



Microstructure and shape memory behavior of $\text{Ti}_{55.5}\text{Ni}_{44.5-x}\text{Cu}_x$ ($x = 11.8\text{--}23.5$) thin films



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ABSTRACT

The microstructure and shape memory behavior of $\text{Ti}_{55.5}\text{Ni}_{44.5-x}\text{Cu}_x$ ($x = 11.8\text{--}23.5$) thin films annealed at 773, 873, and 973 K for 1 h were investigated. None of the films except the $\text{Ti}_{55.4}\text{Ni}_{32.8}\text{Cu}_{11.8}$ film annealed at 773 K for 1 h had any precipitates in the B2 grain interiors and their grain sizes were small (less than 1 μm). Increasing the annealing temperature caused grain growth and thus a decrease in the critical stress for slip and an increase in the martensitic transformation start temperature (M_s). The grain size was also controlled by the growth of a second phase. In the three-phase equilibrium region of Ti_2Ni , Ti_2Cu and TiNi , Ti_2Cu grains grew faster than Ti_2Ni grains, leading to a decrease in the critical stress for slip and an increase in the M_s temperature with increasing Cu content.

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1. Introduction

Ti–Ni–Cu thin films formed by sputtering are attractive candidates for powerful microactuators [1–4]. They have advantages such as a small temperature hysteresis, a stable shape memory effect, a single-stage transformation, a relatively large recoverable strain, and insensitivity of their transformation temperatures to Cu content in comparison with Ti–Ni films. Until now, there have been some reports [5,6] on the shape memory behavior of Ti–Ni–Cu bulk alloys and thin films [4], but their compositions were restricted to approximately 50 at% Ti. The authors investigated the microstructure and shape memory behavior of Ti–Ni–Cu films with 51.5 at% Ti and 6.5–20.9 at% Cu and reported that the shape memory behavior was improved by the presence of coherent precipitates in the B2 grain interior and the solid-solution hardening of Cu in the B2 matrix [7]. However, a recent combinatorial study [8] demonstrated that a much broader composition range of the Ti–Ni–Cu system provides a reversible phase transformation, suggesting that Ti–Ni–Cu films with a higher Ti content are also promising as microactuators. Very recently, Ishida et al. [9] investigated the microstructures of Ti–Ni–Cu thin films with 15.5 at% Cu

and varying Ti content from 44.6 to 55.4 at% and found that the structure of a Ti–Ni–Cu film with 55.4 at% Ti was significantly different from those of films with 51.5 at% Ti; the former films had a duplex structure with a small grain size, whereas the latter films had a large amount of precipitates in the grain interiors. Furthermore, it was reported that a three-phase equilibrium region of TiNi, Ti_2Ni , and Ti_2Cu phases extended over a wide range of Cu content in the Ti-rich Ti–Ni–Cu composition area. These differences in the structure and the phase constitution are expected to modify the shape memory behavior of the Ti-rich Ti–Ni–Cu films reported in a previous work [7]. The objective of the present paper, therefore, is to clarify the effects of annealing temperature and Cu content on the microstructure and shape memory behavior of Ti–Ni–Cu films with a high Ti content (55.5 at% Ti), focusing, especially on the presence of the three-phase region of TiNi, Ti_2Ni , and Ti_2Cu phases.

2. Experimental

Amorphous Ti–Ni–Cu films with a constant Ti content of 55.5 at% and various Cu contents of 11.8–23.5 at% ($\text{Ti}_{55.4}\text{Ni}_{32.8}\text{Cu}_{11.8}$, $\text{Ti}_{55.4}\text{Ni}_{28.9}\text{Cu}_{15.7}$, and $\text{Ti}_{55.4}\text{Ni}_{21.1}\text{Cu}_{23.5}$) were prepared by sputtering. The films were deposited on glass substrates using a carousel-type magnetron sputtering apparatus [7]. Two 4-in. targets of Ti (99.99%) and Ni (99.99%) and a 3-in. target of Cu (99.99%) were used

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to obtain the desired compositions. The direct current (dc) power of the Ni and Cu targets were varied from 196 to 119 W and from 48 to 89 W, respectively, whereas the dc power of the Ti target was maintained at 1000 W. The chamber was pumped down to a base pressure of 3×10^{-5} Pa and deposition was conducted at an Ar gas pressure of 0.13 Pa. The substrate holder was rotated at 60 rpm (1 s^{-1}) to obtain composition homogeneity during sputtering. The deposition time was 2.5 h and the final thickness was $\sim 8 \mu\text{m}$. The film compositions were determined using inductively coupled plasma atomic emission spectroscopy (ICP-AES).

After sputtering, the deposited films were peeled off the glass substrates and then annealed at 773, 873 or 973 K for 1 h to induce crystallization. The heat treatment was conducted in a vacuum furnace equipped with infrared lamps. X-ray diffraction (XRD) with Cu $K\alpha$ radiation and Si powder as an internal standard was employed to identify the phases formed in the films. The microstructure of the annealed films was observed by transmission electron microscopy (TEM; Jeol JEM-2000FXII) with a heating stage. Thin foils for TEM observations were prepared by twin-jet electropolishing in an electrolyte solution consisting of 95% acetic acid and 5% perchloric acid by volume.

The shape memory behavior of the films was measured with a small tensile tester equipped with an automatically controlled heater (Shimadzu TMA-50). The size of the test samples was $0.4 \text{ mm} \times 5 \text{ mm}$ (gauge portion) with a thickness of $\sim 8 \mu\text{m}$. The test involved loading a sample at 473 K, cooling it to 173 K at a rate of 10 K/min, and then reheating it to the original temperature at a rate of 10 K/min. A series of strain–temperature measurements was conducted using the same sample under various constant stresses from 20 to 120 MPa in steps of 20 MPa and then from 120 MPa to 1 GPa in steps of 40 MPa.

3. Results and discussion

3.1. XRD analysis

Fig. 1 shows X-ray diffraction patterns of the films annealed at 773, 873, and 973 K for 1 h (Si reference peaks are not included in the figure because Si peaks are located at 28.47 and 47.30° for the 111 and 220 reflections, respectively.) All the films had a Ti_2Cu phase. In addition, a Ti_2Ni phase was identified in the $\text{Ti}_{55.4}\text{Ni}_{32.8}\text{Cu}_{11.8}$ films annealed at 773, 873, and 973 K and in the $\text{Ti}_{55.4}\text{Ni}_{28.9}\text{Cu}_{15.7}$ films annealed at 873 and 973 K. The peak ratio of the Ti_2Ni phase to the Ti_2Cu phase was higher for the $\text{Ti}_{55.4}\text{Ni}_{32.8}\text{Cu}_{11.8}$ films than for the $\text{Ti}_{55.4}\text{Ni}_{28.9}\text{Cu}_{15.7}$ films, and it

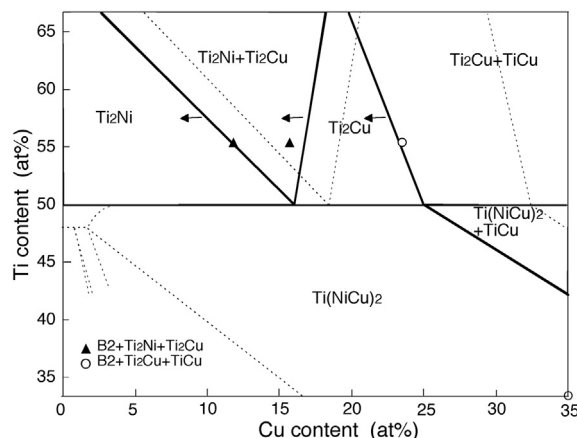


Fig. 2. Equilibrium phase diagram of Ti–Ni–Cu (973 K) (After Fig. 9 in Ref. [7]). Broken lines show the equilibrium phase diagram at 1073 K.[10].

increased with increasing annealing temperature. On the other hand, the $\text{Ti}_{55.4}\text{Ni}_{21.1}\text{Cu}_{23.5}$ films annealed at 773 and 873 K exhibited a TiCu phase, but this phase was not detected in the film annealed at 973 K. All these features can be reasonably explained using the equilibrium phase diagram reported in a previous paper [9].

Fig. 2 shows a part of the equilibrium phase diagram at 973 K. The phase constitution of the present films annealed at 973 K for 1 h is consistent with the phase diagram shown in Fig. 2. In a previous paper [9], it was suggested that the stable region of a Ti_2Cu phase shifts to a lower Cu content with decreasing temperature, as shown by the arrows in the figure. Therefore, the appearance of a TiCu phase in the $\text{Ti}_{55.4}\text{Ni}_{21.1}\text{Cu}_{23.5}$ films annealed at 873 and 773 K and the disappearance of a Ti_2Ni phase in the $\text{Ti}_{55.4}\text{Ni}_{28.9}\text{Cu}_{15.7}$ film annealed at 773 K are rationalized in terms of the phase stability.

The peak positions of the $110_{\text{B}2}$ reflection from the films annealed at 773, 873, and 973 K are shown in Fig. 3. The peak positions of the $110_{\text{B}2}$ reflection from the $\text{Ti}_{50}\text{Ni}_{(50-x)}\text{Cu}_x$ films were also plotted in the figure for comparison. The lattice constants for the $\text{Ti}_{55.4}\text{Ni}_{32.8}\text{Cu}_{11.8}$ and $\text{Ti}_{55.4}\text{Ni}_{28.9}\text{Cu}_{15.7}$ films annealed at 973 K were nearly the same, reflecting the equilibrium among the three phases ($\text{B}2$, Ti_2Ni , and Ti_2Cu). The compositions of these phases are considered to be almost the same for the $\text{Ti}_{55.4}\text{Ni}_{32.8}\text{Cu}_{11.8}$ and $\text{Ti}_{55.4}\text{Ni}_{28.9}\text{Cu}_{15.7}$ films, but the volume ratios between the Ti_2Ni and Ti_2Cu phases were different, as mentioned earlier. On the other

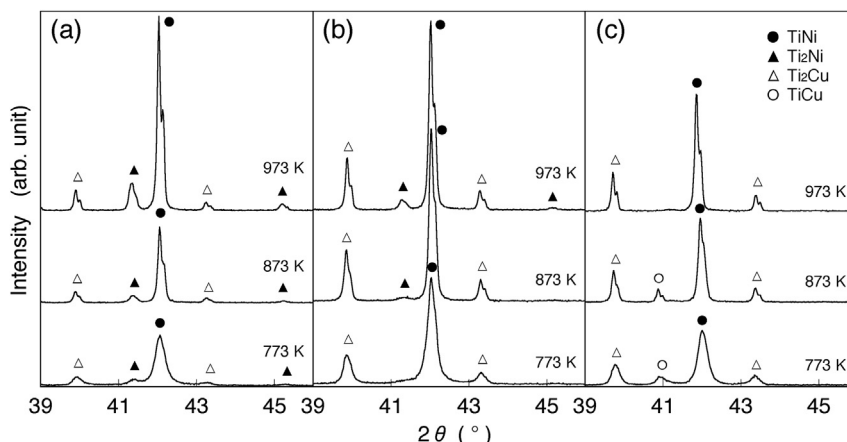


Fig. 1. XRD patterns of (a) $\text{Ti}_{55.4}\text{Ni}_{32.8}\text{Cu}_{11.8}$, (b) $\text{Ti}_{55.4}\text{Ni}_{28.9}\text{Cu}_{15.7}$, and (c) $\text{Ti}_{55.4}\text{Ni}_{21.1}\text{Cu}_{23.5}$ films annealed at 773, 873, and 973 K for 1 h.

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