Annealing effects on the interfacial reactions of Ni on Si 0.76 Ge 0.24 and Si 1xy Ge x C y

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Annealing effects on the interfacial reactions of Ni on $Si_{0.76}Ge_{0.24}$ and $Si_{1-x-y}Ge_xC_y$

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Interfacial reactions of $Ni/Si_{0.76}Ge_{0.24}$ and $Ni/Si_{1-x-y}Ge_xC_y$ by vacuum annealing and pulsed KrF laser annealing were studied. Upon annealing at a temperature of 200–600 °C Ge segregation occurred with the extent becoming more severe at higher temperatures. The temperatures at which phase transformation and the agglomeration structure occurred were higher for $Ni/Si_{1-x-y}Ge_xC_y$ than for $Ni/Si_{0.76}Ge_{0.24}$. Upon pulsed KrF laser annealing the agglomeration structure was considerably improved, however, the retardation of phase transformation in the $Ni/Si_{1-x-y}Ge_xC_y$ system still occurred. C accumulation around the original amorphous/crystal interface formed by C^+ implantation played a significant effect on delaying the phase transformation. For the $Ni/Si_{0.76}Ge_{0.24}$ and $Ni/Si_{1-x-y}Ge_xC_y$ samples annealed at 0.2 J/cm^2 for 20 and 30 pulses, respectively, smooth $Ni(Si_{0.76}Ge_{0.24})_2$ and $Ni(Si_{1-x-y}Ge_xC_y)_2$ films could be grown, meanwhile Ge segregation and strain relaxation of the unreacted $Si_{0.76}Ge_{0.24}$ films were effectively suppressed. © *2000 American Vacuum Society.* [S0734-2101(00)04301-3]

I. INTRODUCTION

Recently, $Si_{1-x}Ge_x$ on Si has been extensively studied for applications in the field of optoelectronics and high speed heterojunction bipolar transistors.[1,2] Since the lattice spacing of Ge is 4.2% larger than that of Si, compressive strains develop in the $Si_{1-x}Ge_x$ overlayer, which create stability problems that limit the thickness of the pseudomorphic $Si_{1-x}Ge_x$ overlayer. Carbon introduced substitutionally into $Si_{1-x}Ge_x$ can reduce the lattice mismatch between $Si_{1-x}Ge_x$ and Si, opening up the opportunities for fabricating thicker pseudomorphic $Si_{1-x}Ge_x$ films with a high Ge content. Besides, carbon introduced substitutionally into $Si_{1-x}Ge_x$ can change the band gap of $Si_{1-x}Ge_x$,[3–5] providing an additional design parameter in band structure engineering on Si.

For device applications the formation of metal/$Si_{1-x}Ge_x$ and metal/$Si_{1-x-y}Ge_xC_y$ ohmic or rectifying contacts is required. Thus, the interfacial reactions of some metals such as Ni,[6–8] Pt,[9,10] Pd,[10–13] Ti,[14–19] Co,[20–24] W,[25,26] Cr,[27] Cu,[28,29] and Zr[30] on $Si_{1-x}Ge_x$, and Ti,[18] Cu,[28] Co,[31] and Zr[32] on $Si_{1-x-y}Ge_xC_y$ by conventional furnace annealing and pulsed laser annealing have been studied, respectively. For conventional furnace annealing the formation of a ternary phase was generally accompanied with Ge segregation. Additionally, an agglomeration structure appeared at higher annealing temperatures. These phenomena could be attributed to the higher heat formation for metal-Si than for metal-Ge.[33] Rapid thermal annealing and pulsed laser annealing could shorten the annealing time, resulting in a reduction of Ge segregation. Furthermore, pulsed laser annealing could produce a smooth and continuous germanosilicide film without inducing strain relaxation in the unreacted $Si_{1-x}Ge_x$ film.[13]

In the present study, the effects of vacuum annealing and pulsed KrF laser annealing on the interfacial reactions of $Ni/Si_{0.76}Ge_{0.24}$ and $Ni/Si_{1-x-y}Ge_xC_y$ were studied by using transmission electron microscopy (TEM) in conjunction with energy dispersive spectrometry (EDS), x-ray diffraction (XRD), and secondary ion mass spectrometry (SIMS). The results show that by multiple pulse laser annealing the phenomena as Ge segregation and strain relaxation of the unreacted $Si_{0.76}Ge_{0.24}$ films, and the formation of agglomeration structure appearing in vacuum annealing can be significantly improved. In addition, C plays an important role on retarding the phase transformation of $Ni/Si_{1-x-y}Ge_xC_y$ upon either vacuum annealing or pulsed laser annealing.

II. EXPERIMENT

Strained and partially relaxed $Si_{0.76}Ge_{0.24}$ films about 100 and 150 nm thick were grown on *n*-type Si(100) at 550 °C by ultrahigh vacuum chemical vapor deposition (CVD), respectively. $Si_{1-x-y}Ge_xC_y$ films were prepared by C ions implanted into the partially relaxed $Si_{0.76}Ge_{0.24}$ films and subsequent pulsed KrF laser annealing at an energy density of 0.8–1.0 J/cm^2. C ions were implanted at 80 keV with a dose of $1.0\times10^{16}/cm^2$. In order to confine most of the implanted ions in the $Si_{0.76}Ge_{0.24}$ films, a SiO_2 overlayer about 150 nm thick was grown on the $Si_{0.76}Ge_{0.24}$ films. The maxi-

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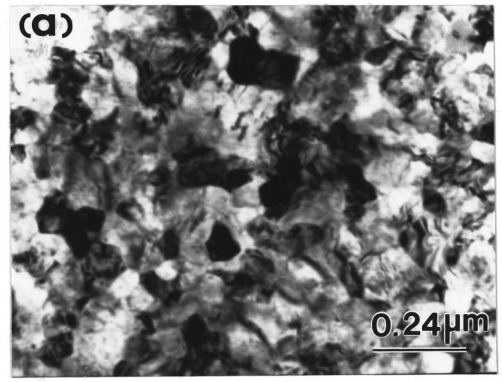
mum of the implanted profile in the $Si_{0.76}Ge_{0.24}$ films was about 90 nm. Before pulsed laser annealing the SiO_2 layer was chemically removed by 5% HF solution. Details of the preparation of $Si_{1-x-y}Ge_xC_y$ films and their characterization were described elsewhere.[34]

Prior to deposition the substrates were cleaned by RCA method[35] and then immediately loaded into the chamber. Ni about 25 nm thick was deposited onto the $Si_{0.76}Ge_{0.24}$ and $Si_{1-x-y}Ge_xC_y$ films at room temperature by electron gun evaporation at a rate of 0.1 nm/s. The base pressure was around $1-2\times10^{-6}$ Torr. Furnace annealing was carried out at a temperature of 200–700 °C in a vacuum of $1-2\times10^{-6}$ Torr. Pulsed KrF laser annealing was performed at an energy density of $0.1-0.4$ J/cm^2 in a vacuum around 2×10^{-2} Torr. The pulse length is 14 ns. The laser beam was focused onto an area of 4×4 mm^2. For laser annealing, the sample was illuminated by a single pulse unless otherwise specified. Phase formation, microstructure, and chemical compositions of the reacted layer were analyzed by EDS/TEM which was equipped with a field emission gun with an electron probe 1.2 nm in size. The strain relaxation of the unreacted $Si_{0.76}Ge_{0.24}$ films after annealing was examined using XRD. The C depth profiles of the $Ni/Si_{1-x-y}Ge_xC_y$ samples after annealing were obtained by SIMS. A sputtering beam of 4.5 keV Cs^+ ions was rastered over an area of $250\times250\ \mu m^2$. The positive secondary ions were collected from the central region of the sputtered crater.



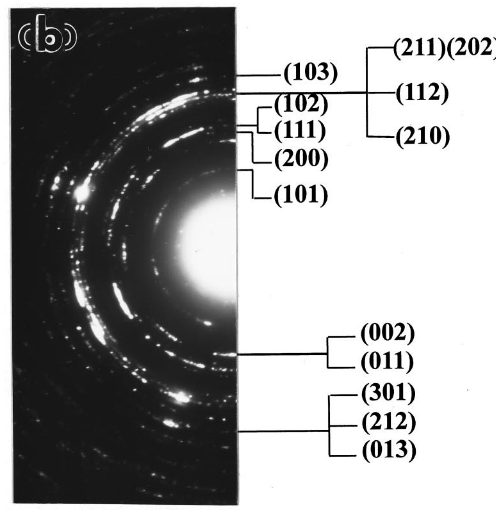


FIG. 1. (a) Plan-view TEM image and (b) electron diffraction pattern (DP) of the $Ni/Si_{0.76}Ge_{0.24}$ sample annealed at 200 °C showing the formation of $Ni(Si_{1-x}Ge_x)$.

III. RESULTS AND DISCUSSION

A. Vacuum annealing

For the $Ni/Si_{0.76}Ge_{0.24}$ films annealed at a temperature of 200–500 °C $Ni(Si_{1-x}Ge_x)$ was formed as shown in Fig. 1. From EDS/cross-section TEM (XTEM) analysis Ge segregation from the $Ni(Si_{1-x}Ge_x)$ layer to the underlying $Si_{0.76}Ge_{0.24}$ substrate apparently appeared at temperatures above 300 °C with the extent becoming more severe at higher temperatures. At temperatures above 400 °C some Ge-rich $Si_{1-x}Ge_x$ grains were apparently formed between the Ge-deficient $Ni(Si_{1-x}Ge_x)$ grains, forming the agglomeration structure as shown in Fig. 2. It seems that Ge is expelled from the $Ni(Si_{1-x}Ge_x)$ grains and diffuses into the $Ni(Si_{1-x}Ge_x)$ grain boundaries to react with Si and Ge from the substrate, causing the formation of the Ge-rich $Si_{1-x}Ge_x$ grains. Similar results have been found in the $Ti/Si_{1-x}Ge_x$ system.[14,15] The heats of formation for NiSi and NiGe have been determined to be about -45 and -32 KJ/mole, respectively.[33] These values suggest that Ni tends to react preferably with Si. Above 550 °C $Ni(Si_{1-x}Ge_x)_2$ was formed, in which only a trace amount of Ge was present.

For the $Ni/Si_{1-x-y}Ge_xC_y$ films annealed at 200 °C $Ni(Si_{1-x-y}Ge_xC_y)$ was formed concurrently with $Ni_2(Si_{1-x-y}Ge_xC_y)$ as shown in Fig. 3. As compared with Fig. 1, the grain size of $Ni(Si_{1-x-y}Ge_xC_y)$ is somewhat smaller than that of $Ni(Si_{1-x}Ge_x)$. After annealing at a temperature of 250–550 °C only $Ni(Si_{1-x-y}Ge_xC_y)$ was present. From EDS/XTEM analysis the $Ni(Si_{1-x-y}Ge_xC_y)$ layer was deficient in Ge with the extent being more severe after annealing at higher temperatures. At 600 °C $Ni(Si_{1-x-y}Ge_xC_y)_2$ was formed concurrently with the appearance of agglomeration.

The above results show that the grain growth, phase transformation, and the appearance of agglomeration are sluggish for the $Ni/Si_{1-x-y}Ge_xC_y$ system in comparison with the $Ni/Si_{0.76}Ge_{0.24}$ system. From SIMS analysis for the $Ni/Si_{1-x-y}Ge_xC_y$ samples annealed at 200 and 400 °C, respectively, in Fig. 4, most C were still accumulated around the original amorphous/crystal interface which was formed by C^+ implantation in the fabricating process of the $Si_{1-x-y}Ge_xC_y$ films.[34] The original amorphous/crystal inter-

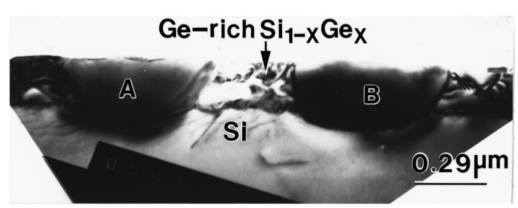


FIG. 2. XTEM image of the $Ni/Si_{0.76}Ge_{0.24}$ sample annealed at 500 °C showing the agglomeration structures. The grains denoted as ''*A*'' and ''*B*'' are of $Ni(Si_{1-x}Ge_x)$.

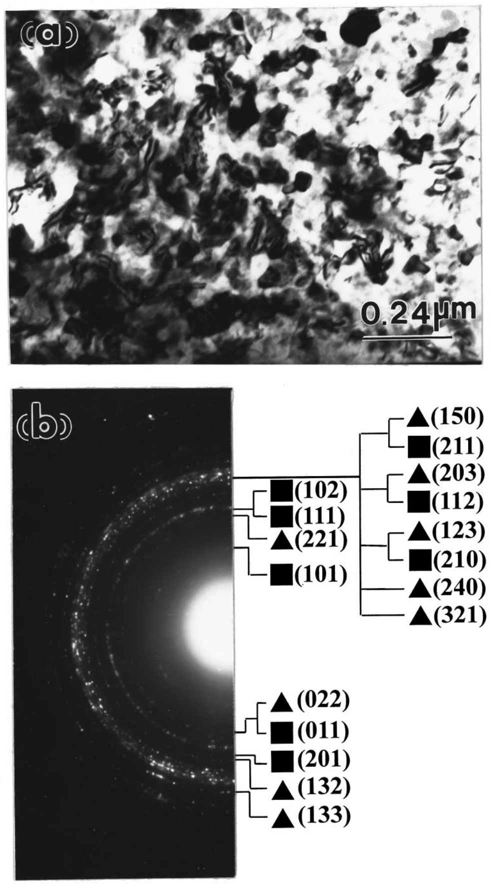


FIG. 3. (a) Plan-view TEM image and (b) DP of the Ni/$Si_{1-x-y}Ge_xC_y$ sample annealed at 200 °C showing the formation of $Ni_2(Si_{1-x-y}Ge_xC_y)$ and $Ni(Si_{1-x-y}Ge_xC_y)$ which are denoted as (▲) and (■), respectively.

face is about 90 nm deep. Upon annealing at higher temperatures, e.g., 600 °C, most C diffused to the film surface. The retardation of phase formation and C accumulation at the germanosilicide/epilayer interface have been observed in the Ti/$Si_{1-x-y}Ge_xC_y$ and Co/$Si_{1-x-y}Ge_xC_y$ systems.[18,31] It is evident that C plays an important role on blocking the interfacial reactions.

B. Pulsed KrF laser annealing

For the Ni/$Si_{0.76}Ge_{0.24}$ films annealed at 0.1 J/cm^2 $Ni_2(Si_{1-x}Ge_x)$ was formed. At an energy density of 0.2–0.3 J/cm^2 two layers were formed as shown in Fig. 5. From microdiffraction analysis, the upper layer was amorphous, while the lower layer was $Ni(Si_{1-x}Ge_x)_2$. From EDS/XTEM analysis the Ni/(Si+Ge) atomic ratios for the upper and lower layers were about 1:1 and 1:2, respectively, and Ge segregation to the $Ni(Si_{1-x}Ge_x)_2$ layer and the underlying $Si_{0.76}Ge_{0.24}$ film occurred. At 0.4 J/cm^2 constitutional supercooling appeared, resulting in the cellular structures of Ge-deficient $Si_{1-x}Ge_x$ islands surrounded by $Ni(Si_{1-x}Ge_x)_2$.

For the Ni/$Si_{1-x-y}Ge_xC_y$ films annealed at an energy density of 0.1–0.2 J/cm^2 $Ni_2(Si_{1-x-y}Ge_xC_y)$ was formed, while significant amount of Ni remained unreacted. For the

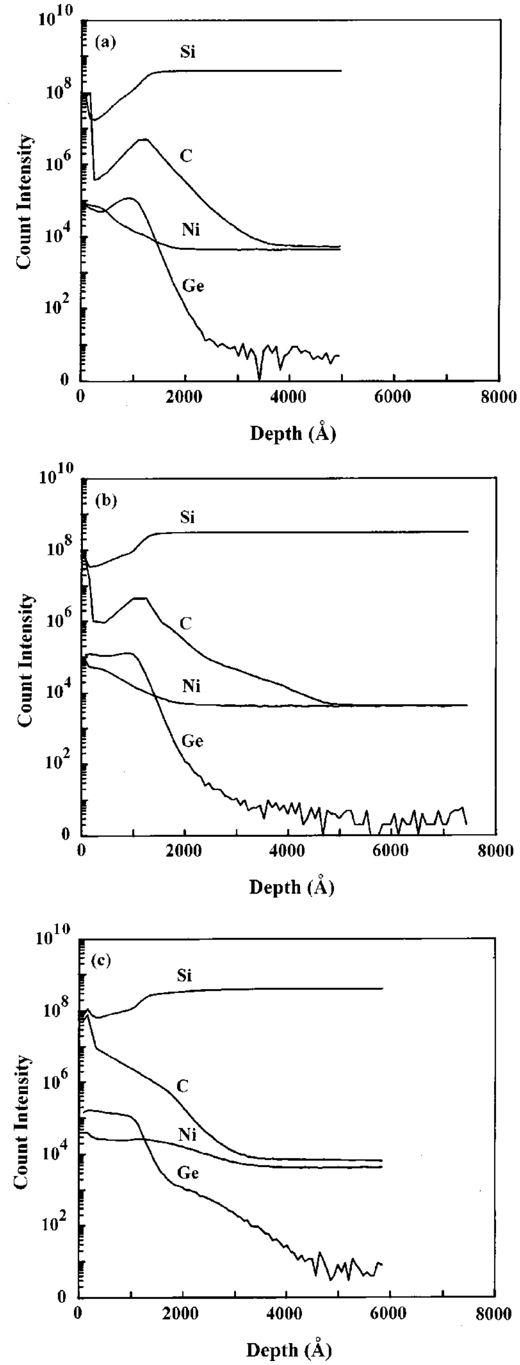


FIG. 4. SIMS depth profiles of the Ni/$Si_{1-x-y}Ge_xC_y$ sample annealed at (a) 200, (b) 400, and (c) 600 °C.

sample annealed at 0.3 J/cm^2 two layers, i.e., an amorphous layer on the $Ni(Si_{1-x-y}Ge_xC_y)_2$ layer similar to those shown in Fig. 5 were formed. Above 0.4 J/cm^2 constitutional supercooling occurred.

In our previous studies for Pd/$Si_{0.76}Ge_{0.24}$ system[13] multiple pulse annealing could homogenize the Pd concentration in the germanosilicide layer without inducing Ge segregation to the underlying $Si_{0.76}Ge_{0.24}$ films and strain relaxation. In the present study, therefore, multiple pulse annealing was also performed. For the Ni/$Si_{0.76}Ge_{0.24}$ films annealed at 0.2 J/cm^2 for 10 pulses $Ni(Si_{1-x}Ge_x)$ was formed concur-

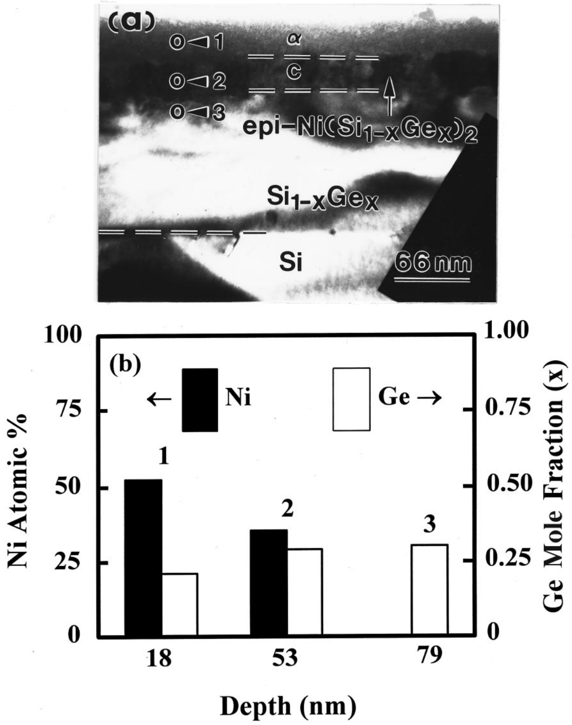


FIG. 5. (a) XTEM image and (b) the depth profiles of the chemical elements for the $Ni/Si_{0.76}Ge_{0.24}$ sample annealed at 0.3 J/cm^2.

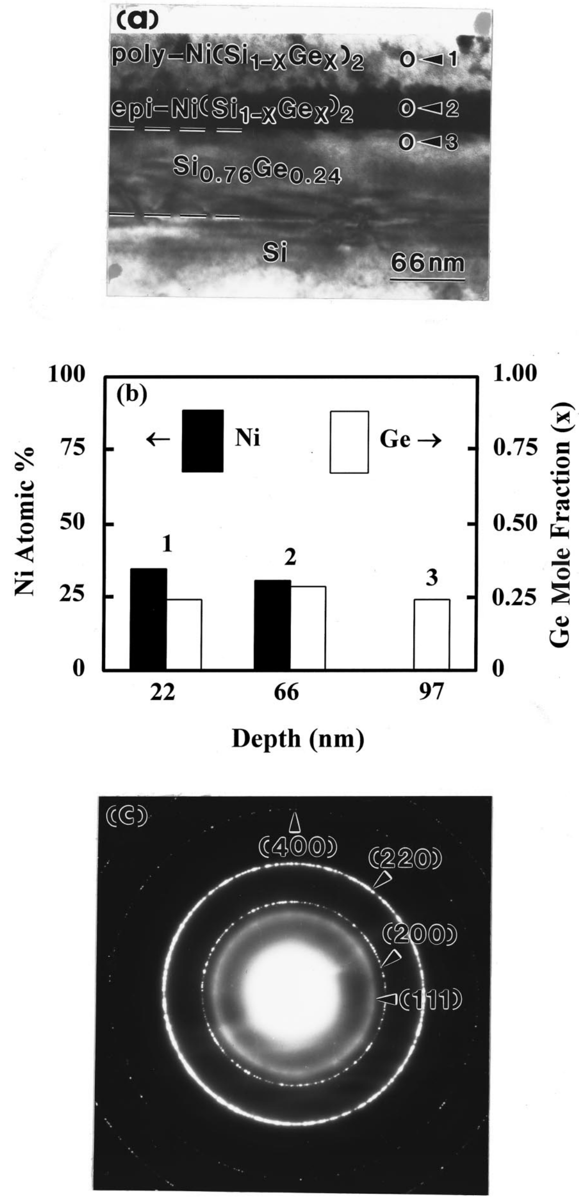


FIG. 6. (a) XTEM image and (b) the depth profiles of the chemical elements for the $Ni/Si_{0.76}Ge_{0.24}$ sample annealed at 0.2 J/cm^2 for 20 pulses, (c) DP of the poly-$Ni(Si_{1-x}Ge_x)_2$.

rently with $Ni(Si_{1-x}Ge_x)_2$. After annealing for 20 pulses a smooth $Ni(Si_{1-x}Ge_x)_2$ layer was formed as shown in Fig. 6, in which the upper layer is polycrystalline, while the lower layer is epitaxial. Meanwhile the EDS/XTEM data show that although some Ge tended to segregate down to the lower part of the germanosilicide layer, Ge segregation out of the germanosilicide to the underlying $Si_{0.76}Ge_{0.24}$ films did not occur. Correspondingly, the XRD pattern of the sample annealed at 0.2 J/cm^2 for 20 pulses is shown in Fig. 7(b). Evidently, the lattice constant of the unreacted $Si_{0.76}Ge_{0.24}$ film in the direction perpendicular to the film surface remained nearly unchanged in comparison with that of the as-grown $Si_{0.76}Ge_{0.24}$ film, revealing that no strain relaxation appeared in the unreacted $Si_{0.76}Ge_{0.24}$ film. Meanwhile, in Fig. 7(c) the XRD pattern of the sample annealed at 400 °C shows the occurrence of strain relaxation. The aforementioned results have shown that upon annealing at temperatures above 300 °C Ge segregation to the unreacted $Si_{0.76}Ge_{0.24}$ film occurred with the extent becoming more severe at higher temperatures. The chemical inhomogeneities present in the surface of the unreacted $Si_{0.76}Ge_{0.24}$ film may induce misfit dislocations and hence cause strain relaxation.[36] As a consequence, it seems that preventing Ge segregation to the unreacted $Si_{1-x}Ge_x$ film can suppress strain relaxation. Similar results have been observed in the $Pd/Si_{0.76}Ge_{0.24}$ system.[13] For the samples annealed at 0.3 J/cm^2 for 5 pulses a single $Ni(Si_{1-x}Ge_x)_2$ film was formed concurrently with Ge segregation to the underlying $Si_{0.76}Ge_{0.24}$ film.

For the $Ni/Si_{1-x-y}Ge_xC_y$ samples annealed at 0.2 J/cm^2 $Ni_2(Si_{1-x-y}Ge_xC_y)$, $Ni(Si_{1-x-y}Ge_xC_y)$, and $Ni(Si_{1-x-y}Ge_xC_y)_2$ were formed for 5, 10, and 30 pulses, respectively. The $Ni(Si_{1-x-y}Ge_xC_y)_2$ layer was very smooth and no apparent Ge segregation into the underlying $Si_{1-x-y}Ge_xC_y$ film occurred as shown in Fig. 8.

The above results reveal that upon pulsed laser annealing the addition of C to the $Si_{1-x}Ge_x$ films also retards the phase transformation. In addition, upon multiple pulse laser annealing smooth $Ni(Si_{0.76}Ge_{0.24})_2$ and $Ni(Si_{1-x-y}Ge_xC_y)_2$ films can be grown without showing Ge segregation to the unreacted $Si_{0.76}Ge_{0.24}$ films and inducing strain relaxation.

IV. SUMMARY AND CONCLUSIONS

(1) For the $Ni/Si_{0.76}Ge_{0.24}$ and $Ni/Si_{1-x-y}Ge_xC_y$ samples annealed at a temperature of 200–600 °C Ge segregation to

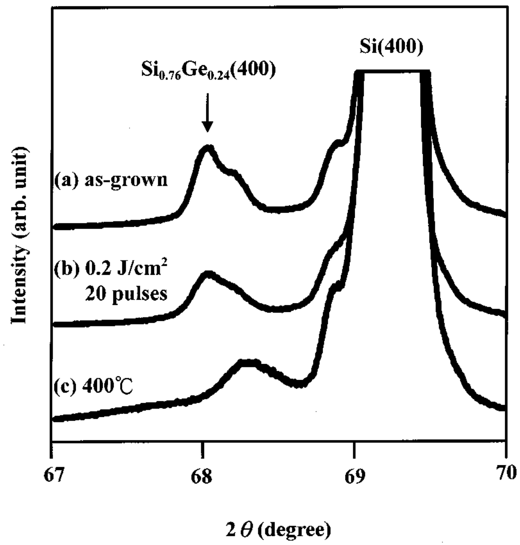


FIG. 7. XRD patterns of (a) the as-grown $Si_{0.76}Ge_{0.24}$ film, (b) the unreacted $Si_{0.76}Ge_{0.24}$ film after annealing at 0.2 J/cm^2 for 20 pulses, and (c) the unreacted $Si_{0.76}Ge_{0.24}$ film after annealing at 400 °C.

the underlying substrates occurred with the extent becoming more severe at higher annealing temperatures. For the Ni/$Si_{1-x-y}Ge_xC_y$ system the temperatures at which phase transformation and agglomeration occurred were higher in comparison with the Ni/$Si_{0.76}Ge_{0.24}$ system.

(2) For the Ni/$Si_{0.76}Ge_{0.24}$ and Ni/$Si_{1-x-y}Ge_xC_y$ samples subjected to pulsed KrF laser annealing the agglomeration structure did not occur, but the phase transformation in the latter was sluggish in comparison with that in the former. It is evident that C plays an important role on delaying phase transformation upon either vacuum annealing or pulsed KrF laser annealing.

(3) Multiple pulse laser annealing can produce smooth Ni$(Si_{0.76}Ge_{0.24})_2$ and Ni$(Si_{1-x-y}Ge_xC_y)_2$ films without inducing Ge segregation and strain relaxation of the unreacted substrates.

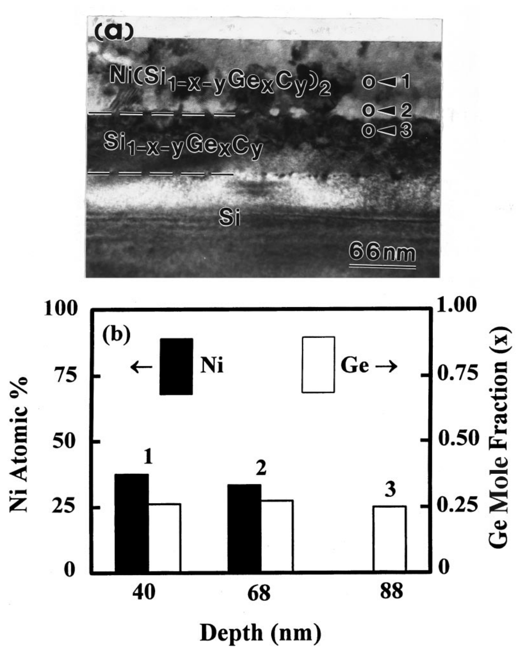


FIG. 8. (a) XTEM image and (b) the depth profiles of the chemical elements for the Ni/$Si_{1-x-y}Ge_xC_y$ sample annealed at 0.2 J/cm^2 for 30 pulses.

ACKNOWLEDGMENT

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