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Effects of silicon nitride interlayer on phase transformation and adhesion of TiNi films

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Abstract

TiNi films with different Ti/Ni ratios were deposited on Si substrates with and without silicon nitride interlayer. Near-equiatomic TiNi films were found to have the lowest residual stress and the highest recovery stress regardless of the existence of silicon nitride interlayer. The addition of silicon nitride interlayer between film and Si substrate did not cause much change in phase transformation behavior as well as adhesion properties. X-ray photoelectron spectroscopy (XPS) analysis revealed that there is significant interdiffusion of elements and formation of Ti–N and Si–Si bonds at TiNi film/silicon nitride interface. Scratch test results showed that adhesion between the TiNi film and substrate was slightly improved with the increase of Ti content in TiNi films.
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1. Introduction

Shape memory alloys (SMAs) possess an array of desirable properties: high power to weight (or force to volume) ratio, thus the ability to recover large transformation stress and strain upon heating and cooling, peudoelasticity (or superelasticity), high damping capacity, good chemical resistance and biocompatibility [1–4]. More recently, thin film SMA has been recognized as a promising and high performance material in the field of microelectromechanical system (MEMS) applications [5–9]. When the TiNi films are deposited on Si substrate, there exist interfacial diffusion and chemical interactions at the interface whereby titanium and nickel silicides may form during high temperature deposition or postdeposition annealing [10,11]. These interfacial reaction products could be complex, heterogeneous and metastable, and a relatively thin reaction layer could have significant adverse effect on adhesion and shape memory properties. In microelectronics and MEMS processes, there need electrically and thermally insulating layer, sacrificial or diffusion barrier layer. Thermally grown SiO_2 is often used for this purpose. However, adhesion of TiNi film on SiO_2 layer (or on glass and polymer substrate) is poor owing to the formation of a thin intermixing fragile and brittle TiO_2 layer [10]. In a significant deformation or during a complex interaction involving scratch, this layer is easily broken, thus causing large-area peeling off. Adhesion of TiNi films on other interlayers (such as silicon nitride, polysilicon, etc.) is important for their successful MEMS applications, but few studies have been performed so far. In this study, TiNi thin films with three different Ti contents were sputtered onto Si wafer with and without silicon nitride (Si_3N_4) layer. Phase transformation characteristics, stress evolution and interfacial adhesion were studied in details.

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2. Experimental details

Single crystalline silicon wafers (100-type with thickness of 0.45 mm) were used as substrates. A layer of silicon nitride (0.2 μm thick) were deposited using low-pressure chemical vapor deposition with SiH_4 and NH_3 as precursors at a high temperature of 1073 K. TiNi films with three different compositions were prepared by cosputtering an equiatomic TiNi target and a pure Ti target. The base pressure of the main chamber was 1.33×10^{-5} Pa. The target-to-substrate distance was 100 mm, and Ar gas pressure was 0.133 Pa with a flow rate of 20 sccm. Plasma power of the Ti target was varied in order to adjust the film composition (see Table 1), and the substrate holder was rotated to enhance film uniformity.

Chemical compositions of the films were investigated by energy dispersive X-ray spectrometry. Measurements were done on five different regions on each of the sample, and the average values are listed in Table 1. Curvature changes of the film-deposited Si wafers were measured as a function of temperature using a Tencor FLX-2908 laser system with a heating and cooling rate of 1 K/min, from which the stress changes were derived [12,13]. Crystalline structures of the TiNi films were characterized using Philips PW3719 X-ray diffraction (XRD, Cu-Kα, 40 kV/30 mA). Interfacial adhesion of the TiNi film on substrate was assessed using a SHIMADZU SST-101 scanning scratch tester. A stylus of 15-μm tip radius was used at a load increased stepwise to a full load of 500 mN. During the scratch operation, the forward speed was set at 1 μm/s, scratch speed 10 μm/s and amplitude of scanning 50 μm. A sudden increase in frequency output indicated adhesion failure, and the normal load at which the damage incurs was used as a measure of the adhesion strength. X-ray photoelectron spectroscopy (XPS) analysis was performed on TiNi/Si and TiNi/Si_3N_4 interfaces using a Kratos AXIS spectrometer with monochromatic Al Kα (1486.71 eV) X-ray radiation. TiNi film was peeled off from both Si and Si_3N_4/Si substrates, and the peeled-off interfaces were immediately analyzed using XPS for interfacial chemistry. Wide-scan spectra were recorded in 1 eV step for all samples. Detailed core level spectra of different elements were recorded in 0.1 eV step. Curve fitting was performed after a Shirley background subtraction by a non-linear least square fit. The spectra were calibrated using the C 1s (284.6 eV) peak as a reference. For elemental depth profiling of the interfacial structure, an ion gun (Kratos MacroBeam) of 4 keV energy was used with high purity Ar gas.

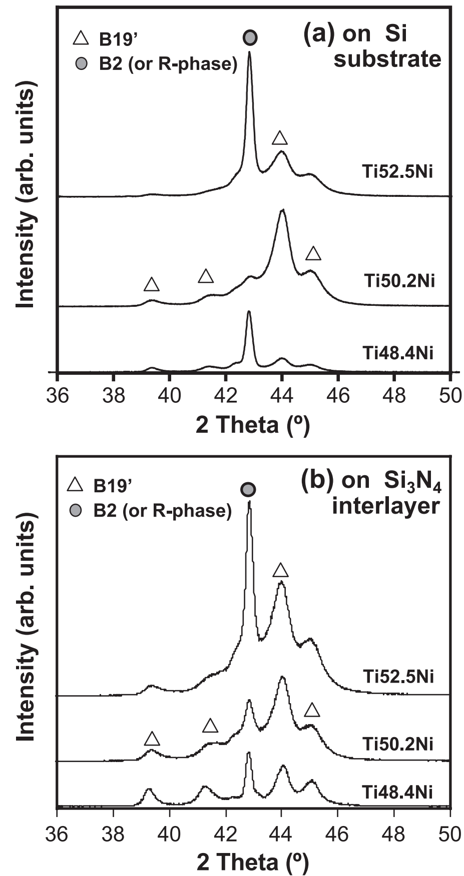

Fig. 1. XRD results of TiNi films with different Ti contents deposited on Si and Si_3N_4 interlayer: (a) on Si substrate (b) on Si_3N_4 interlayer.

3. Results and discussions

Fig. 1(a) shows XRD results of the TiNi films deposited on Si with three different Ti contents. The dominant phase for the near-equiatomic film (Ti50.2Ni) is mainly martensite with some untransformed R-phase/austenite. For the Ti-rich (Ti52.5Ni) and Ni-rich films (Ti48.4Ni), the phases are a mixture of austenite and martensite with more contents of austenite. Fig. 1(b) shows XRD results of the TiNi films deposited on the Si_3N_4 interlayer. There is not much difference for the XRD results for TiNi films on Si substrate or Si_3N_4 interlayers except the relative intensities of

Table 1
Sputtering conditions, film thickness and composition of TiNi films [radio frequency (RF), direct current (DC)]

Power (W)		Deposition temperature (K)	Duration (h)	Ar pressure (Pa)	Film thickness (μm)	Composition (at.%)	
TiNi (RF)	Ti (DC)					Ti	Ni
400	60	723	4	0.133	3.5	48.4	51.6
400	70	723	4	0.133	3.5	50.2	49.8
400	80	723	4	0.133	3.5	52.5	47.5

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