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# High resolution channeling contrast microscopy and channeling analysis of SiGe quantum well structures

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#### Abstract

High resolution channeling contrast microscopy (CCM) and channeling measurements were carried out to characterize SiGe quantum well structures on micron thick graded layers (i.e. virtual substrates). The virtual substrates were grown by gas source molecular beam epitaxy at a pressure of  $10^{-5}$  mbar and low pressure chemical vapor deposition at  $10^{-2}$  mbar on boron doped Si(001) substrates respectively. A homoepitaxial silicon buffer layer was grown prior to the deposition. The nominal structure is a 20 nm Si<sub>0.75</sub>Ge<sub>0.25</sub> layer at the surface, followed by 10 nm pure Si, 500 nm Si<sub>0.75</sub>Ge<sub>0.25</sub> and a 1000 nm thick graded SiGe (0–26%) layer. RBS was used to measure the depth profiles, and angular scans around the (100) axis were carried out to assess crystal and interface quality. CCM was used to acquire depth resolved images of micron-sized lateral inhomogenities ('cross-hatch') present on both samples. © 2002 Elsevier Science B.V. All rights reserved.

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#### 1. Introduction

The control of the band offset at SiGe/Si heterojunctions through either strain or Ge content allows one to improve the performances of Sibased microelectronic and optoelectronic devices [1]. However, the existence of lattice mismatch in Si/Ge-based systems implies that thermally stable

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pseudomorphic layers can be grown only up to a critical thickness before misfit dislocations are introduced which relax the strain between the SiGe film and the Si substrate. Therefore, partially or fully relaxed SiGe layers grown thicker than the critical thickness contain misfit dislocations and these are detrimental to the device performance based on band gap engineering of Si/SiGe heterostructures. It has been shown that linearly graded SiGe layers followed by thick constant composition SiGe buffer layers, often termed "virtual substrates", ensure that the lattice mismatch is relaxed gradually. While this has the advantage of reducing

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the threading dislocation density in the top most part of the buffer layer, a 'cross-hatch' morphology is often generated on the surface. While the exact origin of these feature is not clear it may have detrimental effects on the subsequent processing of the device.

Because of the technological impact of the material, a substantial amount of work was and is carried out to characterize SiGe heterostructures by RBS/channeling [2–4]. Recently, microbeam techniques are also used for the characterization of SiGe thin film structures [5,6]. Here we report on high resolution channeling contrast microscopy (CCM) [7] and channeling/RBS measurements carried out in order to characterize these virtual substrates. The CCM technique allows one to image the cross-hatch features not only at the sample surface but also as a function of depth and therefore offers a unique way to investigate the way in which these patterns develop.

## 2. Experimental

Two SiGe quantum well structures on micron thick graded layers (virtual substrates) were investigated. The virtual substrates were grown by gas source molecular beam epitaxy (GSMBE) at a pressure of  $10^{-5}$  mbar (sample 832) and low pressure chemical vapor deposition (LPCVD) at  $10^{-2}$  mbar (sample 834) on boron doped Si(001) substrates respectively. A homoepitaxial silicon buffer layer was grown prior to deposition. GSMBE was then used to grow a thin Si layer followed by a thin SiGe layer on both samples. The nominal structure is 20 nm Si<sub>0.75</sub>Ge<sub>0.25</sub> layer at the surface, followed by 10 nm pure Si, 500 nm Si<sub>0.75</sub>Ge<sub>0.25</sub> and a 1000 nm thick graded SiGe (0–26%) layer.

The measurements were carried out at the nuclear microscopy facility at the National University of Singapore [8]. The recently installed 3.5 MeV Singletron accelerator [9] was used for the measurements, because it provides highly stable beam currents (typically below 1% intensity variation on a minute time scale) and very high brightness beams. The former implies that alignment procedures for channeling and CCM mea-

surements can be carried out much more efficiently than with van de Graaff-type machines, and the latter allows one to set the aperture slits so that the beam divergence is below  $0.2^{\circ}$  in both planes, i.e. reasonably good channeling conditions are achieved at sub-micron spot sizes with 100 pA beam current.

For the broad beam channeling measurements a 2 MeV He<sup>+</sup> beam of typically 5 nA was used. RBS spectra were recorded with 50 mm<sup>2</sup> PIPS detectors of 14 keV energy resolution at 160° and 110° scattering angle. Vertical slits of 2 and 3 mm widths were used to minimize geometrical straggling effects, resulting in solid angles of 6 and 6.3 msr. The samples were mounted on a eucentric goniometer that has a 14 mm translational range for both the x and y direction and allows rotations up to  $20^{\circ}$  around both the x and the y axis with a resolution of 0.1 mrad. The beam spot size was typically 300 µm. The CCM data was taken with a 300 mm<sup>2</sup> PIPS detector of 19 keV energy resolution at 145° scattering angle. A solid angle of 280 msr was used so that reasonable statistics could be accumulated during 1 h runs with typically 100 pA beam current.

# 3. Results and discussion

Figs. 1 and 2 show the results of 2 MeV broad beam RBS measurements for the two samples, together with the results of a RUMP [10] fit to the data, and the deduced sample structure is sketched as well. Because the thin surface near GeSi and Si structures are not resolved in the geometry with a scattering angle  $\theta$  of 160°, inserts show the highenergy part of spectra taken with a scattering angle  $\theta$  of 110°. In this geometry, the thin surface structures are well resolved. From the sketches of the sample structures it can be seen that the agreement with the nominal structure is good for the sample 832, on which GSMBE was used to grow the virtual substrate in a well-characterized system. The RUMP simulation was calculated assuming a linear decrease of the Ge concentration in the graded layer, in good agreement with the RBS data, as can be seen in the region between channel 85 and 140 in Fig. 1. In the case of sample

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Fig. 1. 2 MeV RBS/channeling results from sample 832.



Fig. 2. 2 MeV RBS/channeling results from sample 834.

834 the graded layer exceeds the nominal thickness substantially, this is probably due to the fact that the LPCVD system used was relatively new and not as well calibrated. Figs. 1 and 2 also show the  $\langle 100 \rangle$  axial channeled spectra. Minimum yield  $\chi_{min}$  values of the order of 10% are found for both samples, indicating that disorder and/or regions with lattice tilt are present.



Fig. 3. Angular scan through the  $\langle 1\,0\,0\rangle$  axis along a (010) direction of sample 834. Plotted are the Ge yields from the indicated depths.

Fig. 3 shows the results of an angular scan through the  $\langle 100 \rangle$  axis along a (010) planar direction of sample 834. The plot shows the Ge yield stemming from the thin surface GeSi layer and also from 60 and 200 nm sub-surface regions, i.e. from the constant concentration GeSi layer. The curves are all similar, implying a homogenous distribution of the disorder in the lattice at least up to 200 nm. The data for sample 832 (not shown) was found to be quite similar to that from sample 834.

The Figs. 4 and 5 display the CCM results. Shown are the total spectra of  $\langle 100 \rangle$  axially channeled  $100 \times 100 \ \mu\text{m}^2$  scans, together with images generated from the windows shown (designated 1, 2 and 3 in Figs. 4 and 5) in the spectra. The cross-hatch structure, bands of high channeling yield, one to a few  $\mu$ m wide, running along the (010) and (001) planar directions, are clearly visible in the CCM maps. These structures are found in the maps generated from the windows 1 and 2, generated by Ge-backscattered particles from the constant concentration layer and also in window 3, stemming from the graded layer. This shows that the cross-hatch present in the surface



Fig. 4. CCM results from sample 832. Plotted is the total spectrum and images generated from the indicated windows.

morphology of the samples originates in the graded layer.

The origin of the cross-hatch seen in Figs. 4 and 5 is not fully understood, one possibility is the presence of regions where a tilt in the lattice planes is present. Fig. 6 shows CCM maps taken from the LPCVD grown sample 834, plotted is the region 2 (see Fig. 5). Together with the  $\langle 100 \rangle$  map two additional images are shown, taken with the sample rotated by  $\pm 0.2^{\circ}$  along the (010) planar direction. The misalignment of the three maps stems from the fact that it is difficult to position the samples precisely at the eucentric point of the goniometer, so that slight lateral shifts occur under rotations. A reversal in the contrast can be seen in the maps, which is consistent with the presence of lattice tilt of around 0.4°. Work is under way to measure a full angular scan, which will reveal the exact amount of tilting. In the

GSMBE grown sample 832 no contrast change was detected under identical conditions, again a full angular scan is needed to will clarify the situation.

#### 4. Conclusion

RBS/channeling and high resolution CCM were used to characterize SiGe quantum well structures on micron thick graded layers (i.e. virtual substrates). The virtual substrates were grown by GSMBE and LPCVD on Si(001) substrates. RBS was used to measure the depth profiles, and angular scans around the  $\langle 100 \rangle$  axis were carried out to assess crystal and interface quality. CCM was used to acquire depth resolved images of micronsized lateral inhomogenities ('cross-hatch') present on both samples. These structures are associated



Fig. 5. CCM results from sample 834. Plotted is the total spectrum and images generated from the indicated windows.

effect of lattice tilt sample 834



Fig. 6. CCM maps from sample 834, plotted is region 2 (see Fig. 5). Shown are maps generated with  $\langle 100 \rangle$  alignment and with the sample rotated by  $\pm 0.2$  along the (010) planar direction.

with the presence of slight lattice tilt in the case of the LPCVD grown sample.

### References

- G.D.M. Dilliway, A.F.W. Willoughby, J.M. Bonar, J. Mater. Sci. 11 (2000) 549.
- [2] S.T. Picraux, B.L. Doyle, J.Y. Tsao, Semiconduct. Semimet. 33 (1991) 139.
- [3] N.P. Barradas, C. Jeynes, O.A. Mironov, P.J. Phillips, E.H.C. Parker, Nucl. Instr. and Meth. B 139 (1998) 239.
- [4] A. Rodriguez, T. Rodriguez, A. Kling, J.C. Soares, M.F. da Silva, C. Ballesteros, Nucl. Instr. and Meth. B 136–138 (1998) 395.
- [5] A. Simon, Z. Kántor, Nucl. Instr. and Meth. B 190 (2002) 351.

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- [6] T. Winzell, J. Pejnefors, M. Elfman, M. Ostling, H.J. Whitlow, Nucl. Instr. and Meth. B 179 (2001) 21.
- [7] M.B.H. Breese, D.N. Jamieson, P.J.C. King, Materials Analysis Using a Nuclear Microprobe, John Wiley & Sons, New York, 1996.
- [8] F. Watt, I. Orlic, K.K. Loh, C.H. Sow, P. Tong, S.C. Liew, T. Osipowicz, T.F. Choo, S.M. Tang, Nucl. Instr. and Meth. B 85 (1994) 708.
- [9] D.J.W. Mous, R.G. Haitsma, T. Butz, R.H. Flagmeyer, D. Lehmann, J. Vogt, Nucl. Instr. and Meth. B 130 (1997) 31.
- [10] L.R. Doolittle, Nucl. Instr. and Meth. B 9 (1995) 344.