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X-ray Diffraction Investigation of Epitaxial Layers of CdTe on Sapphire

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Abstract

The anomalous scattering of X-rays has been used to determine the polarity of CdTe epitaxial layers on sapphire. The results for two samples are presented, one of (111) orientation ('A face'), the other of $(\bar{1}\,\bar{1}\,\bar{1})$ orientation ('B face'). The $(\bar{1}\,\bar{1}\,\bar{1})$ layer is twinned, the two twin species being related by a 180° rotation about the [111] axis. The twin fraction shows considerable variation for different positions on this sample, and must be taken into account when analysing the integrated X-ray intensities, in order to get meaningful Bijvoet ratios. The polarities of the two twin species are found to be the same.

1. Introduction

Stevenson *et al.* (1989) have recently discussed the use of anomalous scattering of X-rays for polarity determination of single-crystal epitaxial layers on substrates. The polarity of $Hg_{1-x}Cd_xTe$ (MCT), for example, has been shown to be important in regard to device performance (Tong *et al.* 1987). Thompson *et al.* (1986), for example, have discussed the quality, in terms of rocking-curve widths, of (111) and $(\bar{1}\,\bar{1}\,\bar{1})$ CdTe layers on sapphire, the former exhibiting better crystal quality. Several methods have been employed to determine polarity and some inconsistency and confusion exists in the literature (see e.g. Fewster and Whiffin 1983; Hewat *et al.* 1988).

In the present study two CdTe/sapphire samples are investigated by X-ray diffraction. One of the samples exhibits twinning, a phenomenon of considerable significance in regard to device performance. A number of workers have observed twinning in CdTe epitaxial layers (e.g. Horning and Staudenmann 1986; Hails *et al.* 1986).

In order to determine the polarity of a twinned epitaxial layer one must establish the degree and nature of the twinning with respect to the experimental configuration being used. Indeed, one cannot assume *ab initio* that the twins have the same polarity. We will discuss a method for determining the twin fraction α (the ratio of the volume of the smaller twin component to the total volume, i.e. $\alpha \leq 0 \cdot 5$) for different positions on the sample, appropriate to the nature of the twinning observed in this case. In interpreting the integrated X-ray intensities of Bragg reflections from a twinned crystal one must be aware that some measurements from, say, the larger twin component may be

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unaffected by the presence of the smaller twin component and some may represent a summation of intensities from both twins (e.g. Grainger 1969; Britton 1972). This will be shown to be the case here and this situation can be used to advantage by establishing α from non-overlapped reflections and then correcting the overlapped reflections (e.g. Sabelli *et al.* 1969).

2. Experimental

The measurements reported in this paper were made on a conventional four-circle X-ray diffractometer. The two samples investigated were both commercially grown CdTe on sapphire [2-inch wafer of (0001) orientation]. Sample A is nominally $6\cdot3\,\mu$m of CdTe of (111) orientation. Sample B is nominally $7\cdot9\,\mu$m of CdTe of $(\bar{1}\bar{1}\bar{1})$ orientation. The samples were mounted and aligned so that the surface was perpendicular to the diffractometer ϕ axis (to within $0\cdot05°$ using the laser technique of Moss and Barnea 1976).

The orientation (or UB) matrix for a given epitaxial layer was determined from a least-squares refinement of the peak positions for several Bragg reflections (taking care that these were associated, in the case of the twinned sample, with the larger twin component only). Integrated X-ray intensity measurements (and thereby Bijvoet-ratio measurements) were made with Cr Kα radiation, for reasons discussed at length by Stevenson *et al.* (1989). A V filter was used to suppress Cr Kβ radiation. The collimator used has a diameter of $0\cdot5$ mm, so that the surface and volume regions being irradiated by the incident X-ray beam are not too extensive.

The method of measuring integrated X-ray intensities is essentially that outlined in Stevenson *et al.* (1989). Each measurement is the result of averaging the (background-corrected) integrated intensities ($\omega/2\theta$ scan mode) for three equivalent reflections; for example, 311, 131 and 113 reflections are used to determine 'I_{311}' (Stevenson *et al.* 1989). The average discrepancy between equivalent-reflection intensities and the mean of the group to which they belong was $0\cdot67\%$ for sample A and $0\cdot23\%$ for sample B, indicating excellent internal consistency. Each of the equivalent-reflection integrated intensities is itself the average of four integrated intensities, measured at azimuthal positions $\psi = 0$, $0\cdot5°$, $180°$ and $180\cdot5°$. This combination of measurements allows us to, firstly, avoid the need to make corrections for the effects of absorption (see Stevenson *et al.* 1989) and, secondly, to make some check for the (unwanted) presence of multiple diffraction effects. [Multiple diffraction effects are less likely to present a problem for the relatively large X-ray wavelength being used here (see e.g. Prager 1971).]

It was found that, for different positions on a given epitaxial layer, the same UB matrix could be used. Each reflection was optimised (in terms of the diffractometer angles 2θ, ω and χ) before the integrated-intensity measurement was made. Hence, for the twinned sample, the 'larger twin' had the same UB matrix (and was indeed the dominant twin component) throughout the experiment.

3. Twinning

Recent studies of CdTe epitaxial layers have shown that one form of twinning which can occur in the {111} planes has the two twin species related by a 180°

rotation about the [111] axis (e.g. Oron *et al.* 1988; Brown *et al.* 1987). If one of the CdTe/sapphire samples is mounted and aligned on the diffractometer and the UB matrix is determined for the layer, one method for checking on the nature and degree of twinning is to carry out a ϕ scan, having set 2θ, ω and χ to the appropriate values for some suitable Bragg reflection; for example, 311 for a (111) layer. The 311 reflection is a suitable choice in that, for an untwinned crystal, we would expect to see three peaks of equal intensity at 120° intervals in ϕ. If the epitaxial layer is twinned in the manner mentioned above we would expect to see six peaks at 60° intervals. If one of these peaks occurs at $\phi = \phi'$, the peaks at ϕ', $\phi'+120°$ and $\phi'+240°$ should have equal intensity and be attributed to one twin component, and those at $\phi'+60°$, $\phi'+180°$ and $\phi'+300°$ should have equal intensity (but possibly different from the first group of three peaks) and be attributed to a second twin component. The selection of the (primary) Bragg reflection for such a ϕ scan is important in that, for example, a $31\bar{1}$ reflection would result in six peaks of equal intensity at 60° intervals in ϕ regardless of whether the sample is twinned or not, each peak being an overlap of two reflections for the twinned case.

In the event that the epitaxial layer is of $(\bar{1}\bar{1}\bar{1})$ orientation or that the two twin species of a twinned sample are of opposite polarities the above arguments still hold, for an appropriate choice of Bragg reflections. In the present work we initially indexed the Bragg reflections as though the epitaxial layer (or at least the larger twin component) was of (111) orientation.

The alignment of the samples on the diffractometer ensures that the surface normal coincides with the ϕ axis. If a 'miscut' exists [for example sample A has been found to be approximately 0·3° off the nominal (111) orientation] the [111] axis will not exactly coincide with the ϕ axis. In cases where the miscut is relatively small, as it is here, one can optimise the peak positions in the ϕ scan (in terms of 2θ, ω and χ) in order to get a better measure of the peak intensities. (It would also be possible to carry out the 'exact' scan, for a larger miscut for example, with appropriate modifications to the computer software used for diffractometer control.)

Fig. 1 shows ϕ scans, as described above and collected with Cr Kα radiation, for the 311 (primary) reflection of (*a*) sample A and (*b*) sample B. We see that sample A shows evidence of twinning, as described above. Both ϕ scans show small peaks at some positions $\phi = \phi'+n30°$, where n is an integer. These may be indicative of other twinning. However, as these peaks are approximately three orders of magnitude weaker than the main peaks, we will not consider them further. We therefore consider that, at the positions in question, sample A is twinned, the relationship between the twins being a 180° rotation about the [111] axis, and sample B is not twinned.

The ϕ-scan technique discussed here is closely related to that used by Oron *et al.* (1988) in their double-crystal X-ray study of CdTe layers. In using the four-circle diffractometer to carry out our ϕ scans we can alter the degree of asymmetry for the primary reflection including, as was used for Fig. 1, symmetric positions. This flexibility may enable one to probe the sample to different depths for a highly absorbing layer.

The ϕ-scan technique will be used in the present work, not only to establish the presence and nature of twinning, but to obtain a quantitative estimate of

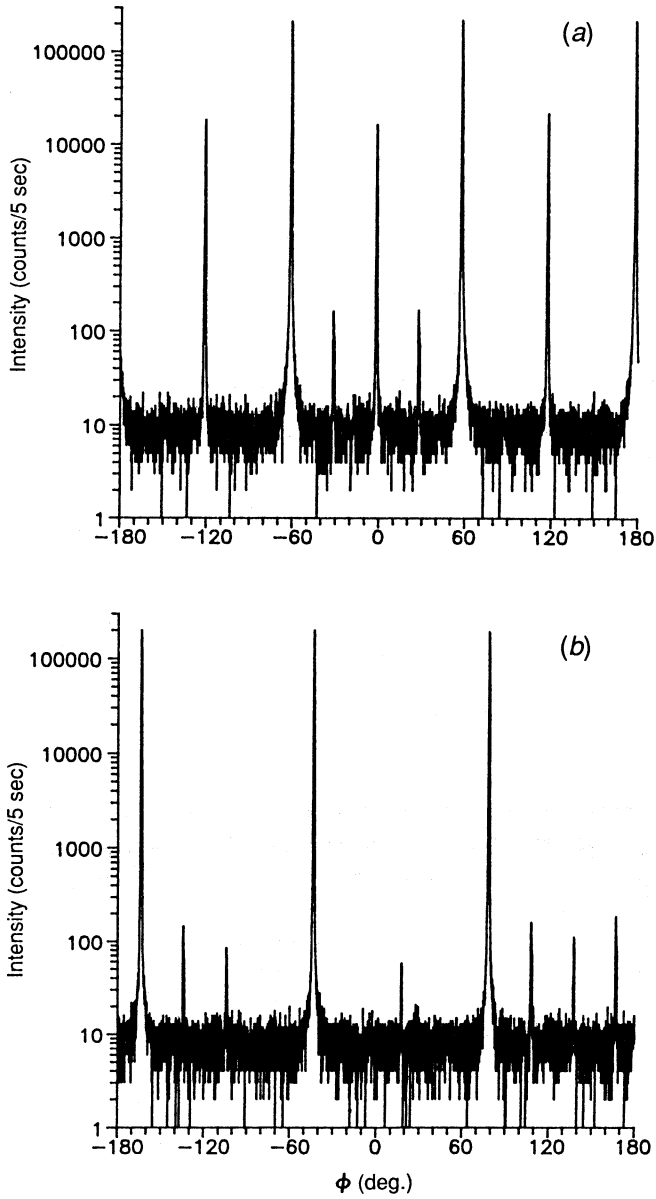

Fig. 1. The ϕ scans collected with Cr Kα X-radiation for the 311 (primary) reflection of (*a*) sample A and (*b*) sample B.

the twin fraction α. This is done using the peak intensities from ϕ scans like Fig. 1*a*. (The possibility of using integrated rather than peak intensities was investigated, but the results were virtually identical.) In determining α we use two ϕ scans, with 311 and 331 primary reflections. Since the volume of crystal irradiated by the incident X-ray beam is somewhat different for different peaks in a single ϕ scan, we get a measure of internal consistency in the α determination. Similarly, comparing the α determinations from two ϕ scans collected with different primary reflections provides a check on consistency (this point will be discussed further in the next section with regard to the relative polarities of the two twin species, i.e. whether they have the same polarity or not). In the present work we found this consistency to be excellent for the α determinations made at a given position on a sample. However, as we shall see, α can change dramatically for different positions on a particular sample.

Table 1. Measured twin fractions and Bijvoet ratios for seven positions on sample A

Position	Twin fraction	$B_{311/\bar{3}\bar{1}\bar{1}}$ (%)	$B_{331/\bar{3}\bar{3}\bar{1}}$ (%)	$B'_{311/\bar{3}\bar{1}\bar{1}}$ (%)
1	0·25 (0·02)	−3·1 (0·7)	−16·8 (1·2)	25·0 (2·7)
2	0·08 (0·01)	17·3 (0·6)	−16·2 (0·8)	25·4 (1·1)
3	0·27 (0·01)	−7·1 (0·5)	−16·9 (1·0)	23·8 (1·6)
4	0·16 (0·01)	9·7 (1·6)	−15·5 (1·5)	26·5 (2·3)
5	0·31 (0·02)	−14·1 (1·4)	−15·8 (1·7)	22·5 (3·0)
6	0·39 (0·01)	−25·6 (1·1)	−17·4 (0·6)	23·2 (1·8)
7	0·48 (0·02)	−43·0 (0·9)	−16·4 (3·0)	22·2 (5·5)
Average	0·28 (0·01)	−9·4 (0·4)	−16·4 (0·6)	24·1 (1·1)

4. Bijvoet Ratio Measurements

The use of Bijvoet-ratio measurements for determining the polarity of an epitaxial layer has been discussed at length by Stevenson *et al.* (1989). Table 1 presents the CdTe Bijvoet-ratio measurements for sample A, collected as previously described, at seven different positions on the wafer. The numbers given in parentheses are estimated standard deviations, based on counting statistics and population statistics (Stevenson *et al.* 1989). The second column gives the values of α, determined as described in the previous section (using 311 and 331 primary-reflection ϕ scans). The first point of note is that the values of α determined from the two primary reflections, for a given sample position, were always in excellent agreement (the average discrepancy being 1·5%, the maximum discrepancy being 3·0%). We are therefore certain that both twins have the same polarity, at least for the seven positions studied. In the event that the twins had opposite polarities these values of α, which would not represent the true twin fraction, would be quite different.

The third column of Table 1 shows a rather large range of values for $B_{311/\bar{3}\bar{1}\bar{1}}$, whereas the fourth column shows a very consistent set of $B_{331/\bar{3}\bar{3}\bar{1}}$ values. It is a straightforward matter to show that, given the 180° rotation about the [111] axis which relates the two twins, the Bragg reflections used to determine I_{311}, I_{331} and $I_{\bar{3}\bar{3}\bar{1}}$ are unaffected by twinning. The Bragg

reflections used to determine $I_{\bar{3}\bar{1}\bar{1}}$, however, are all affected by twinning. (The Bragg reflections used here were chosen so that the values of χ were not too far from 90° in order to avoid problems associated with glancing incidence.) Thus, there is no twinning correction to be made to $B_{331/\bar{3}\bar{3}\bar{1}}$ values, but such a correction is essential for $B_{311/\bar{3}\bar{1}\bar{1}}$ values. The last column of Table 1 lists the twin-corrected values (using the tabulated values of α), labelled $B'_{311/\bar{3}\bar{1}\bar{1}}$. We see that these values are now very consistent.

Table 2. Measured Bijvoet ratios for five positions on sample B

Position	$B_{311/\bar{3}\bar{1}\bar{1}}$ (%)	$B_{331/\bar{3}\bar{3}\bar{1}}$ (%)
1	−10·3 (0·6)	19·9 (0·6)
2	−10·2 (0·9)	20·3 (0·5)
3	−9·7 (0·5)	20·4 (0·6)
4	−9·5 (0·3)	20·0 (0·5)
5	−9·8 (0·4)	20·4 (0·7)
Average	−9·9 (0·3)	20·2 (0·3)

Table 2 presents the CdTe Bijvoet-ratio measurements for sample B, at five different positions on the wafer. The value of α for each position was 0·00, i.e. there is no twinning, at least for the five positions studied. The values of $B_{311/\bar{3}\bar{1}\bar{1}}$ in the second column are very consistent, as are the values of $B_{331/\bar{3}\bar{3}\bar{1}}$ in the third column.

The theoretical values of $B_{311/\bar{3}\bar{1}\bar{1}}$ and $B_{331/\bar{3}\bar{3}\bar{1}}$ are −16·9% and 18·4% respectively, calculated as described by Stevenson *et al.* (1989). A comparison of these values with those in Tables 1 and 2 leads us to the conclusion that sample A is of $(\bar{1}\bar{1}\bar{1})$ orientation (B face) and sample B is of (111) orientation (A face), i.e. the nominal orientations given by the manufacturer are incorrect. It is evident however that there is some remaining discrepancy between theory and experiment. This discrepancy is particularly intriguing in that the average values of $|B'_{311/\bar{3}\bar{1}\bar{1}}|$ for sample A and $|B_{311/\bar{3}\bar{1}\bar{1}}|$ for sample B [24·1(1·1)% and 9·9(0·3)% respectively], when averaged, give 17·0(0·6)%. This is in excellent agreement with theory (16·9%). Similarly, the average values of $|B_{331/\bar{3}\bar{3}\bar{1}}|$ for samples A and B [16·4(0·6)% and 20·2(0.3)% respectively], when averaged, give 18·3(0·3)%, compared with the theoretical value of 18·4%. Thus, the cause of the remaining discrepancy between theory and experiment has an equal and opposite effect on the Bijvoet-ratio magnitude for samples A and B. This point may not be too surprising given that the two samples come from the same manufacturer, have similar epitaxial-layer thicknesses and are of opposite polarities. Possible reasons for this discrepancy between theory and experiment include internal strain effects, bonding effects (e.g. non-spherical distortions of the atomic charge distribution) and anharmonic thermal vibrations (see e.g. Colella 1971; McIntyre and Barnea 1979). Careful investigations of relevant diffracted-beam dimensions, using both film and two-dimensional Bragg intensity distributions (Mathieson 1982; Mathieson and Stevenson 1986; McIntyre and Stevenson 1990), ruled out the possibility of the detector-aperture and scan-range truncations of the X-ray signal for any of the measured reflections being significant here. These investigations also included repeating some Bijvoet-ratio measurements with a 0·1 mm diameter

collimator, the results being quite consistent with those already presented. Further investigation of this discrepancy is beyond the scope of the present work.

5. Conclusions

The anomalous scattering of X-rays has proven to be a very useful tool for non-destructively determining the polarities of two CdTe epitaxial layers on sapphire. One of the samples has exhibited significant twinning, the twins being related by a 180° rotation about the [111] axis. The degree of this twinning has been determined quantitatively for several positions on the sample and this information used to correct measured Bijvoet ratios. The two twin species have been shown to have the same polarity.

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