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Probing the interfacial and sub-surface structure of Si/Si$_{1-x}$Ge$_x$ multilayers

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The ability to determine structural and compositional information from the sub-surface region of a semiconductor material has been demonstrated using a new time-of-flight medium energy ion scattering spectroscopy (ToF-MEISS) system. A series of silicon–silicon/germanium (Si/Si$_{1-x}$Ge$_x$) heterostructure and multilayer samples, grown using both solid source molecular beam epitaxy (MBE) and gas source chemical vapor deposition (CVD) on Si(100) substrates, have been investigated. These data indicate that each individual layer of Si$_{1-x}$Ge$_x$ ($x\sim0.22$) in both two- and three-period samples, can be uniquely identified with a resolution of approximately 3 nm. A comparison of MBE and CVD grown samples has also been made using layers with similar structures and composition. The total Ge content of each sample was confirmed using conventional Rutherford backscattering spectrometry. © *1995 American Institute of Physics.*

The ability to determine structural and compositional information from the sub-surface (or near-surface) region of a semiconductor material is extremely important for the investigation of epitaxial layered structures grown by techniques such as MBE and CVD. A large array of experimental techniques already exist capable of giving high quality structural and chemical information, although probably the most widely used for the investigation of semiconductor materials are x-ray diffraction (XRD) for structural analysis and secondary ion mass spectrometry (SIMS) for chemical analysis. However, these techniques have limitations if the layered structure to be investigated is in the near-surface region, i.e., less than 50 nm below the surface of the material. Both of these techniques require a significant depth of material prior to the structure to be studied. In the case of XRD, the layers must occupy some fraction of the extinction distance, and for SIMS, the sputtering process must reach an equilibrium. Also, although a number of surface structural and chemical techniques [e.g., Auger spectroscopy (AES) and x-ray photoelectron spectroscopy (XPS)] are available, their penetration distance into a material is limited by the mean-free path of the excited electrons and therefore they sample only the surface, i.e., the top 1–2 nm, of the material. Clearly, there is a gap or region of material from which structural and chemical information is somewhat more difficult to obtain.

Medium energy ion scattering spectroscopy (MEIS; 5–50 keV) has for several years been used in the study of surface and near-surface structure.[1,2] However, the recent development of several forms of pulsed ion beam techniques in combination with time-of-flight detection has led to an increased use, particularly of MEIS, as a means of studying sub-surface structure and composition (both *in situ* and *ex situ*) in a non-destructive manner.[3,4] Also, since time-of-flight detection methods are sensitive to both charged and neutral scattering products, it is possible to make use of the majority neutral fraction produced as a result of the interaction between ions and a surface.[5,6] In this letter a series of Si/Si$_{1-x}$Ge$_x$ multilayer samples grown on Si(100) substrates, using both solid source MBE and CVD, have been investigated to demonstrate that structural and compositional information can be obtained from the top 25 nm of these materials.

The MEISS instrument used for these measurements was developed at AEA Technology and is shown schematically in Fig. 1. Ions are generated in a duoplasmatron ion source at a chosen energy in the range 5–30 keV and directed, via a hole in the detector, onto the sample in 10–50 ns pulses by electrostatic switching of the ion beam through a small aperture. A small angular off-set (~2°) serves to remove any background from neutral species eminating from the ion source. The scattering products are detected using a coaxial micro-channel plate detector, and the configuration of the complete ToF-MEISS system is small and compact. Also, since this instrument only requires line-of-sight to the sample, it can be attached to a growth chamber, via a conventional 70 mm flange, to provide *in situ* real-time analysis during thin film growth.

All the samples discussed in this letter were grown on lightly doped Si(100) *p*-type substrates. The MBE samples were grown in a VG V90S reactor using solid sources of Si and Ge, whereas the CVD sample was grown in a custom built low-pressure CVD system using silane and germane sources. Buffer layers of Si and the SiGe layers were grown at 1123 and 883 K, respectively, for the CVD grown sample and at 770 K for the MBE grown samples. All the layers studied were terminated with a 2.5 nm silicon cap.

An example of the time-of-flight spectrum recorded from a three-period (2.5:2.5 nm) Si/Si$_{0.78}$Ge$_{0.22}$ multilayer structure grown by MBE and taken using a 21 keV beam of helium ions incident at an angle of 45° is shown in Fig. 2. The three peaks observed at short flight times (1375, 1400, and 1425 ns) are clearly resolved and assigned to the Ge content present in each of the SiGe layers (the Ge content was independently determined at 22%±2%). The measured signals from each of the three Ge layers are displaced due to the energy loss of the primary ions in passing through the intervening material. Hence, the horizontal time-of-flight scale can, in principle, be related to a depth scale within the

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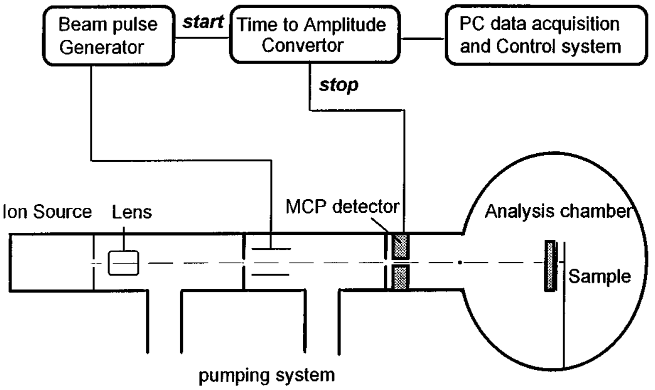

FIG. 1. Schematic of the time-of-flight medium energy ion scattering (ToF-MEIS) spectrometer and data collection system.

material and the scattered yield is proportional to the concentration of the species in the sample matrix. The separation of the Ge features can be used to calculate a value for the thickness of the layers based on the stopping power of He in a solid.[7] The observed Ge peaks are separated by some 25 ns, which equates to an energy difference of the He scatter products of some 1400 eV. In silicon, this energy loss would occur due to the passage of ions through approximately 20 nm of material (stopping power ~75 eV/nm). Since the measurement was performed at 45° incidence to the surface, this penetration represents a depth of approximately 6 nm and is close to the anticipated Ge layer separation of 5.0 nm, calculated on the basis of the calibrated growth rates. For deeper layers, an increase in the Ge peak width is also observed as a result of energy straggling effects due to statistical fluctuations for inelastic loss events. The silicon signal appears as an edge in the spectrum at a later time (~1480 ns). Alignment of the incident ion beam with a principal axis of the material, such as the $\langle 110 \rangle$ axis of the Si(100) substrate, results in the vast majority of ions traveling down the open channels of the single crystal lattice. This channeling effect dramatically reduces the probability of a scattering collision, and hence there is a large reduction in the overall backscattered yield. If amorphous layers are present in the sample this effect will not occur, and hence from the data of Fig. 2, it can be seen that the majority of the grown structure is indeed single crystal. What is delineated, however, is the thin amorphous oxide which grows on the sample surface when exposed to the atmosphere prior to analysis. This gives rise to the small peaks observed in the channeled spectrum, at 1480 and 1640 ns, respectively, which correspond to the time for helium scattering from silicon and oxygen at the surface. The measured full width at half-maximum (FWHM) of the silicon surface peak corresponds to a depth of some 3 nm and can be taken as a practical measure of the depth resolution of the technique in this configuration. However, a small feature at 1360 ns also indicates the presence of surface germanium. If the sample was indeed composed as is shown at the top of Fig. 2, the outermost layer should contain no germanium and the thickness of the native oxide of silicon might be expected to consume only a small fraction of the silicon cap. However, the fact that this germanium feature appears in all the spectra from samples with a silicon cap thickness <10 nm at least indicates the possibility of Ge segregation towards the surface.[8]

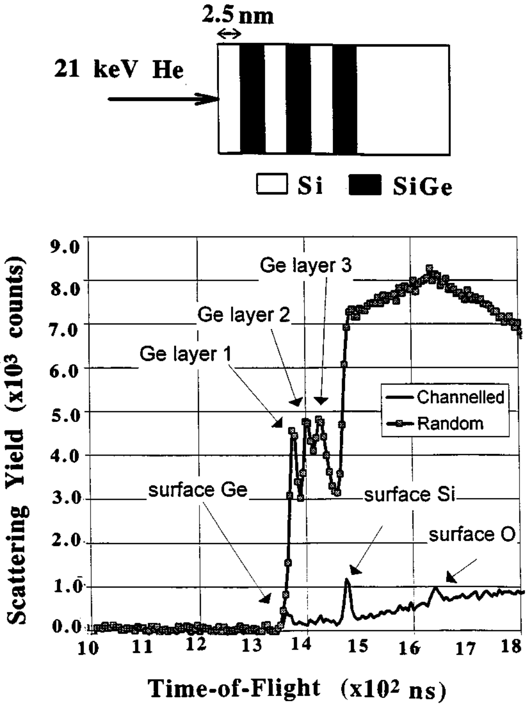

FIG. 2. ToF-MEIS data obtained from a three-period Si/$Si_{1-x}Ge_x$ superlattice (x=0.22) with each layer being only 2.5 nm thick grown on a Si(100).

A comparison has also been made of two SiGe containing samples, grown by MBE and CVD, respectively, to the same specification, in an attempt to determine whether the ToF-MEISS technique could identify any structural and composition differences between them. The structure chosen was grown on a thick Si buffer layer and consisted of two $Si_{1-x}Ge_x$ layers 5.0 and 2.5 nm thick, separated by a 5.0 nm Si layer and capped with a Si layer 2.5 nm thick. Normalized time-of-flight spectra taken with a 21 keV incident He ion beam from each of these samples are shown in Fig. 3. These data indicate the presence of significantly more Ge within the SiGe layers of the MBE grown sample than in the CVD grown sample, although the widths of each peak, at FWHM, are comparable. There are also slight variations in the flight times associated with the two germanium features which would tend to imply that the silicon cap thickness is slightly greater for the MBE grown sample. This difference in Ge content between the two samples is confirmed by the RBS data, shown in Fig. 4. A quantitative measure of the total Ge content in the layers,[9] indicates that there is 2.4±0.2 times as much Ge in the MBE sample than the one grown by CVD. This difference in the Ge concentration of the two samples may reflect either the differences in the detailed kinetics for Ge incorporation in the two growth techniques (for CVD growth the rate at which Ge is incorporated into a growing layer differs as the Ge mole fraction increases), or it may simply reflect a difference in the growth rate calibrations. An approximate ratio of the Ge content for the two samples of 2.1±0.2, in reasonable agreement with the RBS data, can be extracted from the Ge peak areas of the ToF-MEIS spectra in Fig. 3. However, the question still remains as to whether the

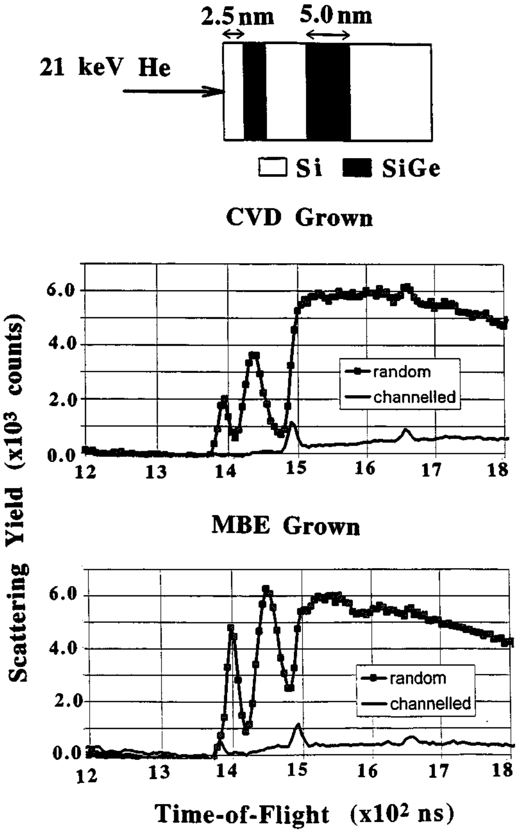

FIG. 3. ToF-MEIS data obtained from two samples with the same layer structure each containing a 2.5 nm and a 5.0 nm Si$_{1-x}$Ge$_x$ layer (target value of $x=0.22$) separated by a 50 nm Si layer and capped by a 2.5 nm layer.

increased Ge signal for the MBE grown sample (compared to the CVD sample) is simply a result of a higher Ge concentration or thicker Ge containing layers. X-ray diffraction data, based on calibration samples grown by MBE, indicates that the two Ge containing layers in the MBE grown sample are separated by a silicon layer 5.4 ± 0.5 nm thick. In addition, since the peak positions and center-to-center separation of the two Ge features in Fig. 3 are approximately the same for both MBE and CVD samples, the layer thicknesses in both must be similar. The majority of the difference between the two samples must therefore be a result of increased Ge concentration.

Channeling data in the $\langle 110\rangle$ direction was also obtained from both of these samples and again shows a high degree of epitaxy and the presence of silicon and oxygen features. However, in these channeled spectra, a surface germanium feature was observed on the MBE grown sample but not for the CVD grown sample. An estimate, from the data in Fig. 3, of the concentration corresponding to this surface feature indicates the presence of $\sim$0.25 monolayers of Ge. Random and channeled spectra were also taken from all of the samples thus far discussed following a HF dip to remove the amorphous oxide layer. In all cases the surface germanium feature also was removed following the HF dip. Had this feature resulted from Ge segregation to the surface from the layer below, it might be expected that a detectable Ge signal would be seen throughout the silicon capping layer.[10] Since no such signal was observed in any of the samples studied, the most likely explanation of this feature is a small quantity of Ge leaking past the shutter covering the appropriate Knudsen source on the MBE reactor during the long substrate cool-down period. This would produce a surface layer rich in Ge at the end of the growth cycle.

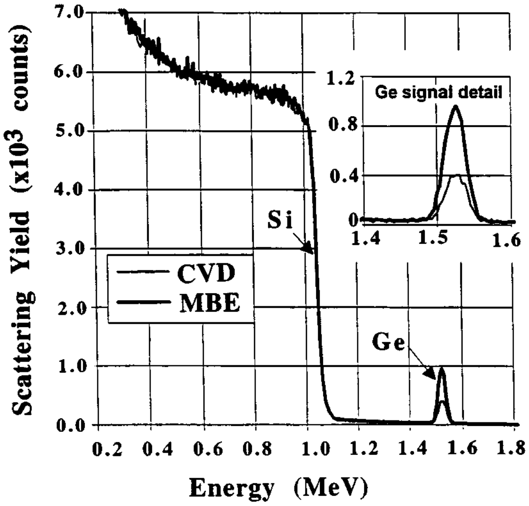

FIG. 4. RBS data comparing the Ge content of the CVD and MBE.

In summary, we have demonstrated that the ToF-MEISS technique can be used to obtain both structural and compositional information from the subsurface region, up to 25 nm below the surface, of a semiconductor material. Examples from a series of Si/SiGe multilayer samples grown by MBE and CVD have been used to demonstrate the non-destructive depth profiling capability of this technique. This ability to obtain structural and composition information, in a region normally inaccessible to the more conventional bulk and surface analysis techniques, should therefore prove to be of considerable value as an *in situ* diagnostic technique for both MBE and CVD growth techniques.

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