High-resolution three-dimensional reciprocal-space mapping of InAs nanowires

S. O. Mariager,^{a*} S. L. Lauridsen,^a A. Dohn,^a N. Bovet,^b C. B. Sørensen,^a C. M. Schlepütz,^c P. R. Willmott^c and R. Feidenhans'l^a

^aNiels Bohr Institute, University of Copenhagen, 2100 Copenhagen, Denmark, ^bNano-Science Center, Department of Chemistry, University of Copenhagen, 2100 Copenhagen, Denmark, and ^cSwiss Light Source, Paul Scherrer Institut, CH-5232 Villigen-PSI, Switzerland. Correspondence e-mail: som@fys.ku.dk

Grazing-incidence X-ray diffraction is combined with a two-dimensional pixel detector to obtain three-dimensional reciprocal-space maps of InAs nanowires grown by molecular beam epitaxy. This rapid data-acquisition technique and the necessary correction factors are described in general terms, as well as for the specific setup used, for which a resolution of $\sim 2 \times 10^{-3}$ Å is computed. The three-dimensional data sets are obtained by calculating the reciprocal space coordinates for every pixel in the detected images, and are used to map the diffuse scattering from the nanowires as both two-dimensional reciprocal-space maps and three-dimensional isosurfaces. The InAs nanowires are shown to consist mainly of wurtzite crystal with a c/a ratio of 1.641. The diffuse scattering reveals two different facet structures, both resulting in hexagonal cross sections of the nanowires.

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1. Introduction

Reciprocal-space maps (RSMs) are well suited to detailed structural studies of imperfections in crystalline materials through the analysis of diffuse scattering around intense Bragg points. For instance, the strain and facet structure of semiconductor nanowires have recently been the target of several investigations employing grazing-incidence diffraction to create RSMs (Keller *et al.*, 2006; Kawamura *et al.*, 2005; Mandl *et al.*, 2006; Mariager *et al.*, 2007; Sakata *et al.*, 2004). Such nanowires have been the target of intense research, owing to their promising electronic, photonic and biological applications (Yang *et al.*, 2002). Structural characterization is especially important in nano-systems, where facets play a crucial role, owing to the high surface-to-bulk ratio, whereby the crystal structure may vary from that of the bulk structure and where interfaces can cause widespread strain. The general use of RSMs, however, spans wider areas, such as the study of strain in thin-layer structures (Masson *et al.*, 2005) or multilayers (Mudie *et al.*, 2004), and the diffuse scattering from single crystals (Boulle *et al.*, 2007; Gerhard *et al.*, 2000) and quantum dots (Schmidbauer *et al.*, 2006). Typically, RSMs have been two-dimensional, and the process of obtaining them is normally very time consuming. The use of point detectors offers high precision but is slow because of the sampling of single data points. To increase the data-acquisition speed, both curved and linear one-dimensional detectors have been employed in various setups. For the same purpose, image plates (Mudie *et al.*, 2004), CCD detectors (Schmidbauer *et al.*,

2006) and, in a grazing-incidence setup, gas detectors (Fontaine *et al.*, 2004) have been applied.

The challenges of obtaining high-resolution RSMs from crystalline structures include the need to measure weak diffuse scattering with high resolution near intense Bragg points, which requires detectors with a large dynamic range and generates large amounts of data. For three-dimensional nanostructures, the full three-dimensional information is an additional requirement. The data should also be imaged in reciprocal-space coordinates rather than the readily accessible angular coordinates, since the former allow for much more straightforward physical interpretation. With the current fast development of detectors, techniques to obtain high-resolution, complete, three-dimensional data sets fulfilling these requirements are becoming viable. Three-dimensional data acquisition has been applied for grazing-incidence small-angle scattering in combination with tomographic methods, to map the crystal truncation planes originating from the edges of SiGe nanoparticles (Vartanyants *et al.*, 2008). Grazing-incidence diffraction, however, allows for a more direct interpretation than small-angle scattering, and three-dimensional acquisition of diffraction data has been performed recently on both semiconductor nanowires (Mariager *et al.*, 2007) and quantum dots (Schmidbauer *et al.*, 2008).

In §2, we first explain in general terms how three-dimensional reciprocal-space maps may be obtained by calculating the reciprocal coordinates of each individual pixel in a two-dimensional detector, and discuss which correction factors are to be applied. We then present the specific setup of the Materials Science (MS) beamline at the Swiss Light Source

(SLS), Villigen, Switzerland, as used for this experiment, and describe how to calculate the resolution of the setup numerically. In order to demonstrate the capabilities of three-dimensional reciprocal-space mapping, in §3 we then apply the method to a sample of InAs nanowires and determine their facets, crystal structure and lattice constants.

2. Method

In order to obtain high-resolution three-dimensional reciprocal-space data of surface structures, a two-dimensional detector is combined with grazing-incidence X-ray diffraction. A grazing-incidence angle minimizes the penetration depth, which emphasizes scattering from the surface. By using a two-dimensional detector, any scan of sample or detector positions will result in a three-dimensional data set.

The choice of scan performed is crucial for the quality of the data. Although the detector encompasses the same angular range for every image in a scan, as given by the detector slits and the position of the detector, the series of images constituting a scan will expand strikingly different volumes in reciprocal space, depending on the scan chosen. This is the origin of the classical Lorentz factor for integrated intensities. In general, the best volume data set is obtained with a scan along the surface normal of the detector plane, which in reciprocal space is given by the outgoing wavevector $\mathbf{k}'$. At a glancing incoming angle for a typical diffraction peak, where the outgoing angle is not too high, a good volume of data is achieved by a rocking scan in which the detector position is kept fixed, while the sample is rotated around its surface normal. The result is a set of intensities as a function of diffraction angles, $I(\alpha, \omega, \delta, \gamma, \nu)$, where the angles of each pixel are determined separately. In order to obtain three-dimensional reciprocal-space maps, these intensities are transformed into a new set as a function of coordinates in reciprocal space, $I(h, k, l)$. The volume data set will then consist of a set of points distributed nonlinearly in reciprocal space, from which lines, planes or three-dimensional surfaces of intensity as a function of reciprocal-space coordinates can be interpolated.

As for all scattering experiments, the intensity $I(\alpha, \omega, \delta, \gamma, \nu)$ must be corrected for polarization, active sample area and integration area. The polarization correction is the same as for any diffraction experiment, but must take into account the specific scattering angles of each pixel. For small detectors that encompass only a few degrees in angular space, the difference in polarization within single images will, however, be negligible. The effect of intensity integration area is known as the Lorentz factor. The scattered intensity I within a given pixel can be calculated as an area integral of the differential cross section $d\sigma/d\Omega$ across the pixel opening angles γ and δ , including the flux Φ , exposure time t and Thomson scattering length r_0 :

$$I(Q) = \Phi t \int \frac{d\sigma}{d\Omega} d\gamma d\delta \simeq \Phi t r_0^2 |F(Q)|^2 |S(Q)|^2 \int d\gamma d\delta. \quad (1)$$

For sufficiently small pixels and large detector-to-sample distances, the angular openings of a single pixel are small enough that both the slowly varying unit-cell structure factor $F(Q)$ and the faster varying lattice structure factor or shape function $S(Q)$ can be assumed to be constant within any given pixel. The integral across the pixels' opening angles is then a constant identical for all pixels, and no angle-dependent Lorentz factor must be applied for the comparison of intensities within the reciprocal-space map. The assumption that a single pixel is small enough for the lattice structure factor to vary only insignificantly across its area is valid for the diffuse scattering near Bragg points but not for the centre of the peaks, where $S(Q)$ changes rapidly. In order to obtain integrated intensities, the detector can instead be used in a stationary mode as described by Vlieg (1997). Finally, the active area, which depends on the specific experiment, must be considered. In the following subsection we describe in detail the setup used in this experiment.

2.1. Setup at the MS diffraction beamline at SLS

The experimental setup at the Surface Diffraction Station of the MS beamline at the SLS (Patterson *et al.*, 2005) consists of a (3D + 2S) surface diffractometer (Vlieg, 1998) and a PILATUS 100K two-dimensional pixel detector, and is ideally suited to obtain high-resolution three-dimensional RSMs. The diffractometer is designed especially for surface studies, but many of its properties are equally useful for epitaxially grown nanostructures, such as the semiconductor nanowires presented here, especially regarding the preference for working at low glancing angles to maximize surface scattering and limit background from the bulk substrate. In addition, the surface normal can be mounted both horizontally and vertically, allowing us to choose the orientation with respect to the mainly horizontal polarization and the larger angular divergence in the horizontal plane of the typical wiggler beam. The horizontal surface normal, which we used, maximizes the in-plane resolution of $\mathbf{Q}_{||}$. Finally, the diffractometer allows for detection of large momentum transfers both in- and out-of-plane, and controls the rotation around the surface normal (ω) by a single motor, which is advantageous for obtaining stable three-dimensional data sets.

The PILATUS 100K detector (Eikenberry *et al.*, 2003) consists of a single silicon module with charge readout on the back in 195×487 pixels, each of size $172 \times 172 \mu\text{m}$. Each pixel works as a single photon counter with a dynamic range of 10^6 photons s^{-1} . An energy filter with a 500 eV resolution can be applied electronically to suppress unwanted fluorescence background, which is an exceedingly important feature for the detection of weak diffuse scattering. In the present case, employing an incident X-ray energy of 16.0 keV, the filter is used to avoid the arsenic K -edge fluorescence. The advantage of working at such high X-ray energies is the access to a large volume in reciprocal space.

The practical use of the diffractometer depends on an efficient calculation of reciprocal space coordinates from the set of angles defining the effective detector position, and *vice*

versa. The (3D + 2S) diffractometer has two circles for the sample. When working with our horizontal surface normal the rotation α determines the incidence angle, while ω rotates the sample around its surface normal. The two primary detector rotations are the horizontal γ and the vertical δ . For the (3D + 2S) diffractometer, the δ circle is mounted on the γ circle, contrary to a z -axis diffractometer. An optional ν circle, which rotates the detector and slits around the detector–sample axis, is available but irrelevant for the discussion presented here. All rotations can be controlled to a precision of around 0.002° . From a given set of angles, a set of reciprocal-space (h, k, l) coordinates in a Cartesian coordinate system fixed to the sample holder can be calculated along the lines of Bunk & Nielsen (2004):

$$\mathbf{Q}_\omega = k \begin{bmatrix} \cos(\omega) \sin(\delta) - \sin(\omega) \{ \cos(\alpha) [\cos(\gamma) \cos(\delta) - 1] + \sin(\alpha) \sin(\gamma) \cos(\delta) \} \\ \sin(\omega) \sin(\delta) + \cos(\omega) \{ \cos(\alpha) [\cos(\gamma) \cos(\delta) - 1] + \sin(\alpha) \sin(\gamma) \cos(\delta) \} \\ - \sin(\alpha) [\cos(\gamma) \cos(\delta) - 1] + \cos(\alpha) \sin(\gamma) \cos(\delta) \end{bmatrix}, \quad (2)$$

where $k = 2\pi/\lambda$ is the length of the wavevector. $\mathbf{Q}_\omega$ can be transformed into the reciprocal lattice of the given sample by use of the experimentally determined orientation matrix $\mathbf{U}$, such that $\mathbf{Q}_\omega = \mathbf{U}\mathbf{B}\mathbf{Q}$. Here, we have kept the standard notation from the literature with a matrix $\mathbf{B}$ which orthonormalizes the reciprocal lattice of the sample, but in practice, $\mathbf{U}\mathbf{B}$ is used as a single orientation matrix. In the calculation of the reciprocal-space coordinates, we have not considered refraction effects at glancing exit angles. Because the refractive index is less than unity at X-ray wavelengths, the maximum scattering of an in-plane Bragg peak is shifted from $\mathbf{Q}_\perp = \mathbf{0}$ to an outgoing angle $\beta = \alpha_c$, the critical angle. The calculations described in this paper are thus restricted to out-of-plane Bragg peaks with an outgoing angle larger than the critical angle.

In order to obtain three-dimensional RSMs from a set of detector images, we use equation (2) and the experimentally determined orientation matrix to convert the intensities and angle settings $I(\alpha, \omega, \delta, \gamma, \nu)$ of single pixels or bins of pixels into intensities $I(h, k, l)$ as a function of reciprocal-space coordinates. The diffractometer angles for each individual pixel in a given image are determined geometrically. Since the rotations α and ω only influence the sample, they are constant for each image, and only δ and γ vary for individual pixels. As Fig. 1 illustrates, the angular offsets $\Delta\delta$ and $\Delta\gamma$ as a function of pixel position are easily determined given the distance from the pixel to the detector centre and the relevant distance to the sample. The latter is given either by the distance D_1 to the sample slits, in the case of narrow slits, or by the distance to the sample D_2 , if wide or no slits are used. If narrow slits are only applied in one direction, the relevant distance used in the angle calculations will be different for the two angles δ and γ . Such a setting is relevant in a grazing-incidence geometry, where the limited height of the X-ray beam ensures a narrow spot size horizontally, while a slit is needed to limit the active

area seen by the detector vertically. A set of *MATLAB* routines, which can perform the conversion from angular to reciprocal coordinates in less than one second per image, are available at the MS beamline at the SLS.

2.2. Active area and resolution

We now calculate the active area and resolution for individual pixels in an image using a ray-tracing technique. The field of view from a pixel corresponds to a rectangle on the sample, which is found by tracing straight lines from each pixel corner through opposing slit corners and onto the sample plane. The overlap of the resulting rectangle with the X-ray beam on the sample then determines the active area, as illustrated in Fig. 1(b). Similar computational techniques have previously been used with grazing-incidence diffraction to study the effect of large exit angles on the measured intensities of crystal truncation rods (Torrelles *et al.*, 1996) and the change in active area during rocking scans (Robach *et al.*, 2000). An example is shown in Figs. 2(a) and 2(b), where the calculated active area in units of mm^2 is compared with the

Figure 1

Schematic drawing of the experimental setup. (a) The relevant angles δ , γ and α for detector and sample. The incoming $\mathbf{k}$ and outgoing $\mathbf{k}'$ wavevectors are shown, as well as the scattering vector $\mathbf{Q}$ for the centre of the detector. Distances from sample to detector (D_1) and slit to detector (D_2) are marked, as well as the slit openings (vertical s_v and horizontal s_h). The sample surface is drawn horizontal for clarity, although the setup used in this paper employed a vertical surface. Finally, the dark stripe on the sample illustrates the area illuminated by the incoming X-ray beam. (b) The ray tracing method. Dotted lines, traced from the pixel through the slit corners, give the field of view on the sample for a particular pixel. The solid lines trace the corners of the active area, corresponding to the overlap between field of view and the area illuminated by X-rays. $\mathbf{k}_1$ and $\mathbf{k}_2$ illustrate the two wavevectors with the largest spread in direction that are able to reach the pixel from the active area. These wavevectors determine the geometric resolution, as illustrated in the insert. For clarity, all lines are drawn terminating at the centre of the pixel, though the ray tracing was performed from the corner of the pixel through opposing corners of the slit, which takes into account the size of the pixel and results in a decrease of the effective resolution.

intensity distribution in a raw image with the same slit settings and a typical set of angles $\delta = 12.705^\circ$ and $\gamma = 4.568^\circ$, corresponding to a GaAs [11 $\bar{1}$] Bragg point. The correspondence between the calculated active area and the actual illumination on the detector is very good and can be reproduced similarly for other angle settings. The active area shows little variation near the centre of the detector and is only relevant if we study features far from the detector centre. The size and shape of the sample must be considered in the calculation, since the field of view for pixels far from the detector centre tends to spill over the sample edges. To include this factor correctly, exact knowledge of both the sample shape and its mounting on the diffractometer is required. For a standard ω scan of a single Bragg point, ω is typically scanned in an interval of $\pm 4^\circ$ with constant δ and γ , and a single area correction is sufficient for all images.

The calculation of the active area allows us to estimate the resolution given by the geometry of the slits placed before and after the sample. Assuming a perfectly collimated beam, the resolution is given by the range of wavevectors that can enter a pixel from the active area on the sample, as illustrated in Fig. 1(b). We calculate the resolution as the largest possible difference between two such $\mathbf{k}$ vectors capable of entering a single pixel, since this translates directly to a spread in $\mathbf{Q}$, as depicted in the insert in Fig. 1(b). Figs. 2(c) and 2(d) show the calculated resolution of $\mathbf{Q}$ in $\text{\AA}^{-1}$ for all pixels in an image, separated into in-plane and out-of-plane components for the same [11 $\bar{1}$] GaAs reflection as used above. The resolution

given by the slits, which are opened 0.5 mm vertically and 10 mm horizontally in this case, is approximately $0.002 \text{ \AA}^{-1}$. This is comparable to the resolution of the RSMs presented in §3. Higher δ and γ angles tend to result in a decrease of the resolution, especially in the in-plane direction. The sizes of the slits are relevant and a smaller horizontal detector slit greatly increases the in-plane resolution. The largest uncertainties arise for the central pixels because of their larger active area and the result is a smearing of the features in our RSMs. Other factors influencing the resolution are the transverse coherence length and energy spread. The transverse coherence length of the beamline has been measured to $19.2 \text{ }\mu\text{m}$, corresponding to an effective source size of $75 \text{ }\mu\text{m}$ (Patterson *et al.*, 2005). Comparing this with our active areas of the order of 1 mm^2 , the latter is the decisive factor. The same is true for the energy spread $\Delta E/E = 0.0139\%$, corresponding to a longitudinal coherence length of $0.39 \text{ }\mu\text{m}$ at 16 keV.

3. InAs nanowires

To demonstrate the possibilities of the described setup, we characterize an ensemble of InAs nanowires grown by molecular beam epitaxy (MBE) on an InAs (111) substrate. Nanowires have attracted great interest in recent years, but the precise details of the growth process and the influence on the resulting crystal and facet structure of the nanowires is still debated, which underlines the need for good characterization tools to supplement existing methods such as scanning electron microscopy (SEM) and transmission electron microscopy.

The InAs nanowires were grown by solid source MBE in a Varian GEN II MBE machine. The substrates used were 'epiready' InAs wafers with a surface orientation of (111). After thorough degassing in a separate buffer chamber, the substrates were transferred to the growth chamber and heated to 808 K in a flux of As_2 to desorb the surface oxide. The As_2 beam equivalent pressure used was $1.33 \times 10^{-3} \text{ Pa}$. After oxide removal, the substrate temperature was lowered to 773 K, and the Au was then deposited directly on the oxide-free surface *in situ* and left to anneal in the As_2 flux for 4 min. The deposition time of the Au determines the resulting size of the catalyst particle; we have not performed a calibration of the Au deposition rate, but a deposition time of 35 s at a source temperature of 1623 K results in an average wire diameter of around 70–80 nm, as shown in Fig. 3. Following the annealing step, the substrate temperature was lowered to the growth temperature of 703 K. The nanowires were grown using an InAs growth rate of $1.5 \text{ }\mu\text{m h}^{-1}$, calibrated by RHEED oscillations on a separate (100) calibration piece prior to growth. The growth time used was 20 min, resulting in an average nanowire length of 4–4.5 μm . All samples were cooled in an arsenic flux after termination of the growth. The substrate temperature used is well below the eutectic temperature of 727 K for the Au–In alloy. The growth mode is thus a vapour–solid–solid mode where the catalyst particle is solid (Dick *et al.*, 2005).

Bulk InAs has a zincblende structure, which corresponds to an ABC stacking of hexagonal close packed (111) bilayers.

Figure 2

(a) Raw detector image with logarithmic intensity scale. (b) Calculated active area displayed as mm^2 per pixel, as obtained from the ray tracing operation. (c) Calculated in-plane resolution for the individual pixels. (d) Corresponding out-of-plane resolution. The axes display pixel number. As displayed here, δ is horizontal, and the images are rotated 90° clockwise. The detector slits are 0.5 mm vertical and 10 mm horizontal and the X-ray beam is 1 mm high on the sample. The angles are chosen for a GaAs [11 $\bar{1}$] Bragg point, but are typical also for the reflections imaged in Figs. 5 and 6.

Typical crystal faults in zincblende crystals correspond to different stacking sequences. A 60° rotation of the crystal structure is a twin fault and corresponds to an ACB stacking, while a two-layer AB stacking sequence corresponds to the wurtzite structure. Since all stacking faults share their in-plane crystal structure, the reciprocal lattices are similar in-plane and a scan along the $[111]$ growth direction is sufficient to identify all present stacking sequences. Such a scan, taken through the $[11\bar{1}]$ bulk Bragg point, is shown in Fig. 4 for two different incoming angles. We clearly identify Bragg points corresponding to the InAs wafer, twin faults and the wurtzite structure. With our large active area, we sample hundreds of thousands of nanowires simultaneously to obtain a statistical average. Additionally, we sample both the nanowires, part of the bulk substrate, and any surface growth. To separate scattering from the substrate surface and the nanowires, we have performed the scan at both an incoming angle $\alpha = 0^\circ$ and a 0.3° glancing angle. While the glancing angle samples the substrate surface and the nanowires, a 0° incoming angle minimizes scattering from the surface growth, while scattering from the sparsely placed wires remains high. In Fig. 4 we see a large decrease in scattering intensity from the twin faults, while both the wurtzite and the wafer peaks remain strong, when moving from an incoming angle of 0.3 to 0° . Integrated intensities corrected for Lorentz factor, polarization and unit-cell structure factors show that, while the wurtzite and zincblende signal is reduced by approximately 50%, the twin signal is diminished by 97%. This implies that the wurtzite structure is the main constituent of the wires, while the twin faults mainly arise in the surface layer grown simultaneously with the wires. Previous results have also shown that InAs nanowires consist mainly of a perfect wurtzite structure (Mandl *et al.*, 2006). At higher incoming angles, we also see weak peaks at intermediate values of $Q_\perp = 0.5, 2.5$ and $3.5 \text{ \AA}^{-1}$. These correspond to a six-layer stacking sequence, as described by Mariager *et al.* (2007), but appear to be present only on the wafer surface.

Figure 3

SEM images of the nanowires seen from the side (a) and top (b). The nanowires grow perpendicular to the (111) surface. Two different facet orientations, marked by circles and squares, are seen in (b).

We also determined the peak positions and thereby the lattice constants of the structures. Since the InAs wurtzite structure only exists in nanowires, a precise determination of the structural parameters is important for theoretical calculations of the wire properties. The wurtzite out-of-plane lattice constant is found from nine different peaks to be $c = 7.028(8) \text{ \AA}$; this is slightly increased with respect to the stacking distance in bulk zincblende, which if maintained in the wurtzite structure would result in an out-of-plane lattice constant of $3^{1/2} \times 6.0583 \text{ \AA} \times (2/3) = 6.996 \text{ \AA}$. Previously, Mandl *et al.* (2006) found $c = 6.985 \text{ \AA}$, but commented that this could be underestimated, while Takahashi & Moriizumi (1966) found $c = 7.02$, in closer agreement with our results. The fact that Takahashi and Moriizumi studied whiskers with a $2\text{--}3 \mu\text{m}$ diameter, while we study wires with nanometre diameters, indicates that the lattice constants are an intrinsic property of the wurtzite structure rather than being influenced by the nanowire dimensions. For the in-plane lattice constants, that is, in the (111) plane, we do not find any difference between the bulk InAs and the wurtzite structure, which results in $a = 4.284 \text{ \AA}$ and a c/a ratio of 1.641. This is higher than the ideal value of 1.633 for a hexagonal close packed structure, but many elements show larger deviations from the ideal c/a ratio. The position of the zincblende peak displays a perfect match with the bulk InAs lattice constant of $6.0583 \text{ \AA}$, and the twin structure displays the same lattice constants well within two standard deviations of the mean.

3.1. Facet structure of InAs nanowires

The SEM images in Fig. 3 show the nanowires to have a hexagonal cross section but cannot discern any micro facets,

Figure 4

Crystal truncation rod scanned through the $[11\bar{1}]$ zincblende Bragg point, located at $Q_\perp = 1$ on the graph. Peaks corresponding to the regular zincblende wafer (Z), twin faults (T) and wurtzite (W) structure are seen. The small peaks at intermediate Q values correspond to six-layer superstructures, which are primarily present as surface growth. The two scans show the same CTR taken at different incoming angles of 0.3 (solid line) and 0° (stippled).

such as those found, for instance, in GaAs wires (Mariager *et al.*, 2007). The SEM images also show two different hexagonal shapes, rotated by 30° with respect to one another, but cannot discern whether or not this is due to different surfaces or a rotation of the entire crystal structure. We can, however, exclude the latter possibility since the Bragg points that would result from such a rotation are absent. In order to determine the exact facet structure of the nanowires, we study the diffuse scattering around the Bragg reflections. Since the shape of the Bragg point is given by the Fourier transform of the crystal shape function, a sharp facet gives rise to a diffuse streak of scattering along the surface normal; such streaks are known as crystal truncation rods (CTRs) and have been used extensively in the study of surfaces (Feidenhans'l, 1989). The broad shoulders of the Bragg peaks in Fig. 4 are the CTRs from the wafer surface and the domain walls between stacking faults within the nanowires. By mapping the diffuse scattering from a given Bragg point in reciprocal space, we thus obtain detailed information on the facet structure of the crystal, which the Bragg point originates from. One advantage of this technique is that different crystal structures are separated in reciprocal space, and hence, the facets of different crystal structures closely spaced in real space can easily be distinguished.

Fig. 5 shows the weak diffuse scattering around the wurtzite Bragg point, located at $Q_\perp = 1.5$ in Fig. 4, as a three-dimensional isosurface. The strong vertical CTR along the $[111]$ direction, also visible in Fig. 4, arises from the domain walls between stacking faults within the wire, while the many weaker rods perpendicular to the wire growth direction correspond to different surface facets. Since no crystal truncation rods from facets extend out of the horizontal (111) plane, which is highlighted by the shaded area, we can conclude that the wurtzite nanowires have no microfacets. The final feature in Fig. 5 is the disc-shaped object intersecting the Bragg point. This is a Debye–Scherrer ring originating from slightly tilted nanowires. In Fig. 6, we plot a two-dimensional RSM of the highlighted (111) plane from the same wurtzite Bragg point. Since the map lies in the (111) plane, any CTR within this plane is perpendicular to the growth direction. The

figure reveals 12 CTRs, corresponding to the two different types of hexagonal wires seen in Fig. 3. Since one Bragg point has all 12 CTRs, the two types of wire facets have crystal structures with the same orientation but different surfaces: one set spanned by six $\{100\}$ wurtzite surfaces and the other set by six $\{110\}$ wurtzite surfaces, corresponding in direction to the cubic $\{11\bar{2}\}$ and $\{01\bar{1}\}$ surfaces. It is also possible to extract one-dimensional scans and thereby to obtain the width of the peaks. Since the intensity in the $[11\bar{2}]$ direction is diminished by the detector slits, we use only the width in the $[2\bar{1}\bar{1}]$, $[1\bar{1}0]$ and $[1\bar{2}1]$ directions to obtain a full width at half-maximum of $\delta Q_\perp = 0.757 \text{ \AA}^{-1}$. From this, the width of the nanowires can be estimated as $d \simeq 0.9(2\pi/\delta Q_\perp) = 75 (3) \text{ \AA}$, in good agreement with the SEM images in Fig. 3.

Both Figs. 5 and 6 display planes with greatly enhanced noise levels. This direction corresponds to the detector images in which the centre of the Bragg point is scanned. Because of the high intensity, transmission filters are inserted in the beam to protect the detector. As a result, the weak diffuse scattering cannot be discerned, and the noise in the low-intensity areas of the detector is enhanced by the transmission factor, which in this specific case amounts to 3.59×10^4 . As a result, weak diffuse scattering like the facet CTRs cannot be detected in images that coincide with the Bragg points. The simplest solution is to take advantage of the symmetry of the sample to explore symmetry-equivalent Bragg points. In our case with a wurtzite crystal structure, we have sixfold symmetry around the c axis. The planes obscured by transmission filters will rotate with the crystal, and by combining data from different Bragg points, all directions in reciprocal space can be explored. Another solution could be to use slits to cut away the Bragg point, at the potential risk of a substantial amount of slit scattering.

A great advantage of the described method is the speed. A full three-dimensional data set around a Bragg point, as displayed in Fig. 5, consists of 80 images with a counting time of 10 s per image, which requires a total scan time of 20 min. In general, of course, this depends on the scattering efficiency of the specific sample. For comparison, obtaining a plot similar to

Figure 5

Three-dimensional isosurface of the weak diffuse scattering near a $[101]$ wurtzite Bragg point, corresponding to the peak at $Q_\perp = 1.5$ in Fig. 4. The directions of the axes are given in cubic coordinates with respect to the InAs wafer, and the shaded area corresponds to the (111) plane mapped in Fig. 6. CTRs corresponding to various facets are seen, as well as parts of a Debye–Scherrer ring.

Figure 6

Two-dimensional RSM of a $[101]$ wurtzite Bragg point. The directions of the axes are given in cubic coordinates with respect to the InAs wafer. 12 different CTRs emanate, corresponding to the six $\{100\}$ and six $\{110\}$ wurtzite surfaces. The colour scale is logarithmic intensity and the CTRs are quenched in the $[11\bar{2}]$ direction by the detector slits.

Fig. 6, that is, only the two-dimensional information, takes approximately 2 h with a similar setup but with a point detector. Considering the value and shortage of synchrotron beamtime, the gains of the method are considerable.

4. Conclusion

The method described for fast and high-resolution three-dimensional reciprocal-space mapping has many advantages. By extracting the full volume data in reciprocal-space coordinates around a Bragg point in one scan, all the required information can be extracted later. This includes both two-dimensional maps and one-dimensional line scans, besides three-dimensional images. Precise details need not be determined online; for instance, a slight offset of a Bragg point is easily located during post analysis and corrected for in scans performed by interpolation of the full data set. The resolution, of the order of 2×10^{-3} Å, is sufficient for mapping fine detail in reciprocal space and can be improved by decreasing the sample and detector slits sizes, but at the cost of intensity. The few necessary correction factors and methods for data extraction have been described. The major improvement of the method is the very fast sampling of large amounts of volume data, which is increasingly important, considering the restrictions on the amount of available synchrotron beamtime. Although we describe the approach in the context of a large diffractometer and synchrotron radiation, the use of an efficient two-dimensional detector, such as the PILATUS 100K, will allow similar volume data sets to be obtained in other diffraction setups.

The method was used to map the crystal and facet structures of MBE-grown InAs nanowires. By varying the incoming angle of the X-rays, the wires were shown to consist mainly of the wurtzite structure with an in-plane lattice constant of $a = 4.284$ Å, dictated by the bulk InAs, and a c/a ratio of 1.641, giving a slightly expanded structure. The visible twin faults were mainly present as surface growth. The wires grew epitaxially on the wafer surface, but displayed two different sets of hexagonal cross section with {100} and {110} wurtzite facets.

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