Resonant scattering in delta-doped heterostructures

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We demonstrate the utility of resonant x-ray scattering in probing the structure of doping layers at a heterostructure interface. The positions of germanium layers inserted at the interface of a silicon epitaxial film assert a strong influence of the phase of the scattered intensity along the crystal truncation rods. The phase of the scattering, and hence the internal structure of the layers, can be determined conveniently by analyzing its energy dependence in the vicinity of the Germanium absorption edge at 11.103 keV. © *2001 American Institute of Physics.* [DOI: 10.1063/1.1415352]

Considerable importance lies in the details of the atomic structure of heterointerfaces. Silicon/germanium interfaces are particularly important because of their application in germanium quantum-well devices used to make high speed transistors. In these, a thin slab of Ge is inserted into a Si crystal during epitaxial growth, with careful control of the growth conditions to minimize the interdiffusion. When the thickness of the inserted layer approaches a single monolayer, the term ''delta doping'' has been adopted. In these structures, it is necessary to bury the quantum well sufficiently deeply inside the semiconductor material that it is not affected by the surface modifications that are associated with the subsequent processing of the material to fabricate devices.

While the structure of Ge layers at Si *surfaces* can be analyzed by a wide variety of surface-science techniques, including x-ray diffraction, the options available for buried *interfaces* are more limited. X-ray diffraction is one of the few techniques with sufficient penetration to reach a deeply buried interface. However, for a Si–Ge–Si heterostructure, the main influence on the resulting diffraction pattern is due to the enlarged spacing of the interface. Such a structure produces strong fringes of intensity along the crystal truncation rods due to diffraction from the surface. The enlarged spacing causes a phase shift between the layers above the doped layer(s) and those below, which causes the fringes. While the spacing change can be determined very accurately, the individual contributions of the Ge layers to the diffraction pattern are relatively small, which means their positions and any detailed structural changes among them are harder to identify.

Crystal truncation rods (CTRs) are continuous lines of diffracted intensity following the direction of the normal to a well-defined surface.[1] An analysis of the distribution of intensity along CTRs yields an interface structure at the atomic level. This method has been extensively used to reveal doping profiles in InAs/InPAs-type heterostructures, where the lattice distortions are minimal and only the chemical distribution can provide contrast.[2]

In this letter, we demonstrate that the relative phase of the Ge contribution to the Si part of the heterostructure can be determined in a straightforward way by the use of the resonant scattering when the beam energy is tuned through the Ge K absorption edge at 11.103 keV. Since the diffraction phase of the thick Si slab varies rapidly with momentum transfer, the shape of the resonance of the Si–Ge–Si heterostructure will be correspondingly sensitive. We will show that dramatically different resonance profiles can arise as the relative phase is tuned arbitrarily.

The sample was grown in a Vacuum Generators V-80 molecular-beam epitaxy system with a base pressure of less than 10^{-10} Torr. A Si(100) wafer was first cleaned by heating at 800 °C to remove the protective oxide. A 700 Å undoped Si buffer layer was then grown at 700 °C. Two monolayers of Ge were then deposited at 275 °C on this substrate, followed by a 100 Å thick Si capping layer. The substrate temperature was ramped from 275 °C to 350 °C during the growth of the cap in order to minimize the interdiffusion. The surface morphology of the Ge layer was monitored during the growth using reflection high-energy electron diffraction and showed a 2×1 surface reconstruction.

Measurements were made at the X16C bending-magnet beamline of the National Synchrotron Light Source (NSLS) at Brookhaven National Lab USA. A sagittally focusing double Si(111) monochromator produced a 0.5×1.0 mm spot on the sample at the center of the Kappa diffractometer.[3] The measurements were made in the symmetric, $\omega = 0$, geometry in order to maximize the penetration to the interface inside the sample. Detector slits of 2×4 mm at distance of 600 mm were sufficient to suppress fluorescent and resonant-Raman contributions to the intensity without the need for an analyzer crystal. The surface sensitivity comes from selection of surface-specific features in the diffraction pattern, such as
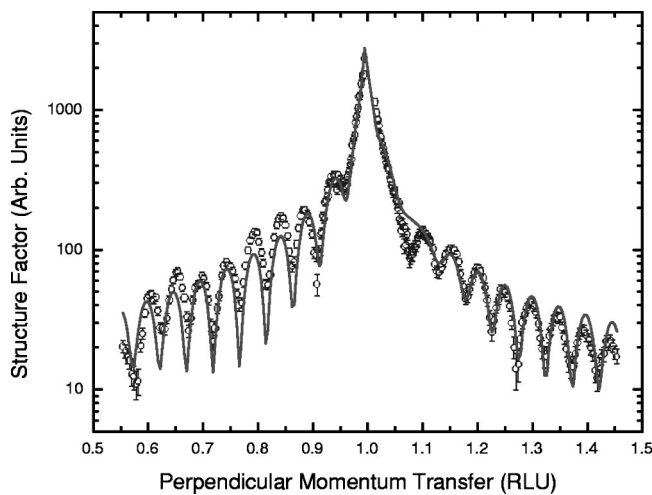
FIG. 1. Structure factor along the $(1,1,L)$ crystal truncation rod measured at $E=11.0$ keV as a function of perpendicular momentum transfer, L. The fit curve comes from a simple structure model based on the composition of the sample with adjustable layer-spacing parameters, d_{Ge} and d_{Si}, at the interface.
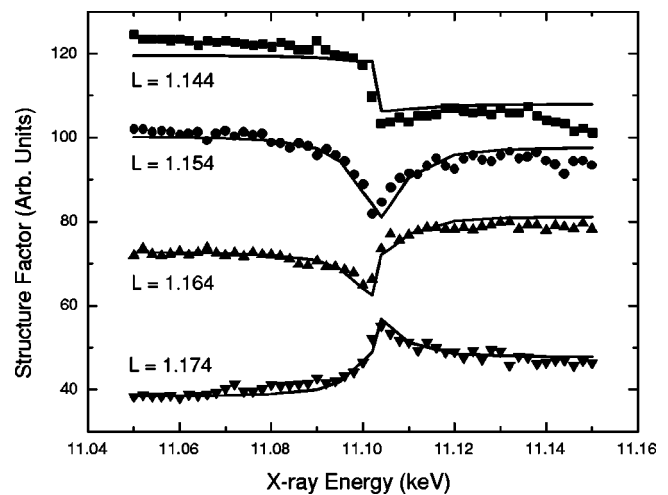

FIG. 2. Resonant scattering measurements at different points along the $(1,1,L)$ crystal truncation rod. The data are corrected for monochromator normalization, background subtracted, and converted to a structure factor. Fit curves are generated using Eq. (1). The curves are offset for clarity.

CTRs. The sample alignment, by means of two Bragg reflections, was sufficiently accurate to measure the intensity along the $(1,1,L)$ CTR as a single scan. Following convention, L denotes the continuous-valued coordinate of momentum transfer perpendicular to the surface of the sample. A q^{-2} intensity background, estimated to represent the thermal diffuse scattering of the substrate, was subtracted. The square root of the resulting intensity was taken to be proportional to the CTR structure factor without any corrections for dynamical effects or absorption. Fitting of this structure factor was achieved by an appropriately modified version of the program ROD.[4]

Figure 1 shows the measured structure factor of the $(1,1,L)$ rod passing through the $(1,1,1)$ bulk Bragg peak. The shape of this curve can be understood to arise from three components interfering with each other: the first is the CTR of the substrate with its characteristic divergence at the Bragg peak and q^{-1} amplitude tails; the second is the thick slab of N layers of Si on top, which gives rise to the fringes of spacing $\Delta L=1/N$. The interference between these first two components is highly sensitive to the interfacial separation: when the separation is zero, the fringes will vanish; when this is half a layer spacing, their amplitude will be a maximum. The phase of the sum changes continuously in L, varying by 2π for every fringe spacing, ΔL. The third component in the sum is the Ge layer at the interface, which, being a thin layer, has a broad featureless structure factor over the whole range of interest in Fig. 1. The phase of the Ge component in the sum is sensitive to the position of the layers as well as, importantly, the x-ray energy near the Ge K edge.

A simple model is used to obtain a crude fit to the measurements of Fig. 1. The model consists of a Si(100) bulk followed by two monolayers of Ge, then 83 monolayers of Si. A second copy of this film structure, raised one monolayer higher, was added with an (intensity) weight of one half. This accounts for the two-fold symmetry of the Si(100) surface due to its directional bonding by creating a four-fold symmetric diffraction pattern, as observed. The only param-

eters in the model were an overall roughness parameter $\beta=0.5$,[1] a variable occupancy of $\theta_{83}=0.13$ of the top-most Si layer, an occupancy of $\theta_{Ge}=0.77$ of the two Ge layers and vertical displacements d_{Ge} and d_{Si} of the Ge and Si layers, moving together as a block. The θ_{Ge} parameter represents substitutional disorder of the Ge layers, probably caused by interdiffusion during growth. The θ_{83} parameter represents the nonintegral number of Si layers grown. This model is a simplified version of that used previously to fit delta-doped structures.[5]

The two structural parameters are d_{Ge} and d_{Si}. The value of $d_{Si}=0.041$ unit cells or 0.23 ± 0.01 Å corresponds to an average 5.5% expansion of the three layer spacings at the interface, consistent with the tetragonal distortion expected there and the lattice mismatch of 4.2%. The model is also sensitive to the positions of the Ge layers but, because these are coupled to all other parameters in the model, the best fit is not very reliable. The best fit of the height of the two Ge layers, $d_{Ge}=0.002\pm0.002$, implies a slight asymmetry between the two Si–Ge interfaces in the structure. Although asymmetry is not expected in a chemically symmetric structure, it can appear because of different degrees of interdiffusion on the two sides of the interface, arising from the growth conditions. Nevertheless in the fit, the value of d_{Ge} was also found to be quite strongly coupled to the other parameters of the model.

Figure 2 shows the main result of this work, the energy dependence measured at strategic positions along the rod, where the phase of the Si part of the structure takes very different values. The structure factor has been derived from the measured intensity as just described. A linear correction for the variation of the monitor normalization with energy has been applied, the same for all curves. It is immediately obvious that the shape of the resonance is quite different for the four L-values shown. At certain L's, the resonance appears as a step, either up or down; at other L's, it appears as a cusp, again either up or down. This is because the phase of the Si part of the structure factor selects various combinations of the real or imaginary part of the Ge atomic form factor in the sum. Apparently, the shape of the resonance

observed can be tuned continuously by the choice of perpendicular momentum transfer, L.

To model the spectra, we use an approximate description of the resonant Ge atomic form factor as a function of photon energy, E, using a sum of a Lorentzian and a step function,

$$f(E) = f_0 - \frac{f_1}{1 + \left(\dfrac{(E-E_0)}{W}\right)^2} + if_2\Theta(E-E_0), \qquad (1)$$

where $f_0 = 27.39 + 0.51i$ electrons[6] is the nonresonant form factor of Ge at $|q| = 1.9$ Å^{-1}, $f_1 = 8$ electrons, and $f_2 = 3.38$ electrons.[5] $\Theta(x)$ is the step function having value unity only for $x > 0$. The values of f_1, the edge position, $E_0 = 11.103$ keV and Lorentzian width $W = 6$ eV, which account mainly for the resolution and calibration of the monochromator, were estimated during fitting the data, while the imaginary components were taken from standard tables.[6]

We used the resonant form of Eq. (1) directly in the ROD model, used to fit the data of Fig. 1, to calculate the E dependence of the structure factor, without adjusting any parameters. The result is overlaid on the spectra of Fig. 2. Because the fit in Fig. 1 is not perfect at the L values chosen, the curves in Fig. 2 were shifted by a scale factor as well as offset for clarity. The important point is that the *shapes* of the resonances are well reproduced by the calculation.

Since it is the shape of the resonance that senses the relative phase of the Si and Ge parts of the structure, this is the part that should be sensitive to the position of the Ge layers in the heterostructure. This is illustrated in Fig. 3 for the case of the $L = 1.144$ spectrum from the top panel of Fig. 2. $L = 1.144$ apparently produces an almost perfect step shape to the resonance, which is reproduced by the position $d_{Ge} = +0.00$ unit cells in the original model. However, as soon as displacements of the two Ge layers are introduced, curved edges start to appear on the step, because the f_1 resonant term starts to contribute. The fact that the data appear to agree better with the curve for $d_{Ge} = -0.02$ probably arises from the imperfect fit in Fig. 1 or an alignment error in the determination of the exact L value.
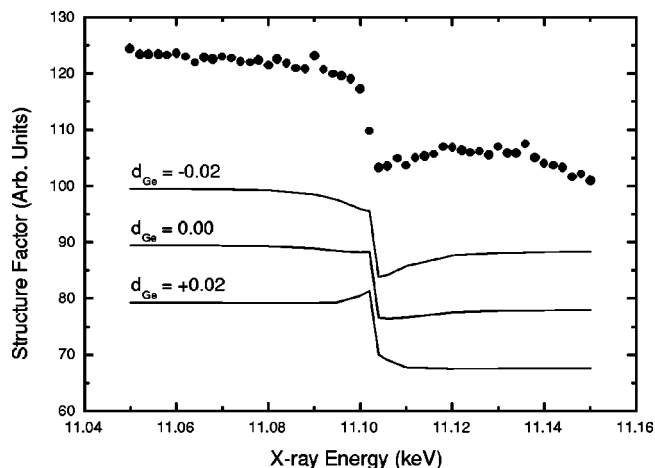

FIG. 3. Sensitivity of the resonant structure factor to the position of the germanium bilayer, d_{Ge}, in units of bulk Ge unit cells ($a_0 = 5.66$ Å). Data (symbols) and calculated structure factors (lines) for the $L = 1.144$ spectrum are offset for clarity.

Depending on which L value is chosen for measuring the resonant spectrum, the interference curve can be made sensitive to the position of the Ge relative to different components of the Si structure: when L is near the bulk Bragg peak, the CTR dominates the structure factor and the Ge distance to the bulk will be probed; when L selects the fringes (as in Fig. 3) where the CTR is relatively weak, the Ge distance to the Si film will be probed instead.

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