



Effect of oxygen concentration on the thermal stability of magnetron sputtered amorphous Ta–Ni thin films

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ABSTRACT

The effect of oxygen concentration on the thermal stability of amorphous Ta–Ni thin film alloy is studied in this work. The films were deposited on Si substrates by co-sputtering of Ta and Ni targets. The oxygen concentration in the Ta–Ni films was controlled by applying radio frequency (RF) substrate bias ranging from 0 W to 100 W. Ta–Ni Films with oxygen concentration from 0.95 to 5.25 at.% were obtained, with lower oxygen concentration obtained at higher RF bias. At the as-deposited state, all the Ta–Ni films are amorphous. Increase of oxygen concentration leads to increased electrical resistivity. The as-deposited amorphous films possess different thermal stability after annealing in vacuum for 30 min at temperatures ranging from 700 °C to 800 °C. Formation of TaSi₂ starts at 750 °C in films formed with lower oxygen concentration (0.95 at.%), while Ta₂O₅ and Ta-based phases are observed in films formed with higher oxygen concentration (4.89 at.% and 5.25 at.%). Our work shows that change of oxygen concentration affects the electrical conductivity and thermal stability of the Ta–Ni films. The presence of varying amount of oxygen also changes the Ta–Ni crystallization behavior as well as the interface stability of the Ta–Ni/Si film on silicon substrate.

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1. Introduction

Amorphous metal alloy, or metallic glass, which consists of at least two metallic components, has been widely studied and applied in various engineering fields. In the past years, effort has been focused on the development and understanding of bulk metallic glasses (BMGs) [1–5]. Because of the simple crystal structure of the constituents, these alloys usually crystallize relatively easily when heated to an elevated temperature, and lose the amorphous structure. Thus thermal stability and glass forming ability (GFA) are among the key properties for amorphous alloys. Inoue [6] has suggested three empirical rules applicable to alloys that have high GFA: 1) at least three alloying elements should be present, 2) there should be a large mismatch in atomic sizes of constituent elements, and 3) large negative heat of mixing among major alloying elements. Besides the major alloy elements, it was noted that the minor alloy elements (impurities) are also important in determining the GFA of BMGs [4,7–10]. For example, it was found that Zr-based BMGs lose their excellent GFA when the oxygen impurity level exceeds 1000 ppm [10]. To the contrary, Zhang et al. [11] reported that the GFA of Zr–Al–Ni–Cu alloys could be improved by alloying with 2–4 at.% oxygen.

In recent years, with the fast development of thin-film technologies, amorphous thin film has shown great potential in areas such as

semiconductor manufacturing and miniaturized device fabrication (e.g. micro-electrics-mechanical-system) [12–16]. Different from the process of bulk amorphous alloy in which melt quenching or fast cooling are usually applied, the amorphous thin film is usually formed by plasma-related processes such as physical vapor deposition (PVD), chemical vapor deposition, and atomic layer deposition. Small amount of other elements such as oxygen from the sputtering chamber is inevitably incorporated in the film during the deposition process. We have reported in a previous study that in the deposited amorphous Ti–Si–N film and Ta–Si–N films, 2–5 at.% of oxygen was introduced, and its presence stabilizes the amorphous phase at elevated temperatures [17–21]. However, the effect of oxygen on the amorphous phase formation and thermal stability of binary metallic thin film has not been well understood.

In this work, Ta–Ni thin films are chosen because of their potential usefulness in Cu diffusion barrier application in the back-end-of-line of advanced semiconductor technology with Cu metallization. In spite of the excellent electrical conductivity and better electromigration resistance, Cu diffuses fast into adjacent dielectric layer or Si leading to damage to the devices. A copper diffusion barrier is needed to prevent Cu diffusion into dielectric layers. For this purpose, a stable amorphous structure is preferred, since an amorphous film has no short diffusion path such as grain boundaries and dislocations which are present in a crystalline barrier. The barrier also requires high electrical conductivity in order to reduce the resistance–capacitance delay. Ta-based binary metallic glass, with stable amorphous structure and low electric resistance, is thus a potential candidate for Cu diffusion barrier to replace

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the conventional crystalline metal nitride systems. Our preliminary research has shown that magnetron sputtered amorphous Ta–Ni films fulfil the requirements such as low electrical resistivity and good thermal stability. Molecular dynamic simulation predicted that Ta–Ni had a wide metallic-glass-forming range from 25 to 75 at.% Ni [22]. Fang et al. demonstrated that the Ta–Ni film acts as a good copper diffusion barrier up to 700 °C [23]. Thus it is both scientifically and technically important to study the effect of oxygen, which will be inevitably introduced during film deposition, on the thermal stability and other related performance of the amorphous Ta–Ni films.

In this work, a series of Ta–Ni films was deposited on Si substrate by magnetron sputtering of Ta and Ni targets. The oxygen concentration in the films was controlled by adjusting the substrate bias during the deposition. The resistivity and microstructure were reported for the as-deposited films. Selected Ta–Ni films with different oxygen concentration were further annealed in vacuum to study their thermal stability and the evolution in microstructure.

2. Experiment

Ta–Ni thin films were deposited at room temperature on Si (100) wafers by magnetron sputtering using Ta and Ni targets with purity of 99.999%. The substrates were diced from Si wafer into pieces of 1 cm by 1 cm, and soaked in SC1 (H₂O₂ (30%): NH₄OH: 5 H₂O) solution for 10 min to remove any possible organic contamination. The substrates were then cleaned with deionized (DI) water before being soaked into SC2 solution (H₂O₂: HCl: 6 DI water) for 10 min to remove inorganic contamination. They were finally cleaned by DI water again and dried with N₂ gas gun. The base pressure of the sputtering chamber was maintained at 5.2×10^{-4} Pa and the working pressure of Ar gas was 2.67 Pa throughout the deposition. The sputtering powers were radio frequency (RF) 200 W for the Ta target and direct current (DC) 80 W for the Ni target. DC power was applied to the Ni target in order to avoid the electromagnetic interference between the RF power and the ferromagnetic target. Previous study reported that applying a DC substrate bias could change oxygen concentration of reactively sputtered ZrN_x films [24]. In this work, we used a RF bias on the substrate to adjust the oxygen concentration. RF bias of 0 W, 25 W, 50 W, 75 W and 100 W was applied on the Si substrate during deposition. Substrate holder was rotated throughout the deposition process to ensure film uniformity and the film thickness was controlled at 800–900 nm. The sputtering rates of Ta and Ni under different substrate bias are shown in Table 1. Additional sets of samples were prepared with a copper top-layer deposited on the Ta–Ni films without breaking the vacuum. The purpose was to prevent oxygen in-diffusion from atmosphere after the deposition, which could provide a reference for the source of oxygen in the film.

To study the thermal stability and microstructure evolution of the Ta–Ni films, the samples were annealed in vacuum at a base pressure of 1.3×10^{-3} Pa at different temperatures at 700 °C, 750 °C, and 800 °C for 30 min. Electrical conductivity was measured using a 4-point probe system. Five to eight measurements were made for each data point. The composition of the films was measured using the JEOL energy dispersive X-ray spectrometer (EDX) attached to scanning electron microscopy JEOL JSM-6360A at an accelerating voltage of 15 kV. The depth profiles of film composition were characterized

using an IONTOF® time-of-flight secondary ion mass spectroscopy (ToF-SIMS) with impact energy of 1 keV Ar[±] ion primary beam. The film crystallinity and phase composition were determined by a Rigaku® X-ray diffraction diffractometer (XRD) with Cu Kα source. The scanning speed is 1° per minute, with glancing angle at 1°. The interfacial stability and microstructure evolution of the film stacks were examined using a JEOL 2100F high resolution transmission microscope (HRTEM) with accelerating voltage of 200 kV.

3. Result and discussion

3.1. Film composition

The variation of the composition with substrate bias is plotted in Fig. 1. Oxygen was observed in all films, with a reduction of oxygen from ~5 at.% to ~1 at.% as substrate bias increases from 0 W to 50 W. With further increase in RF power, there was no significant change in oxygen concentration. Table 1 summarizes the detailed composition of all deposited films.

We need to address two questions first: where does the oxygen come from and what is the mechanism of oxygen concentration reduction with increased substrate bias? The observation of oxygen in sputtered thin films and its effect on film properties were reported in previous reports [17,18,24]. The incorporation of oxygen could be attributed to two possible sources, 1) the incorporation of oxygen and moisture residue inside the chamber during the deposition process; 2) the post-deposition oxidation, where oxygen diffuses from atmosphere and forms oxide after the deposition [25]. Usually it is difficult to differentiate between the two sources. However in this work the task becomes possible as we used a Cu protective layer on the Ta–Ni films to eliminate the incorporation of oxygen from the atmosphere after deposition. Two groups of Cu (200 nm)/Ta–Ni (100 nm)/Si were deposited during which substrate RF bias of 0 W and 100 W were applied during Ta–Ni deposition. The depth profiles of elements were measured by SIMS and shown in Fig. 2. Without application of the bias (0 W), the intensity of oxygen in the Ta–Ni was relatively high, while oxygen intensity was negligible in the Ta–Ni layer under 100 W bias. This shows that substrate bias of 100 W has a clear influence on the amount of oxygen in the Ta–Ni films, and this observation agrees with the findings by EDX. Considering that no oxygen in-diffusion from atmosphere is allowed because of the presence of a Cu protective layer, the oxygen in Ta–Ni films could only be incorporated from the oxygen and moisture residues in sputtering chamber. We believe that the depletion of oxygen from the deposited film is due to the re-sputtering by the applied substrate bias. During the Ta–Ni film deposition, the oxygen and H₂O residues in the chamber are activated with high energy plasma. The activated oxygen atoms are adsorbed on substrate surface and incorporated in the Ta–Ni films. By applying RF power on the substrate, light-weight species like oxygen on the surface will be re-sputtered off by the low energy bombardment, leading to depletion of oxygen in the deposited films. Thus with increasing substrate bias, lower oxygen concentration was obtained in the deposited Ta–Ni films. It is worth mentioning that Ti and Ta possess very high affinity with oxygen, so large amount of oxygen could be incorporated into the Ta or Ti-containing films even when the level of oxygen residues is low inside the sputtering chamber [17,18,26]. For example, 10 at.% oxygen was

Table 1

Films composition, electrical resistivity and sputtering rate of the as-deposited Ta–Ni films under different substrate RF bias power.

Sample	Ta _{66.33} Ni _{28.42} O _{5.25}	Ta _{68.28} Ni _{26.83} O _{4.89}	Ta _{72.58} Ni _{26.47} O _{0.95}	Ta _{71.97} Ni _{26.42} O _{1.61}	Ta _{72.02} Ni _{26.89} O _{1.09}
Substrate bias power (W)	0	25	50	75	100
Composition (at.%)	Ta 66.33; Ni 28.42; O 5.25	Ta 68.28; Ni 26.83; O 4.89	Ta 72.58; Ni 26.47; O 0.95	Ta 71.97; Ni 26.42; O 1.61	Ta 72.02; Ni 26.89; O 1.09
Electrical resistivity (μΩ·cm)	410.4 ± 26.4	217.3 ± 4.8	238.8 ± 4.2	211.5 ± 1.8	217.6 ± 2.5
Sputtering rate (nm/min)	Ta: 10.76 Ni: 4.61	Ta: 10.42 Ni: 4.10	Ta: 10.69 Ni: 3.90	Ta: 10.19 Ni: 3.74	Ta: 9.71 Ni: 3.62

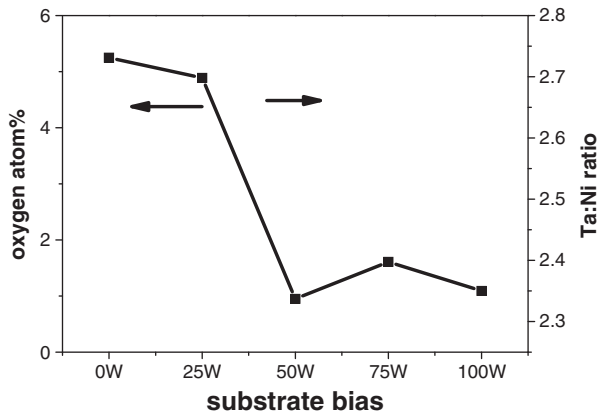


Fig. 1. The variation of oxygen concentration and Ta:Ni ratio with bias power.

found in the Ti underlayer using DC magnetron sputtering [26]. Up to 46 at.% oxygen was present in the Ta–Si–N films [18]. In both cases, no substrate bias was applied.

With the change of oxygen concentration, variation in the Ta and Ni concentrations was also noticed. In order to better understand Ta and Ni composition change, Ta:Ni atomic ratio is also shown in Fig. 1. An increase of Ta to Ni ratio from 2.33 to 2.74 was observed as RF power increases from 0 W to 50 W. The Ta:Ni ratio stabilized when RF bias was 50 W and higher. This change of Ta and Ni composition is also attributed to the sputter etching with the RF substrate bias. With substrate bias applied, Ta and Ni atoms adsorbed on the substrate surface will be partially sputtered off, but at different removal rates. At

a fixed RF bias power, Ni will be sputtered off faster due to its smaller atomic weight. Thus with higher substrate power, a higher Ta:Ni ratio was obtained. However, this effect was found to stabilize at 50 W, i.e., there is no further change in the Ta:Ni ratio. This is also verified by the change of Ta and Ni sputtering rate (Table 1). When the RF bias power was lower than 50 W, Ni sputtering rate decreased with higher RF power, while Ta sputtering rate was not affected. This has resulted in the increase of Ta:Ni ratio. When the RF power was higher than 50 W, the sputtering rate of both Ta and Ni decreased slightly, and the Ta:Ni ratio remained at the same level.

3.2. Electrical resistivity of the as-deposited Ta–Ni films

The variation of electrical resistivity with bias power is shown in Fig. 3, and the measurement details are listed in Table 1. A reduction of electrical resistivity was observed as the substrate bias increases. An obvious factor for the resistivity change is the oxygen concentration as it is widely reported that incorporation of oxygen increases the film resistivity [19]. With relatively high oxygen concentration, the number of free electrons is reduced, which leads to increase of film resistivity. However in the current work, it was also noticed that in the film under 25 W bias, the resistivity was as low as the other samples formed under 50–100 W bias while the sample contains relatively high amount of oxygen (Table 1). We believe this could be due to the densification of the films by the enhanced ion bombardment under the RF bias, but this would require further verification in the future work. Overall, the observed trend of low film resistivity under bias condition could be contributed by both the lower oxygen concentration and densification of the sputtered films. Catania et al. [27] found that the α -Ta phase transformed to β -Ta phase under applied bias, causing an increase in the pure Ta film resistivity. Our observed trend is opposite to their findings, and this is probably due to the dominantly amorphous nature of the films in the current work. Indeed the resistivity of crystalline polymorphs is strongly dependent on the type of crystal structure, but the one for the amorphous phases may be more influenced by the film defect structures (e.g. free volume) and impurity level (e.g. oxygen) since the film contains, at its best, only short-range ordered atomic clusters in the amorphous state.

The XRD patterns of the deposited Ta–Ni films are shown in Fig. 4. In the current work, there was only one broad peak around 39.2° , suggesting that all the as-deposited films are amorphous. It should be noted that the bulk binary metallic glass usually does not have high GFA. In the case of PVD deposited thin films, because of the lack of kinetic energy when atoms are adsorbed on the substrate surface, the atomic movement is largely suppressed, resulting in delay or complete hindrance of crystalline phase formation. In addition, Ta and Ni have a large atomic size difference (29.0%). As suggested by Inoue [6], a large atomic size difference has good glassy formation tendency. As a result,

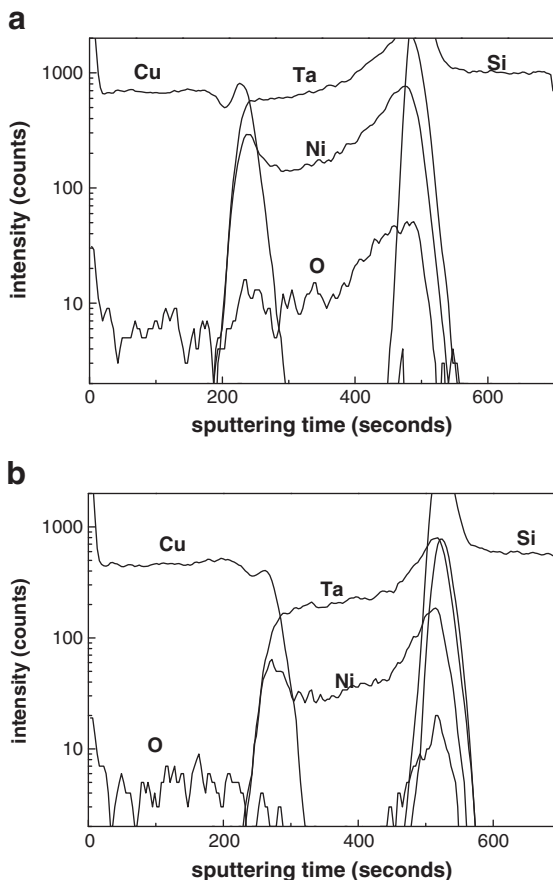


Fig. 2. ToF-SIMS depth profile of elements Cu, Ta, Ni, O, Si of the as-deposited Cu/Ta–Ni/Si stacks with substrate bias of (a) 0 W; (b) 100 W.

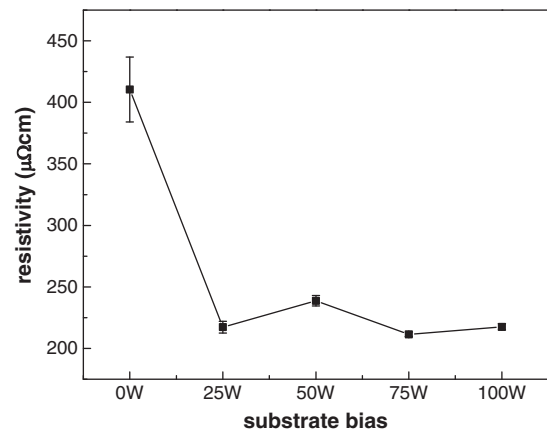


Fig. 3. Resistivity of as-deposited Ta–Ni thin films.

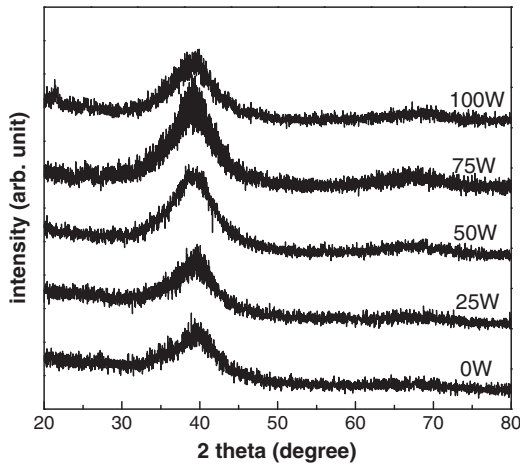


Fig. 4. XRD pattern of as-deposited Ta–Ni thin films.

the formation of amorphous Ta–Ni thin films is kinetically favorable. This is in contrast with previous reports where crystalline phases are formed in the as-deposited pure Ta film [27, 28]. As will be discussed below, the incorporation of small amount of oxygen is also a contributing factor toward the excellent film GFA in the Ta–Ni systems.

3.3. Microstructure evolution of Ta–Ni films

To study the thermal stability of the as-deposited amorphous alloy, the Ta–Ni films were annealed at temperatures of 700 °C, 750 °C and 800 °C for 30 min. The microstructure of the annealed the Ta–Ni films with oxygen concentration of 0.95%, 4.89% and 5.25% (i.e. $\text{Ta}_{72.58}\text{Ni}_{26.47}\text{O}_{0.95}$, $\text{Ta}_{68.28}\text{Ni}_{26.83}\text{O}_{4.89}$ and $\text{Ta}_{66.33}\text{Ni}_{28.42}\text{O}_{5.25}$) were studied to evaluate the effect of oxygen on the film properties and their thermal stability. We found that $\text{Ta}_{72.58}\text{Ni}_{26.47}\text{O}_{0.95}$ is representative of the other low oxygen concentration samples, thus only one condition was investigated in this examination.

Fig. 5 shows the XRD patterns of the Ta–Ni films after annealing. As shown in Fig. 5(a), the amorphous structure of all Ta–Ni films remained after annealing at 700 °C. No reaction between Ta–Ni and Si was observed by XRD. According to the results by Min and co-workers using Ta as a barrier, the reaction between Ta and Si occurs at 650 °C with the formation of TaSi_2 [26]. Our current work suggests that the addition of Ni into Ta has improved the thermal stability of the interface between the barrier film and Si substrate as no crystallization, nor interface reaction was observed at 700 °C. This will be further verified later with TEM observation at the cross section microstructures.

As shown in Fig. 5(b), crystalline peaks were observed after 750 °C annealing. While the XRD peaks observed from 33.5° to 42.5° are attributed to multiple peaks belonging to Ta β -phase, other peaks belonging to different crystalline phases appeared for films with different oxygen concentration. It is likely that Ta α -phase also exists after annealing because it has a strong peak at 38.5°, close to the multiple peaks of Ta β -phase. In addition, it was reported that the β -phase transforms into α -phase after annealing treatment [28]. In the low-oxygen $\text{Ta}_{72.58}\text{Ni}_{26.47}\text{O}_{0.95}$ sample, TaSi_2 peaks appeared at 21.5° and 25.4°, which indicates the loss of interfacial integrity between the Ta–Ni and Si substrate. In oxygen-rich $\text{Ta}_{68.28}\text{Ni}_{26.83}\text{O}_{4.89}$ sample, Ta_2O_5 peaks were observed at 23.1° and 28.3°. In $\text{Ta}_{66.33}\text{Ni}_{28.42}\text{O}_{5.25}$ sample, there was no other crystalline phase except the common Ta-based phases. In the two oxygen-rich films ($\text{Ta}_{68.28}\text{Ni}_{26.83}\text{O}_{4.89}$ and $\text{Ta}_{66.33}\text{Ni}_{28.42}\text{O}_{5.25}$), there was no reaction between the Ta–Ni films and Si substrate. Thus the observation confirms the positive effect of oxygen in helping to retard the reaction between Ta–Ni films and Si substrate. Ta_2O_5 peaks were observed in the $\text{Ta}_{66.33}\text{Ni}_{28.42}\text{O}_{5.25}$ sample after annealing at 800 °C (Fig. 5(c)). On

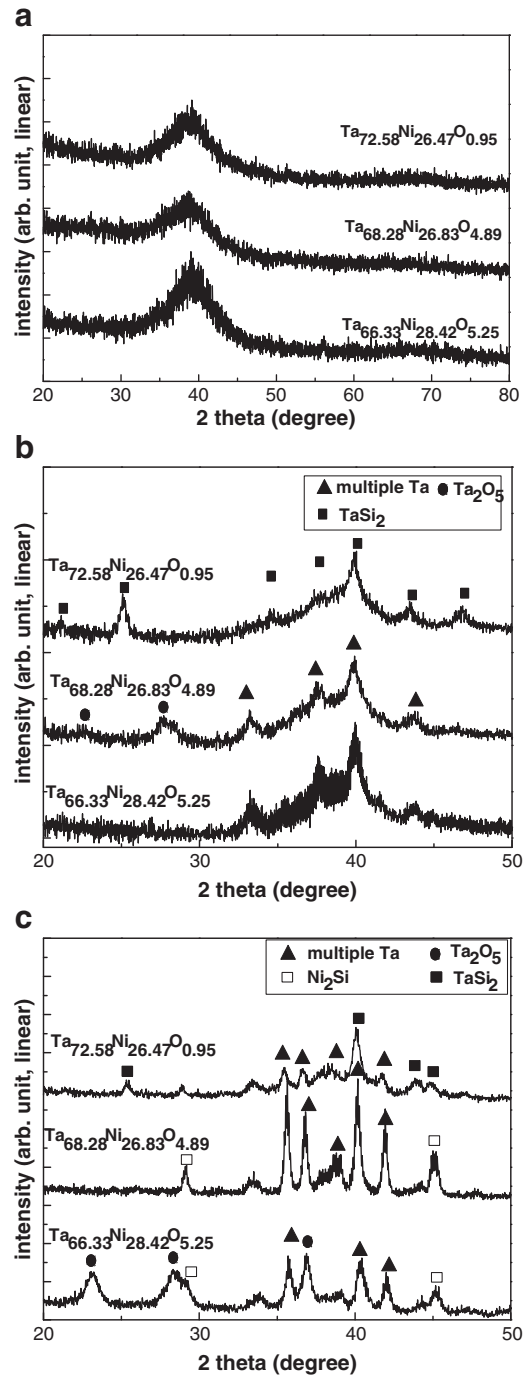


Fig. 5. XRD patterns of the Ta–Ni/Si films after annealing at (a) 700 °C; (b) 750 °C; (c) 800 °C.

the other hand, Ta_2O_5 peaks were not present in the 800 °C annealed $\text{Ta}_{68.28}\text{Ni}_{26.83}\text{O}_{4.89}$ sample. This implies that TaSi_2 is more stable at temperature at 800 °C and above. The Ta_2O_5 that was formed at 750 °C might have been decomposed in favor of TaSi_2 formation. In addition, NiSi_2 was also observed in $\text{Ta}_{66.33}\text{Ni}_{28.42}\text{O}_{5.25}$ and $\text{Ta}_{68.28}\text{Ni}_{26.83}\text{O}_{4.89}$ after 800 °C annealing (Fig. 5(c)).

TEM studies were then used to reveal the details of the interfacial reaction between the Ta–Ni films with Si substrate. Fig. 6 shows the interface evolution of $\text{Ta}_{66.33}\text{Ni}_{28.42}\text{O}_{5.25}/\text{Si}$ and $\text{Ta}_{72.58}\text{Ni}_{26.47}\text{O}_{0.95}/\text{Si}$ at 700 °C, 750 °C and 800 °C. At 700 °C, a layer of amorphous silicon oxide around 3 nm thick was observed in both $\text{Ta}_{66.33}\text{Ni}_{28.42}\text{O}_{5.25}/\text{Si}$ and $\text{Ta}_{72.58}\text{Ni}_{26.47}\text{O}_{0.95}/\text{Si}$ samples. This layer is the native oxide on silicon substrate which was not removed during substrate cleaning.

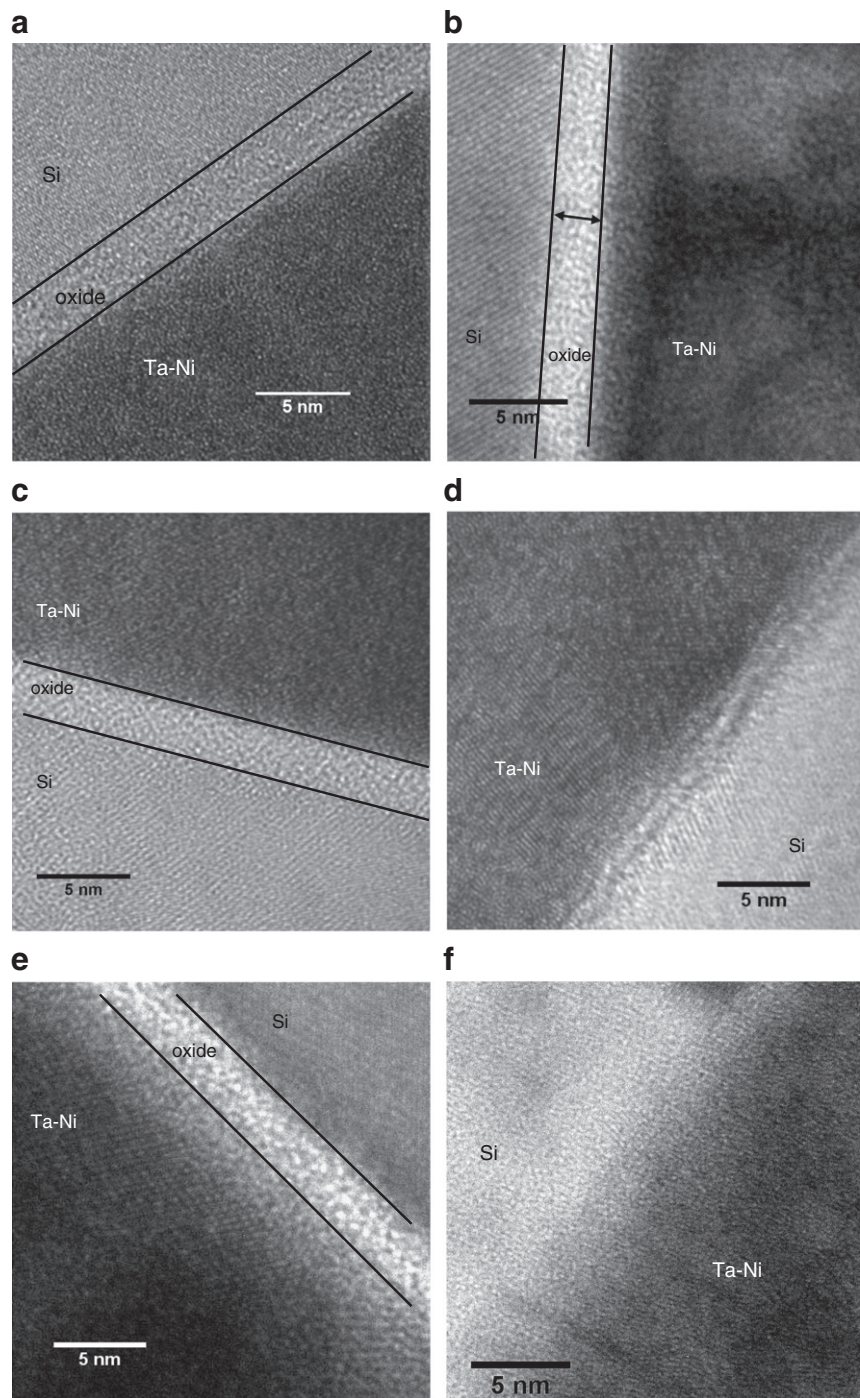


Fig. 6. TEM micrograph of Ta–Ni/Si interface of (a) $\text{Ta}_{66.33}\text{Ni}_{28.42}\text{O}_{5.25}$ annealed at 700 °C; (b) $\text{Ta}_{72.58}\text{Ni}_{26.47}\text{O}_{0.95}$ annealed at 700 °C; (c) $\text{Ta}_{66.33}\text{Ni}_{28.42}\text{O}_{5.25}$ annealed at 750 °C; (d) $\text{Ta}_{72.58}\text{Ni}_{26.47}\text{O}_{0.95}$ annealed at 750 °C; (e) $\text{Ta}_{66.33}\text{Ni}_{28.42}\text{O}_{5.25}$ annealed at 800 °C; (f) $\text{Ta}_{72.58}\text{Ni}_{26.47}\text{O}_{0.95}$ annealed at 800 °C.

No crystalline phases were observed in the Ta–Ni layers in all samples. At 750 °C, the silicon oxide layer in the $\text{Ta}_{66.33}\text{Ni}_{28.42}\text{O}_{5.25}$ /Si remained at the interface, while it became indistinguishable for the $\text{Ta}_{72.58}\text{Ni}_{26.47}\text{O}_{0.95}$ /Si sample. Multiple crystalline phases were observed in the $\text{Ta}_{72.58}\text{Ni}_{26.47}\text{O}_{0.95}$ sample, but only a small amount of nano-crystalline phase was present in the $\text{Ta}_{66.33}\text{Ni}_{28.42}\text{O}_{5.25}$ sample. It suggests that the higher oxygen concentration could enhance the interface stability and thus delays the reaction between the Ta–Ni and Si. At 800 °C, the oxide layer in $\text{Ta}_{66.33}\text{Ni}_{28.42}\text{O}_{5.25}$ /Si remained clearly visible. It was also observed that multiple crystalline phases, identified as Ta_2O_5 , Ta and NiSi_2 , appeared in the Ta–Ni region. The reaction is due to the diffusion of Si through oxide layer into Ta–Ni region. On the

other hand, for $\text{Ta}_{72.58}\text{Ni}_{26.47}\text{O}_{0.95}$, the interface between Ta–Ni and Si was undistinguishable as it has lost its integrity. Diffusion through the interface was more severe than the one in the $\text{Ta}_{66.33}\text{Ni}_{28.42}\text{O}_{5.25}$ sample.

4. Conclusion

Effect of oxygen concentration on the electrical conductivity, thermal stability and microstructure evolution of sputtered amorphous Ta–Ni thin films has been studied. By applying a substrate bias, the oxygen concentration in the sputtered Ta–Ni films varied from 0.95 to 5.25 at.% in our current work. The increase of oxygen concentration leads to the decrease of electrical conductivity. All the films have

shown remarkable glass formation ability: they remained amorphous after annealing at 700 °C for 30 min. Different crystallization behavior was observed when films were annealed at 750 °C for 30 min. In films with low oxygen concentration (0.95%), TaSi₂ formation was observed, while in films with higher oxygen concentration (4.89% and 5.25%), the interface reaction with silicon substrate was not observed till 800 °C. It is shown that oxygen in Ta–Ni/Si systems improves interface stability between Ta–Ni and Si.

Acknowledgments

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