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Evolution of surface roughness during metal silicides phase transformation

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This study monitors systematically the evolution of surface roughness during metal (Ti, Co, and Ni) silicides phase transformation and coupling to other physical parameters. During metal/silicon reaction film surface roughness evolves due to the effect of nucleation and growth of metal silicides and is sensitive to different processing conditions. In general, surface roughness increases as new phases are formed and decreases during grain growth. The difference in roughness between processes indicates whether a process is more inferior for new phase formation. The correlation of surface roughness towards sheet resistance, film thickness, and phase transformation are found to be independent of film thickness in C49-to-C54 TiSi₂ polymorphic transformation but not for Co and Ni silicides. Any abnormalities from the trend indicate that the film is experiencing a physical degradation. © 2004 American Vacuum Society. [DOI: 10.1116/1.1636158]

I. INTRODUCTION

Study of surface roughness is important in characterizing surfaces. Previous reports have shown that the properties of materials can be affected by surface roughness. For example, electrical properties such as ohmic contact resistance of SiC can be seriously affected by microscratches and surface roughness.^{1,2} Furthermore, defects such as atomic height roughness have severe consequences on the spin structures of ferro/antiferromagnetic interfaces.³

In order to characterize surface roughness, various techniques have been developed. For instance, an optical probe-like ellipsometer is able to provide roughness information at the interfaces and on the surfaces of the samples. Haberland *et al.*⁴ have demonstrated the use of reflectance spectroscopy in studying the interface and surface roughness of AlGaAs/AlAs during growth. Atomic force microscope (AFM) has also been widely utilized to measure the surface morphology of different materials. Several groups^{5–7} have reported studies of properties such as composition, adhesion and friction, and grain sizes using AFM. Moreover, Buckley *et al.*⁸ have shown that there is a correlation between the surface roughness and recrystallization of copper metallization.

In this study, we studied the phase transformation of metal (Ti, Co, and Ni)/silicon reaction to form metal silicides. In particular, the root mean square (rms) surface roughness of these silicides during the phase transformation was closely monitored. It was found that surface roughness changes as phase transformation occurs and depends on different processing conditions at different annealing temperature. These processing conditions include amorphization and capping layer during silicidation. Hence, we used this behavior to

explain the nucleation and growth of metal silicides under different processing conditions. The correlation results of surface roughness towards sheet resistance, film thickness, and phase transformation are found to be independent of film thickness in C49-to-C54 TiSi₂ polymorphic transformation but not for Co and Ni silicides.

The study also showed that initial agglomeration of Ni silicide is usually accompanied by a large change in surface roughness that is directly opposite the trend in sheet resistance measurement.

II. EXPERIMENT

Silicidation were carried out for Ti, Co, and Ni films on blanket 8 in. Si(100) wafers. Prior to metal deposition, Si wafers were cleaned using HF 100:1 to remove native oxide on the surface. Subsequently, these wafers were loaded into a sputtering chamber for metal deposition (a) 400 Å of Ti, (b) 130 Å of Co with 125 Å of Ti-capping layer and (c) 160 and 300 Å of Ni films. These films were sputtered under the pressure of 1×10^{-8} Torr in N₂ ambient. Part of the Ti deposited wafers were then amorphized using either indium (dose 1E13 atoms/cm² at 130 keV) or silicon implantation (dose 2E15 atoms/cm² at 40 keV). All wafers were then rapid thermal annealed (RTA) to various temperatures ranging from 400 to 900 °C with ramp rate of 50 °C/s. After RTA, the unreacted Ti, Co, Ni, and TiN layers formed during the RTA were removed by using H₂O₂:NH₄OH:H₂O; 1:1:5 for 3–5 min.

The root mean square (rms) surface roughness of these samples had been characterized by using Digital Instruments Nanoscope IIIa TM (Tapping mode) AFM. The Si tip was operated at its resonance frequency of 284.86 kHz with a spring constant of 23.88 N/m. The oscillation frequency and

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voltage applied to the piezoelectric crystal were adjusted to achieve optimal sample contrast with probing area of $2.5 \mu\text{m} \times 2.5 \mu\text{m}$. In addition, a sampling interval of 512 per line is used. X-ray diffraction (XRD) was used to monitor the different phase transformation for the Ti, Co, and Ni silicides. The energy of the XRD beam was 40 keV with a current of 30 mA and 2° glancing angle. Four point probes had been used to measure all the sheet resistances of the samples at room temperature. Some of the film thicknesses are measured using transmission electron microscope (TEM) while the others are calculated using the sheet resistance results. The specimens were observed in a JEOL 200 TEM (Japan Electron Optics Laboratory, Ltd., Tachikawa, Tokyo) operated at 200 kV.

III. RESULTS AND DISCUSSION

Figure 1 shows the XRD spectra from samples of Ti/Si reaction which have been RTA annealed to temperature ranging from 500 to 850 °C. For the samples without amorphization (standard process), C49-to-C54 phase transformation occurs at around 760 °C (Fig. 1). However, the same phase transformation occurs at a lower temperature of $\sim 700^\circ\text{C}$ for samples with indium [Fig. 1(b)] and silicon [Fig. 1(c)] amorphization. These results are consistent with several other studies.^{9–11} Furthermore, Figs. 1(b) and 1(c) also show that In amorphization produces higher C54(131) intensity than Si amorphization when annealed at 700 and 760 °C. Higher C54(131) peak intensity indicates higher C54–TiSi₂ nuclei formed.

Table I relates the results of different physical parameters of TiSi₂ processed under varied conditions and depicts the thickness of titanium disilicides film remains approximately the same after the C49-to-C54 phase transformation since only 6% of volume change occurs during phase transformation. Sheet resistances decrease significantly as with the nucleation of C54 phase. The sheet resistance reduces to $\sim 3 \Omega/\square$ at 760 and 700 °C for standard and ion implanted process, respectively. This is consistent with the XRD spectrum in Fig. 1. The rms surface roughness of these samples had been measured with AFM. We found that there is a strong correlation between the surface roughness and the XRD peaks. As the surface roughness can be affected by film thickness, we have normalized the surface roughness with film thickness. From the trend shown in Fig. 2(a), we see that normalized rms surface roughness for samples using standard process increases with annealing temperature to about 760 °C. Subsequently, surface roughness starts to decrease as annealing temperature increases to 850 °C. We attribute this trend to the nucleation and growth of C54–TiSi₂. From Fig. 1, standard samples annealed from 600 to 700 °C show only the growth of C49 phase and no C54 phase. At this temperature range, we believe that the increase in surface roughness [Fig. 2(a)] is due to the C49 grain growth that results in induced stress. Figure 3 shows the TEM micrographs of C49–TiSi₂ annealed to 650 °C. Strains are clearly seen (as indicated by the arrows) at the interfaces and grain boundaries. At the grain boundary of two C49 grains, the grains

(a)

(b)

(c)

FIG. 1. XRD results of C49-to-C54 TiSi₂ phase transformation at different peak annealing temperature (a) standard—without amorphization; (b) indium preamorphization at $1\text{E}14 \text{ atoms/cm}^2$ at 130 keV; (c) silicon amorphization at $2\text{E}15 \text{ atoms/cm}^2$ at 40 keV.

appear to grow into one another resulting in reduced stress that causes larger roughness at the surface of the TiSi₂ film.

As annealing temperature increases to 760 °C, C49-to-C54 phase transformation has completed. This can be seen in Fig. 1(a) where C49 peak has been replaced by three C54 peaks. Surface roughness also reaches the maximum at

TABLE I. Results of various TiSi_2 physical parameters annealed under different processing conditions.

Splits	Temperature (°C)	Sheet resistance ($\Omega/\square$)	Surface roughness (nm)	Phase transformation	Thickness (nm)	
					Ave.	Std. dev.
Standard (without implantation)	650	17.5	6.987	C49	46.5	5.15
	700	13.6	8.124	C49	57.4	2.84
	760	3.21	10.02	C54	62.3	1.87
	850	3.15	8.78	C54	63.1	3.24
In-ITM	650	15.1	3.959	C49+C54	61.5	3.12
	700	3.18	5.975	C54	62.8	3.25
	850	3.13	3.31	C54	62.6	2.81
Si-ITM	600	15.2	2.038	C49	45.1	4.15
	650	10.3	3.388	C49+C54	62.4	2.12
	700	3.32	4.261	C54	61.7	3.18
	760	3.15	3.36	C54	63.1	2.89
	850	3.13	3.271	C54	62.2	3.52

760 °C (Fig. 2). We attribute the change in the roughness to the $\sim 6\%$ volume difference during C49-to-C54 nucleation but these volume change does not affects the thickness significantly as seen in Table I. When the C54– TiSi_2 nuclei grow between the triple point junction of C49 phase heterogeneous nucleation,^{12,13} the grain size distribution will result

in large surface roughness. When temperature increases further to 850 °C, the growth mechanism of C54 phase transformation dominates and nuclei grow two dimensionally, thus surface roughness becomes smaller again.

Therefore, surface roughness can be used to monitor C54 phase nucleation and growth. Similar phenomena were observed for indium and silicon implanted samples. However, the maximum surface roughness was found to occur at 700 °C instead of 760 °C indicating that C49-to-C54 TiSi_2 phase transformation occurs at a lower temperature than in samples without amorphization.

Furthermore, it is observed that samples using In amorphization show higher surface roughness than samples with Si amorphization at 700 °C. We believe this can be explained as follows. First, indium precipitation occurs during silicidation. This will result in larger stress in the silicide film causing missing grains (Fig. 4). The voids (missing grains) formation can be caused by the outdiffusion of indium during

FIG. 2. (a) Plot of surface roughness against peak annealing temperature for C49 to C54 phase transformation for different amorphization conditions; (b) sheet resistance of Ti silicides annealed to different temperatures.

FIG. 3. Interfaces of C49– TiSi_2 grains and silicon substrate annealed to 650 °C.

FIG. 4. In-ITM at 650 °C with voids (missing grain) at the size of 94 nm.

RTA. Figure 5 shows the indium depth profile measured with SIMS before and after silicidation. We see that indium atoms move towards the interface of the silicide upon rapid thermal annealing (RTA). Ganin *et al.*¹⁴ have shown that indium precipitation occurs in Si after RTA. They indicated that as indium concentration reaches its solubility limits in silicon during diffusion, phase separation occurs. Eventually indium tends to segregate that will result in larger stress.

Second, the In amorphized samples shows higher C54 nucleation density after annealed to 700 °C. This can be seen in Figs. 1(b) and 1(c) where the C54(131) peak in Fig. 1(b) is about 10% larger than in Fig. 1(c). Hence, higher C54 nucleation leads to larger peak surface roughness.

FIG. 5. SIM profile for indium diffusion (a) before silicidation, (b) after annealed at 760 °C.

Figure 2(b) shows a strong correlation between sheet resistance and the surface roughness [Fig. 2(a)]. As the sheet resistance reaches its minimum, the surface roughness reaches its maximum. Subsequently, the sheet resistance remains constant with increasing annealing temperature but surface roughness decreases. We can conclude that smoother surface is obtained with higher annealing temperature due to increased nucleation density¹⁵ and grain growth of one phase. Moreover, we see that the surface roughness of the samples with amorphization reach the minimum point at a lower temperature compared to the standard sample. This is consistent with the observation that the amorphized samples have the C49-to-C54 transformation occurs at lower temperatures as point out earlier.

More details can be seen when applied to CoSi₂ phase transformation. Table II depicts the correlation of sheet resistance, film thickness, surface roughness, and phase transformation. Here, we see that Co silicides thicknesses increase from ~20 to ~30 nm at annealing temperature of below and above 575 °C, respectively. There is large volume change for CoSi-to-CoSi₂ phase transformation, thus thickness effect can contribute significantly to the roughness. To eliminate the effect of film thickness, Fig. 6 are plotted in normalized rms surface roughness of CoSi₂ transformation with different annealing temperatures. As temperature increases from 450 to 550 °C, CoSi grows with increasing roughness as evidenced by the XRD spectra in Fig. 7. At 575 °C, we see sharp roughness increases due to the initial nucleation of new CoSi₂ phase. Appelbaum *et al.*¹⁶ show initially in their investigation that the formation CoSi/CoSi₂ interface is rough. This sharp increase in roughness can be induced by the large

TABLE II. Results of various CoSi₂ physical parameters annealed under different processing conditions.

Splits	Temperature (°C)	Sheet resistance (Ω/□)	Surface roughness (nm)	Phase transformation	Thickness (nm)	
					Ave	Std. dev.
Co	450	48.98	3.293	CoSi	20.4	7.1
	500	91.11	3.864	CoSi	21.5	8.4
	525	91.23	4.205	CoSi	21.7	8.8
	575	27.96	4.932	CoSi + CoSi ₂	26.4	9.2
	600	5.875	4.863	CoSi ₂	31.8	6.1
	850	5.625	3.514	CoSi ₂	34.1	6.5

FIG. 6. Plot of surface roughness and sheet resistance of cobalt silicidation against peak annealing temperature for different process conditions.

volume change of CoSi:CoSi₂ thickness formed per Å of Co metal on silicon are 2.02:3.52. As temperature increases to beyond 575 °C (Fig. 7), CoSi-to-CoSi₂ transformation is completed and CoSi₂ grows laterally, the rms surface roughness also reduces (Fig. 6). Based on the XRD and surface roughness data and other works,^{16,17} CoSi nucleates and grows when the annealing temperature changes from 450 to 550 °C. This explains why the surface roughness increases sharply at this temperature range. As both CoSi and CoSi₂ exist at temperature 575 °C, rms surface roughness suddenly increased with a peak. This peak cannot be seen if they are not normalized with thickness. Thus changes in film thickness during phase transformation may take away some vital information. At annealing temperature of 600 °C, the drop in rms surface roughness is rapid since CoSi₂ growth is extremely fast at this temperature region¹⁶ and covering the film surface. In the temperature range between 600 and 700 °C, a competing nucleation and growth of CoSi₂ started to cause and the roughness remains relatively unchanged. At a temperature higher than 700 °C, CoSi₂ grows resulting in smaller roughness. Furthermore, it is possible that the cobalt disilicides' tendency to grow epitaxially that led to smaller surface roughness.

FIG. 7. XRD results of Co silicidation annealed to peak temperature from 400 °C to 850 °C with 125 Å of Ti-capped layer.

FIG. 8. A plot of surface roughness and sheet resistance vs different annealing temperature for Ni silicides transformation.

FIG. 9. XRD analysis of Ni silicides transformation.

FIG. 10. Plot of surface roughness and sheet resistance vs different annealing temperature at 300 Å.

FIG. 11. AFM micrographs of Ni silicide (a) 500 °C, (b) 600 °C, (c) 750 °C, and (d) 800 °C.

Figure 6 also shows how sheet resistance of CoSi₂ varies with annealing temperature. Sheet resistance increases as temperature increase and reaches a maximum at ~500–550 °C. Sudden drops in sheet resistance occur at 575 °C further confirms the correlation of sheet resistance and surface roughness to phase transformation. Subsequently, sheet resistance decreases rapidly at around 600 °C where the nucleation of CoSi₂ occurs. Sheet resistance (R_s) then remains unchanged as temperature further increases. Combining with the observations made in the XRD spectrum (Fig. 7), clearly indicates that the nucleation of CoSi₂ will cause the surface roughness to increase. However, as the CoSi₂ lateral grain growth dominates at higher temperatures (~800 °C), surface roughness decreases.

Therefore, from the results of Ti and Co silicides, we can conclude when there is a sharp drop of surface roughness from its maximum during phase transformation, it is an indication that one of the major phases has dominated the growth process.

Figure 8 shows the changes in surface roughness for NiSi

to NiSi₂ phase transformation with increasing annealing temperature. The thickness of the deposited Ni was 160 Å. This trend is similar to what have been observed for both Ti and Co silicide. At temperatures between 500 and 750 °C, NiSi grows with higher rms surface roughness. As the sheet resistance is measured to be relatively the same,¹⁸ the thickness is calculated to be ~30 nm. Here, the sudden increase in surface roughness at 750 °C is a signature of the formation of NiSi₂ evidenced in XRD analysis in Fig. 9. Since NiSi to NiSi₂ transformation is accompanied by a thickness ratio of 2.36, the thickness of NiSi₂ formed becomes ~71 nm. By normalizing rms surface roughness with thickness, the trend obtained is similar to the sheet resistance graph (Fig. 8).

For thicker Ni films, the surface roughness was a function of annealing temperature that behaved differently when compared to thinner films. Figure 10 illustrates that for a 300 Å Ni film, the surface roughness does not produce a trend as in Fig. 8. Instead, the roughness suddenly decrease and increase without a trend. This change is attributed to the agglomeration of the Ni films as shown in Fig. 11 which shows the

FIG. 12. Depth of Ni silicides agglomeration measured using AFM after annealed to 800 °C.

changes of Ni silicide film at different temperatures. Voids [Fig. 11(c)] formation starts to occur at 750 °C.

We have also measured the sheet resistance of the Ni films as shown in Fig. 10. We see that large change in surface roughness occurs during agglomeration. At temperatures of 600–700 °C, comparable changes in both surface roughness and sheet resistance is observed. However, at temperatures above 700 °C, large decrease in surface roughness occurs indicating the onset of Ni silicide agglomeration that is on the opposite trend of the sheet resistance measurement. The sign of severe agglomeration measured by sheet resistance only occurs at 850 °C where it leaped to 160Ω/□ is shown in Fig. 11(a). Here the depth of the agglomeration is ~30 nm that is 25% of the Ni disilicides thickness formed (~130 nm) (Fig. 12).

IV. CONCLUSION

We have demonstrated the correlation between surface roughness and metal silicides phase transformation and

found that higher nucleation density is usually associated with higher surface roughness in C49-to-C54 TiSi₂ formation. In addition, we have used the change in surface roughness to identify the effectiveness of nucleation of C54–TiSi₂ on different implantation conditions and found that indium provides better nucleation than silicon implantation. Although similar observation is seen in CoSi-to-CoSi₂ formation, the change in thickness can affect the sensitivity in detecting any formation of new phase. Here the depth of the agglomeration is ~30 nm that is 25% of the Ni disilicides thickness formed (~130 nm) (Fig. 12).

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