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X-ray Determination of the Dislocation Densities in Semiconductor Crystals using a Bartels Five-Crystal Diffractometer

By P. D. Healey, K. Bao, M. Gokhale, J. E. Ayers* and F. C. Jain

Electrical and Systems Engineering Department, University of Connecticut, Storrs, CT 06269-3157, *USA*

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Abstract

Recently, a general technique for the measurement for the threading dislocation densities in epitaxic semiconductors by high-resolution X-ray diffraction was reported [Ayers (1994). *J. Cryst. Growth*, **135**, 71–77]. Here, this method has been extended to the case of a Bartels five-crystal diffactometer by making use of known instrumental effects for this diffractometer. The usefulness of the method has been demonstrated by application of the technique to epitaxic ZnSe grown on GaAs (001) by photo-assisted metalorganic vapor-phase epitaxy. It is shown that in this case the threading dislocation density of the epitaxic layer can be determined quantitatively. Evidence for the introduction of dislocations in the underlying GaAs substrate is also presented.

Introduction

We recently reported a non-destructive X-ray technique for the determination of threading dislocation densities in semiconductor epitaxic layers (Ayers, 1994). This technique requires the measurement of several *hkl* diffraction profiles at different Bragg angles with a high-resolution diffractometer. The dislocation density can then be readily determined from the observation of two different phenomena: the first is the distortion of the orientation of diffracting planes by the dislocations and the second is the distortion of the spacing of the diffracting planes by the dislocations. In this paper, we have extended the aforementioned technique to the case of a Bartels five-crystal diffractometer (Bartels, 1983*a*,*b*) by accounting for the instrumental effects particular to the Bartels diffractometer. We have applied this technique to the characterization of epitaxic ZnSe grown on GaAs (001) by photo-assisted metalorganic vapor-phase epitaxy.

Theory

The X-ray rocking curve measured from a semiconductor crystal using a Bartels five-crystal diffractometer (Bartels, 1983*a*,*b*) has a characteristic full width at half-maximum (FWHM) which is determined by the specimen crystal and also instrumental effects (Healey & Ayers, 1994). The rocking curve FWHM for the specimen is determined by X-ray extinction, the crystal size, the crystal curvature, and also by the threading dislocation density. Dislocations broaden the rocking curve in two different ways: (i) by angular broadening; (ii) by strain broadening. Angular broadening occurs because each dislocation introduces a rotation of the crystal lattice, thus directly broadening the rocking curve. Strain broadening results from the strain fields surrounding dislocations, in which the Bragg angle of the crystal is non-uniform. The instrumental broadening introduced by the Bartels five-crystal diffractometer (this instrument is shown schematically in Fig. 1) arises from the wavelength spread, the horizontal divergence and the vertical divergence of the probing X-ray beam.

We assume that the X-ray rocking curve is Gaussian in shape and that it represents the convolution of a number

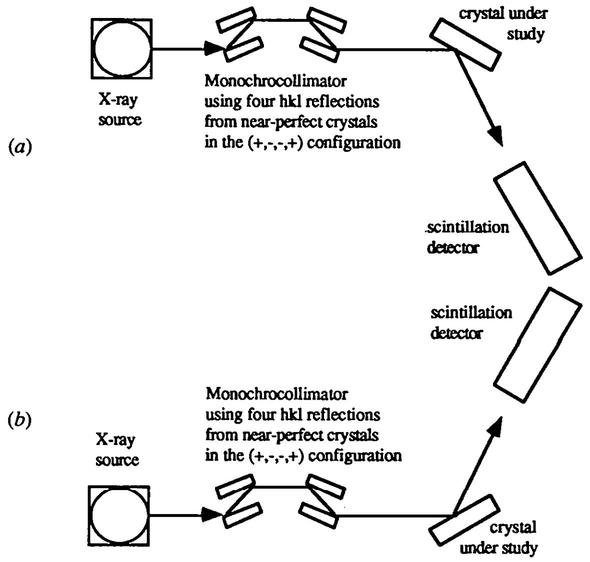

Fig. 1. Schematic of the Bartels five-crystal X-ray diffractometer, showing the two experimental configurations. (*a*) (+, −, −, +, −) configuration and (*b*) (+, −, −, +, +) configuration.

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* Author for correspondence.

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of Gaussian intensity distributions. It is also assumed that the specimen being examined is a uniform single crystal (so that the lattice constant is not graded due to changes in composition). Then, if $\beta_m(hkl)$ is the measured rocking curve FWHM for the hkl reflection, it follows that

$$[\beta_m(hkl)]^2 = [\beta_0(hkl)]^2 + [\beta_L(hkl)]^2 + [\beta_r(hkl)]^2 + [\beta_{\text{dislocations}}(hkl)]^2 + [\beta_{\text{instrument}}(hkl)]^2, \tag{1}$$

where $\beta_0(hkl)$ is the natural rocking-curve width for a perfect crystal of the specimen material, as determined by extinction, $\beta_L(hkl)$ represents the broadening of the rocking curve by finite crystal size, $\beta_r(hkl)$ represents the broadening of the rocking curve introduced by specimen curvature, $\beta_{\text{dislocations}}(hkl)$ represents the broadening of the rocking curve introduced by the threading dislocations in the specimen and $\beta_{\text{instrument}}(hkl)$ represents the broadening of the rocking curve introduced by the instrumental effects. In general, each of the terms in (1) is a function of the indices of the reflection hkl. For the purpose of making the equations more compact, we have omitted explicit references to hkl in our notation for the remainder of this paper.

The natural rocking-curve widths for perfect crystals can be calculated as described by Warren (1969) for $(\Theta_B + \varphi)$ incidence as

$$\beta_0 = [r_e\lambda^2(1 + |\cos 2\Theta_{B,\text{specimen}}|)|F_{hkl}|] \times [\sin(\Theta_{B,\text{specimen}} - \varphi)/\sin(\Theta_{B,\text{specimen}} + \varphi)]^{1/2} \times [\pi a_0{}^3 \sin(2\Theta_{B,\text{specimen}})]^{-1}, \tag{2}$$

where r_e is the classical electron radius, 2.818×10^{-5} Å, λ is the X-ray wavelength, $\Theta_{B,\text{specimen}}$ is the Bragg angle for the crystal being measured, $|F_{hkl}|$ is the magnitude of the structure factor for the hkl reflection, a_0 is the lattice constant of the (cubic) diffracting crystal and φ is the (positive) angle between the crystal surface and the diffracting planes. Values of natural rocking-curve widths can be obtained from standard X-ray tables for semiconductor characterization (*e.g.* Ayers, 1990).

The broadening due to crystal size is usually only affected by the layer thickness in single-crystal heteroepitaxic semiconductors. It is given approximately by (Scherrer, 1918)

$$\beta_L{}^2 = [4\ln 2/(\pi h^2)](\lambda^2/\cos^2\Theta_{B,\text{specimen}}), \tag{3}$$

where h is the thickness of the semiconductor layer. This contribution is usually negligble for reflections from reasonably thick epitaxic semiconductor layers as well as semiconductor substrate crystals.

Rocking-curve broadening due to specimen curvature has been described previously and is given as (Flanagan, 1959; Halliwell, Lyons & Hill, 1984)

$$\beta_r{}^2 = w^2/r^2 \sin^2\Theta_{B,\text{specimen}}, \tag{4}$$

where w is the width of the X-ray beam in the diffraction plane and r is the radius of curvature for the specimen. Usually, mismatched heteroepitaxic layers are deposited on substrates that are one hundred times thicker, so that the curvature and its associated broadening are negligible. Since the curvature broadens the rocking curves from the epitaxic layer and substrate equally, it is possible to make an upper estimate of the effect of curvature by measuring the substrate rocking curve.

The broadening of the rocking curve FWHM associated with the threading dislocation density is given by (Ayers, 1994)

$$(\beta_{\text{dislocations}})^2 = K_\alpha + K_\varepsilon \tan^2\Theta_{B,\text{specimen}}, \tag{5}$$

where $\Theta_{B,\text{specimen}}$ is the Bragg angle of the specimen reflection and K_α and K_ε are constants for the specimen given by (Gay, Hirsch & Kelly, 1953; Dunn & Koch, 1957; Hordon & Avenbach, 1961; Ayers, 1994)

$$K_\alpha = 2\pi \ln 2 b^2 D \qquad \text{(dislocations of any character)}$$

and

$$K_\varepsilon = \begin{cases} 0.160b^2D|\ln(2\times10^{-7}D^{1/2})| & \text{(60° dislocations)}, \\ 0.090b^2D|\ln(2\times10^{-7}D^{1/2})| & \text{(screw dislocations)}. \end{cases} \tag{6}$$

Here, b is the length of the Burgers vector for dislocations in the specimen (for zinc blende semiconductor crystals, $b = a_0/2^{1/2}$, where a_0 is the lattice constant) and D is the threading dislocation density per square centimeter in the specimen. Physically, K_α stems from the angular broadening and the associated effect is independent of the specimen Bragg angle. K_ε is related to the strain broadening, for which the effect is dependent on the specimen Bragg angle, as expected from the differential form of the Bragg equation.

The instrumental broadening of the diffraction profiles compromises three components which are affected by the wavelength spread, horizontal divergence and vertical divergence of the probing X-ray beam (Healey & Ayers, 1994):

$$(\beta_{\text{instrument}})^2 = (\beta_{\text{wavelength spread}})^2 + (\beta_{\text{horizontal divergence}})^2 + (\beta_{\text{vertical divergence}})^2. \tag{7}$$

The wavelength spread of the X-ray beam gives rise to broadening of the measured diffraction profile, which is described by a FWHM that can be determined by differentiation of the Bragg equation and that we denote $\beta_{\text{wavelength spread}}$:

$$\beta_{\text{wavelength spread}} = (\Delta\lambda/\lambda)\tan\Theta_{B,\text{specimen}}. \tag{8}$$

For the Bartels four-crystal monochromator, the wavelength spread of the X-ray beam is given by

$$(\Delta\lambda/\lambda) = \beta_{\text{monochromator}}/2\tan\Theta_{Bm}, \tag{9}$$

where $\beta_{\text{monochromator}}$ is the rocking-curve FWHM for the monochromator crystals and Θ_{Bm} is the Bragg angle for the monochromator reflections.

The horizontal divergence is in the plane of diffraction and therefore the associated profile has a FWHM equal to the horizontal divergence, which is thus equal to half of the rocking-curve width for the monochromator crystals:

$$\beta_{\text{horizontal divergence}} = \beta_{\text{monochromator}}/2. \tag{10}$$

The vertical divergence introduces broadening of the measured diffraction profiles as well, which is given by

$$\begin{aligned}\beta_{\text{vertical divergence}} &= \sin^{-1}[\sin(\Theta_{B,\text{specimen}})/\cos(\Delta\varphi/2)] \\ &\quad - \Theta_{B,\text{specimen}} \\ &\simeq (0.00148)\Delta\varphi\tan(\Theta_{B,\text{specimen}}).\end{aligned} \tag{11}$$

The approximate expression was determined numerically for small values of $\Delta\varphi$ in the following manner. Numerical values for $\beta_{\text{vertical divergence}}$ were plotted *versus* $\tan(\Theta_{B,\text{specimen}})$ for the range $10 \leq \Theta_{B,\text{specimen}} \leq 80°$ and for cases $\Delta\varphi = 0.25$, 0.5, 0.75 and 1.0°. The resulting plots are straight lines, given approximately by (11). The vertical divergence of the Bartels four-crystal monochromator is given by

$$\Delta\varphi = 2\cos^{-1}[\sin(\Theta_{Bm} - \beta_{\text{monochromator}}/4)/\sin(\Theta_{Bm})]. \tag{12}$$

The above results may be summarized for the general case of the Bartels five-crystal diffractometer as follows (Healey & Ayers, 1994):

$$\begin{aligned}(\beta_{\text{instrument}})^2 &= [\beta_{\text{monochromator}}/(2\tan\Theta_{Bm})]^2 \\ &\quad \times \tan^2[\Theta_{B,\text{specimen}}] + (\beta_{\text{monochromator}}/2)^2 \\ &\quad + (8.8\times 10^{-6}\{\cos^{-1}[\sin(\Theta_{Bm} \\ &\quad - \beta_{\text{monochromator}}/4)/\sin(\Theta_{Bm})]\}^2 \\ &\quad \times \tan^2(\Theta_{B,\text{specimen}}).\end{aligned} \tag{13}$$

Now, for the case of a Ge 022 Bartels monochromator used in the (+, −, −, +) configuration at a temperature of 293 K and using the Cu $K\alpha_1$ X-ray line ($\lambda = 1.540594$ Å), the instrumental contributions to the measured width of the diffraction profiles may be calculated using (13) to be

$$\begin{aligned}(\beta_{\text{wavelength spread}})^2 &= 197\tan^2(\Theta_{B,\text{specimen}}), \\ (\beta_{\text{horizontal divergence}})^2 &= 36, \\ (\beta_{\text{vertical divergence}})^2 &= 26\tan^2(\Theta_{B,\text{specimen}}),\end{aligned} \tag{14}$$

where the β^2 terms are in units of $('')^2$. Thus, with the wavelength spread, horizontal divergence and vertical divergence of the instrument using a Ge 022 monochromator taken into account, it has been shown that the instrumental broadening is given by

$$(\beta_{\text{instrument}})^2 = 36 + 223\tan^2(\Theta_{B,\text{specimen}}). \tag{15}$$

Once the instrumental effects are known, the threading dislocation density in a semiconductor specimen crystal can be determined as follows. The diffraction profile widths (FWHMs) are determined for three or more *hkl* reflections at different Bragg angles. Then, for each measurement, the broadening due to threading dislocations is extracted from the measured width by accounting for the natural width of the profile and the instrumental broadening:

$$(\beta_{\text{dislocations}})^2 = \beta_m^2 - \beta_0^2 - (\beta_{\text{instrument}})^2. \tag{16}$$

Tacit here is the assumption that the contributions of the crystal size and curvature may be neglected. Then, if $(\beta_{\text{dislocations}})^2$ is plotted *versus* $\tan^2(\Theta_{B,\text{specimen}})$, a straight line is obtained with intercept K_α and slope K_ε. The threading dislocation density may then be determined independently by two calculations. First, the threading dislocation density may be determined from the amount of angular broadening introduced by dislocations from

$$D = K_\alpha/(4.36b^2) \qquad \text{(angular broadening)}. \tag{17}$$

Secondly, the threading dislocation density may be determined from the amount of strain broadening introduced by dislocations:

$$D = K_\varepsilon/[0.090b^2|\ln(2\times 10^{-7}D^{1/2})|] \qquad \text{(strain broadening)}. \tag{18}$$

This equation must be solved in an iterative fashion for the determination of D. It should be noted that (18) was derived for the case of threading dislocations having screw character. However, strain broadening introduced by the threading dislocations depends rather weakly on their character (Ayers, 1994) so that the above relationship can be used with less than a factor of two error.

Experimental

We have applied the technique described above to epitaxic ZnSe grown by ultraviolet assisted organometallic vapor-phase epitaxy (OMVPE) on GaAs (001). The epitaxic ZnSe on GaAs (001) was prepared as follows. The GaAs substrate was cut from a 3 in wafer, 375 µm thick, grown by the liquid-encapsulated Czochralski technique and supplied by Sumitomo with a dislocation etch-pit density less than 10^4 cm^{-2}. The epitaxic ZnSe (001) was grown by photo-assisted low-pressure (40 kPa) organometallic vapor-phase epitaxy at

673 K using an EMCORE vertical stainless steel reactor with a resistance-heated rotating (400 r min^{-1}) molybdenum susceptor. The carrier gas was palladium-diffused hydrogen at a total flow of 14.25 standard l min^{-1}. The alkyl sources dimethylzinc ($X = 1.0 \times 10^{-4}$) and dimethylselenide ($X = 2.0 \times 10^{-4}$) were employed. The epitaxic layer was cadmium doped by dimethylcadmium ($X = 1.7 \times 10^{-5}$) to a level of approximately 3×10^{20} cm^{-3}. The photo-irradiation during growth was achieved using a Bausch and Lomb SP-200 mercury arc lamp (200 W electrical power). Prior to the photo-assisted growth of ZnSe, an undoped ZnSe buffer was grown without photo-irradiation at 868 K, 40 kPa, 14.25 standard l min^{-1} total flow and a rotation rate of 400 r min^{-1}. The reactant mole fractions were 1.0×10^{-4} (dimethylzinc) and 2.0×10^{-4} (dimethylselenide), resulting in 0.3 µm thickness after 20 min of growth. The total film thickness for the ZnSe (001) epitaxic layer was 1.6 µm. This thickness was determined by the following procedure. Part of the epitaxic layer was masked by the application of melted black wax. Then the masked epitaxic layer was etched in bromine/methanol at a slow rate (0.2 µm min^{-1}) until the interference fringes of the epitaxic layer disappeared. Then the wax mask was removed and the resulting step height was determined using a mechanical profiler (Tencor Instruments Alpha Step).

X-ray diffraction profiles were measured using a Blake high-resolution diffractometer with Cu $K\alpha_1$ radiation ($\lambda = 1.540594$ Å). The Philips X-ray source was operated at 30 kV and 10 mA. The line-focused beam was slit limited to 5 mm length normal to the plane of the diffractometer and 0.5 mm width in the plane of the diffractometer by pairs of slits placed on either side of the monochromator. The spacing between the slits was 210 mm. A four-crystal Bartels-type monochromator was employed using four Ge 022 reflections from Ge (011) crystals arranged in the $(+, -, -, +)$ geometry. Diffraction profiles were obtained for the 113, 004, 115, 335, 444 and 117 reflections with both the $(+, -, -, +, -)$ and $(+, -, -, +, +)$ geometries for both the ZnSe epitaxic layer and its GaAs substrate. For each measurement, the specimen tilt was adjusted to bring the specimen diffraction vector into the plane of the diffractometer. Tilt optimization was performed by adjusting the tilt for maximum peak reflected intensity and with a precision of ±0.5°. Typical intensities measured with a Bicron scintillation counter were 1750 counts s^{-1} for the GaAs 004 reflection and 240 counts s^{-1} for the ZnSe 004.

The widths of the measured diffraction profiles (FWHMs) were determined by least-squares fitting to a Gaussian or Lorentzian profile. The broadening introduced by dislocations was determined from the experimentally determined width by removal of the instrumental broadening and the natural width for the observed rocking curve *via* (16).

Results and discussion

Fig. 2 shows typical diffraction profiles for the GaAs (001) substrate. Diffraction profiles measured at low Bragg angles, such as the 004 profile shown in Fig. 2(*a*), were fit most closely by a Lorentzian profile. The broader profiles measured for the GaAs substrate at higher Bragg angles were best fit using a Gaussian curve. An example is the 117 reflection, as shown in Fig. 2(*b*). All profiles measured for the ZnSe epitaxic layer were best fit using a Gaussian curve. An example is the ZnSe 004 profile shown in Fig. 3.

Fig. 4 shows $(\beta_{\rm dislocations})^2$ *versus* $\tan^2(\Theta_{B,{\rm specimen}})$ for the ZnSe epitaxic layer. The data may be fit by a straight line given by

$$(\beta_{\rm dislocations})^2 = 23\,700 + 2480\tan^2(\Theta_{B,{\rm specimen}}). \quad (19)$$

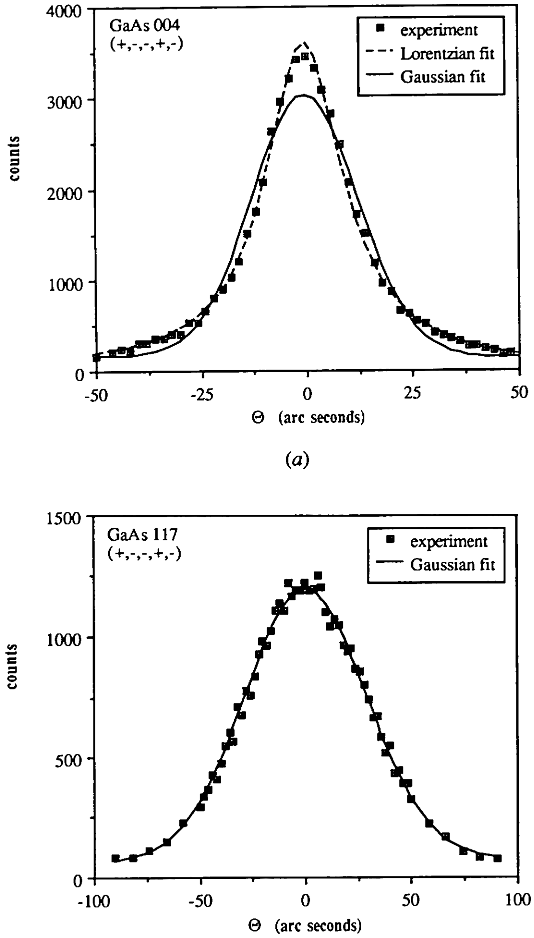

Fig. 2. (*a*) The experimentally measured 004 diffraction profile for the GaAs (001) substrate (points) shown with the Lorentzian best fit (dashed lines) and the relatively poor Gaussian fit (solid line). (*b*) The experimentally measured 117 diffraction profile for the GaAs (001) substrate (points) shown with the Gaussian best fit (solid line).

Thus, the calculated dislocation density is $8.0 \times 10^7\,\mathrm{cm}^{-2}$ (from $K_\alpha = 23700''^2$) and $6.3 \times 10^7\,\mathrm{cm}^{-2}$ (from $K_\varepsilon = 2480''^2$). The two calculated values of the threading dislocation density are in very good agreement. It should be noted that we have assumed there is no broadening of the diffraction profiles from the epitaxic layer due to layer thickness or curvature. The broadening due to the layer thickness is known to be negligible for a 1.6 µm thick layer of ZnSe (Scherrer, 1918; Ayers, 1994). The broadening due to curvature (Halliwell, Lyons & Hill, 1984; Flanagan, 1959) would be introduced equally for the substrate and the epitaxic layer and will be shown to be insignificant.

In Fig. 5, we show the same type of data, $(\beta_{\mathrm{dislocations}})^2$ *versus* $\tan^2(\Theta_{B,\mathrm{specimen}})$ for the GaAs (001) substrate. Here, the data follow a straight line reasonably well. The scatter is more pronounced because the measured widths are less than those for the ZnSe. Thus, the experimental errors, such as those due to imperfect tilt optimization for the specimen, are exaggerated. The least-squares best fit to the data is given by

$$(\beta_{\mathrm{dislocations}})^2 = 269''^2 + 17.0''^2 \tan^2(\Theta_{B,\mathrm{specimen}}). \quad (20)$$

Thus, the calculated dislocation density is $9.1 \times 10^5\,\mathrm{cm}^{-2}$ (from $K_\alpha = 269''^2$) and $3.1 \times 10^5\,\mathrm{cm}^{-2}$ (from $K_\varepsilon = 17''^2$). Once again, crystal-size broadening and curvature have been neglected. Crystal-size broadening is less than 1″ for a 375 µm thick GaAs crystal. The data also show that the broadening due to curvature is negligible because broadening of this type is inversely proportional to the sine of the angle of incidence and would be maximum for the 113 reflection (Halliwell, Lyons & Hill, 1984; Flanagan, 1959).

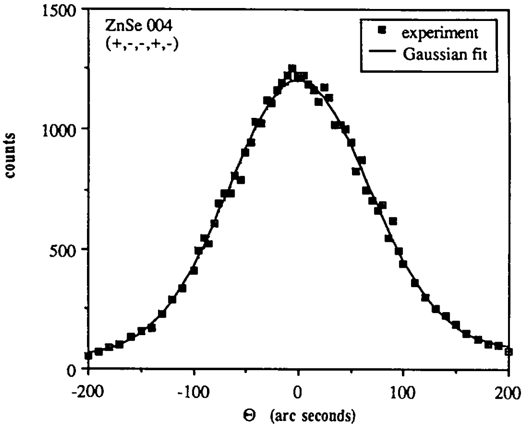

Fig. 3. The experimentally measured 004 diffraction profile for the ZnSe epitaxic layer (points) shown with the Gaussian best fit (solid line).

Care must be exercised in interpreting the substrate data because the threading dislocation density is expected to be highly non-uniform in the substrate. Specifically, the dislocation density is expected to be maximum near the interface. In such a case, the dislocation density determined by the present method is a weighted average and the maximum threading dislocation density (at the interface) is expected to be greater. Moreover, the nature of the weighting in the average depends on the extinction depth and is different for the different X-ray reflections. This, as well as experimental errors, may explain the pronounced scatter of the data and also the marginal agreement between the two calculated values of the threading dislocation density, for the case of the GaAs substrate.

For the purpose of comparison, we determined the same rocking curve FWHM data for a bare GaAs substrate which did not have a ZnSe epitaxic layer. These data are plotted in Fig. 6. The bare GaAs substrate is expected to exhibit negligible broadening of the rocking

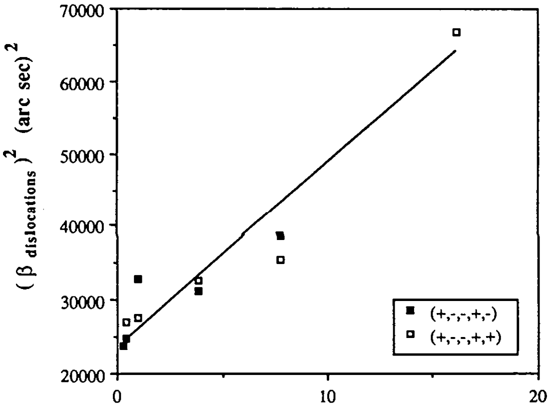

Fig. 4. $(\beta_{\mathrm{dislocations}})^2$ *versus* $\tan^2(\Theta_{B,\mathrm{specimen}})$ for the epitaxic ZnSe on GaAs (001). The staight line is a least-squares best fit to the experimental data, shown as squares.

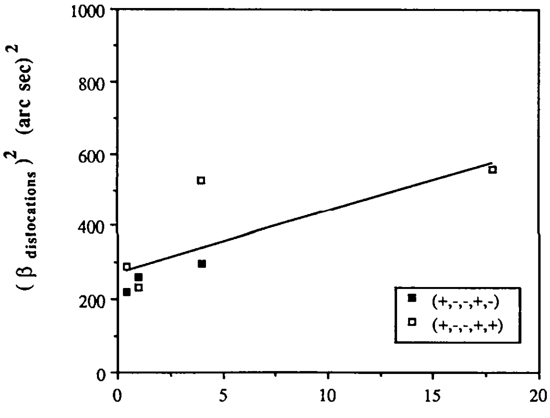

Fig. 5. $(\beta_{\mathrm{dislocations}})^2$ *versus* $\tan^2(\Theta_{B,\mathrm{specimen}})$ for the GaAs (001) substrate. The straight line is a least-squares best fit to the experimental data, shown as squares.

curves due to dislocations because the etch-pit density specified by the manufacturer was < $10^4\,cm^{-2}$. Therefore, it was expected that all values of $\beta_{dislocations}$ would be zero, within the experimental error. In fact, the values of $\beta_{dislocations}$ became increasingly negative with increasing Bragg angle and absolute values as large as 25″ were observed. This indicates that the theory overestimates the instrumental broadening of the Bartels diffractometer by a small amount. This overestimation is probably due to the starting assumption of Gaussian profiles, and it will introduce errors in the threading dislocation density values that have been determined. The error is not important in the case of the epitaxic ZnSe but will be larger for measurements of more perfect crystals. In the case of the GaAs substrate, the error is significant, but less than a factor of two judging from the slopes of the characteristics shown in Figs. 5 and 6. In general, it is expected that the method used here can attain better than a factor of two accuracy for crystals having a dislocation density greater than about $5 \times 10^5\,cm^{-2}$. Furthermore, if the instrumental effects are determined empirically, factor of two accuracy can be attained for dislocation densities almost an order of magnitude lower. Thus, this method may be applied to almost all heteroepitaxic semiconductor materials.

The above results for the GaAs substrate material, with and without a ZnSe epitaxic layer, show conclusively that the process of heteroepitaxic growth introduces threading dislocations into the substrate. For the ZnSe on GaAs (001) studied here, the substrate dislocation density is about two orders of magnitude less than the dislocation density in the epitaxic layer. It is interesting to note that the substrate is also about two orders of magnitude thicker than the epitaxic layer (375 *versus* 1.6 μm). Therefore, it follows that the strain introduced in the substrate during growth would be about two orders of magnitude lower than the strain introduced in the epitaxic layer. This may explain the difference in the densities of dislocations introduced during growth.

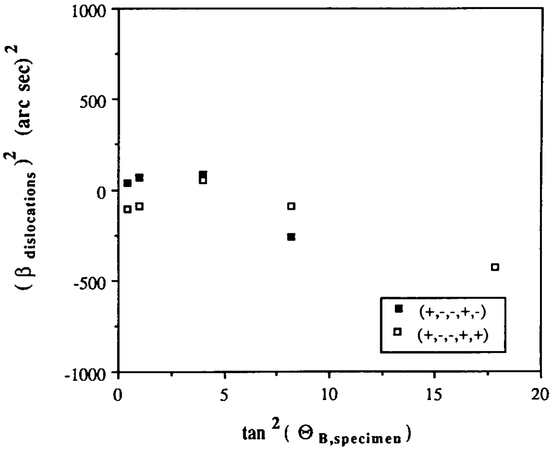

Fig. 6. $(\beta_{dislocations})^2$ *versus* $\tan^2(\Theta_{B,specimen})$ for bare GaAs (001) without a ZnSe epitaxic layer.

Concluding remarks

In conclusion, we have shown how dislocation densities can be determined for semiconductor specimens through the measurement of the widths of several *hkl* reflections using a Bartels-type five-crystal X-ray diffractometer. For the application of this technique, the known instrumental broadening of the diffraction profiles due to the Bartels diffractometer must be removed from the measured widths. Then, the threading dislocation density can be determined from the observation of the angular broadening of the dislocations and also from the observation of the strain broadening of the dislocations.

To demonstrate the usefulness of the method, we have applied this technique to epitaxic ZnSe grown on GaAs (001) by photo-assisted metalorganic vapor-phase epitaxy. We have determined the (weighted average) threading dislocation densities to be $7 \times 10^7\,cm^{-2}$ in the epitaxic layer and approximately $6 \times 10^5\,cm^{-2}$ in the GaAs substrate.

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References

Ayers, J. E. (1990). PhD dissertation, Rensselaer Polytech. Inst., Troy, NY, USA.
Ayers, J. E. (1994). *J. Cryst. Growth*, **135**, 71–77.
Bartels, W. J. (1983*a*). *J. Vac. Sci. Technol.* **B1**, 338–345.
Bartels, W. J. (1983*b*). *Philips Tech. Rev.* **41**, 183–185.
Dunn, C. O. & Koch, E. F. (1957). *Acta Metall.* **5**, 548–554.
Flanagan, W. F. (1959). ScD dissertation, Massachusetts Institute of Technology, Cambridge, MA, USA.
Gay, P., Hirsch, P. B. & Kelly, A. (1953). *Acta Metall.* **1**, 315–319.
Halliwell, M. A. G., Lyons, M. H. & Hill, M. J. (1984). *J. Cryst. Growth*, **68**, 523–531.
Healey, P. D. & Ayers, J. E. (1994). Unpublished.
Hordon, M. J. & Avenbach, B. L. (1961). *Acta Metall.* **9**, 237–246.
Scherrer, P. (1918). *Nachr. Goettingen Ges.* pp. 98–109.
Warren, B. E. (1969). *X-ray Diffraction.* Reading, MA: Addison-Wesley.