Phase evolution of ultra-thin Ni silicide films on CF$_4$ plasma immersion ion implanted Si

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Abstract

We present a systematic study on the effects of CF$_4$ plasma immersion ion implantation (PIII) in Si on the phase evolution of ultra-thin Ni silicides. For 3 nm Ni, NiSi$_2$ was formed on Si substrates with and without CF$_4$ PIII at temperature as low as 400 °C. For 6 nm Ni, NiSi was formed on pure Si, while epitaxial NiSi$_2$ was obtained on CF$_4$ PIII Si. The incorporation of C and F atoms in the thin epitaxial NiSi$_2$ significantly reduces the layer resistivity. Increasing the Ni thickness to 8 nm results in the formation of NiSi, where the thermal stability of NiSi, the NiSi/Si interface and Schottky contacts are significantly improved with CF$_4$ PIII. We suggest that the interface energy is lowered by the F and C dopants present in the layer and at the interface, leading to phase evolution of the thin Ni silicide.

Keywords: silicide, phase, plasma immersion ion implantation, interface energy, Gibbs free energy

(Some figures may appear in colour only in the online journal)

1. Introduction

NiSi is used as Ohmic or Schottky contacts at the source/drain and gate in metal-oxide-semiconductor field effect transistors (MOSFETs) because of its low resistivity, low thermal budget and silicon consumption [1–3]. Ultra-thin silicide layers with high uniformity are required for scaled nanometre devices. However, the instability of NiSi due to low transition temperature to highly resistive NiSi$_2$ phase and grain agglomeration, especially for very thin layers at high temperatures, results in rough interface and surface, causing non-uniform contacts and substantial leakage at shallow junctions [4–6]. The thermal stability of the NiSi layer has been improved by incorporating elements such as Pt [7, 8], C [9, 10] and F [11] in Ni or silicon substrate or adding a new thin layer [12, 13]. With these methods the thermal stability of NiSi was increased at least 100 °C by inhibiting the agglomeration of NiSi grains and delaying NiSi$_2$ nucleation.

The solid-state reaction of Ni with a Si substrate leads to the sequence phase formation of δ-Ni$_2$Si, NiSi and NiSi$_2$. High resistivity phase NiSi$_2$ forms at an annealing temperature normal higher than 700 °C [14–17]. The interface energy plays an important role in phase evolution, especially when the film thickness is very thin [18, 19]. The phase formation sequence can be changed with a decrease of film thickness. Recent reports showed that when the thickness of the deposited Ni film is less than 4 nm, epitaxial NiSi$_2$ layers with high single crystalline quality and high thermal stability are formed even at low temperatures (300 °C) [20–23]. During the silicidation process, no NiSi phase is observed, but epitaxial θ-Ni$_5$Si$_2$ mesophase is found before the formation of epitaxial NiSi$_2$ [22]. The epitaxial
NiSi2 layer with an atomic flat NiSi2/Si interface shows super properties and has been used for advanced nanometre transistors [24–26]. The thickness of the epitaxial NiSi2 is limited to <10 nm due to the limited Ni thickness (<3 nm). Moreover, when the thickness of the Ni film is less than 6 nm, \( \theta \)-NiSi2 replaces \( \delta \)-NiSi2 as the first phase during silicidation [22]. The phase evolution can also be changed by additive elements especially in very thin films. For example, epitaxial \( \theta \)-NiSi2 instead of \( \delta \)-NiSi2 was obtained as the first phase by silicidation of Ni-Pt alloy with Si [27–29]. Geenen et al [30] discovered a broader temperature range for \( \delta \)-NiSi2 formation and low-temperature NiSi2 formation with 10% Co in 9 nm Ni. They also concluded that the NiSi formation is delayed by 10% Al doped in 9 nm Ni. However, for silicidation with C and F incorporations, only the phase formation behaviour was investigated in the case of thick films [9–11]. It should be very interesting to investigate the phase evolution of nickel silicide in the ultra-thin films combining the effect of C and F elements.

This paper presents the phase evolution of Ni silicides with different Ni thicknesses (3 nm, 6 nm and 8 nm) on CF4 plasma pre-implanted Si (100), aiming to study the impact of Ni thickness and the effects of C and F impurities on the phase evolution. This enables us to obtain a comprehensive understanding of silicide formation as a function of film thickness and the effects of impurities.

2. Experimental

Figures 1(a)–(c) show the silicidation process. In this work, 300 nm p-type silicon (100) wafers were used as substrates. Firstly, CF4 plasma immersion ion implantation (PIII) was applied using PULSION plasma implanter developed by Ion Beam Services (IBS) into Si substrates (figure 1(a)) at room temperature at a plasma energy of 2.5 keV to a fluence of \( 5 \times 10^{15} \) cm\(^{-2} \). The distributions of C and F in Si measured by secondary ion mass spectrometry (SIMS) were presented in figure 1(d). It showed very high density of C and F atoms with peak concentrations of \( \sim 2 \times 10^{17} \) cm\(^{-3} \) and \( \sim 6 \times 10^{15} \) F cm\(^{-3} \) in Si within a depth of 20 nm. The F peak is located at a depth of \( \sim 3 \) nm. CF4 PIII offers high efficiency to introduce a high concentration of C and F atoms closer to the surface than the beamline ion implantation.

Subsequently, these wafers were cut into small pieces for further processing. After removing the native SiO2 in 1% HF solution, Ni layers with different thickness (3 nm, 6 nm and 8 nm) were deposited onto the Si substrate by sputtering (figure 1(b)). Then the silicidation process was carried out with rapid thermal annealing (RTA) for 10 s at different temperatures ranging from 300 °C to 750 °C in forming gas (96% N2 and 4% H2) (figure 1(c)). Finally, the unreacted Ni was removed with \( \text{H}_2\text{SO}_4:\text{H}_2\text{O}_2 \) (4:1) solution.

The fabricated silicide layers were characterized with different techniques. The thermal stability was detected by van der Pauw resistance measurements. The morphologies of the layer and the silicide/silicon interface were investigated by transmission electron microscope (TEM). Rutherford backscattering spectrometry (RBS) was used to measure the silicide composition and thickness. The distribution of elements in the sample was measured by SIMS.
3. Results

Because of the large difference in resistivity between NiSi and NiSi$_2$, the sheet resistance measurement could offer a reliable way to study the phase formation. Figures 1(e)–(g) show the thermal stability of the silicide layers by plotting the sheet resistance as a function of silicidation temperature for silicides formed with 3 nm, 6 nm and 8 nm Ni, respectively. Reference samples without PIII are shown for comparison.

3.1. Silicide formed with 3 nm Ni

For the silicide layers formed with 3 nm Ni, the sheet resistance value remains almost constant with the annealing temperature from 400 °C to 750 °C, as shown in figure 1(e). It has already been reported that an epitaxial NiSi$_2$ layer was formed on Si (100) when the Ni layer is ∼3 nm [20]. Experimental results presented in this paper for the 3 nm Ni reference sample are consistent with the published results. In addition, the RBS channelling measurements indicate that a single crystalline NiSi$_2$ layer with a thickness of ∼9 nm was formed on the PIII samples, as indicated in figure 2(b). The cross section TEM image in figure 2(a) further shows an epitaxial NiSi$_2$ layer with a flat surface. However, pyramids with wedge-shaped (111) facets in some regions were observed at the interface, which resulted from the lower surface energy of (111) surfaces. Surprisingly, the sheet resistance of the NiSi$_2$ layer formed on CF$_4$ PIII Si substrate is much lower than the reference sample. At a silicidation temperature of 500 °C, the sheet resistance of the NiSi$_2$ layer formed on CF$_4$ PIII Si substrate is only 18.7 $\Omega/\Box$, corresponding to a specific resistivity of 16.8 $\mu\Omega$ cm which is comparable to the value of NiSi, while the reference sample shows a sheet resistance of 61.9 $\Omega/\Box$ for a specific resistivity of 55.7 $\mu\Omega$ cm. The lower resistivity with CF$_4$ should be related to the C and F incorporation in the NiSi$_2$ layers. We found that C atoms and part of the F atoms distribute in the NiSi$_2$ layer (see also figure 7 for 6 nm Ni), which may reduce the point defects in the layer, similar to the behaviour found for NiSi [9]. The effects of C and F in the layer for the low resistivity are still not very clear and requires more investigation. In addition, at a higher silicidation temperature of 750 °C, the epitaxial layer with perfect uniformity and atomic flat surface/interface was achieved, as indicated in figure 2(c).

3.2. Silicide formed with 6 nm Ni

For 6 nm Ni samples, a much larger thermal stability window is found for the layers formed on PIII treated substrates, as shown in figure 1(f). Figure 3(a) shows the RBS random and channelling spectra for the Ni silicide layers formed with 6 nm Ni at 450 °C. It is clearly seen that a NiSi layer with a thickness of ∼12 nm was formed on pure Si (100) without PIII, which is indicated by the higher Ni signal in the RBS spectrum (green). However, the RBS channelling spectrum indicates that an epitaxial NiSi$_2$ layer with a thickness of ∼19 nm was formed on CF$_4$ PIII Si (100) at 450 °C. The single crystalline NiSi$_2$ layer shows higher thermal stability than the NiSi layer as shown in figure 1(f). At 450 °C, the NiSi$_2$ layer shows a sheet resistance of 14.2 $\Omega/\Box$ corresponding to a specific resistivity of 27 $\mu\Omega$ cm, which is higher than the layer formed with 3 nm Ni due to more defects, as compared by the TEM images in figures 2 and 3, but lower than the NiSi$_2$ layer formed with 3 nm Ni on the reference sample. The reference NiSi layer has a sheet resistance of 17 $\Omega/\Box$ for a resistivity of 20.4 $\mu\Omega$ cm. The NiSi layer formed on pure Si suffers agglomeration at temperatures >600°C, thus causing the degradation of sheet resistance.

Figures 3(b) and (c) show the cross section TEM images for NiSi$_2$ layers formed with 6 nm Ni at silicidation temperature of 500 °C and 650 °C, respectively. The comparison of these two TEM images shows that the NiSi$_2$ (111) facets for the layer formed at 650 °C annealing temperature is less than that at 500 °C, indicating that higher annealing
temperature is needed for higher single crystalline quality of the NiSi$_2$ layer, which is similar as the case of 3 nm Ni. The formed NiSi$_2$ layer shows much better surface compared to the NiSi layer formed on pure Si(100) due to the single crystallinity.

X-ray reciprocal space mapping (RSM) measurement further shows that the NiSi$_2$ layer grows pseudomorphically and forms single crystal on the CF$_4$ PIII silicon substrate with tensile strain, as demonstrated in figure 4. NiSi$_2$ has a cubic structure with a lattice constant of 5.406 Å [31]. However, the lattice constant of NiSi$_2$ in the surface perpendicular direction is about 5.37 Å, smaller than the value in the reference. Due to the relatively low single crystalline quality and thin layer of NiSi$_2$, the NiSi$_2$ signal is not sharp compared to the Si substrate signal. The incorporation of C and F in the NiSi$_2$ layer and the relatively low single crystalline quality could cause a smaller measured lattice constant of NiSi$_2$ in the surface perpendicular direction.

3.3. Silicide formed with 8 nm Ni

From the RBS measurements (not shown) for 8 nm Ni samples, we found that ~15 nm thick NiSi layers were formed on both the pure Si and CF$_4$ PIII Si substrates at silicidation temperatures ranging from 400 °C to 650 °C. Figure 5 shows cross section TEM images for the NiSi layers formed on Si substrates with and without CF$_4$ PIII. The NiSi/Si interface is much smoother on the CF$_4$ PIII Si substrate, which leads to an increase of the thermal stability of NiSi as shown in figure 1(g).

Table 1 summarizes the Ni silicide phase formed on Si(100) at a temperature range from 400 °C to 650 °C, in which all the layers are thermally stable as shown in figures 1(e)–(g). From table 1, we can see that CF$_4$ PIII increases the critical thickness of Ni for direct formation of NiSi$_2$ from 3 nm to 6 nm. Figure 6 displays the phase evolution and the corresponding specific resistivity for silicides formed at 500 °C on Si substrates with and without CF$_4$ PIII. It is very interesting that the NiSi$_2$ layer formed with 3 nm Ni on CF$_4$ PIII Si substrate shows a very low resistivity which is comparable and even lower than that of NiSi formed with 6 and 8 nm Ni on pure Si substrates. This is a big advantage for applications because the epitaxial NiSi$_2$ is much more stable and uniform.

4. Discussion

It is known that the growth kinetics of NiSi$_2$ is nucleation controlled, while NiSi is controlled by diffusion with Ni as the diffusing specie [15, 16]. However, when the initial Ni film is thinner than 4 nm, reactions are no longer limited by atomic diffusion [19, 32, 33]. Interface energy plays a vital
role for ultra-thin films. Consequently, epitaxial NiSi₂ instead of polycrystalline NiSi is formed at low temperatures (400 °C). From the nucleation theory the silicide phase should be formed with a larger Gibbs free energy ($\Delta G$) reduction which is given by [34]:

$$\Delta G = \Delta \sigma + \Delta G_V = \Delta \sigma + (\Delta H - T \Delta S)$$  \hspace{1cm} (1)

where $\Delta \sigma$ is the interface energy change in which the grain boundary energy and the interface energy changed by strain are included. $\Delta G_V$ is the free energy difference in volume and $\Delta H$ is the enthalpy change, $T$ is the temperature, and $\Delta S$ is the entropy difference. Since $\Delta S$ is usually very small, $\Delta G$ for both NiSi and NiSi₂ phase can be written as the following:

$$\Delta G_1 = \Delta \sigma_1 + 2.01t\Delta H_1 \text{ for NiSi}$$  \hspace{1cm} (2)

$$\Delta G_2 = \Delta \sigma_2 + 3.59t\Delta H_2 \text{ for NiSi}_2$$  \hspace{1cm} (3)

where $t$ is the thickness of the Ni film. The factors 2.01 and 3.59 are the corresponding silicide/Ni thickness ratio [31]. The heat of formation $\Delta H$ for NiSi and NiSi₂ are $-10.3 \text{ kcal/mol.at}$ and $-7.2 \text{ kcal/mol.at}$, respectively [35]. Taking the molecular density of $4.552 \times 10^{22} \text{ cm}^{-3}$ for NiSi and $2.457 \times 10^{22} \text{ cm}^{-3}$ for NiSi₂ into account we can obtain $\Delta H_1 = -0.78 \text{ kcal cm}^{-3}.\text{at}$ and $\Delta H_2 = -0.29 \text{ kcal cm}^{-3}.\text{at}$. In addition, $\Delta \sigma_2$ is usually less than $\Delta \sigma_1$ due to the epitaxy of NiSi₂. Therefore we can conclude $|\Delta G_1| > |\Delta G_2|$, favoring the NiSi formation when $t$ is larger, as demonstrated schematically by solid lines in figure 8. For a very thin Ni layer, the interface energy ($\Delta \sigma$) contributes more to $\Delta G$ according to equations (2) and (3), which means $|\Delta G_1| < |\Delta G_2|$ when the film thickness is less than a critical thickness $t_c$. The epitaxial atomic alignment of NiSi₂ reduces the interface energy and is thus energetically favourable for its formation for a small thickness of Ni film.

It is likely that dopants like C and F change the silicide formation behaviour by affecting the interfacial energy. Figure 7 shows the SIMS profiles of C and F in NiSi₂ formed with 6 nm Ni. It is found that the distribution of C atoms is almost independent of silicidation temperature. More F atoms segregate at the interface by increasing temperature, as indicated by the higher F peak at the NiSi₂/Si interface. F segregation results are also found for other samples with different Ni thicknesses (not shown). Most C, and a portion of F atoms, exist in the silicide, refining the grain boundaries of silicide and reducing the defects. A large density of F atoms segregate at the silicide/Si interface, which enhances the interface cohesion and makes the interface smoother [9–11]. Therefore, for the formation of NiSi₂ with 3 nm Ni, CF₄ PIII caused a lower density of the pyramids at the interface and decreased the sheet resistance of NiSi₂. For the formation of NiSi with 8 nm Ni, the smoother surface and interface increase the nucleation barrier of NiSi₂ and thus enhance the thermal stability of NiSi [36].

We suppose that the entropy and enthalpy changed by C and F atoms are very small. The slope of the lines is then still dominated by the heat of formation. Our experimental results indicate a larger critical thickness in PIII samples. Thus we can conclude a smaller $\Delta \sigma_1$ and a larger $\Delta \sigma_2$ decrease, as illustrated in figure 8.
Moreover, by using equations (2) and (3), the difference of interface energy change between reaction \( \text{Ni} + \text{Si} \rightarrow \text{NiSi} \) and \( \text{Ni} + \text{Si} \rightarrow \text{NiSi}_2 \) can be obtained at a known critical thickness \( t_c \):

\[
\Delta \sigma^* = \Delta \sigma_1 - \Delta \sigma_2 = t_c \left( 2.01 \Delta H_1 - 3.59 \Delta H_2 \right)
\]

Then the ratio of \( \Delta \sigma^* \) between the reactions with and without CF4 PIII is simply expressed as:

\[
\frac{\Delta \sigma^*_A}{\Delta \sigma^*_B} \propto t_A / t_B
\]

From our experimental results, \( t_A \) and \( t_B \) correspond to approximately 3 nm and 6 nm, respectively. In consequence, a factor of \( \times 2 \) is obtained for the \( \Delta \sigma^* \) change by CF4 PIII.

As an application test we characterized the NiSi/p-Si Schottky contacts formed with 8 nm Ni at 500 °C. Figure 9 shows the measured reverse currents of two back-to-back Schottky diodes. It is interesting that the diode on the CF4 PIII p-Si substrate shows a lower Schottky barrier height (SBH) which is indicated by the higher reverse currents. We extracted from the I–V characteristics a lower SBH of 0.410 eV on the CF4 PIII substrates compared to the SBH = 0.458 eV on Si without CF4 PIII. The improved NiSi/Si interface by CF4 PIII is reflected by the smaller ideal factor of \( n = 1.02 \) with respect to \( n = 1.06 \) for the diode on pure Si(100) substrate. The segregation of C and F atoms at the interface passivate the interface and lower the SBH. The lower SBH and \( n \) can improve the contacts for nanoelectronics.

5. Conclusions

In conclusion, phase evolution of Ni silicides formed with ultrathin Ni films on pure Si (100) and CF4 PIII treated Si (100) have been systematically studied. It is revealed that both film thickness and CF4 incorporation can affect the phase evolution and the morphology of Ni silicides. C and F atoms distributed in the silicide layer and at the silicide/Si interface decrease the interface energy, thus resulting in an increase of the critical thickness \( t_c \) of direct formation of NiSi2 phase at low temperatures. With the CF4 PIII condition applied in this work \( t_c \) is doubled by CF4 PIII. With a Ni layer thickness \( > t_c \), for example 8 nm Ni, CF4 PIII improves the NiSi layer uniformity and the interface smoothness. In improved layer uniformity, resistivity and SBH offer benefits for nanoelectronic applications.

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