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Characterization of thick graded $Si_{1-x}Ge_x$/Si layers grown by low energy plasma enhanced chemical vapour deposition

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Abstract

Thick, linearly graded-composition strained $Si_{1-x}Ge_x$/Si layers were recently developed for proton beam bending and extraction experiments. Such unrelaxed layers which are many microns thick necessitate a low maximum germanium content. Here, graded $Si_{1-x}Ge_x$ epilayers, 5–20 μm thick with maximum Ge compositions of $x = 0.5$–1.7%, grown by low energy plasma enhanced chemical vapour deposition were characterized using a recently developed mode of ion channeling analysis which is capable of quantifying the small lattice rotations along off-normal planar directions. High-quality 10 μm $Si_{1-x}Ge_x$ epilayers with bend angles along off-normal directions which agree very well with those of fully strained layers are successfully grown.

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1. Introduction

Channeling into mechanically bent silicon crystals have been used for bending and extraction of GeV proton beams from high energy accelerators [1–4] since they are very compact and produce a very large equivalent magnetic field strength. They are, however, limited by a minimum bending length of a few mm, which limits their efficiency at beam energies below a few GeV. An alternative scheme for bending MeV to GeV protons was recently demonstrated [5], using a graded composition strained $Si_{1-x}Ge_x$/Si layer in which the germanium content was increased linearly from zero at the silicon substrate to a maximum of x at the epilayer surface. Due to the linearly increasing tetragonal distortion, off-normal lattice planes are bent gradually with a constant radius of curvature over the full epilayer thickness. The total bend angle along the off-normal lattice planes is the same as that at the interface for a uniform composition strained layer. Incident ions which are initially channeled into off-normal {1 1 0} or

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{1 1 1} planes are steered into the corresponding substrate lattice planes with minimal dechanneling at the epilayer/substrate interface because there is no abrupt change in the lattice direction.

In a previous beam bending experiment using a graded composition $Si_{1-x}Ge_x$ layer [5], the MBE-grown epilayer thickness was 200 nm and the maximum Ge composition was $x = 15\%$, equivalent to a radius of curvature of the {1 1 1} epilayer planes close to the [1 1 2] axis of 0.05 mm. This radius of curvature was sufficiently large to bend a 2 MeV proton beam. However, for bending proton beams of energies of hundreds of MeV to a few GeV, bending radii of many millimeters are required, necessitating much thicker epilayers, which in turn requires that the maximum surface Ge composition is sufficiently low (a few per cent) to avoid strain relief.

The problem thus becomes one of growing suitably thick, graded layers which are highly perfect in a reasonable period. Growth by molecular beam epitaxy (MBE) has an upper limit of deposition rate of less than 1 nm/s, and more importantly, is subject to rapid source depletion. Hence it is too slow and expensive for layers which are many microns thick. Low energy plasma enhanced chemical vapour deposition (LEPECVD) has been used to grow thick graded $Si_{1-x}Ge_x$ layers with adequate control over the composition and a reasonably fast growth rate on the order of 3 nm/s for pure Si [6].

2. Experimental

In LEPECVD, a high density but low energy plasma is used to efficiently crack the precursor gases, such as silane and germane. For the low Ge concentrations required for the present work, a mass flow controller with a maximum capacity of 20 sccm was used for pure silane. A second one with a capacity of 4.5 sccm was used for 10% germane diluted in silane. The initial substrate temperature of 720 °C was gradually lowered to 670 °C during alloy layer growth.

A common problem using this method is Ge segregation during growth, which may lead to significant surface Ge enrichment. We have tried to overcome this with additional hydrogen during growth, at typical flow rates of 5 sccm after attaining a substrate temperature of 675 °C. Four wafers were grown with nominal epilayer thicknesses and maximum Ge compositions, either with or without addition of hydrogen during growth, as shown in Table 1.

Thick silicon–germanium layers graded to much higher Ge contents than those used here find applications in strained silicon technology [7]. For layers with low Ge concentrations an application has been suggested in the form of crystalline undulators [8], but otherwise there has been little previous interest in the growth and characterization of such thick graded epilayers. These epilayer thicknesses are all below that at which significant relaxation is expected to occur [5,9] though such a

Table 1
Sample growth conditions and the measured values

Sample	Hydrogen flow rate (sccm)	Nominal epilayer thickness (μm)	Nominal surface Ge composition x (%)	Nominal Ge grading rate (%/μm)	Minimum channeling yield, χ_{min} (%)
5787	None	5	1.7	0.340	4.5
5785	None	10	1.0	0.100	4.4
5789	10	10	1.0	0.100	4.9
5794	5	20	0.5	0.025	4.4
Sample	**Measured surface Ge composition x (%)**	**Measured Ge grading rate (%/μm)**	**Nominal rotation angle $\Delta\psi$ (°)**	**Measured rotation angle $\Delta\psi$ (°)**	**Radius of curvature (mm)**
5787	2.0	0.31	0.042	0.034 ± 0.006	10
5785	1.2	0.12	0.025	0.025 ± 0.004	28
5789	1.2	0.12	0.025	0.020 ± 0.005	36
5794	0.6	0.04	0.013	–	100

model of strain build-up was not developed for such low Ge compositions in very thick, graded layers. Hence the optimal growth conditions need to be determined, and the grown layers need to be properly characterized.

3. Results and discussions

Nomarski interference optical microscopy and atomic force microscopy (AFM) were used to assess the quality of the epilayers. Both samples 5787 and 5785 showed a smooth surface morphology across large areas with root mean square (RMS) roughness of around 1.7 nm. Across the other wafer surfaces some areas appeared smooth and other areas were rough and highly dislocated, suggesting partial relaxation of these wafers. Typical AFM images and the RMS roughness obtained from defective regions of sample 5789 and 5794 are given in Fig. 1. Cross-hatch patterns, along {1 1 0} directions, corresponding to a network of interface dislocations, were observed for sample 5789 (Fig. 1(a)) and 5794 (Fig. 1(b)) which was also decorated with pits.

Fig. 2 shows Rutherford backscattering (RBS) spectra taken with (a) 2 MeV helium and (b) proton beams in a nonchanneled alignment. In Fig. 2(a) the Ge profiles can be observed over a limited range of analytical depth of ~0.8 μm before the Si signals starts to interfere. However, the use of helium beams for analysis gives better depth resolution than for protons, enabling any Ge enrichment at the surface to be observed more easily. The Ge profile for each sample appears linear in Fig. 2(a), except for 5794 where certain regions show evidence of a Ge segregation peak at the surface, as indicated by the second spectrum from this wafer. This is interpreted as resulting from a significant lack of homogeneity across this wafer, with no regions appearing to be free of defects. The axial minimum channeling yield, χ_{min}, was measured for each sample in order to determine the crystalline quality of the upper 0.2 μm of the epilayer. The channeled and nonchanneled RBS spectrum of 5787 were shown in Fig. 2(c) and the χ_{min} values measured for all samples are given in Table 1. The low minimum yield obtained for

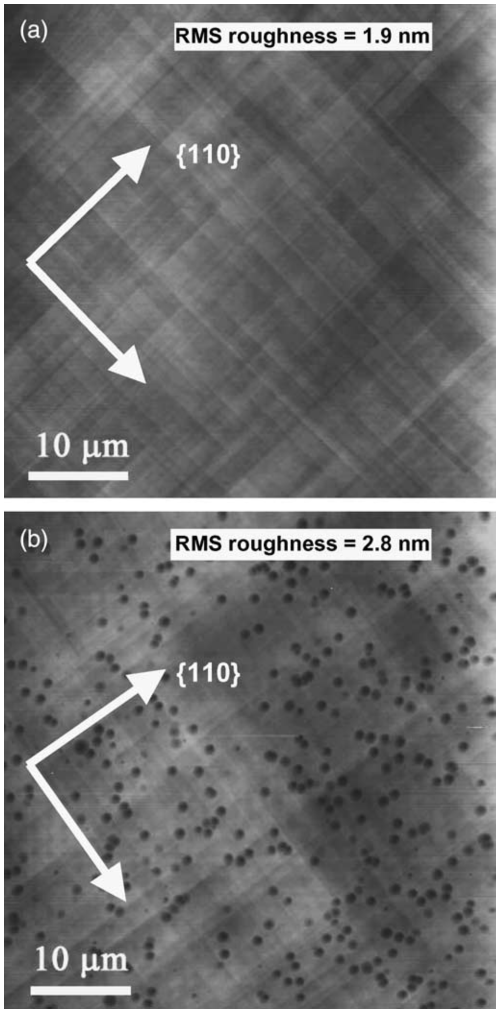

Fig. 1. AFM images of sample 5789 (a) and 5794 (b).

5794, together with poor channeling behaviour observed in Fig. 3(a) again demonstrates the wide range of crystal quality from this wafer.

The proton RBS spectra in Fig. 2(b) have a greater analytical depth of ~2.0 μm of the Ge profile, so gives a better measure of the Ge depth variation. A constant Ge concentration through the epilayer is simulated using RUMP [10] for 5787 and 5789 in Fig. 2(b), from which it can be seen that the measured Ge concentration does indeed vary linearly with depth. All the spectra were then fitted using a decreasing Ge gradient from the surface, and the simulated curve for sample 5787 is

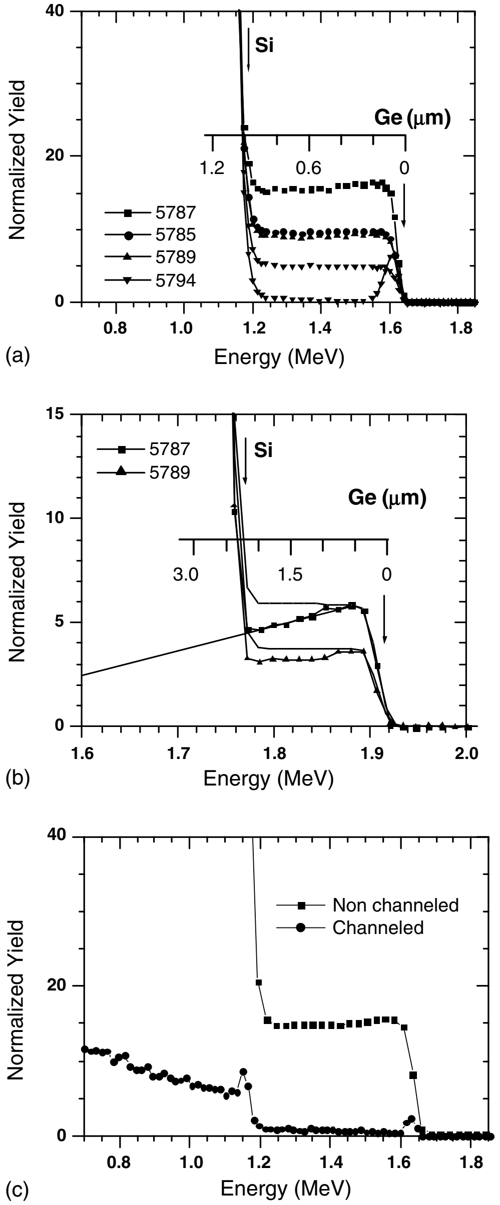

Fig. 2. RBS spectra taken with 2 MeV helium (a) and protons (b) in nonchanneled direction, with the Si and Ge surface energy and the Ge depth scales indicated. (c) 2 MeV helium channeled and nonchanneled RBS spectrum for sample 5787.

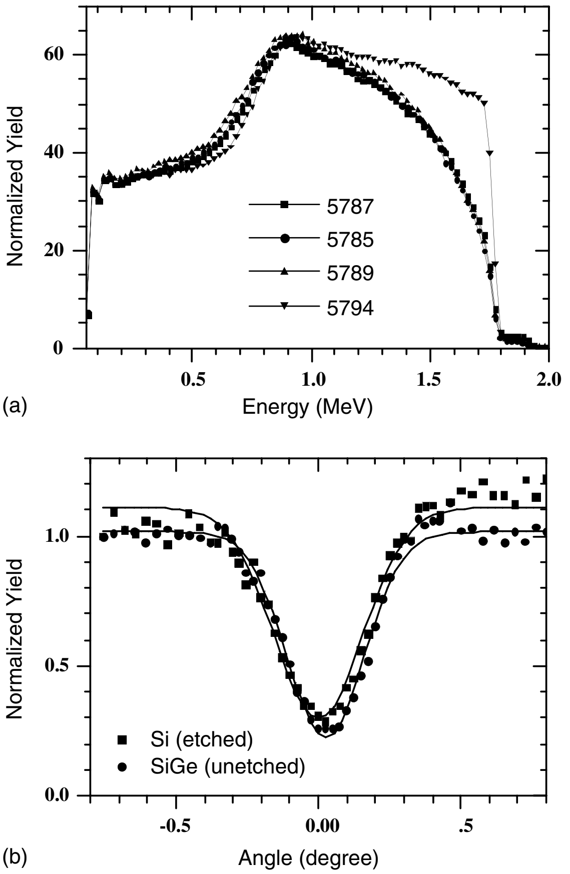

Fig. 3. (a) Proton RBS spectra for all wafers in {1 1 1} planar alignment close to [1 1 2] axis. (b) Beam rocking angular line scans taken from the underlying silicon substrate (etched) and the graded $Si_{1-x}Ge_x$ epilayer (unetched) for sample 5785. Solid lines are the gaussian fit to the corresponding data points.

shown in Fig. 2(b). The measured surface Ge compositions and grading rate for all samples shown in Table 1, are found to be in excellent agreement with the nominal values. Assuming a fully strained epilayer, the nominal angular rotations, $\Delta\psi$ along {1 1 1} planes near to the [1 1 2] axis for these measured compositions are given in Table 1. These nominal rotation angles are too small to be accurately measured using conventional RBS/channeling analysis with an unfocused, broad beam, and the thick, graded epilayer also makes measurements using X-ray diffraction ambiguous. Here we use the method described in [11] for measuring small interface rotations in strained layers. In order to measure the bend angle of the graded epilayers, a portion of the sample surface was etched to expose the underlying silicon substrate. Planar channeling angular scans recorded at alignment with the {1 1 1} planes near the [1 1 2] axis, were obtained using the beam rocking technique [12,13] with a focused 2 MeV proton beam scanning over the sample surface, without translating the sample. A scanned beam displacement of 1 mm corresponds to a change in the beam/crystal alignment of ±0.85°, compared with a critical angle of 0.16° for 2 MeV protons in Si{1 1 1} planes, so an angular channeling dip is collected simply by continuous deflection of the focused beam. The focused beam was first scanned horizontally over the $Si_{1-x}Ge_x$ epilayer region and then deflected vertically to collect similar angular line scans from the exposed silicon substrate. Since the sample remained fixed, any translations introduced by the mechanical backlash of the goniometer are avoided. Hence, small angular rotations between the epilayer and the substrate planes are able to be measured accurately.

Fig. 3(a) shows proton backscattering spectra with all four wafers in {1 1 1} planar alignment. Good planar channeling behaviour is observed from each sample, except 5794. Fig. 3(b) shows two beam-rocking angular line scans from 5785, recorded from the underlying silicon substrate (etched) and the $Si_{1-x}Ge_x$ epilayer surface (unetched). In order to accurately determine the positions of the two channeling minima, a gaussian profile is least-squares fitted to each curve. The measured rotation angles $\Delta\psi$, obtained from the difference between the two minima are shown in Table 1. Each wafer, except for 5794, gives lattice rotations which are in good agreement with the nominal values assuming a fully strained layer. Table 1 shows the radius of curvature of the {1 1 1} planes close to the [1 1 2] axis in each sample, based on the measured value of $\Delta\psi$ and the nominal layer thickness. We were not able to measure a rotation angle from 5794, since the planar channeling behaviour shown in Fig. 3(a) is very poor; Table 1 thus shows a radius of curvature for this wafer based on the nominal rotation angle, for comparison.

4. Conclusions

We have used LEPECVD to grow 10 μm thick, graded $Si_{1-x}Ge_x$ layers (wafer 5785) in which the Ge composition reaches a maximum of $x = 1.2\%$ at the epilayer surface, to give a bending radius of curvature of ~28 mm. This radius of curvature is sufficient to bend proton beams of hundreds of MeV to a few GeV. Addition of hydrogen in the growth (wafer 5789) caused no discernible improvement in the sample quality. The growth of a 20 μm thick, graded layer of ~100 mm radius of curvature was also not successful under the growth conditions used. These thicker layers were partially relaxed and contained many defects and dislocations due to strain relief. We have also demonstrated that the small angular rotations of ~0.02° of the graded epilayer surface with respect to off-normal substrate planes can be measured by a combination of beam rocking and partial etching.

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