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The measurement of threading dislocation densities in semiconductor crystals by X-ray diffraction

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The measurement of threading dislocation densities in heteroepitaxial semiconductor layers is important for the development of injection lasers, microwave transistors, and also the integration of devices in disimilar semiconductors. Dislocation density measurements have been made by destructive techniques such as etching and by transmission electron microscopy (TEM). However, X-ray rocking curves provide non-destructive measurements of dislocation densities with accuracy equal to crystallographic etches or TEM. The theory of this technique has been described by Gay, Hirsch, and Kelly [P. Gay, P.B. Hirsch, and A. Kelly, Acta Met. 1 (1953) 315] and Hordon and Averbach [M.J. Hordon and B.L. Averbach, Acta Met. 9 (1961) 237], for the case of highly dislocated metal crystals. In this paper, the theory of dislocation density measurement from rocking curves is extended to the case of (001) zinc-blende semiconductors. It is shown that the measurement of several (*hkl*) rocking curve widths with a particular X-ray wavelength allows the calculation of the dislocation density by two independent techniques, thus allowing for a check of self-consistency. It is shown that for the case of epitaxial GaAs on Si(001), dislocation densities determined by these two methods are in good agreement.

1. Introduction

In recent years, there has been a growing interest in the application of mismatched heteroepitaxial semiconductors for electronic and optoelectronic devices. Examples include GaAs on Si, InP on Si, and ZnSe on GaAs. There are many advantages to the use of such materials, including integration of devices in different semiconductors, improved thermal performance and yield, and reduced cost. However, the use of mismatched heteroepitaxial semiconductors in devices is often hindered by high threading dislocation densities. It is therefore important to develop experimental techniques for the accurate determination of threading dislocation densities.

Transmission electron microscopy (TEM) and etch pit densities (EPDs) have been used to determine threading dislocation densities in mismatched heteroepitaxial layers. A disadvantage of these techniques is their destructive nature. Also, EPD measurements tend to underestimate the dislocation density for dislocation densities greater than about 5×10^6 cm^{-2} [1–3]. TEM cross-sectional measurements, on the other hand, only provide an accurate measurement of the dislocation density in highly defected crystals, due to the small crystal volume which is examined.

The X-ray rocking curve technique is complementary to the TEM and EPD methods because it is non-destructive and can be used to accurately determine dislocation densities in the range from 10^5 to 10^9 cm^{-2}. Gay, Hirsch and Kelly [4] and Hordon and Averbach [5] have described the theory of X-ray rocking curve broadening in dislocated metal crystals. In this paper, this theory is extended to the case of zinc-blende semiconductors. It is shown that the measurement of several (*hkl*) rocking curve widths with a particular X-ray wavelength allows the calculation of the dislocation density by two independent techniques, thus allowing for a check of self-consistency. It is

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shown that for the case of epitaxial GaAs on Si(001), dislocation densities determined by these two methods are in good agreement.

2. Theory

The X-ray rocking curve measured from a semiconductor crystal using a Bartels five-crystal diffractometer [6,7] or a double-crystal diffractometer in the parallel position has a characteristic full width at half maximum (FWHM) which depends on the crystal being examined and the examining wavelength, but which is insensitive to instrumental effects. Dislocations broaden the rocking curve in three ways: (i) the dislocation introduces a rotation of the crystal lattice, thus directly broadening the rocking curve; (ii) the dislocation is surrounded by a strain field, in which the Bragg angle of the crystal is nonuniform; (iii) in grossly defected crystals, dislocations can form the walls between small polycrystals, which give rise to crystal size broadening. In single-crystal semiconductor specimens, only the first two effects (rotational and strain broadening) are important.

The X-ray rocking curve is assumed to be Gaussian in shape, and to represent the convolution of a number of Gaussian intensity distributions. The assumption of a Gaussian rocking curve is supported by experimental evidence in the case of GaAs on Si(001). For example, fig. 1 compares the experimental (004) rocking curve for a 1.5 μm thick GaAs/Si(001) epitaxial layer with a Gaussian profile of the same width. The two profiles are indistinguishable, except in the tail regions. Similarly, it was found that all of the rocking curves measured for this sample, including the (002), (113), (224), (115), (006), (026), (444), and (117), were approximately Gaussian. Therefore, for the GaAs on Si(001) system, the rocking curves can be considered to be approximately Gaussian. As will be shown, isolation of the various broadening effects shows that the dislocations give rise to Gaussian intensity distributions as well. It is reasonable to expect similar behavior in other heteroepitaxial systems.

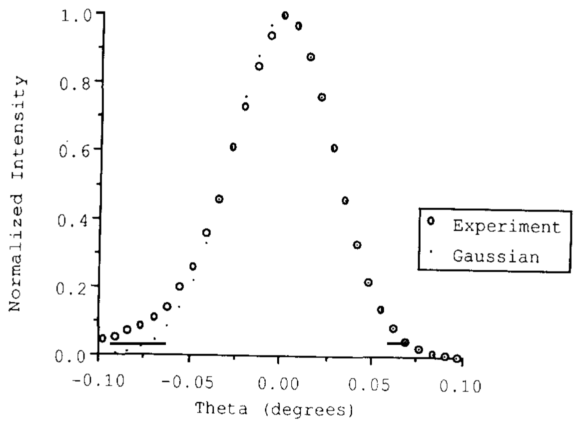

Fig. 1. GaAs(004) rocking curve for a 1.5 μm GaAs/Si(001) sample, from the data of Ayers, Schowalter, and Ghandhi [18].

Thus if $\beta_m(hkl)$ is the measured rocking curve FWHM for the (hkl) reflection, then [5]

$$\beta_m^2(hkl) = \beta_0^2(hkl) + \beta_d^2(hkl) + \beta_\alpha^2(hkl) + \beta_\epsilon^2(hkl) + \beta_L^2(hkl) + \beta_r^2(hkl), \quad (1)$$

where $\beta_0(hkl)$ is the intrinsic rocking curve width for the crystal being examined, $\beta_d(hkl)$ is the intrinsic rocking curve width for the first crystal (in the case of the double-crystal diffractometer) or the Bartels monochromator [6,7] (in the case of the five-crystal diffractometer), $\beta_\alpha(hkl)$ is the rocking curve broadening caused by angular rotation at dislocations, $\beta_\epsilon(hkl)$ is the rocking curve broadening caused by the strain surrounding dislocations, $\beta_L(hkl)$ is the rocking curve broadening due to crystal size, and $\beta_r(hkl)$ is the rocking curve broadening due to curvature of the crystal specimen.

The intrinsic rocking curve widths for $(\theta + \phi)$ incidence may be calculated as [8]

$$\beta_0(hkl) = \left[r_e\lambda^2(1 + |\cos 2\theta|)\,|F_{hkl}|\right] \times \left[\sin(\theta - \phi)/\sin(\theta + \phi)\right]^{1/2} / \left[\pi a_0^3 \sin(2\theta)\right], \quad (2)$$

where r_e is the classical electron radius, 2.818×10^{-5} Å, λ is the X-ray wavelength, θ is the Bragg angle, $|F_{hkl}|$ 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 angle between the crystal surface and the diffracting planes. Intrinsic rocking curve widths are usually less than 10 arc sec (0.0028°) and can often be neglected. For example, the intrinsic rocking curve widths for Gaas using Cu Kα radiation are 0.5 arc sec for the (002) reflection, 8.7 arc sec for the (004) reflection, and 3.5 arc sec for the (115) reflection [9]. For the Ge (220) reflection used in most Bartels monochromators, the intrinsic rocking curve width is 12 arc sec [7].

The broadening due to angular rotation at the dislocations has been described by Gay, Hirsch, and Kelly [4]. In this treatment the single crystal is considered to comprise an arrangement of subsidiary mosaics which are tilted with respect to each other. Each subsidiary mosaic is associated with a dislocation, which tilts the mosaic with respect to its neighbors. It is assumed that the orientations of the mosaics have a Gaussian distribution, so that the probability that a mosaic is tilted by an angle θ from the mean orientation is equal to $(\sigma\sqrt{2\pi})^{-1}\exp(-\theta^2/2\sigma^2)$, where σ is the standard deviation. The probability for two mosaics to be tilted with respect to each other by an angle $|\theta-\phi|$ is equal to $(\sigma\sqrt{2\pi})^{-2}\exp[-(\theta^2+\phi^2)/2\sigma^2]$. Therefore the mean disorientation between mosaics is given by

$$\overline{|\theta-\phi|} = \frac{\int_{-\infty}^{\infty}\int_{-\infty}^{\infty}|\theta-\phi|\exp\left[-(\theta^2+\phi^2)/2\sigma^2\right]\,\mathrm{d}\theta\,\mathrm{d}\phi}{\int_{-\infty}^{\infty}\int_{-\infty}^{\infty}\exp\left[-(\theta^2+\phi^2)/2\sigma^2\right]\,\mathrm{d}\theta\,\mathrm{d}\phi}. \tag{3}$$

The value of the denominator is $2\pi\sigma^2$, and Dunn and Koch [10] have integrated the numerator explicitly to obtain $4\sigma^3\sqrt{\pi}$. Therefore,

$$\overline{|\theta-\phi|} = 2\sigma/\sqrt{\pi}. \tag{4}$$

For a Gaussian distribution, the relationship between the standard deviation and the FWHM is given by $\sigma=\beta/(2\sqrt{2\ln 2})$, so that the mean disorientation between mosaics can be related to the measured FWHM by

$$\overline{|\theta+\phi|} = \beta_\alpha/(\sqrt{2\pi\ln 2}). \tag{5}$$

Now if the dislocations are arranged in a random network, with an average spacing of $1/\sqrt{D}$, where D is the dislocation density, then the average angle between neighboring mosaics is approximately

$$\overline{|\theta-\phi|} \approx b\sqrt{D}, \tag{6}$$

where b is the length of the Burgers vector. Equating (5) and (6), we can relate the measured FWHM to the dislocation density by

$$D \approx \beta_\alpha^2/(2\pi\ln 2b^2) \approx \beta_\alpha^2/(4.36b^2). \tag{7}$$

Thus the angular broadening due to dislocations is therefore given by

$$\beta_\alpha^2 = 2\pi\ln 2b^2D = K_\alpha. \tag{8}$$

The strain broadening due to dislocations has been described by Warren [11] and Hordon and Avenbach [5]. If it is assumed that the random array of dislocations gives rise to a Gaussian distribution of local strain, then this strain gives rise to rocking curve broadening given by

$$\beta_\epsilon^2 = \left[8\ln 2\left(\overline{\epsilon_N^2}\right)\right]\tan^2\theta = K_\epsilon\tan^2\theta, \tag{9}$$

where $\overline{\epsilon_N^2}$ is the mean square strain in the direction of the normal $\overline{N}$ to the diffracting planes. $\overline{\epsilon_N^2}$ may be found as follows. If it is assumed that the Poisson ratio is 1/3, then from the known strain distribution around the edge dislocation it is found that [5,12]

$$\overline{\epsilon_{N_e}^2} = \left[5b^2/(64\pi^2r^2)\right]\ln(r/r_0) \times(2.45\cos^2\Delta + 0.45\cos^2\psi), \tag{10}$$

where Δ is the angle between the dislocation glide plane normal and the normal to the diffracting planes, ψ is the angle between the dislocation Burgers vector and the normal to the diffracting planes, and r and r_0 are the upper and lower limits for integration of the strain field in the radial direction from the dislocation core. Similarly, for the pure screw dislocation, it is found that [5,13]

$$\overline{\epsilon_{N_s}^2} = \left[b^2/(4\pi^3 r^2)\right] \ln(r/r_0)(\sin^2\psi). \tag{11}$$

Eqs. (10) and (11) can be applied to 60° dislocations as follows:

$$\begin{aligned}\overline{\epsilon_N^2} &= \overline{\epsilon_{N_e}^2} \sin^2 60° + \overline{\epsilon_{N_s}^2} \cos^2 60° \\ &= \left[b^2/(\pi^2 r^2)\right] \ln(r/r_0) f(\Delta, \psi).\end{aligned} \tag{12}$$

Now first suppose that the threading dislocations in the (001) semiconductor all have 60° character. There are sixteen distinct dislocation systems as classified by line vector, glide plane, and Burgers vector. (These systems can be enumerated in a manner similar to that in ref. [14]. The difference is that the line vectors of the threading dislocations have a non-zero component perpendicular to the interface.) If symmetric X-ray reflections are considered, then for eight of these slip systems, $\psi = 45°$ and $\Delta = 60°$. For the other eight systems, $\psi = 90°$ and $\Delta = 60°$. Thus, for 60° threading dislocations, $f(\Delta, \psi) = 0.070$ for symmetric X-ray reflections. Then, if it is assumed that $r_0 = 10^{-7}$ cm [5] and that $r = 0.5/\sqrt{D}$, or one-half the average dislocation spacing, it is found using eq. (9) that

$$\begin{aligned}\beta_\epsilon^2 &= 0.160 b^2 D \,|\ln(2 \times 10^{-7}\ \mathrm{cm}\sqrt{D}\,|\tan^2\theta \\ &= K_\epsilon \tan^2\theta \quad (60° \text{ dislocations}),\end{aligned} \tag{13}$$

If the threading dislocations have screw character instead, then there are eight distinct slip systems, all of which have $\psi = 45°$ and $\Delta = 54.7°$. Thus, for screw type threading dislocations, it is found using eq. (9) that

$$\begin{aligned}\beta_\epsilon^2 &= 0.090 b^2 D \,|\ln(2 \times 10^{-7}\ \mathrm{cm}\sqrt{D}\,|\tan^2\theta \\ &= K_\epsilon \tan^2\theta \quad (\text{screw dislocations}).\end{aligned} \tag{14}$$

This shows that the actual character of the threading dislocations will have only a weak effect on the strain broadening observed in the experimental rocking curve. Although expressions (13) and (14) were developed for the symmetric reflections such as (002), (004), and (006), they may be applied approximately to the reflections (113), (115), and (117), with less than a twenty percent error.

The broadening due to crystal size is usually only affected by the layer thickness in single crystal heteroepitaxial semiconductors. It is given approximately by [15]

$$\beta_L^2 = \left[4 \ln 2/(\pi h^2)\right](\lambda^2/\cos^2\theta), \tag{15}$$

where h is the thickness of the semiconductor layer. For example, for the (004) reflection from GaAs, β_L is 35″ for a 1.0 μm thick layer, 70″ for a 0.5 μm thick layer, and 350″ for a 0.1 μm thick layer. Thus, depending on the dislocation density, crystal size broadening can often be neglected for layers thicker than 1.0 μm.

Rocking curve broadening due to specimen curvature has been described by Halliwell, Lyons and Hill [16] and by Flanagan [17]. It is given by

$$\beta_r^2 = w^2/(r^2 \sin^2\theta) = K_r/\sin^2\theta, \tag{16}$$

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 heteroepitaxial layers are deposited on substrates which are one hundred times thicker, so that the curvature and its associated broadening are negligible. Because the curvature broadens the rocking curves from the epitaxial layer and substrate equally, it is possible to make an upper estimate of the effect of curvature by measuring the substrate rocking curve.

Combining the relationships above, eq. (1) can be rewritten as

$$\begin{aligned}\beta_m^2(hkl) &= \beta_0^2(hkl) + \beta_d^2(hkl) + K_\alpha + K_\epsilon \tan^2\theta \\ &\quad + \left[4 \ln 2/(\pi h^2)\right](\lambda^2/\cos^2\theta) \\ &\quad + K_r/\sin^2\theta.\end{aligned} \tag{17}$$

It is thus possible to measure three different reflections from a specimen at different values of θ and determine the three unknowns K_α, K_ϵ, and K_r, because the intrinsic rocking curve widths and the crystal size broadening are known. Then the dislocation density may be determined independently from K_α and from K_ϵ, providing a check for internal consistency.

In cases for which the broadening due to curvature and crystal size is negligible (i.e., cases for which $\beta_r^2 \ll \beta_m^2$ and $\beta_L^2 \ll \beta_m^2$) the analysis is considerably more simple. In such cases,

$$\beta_m^2(hkl) - \beta_0^2(hkl) - \beta_d^2(hkl) = \beta_{adj}^2 \approx K_\alpha + K_\epsilon \tan^2\theta, \tag{18}$$

where β_{adj} is the rocking curve width adjusted to account for the intrinsic rocking curve widths. If one measures rocking curve FWHMs for three or more rocking curves at different values of θ, and plots β_{adj}^2 versus $\tan^2\theta$, the result is a straight line with intercept K_α and slope K_ϵ. Then the dislocation density may be determined independently from the rotational broadening and from the strain broadening as follows:

angular broadening,

$$D = K_\alpha / 4.36 b^2; \tag{19}$$

strain broadening,

$$D = K_\epsilon / 0.090 b^2 \,|\ln(2 \times 10^{-7}\ \text{cm}\sqrt{D})|, \tag{20}$$

Eq. (19) is now well established and has been applied to a number of heteroepitaxial semiconductor systems [1,9,18–21] although sometimes without the correction due to Dunn and Koch [10]. On the other hand, there has been no application of eq. (20) to semiconductors, to the author's knowledge. The simultaneous application of both equations (19) and (20) may provide more reliable dislocation density data, however.

As an example, consider the case of GaAs on Si(001). Complete rocking curve data have already been obtained for this system by Ayers, Ghandhi and Schowalter [9,18]. These data are reproduced in table 1 for the case of a 3.0 μm thick layer on GaAs on Si(001) which was annealed for 30 min at 850°C. The rocking curve widths were measured by a Bede model 150 double crystal diffractometer ($\beta_0 = \beta_d$) with CuKα radiation (λ = 1.54 Å) and using the $(\theta + \phi)$ angle of incidence, where θ is the Bragg angle and ϕ is the angle between the surface of the crystal and the diffracting planes. Fig. 2 shows the adjusted values of β_{adj}^2 as a function of $\tan^2\theta$. It can be seen that the data fall on a straight line given by

$$\beta_{adj}^2 = 41{,}600(\text{arc sec})^2 + 5850(\text{arc sec})^2(\tan^2\theta). \tag{21}$$

Thus K_α = 41,600 (arc sec)^2 = 9.78×10^{-7} rad^2, and from eq. (19), the threading dislocation den-

Table 1
X-ray rocking curve data for a GaAs/Si(001) specimen, produced from the data of Ayers, Ghandhi and Schowalter [9,18]; the GaAs layer was 3.0 μm thick, and was annealed after growth for 30 min at 850°C; in the case of asymetric reflections, the measured and intrinsic rocking curve widths are all for the $(\theta + \phi)$ angle of incidence, where ϕ is the angle between the surface and the diffracting planes; β_m is the measured rocking curve FWHM, and β_0 is the intrinsic rocking curve width for the (hkl) reflection from (001) GaAs with $(\theta + \phi)$ incidence

(hkl)	θ deg	$\tan^2\theta$	β_m (arc sec)	β_0 (arc sec)
(002)	15.8	0.080	220	0.5
(113)	26.9	0.257	205	1.5
(004)	33.0	0.422	220	8.7
(224)	41.9	0.805	210	2.4
(115)	45.1	1.007	210	3.5
(044)	50.4	1.461	235	2.0
(006)	54.9	2.02	220	0.3
(026)	59.5	2.88	235	5.4
(444)	70.7	8.15	295	4.7
(117)	76.7	17.90	385	7.7

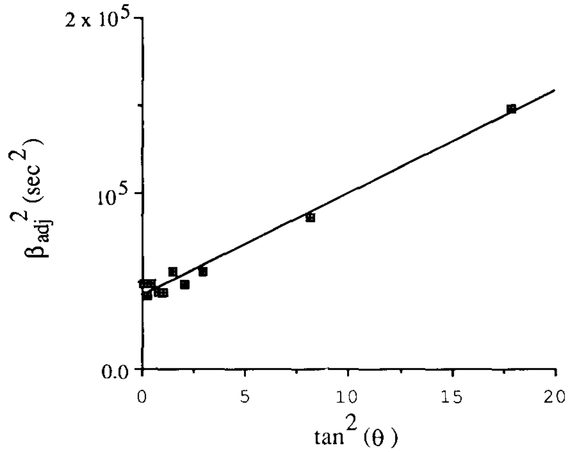

Fig. 2. β_{adj}^2 versus $\tan^2\theta$ for a 3.0 μm GaAs/Si(001) specimen, from the data of table 1.

sity can be calculated as 1.4×10^8 cm^{-2}. Using the second method, $K_\epsilon = 5850$ (arc sec)$^2 = 1.375 \times 10^{-7}$ rad^2, and from eq. (20), the threading dislocation density is calculated to be 1.5×10^8 cm^{-2}, which is in good agreement with the previous calculation. If 60° dislocations are assumed, then the threading dislocation density calculated from the strain broadening is reduced by a factor of two. However, differences of a factor of two or less between the K_ϵ and K_α calculations should be considered excellent agreement due to the approximate treatment of r_0 used in eqs. (10) through (14). Thus it does not appear possible to distinguish between screw and 60° dislocations from these measurements.

The good agreement of the two independent calculations is significant for the following reason. Sources of broadening other than dislocations tend to give rise to different relative amounts of rotational and strain broadening. For example, point defects contribute primarily strain broadening while curvature contributes mostly rotational broadening. Thus, agreement between the two calculations can be used to rule out significant broadening from such sources.

It should be pointed out also that the measurement of as few as three rocking curves allows application of these methods, as long as the reflections, are chosen with a sufficient range of $\tan^2\theta$. For most (001) semiconductor crystals, it is sufficient to measure only the (004), (115) and (117) rocking curves for accurate determination of the dislocation density. The (004) rocking curve width is determined primarily by the angular broadening of dislocations, and the (117) rocking curve width is determined primarily by the strain broadening of dislocations.

At this point it is worthwhile to comment on the starting assumption of Gaussian intensity profiles. The (004) rocking curves for the GaAs/Si samples are experimentally found to be Gaussian (for example, see fig. 1). Then, because the (004) profile is determined primarily by the angular broadening of dislocations, that distribution must be Gaussian. Also, the (117) rocking curves for GaAs/Si samples are experimentally found to be Gaussian (this was true for the experimental profiles obtained here). Then, because the (117) profile is determined primarily by the strain broadening of dislocations, the associated distribution must also be Gaussian. It is reasonable to expect similar behavior in heteroepitaxial systems other than GaAs/Si(001).

An additional complication of this analysis is that the dislocation density may vary with depth in the samples under study. This behavior is not well understood at present, but it is a generally accepted fact that the dislocation density decreases with distance from the interface in a single heteroepitaxial structure. In addition, the weighting of diffracted intensity from different parts of the crystal will be such that the measured rocking curve width will be indicative of the *lowest* dislocation density in the crystal. This is analogous to the case of the particle-size broadening, which also gives heavy weighting to the most perfect parts of the crystal (largest particles). Thus, the measured rocking curve width is expected to correspond to the surface dislocation density. This is fortunate, because it is the density of dislocations at the surface which is most important to the fabrication of electronic devices.

Finally, it should be noted that the strain broadening due to dislocations may be used via eq. (20) only for experimental cases in which X-ray wavelength dispersion is absent. These include the Bartels diffractometer [6,7] or the case of the double crystal diffractometer used in the (+, −) parallel setting [22]. When wavelength dispersion introduces broadening in the rocking curve, it is proportional to $\tan^2\theta$ [23] and can not be easily separated from the strain broadening of the dislocations.

3. Conclusion

In conclusion, the theory for the determination of dislocation densities in semiconductor crystals from rocking curve widths has been described. It has been shown that the measurement of several (hkl) reflections from a single crystal allows the determination of the dislocation density by two independent methods, thus allowing for a check of self-consistency. It was also shown for the case of heteroepitaxial GaAs/Si(001) that the results of these two methods are in good agreement. The simultaneous application of the two methods may allow more accurate dislocation density determination. In the general case of most (001) semiconductors, it is sufficient to measure three rocking curves such as the (004), (115) and (117) for accurate determination of the threading dislocation density. Similar methods can be applied to crystals of other orientations using the appropriate reflections. Application to thin or multiple layers requires consideration of dynamical diffraction effects but is also possible.

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