Sputter Epitaxial Growth of Flat Germanium Film with Low Threading-Dislocation Density on Silicon (001)

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Fast epitaxy of Ge on Si(001) was realized by DC sputtering at 2.1 nm·s⁻¹ and 360°C; the resulting film was optically flat without a cross-hatch structure. After annealing at 700°C, 90°-full-edge dislocations dominated the Ge–Si interface and the threading-dislocation density (TDD) of the Ge film was below 10⁶ cm⁻², which is three orders of magnitude lower than the value of Ge films prepared by other methods. The extremely low TDD might be attributable to the spaces vacated by desorbed Ar within the film that served as dislocation sinks during sputtering. Acceptor-band conduction, which was at 0.02 eV above the valence band and was induced by dislocations, was observed. The absolute mobility, which was 0.02 eV above the valence band, was 6.2 cm²·N⁻¹·s⁻¹ at 300 K. After annealing at 700°C, the ionized-defect scattering in the film was considerably decreased and a mobility of 1180 cm²·V⁻¹·s⁻¹ was obtained. The direct band gap energy of the film prepared without annealing was 0.81 eV, and became 0.79 eV after annealing.

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Manuscript submitted June 17, 2014; revised manuscript received July 21, 2014. Published August 1, 2014.

Germanium films on Si substrates have attracted considerable interest because of their important roles in optoelectronic devices and high-speed transistors. For solar cells (SC) in particular, their optical responsivity can be expanded to ∼1500 nm by using Ge as the light-absorbing material. Owing to Ge’s low absorption coefficient at near-infrared wavelength, however, light-absorbing layers with a total thickness of several micrometers are necessary, which require the Ge films to be grown on Si at high speed. So far, several growth techniques such as chemical vapor deposition (CVD), vacuum evaporation (VE), and sputter epitaxy (SE) have been applied to Ge epitaxy on Si. Among these techniques, SE takes advantage of surface bombardment by low-energy particles, which enables epitaxy at low temperature, at high growth rate, and with high critical thickness. By applying DC power for the sputtering process, 10-μm-thick Si have been grown on Si(001) at a rate of 3.3 nm·s⁻¹ and under high-vacuum back pressure. Furthermore, the potential of SE in SC fabrication has been demonstrated by the recent production of a single-crystalline-Si solar cell with an n-type emitter, whose internal quantum efficiency is as high as 90%.

The main challenge of Ge epitaxy on a Si substrate is the 4.2% lattice mismatch, which can cause a cross-hatch pattern resulting from the Stranski–Krastanov (SK) growth mode. Using a compositionally graded buffer layer, in which the lattice mismatch strain is relieved by a fractional Ge content of 0 to 1, is effective for growth in the Frank–van der Merwe (FM) mode. However, the buffer layer thickness can exceed several micrometers, which results in an unavailing light absorption in the buffer layer for solar cells and an elongated deposition time. Another approach is to use a thin SiC or GeC buffer layer, which can be epitaxially grown on Si, and Ge is grown on this buffer layer. Additionally, low-temperature and ultra-high vacuum chemical vapor deposition (UHVCVD) is another method for growing Ge in the FM mode. In this approach, when the temperature is below 350°C, Ge grows in the FM mode because the low surface diffusivity of Ge kinetically suppresses the formation of Ge islands. As the growth rate is extremely low (∼0.007 nm·s⁻¹) at low temperature, a so-called low-temperature/high-temperature (LT/HT) two-step growth technique has been developed: once a thin and flat Ge layer is formed at LT, Ge grows faster on the thin Ge template at HT. On the other hand, the growth rate of Si in SE method can be as high as 3.3 nm·s⁻¹ at temperatures below 360°C and the FM mode growth of Ge on Si with a corresponding high growth rate is thus expected.

In this study, flat Ge films were grown at 2.1 nm·s⁻¹ and 360°C on Si(001) by SE for the first time. The drastically improved crystalline quality as well as optical and electrical characteristics by post-growth thermal annealing will be demonstrated here.

Experimental

The Si(001) wafer was cleaned with 40-ppm ozonized deionized (DI) water for 30 min, treated in a 1% HF solution to remove surface oxides, and then rinsed in DI water. It was then loaded into the sputtering chamber and heated to 360°C, and the temperature of the wafer was kept below 360°C during sputtering. No in situ cleaning was performed before epitaxy. The base pressure was ∼5 × 10⁻² Torr, and 99.9999% Ar was introduced into the chamber under a pressure of 2.5 mTorr during sputtering. The sputtering target was a non-doped Ge wafer with a resistivity of 40-Ω·cm. The film was evaluated using reflectance high-energy electron diffraction (RHEED), transmission electron microscopy (TEM), X-ray diffraction (XRD), Expert, Philips, The Netherlands, Raman scattering spectroscopy (hereafter referred to as simply Raman; System 1000, Renishaw, UK), van der Pauw Hall measurements, and visible–infrared light spectroscopy (U4000, Hitachi, Japan). A Si wafer with a resistivity of 5000–10000 Ω·cm was used as the substrate for the Hall measurements.

Results and Discussion

The initial experimental results showed that the Ge growth rate was 0.19 nm·s⁻¹ at a plasma power P of 10 W and increased linearly to 2.1 nm·s⁻¹ at P of 100 W. For the remaining experiments, P was fixed at 100 W. Figure 1a shows a scanning-electron-microscope (SEM) image of a 1000-nm-thick Ge film, with a RHEED image at the [001] azimuthal angle in the inset that shows the single-crystalline film; Figure 2a shows a cross-sectional TEM image of the film. The film surface was flat and no cross-hatch structure was observed.

Figure 3a shows the Raman spectra of the as-deposited Ge films whose thickness d ranged from 10 to 10000 nm; the spectrum of a Ge wafer is also shown for reference. A sharp and symmetric Raman shift as observed at ∼301 cm⁻¹ corresponding to crystalline Ge, without the shoulder at lower wavenumbers, indicates that the thickness of the Ge crystalline phase was absent. In the films with d ≤ 50 nm, a phonon peak at 520 cm⁻¹ from the underlying Si substrate appeared. In inset of Figure 3a, it can be seen that the peak intensity depends on d. If Ge was grown on Si in the island mode, the signal from Si would have been proportional to the Ge coverage ratio and it would have decreased linearly with increasing d. On the other hand, the experimental data shown in Fig. 3a can be fitted by a solid line of exp(−2αd), where α is the absorption coefficient of Ge (5 × 10⁴ cm⁻¹) at the Raman laser wavelength (514.5 nm) and 2α is the sum of incident light path and escaping light path through the Ge film. This non-linear relationship indicates
that Ge was optically flat. The Ge–Si interface was determined to be abrupt since there are no peaks corresponding to Si–Ge bonds in the spectrum at 400 cm\(^{-1}\).

The full width at half maximum (FWHM) of the Raman shift \(\Delta \omega\) at 301 cm\(^{-1}\), \(W_R\), is shown in Fig. 3b to be dependent on \(d\). The value of \(W_R\) for a Ge wafer is indicated by the red tick mark on the y-axis. The value of \(W_R\) for the Ge film was 8.4 cm\(^{-1}\) at \(d = 10\) nm; it decreased sharply with increasing \(d\) for 10 nm < \(d\) < 50 nm because of phonon confinement, \(^{25-27}\) whereas the decrease became more gradual for \(d > 50\) nm because of improving crystallinity. \(^{25-27}\) At \(d > 100\) nm, \(W_R\) was asymptotically close to that of bulk Ge (4.05 cm\(^{-1}\)). Figure 3c shows the Raman shift \(\Delta \omega\) in relation to \(d\). The in-plane strain \(e\) was calculated from the equation \(e [\%] = 0.25(300.9 – \Delta \omega)\), and it is shown on the right axis of Fig. 3c. \(^{28}\) The laser wavelength of the Raman spectrometer was 514.5 nm, so the data corresponds to film properties at a depth of 50 nm from the surface. A schematic of the in-plane strain in the Ge film is shown in the inset of Fig. 3c. The in-plane strain was –0.24% (compressed) at the initial growth stage, and it relaxed with increasing \(d\). As can be seen in Fig. 2a, there was a strain field and dislocation cluster around the Ge–Si interface. As the substantial lattice mismatch between Ge and Si was accommodated by the dislocation layer, the strain in the overlying Ge layer was relaxed, which yielded better crystalline quality. The strain was ∼0% at \(d = 5000\) nm and turned to 0.07% tensile strain at \(d = 10000\) nm. This transition was thermally induced in the Ge epilayer during cooling after growth owing to the larger thermal expansion of Ge.

Figure 4 shows the \(d\)-dependence of the Hall hole concentration \(p\) (in log scale) and mobility \(\mu\) (in linear scale). Even though the sputtering target was non-doped Ge, the SE-Ge film was p-type owing to structural defects \(^{29}\) such as dislocations. The values of \(p\) and \(\mu\) improved with increasing \(d\) because of the improved crystalline quality,
as have been shown in Fig. 3b. This tendency was the same as that observed in CVD and VE films. The 1000-nm-thick as-deposited SE-Ge film had a $p$ value of 4 to $7 \times 10^{16}$ cm$^{-3}$, which is comparable to that of VE-Ge and two orders of magnitude lower than that of CVD-Ge at a similar deposition temperature. The SE-Ge film had $\mu$ values in the range of 200–300 cm$^2$·V$^{-1}$·s$^{-1}$, which is comparable to that of CVD-Ge but lower than that of VE-Ge. The electrical properties were drastically improved after thermal annealing, as will be shown below.

Various SE-Ge films were annealed at different temperatures ($T_A$) between 300 and 900°C for 30 min under a forming gas (FG: 3.8% H$_2$ in N$_2$) atmosphere at one atm. From this point forward, $d$ was fixed at 1000 nm unless otherwise noted. Figure 2b, 2c, and 2d show cross-sectional TEM images of annealed Ge films with $T_A$ of 600, 700, and 900°C, respectively. The dislocations and strain field decreased considerably with increasing $T_A$; at 800°C, only misfit dislocations at the Ge–Si interface were observed, as shown in the cross-sectional [110] HR-TEM image in Fig. 2e. Burgers circuits were drawn around the misfit dislocations, as shown by the dashed rectangles in Fig. 2e. Full-edge dislocations with the Burger’s vector inclined at 90° dominated, which accounts for the Ge film’s flatness and the observation of few dislocations. The Ge films prepared with and without annealing were treated in a Dash etchant (CH$_3$COOH:HNO$_3$:HF:I$_2$ = 40 mL:10 mL:54 mg) for 30 s to remove surface defects. Subsequent SEM observations revealed etch pits, whose areal density has been shown to be correlated to the threading-dislocation density (TDD) measured in plan-view TEM images. As the etching rate was ∼20 nm·s$^{-1}$ for the as-deposited Ge film, a Ge film with a thickness of ∼400 nm remained on Si. Figure 1b and 1c show plan-view SEM images of the as-deposited film and the film that was annealed at 700°C, respectively, with high-magnification images in the insets. The TDD, calculated as the etch-pit density, was $5 \times 10^6$ cm$^{-2}$ in the as-deposited film, but it was reduced to $\leq 1 \times 10^5$ cm$^{-2}$ after annealing at 700°C. These values will be discussed later in a comparison of SE-Ge films with Ge films produced by VE and CVD.

Figure 5 shows XRD $\omega$ scans featuring the Ge(004) peak and coupled $\omega$–2$\theta$ scans of films annealed at different values of $T_A$. The crystallinity was evaluated from the FWHM of the $\omega$ scans, and the in-plane strain was estimated from the peak positions of the $\omega$–2$\theta$ scans. The estimated values of the in-plane strain are shown in Fig. 6a as a function of $T_A$, together with the strain estimated from the Raman shift in Fig. 3c. The error bars reflect the spread of the $\omega$–2$\theta$ scans. It can be seen that the films without annealing and annealed at $T_A \leq 400°C$ were compressively strained. The strain was relaxed toward the upper surface of the film, and because of the broad distribution of strains, the error bars were large for films with $T_A \leq 400°C$. As $T_A$ increased, the strain changed from negative values toward zero and became tensile at $T_A \geq 600°C$. This tensile strain was induced after high-temperature annealing because of the difference between the thermal expansion of Si and Ge and the release of adsorbed gases within the film. The strains estimated from the Raman shift were lower than those from the $\omega$–2$\theta$ scans because the Raman data only contained information on the film surface at which the strain was relaxed.

Figure 6b shows the FWHM of the Ge(004) peak in XRD $\omega$ scans, $W_w$, and FWHM of the Raman shift, $W_R$, as functions of $T_A$. Both $W_w$ and $W_R$ displayed the same trends in dependence on $T_A$, but $W_w$ exhibited smaller fluctuations. The value of $W_w$ was $2000$ arcsec in the as-deposited film, and it started to decrease at $T_A = 460°C$ and reached $252$ arcsec at $T_A = 800°C$. As $W_w$ reflects the fluctuations in measured values of a lattice constant, a comparison with $W_w$ obtained for Ge films deposited on Si by other methods would reveal interesting information on sputtering epitaxy of Ge. For Ge films deposited at a temperature of ∼360°C via VE, $W_w$ is in the range of 660–2520 arcsec. When higher deposition temperatures or high-temperature post-deposition annealing is used, $W_w$ is 240–490 arcsec for VE-Ge$^{11,12}$ and 30–100 arcsec for CVD-Ge.$^{9}$ These values show that the lattice-constant fluctuations of the SE-Ge films are similar to those of VE-Ge films, but worse than those of CVD-Ge films. The larger lattice-constant fluctuations can be attributed to the higher deposition rate (∼2 nm·s$^{-1}$) and lower deposition temperature (340–440°C) of SE and VE compared to CVD (0.5 nm·s$^{-1}$; 750°C). On the other hand, after our SE-Ge films were annealed at 700°C, the value of TDD was $< 10^4$ cm$^{-2}$, which is more than three orders of magnitude lower than TDD of VE-Ge or CVD-Ge films$^{5,9-11}$ despite comparable lattice-constant fluctuations of the SE-Ge and VE-Ge films. It has been reported that dislocations will glide toward the sidewall of a mesa-structured Ge film and disappear after thermal annealing.$^8$ Furthermore, Ar sputtering gas has been known to be adsorbed by the deposited film during sputter deposition. Therefore, the spaces vacated by desorbed Ar within the Ge film would have served as dislocation sinks during thermal annealing.

The Hall hole concentration $p$ (Fig. 6c) and mobility $\mu$ (Fig. 6d) were $5 \times 10^{16}$ cm$^{-3}$ and 300 cm$^2$·V$^{-1}$·s$^{-1}$, respectively, for as-deposited film and films annealed at temperatures below 350°C, but they declined abruptly as $T_A$ was increased from 400 to 440°C. The declining values should have resulted from the generation of dangling bonds because of the desorption of Ar. With further increase in $T_A$ from 500 to 600°C, $p$ and $\mu$ improved sharply; at $T_A = 700°C$, $p$ and $\mu$ reached $1.3 \times 10^{16}$ cm$^{-3}$ and 1180 cm$^2$·V$^{-1}$·s$^{-1}$, respectively. This improvement can be attributed to the removal of dislocations and decreased lattice-constant fluctuations, as have shown in Fig. 2a–2d and Fig. 6b. A further increase in $T_A$ to 900°C resulted in roughening of the film surface, presumably owing to alloying between Ge and Si, and a decrease in $\mu$. 
The temperature dependence of the Hall mobility and carrier concentration were investigated to understand the scattering of carriers (holes) before and after thermal annealing. Figure 7 shows the Hall mobility and hole concentration of Ge films with and without thermal annealing at 700 °C (973 K) at a range of film temperatures T. In the Ge film prepared without annealing, the mobility $\mu$ increased with decreasing $T$ as a function of $T^{-3.5}$ for 370 K > $T$ > 300 K owing to acoustic and optical-phonon scattering of holes. The value of $\mu$ decreased with decreasing $T$ as a function of $T^{-5}$ for 300 K > $T$ > 190 K because of ionized-defect scattering. Even at 300 K, $\mu$ increased by ion scattering, indicating a high concentration of ionized defects in the Ge film. The hole concentration $n_{\text{PA}}$ decreased over this entire range as $T$ decreased from 370 to 190 K. The activation energy also decreased from 0.19 to 0.02 eV between 370 and 240 K and remained at 0.02 eV as $T$ was further decreased from 240 to 190 K. As $T$ decreased from 190 to 100 K, $n_{\text{PA}}$ continued to decrease and dropped to the vicinity 10 cm$^{-2}$·s$^{-1}$, while $n_{\text{PA}}$ increased again to $2 \times 10^{15}$ cm$^{-2}$ at $T$ < 100 K (which can be explained by carrier conduction in the acceptor band) as schematically illustrated in the inset of Fig. 7. The acceptor band, which was located 0.02 eV above the valence band $E_v$, was introduced by defects since the Ge target, and thus the Ge film, was not doped. Therefore, $\mu_{\text{PA}} = 2 \times 10^{16}$ cm$^{-3}$ and $\mu_{\text{PA}} = 3–10$ cm$^{-2}$·s$^{-1}$ at $T$ < 100 K correspond to the hole concentration and hole mobility, respectively, in the defect-induced acceptor band.

In the Ge film that was annealed at 700 °C (973 K), the mobility $\mu_{700}$ increased with decreasing $T$ as a function of $T^{-1.5}$ for 300 K > $T$ > 80 K because of acoustic phonon scattering, and decreased with decreasing $T$ as a function of $T^{-5}$ for $T$ < 80 K because of ionized-defect scattering. The temperature at which $\mu_{700}$ reached a maximum shifted to 80 K from 300 K for the maximum $\mu_{\text{PA}}$, because of a considerably reduced ionized-defect scattering. Besides, the defect-induced acceptor band annihilated since $\mu_{P_{700}}$ didn’t increased again with decreasing $T$ for 300 K > $T$ > 20 K. The value of $\mu_{P_{700}}$ have a constant activation energy of ~0.02 eV. From the above results, the schematic image of acceptor states within band gap after annealing are shown in the inset of Fig. 7. The defect-induced acceptor states were discretely-distributed at ~0.02 eV above the valence band and have no longer form acceptor band. From cross-sectional TEM image in Fig. 2a–2d, these defect-induced acceptor band and states were originated from dislocations, and so they decreased considerably after annealing.

Finally, the absorption coefficient $\alpha$ of the SE-Ge film on Si was estimated. It was obtained by measuring the top-surface reflectivity $R_{\text{GeSi}}$, bottom-surface reflectivity $R_{\text{SiGe}}$, transmissivity of the Ge/Si structure $T_{\text{GeSi}}$, and reflectivity of the Si wafer $R_{\text{Si}}$, as shown in the inset of Fig. 8a. A high film thickness $d$ of 5000 nm was used to ensure high accuracy of $\alpha$. Figure 8a and 8b show the $\alpha$–$h\nu$ and $h^2\alpha^2$–$h\nu$ relations, respectively, of 5000-nm-thick Ge films with and without annealing, where $h\nu$ is the photon energy, $h$ is the Planck’s constant, and $\nu$ is the photon’s frequency. The value of $\alpha$ was ~5000 cm$^{-1}$ at 0.83 eV for Ge film with annealing, means a spectral response of the Ge film can be expanded to 1500 nm by using 2 μm-thick film even without light trapping technique. In Fig. 8a, the slope of the exponential edge ($0.8 \text{ eV} > h\nu > 0.75 \text{ eV}$) increased and the sub-gap absorption tail ($h\nu < 0.75 \text{ eV}$) decreased with increasing $T_A$, which are attributed to the improved crystal periodicity and reduced interband defect states, respectively. From Fig. 8b, the direct band-gap energy $E_g$ of Ge was estimated to be 0.81 eV before annealing and 0.79 eV after annealing. The value of $E_F$ was reduced by annealing because the strain in Ge changed from being compressive to tensile after thermal annealing.

Acknowledgments
We thank Prof. Y. Kajikawa at Shimane University and Prof. Y. S. Huang at National Taiwan University of Science and Technology for helpful discussions. This work received financial support from the Japan Science and Technology Agency (JST) for the ALCA project.

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