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Quantitative Evaluation of an Epitaxial Silicon-Germanium Layer on Silicon

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Abstract

An epitaxial Si_xGe_y layer on a silicon substrate was quantitatively evaluated using rocking curve (RC) and reciprocal space map (RSM) obtained by powder X-ray diffraction (XRD), energy-dispersive X-ray spectroscopy (EDS) in conjunction with transmission electron microscopy (TEM), and EDS in conjunction with scanning electron microscopy (SEM). To evaluate the relative deviation of the quantitative analysis results obtained by the RC, RSM, SEM/EDS, and TEM/EDS methods, a standard sample comprising a $Si_{0.7602}Ge_{0.2398}$ layer on a Si substrate was used. The correction factor (*K*-factor) for each technique was determined using multiple measurements. The average and standard deviation of the atomic fraction of Ge in the $Si_{0.7602}Ge_{0.2398}$ standard sample, as obtained by the RC, RSM, TEM/EDS, and SEM/EDS methods, were 0.2463 ± 0.0016, 0.2460 ± 0.0015, 0.2350 ± 0.0156, and 0.2433 ± 0.0059, respectively. The correction factors for the RC, RSM, TEM/EDS, and SEM/EDS methods were 0.9740, 0.9740, 1.0206, and 0.9856, respectively. The Si_xGe_y layer on a silicon substrate was quantitatively evaluated using the RC, RSM, and EDS/TEM methods. The atomic fraction of Ge in the epitaxial Si_xGe_y layer, as evaluated by the RC and RSM methods, was 0.1833 ± 0.0007, 0.1792 ± 0.0001, and 0.1631 ± 0.0105, respectively. After evaluating the results of the atomic fraction of Ge in the epitaxial layer, the error was very small, *i.e.*, less than 3%. Thus, the RC, RSM, TEM/EDS, and SEM/EDS methods are suitable for evaluating the composition of Ge in epitaxial layers. However, the thickness of the epitaxial layer, whether the layer is strained or relaxed, and whether the area detected in the TEM and SEM analyses is consistent must be considered.

Keywords

Silicon-Germanium, Epitaxial Layer, Rocking Curve, Reciprocal Spacing Map, TEM, SEM, EDS

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1. Introduction

Over the past several decades, heteroepitaxial structures composed of silicon-germanium on a silicon substrate (Si_xGe_y/Si) have been investigated and successfully applied in complementary metal oxide semiconductors (CMOS) [1] [2], sensors [3], photodetectors and modulators for optical interconnections [4] [5], and heterojunction bipolar transistors [6], among other devices. The strained Si_xGe_y/Si heterostructures will change the band structure and the density of stages because of an enhancement in the mobility of charge carriers [7]. Both silicon (Si) and germanium (Ge) are isostructural with diamond, with lattice constants of a = 5.431 and 5.658 Å, for silicon (Si) and germanium (Ge), respectively; their lattice mismatch is approximately 4.17%. Different Si/Ge ratios in Si_xGe_y will result in variations in the lattice mismatch and in strain relaxation, thereby affecting device performance. Thus, accurate quantitative analysis of Si_xGe_y is critical. In this study, we performed non-destructive high-resolution X-ray diffraction (XRD). Transmission electron microscopy (TEM) in conjunction with energy-dispersive X-ray spectroscopy (EDS) and scanning electron microscopy (SEM) in conjunction with EDS were performed to quantitatively analyze an epitaxial Si_xGe_y layer on a Si substrate. Determining the exact composition of epitaxial Si_xGe_y required the use of a standard sample, $Si_{0.7602}Ge_{0.2398}$, to evaluate the relative deviation of the quantitative analysis results obtained using the aforementioned techniques.

2. Experimental Procedure

An epitaxial Si_xGe_y layer was deposited onto a Si substrate by ultrahigh vacuum chemical vapor deposition (UHVCVD) under a base pressure of 2×10^{-8} Torr. The reactive gases for the growth of Si and Ge were disilane (Si_2H_6) and germane (GeH_4), respectively. The Si_xGe_y layer was grown on the silicon substrate at 400˚C and then annealed at 750˚C/15min to improve the crystallinity. The thickness of the deposited film was approximately 50 nm. The epitaxial Si_xGe_y was quantitatively analyzed using a high-resolution X-ray diffractometer (PANalytical MRD X'Pert) equipped with a Cu-$K_{\alpha 1}$ radiation source (λ = 1.5406 Å), a transmission electron microscopy (model JEM 2010Fx, JEOL, Ltd., Tokyo, Japan) equipped with an energy-dispersive X-ray spectrometer (model X-Max 80, Oxford Instruments, Inc., London, UK), and a scanning electron microscope (JEOL JSM 6500-F) also equipped with an energy-dispersive X-ray spectrometer (model X-Max 80). The cross-sectional TEM specimen of epitaxial Si_xGe_y on Si was prepared using a focused ion beam (FIB, FEI NovaLab 600). The rocking curve (RC) and reciprocal spacing map (RSM) of XRD have been performed to evaluate the composition of Si_xGe_y. As for SEM and TEM, the thickness of the detected areas should be consistent in order to decrease the error. To evaluate the relative deviation of the quantitative analysis results for Si_xGe_y obtained by XRD, TEM/EDS, and SEM/EDS, the analyses of the Si_xGe_y layer were performed multiple times for comparison. Moreover, a standard sample—an $Si_{0.7602}Ge_{0.2398}$ layer with a thickness of 5 μm on Si (reference #8095, National Institute of Standards and Technology (NIST))—was used to evaluate and compare the quantitative errors associated with the aforementioned techniques.

3. Results and Discussion

3.1. Quantitative Evaluation Using XRD Rocking Curve and Reciprocal Space Maps

Figure 1 shows the rocking curve (RC) (ω-2θ) pattern for the (004) reflections of the $Si_{0.7602}Ge_{0.2398}$ layer and the Si substrate of the $Si_{0.7602}Ge_{0.2398}$ standard sample on Si. The $Si_{0.7602}Ge_{0.2398}$ layer on Si is fully relaxed, as evidenced by the lack of periodic interference fringes in **Figure 1**. Strain caused by structural deformation at the interface of SiGe/Si can be relaxed through various mechanisms related to the fabrication temperature [8], time [9], and a layer thickness greater than the critical thickness [10]. From the RC, the composition of a standard sample can be calculated according to Vegard's law on the basis of the relative angle between the substrate peak and the epitaxial-layer peak [11] [12]. From the RC pattern, the composition of the $Si_{0.7602}Ge_{0.2398}$ standard sample was calculated from the relative angle between the (004) substrate peak and the (004) epitaxial-layer peak according to Vegard's law, as stated in Equation (1) [11] [12], from which the lattice constant a_{SiGe} was obtained by linear interpolation. To enhance the accuracy of the method, the modified Vegard's law [13] in Equation (2) [14] [15] was used in this study. In Equation (2), a_{SiGe} is the average lattice constant of the fully relaxed SiGe layer, a_{Si} is the lattice constant of the Si substrate, and a_{Ge} is the lattice constant of pure Ge.

$$a_{\text{SiGe}}(x) = a_{\text{Si}}(1-x) + a_{\text{Ge}}x \tag{1}$$

$$a_{\mathrm{SiGe}}(x) = a_{\mathrm{Ge}}x + a_{\mathrm{Si}}(1-x) - 0.0272x(1-x) \quad (2)$$

In Vegard's law, the relaxation (R) of the layer is strained (R = 0%) or relaxed (R = 100%) to simulate the experimental RC. Thus, in the case of the standard specimen, R was assumed to be 100% because the thickness of the epitaxial layer (4 μm) exceeded the critical thickness, resulting in the lack of interference fringes in the RC pattern in **Figure 1**. The atomic fractions of Ge were calculated as 0.1407 for R = 0% and 0.2463 for R = 100%. Obviously, the value of 0.2463 is similar to the atomic fraction of Ge in the $Si_{0.7602}Ge_{0.2398}$ standard sample. To accurately determine the correction factor (K) in the RC analysis, ten measurements were performed and the average value was calculated, as reported in **Table 1**. A comparison of the two values (0.2463, calculated from the average of ten measurements, and 0.2398, provided by NIST) in **Table 1** indicates that the correction factor for the RC analysis is K_{RC} = 0.2398/0.2463 = 0.9736.

Figure 2 shows the RC (ω-2θ) for the (004) reflection of epitaxial Si_xGe_y. The solid and dashed curves represent the experimental and simulation data, respectively. The periodical interference fringes shown in **Figure 2** indicate that the Si_xGe_y layer is a thin, nearly perfect heteroepitaxial layer and a strain layer [15]. Thus, R should

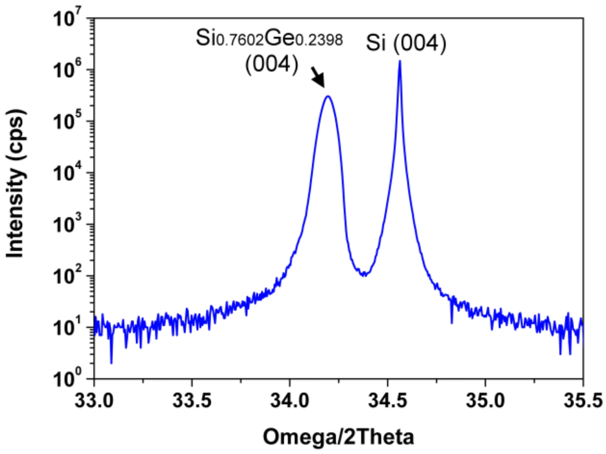

Figure 1. RC (ω-2θ) (004) rocking curve for the standard sample of $Si_{0.7602}Ge_{0.2398}$ on a Si substrate.

Table 1. The correction factor (K) for the $Si_{0.7602}Ge_{0.2398}$ standard sample, as calculated from ten measurements using the RC, RSM, TEM/EDS, and SEM/EDS techniques.

RC Ge (atomic fraction)	RSM Ge (atomic fraction)	TEM/EDS Ge (atomic fraction)	SEM/EDS Ge (atomic fraction)
0.2446	0.2459	0.2378	0.2354
0.2461	0.2474	0.2049	0.2371
0.2479	0.2465	0.2362	0.2444
0.2489	0.2458	0.2187	0.2378
0.2439	0.2476	0.2592	0.2375
0.2471	0.2480	0.2411	0.2448
0.2454	0.2463	0.2389	0.2469
0.2466	0.2450	0.2336	0.2527
0.2450	0.2435	0.2269	0.2493
0.2475	0.2438	0.2523	0.2474
Average = 0.2463 Stdev = 0.0016 K_{RC} = 0.9736	Average = 0.2460 Stdev = 0.0015 K_{RSM} = 0.9749	Average = 0.2350 Stdev = 0.0156 $K_{TEM/EDS}$ = 1.0206	Average = 0.2433 Stdev = 0.0060 $K_{SEM/EDS}$ = 0.9856

be set as 0%. Furthermore, the thickness of the epitaxial Si_xGe_y layer was evaluated to be approximately 50.25 nm on the basis of the spacing of the fringes ($\Delta\theta$) of the RC in **Figure 2**. The thickness was calculated on the basis of the fringe spacing using Equation (3) as follows:

$$\delta\omega = \frac{\lambda \cdot \sin\varepsilon}{t \cdot \sin 2\theta}, \tag{3}$$

where t is the thickness of the Si_xGe_y layer, λ is the wavelength, $\delta\omega$ is omega spacing of the fringes, and ε is the angle between the diffracted beam and the sample surface. The correction factor of quantitative analysis obtained from the RC (K_{RC}) is 0.9736, as discussed previously and shown in **Table 1**. Thus, the composition of Si_xGe_y was corrected by multiplying K_{RC}; the result, along with the standard deviation (Stdev) calculated from the results of ten measurements, are reported in **Table 2**. Finally, the atomic fraction of Ge in the epitaxial Si_xGe_y layer, as calculated from the RC, is 0.1833 ± 0.0007.

The RC of XRD is a fast method for measuring the compositions of epitaxial layers; however, the assumption of R introduces uncertainty. Normally, R = 0% is assumed for a strained epitaxial layer and R = 100% is assumed

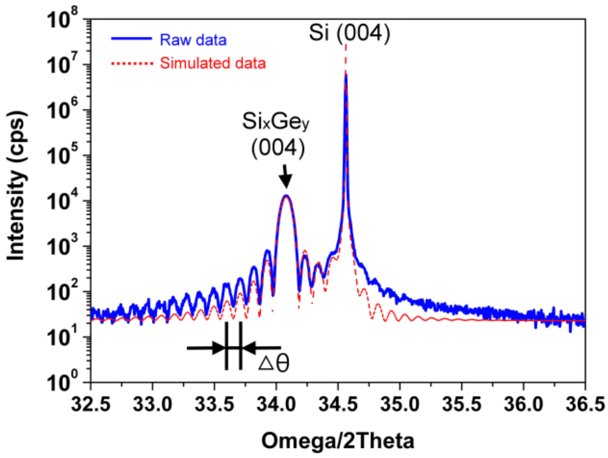

Figure 2. RC (ω-2θ) (004) rocking curve for Si_xGe_y; the solid and dashed curves represent the experimental and simulated results, respectively.

Table 2. Composition of the epitaxial Si_xGe_y, as calculated from the average of ten measurements multiplied by the corresponding correction factor (K) for the RC, RSM, and TEM/EDS methods.

RC Ge (atomic fraction)	RSM Ge (atomic fraction)	TEM Ge (atomic fraction)
0.1887	0.1838	0.1635
0.1870	0.1838	0.1616
0.1897	0.1836	0.1592
0.1888	0.1840	0.1570
0.1879	0.1839	0.1442
0.1883	0.1837	0.1721
0.1885	0.1837	0.1552
0.1881	0.1836	0.1668
0.1878	0.1838	0.1744
0.1883	0.1838	0.1439
Average × K_{RC} = 0.1833 Stdev = 0.0007	Average × K_{RSM} = 0.1792 Stdev = 0.0001	Average × $K_{TEM/EDS}$ = 0.1631 Stdev = 0.0105

for a relaxed layer. In general, whether an epitaxial layer is strained or relaxed depends on several factors, including the thickness of the epitaxial layer, lattice mismatch between epitaxial layer and substrate, and various experimental parameters such as the deposition rate, growth temperature, SiH_4 and GeH_4 flow rates, annealing temperature, and time. However, if the epitaxial layer is partially strained, the relaxation parameter is difficult to estimate and this uncertainty will affect the accuracy of the measurement. Thus, an accurate evaluation of the strain relation and composition of Si_xGe_y requires reciprocal space map (RSM) of XRD with symmetric and asymmetric planes. **Figure 3(a)** and **Figure 3(b)** show the symmetric (004) and asymmetric (224) RSM of the $Si_{0.7602}Ge_{0.2398}$ standard sample, revealing that the layer is fully relaxed. The statistical quantitative analysis of Ge in the standard sample after ten measurements resulted in an average Ge composition of 0.2460, as shown in **Table 1**. *R* can be calculated from **Figure 3(a)** and **Figure 3(b)** was 99.6%. A comparison of the values of 0.2460 (obtained from the measurements) and 0.2398 (reported by NIST) for the atomic fraction of Ge indicate that the correction factor for RSM (K_{RSM}) is the ratio 0.2398/0.2460 equal to 0.9749, which is similar to the K_{RC} value.

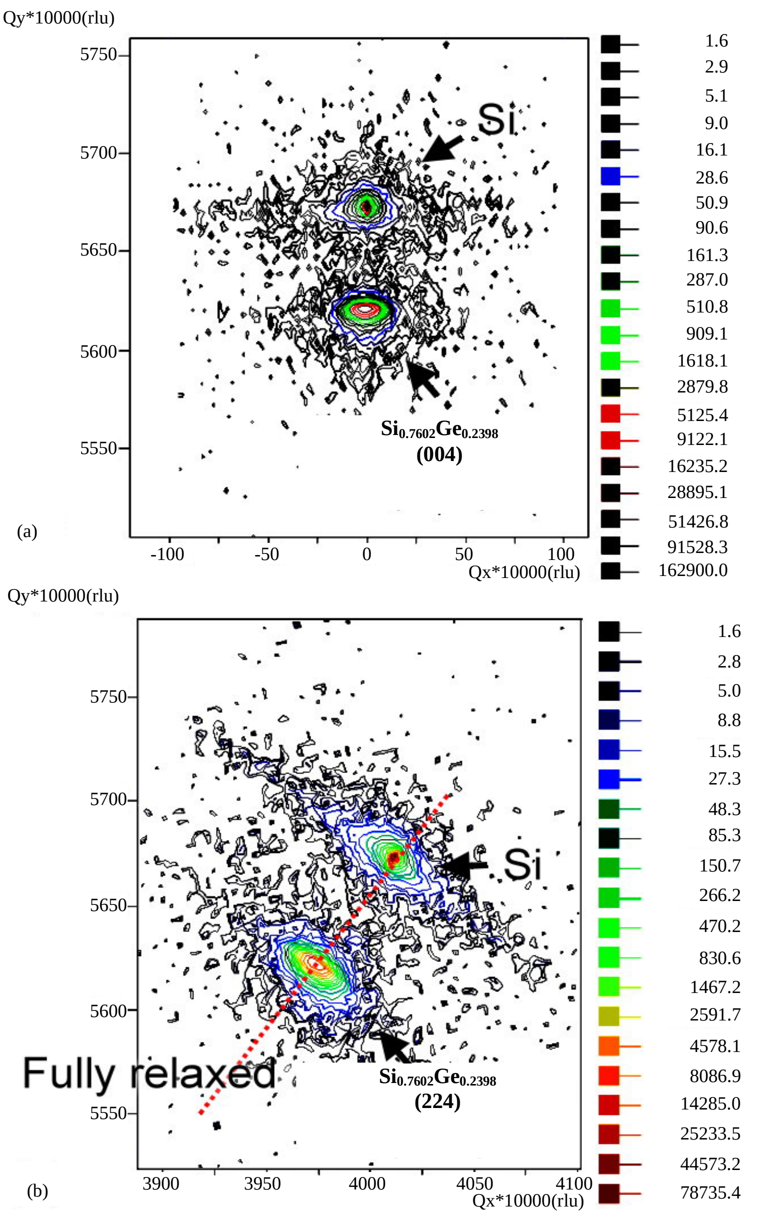

Figure 3. The (a) (004) and (b) (224) RSM of the $Si_{0.7602}Ge_{0.2398}$ standard sample.

Figure 4(a) and **Figure 4(b)** show the symmetric (004) and asymmetric (224) of RSM for the epitaxial Si_xGe_y, displaying the strained epitaxial layer. Ten measurements were performed; the calculated results are shown in **Table 2**. According to these results, the atomic fraction of Ge in Si_xGe_y, after being averaged and multiplied by the K_{RSM}, is 0.1790 ± 0.0001 and R is 0.6%. A comparison of the atomic fractions of Ge in Si_xGe_y obtained from the RC of XRD (0.1833) and RSM (0.1790), as reported in **Table 2**, reveals a difference of approximately 2.4%; this difference is likely a consequence of the assumption of relaxation for the RC. Even though the atomic fraction of Ge in Si_xGe_y obtained using RC differs from that obtained using RSM, the RC technique is faster than the RSM technique. Measurement of the atomic fraction of Ge in Si_xGe_y is time consuming; however, the measurement results are very accurate.

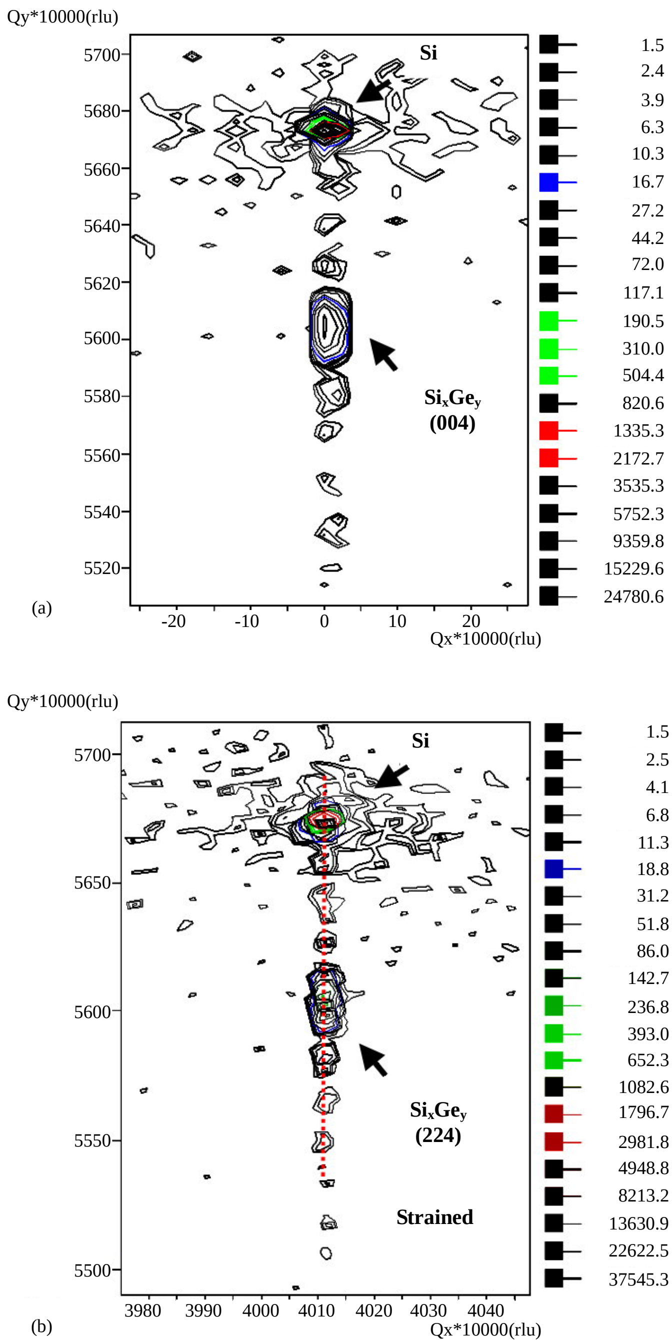

Figure 4. The (a) (004) and (b) (224) RSM of Si_xGe_y.

3.2. Quantitative Evaluation Using TEM/EDS and SEM/EDS

Figure 5(a) shows a transmission electron micrograph (bright-field image, BFI) of the $Si_{0.7602}Ge_{0.2398}$ standard sample on a Si substrate. In this figure, dislocations are evident in the $Si_{0.7602}Ge_{0.2398}$ layer; these dislocations are normally caused by stress release resulting from the lattice mismatch between $Si_{0.7602}Ge_{0.2398}$ and the Si substrate. To accurately and quantitatively analyze the composition of $Si_{0.7602}Ge_{0.2398}$ using the energy-dispersive X-ray spectrometer of the transmission electron microscope, we collected EDS spectra at ten points on the sample, as indicated in **Figure 5(a)**. TEM/EDS quantitative analysis is affected by the interaction volume, which is determined by the interaction of the electron beam with the specimen. Thus, ideally, the regions analyzed by EDS should exhibit a consistent thickness. Here, ten measurements were performed at ten regions with approximately the same thickness, as indicated in **Figure 5**. The average atomic fraction of Ge in the $Si_{0.7602}Ge_{0.2398}$ standard sample was 0.2350. A comparison of this value with the value of 0.2398 provided by NIST results in a correction factor for EDS ($K_{TEM/EDS}$) of 1.0206 (the ratio of 0.2398/0.2350), as reported in **Table 1**. **Figure 5(b)** shows a transmission electron micrograph (BFI) of an epitaxial Si_xGe_y layer on a Si substrate. The thickness of the Si_xGe_y layer was determined to be 48.47 nm, similar to the thickness calculated from the XRD pattern. Moreover, no obvious dislocations were observed at the interface between the epitaxial Si_xGe_y layer and the Si substrate. Thus, the epitaxial Si_xGe_y layer on Si is likely strained, consistent with the results of the RC and RSM analyses. The ten points on the epitaxial Si_xGe_y layer indicated in **Figure 5(b)** were analyzed. The average (multiplied by $K_{TEM/EDS}$) and the Stdev were 0.1631 and 0.0105, respectively.

Figure 6 shows a top-down scanning electron micrograph (secondary electron image, SEI) of the $Si_{0.7602}Ge_{0.2398}$ standard sample on a Si substrate, performed by the voltage of 15 kV and working distance of 9.9 mm. The ten points on the sample indicated in **Figure 6** were analyzed using the EDS system of the scanning electron microscope. The average for the atomic fraction of Ge in the $Si_{0.7602}Ge_{0.2398}$ standard sample was 0.2433. A comparison of this value with that provided by NIST (0.2398) indicates that the correction factor for EDS ($K_{SEM/EDS}$) is 0.9856, calculated by the ratio of 0.2398/0.2433, as reported in **Table 1**. Normally, if the epitaxial layer on a Si substrate is less than 1-μm thick, the interaction volume will expand to approximately a 1-μm droplet from the layer to the substrate when the SEM acceleration voltage is 15 kV. Thus, SEM/EDS will not only detect the layer but will also detect the substrate, resulting in inaccurate quantitative analysis results for the layer. Thus, SEM/EDS is not suitable for evaluating thin epitaxial layers with a thickness of 1 μm or less on a Si substrate.

The atomic fractions of Ge and the correction factors for the RC, RSM, TEM/EDS, and SEM/EDS techniques are summarized in **Table 3**. The average and standard deviation of the atomic fractions of Ge in the $Si_{0.7602}Ge_{0.2398}$ standard sample are 0.2463 ± 0.0016, 0.2460 ± 0.0015, 0.2350 ± 0.0156, and 0.2433 ± 0.0060 for RC, RSM, TEM/EDS, and SEM/EDS, respectively. Among these techniques, the error was less than 3%. The correction factors for RC, RSM, TEM/EDS, and SEM/EDS are 0.9736, 0.9749, 1.0206, and 0.9856, respectively. To obtain the exact composition for the Si_xGe_y layer, the correction factor needs to be applied to each measured value. The atomic fractions of Ge in the epitaxial Si_xGe_y layer, as determined by RC, RSM, and TEM/EDS, are 0.1833 ± 0.0007, 0.1792 ± 0.0001, and 0.1631 ± 0.0105, respectively. In the case of the TEM/EDS analysis, the preparation of the TEM samples is more time consuming compared with the sample preparation times required in the

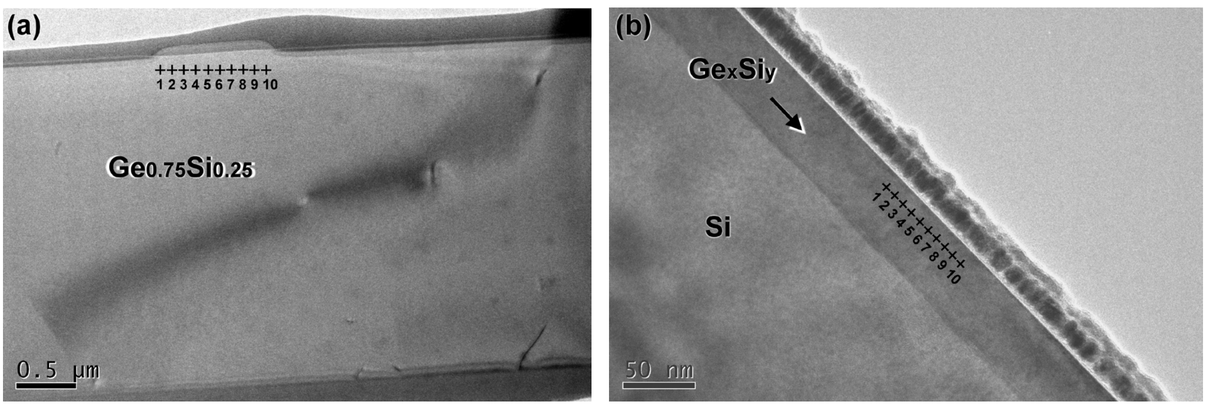

Figure 5. Transmission electron micrograph (bright-field image, BFI) of (a) the $Si_{0.7602}Ge_{0.2398}$ standard sample and (b) Si_xGe_y on a Si substrate.

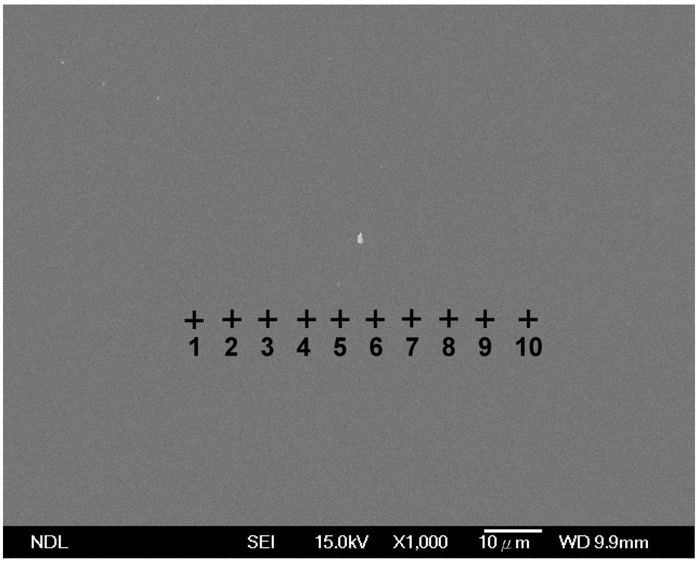

Figure 6. Scanning electron micrograph (secondary electron image, SEI) of the $Si_{0.7602}Ge_{0.2398}$ standard sample.

Table 3. Comparison of the quantitative evaluation results for the $Si_{0.7602}Ge_{0.2398}$ (numbers represent compositions in atomic %) standard sample, as obtained using RC, RSM, TEM/EDS, and SEM/EDS.

Technique	Atomic fraction of Ge (Standard: 0.2398)	Error (%)	Sample preparation/Note
RC	0.2463	2.71	Fast/assume relaxation (*R*)
RSM	0.2460	2.59	Fast/requires long sample preparation time
TEM/EDS	0.2350	2.00	Slow/consistent beam intensity and thickness of detected area
SEM/EDS	0.2433	1.46	Fast/layer larger than 1 μm; consistent beam intensity and thickness of detected area

other techniques. In the cases of the RC and RSM techniques, the samples are quickly and easily cut to prepare them. In the RC measurement, *R* of the layer must be assumed to be strained (*R* = 0%) or relaxed (*R* = 100%). Thus, if the Si_xGe_y layer is partially strained, the RC measurement probably will not provide an accurate result. The RSM method can be used to resolve this issue of partial strain in a Si_xGe_y sample; however, in such cases, the measurement will necessarily require more time. If the thickness of the Si_xGe_y layer is less than 1 μm, SEM-based EDS will not be a suitable method for characterizing the layer because the electron beam will detect elements in the substrate because of the interaction volume depth being approximately 1 μm. Moreover, in the cases of analyses by TEM/EDS and SEM/EDS, the thickness of the detected area and the beam intensity could increase the error of the quantitative analysis results.

4. Conclusion

Quantitative evaluations of epitaxial Si_xGe_y layers on silicon substrates using the RC, RSM, SEM/EDS, and EDS/TEM techniques were performed in this study. A standard sample, $Si_{0.7602}Ge_{0.2398}$, was used to evaluate the relative deviation of the RC, RSM, SEM/EDS, and TEM/EDS quantitative analysis results. The average and standard deviation of the atomic fraction of Ge in the $Si_{0.7602}Ge_{0.2398}$ standard sample, as determined by the RC, RSM, TEM/EDS, and SEM/EDS methods, were 0.2463 ± 0.0016, 0.2460 ± 0.0015, 0.2350 ± 0.0156, and 0.2433 ± 0.0059, respectively. The correction factors for each technique were determined using the standard sample of $Si_{0.7602}Ge_{0.2398}$ on Si; the determined factors were 0.9740, 0.9740, 1.0206, and 0.9856 for RC, RSM, TEM/EDS, and SEM/EDS, respectively. An epitaxial Si_xGe_y layer on silicon was quantitatively evaluated using the RC, RSM, TEM/EDS methods, and the atomic fractions of Ge in the epitaxial Si_xGe_y layer were determined as 0.1833 ± 0.0007, 0.1792 ± 0.0001, and 0.1631 ± 0.0105, respectively. We determined that SEM/EDS was not suitable for evaluating Si_xGe_y layers on Si substrates in cases where the layer thickness was less than 1 μm. The error associated with our determination of the atomic fraction of Ge in the epitaxial layer was very small, *i.e*., less than 3%. Thus, the RC, RSM, TEM/EDS, and SEM/EDS methods are suitable for evaluating the composition of Ge in an epitaxial layer; however, the thickness of the epitaxial layer, whether the layer is strained or relaxed, and whether the thickness of the detected areas in the TEM and SEM measurements is consistent must be considered.

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