

# Sputtering deposited TiNi films: relationship among processing, stress evolution and phase transformation behaviors

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## Abstract

TiNi films were prepared by co-sputtering a Ti50Ni50 target and a Ti target under different process parameters. Stress in TiNi film on silicon substrate were evaluated using curvature method. A wide range of residual stress levels (either tensile or compressive) were found in the deposited films, depending significantly on Ti/Ni ratio, gas pressure, deposition temperature and/or annealing conditions. Stress evolution for as-deposited amorphous films during annealing process were systematically studied with the consideration of residual stress in as-deposited films; thermal stress; tensile stress due to densification crystallization; stress relaxation due to martensite transformation, etc. Upon heating, the crystallized TiNi films generated large tensile stresses when transforming from martensite to austenite, whereas during cooling, the stress relaxed significantly when austenite transformed back to the ductile martensite. The stress generation and relaxation behavior was significantly affected by film composition, deposition temperature and/or annealing temperature.

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**Keywords:** Sputtering; TiNi thin film; Shape memory; Martensite transformation; Stress; Curvature method; Annealing

## 1. Introduction

Thin film shape memory alloys (SMA) have the potential to become a primary actuating mechanism for micro-actuators and biomedical applications [1,2] and great effort has been made to produce TiNi based thin films using a sputtering technique [3,4]. However, successful implementation of micro-actuators using TiNi based films requires a good understanding of the relationship among the processing, microstructure and properties of TiNi films [3,5–11]. The establishment of a deposition process for precise control of composition and film quality is the primary challenge in creating SMA micro-actuators. Elaboration of various properties (such as shape memory effect, mechanical and fatigue properties, performance degradation, adhesion, uniformity, etc.) is necessary for the successful application of TiNi based micro-actuator application. A good thermo-mechanical modeling for martensitic transformation and shape memory effects will be beneficial for the design

of integrated micro-devices. There are some basic requirements for the successful application of TiNi films, such as the minimum thickness, composition uniformity across a large area, a good adhesion with substrate and free of macroscopic defects (such as pores and cracks, etc.).

Residual stress in the deposited films could become one of potential problems for their wide application, as it may influence not only adhesion between film and substrate, but also deformation of micro-electro-mechanical system (MEMS) structure, mechanics and thermodynamics of displacive transformation, as well as superelasticity effects, etc. [12]. Large residual stress could lead to either film cracking and decohesion in tension, or film delamination and buckling in compression [13]. Deposition conditions and composition in TiNi films could have important consequences with respect to the development of residual stress [14]. For a film–substrate system, possible origins of stress in thin films can be divided into three groups, i.e., thermal stress, intrinsic stress and phase transformation stress. Fig. 1 shows stress evolution during martensite transformation process as a function of temperature for a 4  $\mu\text{m}$

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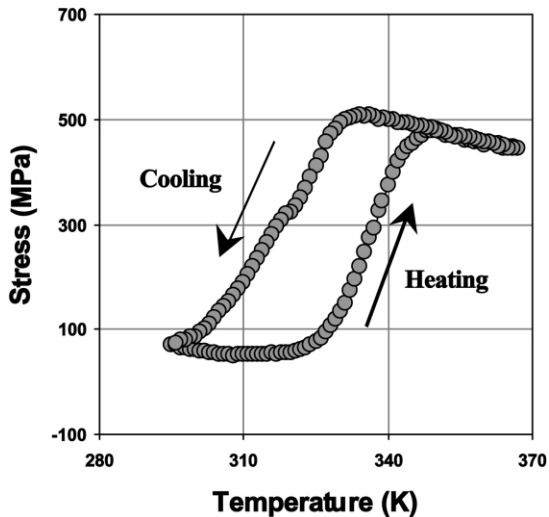


Fig. 1. Stress evolution curves during martensite transformation as a function of temperature for 4  $\mu\text{m}$  Ti50·2Ni film deposited at 723 K with Ar gas pressure of 3.2 mtorr.

Ti50·2Ni film on silicon substrate deposited at 723 K with an Ar gas pressure of 3.2 mtorr. The stress vs. temperature plot shows a closed hysteresis loop. At room temperature, tensile stress exists in the films. During heating, the stress increases significantly due to phase transformation from martensite to austenite. During cooling, martensitic transformation occurs and the tensile stress relaxes significantly due to the formation and alignment of twinning structures and shear-variant boundary motion [8,4,14]. Transformation temperatures and hysteresis, multi-stage transformation, magnitude of stress generation and relaxation rate can be obtained from the stress–temperature curves [14–16]. The recovery stress, given by the difference between the maximum and minimum stress, is an indication of actuating ability during application. Large recovery stress is preferred for the actuation purpose in MEMS applications. However, if the transformation temperatures (especially for martensite transition finish temperature,  $M_f$ ) are lower than room temperature, the stress–temperature evolution curve will not be closed at room temperature, and a certain value of un-recovered stress will contribute to the total residual stress in the deposited films.

There are many process parameters which significantly affect the phase transformation behavior and thus stress generation and recovery during phase transformation. A systematic study on their effects and precisely control of stress evolution is necessary. In this study, TiNi films with different Ti/Ni ratios were prepared under different process parameters. Evolution of stress values during film growth was measured and discussed as a function of gas pressure. Stress generation and

relaxation of as-deposited and/or post-annealed films during martensite transformation were studied.

## 2. Experimental

TiNi films were prepared by co-sputtering a Ti50Ni50 (at.%) target and a pure Ti target using magnetron sputter. TiNi target power was fixed at approximately 400 W (RF), while Ti target power was varied from 0 to 70 W (DC) to obtain films with different Ti contents [14,15,17]. The base pressure of main chamber was  $1 \times 10^{-4}$  mtorr. Four-inch (100)-type silicon wafers (with a thickness of 0.45 mm) were used as substrates. The substrate holder was rotated during deposition to achieve uniform deposition. Ar gas pressure varied from 0.8 to 10 mtorr. Films were either deposited at room temperature (with post-annealing at different temperatures) or at 723 K. The substrate-to-target distance was fixed at 100 mm. Surface and cross-section morphology was studied using a scanning electron microscope (SEM, Hitachi S5300). Surface morphology and roughness were obtained using a Shimadzu SFT-9800 atomic force microscope. Film composition was determined by energy dispersive X-ray microanalyzer. The change of crystalline structures for the deposited films was investigated using a Philips PW3719 X-ray diffractometer (XRD) at different temperatures between 303 and 423 K. The martensitic transformation temperatures were measured using a TA instrument 2920 differential scanning calorimeter (DSC) at a heating/cooling rate of 5 K/min. Curvature changes of film-deposited Si wafers were measured using a Tencor FLX-2908 laser system with a heating and cooling rate of 0.5 K/min, and the stress changes as a function of temperature were derived [8,4,14,15].

## 3. Results and discussions

### 3.1. Residual stress for Ti50Ni50 film deposited at room temperature and different pressures

Since the films were deposited at room temperature and substrate heating due to plasma was not significant, thermal stress has little contribution to residual stress. Residual stress in the deposited films is mainly attributed to the intrinsic stress formed during film nucleation and growth. Fig. 2 shows evolution of residual stress for the films deposited under different Ar gas pressures as a function of film thickness (corresponding to different deposition durations). The stress values are found to be sensitive to gas pressure.

With a gas pressure of 0.8 mtorr, the stress fluctuates but remains compressive. At a low gas pressure, the sputtered atoms pass to substrate with little loss of kinetic energy, causing much more atomic peening effect

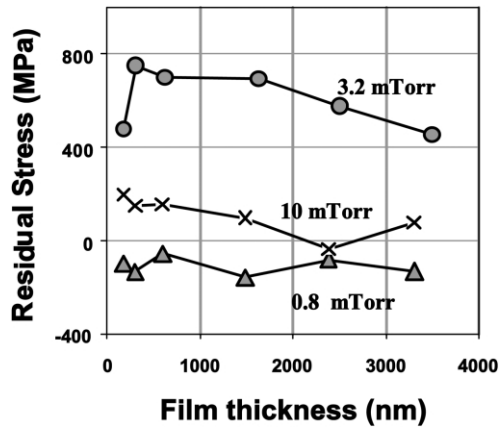
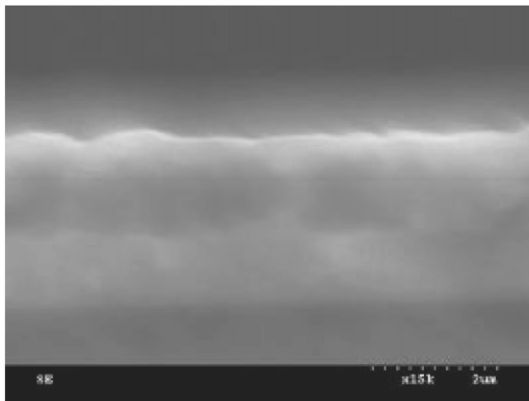


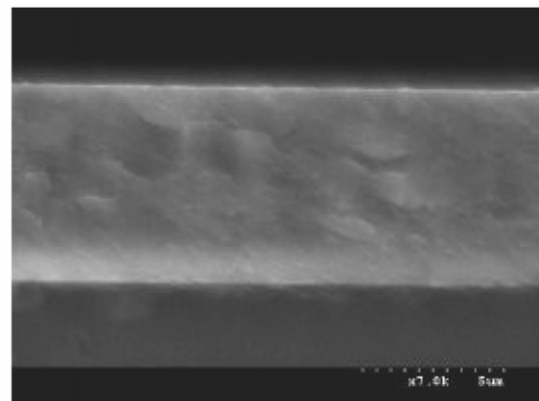
Fig. 2. Changes of residual stress for the as-deposited films under different Ar gas pressures with a function of film thickness.

and enhancing the mobility of adatoms by transferring kinetic energy to them. These could promote the generation of compressive stress. Fig. 3a shows cross-section morphology of the deposited film, revealing a dense and featureless structure. Compressive stress in film is often desired than tensile stress in applications, because most coating materials are stronger in compression than in tension.

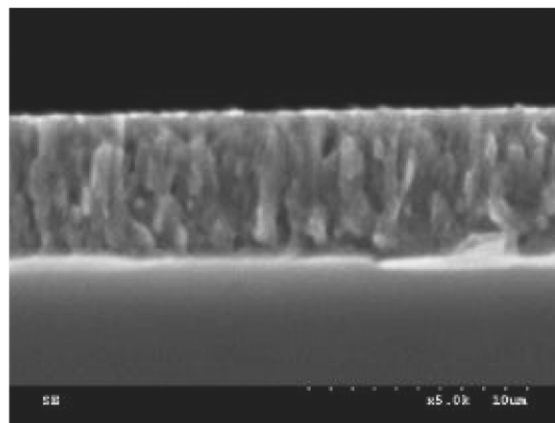
With the gas pressure increased to 3.2 mtorr, the stress values are all tensile. With increase of deposition duration, tensile stress is quite large at the initial deposition stage, reaches to a maximum value at a thickness of 300–400 nm, then slightly decreases with increase in film thickness (Fig. 2). At this gas pressure, compressive stress due to atomic peening effect is probably not so significant as those of films deposited at 0.8 mtorr, since the large number of gas molecules could collide with the sputtered atoms, reducing peening effect. The large tensile stress at the initial stage is probably induced by the island coalescence during film nucleation and for-



(a)



(b)



(c)

Fig. 3. Cross-section morphology of the deposited film (a) 0.8 mtorr; (b) 3.2 mtorr; (c) 10 mtorr.

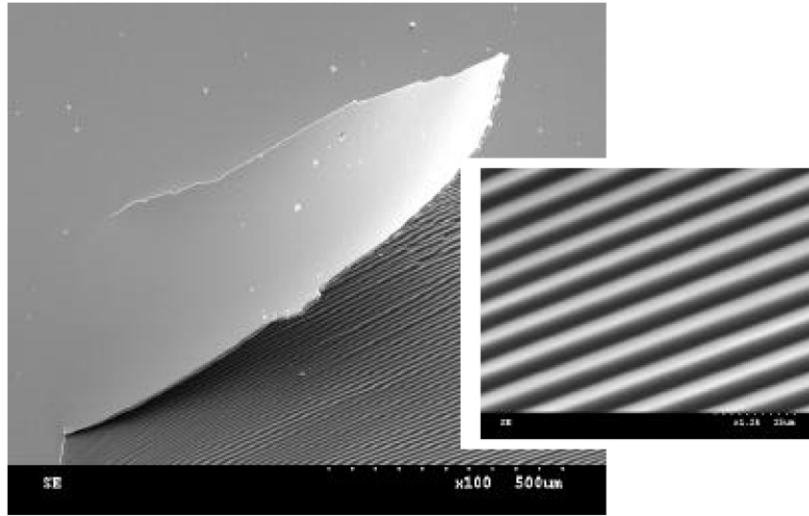


Fig. 4. Spallation of TiNi films due to large tensile stress, and cracks propagation in Si substrate.

mation [18–20]. Since the room temperature deposited TiNi films are usually in amorphous state, the stress evolution models for amorphous clusters in thin films can be used to explain the generation of large tensile stress during island (or nano-crystal) coalescence process at the initial film formation stage (due to local energy minimization in a dynamic equilibrium of stress generation and surface energy reduction) [21–23]. During later stages of film growth, relaxation of tensile stresses can be observed because of surface reconstruction, reduction or diffusion of the grain boundary structures, grain growth, and formation of disordered structures. The film prepared at 3.2 mtorr shows featureless morphology as shown in Fig. 3b.

With gas pressure increased to 10 mtorr, the residual stress is still tensile, but its magnitude decreases signif-

icantly as compared to those deposited with a gas pressure of 3.2 mtorr. Relatively large tensile stress exists at the initial stage of film growth, which can also be attributed to coalescence process. The stress gradually decreases due to the relaxation mechanisms. At a high Ar gas pressure, the energy of sputtered atoms decreases due to collision, resulting in decrease in surface diffusion and development of columnar structure. Argon atoms trapped in the films make the film partially porous and fragile [24,25]. SEM observation can verify these results as shown in Fig. 3c. The film surface shows coarse microstructure, while cross-section observation reveals columnar structure. Therefore, it can be concluded that the lower value of tensile stress at a higher Ar gas pressure is caused by the formation of columnar structure with a lower density due to intercolumnar porosity and entrapment of argon gas in film [24,25].

Large stress can cause creep, cracking and even spallation of films. After deposition, peeling of films was sometimes found, especially for films prepared at a gas pressure of 3.2 mtorr as shown in Fig. 4. Detailed observation reveals that the cracks propagate in Si substrate near to TiNi film, instead at the interface between TiNi film and Si substrate. The fracture morphology shows wavy feature, and cracks propagate through (1 1 1) crystalline plane, along [1 1 0] directions of Si (1 0 0) wafer. The above results indicate that the adhesion strength between TiNi/Si interface (or called interfacial toughness) is higher than the bulk fracture toughness of (1 0 0) silicon substrate [5].

In brief, stress evolution in the room-temperature deposited films is governed by the competition among: (1) compressive stress induced by atomic peening; (2) tensile stress caused by the coalescence process during film formation; (3) relaxation of stress caused by the microstructure evolution and defects formation during

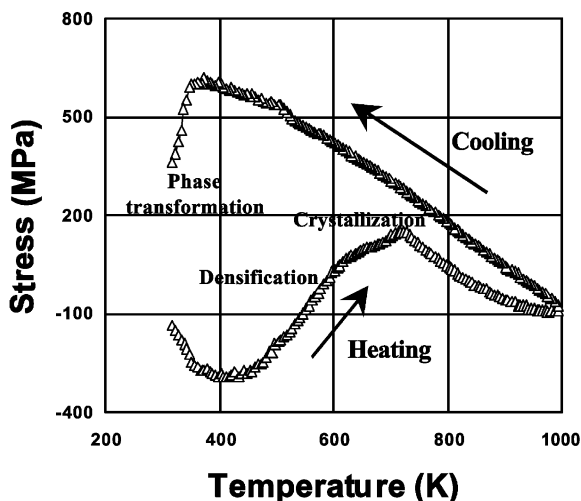


Fig. 5. Residual stress evolution curve during annealing for the amorphous film deposited at a gas pressure of 0.8 mtorr.

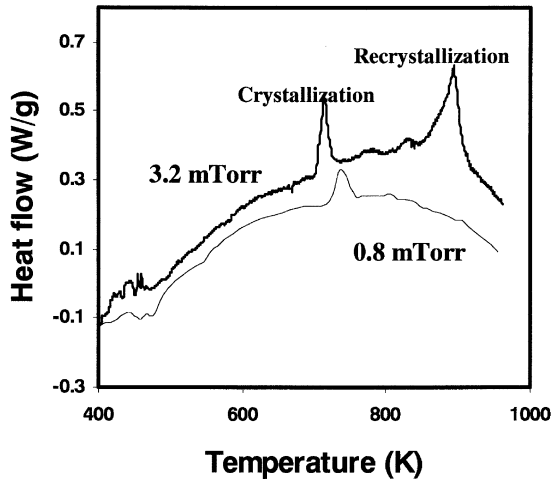


Fig. 6. DSC curves during annealing for the amorphous films deposited at different gas pressures.

film growth. The final stress is strongly influenced by gas pressure as discussed above.

### 3.2. Stress evolution during annealing process for amorphous Ti50Ni50 film

Stress evolution of an as-deposited amorphous film deposited with gas pressures of 0.8 mtorr during annealing is shown in Fig. 5. At room temperature, compressive stress exists in the film. With an increase in temperature, the compressive stress increases due to thermal stress, since thermal expansion of coefficient of TiNi films is much larger than that of Si substrate. However, above 400 K, tensile components increase due to densification of TiNi film [8,6]. With further heating to approximately 713 K, a peak can be found due to densification caused by the crystallization of TiNi film [8]. Fig. 6 shows the corresponding DSC curve, showing film densification and crystallization behavior during annealing process. When the crystallization event completes, development of compressive stress occurs due to generation of thermal stress. Cooling from high temperature results in the development of tensile stresses caused by the difference in thermal expansion coefficient between TiNi film and Si substrate. Cooling to below  $M_s$ , martensitic transformation occurs and tensile stress drops significantly as explained before.

For the Ti50Ni50 film deposited at a gas pressure of 3.2 mtorr, stress evolution curve vs. annealing temperature is shown in Fig. 7. At room temperature, large tensile stress exists in the film. With an increase in temperature, tensile stress decreases significantly due to thermal stress. However, the decrease rate in tensile stress decreases above 400 K, due to the gradual densification of the film. There is a sudden jump in tensile stress at 714 K corresponding to film crystalli-

zation temperature obtained from DSC tests shown in Fig. 6. When the crystallization event completes, the development of compressive stress occurs due to thermal stress. At a temperature approximately 890 K, there is an increase in tensile stress, which is probably due to the re-crystallization of the films, which needs further experimental verification. DSC result in Fig. 6 also shows a strong peak at approximately 893 K corresponding to this re-crystallization process. Cooling from high temperature results in the development of tensile stress. It is this rapidly increased tensile stress that may cause the spallation of thin films and even cracking of Si wafer during experiment. Cooling below  $M_s$ , martensitic transformation occurs and the tensile stress drops significantly.

In brief, during annealing process of as-deposited amorphous films, stress evolution is governed by the competition among: (1) residual stress in as-deposited films; (2) thermal stress (compressive stress during heating, while tensile during cooling); (3) tensile stress due to densification of structure; (4) tensile stress due to film crystallization; (5) relaxation of stress due to martensite transformation if available above room temperatures; (6) internal stress changes due to reduction of defects, or formation of precipitates, which is rather difficult to evaluate.

### 3.3. Effects of annealing temperatures on stress evolution of annealed Ti50Ni50 films at a gas pressure of 3.2 mtorr

Fig. 8 displays DSC results of Ti50Ni50 films annealed at different temperatures. The stress evolution curves vs. temperature for the annealed films are shown in Fig. 9. At annealing temperature of 673 K, there is no apparent peak in DSC curve. Stress evolution curve

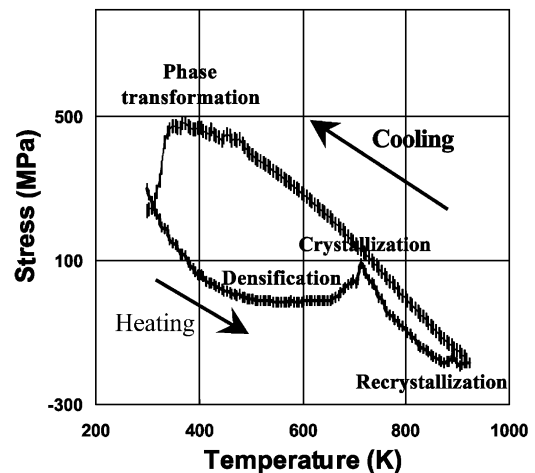


Fig. 7. Residual stress evolution curve during annealing for the amorphous film deposited at a gas pressure of 3.2 mtorr.

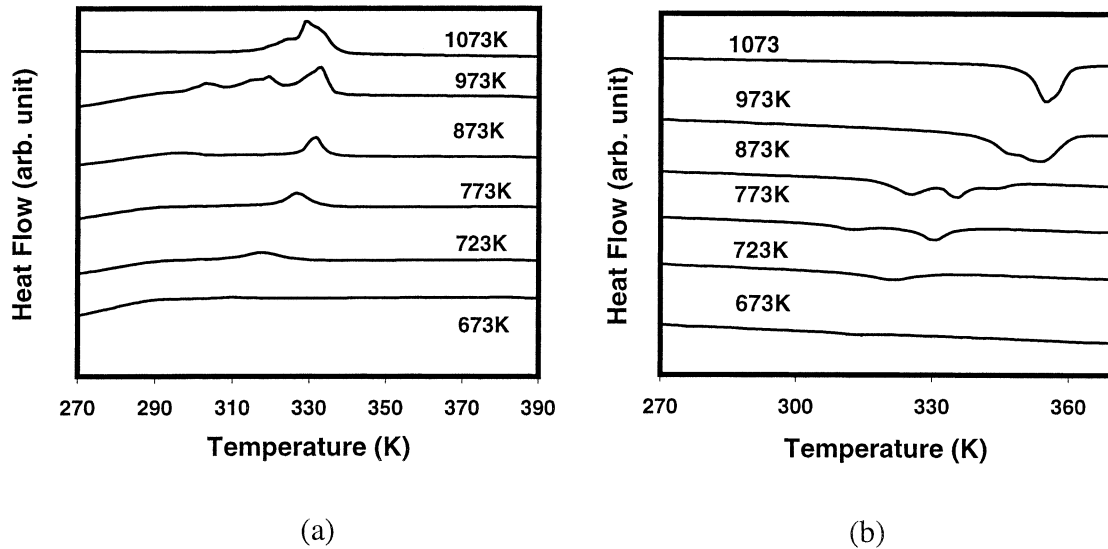


Fig. 8. Heating and cooling DSC curves of Ti50Ni thin film at various annealing temperatures for 1 h (a) cooling curve (b) heating curve.

only shows a linear line, indicating that only thermal stress changes during heating and cooling. After annealing at 723 K, DSC heating/cooling curves show weak peaks (Fig. 8). Stress evolution curve (Fig. 9) shows a partially closed loop, indicating phase transformation is far from finish at room temperature, thus large stress is not relaxed. Both DSC and curvature measurement results show that transformation temperatures are shifted to higher values with increase of annealing temperature. Recovery stress in the film increases significantly, and heat flow peaks become more defined, both indicating that phase transformation becomes significant. The higher is the annealing temperature, the higher fraction is the crystallized phases, thus more significant for martensite phase transformation [26,27]. At an annealing tem-

perature between 873 and 973 K, DSC results show that there are several peaks, indicating a multi-stage transformation. For the films annealed at 873 K, the stress evolution curve clearly shows a closed hysteresis loop (Fig. 9). DSC results are shown in Fig. 8, with only one peak observed, corresponding to M and A transformation.

3.4. Residual stress and stress evolution for post-annealed films with different Ti/Ni ratios at a gas pressure of 3.2 mtorr

Influences of annealing and composition on the transformation behavior of sputtered amorphous TiNi films have been extensively studied using DSC tests

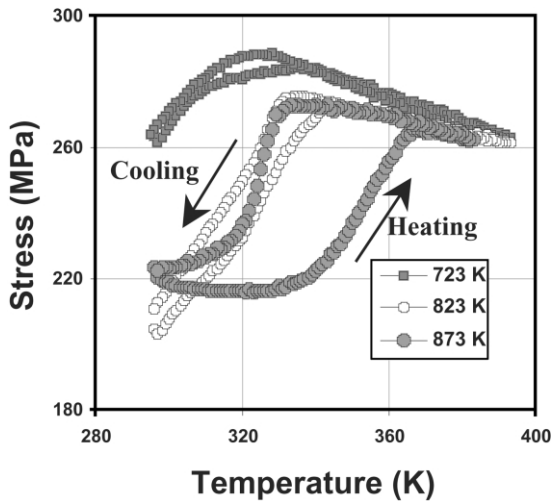


Fig. 9. Stress evolution curves vs. temperature for the room-temperature deposited films annealed at different temperatures.

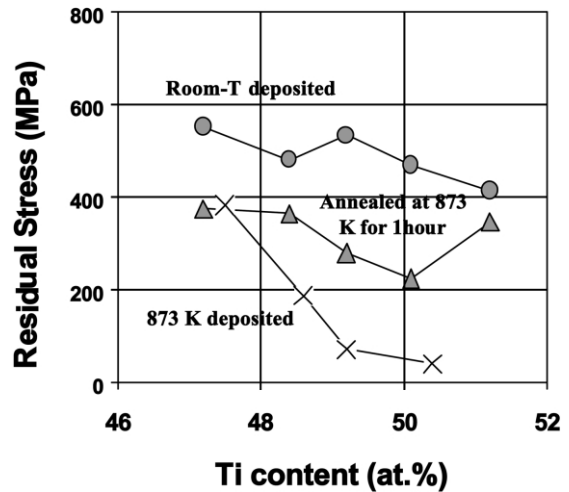


Fig. 10. Residual stress for TiNi films with different Ti/Ni ratios at an Ar gas pressure of 3.2 mtorr.

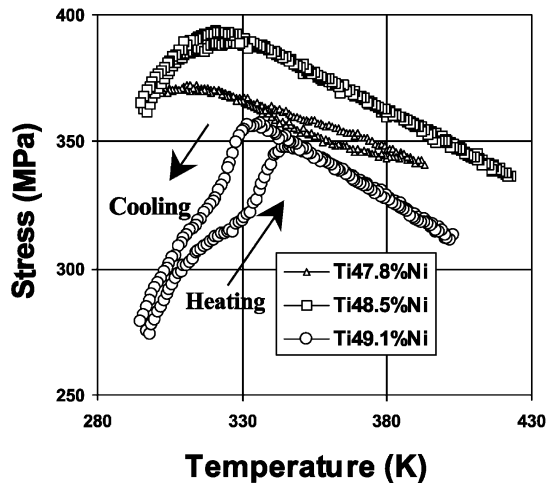


Fig. 11. Stress evolution curves for the post-annealed films with different Ti/Ni ratios.

[3,13,27,28]. In this paper, we will focus more on the effects of annealing and composition on residual stress and stress evolution during martensite transformation. Fig. 10 shows residual stress for TiNi films with different Ti/Ni ratios with and without annealing at 873 K at an Ar gas pressure of 3.2 mtorr. Residual stresses in as-deposited amorphous films are tensile, and the intrinsic stress in the as-deposited films are more or less the same among each other. After annealing these films, the stress decreases, but is quite sensitive to film composition. The stresses decrease with increase in Ti content up to 50.1%. The difference in residual stress for films with different Ti contents can be explained from the differences in phase transformation behavior, intrinsic stress in the films, and/or precipitates in the films [29].

Fig. 11 shows the stress evolution curves for the post-annealed (at 873 K) films with different Ti/Ni ratios. For the film 47.8% Ti, there is only a small hump in the stress evolution curve, indicating that the martensite transformation is not so significant. One possible reason for the high residual stress in film and weak phase transformation could be related to high Ni content in films and R-phase transformation. With the increase of Ti content to 48.5%, transformation temperatures increase, and recovery stress increases. The stress evolution curve shows a partially closed loop. Heating and cooling curves are almost overlap, but the curves are not close at room temperature. From the small hysteresis, it is easy to regard it as R-phase transformation. When cooling down to room temperature, the transformation does not finish, and large stress is only partially released. For the film with a Ti content of 49.1%, an apparent two-step transformation during both heating and cooling process can be observed as shown in Fig. 11, corresponding to martensite and R-phase transformations.

Two-stage transformation behavior in TiNi films has been studied by many researchers, and it can be attributed to the fine internal structure, small grain sizes, distribution of dislocations, non-uniform stress distributions, and/or precipitates, such as  $Ti_3Ni_4$ , or  $Ti_2Ni$ , etc. [30–32]. For the film with a Ti content of 50.2%, stress vs. temperature plot shows a closed hysteresis loop, i.e., perfect shape memory effect as shown in Fig. 9. At room temperature, the residual stress is quite low due to the significant relaxation of residual stress by martensite transformation, and also low intrinsic stress (without many precipitates). With the further increase in Ti content, martensitic transformation temperatures decrease, and only partial relaxation of stress is observed above room temperature. Therefore, the stress increases slightly. Precipitation in the films also contributes to the increase in stress.

### 3.5. Residual stress and stress evolution for films deposited at 723 K and a gas pressure of 3.2 mtorr

Fig. 10 shows the residual stress values for TiNi films deposited at 723 K with different Ti/Ni ratios at a gas pressure of 3.2 mtorr. The residual stress values decrease dramatically if compared with those deposited at room temperature and annealed at higher temperature, but still are sensitive to Ti content. Increasing substrate temperature causes the effect of rarefying Ar gas in the vicinity of both cathode and substrate, thus increases the mean free path for the energetic atoms striking on the surface of the growing films. Therefore, the compressive intrinsic stress increases significantly. Also the defects formed at a high temperature could be much less than those deposited at room temperature, and thus the intrinsic stress could be lower. For the as-deposited film (723 K deposition) with a Ti content of 47.2%, the residual stress is quite high. With the gradual increase in Ti content, the residual stress decreases significantly. The dramatic differences in residual stress for different Ti/Ni films are due to the differences in (1) phase transformation behaviors, (2) intrinsic stress, and (3) precipitation as explained in last section.

Fig. 12 shows the stress evolution curves for the films with different Ti/Ni ratios. For the film with a Ti content of 47.8%, there is only a partial closed loop, and large residual stress is just partially released by R-phase transformation. With the increase of Ti content to 48.5%, transformation temperature increases, and recovery stress also increases as shown in Fig. 12. However, there is still a partially closed loop when cooling down to room temperature. For the film with a Ti content of 49.1%, an apparent two-step transformation can be observed during both heating and cooling process, which is corresponding to martensite, R-phase and austenite transformation. The differentiated stress vs. temperature

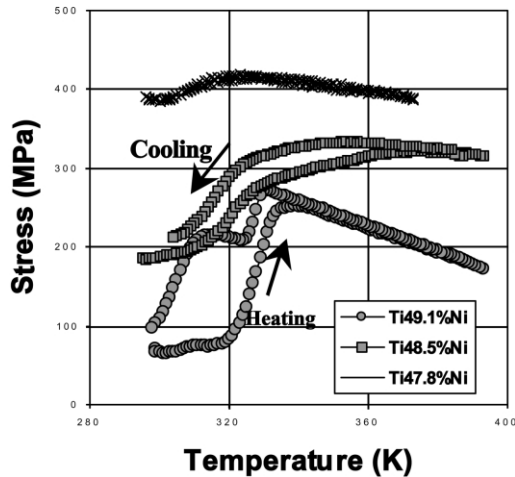


Fig. 12. Stress evolution curves for the films with different Ti/Ni ratios for the films deposited at a temperature of 723 K. (a) 47.8% Ti; (b) 48.5% Ti; (c) 49.1% Ti; and (d) differentiated stress vs. temperature for 49.1% Ti film.

curve reveals clearly this two-step transformation behavior. Based on XRD results, the peaks are associated with transformations among martensite, R-phase and austenite. For the film with a Ti content of 50.4%, stress vs. temperature plot shows a closed hysteresis loop shape, i.e., perfect shape memory effect as shown in Fig. 1. At room temperature, the residual stress is quite low due to the significant relaxation of residual stress by martensite transformation, and also low intrinsic stress (without many precipitates).

The recovery stress for films deposited at 723 K seems much larger than those of films with similar composition but deposited at room temperature and post-annealed at high temperatures. Since we took out the room-temperature deposited film from vacuum chamber to measure the residual stress before putting them back into chamber, and this process could deteriorate the shape memory effect of the film as already pointed out in Ref [4]. Also AFM observation on the surface morphology of the film deposited at 723 K reveals the crystals formed are much larger than those deposited at room temperature and annealed at higher temperatures. This can also be used to explain why the recovery stress is much higher for the films deposited at 723 K.

Successful application of TiNi thin films in MEMS requires elaboration of crystallized films using a heat treatment compatible with microelectronic process. It is better to use a lower temperature, in order to reduce thermal stress, minimize the reactions between the film and the Si substrate, and prevent the deterioration of materials properties as well as deformation of MEMS structure. Depositing SMA films under a medium high substrate temperature can avoid high-temperature post-

annealing, thus is more suitable for MEMS applications. However, the deposition temperature should be high enough to generate a certain content of crystallized phases with a good phase transformation behavior.

In brief, large range of stress values could exist in the deposited TiNi films, either compressive or tensile. The stress generation and relaxation behaviors were significantly affected by film composition, deposition and/or annealing temperatures, which strongly control the intrinsic stress, thermal stress and phase transformation behaviors. To minimize the residual stress, while to maintain a large recovery stress during martensitic transformation for actuation purpose, it is necessary to: (1) precisely control the Ti/Ni ratio; (2) deposit films at a possible lower pressure; (3) select a suitable deposition temperature or annealing temperature, with a compromise between thermal stress and intrinsic stress; (4) to use some interlayers (with possible compressive stress) to reduce large tensile stress in some TiNi films.

#### 4. Conclusions

A wide range of residual stress levels (either tensile or compressive) were found in the deposited films, depending significantly on Ti/Ni ratio, gas pressure, deposition temperature and/or annealing conditions. During annealing of as-deposited amorphous films, stress evolution is related to residual stress in as-deposited films; thermal stress; tensile stress due to densification crystallization; stress relaxation due to martensite transformation. Upon heating, crystallized TiNi films generate large tensile stresses when transforming from martensite to austenite, whereas during cooling, the stress relaxed significantly when the films transformed back to the ductile martensite phase. The stress evolution during martensite transformation depend significantly on some dominant factors such as Ti/Ni ratios, deposition and/or annealing temperatures or durations.

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