On the microstructure and interfacial properties of sputtered nickel thin film on Si (1 0 0)

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Abstract. Ni films of thickness ranging from 150 to 250 nm were deposited by DC magnetron sputtering on to Si (100) substrates maintained at room temperature and followed by post-annealing at 300 and 500 °C for 30 min. Other set of Ni films were deposited on to Si (1 0 0) substrates held at annealing temperature of 300 and 500 °C for 30 min. Microstructural investigation by field emission scanning electron microscope (FE-SEM) and atomic force microscope (AFM) revealed columnar morphology with voided boundaries for films deposited at room temperature and was retained after post-deposition annealing at higher temperatures. Nickel silicide formation with isosceles triangle diffusion front was confirmed by cross-sectional high-resolution transmission electron microscopy (X-HRTEM) for post-annealed Ni films. Thin film deposited at high substrate temperatures having near-equiaxed structure found to be the best route to fabricate thin films without silicide formation.

Keywords. Sputtering; thin film; diffusion; morphology.

1. Introduction

Thin film shape memory microactuators are gaining technological interest in micro-electro-mechanical systems (MEMs) due to the intrinsic property of one-way shape memory effect (Kohl 2004). One such material of interest is Ti–Ni alloy thin film, which is nowadays being incorporated as MEMS-based microactuators and transducers (Walker *et al* 1990; Johnson 1991; Fu *et al* 2004). For a Ti–Ni thin film to work as micro-device, control over working temperature and compositional tuning during deposition are the prime requisites. Due to inflexibility in composition adjustment, the fabrication of the Ti–Ni thin film by sputter deposition with Ti–Ni alloy target continues to be a challenge. Several works were carried out to combat the composition inflexibility either by co-sputtering Ti–Ni alloy target with Ti targets (Takabayashi *et al* 1997; Shih *et al* 2001; Fu *et al* 2003) or by incorporating the Ti plates which required a number of adjustments for size, shape, geometry, number and position of the plates (Wolf and Heuer 1995; Ishida *et al* 1999; Chu *et al* 2000; Surbled *et al* 2001). Further, works were developed by separately sputtering elemental targets of Ni and Ti and adjusting the individual powers (Krulévitch *et al* 1996; Ohta *et al* 2000; Sanjabi *et al* 2005). This method of sputtering was remarkably flexible and simple

to control the composition of the resulting Ti–Ni thin film.

Ni–Ti shape memory alloy films sputter deposited on Si substrates are predominantly amorphous when deposited at room temperature, and annealing at temperatures above 500 °C is necessary to crystallize the films. Crystallization of Ti–Ni films by *in situ* heating during deposition and post-deposition annealing of the films often lead to the formation of reaction products at the interface (Stemmer *et al* 1997; Chen and Wu 2000; Wu *et al* 2001; Qiu *et al* 2003). These metal silicides were reported to have an adverse effect on the shape memory behaviour which in turn influenced the performance of microactuators (Wu *et al* 2004). Ni is the primary diffusing species among Ti and Si. During annealing, silicide reaction is initiated between the Ni–Ti and Si interface (Wu *et al* 2001). Further, they observed reaction products such as NiSi₂ and TiNiSi being formed with increase in the annealing temperature. Recently, Martins *et al* (2008) reported the formation of interfacial diffusion layers of NiSi₂ and Ti₄Ni₄Si₇ that render high residual stress and low recovery stress in Ni–Ti shape memory alloy thin film.

Atomic diffusion and interface reaction can be controlled by appropriate choice of reacting components, reaction paths and products. There are reports on restricting the interfacial reactions in Ni–Ti/Si interfaces during crystallization process by depositing thin layer of SiN by PECVD on Si substrate (Wu *et al* 2004; Fu *et al* 2005).

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Out of Ni and Ti elemental targets, Ni was chosen in the present study since they are primary diffusing species at lower temperatures and their existence needs to be understood. Metal motion is dominant in the formation of silicides with the diffusion coefficient for Ni atoms being greater than the coefficients for Si atoms (D'Heurle *et al* 1984; Julies *et al* 1999). In Ni/Si system, the formation of nickel silicides is a sequential growth of three phases, namely Ni_2Si , NiSi and NiSi_2 . This is based upon the temperature of formation (Poate *et al* 1978). The driving force for the silicide formation is decrease in free energy and the external energy input accelerating the silicide formation by changing the thermodynamic energy of the system. Nickel disilicides (NiSi_2) are being largely employed as metal–oxide–semiconductor field-effect transistors (MOSFETs) which form at about 800 °C and grow epitaxial on (1 0 0), (1 1 1) and (1 1 0) silicon faces (Abhaya *et al* 2007). It is important to point out here that, though there are reports on formation of nickel silicides at temperatures as low as 200 °C, in the present study we prefer to investigate Ni–Si interactions at 300 °C. The reason being that as-deposited amorphous Ni–Ti thin film begins to crystallize only at temperatures greater than 300 °C.

The authors believe that the present study on the interfacial reactions Ni/Si is significant with respect to shape memory behaviour of near equiatomic Ni–Ti thin film which is highly sensitive to compositional change. In this context, Ni layers of different thicknesses, when introduced as interlayer, were found to restrict the diffusion of Cu into Si (Abdul-Letif 2007) and Au into Cu promoting good adhesion and excellent diffusion barrier (Chang 1986).

The work reported here is an effortless method to minimize the interfacial reactions in Ni–Ti/Si by introducing Ni layer as a diffusion barrier instead of incorporating SiN and TiN layers. This study is considered to be

the basis for further investigation on the interfacial reactions in Ni–Ti/Ni/Si thin film which can eventually lead to reproducible and reliable shape memory effect.

2. Materials and method

A schematic diagram of the RF and DC magnetron sputtering system with sputter-down assembly employed to deposit Ni films is shown in figure 1(a). The sputtering targets are 60° inclined to each other in confocal geometry located on the top of the chamber facing the substrate platform (figure 1b). Ni films of thickness varying from 150 to 250 nm were deposited from pure 3 inch Ni target (99.99%) onto 1'' × 1'' Si (1 0 0) substrate by DC magnetron sputtering method. Prior to the depositions, Si substrates were ultrasonically cleaned with acetone and alcohol to remove the surface contaminants. Thereafter, Si substrates were etched in hydrofluoric acid in order to remove the surface oxide. After being dried, they were loaded into the vacuum chamber immediately. The target–substrate distance was fixed at 150 mm for all the depositions. Substrates were rotated at a speed of 26 rpm in order to maintain the uniformity in the resulting films. Ar gas with a constant flow of 50 sccm was used as the sputtering gas. The chamber was pumped down to 2×10^{-6} Torr by turbo molecular pump and Ar gas was backfilled. Sputtering gas pressure of 20 mTorr was maintained during the deposition. The deposition conditions of Ni films are shown in table 1. The substrate temperature was maintained at room temperature, 300 and 500 °C. The room temperature deposited films further were annealed at 300 and 500 °C for 30 min in vacuum. Annealing was carried out after the deposition without breaking the vacuum (under base pressure of 2×10^{-6} Torr) to rule out the possibility of oxidation in Ni films at higher temperatures.

Figure 1. Schematic diagram of (a) RF–DC magnetron sputtering system and (b) location of target, substrate, and heater.

Table 1. Experimental conditions adopted for Ni to study the influence of *in situ* substrate temperature and post-annealing temperature.

Argon gas pressure (mTorr)	STD (mm)	DC power (W)	<i>In situ</i> substrate temperature (°C)	Post-annealing temperature (°C)	Annealing time (min)	Deposition time (min)	Deposition rate (± 3 nm/min)
20	150	300	27	–	–	10	17
20	150	300	27	300	30	10	17
20	150	300	27	500	30	10	17
20	150	300	300	–	–	30	13
20	150	300	500	–	–	30	13

Figure 2. Cross-sectional TEM sample preparation.

The film thickness was measured using step height on a masked silicon substrate by surface profiler (Veeco-Dektak 150). Grazing incidence X-ray diffraction (GIXRD–Philips X’Pert) was employed to determine crystalline phases generated. The grazing incidence angle is fixed to 0.5° for scan range (2θ) 30 – 100° . The surface studies were observed by atomic force microscopy (AFM) (Veeco-Nanoscope IV) under tapping mode. The microstructural studies were carried out on planar and cross-sectional films by field emission scanning electron microscope (Carl Zeiss-SUPRA40).

JEOL-JEM 2100 high-resolution transmission electron microscope (HR-TEM) was employed to characterize planar and cross-sectional Ni samples for microstructure. Cross-section TEM samples were prepared for the detailed structural analysis at the interface. The sample was cut into 6 pieces of size 5×4 mm² using Gatan model 150 ultra-sonic disc cutter. Film to film and Si to

Si sides were sandwiched together with an adhesive. The schematic of one such block (film to film and Si to Si) is represented in figure 2. The sample was then glued to the hollow stainless-steel tube in order to further reduce the diameter to 2.3 mm by using ultrasonic disc cutter. The cylindrical sample with dimension 5×2.3 mm² was fitted into brass tube and cut into pieces using Isomet cutter 1000. After cutting, the samples were carefully polished in rotopol grinder fitted with fine emery paper to reduce the thickness to < 100 μ m. Finally, the samples were thinned by ion milling for 2 h with ion beam energy of 4.8 keV.

3. Results and discussion

3.1 Grazing incidence X-ray diffraction analysis

Figure 3 shows the GIXRD pattern for Ni films deposited at room temperature and followed by post-annealing at temperatures 300 and 500 °C. At temperature greater than 400 °C, the interfacial reaction between Ni and Ti film and silicon substrate occurs leading to the diffusion of Ni species into the silicon to form Ni₂Si at the interface (Wu *et al* 2001). The GIXRD pattern of Ni film annealed at 500 °C for 30 min show the clear evidence of Ni₂Si phase formation along with the presence Ni cubic-phase. Thus it can be understood that the formation of metal–silicide is initiated at 500 °C without much consumption of Ni concentration at the film–substrate interface. The crystallite size was calculated from (1 1 1) peak by using well-known Scherer’s formula (1) and found to increase from 10 to 17 nm with the post-annealing temperature

$$G = \frac{0.9\lambda}{\text{FWHM} \cdot \cos \theta_B}. \quad (1)$$

GIXRD patterns for Ni films deposited at 300 and 500 °C substrate temperatures are shown in figure 4. Even at high substrate temperature, the texturing of grains was favourable in the (1 1 1) direction in Ni films. However, GIXRD pattern for Ni films fail to show any evidence of nickel silicide formation at high substrate temperature near the film/substrate interface. The grain size was found to increase from 18 to 33 nm for Ni films as the

Figure 3. GIXRD plot for Ni films at different post-annealing temperatures.

Figure 4. GIXRD plot for Ni films at different substrate temperatures.

deposition temperature was raised from 300 to 500 °C. Similar observation has been reported (Chawla *et al* 2008) in case of Ti films, which suggest that increase in surface and grain boundary diffusivity of adatoms causes the grain growth at high substrate temperature leading to the increase in the grain size and the roughness of the films. This discussion seems to be relevant in the present case too.

3.2 Atomic force microscopy analysis

To investigate the effects of substrate temperature on the surface topography and roughness of Ni films, AFM analysis was carried out on Ni films. Figure 5(a)–(c) shows the AFM images of the Ni films deposited on Si substrates held at 30, 300 and 500 °C, respectively. As it is evident from the AFM images, the grain size increases with the increase in substrate temperature during deposition. The root mean square (rms) roughness values were calculated for Ni films. Room temperature deposited Ni

Figure 5. AFM images of Ni films deposited at (a) room temperature (scan size: $2 \times 2 \mu\text{m}$) (b) 300 °C and (c) 500 °C (scan size: $4 \times 4 \mu\text{m}$).

film has an rms roughness value of 3.85 nm. Further, rms roughness values were found to increase from 10 to 23 nm as the substrate temperature was increased from 300 to 500 °C. The variation in surface topography with substrate temperature is the consequence of grain growth taking place due to the increase in rate of surface

Figure 6. AFM images of Ni films deposited at room temperature followed by post-annealing at (a) 300 °C and (b) 500 °C.

migration of adatoms. When films are sputter deposited on substrate held at higher temperature, surface migration of the adatoms with higher energy takes place. This is due to the fact that the adatoms gain additional energy apart from the kinetic energy while they reach the growing film surface. This would result in the promotion of larger grains due to higher surface migration of adatoms. The increase in film roughness is probably due to the increase in the lateral Ni grain size. It should also be noted that the normal grain growth is not expected in thin films, since the microstructural evolution in such thin films is governed by the average grain size comparable with the film thickness and where the grain boundaries are perpendicular to the film surface and across its whole thickness (Di Nunzio 2003). Instead, abnormal grain growth takes place often leading to bimodal grain size distribution caused by secondary grain growth, in which case, some grains may grow excessively large relative to surrounding ones.

Figure 6(a) and (b) represents the AFM images of Ni films post-annealed at 300 and 500 °C for 30 min after deposition. From the topographical details of Ni films it is evident that the grain growth had taken place upon annealing treatment. The roughness was found to increase from 5.42 to 7.14 nm as the annealing temperature was increased from 300 to 500 °C. There is also a clear evidence of the presence of wide channels in between the grains suggesting the grain coalescence at high temperature.

3.3 Field emission-scanning electron microscopy (FE-SEM) analysis

Figure 7(a) and (c) shows the planar FE-SEM images of Ni films post-annealed at 300 and 500 °C for 30 min. From the micrograph it is clear that there is negligible

improvement in the microstructure for Ni films post-annealed at 300 °C. However, at annealing temperature of 500 °C, the grain size was found to be $\sim 0.1 \mu\text{m}$. The cross-sectional FE-SEM micrographs of the films annealed at 300 and 500 °C shown in figure 7(b) and (d) prove the coalescence of individual islands leading to columnar microstructure separated by grain boundaries. In addition, the channels appear to be wider with reduction in their number density.

As pointed out earlier, during room temperature deposition of Ni films, the lack of atomic mobility on the substrate surface restricts nucleation at most favourable sites (valleys) on the substrate. This causes the film to grow vertically instead of laterally. An ideal growth leads to smooth-continuous film even when deposited at low substrate temperature provided deposition rate of the film is nearly equal to the film nucleation rate. However, in the present case, it is presumed that the rate at which adatoms deposit on the substrate (deposition rate) is lower than nucleation rate. Due to this, the deposited adatoms grow as individual islands resulting in less-denser film microstructure. This justifies the presence of wide channels in between the grains, even after coalescence of neighbouring grains, when the films are subjected to the post-annealed Ni films at high temperature.

The crack-like microstructure which was previously observed in as-deposited Ni films was not observed in Ni films deposited on Si substrate held at 300 and 500 °C as shown in figure 8(a) and (c). At substrate temperatures of 300 and 500 °C, the T/T_m ratio of Ni films was calculated to be 0.33 and 0.45, respectively. These ratios lie in zone I (transition zone) and zone II of Thornton's structure zone model (SZM) and the expected microstructure is large columnar structure with densely packed grain boundaries without channels in between the islands (Inoue *et al* 2009). Atomic mobility on the substrate

Figure 7. Planar and cross-sectional FE-SEM images of post-annealed Ni films: (a) and (b) 300 °C, and (c) and (d) 500 °C for 30 min.

Figure 8. Planar and cross-sectional FE-SEM images of Ni films deposited at substrate temperature of (a) and (b) 300 °C, and (c) and (d) 500 °C.

surface increases thereby adatom condensation occurs preferentially at the surface concavities leading to the smoother surface and denser-Ni film. Figure 8(b) and (d) shows the cross-sectional FE-SEM microstructure of Ni films deposited at substrate temperature 300 and 500 °C. A near-equiaxed grain structure with densely packed microstructure comprising large lateral grain size was observed. This type of film microstructure shows the combination of layer-by-layer growth accompanied with island formation obeying Stranski–Krastanov growth model.

3.4 Transmission electron microscopy analysis

In order to investigate any silicide formations, cross-sectional TEM studies were carried out on Ni film deposited at 500 °C substrate temperature. However, results did not reveal the formation of nickel silicides or any type of interfacial reaction between Ni and Si as shown in figure 9(a). The corresponding selected area electron diffraction pattern (SAED) contains low intensity rings which are indexed to Ni phase and the spots are indexed to Si substrate (figure 9b). At substrate temperature of 500 °C, the metal supply is enormous with insignificant metal consumption due to the relative lowering of deposition temperature. In present case, the deposition rate of *in situ* heated Ni films (13 ± 3 nm/min) is higher than that deposition rate reported in literature; this might have delayed the formation of silicides at the interface. Similarly when compared to the electron beam evaporated Ni films, this value is high enough to restrict the interfacial reactions as reported (Julies *et al* 1999). The basic principles behind the formation of metal-silicide on Si substrates are: (i) solid state diffusion yielding sequential silicides formation; and (ii) deposition at extremely low rates on heated substrates allowing instantaneous

reaction of film and substrate at the time of deposition itself.

The principle behind the latter process is to reduce the concentration of metal at the growth interface during compound phase formation by performing the deposition slowly while keeping the substrate at high temperature. Therefore, in the present case, deposition of Ni films on heated substrates at temperatures 300 and 500 °C has negligible effect on the nickel silicide phase formation. This shows that the formation of nickel silicide depends not only on the kinetic energy of the sputtered Ni atoms but also depend upon the morphology of the films based on diffusion temperature and time. However, formation of such products in small amount cannot be totally ruled out and further investigations are necessary to confirm the presence or absence of such silicides.

The formation of metal-silicides which were previously detected in GIXRD analysis (figure 3) for Ni film post-annealed at temperature of 500 °C was thoroughly examined by TEM analysis as shown in figure 10(a) and (b). On closer observation, the presence of a homogeneous thin region growing adjacent to Ni and Si interface (represented by thick arrow) was evident and considered as a part of interfacial diffusion layer. The diffusion front at the interface appears to be a series of isosceles triangles with vertex 70° and size of the triangles appears to be non-uniform as represented by dotted circle and triangle in figure 10(b). The formation of such diffusion region at the film–substrate interface is believed to set a pathway for the interdiffusion of Ni atoms through the silicide/Si interface resulting in faster growth of nickel silicides. Qiu (2003) observed similar formation of silicides in reactive-sputter deposition of Ni films on Si at 200 °C and accounted for the formation of isosceles triangle diffusion front as the diffusion of Ni atom along the (1 1 1) planes of Si substrate. Figure 10(c) represents the electron

Figure 9. Ni film deposited on Si substrate at 500 °C: (a) cross-sectional TEM micrograph and (b) corresponding SAED.

Figure 10. Post-annealed Ni films at 500 °C: (a) cross-sectional bright field transmission electron micrograph at 50 kV, (b) cross-sectional bright field transmission electron micrograph at 150 kV and (c) corresponding SAED.

diffraction pattern taken close to the film–substrate interface area. The weak and diffused rings are indexed to Ni phase and the low intensity spots are indexed to Si substrates. The presence of faded or very diffused rings in electron diffraction pattern signifies that Ni is un-reacted which constitutes the major part of the film. On the other hand, some of the spots in the diffraction pattern could not be indexed as the diffraction spots from Ni_2Si .

This indicates that the interface is not pure Ni_2Si , but is a mixture of Ni, Si and Ni_2Si . However, a few researchers have observed the formation of Ni_2Si at low temperature (200 °C) followed by subsequent high temperature annealing which leads to the conversion of Ni_2Si into NiSi and NiSi_2 phases (Kim and Cooley 1987; Vantomme *et al* 1999). In metal–silicon interaction, the formation of silicides is usually initiated at 200 °C at metal/Si interface. Metal being primary diffusing species diffuse into the Si substrate at low temperature forming M_2Si as

initial interfacial product. The diffusion front at the interface is formed by a series of isosceles triangles, which enables further diffusion of Ni atoms into the Si substrates. At temperature greater than 400 °C, the entire metal is converted to M_2Si and further, the reaction proceeds to form mono-silicides. At temperatures greater than 600 °C, di-silicides are formed by consuming all of the metal film. This particular case was observed for the electron beam evaporated films with metal film thickness of 10–50 nm (Julies *et al* 1999).

However, the scenario might be slightly different when considering higher film thickness. In addition, the fact that the Ni films of thickness of the order of 10–50 nm deposited using electron beam evaporation and, subsequently, subjected to rapid thermal annealing treatments, explains the delay in silicide formation at temperature around 500 °C in the present case where the sputtered deposited films are of ~180 nm film thickness. Both

GIXRD and cross-sectional TEM analysis in the present study revealed the formation of Ni₂Si in the form of triangular diffusion fronts at the film–substrate interface for Ni films deposited on unheated substrate followed by solid-phase reaction. Therefore, this process might be beneficial especially while producing Ni–Ti shape memory alloy films which have to undergo the process of crystallization at temperatures around 700 °C where the metal-interactions with Si are more dominant.

4. Conclusions

This study provides an understanding of the interactions between Ni and Si based on the growth morphology of Ni films at different substrate conditions. The work represented here is explicitly different from the previously reported work on nickel silicide formation in terms of thickness and growth morphology. Ni films deposited at room temperature revealed columnar morphology and tend to retain the morphology even at high post-annealing temperature. This columnar morphology of sputtered Ni films is believed to be the driving force for the silicide formation in the present case. On the contrary, Ni films deposited *in situ* at higher substrate temperature revealed smooth and continuous grain morphology which did not show any trace of silicide formation. Hence, it is concluded that columnar morphology is an additional factor apart from thickness, deposition rate and temperature which affect the interfacial reactions between Ni and Si.

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