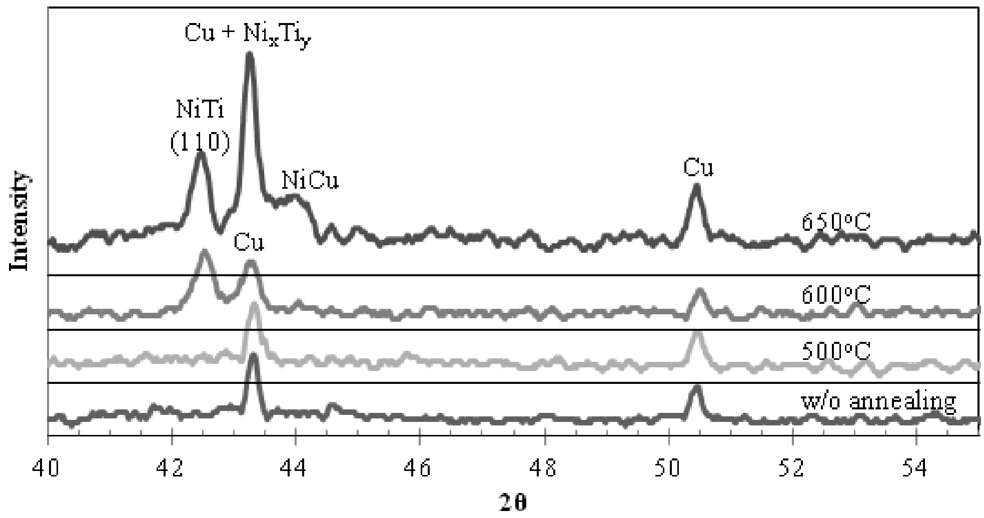

Figure 3 X-ray diffraction pattern of bimorph cantilever under room temperature test for various annealing conditions; without annealing, annealing at 500, 600 and 650^{o}C.

Figures 4a and b show EDS results comparing the composition on a surface of Ni-Ti film (depth of 1-2 μm from its surface) before and after annealing at 650^{o}C, respectively. Before annealing, or for as deposited Ni-Ti thin film, the composition was 38.9%-titanium and 61.1%-nickel. On the other hand, after annealing at 650^{o}C, the composition was changed to 38%-titanium, 33.5%-nickel and 28.5%-oxygen. These compositions were varied around 2-3% on different locations on the Ni-Ti film. From the results, it was implied that oxidation was not avoidable with argon overflow technique. In addition, the ratio between titanium and nickel was changed from 38.9:61.1 before annealing to 53.1:46.9 after annealing at 650^{o}C, or nickel content was decreased after the annealing. One possibility was a diffusion of nickel content into copper structure as NiCu alloy structure found in XRD experiments.

Pre-Stressing Conditions

In this experiment, the bimorph micro cantilevers were annealed at 650^{o}C in two pre-stressing conditions, i.e. a flat and curve refractory concrete mold. According to the curve mold, it had a radius of curvature around 22 mm. To investigate the effects of pre-stressing condition, the deflections of three bimorph micro cantilevers, i.e. without annealing, with annealing in flat mold and curve mold, were examined. Figure 5 shows a schematic of the experimental setup. The bimorph micro cantilevers were clamped with glass slides as their lengths to the tip equal to 22.5 mm. Then, the cantilevers were placed on a hotplate and covered by a glass box in order to precisely control temperature inside. After turning on the hotplate, temperature inside the glass box as well as that of the bimorph micro cantilevers increased and it was monitored using an infrared thermometer. The heating rate was controlled at around 1.4^{o}C/minute. To cool down the cantilevers, the hotplate was turned off, and the temperature then naturally dropped at a cooling rate of around 1.3^{o}C/minute. The photographing technique using a micro-digital camera was employed for measuring the instantaneous deflection while heating up and cooling down with uncertainty of 100 μm. In all experiments, the temperature was controlled between 30 and 125^{o}C.

Figures 6a and b show the captured images of the bimorph micro cantilevers with two conditions of pre-stressing comparing with that without annealing, when their temperature was at room temperature and at 125°C, respectively. Shapes of the cantilever at different pre-stressing conditions differed after annealing. The shape of the cantilever with annealing in curve mold was similar to that without annealing, while that with annealing in straight mold bended in an opposite direction. After heating, all cantilevers tended to bend to the right or toward Ni-Ti surface. The deflection of these cantilevers was calculated by comparing these two images and measuring the lateral distance where the tip was deflected. Three tests had been done for each condition, and the averaged deflections of cantilevers with two pre-stressing conditions while heating up and cooling down comparing with that without annealing are shown in Figure 7.

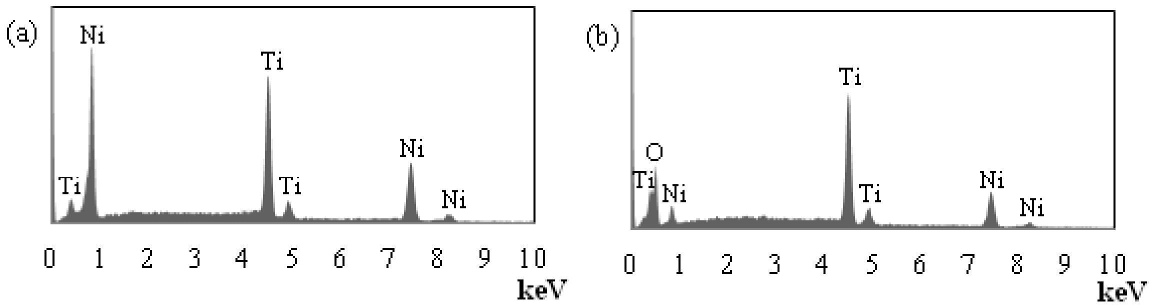

Figure 4 Energy dispersive spectroscopy of Ni-Ti thin film on a surface of bimorph micro cantilever; a) before annealing, b) after annealing at 650°C.

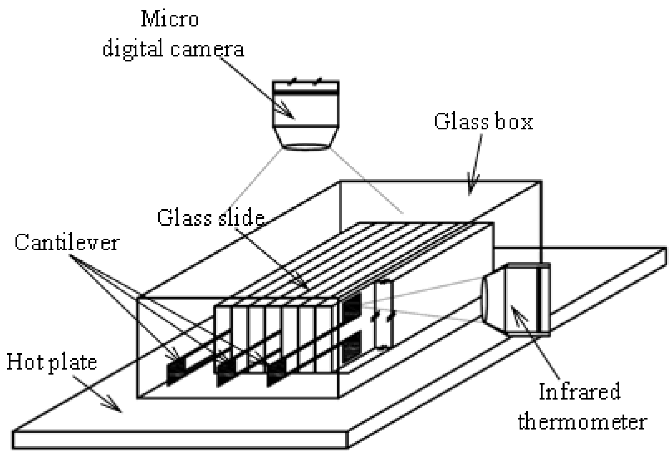

Figure 5 Schematic of the experimental setup.

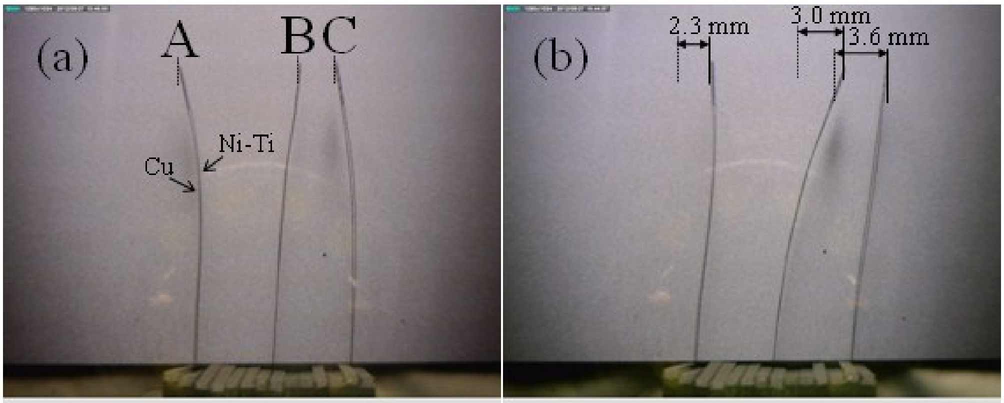

Figure 6 Photographs of each bimorph cantilever - A: w/o annealing, B: annealed with flat mold and C: annealed with curve mold; a) at 30°C, b) at 125°C.

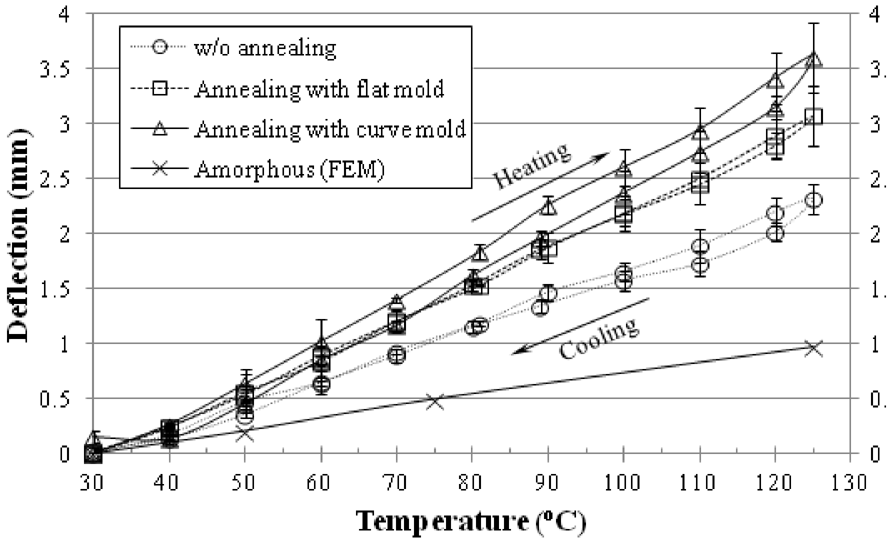

Figure 7 Averaged deflection of each bimorph micro cantilever for two pre-stressing conditions. FEM results were calculated with Young's modulus equals to 120 and 110 GPa, while the thermal expansion coefficient equals to 16.5 and 14.5 ppm/°C, for copper and Ni-Ti, respectively.

From the graph, the hysteresis of the deflection while heating up and cooling down was quite small. The results also showed that the deflection of the bimorph micro cantilever annealed with curve mold was slightly larger than that annealed with flat mold. On the other hand, the deflection of the bimorph micro cantilever without annealing was relatively small comparing with the others. At 125°C, the bimorph micro cantilever without annealing, with annealing in flat mold and in curve mold had deflection about 2.3 mm, 3.0 mm and 3.6 mm, respectively. From the results, it was implied that the annealing process, or the occurrence of oriented micro structures, had significant effects on the deflection while the effects of pre-stressing condition were relatively small.

Conclusion

This work presents a parametric study of an annealing process in a quartz tube furnace with argon overflow that is very important process for a fabrication of bimorph nitinol-copper (NiTi-Cu) micro cantilever. After depositing Ni-Ti thin film with its ratio of 1.0 on the copper structure, the effects of annealing temperature, i.e. 500, 600 and 650^{o}C, and pre-stressing conditions, i.e. a flat and curve mold, were investigated. The conclusions can be drawn as follows.

1. The annealing temperature affects the formation of crystalline structure of as deposited Ni-Ti. From this study, the temperature was higher than 600^{o}C where the NiTi crystalline structure started orienting, and it was in austenite phase at room temperature. Moreover, at higher annealing temperature, the NiTi crystalline structure oriented more completely. However, at 650^{o}C, the occurrence of NiCu and Ni_3Ti was also observed.

2. The nickel content near a surface of Ni-Ti film after annealing at 650^{o}C was decreased. Nickel probably diffused toward copper layer underneath and reacted to form NiCu. In addition, the oxide was also observed after annealing at this high temperature.

3. Comparing between annealing temperature and pre-stressing condition, the latter one had less effect on the deflection of bimorph micro cantilever. After heating to 125^{o}C, the cantilever with annealing in curve mold provided the largest deflection, and the deflection became smaller for the cantilever with annealing in flat mold and that without annealing, respectively. For 22.5 mm long cantilever, the deflection for those conditions was 3.6, 3.0 and 2.3, respectively.

Acknowledgements

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