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Monitoring growth with X-ray diffraction

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With the introduction of high intensity synchrotron radiation sources, X-ray diffraction has become established as a powerful probe of surface crystallography. Many surface structures have been solved with progressively larger unit meshes. More recently, the technique has been extended to provide real time monitoring of surfaces during epitaxial growth. The weak scattering of X-rays allows a simple interpretation of measured intensities through the kinematic approximation, thus giving a direct and immediate insight into the atomic geometry. The technique is not limited to the solid–vacuum interface and has potentially wide application. Thus it may be used to monitor the growth in a OMVPE reaction chamber and can probe atomic arrangements at buried interfaces such as a delta doping layer. The use of X-ray scattering for the study of epitaxial growth on semiconductor surfaces is reviewed with examples taken from recent work.

1. Introduction

The characterization of the atomic structure of semiconductor surfaces and interfaces is of considerable fundamental and technological interest. Methods, such as molecular beam epitaxy (MBE) and organo-metallic vapour phase epitaxy (OMVPE), are now widely used to produce structures of novel design and unprecedented perfection. The details of the atomic mechanisms involved, how they are related to the surface reconstructions, the presence of steps and other morphological features, are, nevertheless, still not well understood. A precise knowledge of the atomic structure of semiconductor surfaces during growth will lead to a better control of the complex processes and thus to the development of improved semiconductor devices.

With the introduction of intense sources of synchrotron radiation, X-ray diffraction has, in the past ten years, become established as a powerful probe of the atomic structure at surfaces and interfaces (Feidenhans'l 1989; Robinson & Tweet 1992). The power of the technique derives from the weak scattering of X-rays by matter. This allows a simple data analysis based on a Fourier sum of plane waves which gives a direct and immediate insight into the atomic geometry. The ability of X-rays to penetrate matter further means that the technique is not limited to the vacuum–solid interface but can be used in the hostile environment of an OMVPE reaction chamber and offers a method of probing layers buried deep within a solid structure. It is characterized by high momentum resolution enabling positional correlations to be monitored over distances in excess of the largest domain size expected on a solid surface.

In the following I will outline the principles of the technique and describe its application to the study of epitaxial growth on semiconductor surfaces with examples from recent work including real time studies.

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2. Fundamentals

In surface X-ray diffraction (SXRD), a highly collimated beam from a synchrotron or storage ring is made incident on a surface and the intensity of the elastically scattered radiation is recorded as a function of the momentum transfer $\boldsymbol{Q} = \boldsymbol{k}_f - \boldsymbol{k}_i$ (Norris *et al.* 1992). At incident and exit grazing angles well above the critical angle for total external reflection, typically less than 0.4° the weak scattering (kinematic) approximation applies. Accordingly, the scattered amplitude is written (ignoring prefactors) as a simple Fourier transform of the electron density $\rho(\boldsymbol{r})$:

$$E(\boldsymbol{Q}) = \int \rho(\boldsymbol{r}) \exp(-\mathrm{i}\boldsymbol{Q}\cdot\boldsymbol{r})\,\mathrm{d}\boldsymbol{r}. \tag{1}$$

For a regular array of unit cells, defined by the base vectors $\boldsymbol{a}_i$ (usually defined in terms of the surface periodicity) and volume V_u, this becomes

$$E(\boldsymbol{Q}) = \frac{1}{V_u}[\boldsymbol{F}(\boldsymbol{Q}) \sum_{\boldsymbol{H}} \delta(\boldsymbol{Q}-\boldsymbol{H})] * S(\boldsymbol{Q}). \tag{2}$$

Q is expressed in the usual way as

$$\boldsymbol{Q} = h\boldsymbol{b}_1 + k\boldsymbol{b}_2 + l\boldsymbol{b}_3, \tag{3}$$

where $\boldsymbol{b}_j$ are the base vectors of the reciprocal lattice related to that of the bulk by $\boldsymbol{a}_i \cdot \boldsymbol{b}_j = 2\pi\delta_{ij}$. The reciprocal lattice vector $\boldsymbol{H}$ corresponds to integral values of the Miller indices h, k and l. $\boldsymbol{F}(\boldsymbol{Q})$ is the structure factor for the unit cell which may be written in terms of the atomic form factors $\boldsymbol{f}_i$ as

$$\boldsymbol{F}_{hkl} = \sum_i \boldsymbol{f}_i \exp(-2\pi\mathrm{i}(hx_i + ky_i + lz_i)). \tag{4}$$

$S(\boldsymbol{Q})$ is the Fourier transform of the shape function $s(\boldsymbol{r})$ $(0 \leqslant s(\boldsymbol{r}) \leqslant 1)$, which defines the physical extent of the ordered region (domain).

For an idealized bulk terminated surface, $s(\boldsymbol{r})$ is a step function and the Fourier transform varies as $1/q_3$ where $\boldsymbol{q}_3$ is the component of $\boldsymbol{Q} = \boldsymbol{q}_1 + \boldsymbol{q}_2 + \boldsymbol{q}_3$, which is normal to the surface. Equation (2) states that each reciprocal lattice point due to the bulk is extended along the surface normal and the total scattering is the sum of contributions from all Bragg points. The continuum of scattering along the normal is referred to as a 'streak' (Andrews & Cowley 1985) or crystal truncation rod (CTR, Robinson 1986). Real surfaces are not flat but contain steps, kinks, islands and other morphological defects which modify the CTR from this simple form.

A reconstructed surface layer is characterized by a surface mesh which has in-plane base vectors which are larger (usually by a simple multiple) than those of the underlying bulk. Since there is no periodicity perpendicular to the surface, the scattering amplitude is finite along rods which are again continuous but vary relatively slowly with a periodicity which reflects the number of bulk planes involved in the reconstruction.

The total scattering amplitude is given by the sum of surface and bulk contributions:

$$E(\boldsymbol{Q}) = E^{\mathrm{surf}} + E^{\mathrm{bulk}}. \tag{5}$$

Along fractional order rods (corresponding to fractional values of h and k) $E^{\mathrm{bulk}} = 0$ and the scattered amplitude is determined solely by the surface arrangement. The

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integral order reflections, by contrast, contain scattering from the bulk and the surface. The interference between the two terms is determined by the atomic arrangement within the surface region and to the positions of the surface atoms relative to the underlying substrate. Thus, the well-ordered bulk structure provides a 'template' with which the surface structure can be determined. By recording several integral order rods, a detailed picture of the atomic ordering at a surface in equilibrium and during growth can be built up (van Silfhout *et al.* 1990).

3. X-ray scattering from rough surfaces

The manner of the growth depends on the presence of steps, vacancies and other defects. Growth, which proceeds through the nucleation of two-dimensional islands, itself continues to change the shape of the surface. To understand the growth process in detail we must monitor the surface morphology *in situ* during deposition.

X-ray diffraction from surfaces which are rough on an atomic scale has been discussed by Vlieg *et al.* (1989). In the simplest case, the structure is described as an assembly of columns of identical unit cells without relaxation or reconstruction at the surface. For the two level model, which describes a flat surface covered by islands of monolayer thickness, it is shown that the scattered intensity around an integral-order rod comprises two components:

$$I = I_{\mathrm{Bragg}} + I_{\mathrm{diff}}, \tag{6}$$

where
$$I_{\mathrm{Bragg}} = |F_{hkl}|^2 \, |F_{\mathrm{CTR}}|^2 \, (A/A_{\mathrm{u}}) \, [1 - 2\theta(1-\theta)\,(1-\cos 2\pi l)] \tag{7}$$

and $I_{\mathrm{diff}}(\Delta h, l) = \int I_{\mathrm{diff}}(\Delta h, \Delta k, l)\,\mathrm{d}k$, the intensity integrated over k,

$$= |F_{hkl}|^2 \, |F_{\mathrm{CTR}}|^2 \frac{A}{A_{\mathrm{u}}^2} 2\theta(1-\theta)\,(1-\cos 2\pi l)\frac{2a_2}{L}\frac{1}{L^{-2} + (2\pi\Delta h/a_1)^2}. \tag{8}$$

Here $|F_{hkl}|^2 \, |F_{\mathrm{CTR}}|^2$ is the contribution of a single column of unit cells, θ is the fractional coverage of islands A is the illuminated area and A_{u} is the area of the surface unit mesh. L is the height–height correlation length corresponding to a simple exponential correlation function.

It is well known that during MBE layer by layer growth, pronounced oscillations occur in the specular reflected intensity of high energy electrons (Neave *et al.* 1983). Gross features such as the periodic roughening due to two-dimensional island nucleation and step flow are readily identified. Details of the microscopic processes involved in the growth layers are, however, difficult to reveal since multiple scattering plays an important role in electron diffraction. X-ray scattering offers a more direct understanding of these atomic mechanisms. To date, the majority of the studies have monitored the central Bragg component of the CTR but, as is evident from equation (8), useful information can also be derived from the time variation of the distribution of diffuse scattering.

4. Selected examples

(*a*) Ge–Ge(111)

Vlieg *et al.* (1988) were the first to demonstrate that at reciprocal lattice positions between bulk points the intensity of the central Bragg component oscillates in a manner suggested by simple kinematic theory and similar to that of RHEED

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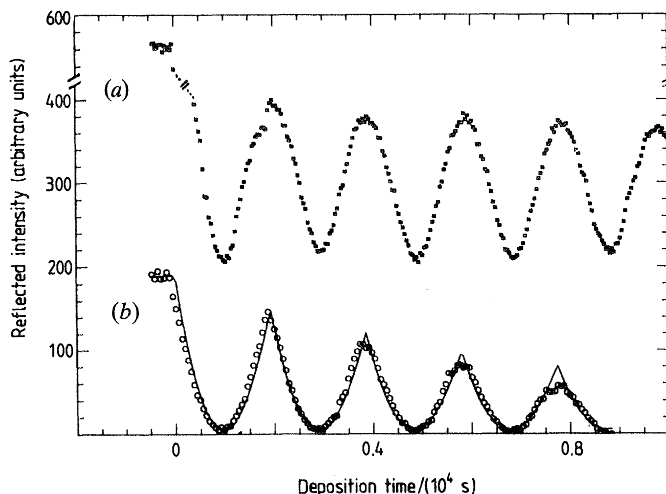

Figure 1. A comparison of the specularly reflected electron (*a*) and X-ray (*b*) intensities from Ge(111) during Ge deposition at 200 °C. The full curve is the fit to the data using a multi-level model. Reproduced from van Silfhout *et al.* (1989*a*) with permission.

intensities. This is shown clearly in figure 1 reproduced from a later study by van Silfhout *et al.* (1989*a*). It compares X-ray and electron specularly reflected intensities from Ge(111) surface during growth of Ge at 200 °C which is known to produce layer by layer growth. Both sets of data were taken at the anti-Bragg condition $l = \frac{3}{2}$ which corresponds to destructive interference between reflections from successive double layers, that is, the maximum sensitivity to double layer islands which are the basic building units of this surface.

It is seen that X-ray intensity follows a series of well-defined parabolas as predicted by equation (7), each peak corresponding to the completion of one bilayer. By contrast the reflected electron intensity plot is more sinusoidal in shape. The steady damping is due to imperfect growth, specifically the starting of a layer before the completion of the previous one. By recording the growth curves for several different values of l, van Silfhout *et al.* (1989*b*) were able to fit the data to multiple level model and to determine the occupancy of the levels. The analysis showed that the damping of the oscillations is caused by a small amount (5%) of the atoms nucleating on top of the almost filled first layer, confirming how sensitive X-ray scattering is to surface morphology on an atomic scale. The greater damping observed for X-rays compared to electrons is a consequence of the larger in-plane coherence length of the X-ray beam, typically 5000 Å, in contrast to that of the electron beam which is of the order of 200 Å. The more constant reflected electron intensity gives an exaggerated impression of the perfection of the growth.

The specular ($h = 0, k = 0$) rod is sensitive to all contributions to the averaged vertical distribution of atoms, irrespective of the in-plane positional registry. A more complete picture of the growth process is obtained by simultaneously measuring the scattering along non-specular integer-order rods for which the in-plane component of the momentum transfer is greater than zero. Figure 2 shows the variation of scattered intensity measured along the (10) rod following deposition of 0.28 and 0.68 monolayers (ML) of Ge on Ge(111) c2 × 8 at room temperature (van Silfhout *et al.* 1992). 1 ML corresponds to the density of a single (111) layer of bulk Ge(111). By

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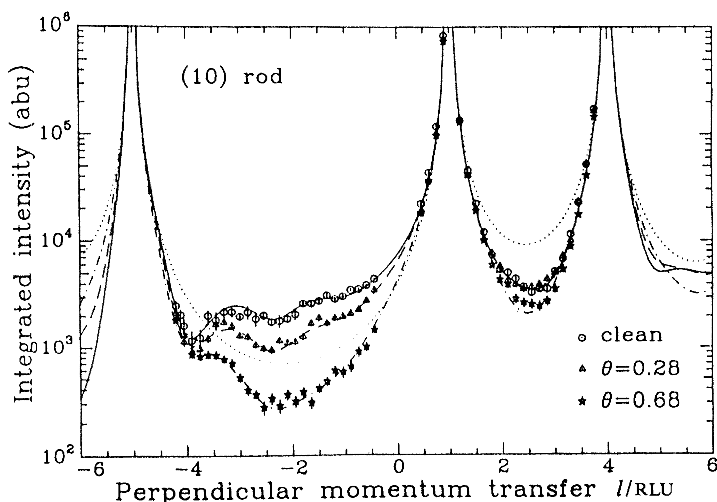

Figure 2. Distributions of integrated intensity along the (10) rod for the clean Ge(111) c2 × 8 surface (open circles) and for the surface after deposition of 0.28 and 0.68 monolayers of Ge at room temperature (triangles and stars respectively). The dotted curve represents the structure factor intensity calculated for a flat bulk-like surface. Reproduced from van Silfhout *et al.* (1992) with permission.

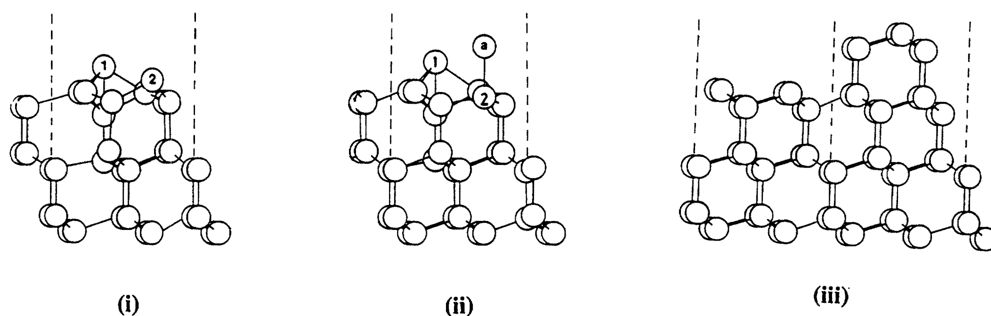

Figure 3. Sequence of growth of Ge on Ge(111) c2 × 8 at room temperature proposed by van Silfhout *et al.* (1992).

fitting plausible models to the data, the growth sequence indicated in figure 3 was deduced. Initially the clear surface is covered by a 25% monolayer of adatoms (1) in T4 sites, that is, one adatom in each 2 × 2 unit cell shown by the broken lines. The first atoms (a) that are landed attach to the rest atoms (2), which on the clean surface are not bonded to the ad-atoms. Continuing the deposition causes a further change with the growth of bi-layer islands as shown in (iii). The high reactivity of the rest atom is confirmed by analysis of the X-ray diffraction data which indicates that it moves from being 0.5 Å above its bulk position on the clean surface to 0.2 Å when it is bonded to a deposited atom.

(*b*) In–Si(111)

An example of hetero-epitaxial growth is that of In on Si(111)7 × 7 reported by Finney *et al.* (1992). The change of X-ray specular intensity during In deposition on a Si(111)7 × 7 substrate held at 350 °C is shown in figure 4. The solid curve is the best

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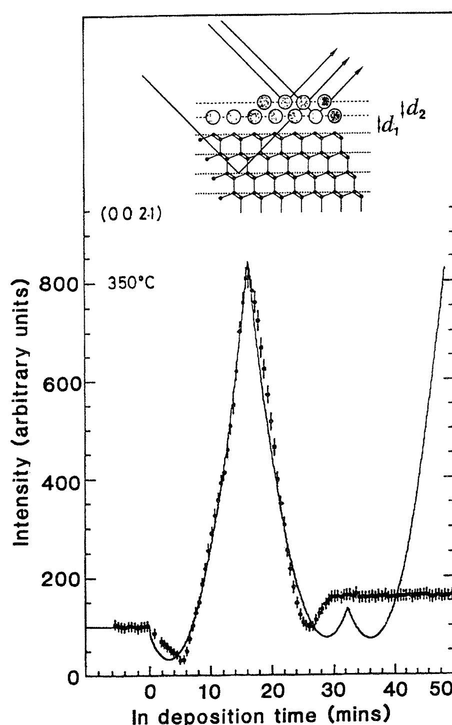

Figure 4. The specularly reflected X-ray intensity during In deposition on Si(111) at 350 °C. The solid curve is the best fit to the data using the growth model described in the text.

fit to the data using a growth model in which it is assumed that the In grows in dense close packed layers on top of a bulk terminated Si(111) substrate. Accordingly, the total structure factor can be written as

$$\boldsymbol{F}_{00l}^{\mathrm{tot}} = \boldsymbol{F}_{00l}^{\mathrm{bulk}} + \sum_n \theta_n \boldsymbol{f}^{\mathrm{In}} \exp\left[2\pi i l \frac{d_n}{|\boldsymbol{a}_3|}\right], \tag{9}$$

where $\boldsymbol{F}_{00l}^{\mathrm{bulk}}$ is the contribution of a single column of unit cells of the bulk and the sum is over indium layers of coverage θ_n; d_n is the displacement of the nth layer from the silicon surface and $\boldsymbol{f}^{\mathrm{In}}$ is the atomic scattering factor for indium. Since the scattering of a single indium layer is stronger than a silicon bilayer, the simple picture shown by uniform homoepitaxial growth is modified. The first minimum occurs at $\theta < 0.5$ and is most pronounced at values of $l > \frac{3}{2}$, the anti-Bragg position. The maxima, are of alternative height, the strongest corresponding to the completion of odd numbers of indium layers. The data points initially follow this pattern. The first peak flags the completion of the first indium layer but, before a second layer is fully formed, the structure relaxes with the nucleation of three dimensional islands. The small difference between experiment and theory, immediately after the shutter is opened, can be attributed to the steady deconstruction of the original 7×7 surface.

At this peak the LEED pattern is 1×1 and it is reasonable to assume that the layer is pseudo-morphic with a largely undisturbed bulk terminated silicon surface. The best fit to the measured reflectivity curve was achieved with the height of the silicon layer $d_1 = 2.63 = \pm 0.07$ Å. This agrees with the sum of the covalent radii of the silicon and indium atom and strongly suggests that the indium atoms occupy 'on-

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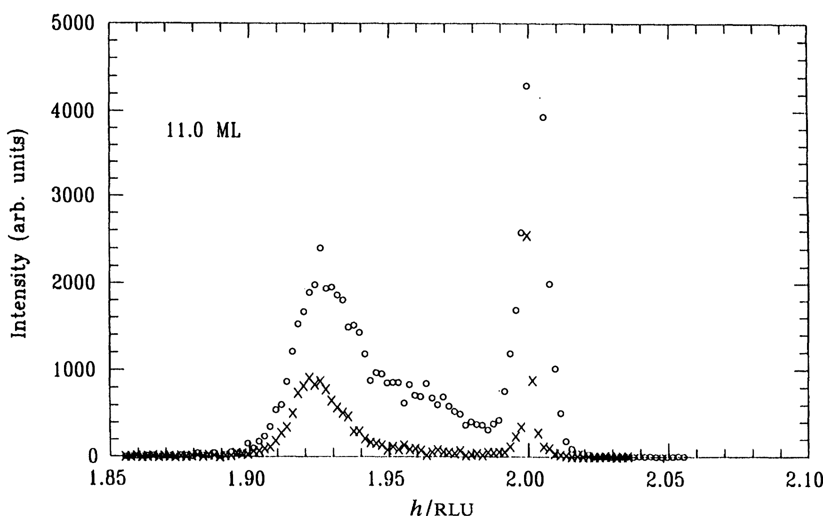

Figure 5. Radial scans through the (2, 0) Bragg peak after deposition of 11 ML of Ge on Si(001). The crosses represent the same scan at lower grazing angles and thus a reduced penetration depth. It confirms that the partly relaxed material is near the interface rather than at the surface. Reproduced from William *et al.* (1991) with permission.

top' T1 sites, as shown in the inset. For an unreconstructed Si(111) surface with dangling bonds oriented vertically, this is the most energetically favoured bonding site for adatoms. At higher temperatures, the surface reconstructs to a lower energy configuration with $\frac{1}{3}$rd of a monolayer of indium atoms in T4 sites in a $\sqrt{3}\times\sqrt{3}$ symmetry (Finney *et al.* 1993).

Since the epitaxial growth is interrupted before the second layer is complete, the height of this layer was estimated with less accuracy. The best fit value of the separation of the two indium layers $d_2 = 2.60 = \pm 0.13$ Å compares with the value 2.48 Å, expected for the close packing of spheres with the average atomic radius of the bulk 3.33 Å.

(*c*) Ge–Si(001)

In on Si(111) is an extreme case of an interface formed between two elements characterized by very different chemical bonding and crystallographic structures. In recent years considerable effort has focused on 'strained layer' growth of covalent semiconductors with the same crystallographic structure and lattice parameters which are equal to within a few percent. The uniaxial distortion induced by epitaxy can modify the electronic structure in a way which is potentially useful for the operation of an electronic device. As the overlayer develops the strain energy progressively increases until, at a 'critical thickness', the strain energy is released and in-plane lattice parameter relaxes towards the value of the bulk phase. Attention has been directed to the value of this parameter and the nature of the relaxation mechanism.

The high sensitivity of SXRD to the in-plane lattice spacing has been exploited in a study of the initial stages of growth of Ge on Si(001) by Williams *et al.* (1991). Figure 5 shows radial scans through the (2 0) CTR for a Si(001) wafer on which 11 ML of Ge has been deposited at 550 °C before cooling to room temperature. For $\theta < 3$ ML, the profile was indistinguishable from that of the clean Si shown by the strong peak at 2.0 reciprocal lattice units (RLU), indicating that the overlayer was pseudomorphic

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with an in-plane lattice spacing identical to that of the Si substrate. Beyond 3.9 ML, the profile progressively extended to lower values of q_{par} as in the in-plane lattice spacing relaxed; at 11 ML, a subsidiary peak is clearly seen near 1.92 RLU, the position expected for bulk Ge.

The onset of relaxation is consistent with earlier work which showed that beyond 3 ML, islands form on the surface of the Ge overlayer (Asai *et al.* 1985). The plateau of scattering in the region 1.95–2.00 RLU indicates that the relaxation is not complete. By reducing the input and exit angles, and therefore the probe depth, the intermediate scattering almost disappears, confirming that the partial relaxed layers are at the interface with the substrate rather than distributed throughout the overlayer. Thus even when islanding has occurred the relaxation is not complete, rather much of the overlayer remains highly strained. Further measurements of the scattering along the specular (00) rod are consistent with this picture and indicate a rapid increase in the island height for coverages above 6 ML. Subsequent measurements by Thornton *et al.* (1991) showed that the growth mode is significantly affected by the presence of an Sb surfactant layer. The modification to the surface energy causes the onset of strain relaxation to be delayed from 3–4 ML to 9–10 ML and to gradually increase thereafter. This would suggest that island formation is inhibited and that the strain is relieved by another mechanism, possibly involving dislocations.

(*d*) *Other systems*

Several other X-ray diffraction studies of semiconductor MBE growth have been reported (Jedrecy *et al.* 1990 (GaAs–Si(001)); Robinson *et al.* 1992 (Pd_2Si–Si(111))). The ability of X-radiation to penetrate matter has been exploited in measurements of OMVPE growth of ZnSe on GaAs(001) (Kisker *et al.* 1990). All of these studies, like the others above, monitored only the time dependence of the central Bragg peak.

The value of simultaneously recording the weaker diffuse scattering has been demonstrated by Fuoss *et al.* (1992) in a study of OMVPE growth of GaAs on GaAs(001). The high brilliance X-ray undulator source on the PEP storage ring at Stanford Synchrotron Radiation Laboratory allowed measurements to be made on a scale of less than 0.1 s and with growth rates of the order of 1 ML s^{-1}, which is typical of that used to grow device structures. At 540 °C and at the time when the intensity in the Bragg component is a minimum, that is when 50 % of the surface is covered with islands, a maximum in the diffuse scattering is seen at $\Delta q_{par} = 0.02$ Å^{-1}. This contrasts with the simple lorentzian form of equation (8) and indicates a non-random distribution of islands with an average spacing (given by $2\pi/\Delta q_{par}$) of 300 Å. The average spacing increases steadily to 800 Å as the substrate temperature is raised to 590 °C. At 625 °C the growth proceeds by step-flow. Similar measurements of the diffuse scattering in an orthogonal direction indicated that the island shape during growth is anisotropic.

5. Outlook

In this brief review I have attempted to illustrate the useful role that X-ray diffraction can play in obtaining a better understanding of the atomic mechanisms involved in the growth of ultra-thin films. The introduction of new synchrotron radiation sources of ever increasing brilliance offers further opportunities for determining more complex structures characterized by larger unit cells and for real time studies with smaller time resolution and therefore growth rates more typical of

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those used in device fabrication. More detail will be obtained concerning the growth kinetics: the shape and size distribution of islands and the role of surface features such as steps and surface reconstructions. Finally, the next few years will witness increasing emphasis on the structure and phase transitions of buried interfaces.

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