Sensitivity of Bragg surface diffraction to analyze ion-implanted semiconductors

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(Received 12 May 1997; accepted for publication 3 September 1997)

A special case of the x-ray multiple diffraction phenomenon, the Bragg surface diffraction (BSD), has been investigated under lattice damage due to ion implantation in GaAs (001) samples. The BSD profile is very sensitive to the diffraction regime (dynamical or kinematical) and provides information regarding crystalline perfection and lattice strains in both directions—parallel and perpendicular—to the sample surface. Results from grazing-incidence x-ray diffraction and reciprocal space mapping are also reported. © 1997 American Institute of Physics. [S0003-6951(97)00844-9]

Multiple diffraction of x rays in crystals is a phenomenon which occurs when several sets of atomic planes simultaneously satisfy Bragg’s law.1 The most important application of this phenomenon is the experimental determination of the x-ray reflection phase,2–5 where the interference of the multiply diffracted amplitudes inside the crystal gives rise to asymmetries in the intensity profiles. From these asymmetries, the phase of the structure factor for the involved reflection can be extracted.6

Beyond the occurrence of hybrid multiple diffraction in epitaxial heterostructures,7,8 the investigation of Bragg surface diffraction (BSD) provides a new perspective to analyze crystal surfaces. When a BSD with forbidden or very weak Bragg reflection is chosen, the peak profile is exclusively affected by the in-plane crystal surface perfection which affects the regime of diffraction, dynamical or kinematical. Under dynamical (kinematical) diffraction, the momentum is transferred by the surface in a primary (secondary) extinction process.

Recently,9 the effects of surface finishing processes in semiconductors have been investigated by mapping the BSD (MBSD). Except for porous silicon, the broadening of the BSD peaks were attributed to the in-plane misorientation of dynamically diffracting regions at the surface.

In this letter, we further investigate BSD profile under lattice damage due to Se+ ion implantation in GaAs (001) crystals. The ion implantation is just a tool used to produce surface defects, without introducing any significant changes in the misorientation of the diffracting regions (in-plane mosaic spread), checked by grazing-incidence x-ray diffraction (GIXD).10,11 The anomalous broadening observed in the BSD profile is the focus of our discussion. Its interpretation allows semi-quantitative analysis of the lattice damage due to the ion implantation.

In order to generate a three-beam simultaneous case the crystal is first aligned by ω rotation for the symmetric Bragg reflection, the primary reflection. The rotation (φ axis) around the reciprocal lattice vector of the primary reflection brings the additional set of planes, the secondary reflection, also to diffract the incident beam. Secondary beams in the surface-parallel direction are BSD cases. The full width at half maximum (FWHM) measured in combined ω and φ scans (i.e., MBSD) are hereafter labeled as Wω and Wφ, respectively.

The MBSD (ω,φ scan) were performed using a collimated x-ray beam from a microfocus generator. It provides an effective circular divergence of 63″ and an illuminated area at the sample of π×(0.17/sinθc)×0.17 mm2 (~0.33 mm2). The 002/111 (G/L reflections) BSD case was chosen for our measurements.

The contribution of both Cu Kα1 and Cu Kα2 wavelengths are present in these maps. While in Figs. 1(a) and 1(b) only the peaks of the GaAs lattice matrix appear, in Figs. 1(c), 1(d), and 1(e) extra peaks due to the distorted lattice region (implanted layer) appear in doublet together with those from the GaAs matrix.

The fundamental point here is the marked differences in the BSD profiles as a function of the implantation conditions. These profiles are quantitatively compared in terms of Wφ and Wω values (Table I). Since the Cu Kα1 matrix peak partially overlaps the Cu Kα2 layer peak, in Figs. 1(c), 1(d), and 1(e), the FWHM of the matrix ones were measured from their Cu Kα2 contribution. The most significant difference from one sample to another is in the Wφ value since it changes in a range from ~85° to ~270°. Moreover, in all measurements Wφ is always wider than Wω, which has practically the same value, being Wω for all matrix (layer) peaks about 40° (~60°). Then, these results are another experimental observation of the anomalous Wφ broadening of the BSD condition. As far as we know, it has only been identified in porous silicon.9

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Such broadening can only occur under a dynamical diffraction, i.e., when primary extinction is the dominant process in which the momentum is transferred by the surface-detour reflection. If secondary extinction (kinematical diffraction) would dominate, \( W_\phi \) would be wider than \( W_\phi \). Therefore, we are reporting here changes in the BSD profile even when only the dynamical regime of diffraction is taking place.

In order to verify the nature of the \( W_\phi \) broadening, GIXD was employed. The rocking curves around the in-plane (220) diffraction vector were performed in the same setup used for MBSD. The incidence and reflection angles are about 0.4°. In all samples, the FWHM of the in-plane rocking curve (\( W_{\text{GIXD}} \) in Table I) is much narrower than the respective \( W_\phi \). Therefore, an in-plane mosaic spread is unable to explain the \( W_\phi \) broadening. Moreover, reciprocal space mapping was also performed in one sample (S08) in order to distinguish between mosaic spread and lattice strain peak broadening. [The diffractometer of the x-ray diffraction beam line (at LNLS) has been used for this measurement.] It is shown in Fig. 2, in the range where the 004 matrix (Cu \( K\alpha_1 \)) and layer (Cu \( K\alpha_2 \)) rods are present. Contributions from both wavelengths are observed since just a linear slit was used to collimate the incident beam from the x-ray tube. A Si (111) analyzer crystal was used in the measurement. From this figure, only lattice strain is observed along the rods.

In two-beam x-ray diffraction experiments, the reciprocal lattice points (r.l.p.) are better described by rods, as the one in Fig. 2 (GaAs). The rods (volume of the r.l.p.) do exist, even for a perfect crystal, as a consequence of a finite length-scale of the diffracting lattice. This finite lengthscale (with length= \( D \)) is responsible for the broadening of the rod according to \( W_\phi(D) = 0.5/\sin \alpha \xi(D) \), where \( \xi = \Delta \omega - \Delta \theta/2 \) and \( \Delta \theta \) are the angles used to map the r.l.p.

Essentially, in this letter we investigate how a finite lengthscale in the surface-parallel direction (introduced by ion implantation) does broaden the BSD profile. In a two-beam experiment, the Ewald sphere construction allows us to visualize broadening of the diffraction condition in the reciprocal space. This construction does not clearly express the broadening effects of the BSD condition, which comes from the surface-detour reflection where two consecutive reflections are involved. The BSD profile is the convolution of the

![Fig. 1. Mapping of the 002/111 Bragg surface diffraction (MBSD) for the samples in Table I: (a) S12, (b) S02, (c) S01, (d) S09, and (e) S08. Cu \( K\alpha_1 \) and Cu \( K\alpha_2 \) contributions are present.](image)

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**TABLE I.** Identification of the used samples with the implantation conditions, FWHM measured from MBSD (\( W_\phi \) and \( W_\omega \)) and GIXD (\( W_{\text{GIXD}} \)), and the lattice coherence length in the surface-parallel direction, \( D \), according \( W_\phi(D) \). GaAs (001) samples were implanted with Se\(^{79} \) at room temperature and normal incidence geometry.

<table>
<thead>
<tr>
<th>Sample</th>
<th>MBSD</th>
<th>Energy (keV)</th>
<th>Dose (( 10^{14} ) ions/cm(^2 ))</th>
<th>Matrix ( W_\phi ) (arcsec)</th>
<th>Matrix ( W_\omega ) (arcsec)</th>
<th>Layer ( W_\phi ) (arcsec)</th>
<th>Layer ( W_\omega ) (arcsec)</th>
<th>( D ) (nm)</th>
</tr>
</thead>
<tbody>
<tr>
<td>S12</td>
<td>Fig. 2(a)</td>
<td>—</td>
<td>—</td>
<td>120</td>
<td>40</td>
<td>457</td>
<td>—</td>
<td>—</td>
</tr>
<tr>
<td>S02</td>
<td>Fig. 2(b)</td>
<td>80</td>
<td>6</td>
<td>196</td>
<td>39</td>
<td>280</td>
<td>—</td>
<td>70</td>
</tr>
<tr>
<td>S01</td>
<td>Fig. 2(c)</td>
<td>80</td>
<td>15</td>
<td>88</td>
<td>40</td>
<td>623</td>
<td>83</td>
<td>60</td>
</tr>
<tr>
<td>S09</td>
<td>Fig. 2(d)</td>
<td>120</td>
<td>3</td>
<td>266</td>
<td>39</td>
<td>206</td>
<td>125</td>
<td>58</td>
</tr>
<tr>
<td>S08</td>
<td>Fig. 2(e)</td>
<td>160</td>
<td>3</td>
<td>165</td>
<td>37</td>
<td>332</td>
<td>144</td>
<td>66</td>
</tr>
</tbody>
</table>
diffraction condition for the secondary and coupling reflections. Such convolution has been carried out in Appendix B of Ref. 9. There it has been shown that in a perfect crystal region with columnar shape, where its length is longer than the diameter (D), \( W_T \) depends on D according to \( W_T(D) = 0.33/a \sin \theta_0 (D/a) \), \( \gamma \) being the angle between the reciprocal lattice vectors of \( G \) and \( L \) reflections. To clarify this, the meaning of \( D \) is the dimension of the perfect diffraction regions in the surface-parallel direction or, in other words, the lattice coherence length in this direction. In a crystal with a long range order of perfection, \( D \) would be assigned to the spatial coherence length of the incoming x rays.

Since the \( W_T \) broadening reflects lattice coherence reduction to the ion implantation damage, the MBSD measurements appearing in Fig. 1 are interpreted as written below. For each sample, the surface lattice coherence length (\( D \)) is schematically illustrated in the figures.

Broadening of the BSD peaks is observed for GaAs (001), as in Fig. 1(a), even in the absence of any ion implantation. It is an in-plane imperfection introduced by the surface finishing process. Although an in-plane mosaic spread could explain this type of effect, GIXD measurements have excluded this possibility since \( W_T(120°) > W_{\text{GIXD}}(45°) \) (Table I). On the other hand, from \( W_T(D) \), a lattice coherence length of 457 nm can be assigned to the surface of this crystal.

In Fig. 1(b), an accentuated \( W_T(196°) \) broadening is observed for the S02 implanted sample. A \( D \) value of 280 nm is obtained on this surface. It confirms that implantation damage enlarges \( W_T \) as a consequence of the reduction (61% in this case) in the surface lattice coherence length. It should be noted that this reduction also enlarges \( W_{\text{GIXD}} \) at the same rate (\( \sim 1.6 \)) of \( W_T \), but the GIXD result is less sensitive to it.

For the next three samples (S01, S08, S09), the implanted layer peaks are present. From Table I (\( D \) value), it is observed that the coherence length of the layers are, in all cases, longer than the ones assigned to the respective matrix. The release of the constraints with the matrix (lattice mismatch) and local heating gives rise to a mechanism of self-organization in the damaged lattice. It is reflected as an increase in the surface coherence length of the layer lattice.

The longest coherence lengths, 660 and 623 nm, are observed in sample S01 [Fig. 1(c)] for layer and matrix lattices, respectively. They show that most of the damage has not been propagated to the matrix below the implanted layer where it has been minimized by the self-organization mechanism. On the other hand, the coherence length of the matrix is shortened when the implantation takes place at higher energy as in samples S09 and S08 [Figs. 1(d) and 1(e)]. Then, the damage is not limited to the distorted layer lattice in these cases.

The BSD intensity from the implanted layer has been detected in a ratio of about 1/2 regarding the matrix contribution in all cases, whereas 004 symmetric rocking curves have provided a smaller ratio of about 1/8. The BSD probed depth is of the order of the layer thickness (\( \sim 1 \mu m \)) estimated from TRIM code\(^\text{12} \)) and it shows that the BSD technique is more sensitive to detect surface defects in comparison with the two-beam symmetric diffraction.

In conclusion, we have shown experimental evidence of the reduction of the lattice coherence length (perfect diffraction block dimension) along the surface-parallel direction to be responsible for the broadening of the BSD profile only in \( \phi \) scan. This effect does occur only under dynamical diffraction and therefore the reduction should not be severe enough to give rise to a total kinematical diffraction. The range of sensitivity goes from the minimum block dimension necessary to avoid secondary extinction (\( \sim 200 \) nm) to the x-ray coherent length of a given setup (\( \sim 700 \) nm in our case). In this range, a mechanism of lattice self-organization to increase long range order has been observed. The best example is in Fig. 1(d) where \( D \) increases from 206 to 438 nm.

The MBSD profile sensitivity to detect changes in implantation conditions can be used as an alternative method to probe surface damage in semiconductors with a very simple experimental setup where monochromator and analyzer crystals are not needed.

The authors would like to thank CNPq, FAPESP, and FAEP/UNICAMP for financial support and especially RHAE/CNPq (Project No. 460320/96-3) for a fellowship to one of us (SLC).

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