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# Characterization of Ge $\delta$-doped Si(111) with RBS channelling

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**The crystalline quality of the Si cap layer, the lattice location and the depth distribution of $\delta$-doped Ge layers in Si(111) crystals grown by molecular beam epitaxy at low and high temperatures have been studied by means of double-grazing-angle Rutherford backscattering spectrometry (RBS), RBS channelling and atomic force microscopy. The crystalline quality of the Si cap layer grown at high temperature is excellent, but there are some lattice defects in the interface or in the cap layer grown at low temperature. The RBS channelling measurements show that most of the Ge atoms occupy the Si substitutional sites. The full width at half-maximum in the depth distribution of $\delta$-doped Ge in the Si(111) crystal is determined to be 13 ± 5 Å.** 

**KEYWORDS:** RBS; AFM; high energy ion scattering (HEIS); channelling; atomic force microscopy; $\delta$-doped; surface roughness; growth; silicon; germanium

## INTRODUCTION

In recent years, fabrication of a $\delta$-like doping profile of a foreign element in a semiconductor has become a subject of major interest in terms of the fundamentals and technologies. So far, Ge $\delta$-layers up to 14 monolayers (ML) thick in Si(111) crystals grown by surfactant-mediated epitaxy (SME) have been studied using medium-energy ion scattering (MEIS), spot profile analysis of low-energy electron diffraction (SPALEED) and transmission electron microscopy (TEM).[1–3] It has been shown by MEIS that Ge films up to 8 ML thick are pseudomorphic on the Si substrate and are adjusted to the smaller substrate lattice constant of the substrate with tetragonal distortion.[1] However, it is known that pseudomorphic SME does not produce Ge films with a smooth surface, especially at intermediate thicknesses.[3]

In the case of molecular beam epitaxy (MBE) without surfactant, thin Ge $\delta$-layers up to 3 ML in Si(111) crystal have been studied by means of SPALEED and x-ray standing-wave (XSW).[2] The first two Ge layers grow in a double bilayer fashion,[2] as already found for Si homoepitaxy,[4,5] although Si-MBE on bulk like Ge proceeds in Volmer–Weber mode.[6] On the other hand, in the case of Si growth on thin strained Ge films, Si grows in bilayer-by-bilayer mode by MBE at a low temperature of 490 °C.[2] It has been shown by XSW that a high crystalline quality at Ge/Si interfaces is achieved at such a low temperature.[2] Because XSW is insensitive to depth, it is necessary to complement the result by direct depth measurements such as Rutherford backscattering spectrometry (RBS) and MEIS.[1,7–9]

In this paper, the crystalline quality of the Si cap layer, the Ge lattice location and the depth distribution of $\delta$-doped Ge in Si(111) crystal by MBE at low (490 °C) and high (700 °C) temperatures have been studied by means of double-grazing angle RBS, RBS channelling and atomic force microscopy (AFM).

## EXPERIMENTAL

The specimens used were mirror-polished n-type Si(111) wafers, $10^{-3}\ \Omega\cdot$cm, with a size of $15 \times 5 \times 0.5$ mm$^3$. The specimen was placed on a manipulator in an ultrahigh vacuum (UHV) system equipped with electron beam deposition sources of Ge and Si at a base pressure of $<7.5 \times 10^{-11}$ Torr. The specimen surface was cleaned by repeated direct current heatings for 20 s at ~1200 °C. After the cleaning process, a bright $7 \times 7$ LEED pattern was observed. Germanium growth and subsequent Si deposition were monitored *in situ* by SPALEED, i.e. the 00-beam LEED intensity was measured as a function of coverage. Details of the SPALEED analysis are published elsewhere.[2] The specimens of Ge $\delta$-doped Si(111) crystal were prepared by MBE at low (490 °C) and high (700 °C) temperatures. Germanium films 1 ML thick (1 ML for Si(111) face = $7.8 \times 10^{14}$ atoms cm$^{-2}$) were deposited onto the Si(111)-$7 \times 7$ surface. The Si cap layer 100 Å thick was grown on the Ge/Si(111) substrate, which is sufficiently thick for protection of the Ge layer from oxidation during air transfer. The Ge and Si growth rates were 0.5 and 4 ML min$^{-1}$, respectively.

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The RBS channelling measurements were carried out in a different UHV chamber that was connected to a beam line of a 2 MeV Van de Graaff accelerator. The $\delta$-doped Ge crystal was mounted on a three-axes rotatable goniometer with two-axes linear motion and an angular resolution of 0.025°. For the measurements, we used a well-collimated (<0.01°) 1.5 MeV $He^+$ beam of 1 mm in diameter. The backscattered $He^+$ ions were detected with silicon surface barrier (SSB) detectors at scattering angles of $153 \pm 1.5°$ and $99 \pm 1.5°$, with an energy resolution of 12 keV in full width at half-maximum (FWHM). The FWHM energy resolution of the system was estimated to be $13.8 \pm 0.2$ keV from the peak of the monolayer Au film on Si substrate at random incidence,[10] thus deriving from the fluctuation in the incident $He^+$ beam energy, the SSB detector resolution, the linearity of the preamplifier and the main amplifier and the analog-to-digital converter in the multichannel analyser.

The beam current was measured with a bias of +90 V supplied to the sample to suppress the secondary electron emission. The beam current was typically 5 nA and a total $He^+$ ion fluence of $\sim 4 \times 10^{15}$ ions $cm^{-2}$ was needed to obtain one RBS spectrum. Angular scans of the backscattering yield were run around the ⟨111⟩ and ⟨110⟩ directions. In the ⟨111⟩-aligned RBS channeling measurements, scattered $He^+$ ions were measured simultaneously with two SSB detectors at scattering angles of 99° and 153° (namely off-planar channelling directions) in order to evaluate both the crystalline quality of the Si cap layer and the amounts of surface impurity atoms such as oxygen and carbon. A rather high depth resolution of 15 Å per channel was achieved at the scattering angle of 99° (57 Å per channel at the scattering angle of 153°). The random spectra were obtained by a polar rotation of 4.0° relative to the channelling direction, which is not a condition for planar channelling, and the direction of detection is not in the plane of incidence. Moreover, in order to measure the Ge depth distribution with the higher depth resolution, both the incidence angle of the $He^+$ beam and the emergence angle of the scattered $He^+$ ions were set at 85°

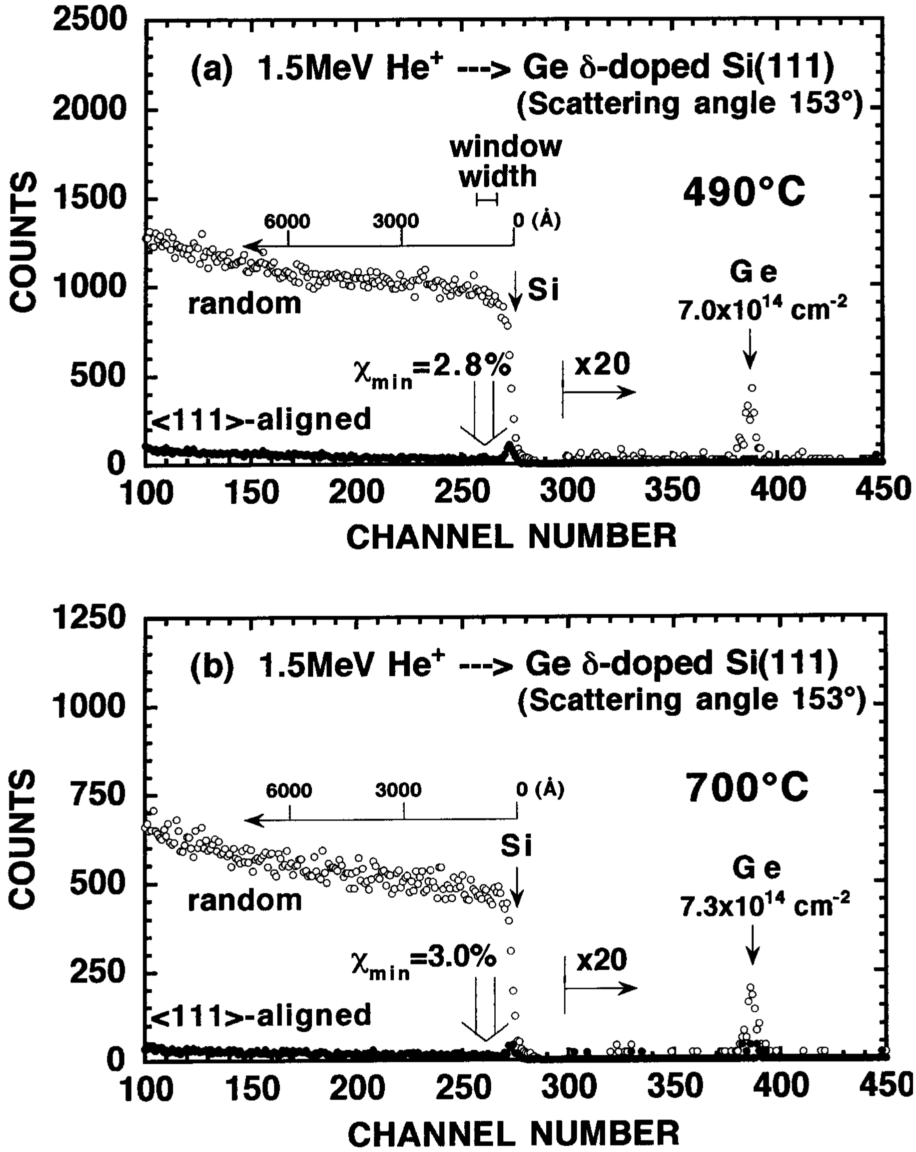
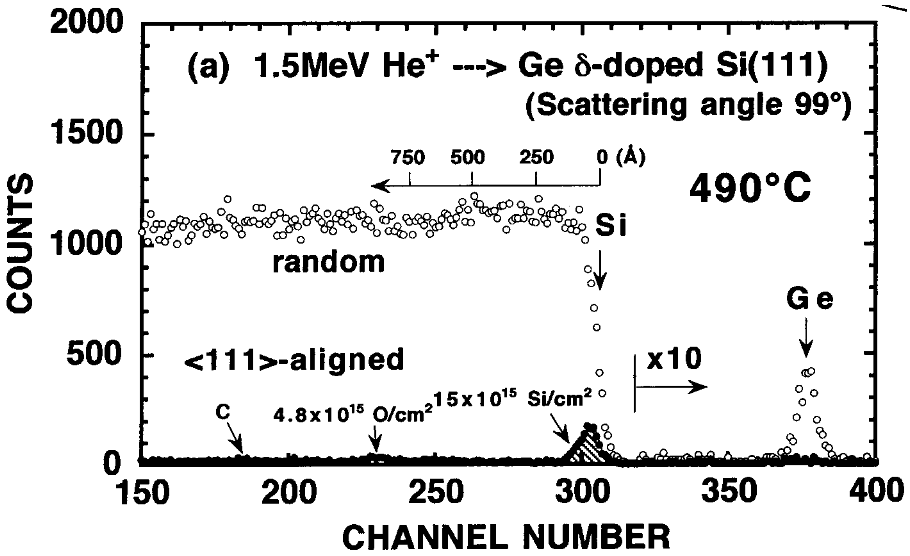



**Figure 1.** The RBS spectra of 1.5 MeV $He^+$ ions from the Ge $\delta$-doped Si(111) substrate grown at 490 °C (a) and 700 °C (b) at random incidence (○) and at the ⟨111⟩-aligned incidence (●), in which the scattering angle is 153°.



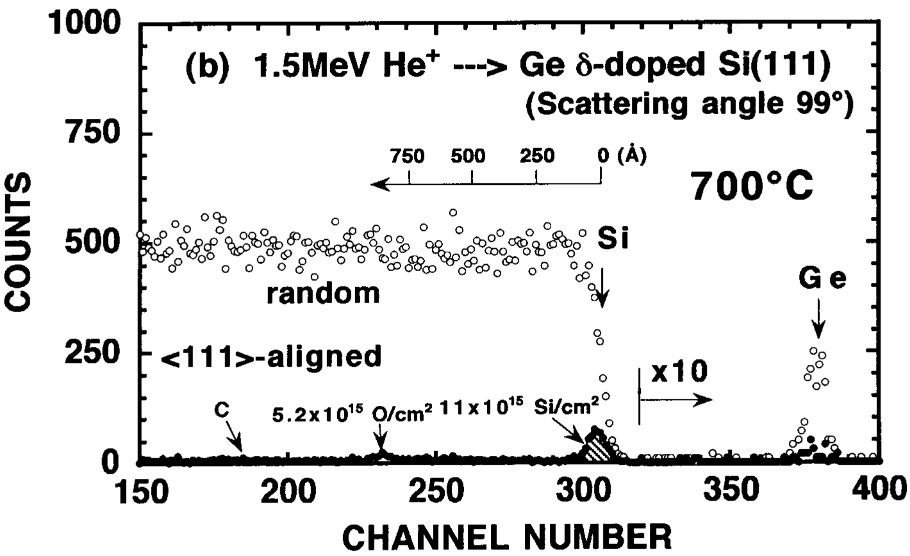




**Figure 2.** The RBS spectra of 1.5 MeV $He^+$ ions from the Ge $\delta$-doped Si(111) substrate grown at 490 °C (a) and 700 °C (b) at random incidence (○) and at the ⟨111⟩-aligned incidence (●), in which the scattering angle is 99°.

to the surface normal—the so-called double-grazing-angle technique—and the azimuth angle of the incident beam and the scattering angle were 3° to the ⟨011⟩ plane and 150°, respectively. An extremely high depth resolution of 5.4 Å per channel was nominally achieved under this condition. The surface roughness was evaluated by atomic force microscopy (AFM) (Olympus model NV2000) in order to estimate the Ge depth distribution from the double-grazing-angle RBS spectrum. The AFM images were taken in air.

## RESULTS

Typical RBS spectra of $He^+$ ions backscattered towards an angle of 153° from the Ge $\delta$-doped Si(111) substrates prepared by MBE at 490 °C and 700 °C are shown in Fig. 1. Open and closed circles represent the random and the ⟨111⟩-aligned spectra. From the random spectra, the concentrations of $\delta$-doped Ge in the Si(111) crystal were determined to be $(7.0 \pm 0.6) \times 10^{14}$ $cm^{-2}$ (at 490 °C) and $(7.3 \pm 0.9) \times 10^{14}$ $cm^{-2}$ (at 700 °C). From the ratio of the aligned to the random yield, the average minimum yields $\chi_{min}$ for the Si substrate, obtained from the total counts in the window width between channels 260 and 270, were measured to be $2.8 \pm 0.2\%$ (at 490 °C) and $3.0 \pm 0.2\%$ (at 700 °C). Similarly, the Ge yields at the aligned incidence are found to be lower than those at random incidence and the average minimum yields $\chi_{min}$ for Ge atoms were estimated to be $4 \pm 0.3\%$ (at 490 °C) and $16 \pm 0.3\%$ (at 700 °C).

In order to evaluate the crystalline quality of the Si cap layer grown at 490 °C and 700 °C, RBS channelling spectra were measured at the scattering angle of 99° (Fig. 2). The Si surface peak yields were converted into the areal densities of $(15 \pm 0.4) \times 10^{15}$ (at 490 °C) and $(11 \pm 0.5) \times 10^{15}$ Si $cm^{-2}$ (at 700 °C), which are discussed later. The existence of oxygen at the surfaces of both specimens is seen from Fig. 2. For the low-temperature substrate a small peak of carbon also is seen. The presence of oxygen and carbon at the surface can be attributed to oxidation and contamination, respectively, during air transfer and storage.

The angular scans of the scattering yields from Ge and Si atoms in the low-temperature specimen were measured around the $\langle 111 \rangle$ and $\langle 110 \rangle$ directions by polar rotation to determine the lattice site of Ge, as shown in Fig. 3. The Si and Ge normalized yields are defined as the backscattering yield at each tilt angle divided by that in random geometry. The Si normalized yields were obtained from the total counts in the window width between channels 260 and 270 of the RBS spectra in Fig. 1. The angular dependence of the Si yields to the $\langle 111 \rangle$ direction shows somewhat planar channelling effects at large tilt angles, as seen from Fig. 3(a). However, the angular profiles at small tilt angles near the $\langle 111 \rangle$ direction are not influenced by the planar channelling effects. The depth dependence of the channelling critical angles (which are half the width of the tilt angle at half-maximum of the normalized random yields) on the $\langle 110 \rangle$ axis of the Ge $\delta$-doped Si(111) substrate is shown in Fig. 4. It is seen from Fig. 4 that the critical angle for Si lattice atoms increases with decreasing depth and coincides with that for $\delta$-doped Ge atoms.

The Ge depth distributions were evaluated by double-grazing angle RBS in combination with AFM. Typical RBS spectra at double grazing angles from the Ge $\delta$-doped Si(111)

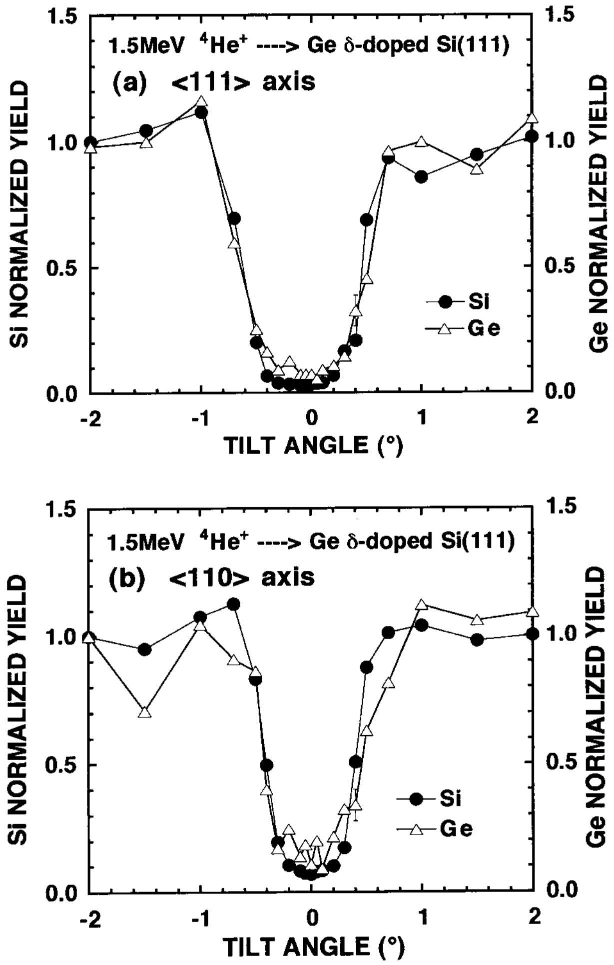



**Figure 3.** Angular scans of the backscattering yield of 1.5 MeV He$^+$ ions from the Ge $\delta$-doped Si(111) substrate, which are obtained around the $\langle 111 \rangle$ (a) and the $\langle 110 \rangle$ (b) axes: ($\triangle$) from the Ge atoms; ($\bullet$) from the Si atoms.

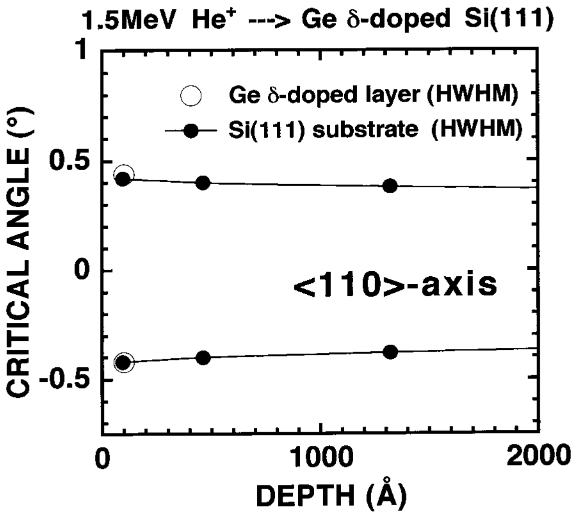



**Figure 4.** Depth dependence of channelling critical angles (which are half the width of the tilt angle at half-maximum of the normalized random yields) of 1.5 MeV He$^+$ ions to the $\langle 110 \rangle$ axis of the Ge $\delta$-doped Si(111) substrate.

substrates at 490 °C and 700 °C are shown in Fig. 5. Most of the Ge atoms are located at the interface and the mean depths of the $\delta$-doped Ge layers were estimated to be $110 \pm 5$ Å (at 490 °C) and $100 \pm 5$ Å (at 700 °C) from Fig. 5(a) and 5(b), respectively. The FWHM was estimated to be $25 \pm 3$ keV for both samples. On the other hand, the surface roughnesses were estimated to be $7 \pm 1$ Å (at 490 °C) and $6 \pm 1$ Å (at 700 °C) by AFM measurements. Using these data, the broadness of the Ge $\delta$-doped layer is estimated below.

## DISCUSSION

The crystalline quality of the Si cap layer can be evaluated from the integrated yield of the Si surface peak. The Si surface peak yield represents the total number of Si atoms not shadowed by the topmost atoms of the Si$\langle 111 \rangle$ atomic rows and deviated due to thermal vibration and lattice defects in the bulk and Si atoms in amorphous layers of the oxidized surface. As described in the preceding section, the Si surface peak yields of the spectra at 490 °C and 700 °C in Fig. 2 were converted into areal densities of $(15 \pm 0.4) \times 10^{15}$ and $(11 \pm 0.5) \times 10^{15}$ Si cm$^{-2}$, respectively. On the other hand, the concentrations of oxygen at the surface were estimated from Fig. 2 to be $(4.8 \pm 0.4) \times 10^{15}$ (at 490 °C) and $(5.2 \pm 0.4) \times 10^{15}$ (at 700 °C). If the composition of the oxidized surface is assumed to be SiO2, the numbers of displaced Si atoms in the layers are estimated to be $(2.4 \pm 0.2) \times 10^{15}$ (at 490 °C) and $(2.6 \pm 0.2) \times 10^{15}$ Si cm$^{-2}$ (at 700 °C). Therefore, the contributions of displaced Si atoms in the Si cap layer to the surface peak yields are calculated to be $(12.6 \pm 0.6) \times 10^{15}$ and $(8.4 \pm 0.7) \times 10^{15}$ Si cm$^{-2}$, respectively. According to comparison of the 'universal' curve with the experimental values for a number of different 'bulk-like' surfaces, which were determined from the RBS channelling measurements by Feldman,[11] the intrinsic surface peak yield of the Si(111) crystal for 1.5 MeV He$^+$ ion beam is estimated to be $8.4 \times 10^{15}$ Si cm$^{-2}$. The latter fact is concluded to indicate that there are

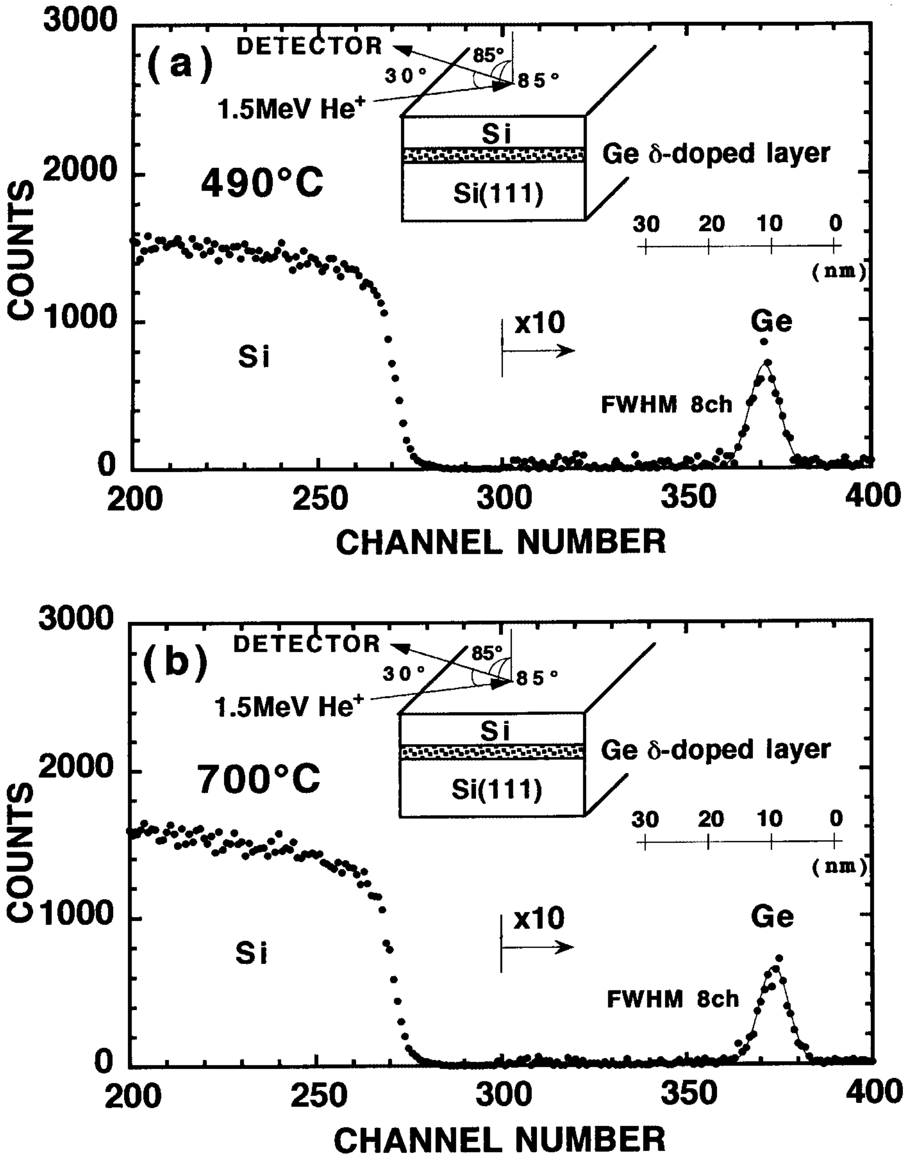



**Figure 5.** The RBS spectra of 1.5 MeV $He^+$ ions from the Ge $\delta$-doped Si(111) substrates grown at 490 °C (a) and 700 °C (b) in double-grazing-angle geometry, where the incidence angle of the $He^+$ beam and the emergence angle of the scattered $He^+$ ions are 85° to the surface normal and the scattering angle is 150°.

almost no lattice defects in the Si cap layer grown at 700 °C, but there are extra displacement atoms—$(4.2 \pm 0.6) \times 10^{15}$ Si $cm^{-2}$—in the Si cap layer grown at 490 °C. The relative number of extra displacement atoms is estimated to be ~7 at% in the whole cap layer of ~11 nm thick.

Angular scans of RBS channelling experiments were performed around the ⟨111⟩ and ⟨110⟩ directions to locate the lattice sites of $\delta$-doped Ge atoms. It is seen from Fig. 3 that the curves of the Ge yields are almost identical to the curves of the Si yields, although the Ge yields in the ⟨110⟩ direction are slightly higher than the Si yields. It is also seen from Fig. 4 that the values of the half-width at half-maximum of the dip curve for the Ge yields agree with those for the Si yields. These data indicate that most of the Ge atoms substantially occupy the Si lattice sites. Moreover, as seen from Figs 5(a) and 5(b), most of the Ge atoms are confined at the interface. Therefore, it is concluded that Ge atoms do not segregate to the surface at these temperatures. In the case of the Si(001) substrate, it has been reported that some of the $\delta$-doped Ge atoms formed by Si MBE growth without surfactant atoms segregate to the surface.[12–15] Nakagawa and Miyao showed that the Ge segregation was reduced with increasing deposition rate of Si[16] therefore the Si deposition rate as well as the crystallographic orientation of the sample may be correlated to the Ge segregation.

It is also seen from Fig. 3 that the Ge yields around the ⟨111⟩ and ⟨110⟩ directions, at tilt angles between −0.2 and 0.2, are $\sim 4 \pm 2$ and $8 \pm 4\%$ higher than the Si yields. The slightly higher Ge yields can be attributed to relaxation of the strained Ge $\delta$-layer formed during the growth of the Si cap layer, as is found also in Ge layers $\delta$-doped in Si(100) crystals.[17,18] The minimum yields of the Ge layer in the ⟨111⟩ direction were estimated to be 4% at 490 °C and 16% at 700 °C, from Figs 1 and 2.

The energy distribution of scattered $He^+$ from the Ge layer in double-grazing-angle RBS is totally composed of contributions from seven factors: the detector energy resolution, $\delta E_d$; the straggling in the energy-loss processes, $\delta E_s$; the energy fluctuations by surface roughness, $\delta E_{x\,in}$ and $\delta E_{x\,out}$; the kinematic energy spread due to the variation in scattering angle, $\delta E_a$; the kinematic energy fluctuation due to the isotope effects of Ge, $\delta E_i$; and the Ge depth distribution, $\delta E_{Ge}$. Assuming that the seven contributions are independent and satisfy Poisson's statistics, the total squared fluctuation around the average energy loss $\delta E$ is given by

$$(\delta E)^2 = (\delta E_d)^2 + (\delta E_s)^2 + (\delta E_{x\,in})^2 + (\delta E_{x\,out})^2 + (\delta E_a)^2 + (\delta E_i)^2 + (\delta E_{Ge})^2$$

For example, for a model of the $\delta$-doped Ge layer with FWHM 5–20 Å and at a depth of 100 Å in thickness, the contributions of each factor to the energy-loss fluctuation for a 1.5 MeV $He^+$ ion beam and the total amount are calculated with a detector energy resolution of 13.8 keV and a surface roughness of 7 Å. Here, the incidence angle of the $He^+$ beam was set equal to the emergence angle of the scattered $He^+$ ions to compare with the present experimental condition. Because the scattering angle is $153 \pm 1.5°$, the kinematic energy spread is estimated to be 2 keV. The kinematic energy fluctuation due to the isotope effects of Ge is estimated to be 4 keV. The calculated values are shown as a function of the angle in Figs 6 and 7. It is seen clearly from Fig. 7 that the depth resolution of the $\delta$-doped Ge layer increases with decreasing angle. Moreover, it is also seen from Fig. 7 that the FWHM of the $\delta$-doped layer can be determined within the experimental error of 5 Å when the angles are set at 5°.

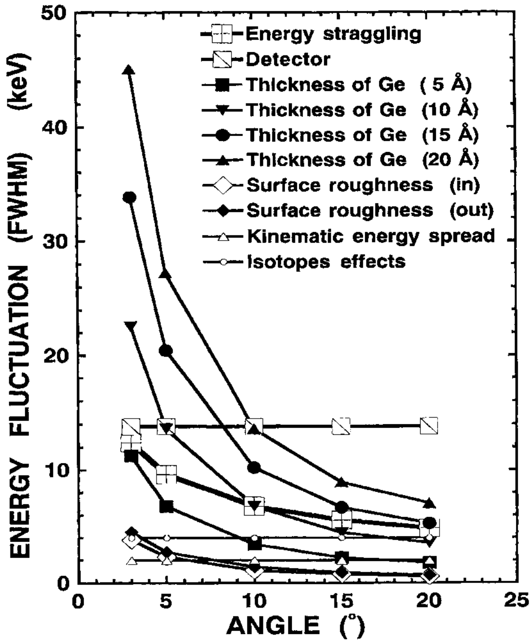



**Figure 6.** The contributions of each factor (the straggling in the energy loss processes, the detector energy resolution, the Ge depth distributions from 5 to 20 Å, the energy fluctuations by surface roughness, the kinematic energy spread and the isotope effects) to the energy-loss fluctuation as a function of the incidence angle of the $He^+$ beam and the emergence angle of the scattered $He^+$ ions.

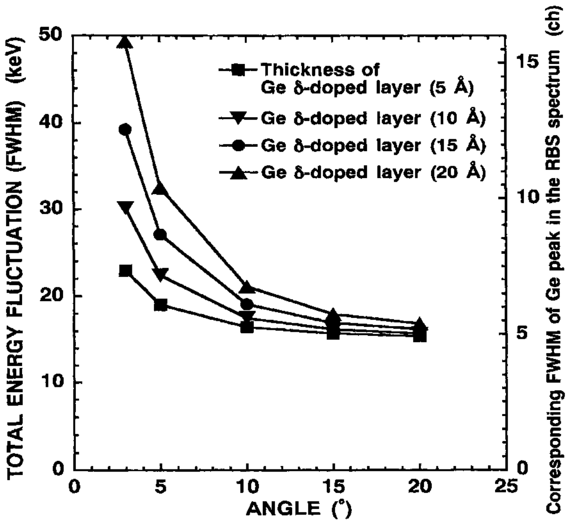



**Figure 7.** The total energy-loss fluctuation at the Ge depth distributions from 5 to 20 Å as a function of the incidence angle of the $He^+$ beam and the emergence angle of the scattered $He^+$ ions.

Here, the FWHM values of the $\delta$-doped Ge layers in the present study are evaluated from the FWHM of the Ge peaks in Fig. 5. From the double-grazing-angle RBS data (in Fig. 5) the mean depths of the $\delta$-doped Ge layers grown at 490 °C and 700 °C were estimated to be $110 \pm 5$ Å (at 490 °C) and $100 \pm 5$ Å (at 700 °C). These values are very close to those monitored *in situ* by SPALEED.[2] The surface roughnesses were estimated to be $7 \pm 1$ Å (490 °C) and $6 \pm 1$ Å (700 °C) from the AFM measurements. This is consistent with the results that Si homoepitaxy proceeds in a double bilayer (6.3 Å) fashion.[4,5] The contributions of the surface roughness to fluctuations in the energy loss of the $He^+$ ions under the condition of double grazing angles are evaluated to be $2.3 \pm 0.3$ and $2.7 \pm 0.4$ keV at 490 °C and $2.0 \pm 0.3$ and $2.3 \pm 0.3$ keV at 700 °C. Because the angles of the incident $He^+$ beam and the detector were set at 85° to the surface normal, the path length of the $He^+$ ions in the Si crystal is $>$1000 Å and the contribution to straggling in the energy-loss processes is calculated to be 10 keV. Experimental data in Fig. 5 show that the Ge peaks are symmetric in shape and the FWHM is $25 \pm 3$ keV. Therefore, the FWHM of the depth distributions of Ge is determined to be to $13 \pm 5$ Å. However, the details of the Ge depth distribution are not clear yet because of the resolution limit. Because the first Ge layers grow in a double bilayer (6.3 Å) fashion,[2] it is considered that some of the Ge exchanges with Si substitutional sites during the Si cap layer growth. This is in quite good agreement with the XSW results,[2] which show that the Si–Ge site exchanges are partly expected during the Si cap layer growth.

## CONCLUSION

The $\delta$-doped Ge layers in the Si(111) crystals grown by MBE at low (490 °C) and high (700 °C) temperatures were studied by means of double-grazing-angle RBS, RBS channelling and AFM. The mean depths of the $\delta$-doped Ge layers grown at 490 °C and 700 °C were estimated to be $110 \pm 5$ Å (at 490 °C) and 100 ± 5 Å (at 700 °C). The RBS channelling measurements showed that most of the Ge atoms occupied Si substitutional sites. The FWHM in the depth distributions of $\delta$-doped Ge in Si(111) crystals was determined to be 13 ± 5 Å. There were almost no lattice defects in the Si cap layer introduced during growth at 700°C, but there were extra displacement atoms—$(4.2 \pm 0.6) \times 10^{15}$ Si $cm^{-2}$—in the Si cap layer grown at 490°C. The surface roughnesses were estimated to be 7 ± 1 Å (at 490 °C) and 6 ± 1 Å (at 700 °C) from the AFM measurements.

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