Epitaxial Growth and Crystalline Properties of $\text{Ge}_{1-x-y}\text{Si}_x\text{Sn}_y$ on Ge(001) Substrates

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Abstract

We have investigated the influence of tensile and compressive strain on the crystalline structures of $\text{Ge}_{1-x-y}\text{Si}_x\text{Sn}_y$ epitaxial layers. The tensile strain in $\text{Ge}_{1-x-y}\text{Si}_x\text{Sn}_y$ induces a non-uniform crystallinity of (220) lattice planes and surface roughening despite the strain magnitude is as small as 0.20%. In contrast, the unstrained or compressive strained $\text{Ge}_{1-x-y}\text{Si}_x\text{Sn}_y$ layer exhibits a flat and uniform surface and high crystallinity. We found that the control of [the sign of the strain](#) is an important factor to obtain a high quality $\text{Ge}_{1-x-y}\text{Si}_x\text{Sn}_y$ layer. Furthermore, substitutional Sn atoms in $\text{Ge}_{1-x-y}\text{Si}_x\text{Sn}_y$ epitaxial layer with a Sn content of 10% are thermally stable for annealing at 500°C.

Keywords

Germanium, Tin, Silicon, Epitaxial growth, Strain

1. Introduction

Recently, the monolithic integration of optoelectronic devices, such as photodetectors and lasers, on Si large-scale integrated circuits has attracted much attention [1,2]. For realizing such device applications using group-IV semiconductors, $\text{Ge}_{1-x}\text{Sn}_x$ alloy has been regarded as an important material because the direct transition $\text{Ge}_{1-x}\text{Sn}_x$ semiconductor is promised with a Sn content higher than 10% [3]. Here, $\text{Ge}_{1-x-y}\text{Si}_x\text{Sn}_y$ ternary alloy is also an attractive material because the energy band structure of $\text{Ge}_{1-x-y}\text{Si}_x\text{Sn}_y$ can be designed independently on the lattice constant by controlling the contents of Ge, Si, and Sn as well as III-V compound semiconductors [2,4]. The energy bandgap of $\text{Ge}_{1-x-y}\text{Si}_x\text{Sn}_y$ can be controlled up to 1.0 eV under the conditions of Sn content of 15% and lattice matching to Ge. For example, considering laser application, the Sn content higher than 6% in $\text{Ge}_{1-x-y}\text{Si}_x\text{Sn}_y$ alloy is required to form a quantum well structure for $\text{Ge}_{1-x}\text{Sn}_x$ [2].

It is generally known that the thermal equilibrium solubility of Sn in bulk Ge and Si are as small as 1.0 at.% and 0.1 at.%, respectively [5,6]. This fact means that the formation of $\text{Ge}_{1-x}\text{Sn}_x$ or $\text{Ge}_{1-x-y}\text{Si}_x\text{Sn}_y$ with a high Sn content is difficult under the thermal equilibrium condition, and Sn precipitation from the alloys could occur during the crystal growth and post deposition annealing (PDA). On the other hand, we previously reported that a low temperature molecular beam epitaxy (MBE) is an effective technique to form a high-Sn-content $\text{Ge}_{1-x}\text{Sn}_x$ layer [7,8]. Furthermore, we clarified that the strain in the layer induces Sn precipitation to reduce the strain energy [9]. Recently, we also examined the MBE growth of $\text{Ge}_{1-x-y}\text{Si}_x\text{Sn}_y$ epitaxial layer with a high Sn content, whose lattice constant was matching to Ge substrate for minimizing the misfit strain between the epitaxial layer and substrate [10]. We found that $\text{Ge}_{1-x-y}\text{Si}_x\text{Sn}_y$ epitaxial layer can be grown without Sn precipitation for a wide Sn content range up to 8% [10]. In addition, the small strain in $\text{Ge}_{1-x-y}\text{Si}_x\text{Sn}_y$ layer leads to a flat surface and a small full width at half maximum in X-ray diffraction (XRD) omega rocking curve [10].

On the other hand, it is generally known that the content and strain influence on the epitaxial growth. For example, $\text{Si}_{1-x}\text{Ge}_x$ epitaxial layer under the compressive strain condition exhibits rough surface than that under tensile strain [11]. However, the influences of the content and strain on the crystalline quality and surface morphology of $\text{Ge}_{1-x-y}\text{Si}_x\text{Sn}_y$ epitaxial layer has not yet been understood in detail. Furthermore, we previously reported that substitutional Sn atoms in $\text{Ge}_{1-x}\text{Sn}_x$ epitaxial layers after the growth are thermally more stable for precipitation compared to those during the epitaxial growth process. We must also elucidate the thermal robustness of Sn precipitation for $\text{Ge}_{1-x-y}\text{Si}_x\text{Sn}_y$ epitaxial layer during PDA. In this study, we investigated the crystalline structures and surface morphologies of tensile and compressive strained $\text{Ge}_{1-x-y}\text{Si}_x\text{Sn}_y$ epitaxial layers with various Sn contents. Thermal stability of Sn atoms in $\text{Ge}_{1-x-y}\text{Si}_x\text{Sn}_y$ layers was also investigated.

2. Experimental procedure

Ge(001) wafers were chemically cleaned with a 20% NH_4OH solution and then with a 13% H_2SO_4 solution. After the wafer was loaded into a MBE ultra-high vacuum chamber with a base pressure less than 1×10^{-8} Pa, the chemical oxide layer on the Ge surface was removed by a thermal treatment at 430°C for 30 min in the chamber. Ge and Sn were deposited with Knudsen cells and Si was deposited with e-gun evaporation. Each a 200-nm-thick $\text{Ge}_{1-x-y}\text{Si}_x\text{Sn}_y$ or $\text{Ge}_{1-x}\text{Sn}_x$ layer was grown on the substrate at 200°C . The target contents of Si and Sn were ranging of 21–46% and 4–13%, respectively. After the growth, samples were taken out to atmosphere and PDA was performed for some samples at 500 and 600°C for 10 min in N_2 atmosphere. [The substitutional Sn and Si contents were evaluated using Raman scattering spectroscopy \(Nanophoton RAMAN-11, wavelength: 532 nm\) \[12\].](#)

Four-crystal X-ray diffraction two-dimensional reciprocal space mapping (XRD-2DRSM) measurements around reciprocal lattice points of $\text{Ge}\bar{2}\bar{2}4$ were performed using Philips X'Pert PRO MRD with $\text{Cu K}\alpha 1$ X-ray source to characterize the lattice spacing of the epitaxial layers. The crystallinity of epitaxial layer was characterized by measuring XRD omega rocking curve of $\text{Ge}_{1-x-y}\text{Si}_x\text{Sn}_y$ or $\text{Ge}_{1-x}\text{Sn}_x$ 004 diffraction spot. Furthermore, transmission electron microscopy (TEM) observations were performed to observe the crystalline structures of $\text{Ge}_{1-x-y}\text{Si}_x\text{Sn}_y$ layers using Hitachi H-800 TEM with an operating voltage of 200 kV. TEM specimens were prepared by an ion-milling method. Atomic force microscopy (AFM) was carried out using JEOL JSPM-4200 in AC mode to characterize the surface morphologies of $\text{Ge}_{1-x-y}\text{Si}_x\text{Sn}_y$ layers.

3. Results and discussion

Firstly, the strain structure in $\text{Ge}_{1-x-y}\text{Si}_x\text{Sn}_y$ epitaxial layer was characterized with comparison to $\text{Ge}_{1-x}\text{Sn}_x$. Figures 1(a) and 1(b) show the XRD-2DRSM results of $\text{Ge}_{0.75}\text{Si}_{0.21}\text{Sn}_{0.04}$ and $\text{Ge}_{0.947}\text{Sn}_{0.053}$ epitaxial layers grown on Ge(001) substrates at 200°C . The intensity of these contour maps is drawn with logarithmic graduation. The diffraction peaks related to $\bar{2}\bar{2}4$ Bragg reflection of $\text{Ge}_{1-x-y}\text{Si}_x\text{Sn}_y$ and $\text{Ge}_{1-x}\text{Sn}_x$ are clearly observed for both samples. The each in-plane reciprocal lattice Q_x value of $\text{Ge}_{1-x-y}\text{Si}_x\text{Sn}_y$ and $\text{Ge}_{1-x}\text{Sn}_x$ is identical with that of a Ge substrate. These results mean the pseudomorphic growth of both $\text{Ge}_{1-x-y}\text{Si}_x\text{Sn}_y$ and $\text{Ge}_{1-x}\text{Sn}_x$ epitaxial layers on a Ge(001) substrate. Also, the strain values in the $\text{Ge}_{1-x-y}\text{Si}_x\text{Sn}_y$ and $\text{Ge}_{1-x}\text{Sn}_x$ layers were evaluated to be +0.20% and -0.78%, respectively. Here, we defined plus and minus signs as the tensile and compressive strains in the epitaxial layer, respectively. Because of the strain compensation between Si and Sn in Ge matrix, the strain in $\text{Ge}_{1-x-y}\text{Si}_x\text{Sn}_y$ is small even though the Sn content is 4% compared to $\text{Ge}_{1-x}\text{Sn}_x$. However, it should be noted that the weak long streak along the [110] direction is observed only for the $\text{Ge}_{1-x-y}\text{Si}_x\text{Sn}_y$ diffraction peak as shown in Fig. 1(a). This result indicates that the variability of (220) lattice planes exists in the $\text{Ge}_{1-x-y}\text{Si}_x\text{Sn}_y$ layer. Here, we also measured XRD omega rocking curves for these samples to consider the in-plane (004) lattice planes separately from out-of-plane

(220) lattice planes. Figure 1(c) shows the omega rocking curves of the diffraction peaks for $\text{Ge}_{1-x-y}\text{Si}_x\text{Sn}_y$ and $\text{Ge}_{1-x}\text{Sn}_x$ 004 Bragg reflections. The peak width of the $\text{Ge}_{1-x-y}\text{Si}_x\text{Sn}_y$ diffraction peak is apparently smaller than that of $\text{Ge}_{1-x}\text{Sn}_x$. This means that the tilting angle of $\text{Ge}_{1-x-y}\text{Si}_x\text{Sn}_y$ (004) lattice planes is smaller than that of $\text{Ge}_{1-x}\text{Sn}_x$, which would be due to the small stain in $\text{Ge}_{1-x-y}\text{Si}_x\text{Sn}_y$ layer.

We also prepared a compressive strained $\text{Ge}_{1-x-y}\text{Si}_x\text{Sn}_y$ layer to elucidate the influence of [the sign of the strain](#), which means tensile or compressive, on the crystallinity of $\text{Ge}_{1-x-y}\text{Si}_x\text{Sn}_y$. Figure 2(a) shows the XRD-2DRSM result for the compressive strained $\text{Ge}_{0.67}\text{Si}_{0.26}\text{Sn}_{0.07}$ layer on Ge(001) substrate. This result shows the pseudomorphic growth of the $\text{Ge}_{1-x-y}\text{Si}_x\text{Sn}_y$ layer on Ge(001) substrate with a compressive strain of -0.31% . It should be noted that the broadening of $\text{Ge}_{1-x-y}\text{Si}_x\text{Sn}_y$ diffraction profile along the [110] direction is suppressed compared to the case of tensile strain as shown in Fig. 1(a). To clarify the difference of the broadening and intensity of the peak in the tensile and compressive strained $\text{Ge}_{1-x-y}\text{Si}_x\text{Sn}_y$ layers, the profiles extracted along the [110] directions are shown in Fig. 2(b). We observe the scattering peaks only for the tensile strained $\text{Ge}_{1-x-y}\text{Si}_x\text{Sn}_y$ layer in the tails of the diffraction profile as shown by arrows in Fig. 2(b). [Because a broadening along the \$Q_x\$ direction in a reciprocal space map is generally associated with a lateral correlation length \[13\], we speculate that the scattering peak is also related to a lateral structure of \$\text{Ge}_{1-x-y}\text{Si}_x\text{Sn}_y\$ layer. The possible reason of the peak is the presence of an elastic strain and/or content fluctuation in a lateral direction as reported for III-V semiconductors, such as in ref. \[14\].](#) These results imply that [the sign of the strain](#) of tensile or compressive strongly influences on the crystalline structure of $\text{Ge}_{1-x-y}\text{Si}_x\text{Sn}_y$.

To elucidate the origin of the streaks in the XRD-2DRSM and the impact of [the sign of the strain](#) on the crystalline structure of $\text{Ge}_{1-x-y}\text{Si}_x\text{Sn}_y$, we observed the cross-sectional crystalline structures using TEM. Figures 3(a)-3(c) show the dark-field cross-sectional TEM images of the tensile strained $\text{Ge}_{0.75}\text{Si}_{0.21}\text{Sn}_{0.04}$ and compressive strained $\text{Ge}_{0.67}\text{Si}_{0.26}\text{Sn}_{0.07}$ layers on Ge(001) substrates grown at 200°C . In the image of the tensile strained $\text{Ge}_{1-x-y}\text{Si}_x\text{Sn}_y$ with the diffraction vector of g_{004} , we can see the relatively uniform contrasts (Fig. 3(a)), indicating that the (004) lattice plane in the layer is uniformly formed without the lattice tilting, domain structure, and/or strain fluctuation. In contrast, we can observe the non-uniform contrasts for the tensile strained $\text{Ge}_{1-x-y}\text{Si}_x\text{Sn}_y$ layer, which are composed of columnar structures, in the dark filed TEM image with the diffraction vector of g_{220} (Fig. 3(b)). Because there are few strong contrasts of the lattice defects such as threading or misfit dislocations, which cause a crystalline variability in an epitaxial layer, we consider that these contrasts were caused by the non-uniformity of (220) lattice planes. On the other hand, as shown in Fig. 3(c), the contrast of the compressive strained $\text{Ge}_{1-x-y}\text{Si}_x\text{Sn}_y$ layer is quite uniform with comparison to that of the tensile strained one. These results mean that the crystallinity of $\text{Ge}_{1-x-y}\text{Si}_x\text{Sn}_y$ is significantly improved by

the growth under the compressive strain. Furthermore, these results of the non-uniformity for (220) lattice planes observed by TEM for the tensile strained $\text{Ge}_{1-x-y}\text{Si}_x\text{Sn}_y$ layer consist with the XRD-2DRSM results.

The surface morphologies of the tensile and compressive strained $\text{Ge}_{1-x-y}\text{Si}_x\text{Sn}_y$ layers were observed using AFM as shown in Figs. 4(a) and 4(b), respectively. For the tensile strained $\text{Ge}_{0.75}\text{Si}_{0.21}\text{Sn}_{0.04}$ layer (Fig. 4(a)), we can see the rough surface even though the strain is as small as +0.20%. The root mean square (RMS) roughness was evaluated to be 0.64 nm. In contrast, a flat and uniform surface with a RMS roughness as small as 0.17 nm is observed for the compressive strained $\text{Ge}_{0.72}\text{Si}_{0.20}\text{Sn}_{0.08}$ layer (Fig. 4(b)). Figure 4(a) indicates the three-dimensional island growth of the tensile strained $\text{Ge}_{1-x-y}\text{Si}_x\text{Sn}_y$ layer, [which should be attribute to the columnar structure in the \$\text{Ge}_{1-x-y}\text{Si}_x\text{Sn}_y\$ layer observed in the TEM image as shown in Fig. 3\(b\).](#) The RMS roughness was plotted in Fig. 4(c) as a function of the magnitude of strain for compressive and tensile conditions in order to elucidate the relationship between the strain and surface roughening. Each Sn content in the $\text{Ge}_{1-x-y}\text{Si}_x\text{Sn}_y$ layers is also described in the figure. This figure reveals that a flat surface can be obtained for the unstrained or compressive strained $\text{Ge}_{1-x-y}\text{Si}_x\text{Sn}_y$ layers, while the RMS roughness significantly increases with the magnitude of tensile strain due to the island growth. These results indicate that the growth mode is dominantly determined by [the sign of the strain](#) rather than the Sn content in $\text{Ge}_{1-x-y}\text{Si}_x\text{Sn}_y$ epitaxial layer. Furthermore, the influence of [the sign of the strain](#) for surface roughening is different from the system of $\text{Si}_{1-x}\text{Ge}_x/\text{Si}$ [11]. [Nevertheless, we need further investigation to elucidate the mechanism in detail. The study on the initial growth and thickness dependence of the growth mode of \$\text{Ge}_{1-x-y}\text{Si}_x\text{Sn}_y\$ is considered to be beneficial to clarify how the non-uniform crystallinity is formed. Furthermore, the local strain and content in the lateral structure should be investigated to elucidate the crystalline structure of \$\text{Ge}_{1-x-y}\text{Si}_x\text{Sn}_y\$ layer.](#)

There is a concern that high temperature process after the epitaxial growth often causes Sn precipitation from $\text{Ge}_{1-x}\text{Sn}_x$ or $\text{Ge}_{1-x-y}\text{Si}_x\text{Sn}_y$ epitaxial layer with a high Sn content. We investigated the thermal stability of substitutional Sn atoms in $\text{Ge}_{1-x-y}\text{Si}_x\text{Sn}_y$ layers for various Sn contents using XRD-2DRSM. When the Sn precipitation from $\text{Ge}_{1-x-y}\text{Si}_x\text{Sn}_y$ layer occurs, the lattice constant of $\text{Ge}_{1-x-y}\text{Si}_x\text{Sn}_y$ along the out-of-plane direction increases with a decrease in a substitutional Sn content, assuming the elastic deformation and Vegard's law for $\text{Ge}_{1-x-y}\text{Si}_x\text{Sn}_y$. Figures 5(a) and 5(b) show the results of XRD-2DRSM of as-grown and annealed $\text{Ge}_{0.54}\text{Si}_{0.36}\text{Sn}_{0.10}/\text{Ge}(001)$ samples. The annealing condition was at 500°C for 10 min. For the as-grown sample, the diffraction peak of $\text{Ge}_{1-x-y}\text{Si}_x\text{Sn}_y$ overlaps with that of Ge (Fig. 5(a)). This means that the lattice constants of $\text{Ge}_{1-x-y}\text{Si}_x\text{Sn}_y$ layer perfectly matches to that of Ge substrate along the both directions of in-plane and out-of-plane. As shown in Fig. 5(b), the position of the overlapped $\text{Ge}_{1-x-y}\text{Si}_x\text{Sn}_y$ diffraction peak hardly changes even after the annealing at 500°C for 10 min. Thus, we consider that remarkable Sn precipitation hardly occurs in the $\text{Ge}_{1-x-y}\text{Si}_x\text{Sn}_y$ layer after the annealing.

Figure 5(c) shows the annealing temperature dependence of the lattice constant along the out-of-plane direction of $\text{Ge}_{1-x-y}\text{Si}_x\text{Sn}_y$ layers with Sn contents of 4%, 7%, 10%, and 13%. Here, the annealing time for all samples was 10 min. For the $\text{Ge}_{1-x-y}\text{Si}_x\text{Sn}_y$ layer with a Sn content of 4%, the lattice constant hardly changes even after the annealing at a high temperature of 600°C, while those of the samples with Sn contents of 7% and 10% obviously decrease. Furthermore, this figure also shows that no significant change of the lattice constants of $\text{Ge}_{1-x-y}\text{Si}_x\text{Sn}_y$ layers with 4%–10% Sn contents even after the annealing at 500°C except for the 13%-Sn sample. Therefore, $\text{Ge}_{1-x-y}\text{Si}_x\text{Sn}_y$ layers with a high Sn content is thermally stable for PDA. This would be attributed to the small strain due to the strain compensation of Si and Sn in $\text{Ge}_{1-x-y}\text{Si}_x\text{Sn}_y$ layer [15].

Conclusions

We investigated the influence of strain in $\text{Ge}_{1-x-y}\text{Si}_x\text{Sn}_y$ epitaxial layers grown on Ge(001) substrates on the crystallinity and surface morphology. The crystalline non-uniformity is observed using XRD-2DRSM and TEM for the (220) lattice planes in the tensile strained $\text{Ge}_{1-x-y}\text{Si}_x\text{Sn}_y$ layer even with a small strain of 0.20%. In contrast, the epitaxial growth under compressive strain condition significantly improves in the crystallinity of $\text{Ge}_{1-x-y}\text{Si}_x\text{Sn}_y$ epitaxial layer. We found that the control of [the sign of the strain](#) is an important factor to obtain a high quality $\text{Ge}_{1-x-y}\text{Si}_x\text{Sn}_y$ epitaxial layer. Furthermore, XRD-2DRSM measurements revealed that substitutional Sn atoms in $\text{Ge}_{1-x-y}\text{Si}_x\text{Sn}_y$ layer with a Sn content less than 10% is thermally stable for annealing at 500°C.

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Figure captions

Fig. 1: (a), (b) XRD-2DRSM results around the Ge $\bar{2}\bar{2}4$ reciprocal lattice points of $\text{Ge}_{0.75}\text{Si}_{0.21}\text{Sn}_{0.04}$ and $\text{Ge}_{0.947}\text{Sn}_{0.053}$ epitaxial layers on Ge(001) substrates. (c) XRD omega rocking curves of $\text{Ge}_{1-x-y}\text{Si}_x\text{Sn}_y$ and $\text{Ge}_{1-x}\text{Sn}_x$ 004 diffraction peaks.

Fig. 2: (a) XRD-2DRSM result around the Ge $\bar{2}\bar{2}4$ reciprocal lattice points for compressive strained $\text{Ge}_{0.67}\text{Si}_{0.26}\text{Sn}_{0.07}$ layer on Ge(001) substrate. (b) Peak profiles of $\text{Ge}_{1-x-y}\text{Si}_x\text{Sn}_y$ $\bar{2}\bar{2}4$ diffraction peaks along the [110] direction for tensile strained $\text{Ge}_{0.75}\text{Si}_{0.21}\text{Sn}_{0.04}$ and compressive strained $\text{Ge}_{0.67}\text{Si}_{0.26}\text{Sn}_{0.07}$ layer.

Fig. 3: Dark-field cross-sectional TEM images of (a), (b) tensile strained $\text{Ge}_{0.75}\text{Si}_{0.21}\text{Sn}_{0.04}$ and (c) compressive strained $\text{Ge}_{0.67}\text{Si}_{0.26}\text{Sn}_{0.07}$ layers on Ge(001) substrates. The diffraction vectors were (a) g_{004} , and (b), (c) g_{220} .

Fig. 4: AFM images and surface roughness profiles of (a) tensile strained $\text{Ge}_{0.75}\text{Si}_{0.21}\text{Sn}_{0.04}$ and (b) compressive strained $\text{Ge}_{0.72}\text{Si}_{0.20}\text{Sn}_{0.08}$ layers. (c) The strain magnitude dependence of the RMS roughness for compressive and tensile strained $\text{Ge}_{1-x-y}\text{Si}_x\text{Sn}_y$ layers on Ge(001). The Sn content in each sample is also shown.

Fig. 5: XRD-2DRSM results around the Ge $\bar{2}\bar{2}4$ reciprocal lattice points of (a) as-grown and (b) 500°C-annealed $\text{Ge}_{0.54}\text{Si}_{0.36}\text{Sn}_{0.10}$ /Ge(001) samples. (c) The annealing temperature dependence of the lattice constant of $\text{Ge}_{1-x-y}\text{Si}_x\text{Sn}_y$ layers along the out-of-plane directions. The Sn contents $\text{Ge}_{1-x-y}\text{Si}_x\text{Sn}_y$ samples are 4%, 7%, 10%, and 13%.

1 **Figure 1**

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1 **Figure 2**

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1 **Figure 3**

1 **Figure 4**

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1 **Figure 5**

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