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Laser-Induced Melt-Mediated Ni(Pt) Germanosilicide Formation on Condensed Si_{1-x}Ge_x/Si Substrates

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Laser-induced melt-mediated Ni-based germanosilicide has been formed on condensed SiGe substrates (with 40% Ge). Pure Ni germanosilicide formation was plagued by the presence of amorphous oxygen-rich globules. Platinum alloying has greatly improved the morphology of the laser-induced Ni(Pt) germanosilicide with the alleviation of oxygen incorporation during laser annealing. This results in an improvement in the surface roughness and electrical conductivity of the germanosilicide film. Low affinity of Pt toward oxygen is believed to have resulted in this improvement.

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Utilization of SiGe either as a virtual substrate, channel stressor, or channel materials¹⁻⁶ requires Ni germanosilicide to be used as the contact material. As compared to Ni silicide, Ni germanosilicide suffers poorer morphological stability during processing at an elevated temperature.^{7,8} It is aggravated by the Ge out-diffusion as well as the presence of mechanical stress in the SiGe layer.^{7,9,10} Addition of alloying element (i.e., Pt), incorporation of buffer layer, or ion implantation has been reported beneficial in reducing these problems to some extent.¹¹⁻¹³ In addition, Pt alloying also plays a role in reducing the oxygen contamination of the silicide film during the annealing process.¹⁴ Besides the above-mentioned conventional rapid thermal annealing (RTA) process to induce silicide formation, several annealing methods like spike or laser annealing are gaining much attention.¹⁵⁻¹⁸ Recently, we have reported the possibility of forming an atomically abrupt germanosilicide/Si interface through a self-limiting melting phenomenon by utilizing laser irradiation.¹⁹ In this paper, the effect of Pt alloying during laser-induced melt-mediated Ni(Pt) germanosilicide formation on a Si_{1-x}Ge_x/Si substrate was investigated.

The SiGe substrate was fabricated through a condensation process which is basically a series of oxidation and annealing steps.²⁰ The oxidation step was performed at 900°C for 9 h with an oxygen flow rate of 7500 sccm, whereas the annealing step was performed at 950°C for 45 min with a nitrogen flow rate of 10,000 sccm. These processes were repeated twice. The final Si_{1-x}Ge_x/Si substrate has a graded Ge concentration with the highest concentration of 40% Ge at the surface. The Ge content in the SiGe layer is relatively stable (40–30%) for the first 50 nm from the surface where most of the reaction is expected to occur during silicide formation.

Prior to a deposition of 20 nm Ni or Ni_{1-u}(Pt)_u ($u = 0.1$) film, the surface of the Si_{1-x}Ge_x/Si substrate was cleaned using piranha solution (H₂SO₄:H₂O₂ = 4:1) for 10 min followed by 1 min dip in diluted HF (1:50) solution to remove the native oxide. Excimer laser irradiation (λ :248 nm; full width at half maximum of the pulse duration: 23 ns) was then carried out under a continuous purified N₂ purging. Single-pulsed laser annealing (LA) using laser fluences ranging from 0.2 to 0.6 J cm⁻² was performed to induce the formation of the germanosilicide film. Identification of phase and crystallographic orientation of the germanosilicide film formed was carried out using X-ray diffraction (XRD) with Cu K α radiation in 2 θ geometry. Cross-sectional transmission electron microscopy (XTEM) analyses using a JEM 2100F were employed to evaluate the microstructure of the germanosilicide formed. Depth profiling Auger electron spectroscopy (AES) was used to trace the elemental

distribution in the germanosilicide film. Four-point probe measurements were performed on the laser-annealed samples to monitor their sheet resistance evolution.

The XRD spectra of both Ni and Ni_{1-u}(Pt)_u, $u = 0.1$ [henceforth referred as Ni(Pt) for simplicity] on Si_{1-x}Ge_x/Si substrate after single-pulsed LA at different laser fluences are shown in Fig. 1a and b, respectively. In both samples, the Ni- or Ni(Pt)-germanosilicides

Figure 1. (Color online) XRD spectra of the (a) Ni/Si_{1-x}Ge_x and (b) Ni(Pt)/Si_{1-x}Ge_x samples after single-pulsed LA at different laser fluences ranging from 0.2 to 0.6 J cm⁻². The calculated diffraction peaks of pure NiSi and Ni₂Si around the 2 θ angle of 45–50° were added to indicate the multiple phases germanosilicide formed during the LA process.

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formation can be detected in the sample after single-pulsed LA at laser fluence as low as 0.2 J cm^{-2} . As compared to the laser fluence needed to induce the reaction in the Ni-based film on Si or $\text{Si}_{1-x}\text{Ge}_x$ ($x < 0.15$) sample, this reaction threshold is relatively low.^{19,21,22} In the Ni-based film on Si sample, silicide does not usually form during a single-pulsed LA at laser fluence lower than 0.4 J cm^{-2} .²¹ During laser irradiation of the Ni/ $\text{Si}_{1-x}\text{Ge}_x$ or Ni(Pt)/ $\text{Si}_{1-x}\text{Ge}_x$ sample, most of the laser energy would have been absorbed in the top 10 nm of the metal film (approximated using the Lambert–Beer equation). The absorbed laser energy will then be converted into thermal energy, which travels further down to the substrate through the conduction mechanism (heat lost due to radiation and/or convection processes is negligible). It is known that $\text{Si}_{1-x}\text{Ge}_x$ has a melting temperature lower than pure Si.²³ Furthermore, the thermal conductivity of the $\text{Si}_{1-x}\text{Ge}_x$ ($x = 0.4$) alloy is approximately 13 times poorer than that in the pure Si.²⁴ Due to its poor thermal conductivity, the presence of a $\text{Si}_{1-x}\text{Ge}_x$ layer underneath the Ni layer will inhibit the conduction of heat and hence, there will be a buildup of thermal energy at the surface area of the sample, creating a thermal confinement effect. This results in a generation of higher temperature in the $\text{Si}_{1-x}\text{Ge}_x/\text{Si}$ sample as compared to the pure-Si-based sample during LA at the same laser fluence. This explains a much lower reaction threshold observed in the $\text{Si}_{1-x}\text{Ge}_x/\text{Si}$ sample with $x \approx 0.4$.

In general, the XRD spectra of the Ni(Pt)/ $\text{Si}_{1-x}\text{Ge}_x$ samples depicted in Fig. 1b exhibit very similar diffraction peaks with that of the Ni/ $\text{Si}_{1-x}\text{Ge}_x$ sample. A similar germanosilicide formation process should have taken place in the Ni(Pt)/ $\text{Si}_{1-x}\text{Ge}_x$ sample. It is apparent that after a single-pulsed LA at $0.2\text{--}0.4 \text{ J cm}^{-2}$, a broad and strong diffraction peak can be observed in the XRD spectra of Ni/ $\text{Si}_{1-x}\text{Ge}_x$ in Fig. 1a. The presence of the broad peak indicates the formation of multiple germanosilicide phases (Ni-rich germanosilicide and Ni mono-germanosilicide have several diffraction peaks within the range of the broad peak) and/or small crystallites in the sample. Multiple phase formation is expected during a single-pulsed LA process due to fast melting and solidification processes such that the elemental mixing cannot be fully completed. A narrower diffraction peak at a 2θ angle of 54.56° suggests the formation of a highly textured germanosilicide phase with a big grain size and was attributed to the $\text{Ni}(\text{Si}_{1-y}\text{Ge}_y)$ phase. The simultaneous formation of both polycrystalline and textured grain in one sample is intriguing. This will be elaborated in the next section with the analysis of the XTEM micrograph of the sample.

A more uniform $\text{Ni}(\text{Si}_{1-y}\text{Ge}_y)$ formation can be achieved during LA at higher laser fluence (i.e., more than 0.3 but less than 0.5 J cm^{-2}) due to a slightly deeper and longer melting process. Increasing the laser fluence to 0.5 J cm^{-2} , however, has resulted in the formation of $\text{Ni}(\text{Si}_{1-z}\text{Ge}_z)_2$ phase at an expense of $\text{Ni}(\text{Si}_{1-y}\text{Ge}_y)$ phase. Deeper melting is attributed to the formation of the Si-rich phase. The position of the $\text{Ni}(\text{Si}_{1-z}\text{Ge}_z)_2$ peak detected is very similar to the pure NiSi_2 , which indicates that most of the Ge atoms were lost from the germanosilicide during LA. The Ge loss might be caused by a high temperature generated in the sample during annealing at high laser fluence. After LA at a higher laser fluence of 0.6 J cm^{-2} , the crystal size of the NiSi_2 phase became so huge (judging from the full width half maximum of the XRD peak of the NiSi_2) and it appears to be highly textured with the substrate.

The transmission electron microscopy (TEM) micrographs of the Ni/ $\text{Si}_{1-x}\text{Ge}_x$ sample after a single-pulsed LA at 0.2 J cm^{-2} is shown in Fig. 2a. The Ni germanosilicide film exhibits a dual layered structure with a thin amorphous layer sandwiched in between. The bottom layer has a better crystalline quality in which a large continuous crystal with a very flat Ni germanosilicide/Si interface could be observed. The top layer is polycrystalline in nature with a presence of discontinued amorphous globules inside it. The polycrystalline top layer was detected as a broad peak in the XRD spectra, whereas the highly textured bottom layer resulted in the appearance of a sharp diffraction peak at around 2θ angle of 54.56° . The orientation

Figure 2. (a) XTEM micrograph of the Ni/ $\text{Si}_{1-x}\text{Ge}_x$ sample after single-pulsed LA at 0.2 J cm^{-2} . The inset shows a high-resolution TEM (HRTEM) of the interface region. (b) XTEM micrograph of the Ni(Pt)/ $\text{Si}_{1-x}\text{Ge}_x$ sample after single-pulsed LA at 0.2 J cm^{-2} . Inset I shows a HRTEM of the interface region, and inset II is the SAED of the Ni(Pt) germanosilicide film, which exhibits a single-crystal-like diffraction pattern. Notice the absence of amorphous globules in the sample with Pt alloying.

relationship between the germanosilicide and substrate determined by TEM selected area electron diffraction (SAED) pattern is $\text{NiSiGe}[010]\|\text{SiGe}[110]$ and $\text{NiSiGe}(100)\|\text{SiGe}(001)$. The AES depth profiling analysis of the Ni/ $\text{Si}_{1-x}\text{Ge}_x$ sample after LA at 0.2 J cm^{-2} in Fig. 3a revealed that the top layer has a considerable amount of oxygen, especially in the first 10 nm from the surface where the oxygen content reached up to 10 atomic %. The oxygen signal peaked up at the region where the interface layer was present but diminished in the lower layer. The formation of this multilayered structure can be explained as follows.

Enhanced by the thermal confinement effect due to the presence of $\text{Si}_{1-x}\text{Ge}_x$ substrate, a single-pulsed LA at 0.2 J cm^{-2} was able to cause melting of both the $\text{Si}_{1-x}\text{Ge}_x$ layer and the Ni layer. Because the annealing was not performed in a high-vacuum condition, oxygen incorporation inevitably occurred. It was verified by AES (not shown) that the condensed $\text{Si}_{1-x}\text{Ge}_x$ substrate was not the primary

Figure 3. (Color online) AES depth profiling of (a) the $\text{Ni}/\text{Si}_{1-x}\text{Ge}_x$ and (b) $\text{Ni}(\text{Pt})/\text{Si}_{1-x}\text{Ge}_x$ sample after single-pulsed LA at 0.2 J cm^{-2} . The depth of the AES profile was approximated from the sputtering rate of a SiO_2 film.

source of the oxygen found in the germanosilicide film. Oxygen atoms were able to travel throughout the whole thickness of the sample due to its high diffusivity in a molten Ni-Si-Ge phase. During solidification, the $\text{Ni}(\text{Si}_{1-y}\text{Ge}_y)$ and not $\text{Ni}(\text{Si}_{1-z}\text{Ge}_z)_2$ was preferably formed from the molten $\text{Ni-Si-Ge}/\text{Si}_{1-x}\text{Ge}_x$ interface because of two reasons. First, at low laser fluence at 0.2 J cm^{-2} the melting of the $\text{Si}_{1-x}\text{Ge}_x$ substrate is limited only to the top layer where Ge content is high; thus, the $\text{Ni}(\text{Si}_{1-z}\text{Ge}_z)_2$ has a less probability to form. Second, the presence of Ge inhibits the formation of NiSi_2 -based phase because there is no equilibrium NiGe_2 phase available. Solubility of oxygen inside the $\text{Ni}(\text{Si}_{1-y}\text{Ge}_y)$ is very low thus during the solidification of $\text{Ni}(\text{Si}_{1-y}\text{Ge}_y)$ phase; the oxygen atoms were expelled out from the growing $\text{Ni}(\text{Si}_{1-y}\text{Ge}_y)$ layer to the remaining Ni-rich molten Ni-Si-Ge layer on the top. The solidification velocity was faster than the rate of oxygen diffusion in the remaining Ni-rich molten Ni-Si-Ge , thus resulting in a buildup of oxygen concentration at the $\text{Ni}(\text{Si}_{1-y}\text{Ge}_y)$ solidification front. Initially this oxygen-rich layer was relatively thin and did not disrupt the supply of the Ni-Si-Ge atoms to the already-nucleated and grown $\text{Ni}(\text{Si}_{1-y}\text{Ge}_y)$ layer. However, as the oxygen snow-ploughing continues, the oxygen-rich layer became considerably thick that it blocked the supply of Ni-Si-Ge atoms. Because there was no template available for the subsequent $\text{Ni}(\text{Si}_{1-y}\text{Ge}_y)$ to grow, new grains randomly nucleated adjacent to the oxygen-rich interfacial layer, which resulted in the formation of a polycrystalline film. The grain boundary rich nature of polycrystalline film facilitated the rejection of oxygen atoms during the formation of polycrystalline Ni-rich germanosilicide. The most probable site for these rejected oxygen atoms was the triple-grain-boundary sites, which resulted in a for-

Figure 4. (Color online) Sheet resistances of both $\text{Ni}/\text{Si}_{1-x}\text{Ge}_x$ (■) and $\text{Ni}(\text{Pt})/\text{Si}_{1-x}\text{Ge}_x$ (▲) after single-pulsed LA at different laser fluence ranging from 0.2 to 0.6 J cm^{-2} .

mation of germanosilicate compound (Ni-Si-Ge-O) in the form of an amorphous pocket. In addition, a faster solidification rate experienced by the top Ni-rich molten Ni-Si-Ge layer due to the larger undercooling experienced by this layer also resulted in the formation of a polycrystalline layer with a small grain size.

Alloying the Ni film with 10 atomic % Pt significantly reduced the problem with oxygen incorporation in the molten Ni-Si-Ge film. The AES depth profiling $\text{Ni}(\text{Pt})/\text{Si}_{1-x}\text{Ge}_x$ sample after single-pulsed LA at 0.2 J cm^{-2} in Fig. 3b no longer detected the presence of oxygen in the top Ni(Pt)-rich germanosilicide layer. The XTEM micrograph of the sample shown in Fig. 2b reveals almost no formation of an interfacial layer, indicating that the oxygen contamination was tremendously reduced in the molten film with Pt alloying. Although the diffusivity of an element increases significantly (up to 5–8 orders in magnitude higher) in molten form, Pt atoms which have slower diffusivity than the Ni were expected to be accumulated near the surface area. Due to a low reactivity between Pt and oxygen, the presence of Pt atoms during the melting process resulted in a reduction of the oxygen diffusion into the molten Ni-Pt-Si-Ge layer, which resulted in less oxygen contamination of the $\text{Ni}(\text{Pt}) \times (\text{Si}_{1-y}\text{Ge}_y)$ layer. The same orientation relationship of $\text{Ni}(\text{Pt})\text{SiGe}[010]||\text{SiGe}[110]$ and $\text{Ni}(\text{Pt})\text{SiGe}(100)||\text{SiGe}(001)$ was also observed in the Ni(Pt) sample. Our previous work using 5% Pt sufficiently avoided oxygen contamination on the 15% SiGe substrates.¹⁹

To further investigate the effect of oxygen alleviation toward the quality of the germanosilicide film, the surface roughness of the sample was evaluated using atomic force microscopy. It was found that after single-pulsed LA at 0.2 J cm^{-2} , the surface roughness of the $\text{Ni}(\text{Pt})/\text{Si}_{1-x}\text{Ge}_x$ sample was approximately 3 times smoother than that in the $\text{Ni}(\text{Si}_{1-y}\text{Ge}_y)$ (2.68 vs 0.919 nm). Surface degradation experienced by the pure-Ni sample arises from the presence of amorphous globules inside the top Ni-rich germanosilicide layer. Removal of these amorphous globules in the sample with Pt alloying led to a significant reduction of its surface roughness. The improved germanosilicide quality can also be observed in the sheet resistance result of the sample with Pt alloying as plotted in Fig. 4. The overall trend of the sheet resistance measurement of the sample with Pt alloying exhibits a downward trend which can be attributed to the formation of amorphous-globule-free $\text{Ni}(\text{Pt})$ germanosilicide film after single-pulsed LA at 0.2 and 0.3 J cm^{-2} , a thicker $\text{Ni}(\text{Pt}) \times (\text{Si}_{1-y}\text{Ge}_y)$ after 0.4 J cm^{-2} , and thick $[\text{Ni}(\text{Pt})]\text{Si}_2$ after $<0.5 \text{ J cm}^{-2}$ LA. The sheet resistance of a pure Ni sample was slightly increased after single-pulsed LA at 0.2 and 0.3 J cm^{-2} , which indicates the degradation of Ni germanosilicide film due to

the presence of amorphous globules. Finally, at higher fluence, NiSi₂ was formed and brought down the sheet resistance of the sample. As a comparison, the sheet resistance of the same samples after the RTA process was also measured. The sample without and with Pt alloying reached its lowest sheet resistance of 5.4 and 6.8 $\Omega/\square$, respectively. However, although the Pt alloying is able to significantly reduce the formation of amorphous pockets, it is believed that some smaller pockets remain in the film. These micropockets act as a scattering center for the electron flow, which results in a higher resistivity of the germanosilicide film.

The presence of a Si_{1-x}Ge_x layer with high Ge content resulted in a thermal confinement effect which caused a much lower reaction threshold as compared to Ni/Si sample. Due to a melt-phase-reaction process, the oxygen contamination is significant, which manifests itself in the formation of a thin interfacial germanosilicate layer as well as amorphous pockets on the top germanosilicide surface. Due to its low affinity to oxygen, Pt alloying alleviates the oxygen incorporation in the Ni germanosilicide. This significantly reduces the amorphous pocket formation which results in a smoother film surface and an improvement in the electrical conductivity of the Ni(Pt) germanosilicide.

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