



Different mechanical response of TiNi film induced by the shape of indenter during nanoindentation

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ABSTRACT

Nanoindentation was conducted for magnetron sputtered TiNi thin film, which exhibits different mechanical responses induced by varied indenter shapes. Superelasticity and incomplete phase transition ($A \rightarrow R$) were clearly depicted by spherical indentation. Under the peak load of 500 μN , the depth recovery ratios are 100% and 68% determined by spherical and Berkovich indentations, respectively. The different response is due to the varied stress distribution beneath two types of indenters. Large plastic deformation hinders the reverse phase transition ($R \rightarrow A$) in Berkovich indentation, resulting lower recovery. Moreover, significant difference in indentation size effect for TiNi film was revealed comparing to traditional metals. And phase transition contributed considerably to the abnormal hardness evolution.

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1. Introduction

TiNi thin films become promising materials for sensors or actuators in micro-electro-mechanical systems (MEMS), because they show the characteristics of large recovery stress and strain, high work density and high corrosion resistance [1–3]. For MEMS applications, the TiNi films are usually deposited on Si or other substrates [4]. The conventional characterization methods, such as tensile test (stress–strain curves) and differential scanning calorimetry (DSC) are no longer applicable to characterize the shape memory effect (SME) of constrained thin films. Therefore, how to correctly evaluate the SME and mechanical properties of constrained thin films on substrates becomes one of the important issues in characterization of TiNi films for MEMS applications [5].

With contributions from Oliver and Pharr and many others [6–9], the nano indentation technique has become an effective approach to test mechanical properties of elastic or elastoplastic materials. Nanoindentation testing could reveal the different elastic and plastic deformation behaviors of austenite and martensite, which is also promising for characterization of superelasticity (SE), phase transition, the SME and mechanical properties of constrained thin films [10–12]. Usually, nanoindentation instrument will be equipped with Berkovich and spherical indenters, both of which can be utilized to characterize mechanical properties of SMAs. In

the past years, researchers employed both Berkovich [13–18] and spherical [10,12,19] indenters to characterize SE and SME property of TiNi thin films. However, due to the shape difference of two indenters, obvious differences in load–displacement curves and deformation behaviors were observed [18,20–22]. According to the varied response caused by shape of indenters, some researchers gave their preferences [19,23] in selecting indenters for SME or SE characterization. However, no extensive comparison or interpretation has been made for the response of TiNi thin films to indenters and related physical mechanism.

In order to correctly evaluate the SME/SE and mechanical properties of constrained thin films with the help of nanoindentation, we conducted extensive examination of mechanical response of superelastic TiNi films to the two indenters. In the present research, we use nanoindentation to characterize mechanical properties superelastic thin films, comparing their mechanical responses induced by Berkovich and spherical indenters.

2. Experimental

The TiNi films were deposited on $4 \times 12 \text{ mm}^2$ p-type Si (111) substrates, using DC magnetron sputtering system. The Ni and Ti targets with 99.99% purity were employed. In order to ensure the purity of films, these targets were pre-sputtered for about 20 min to remove surface oxides. The substrates were rotated at 7 rpm during deposition to maintain the uniformity in the film thickness.

The TiNi thin film composition ($\text{Ti-}48 \pm 0.3 \text{ at.}\% \text{ Ni}$) was analyzed by energy dispersive spectroscopy attached to scanning

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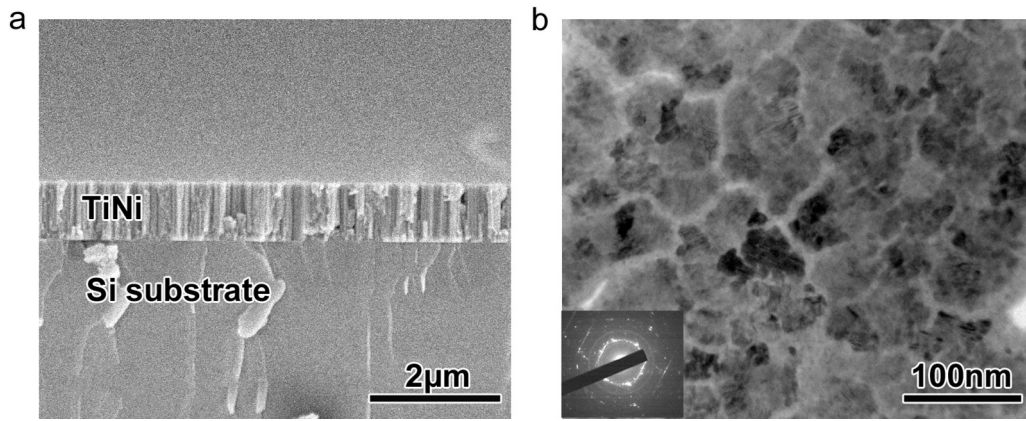


Fig. 1. (a) SEM cross section view of as-deposited 1 μm -thick TiNi film; (b) TEM micrograph of annealed 1 μm -thick TiNi film with grain size of ~ 60 nm (the inset is the corresponding SAED).

electron microscope (SEM, Hitachi S-4800, Hitachi, Japan). The thickness is about 1 μm . The as deposited film was annealed in 550 $^{\circ}\text{C}$ for 1 h under vacuum higher than 1×10^{-5} Pa. The film structures were characterized by X-ray diffraction (XRD) with Cu-target ($k\alpha_1 = 1.54060 \text{ \AA}$, Rigaku Ultimate III, Japan) and transmission electron microscope (TEM, JEM-2100, JEOL, Japan). The physical property measurement system (PPMS, Quantum Design Inc., USA) was employed to measure the characteristic phase transition temperatures of TiNi thin film. The heating/cooling rate is 2 $^{\circ}\text{C}/\text{min}$ in the temperature range from -200 to 100 $^{\circ}\text{C}$.

The film mechanical properties were characterized using nanoindentation (TI 950 Triboindenter, Hysitron Inc., USA). Both Berkovich and spherical indenters were used in the test. The radius of Berkovich tip is around 150 nm and that of spherical tip is about 1 μm . Peak load of 500, 700 and 1000 μN were selected for both types of indenters.

3. Results and discussion

The cross section view of as deposited TiNi thin film was shown in Fig. 1a, from which we can see a film with homogenous thickness of 1 μm . The as deposited TiNi film is in an amorphous state and after annealed in 550 $^{\circ}\text{C}$ (1 h) obvious nanocrystals (~ 60 nm) could be observed (Fig. 1b). The phase transition occurred between austenite (A) and rhombohedral (R) phase which has a negative temperature coefficient, as shown in Fig. 2a. The austenite finish temperature (A_f) is 22 $^{\circ}\text{C}$, therefore the TiNi film is expected in

almost complete austenite state at room temperature ($\text{RT} = 25^{\circ}\text{C}$). The phases were confirmed in XRD pattern (Fig. 2b).

Fig. 3 shows the load–displacement curves by Berkovich and spherical indenter under the peak force of 500 μN , 700 μN and 1000 μN . It's obvious that Berkovich indenter produced much deeper penetration depth than spherical one under the same load. Taking peak force of 1000 μN for instance, the maximum penetration depth in Berkovich indentation is 62 nm, which is about as twice deeper as in spherical indentation (37 nm). In addition, large residual depth was shown for Berkovich indentations. Comparably, the deformation with spherical indenter is almost recoverable. The differences between load–displacement curves of two indentations could be quantified by the depth recovery ratio η_h [24],

$$\eta_h = \frac{h_{\max} - h_r}{h_{\max}} \quad (1)$$

where $h_{\max}$ is the maximum penetration depth, h_r is residual depth after unloading. The depth recovery ratios were presented in Fig. 3a and b as insets. For both types of indenters, depth recovery ratio shows an increasing trend as peak force decreases. Spherical indentations always exhibit much higher recovery ratio than Berkovich ones under the same peak force. It should be noted that the spherical indentation reached the complete recovery when load is 500 μN .

The considerable difference in TiNi film response for two indenters under same load is due to the varied stress distribution beneath two indenters during indentation process. The distribution

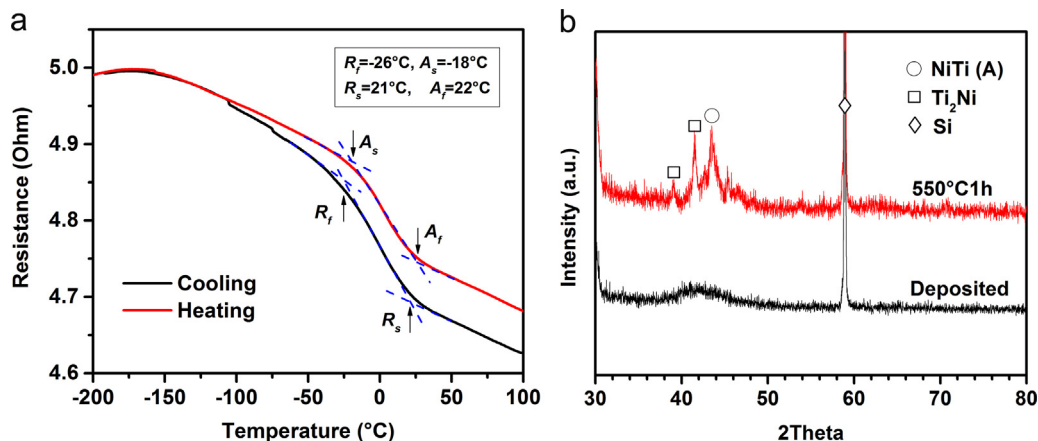


Fig. 2. (a) Resistance versus temperature curves of annealed TiNi film, (b) XRD patterns of annealed TiNi film.

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