

STRUCTURAL CHARACTERIZATION OF HgTe/CdTe SUPERLATTICES

B. M. PAINE*, T. VREELAND, JR.* AND J. T. CHEUNG**

* Division of Engineering and Applied Science, California Institute of Technology, Pasadena, CA 91125

** Rockwell International Science Center, Thousand Oaks, CA 91360

ABSTRACT

Superlattices of HgTe and CdTe, grown on (100) and (111) CdTe, have been characterized by He ion backscattering spectrometry and x-ray double crystal diffractometry. Backscattering spectrometry gave basic information about the depth-distribution of layers, i.e. the number of layers in the superlattice, the thickness of CdTe overlayer and the location of damage in the underlying CdTe. Symmetric and asymmetric rocking curves gave information about crystal quality and layer thicknesses. Average strains in the directions perpendicular and parallel to the layer interfaces were obtained directly from the rocking curves. Strains and thicknesses of the HgTe and CdTe layers, as well as variations from layer to layer, were found by fitting the symmetric rocking curves with theoretical calculations based on a kinematic model for the diffraction. A tilt of the atomic planes in the epitaxial layers, relative to those in the substrate, was observed by means of rocking curves recorded from the same diffracting planes, but with reversed incident and diffracted beam directions.

INTRODUCTION

Superlattice structures of HgTe and CdTe are currently of considerable interest for detection of infra-red radiation [1], due to the prediction of more desirable properties than are found for alloyed materials [2,3]. Since the substrates used for growth of such films, as well as the films themselves, are frequently of uncertain crystal quality, it is desirable to have a sensitive technique for their non-destructive physical characterization. In particular, one would like to have a measure of strains and dislocations in substrates, "buffer" layers, and superlattice layers. Layer thicknesses are also important because of their strong effect on device performance.

One of the most powerful tools for monitoring the dimensions and crystalline structure of thin films is x-ray double crystal diffractometry (DCD). This technique is reviewed briefly elsewhere in these proceedings [4]. We have applied DCD to the HgTe/CdTe system, and in this paper we report results for superlattices grown by laser molecular beam epitaxy (Laser MBE) on (100) and (111) CdTe substrates. In particular, we have recorded symmetric and asymmetric rocking curves, from which we deduced average parameters of the superlattice. We have also conducted detailed fitting of the data with a theoretical model for the diffraction process, from which we deduced the separate strains and thicknesses of the HgTe and CdTe layers. In addition, we report on complementary analyses by the well-known technique of backscattering spectrometry (BSS).

SAMPLE PREPARATION AND ANALYSIS

The laser MBE deposition was made in vacuum, from three sources: a thermal effusion Hg source, which was left on throughout the deposition, and CdTe and Te sources consisting of pieces of these materials in Ta boats, which were exposed alternately to a rastered laser beam. Laser evaporation of CdTe leads to deposition of CdTe only; evaporation of Te leads to combination with Hg atoms and deposition of HgTe. Details of the technique, as

well as a discussion of the mechanism of the deposition are given elsewhere [5].

Backscattering spectrometry was conducted in vacuum with 2 MeV He ions. The particle detector was mounted at a scattering angle of 170° , in a vertical plane. For optimal depth resolution, the sample was tilted about a vertical axis such that it was 45° from the incident beam. Channeling experiments were also conducted with the incident ions channelled along the axis lying most nearly normal to the sample surface. "Random" orientations were obtained by tilting the sample 7° away from the channeling direction and rotating continuously about the sample normal.

The x-ray double crystal diffraction measurements were conducted in air, with collimated $\text{FeK}\alpha_1$ radiation, selected by means of a (400) diffraction in a (100) GaAs first crystal. Since this is a dispersive combination for CdTe, special care was taken to remove $\text{K}\alpha_2$ radiation, with slits mounted between the first crystal and the sample. The incident x-ray beam, detector, and normal to the mounted sample, all lie in the horizontal plane. The measurement is made by rotating the sample about a vertical axis close to a Bragg condition (within 2°), and measuring diffracted intensity as a function of the rotation angle θ . This is executed with a step-scan apparatus under microprocessor control. Before each scan, the desired Bragg diffraction was brought into the horizontal plane by rotating the sample about its surface normal (angle ϕ), under manual control. Orientation of the crystal axes of a sample, relative to its surface normal, was measured first roughly by Laue back-diffraction, and then accurately by finding the ϕ angle for minimum incident grazing angle of the beam with respect to the surface, at the Bragg peak. The diffracted intensity is divided by incident intensity and plotted against $\Delta\theta$, the deviation from θ_B , to give the so-called "rocking curve".

We now describe the technique for measuring average strains and tilt angles in an epitaxial layer relative to the substrate. The general situation for a DCD measurement is illustrated in Fig. 1. The diffracting planes in the substrate have an inclination angle ψ relative to the surface; the chosen Bragg angle for them is θ_B . The same planes in the epitaxial layer are mis-oriented from those in the substrate, with a projection in the plane of the diffraction experiment of ξ_ϕ . We will use the term "strain" in a general sense, to mean deviations in the lattice parameter of the epitaxial layer from that of the substrate, independent of their unstressed values.

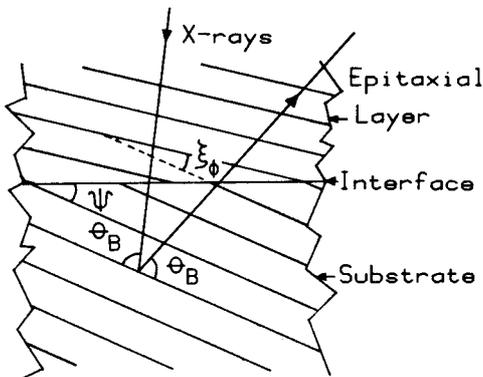


Fig. 1. Schematic diagram showing the atomic planes for a particular Bragg reflection near the interface between a single-crystal substrate, and an epitaxial layer.

We assume the epitaxial layer is much thinner than the substrate, so we can neglect strains in the latter. For cubic crystals with the interface parallel to the {100} plane, the strain tensor is expected to have rotational symmetry about the normal to the interface, because of the 4-fold crystal symmetry. We therefore assume it is fully defined by the strains perpendicular and parallel to the interface, ϵ^\perp and ϵ^\parallel respectively. This may be a good approximation in most cases if the loss of coherency is small, since the interface planes usually deviate from the major crystal planes by less than 3°.

The rocking curve from a sample containing a layer that is uniformly or periodically strained usually shows a well-defined "zero-order" peak. The angular separation of this peak from the substrate peak depends on the average strains $\langle\epsilon^\perp\rangle$ and $\langle\epsilon^\parallel\rangle$ [6]:

$$\Delta\theta_0 = -k_1\langle\epsilon^\perp\rangle - k_2\langle\epsilon^\parallel\rangle \pm \xi_\phi \quad (1)$$

The constants k_1 and k_2 each have two components, the first describing changes in the diffraction angle due to strain perpendicular to the diffracting planes, and the second describing rotation of the lattice planes caused by perpendicular and parallel strain:

$$k_1 = \cos^2\psi \tan\theta_B \pm \sin\psi \cos\psi \quad (2)$$

$$k_2 = \sin^2\psi \tan\theta_B \mp \sin\psi \cos\psi \quad (3)$$

The lower signs in Eqs. 1-3 apply for the x-ray beam directions of Fig. 1; the upper signs apply for the beam directions reversed, i.e. the sample rotated through 180°. Let $\Delta\theta_0^a$ and $\Delta\theta_0^b$ be the values measured from rocking curves for these two conditions, respectively. Then

$$(\Delta\theta_0^a + \Delta\theta_0^b)/2 = -\tan\theta_B(\cos^2\psi\langle\epsilon^\perp\rangle + \sin^2\psi\langle\epsilon^\parallel\rangle) \quad (4)$$

$$(\Delta\theta_0^a - \Delta\theta_0^b)/2 = \sin\psi \cos\psi(\langle\epsilon^\perp\rangle - \langle\epsilon^\parallel\rangle) - \xi_\phi \quad (5)$$

The procedure for extracting $\langle\epsilon^\perp\rangle$, $\langle\epsilon^\parallel\rangle$ and ξ_ϕ is as follows. θ_B is calculated from the known lattice parameter of the substrate; ψ is calculated from the known crystal orientation. A nearly-symmetric reflection is chosen, so the $\langle\epsilon^\parallel\rangle$ in Eq. 4 can be neglected. $\Delta\theta_0^a$ and $\Delta\theta_0^b$ are measured and $\langle\epsilon^\perp\rangle$ calculated from Eq. 4. Then an asymmetric reflection is chosen and $\langle\epsilon^\parallel\rangle$ found from Eq. 4. Then the values of ξ_ϕ can be found from Eq. 5. Similar methods have been used by Nagai [7] and Neumann et al. [8].

In addition to this analysis, we have fitted the rocking curves with theoretical calculations based on the kinematic model developed by Speriosu [4,9].

RESULTS

Fig. 2 shows a representative backscattering spectrum, recorded from a (111) sample, with a non-channelled orientation. In this figure, the upper inset shows the nominal structure of the sample, while the lower one shows schematically the component signals in the spectrum, ignoring the oscillations arising from the layer periodicity. The oscillations in the Hg signal dominate, because of the higher atomic number of Hg. The data suggest that 14 periods are present: 12 are clear, while the deepest two are not resolved but may be inferred from the doubly-broad signal. The shift of the Hg edge from that expected for exposed material confirmed the thickness of the surface CdTe layer. The channeling yield of this sample was 50% of the random yield.

Channeling measurements on a sample consisting of only the buffer layer, on top of bulk CdTe, revealed the presence of about 1000 Å of highly

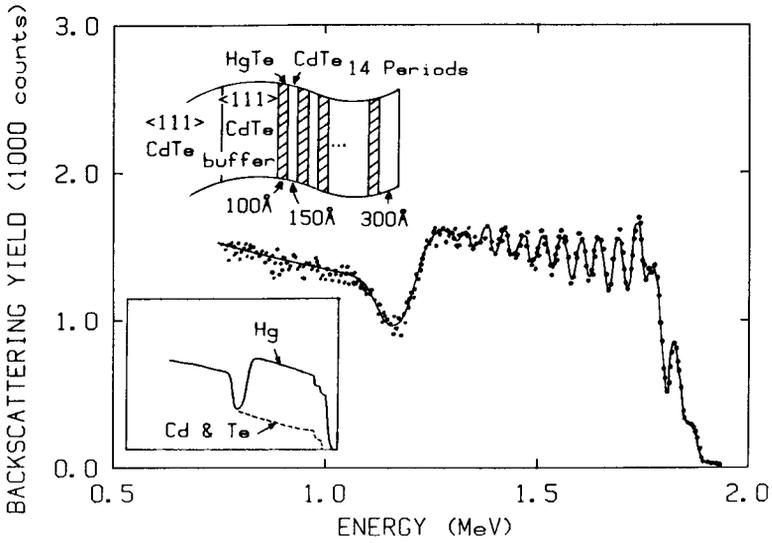


Fig. 2. Backscattering spectrum for the HgTe/CdTe superlattice sample shown in the upper inset. The lower inset shows the components of the signals in the spectrum, ignoring oscillations arising from the sample periodicity.

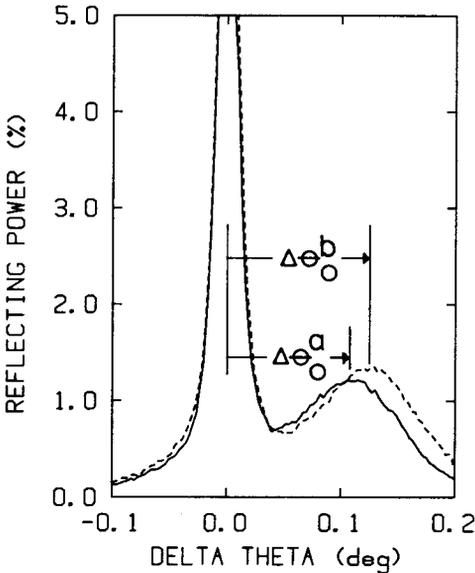


Fig. 3. Sections of the (400) rocking curves recorded for a (100) sample, with two orientations about the sample normal, 180° apart.

defective material lying close to their interface, probably arising from damage or impurities on the surface of the starting material. This was confirmed by DCD, which sensed a region of very high strain (up to 1%). Similar structure was observed by DCD with several other samples, but with smaller magnitude, such that it would not have been observable by BSS.

X-ray DCD studies were made on both (111) and (100) samples, but since the (100) samples proved to have superior electrical performance [5], we will concentrate here on the latter orientation. Fig. 3 shows the substrate and zero-order peaks from (400) rocking curves measured from such a sample, at orientations of ϕ and $\phi + 180^\circ$. $\Delta\theta_0^a$ and $\Delta\theta_0^b$ were measured between the centroids of the two peaks in each curve; their average gave $\langle\epsilon^\perp\rangle = -(0.27 + 0.02)\%$. Two asymmetric reflections were used to find $\langle\epsilon^\parallel\rangle$: the (440), which yielded a value of $-(0.17 \pm 0.04)\%$ and the (422), which yielded a value of $-(0.10 + 0.05)\%$. Repeated measurements reproduced these results closely. The fact that parallel strain is non-zero indicates that there is some de-coupling of the superlattice from the substrate, driven by the stresses in the HgTe layers. This is probably the result of the formation of misfit dislocations.

The measurable difference between $\Delta\theta_0^a$ and $\Delta\theta_0^b$ in Fig. 3 shows that there is a significant mis-orientation angle ϵ_ϕ . Since the $\langle 100 \rangle$ direction in the sample was found to be mis-oriented from normal by only 2.7° , the $(\langle\epsilon^\perp\rangle - \langle\epsilon^\parallel\rangle)$ term in Eq. 5 for a (400) reflection introduces only a relatively small correction. From two measurements of ϵ_ϕ at near right angles, the mis-orientation angle was found to be (40 ± 15) arcsec.

The full measured (400) rocking curve for this sample is shown as the dashed curves in Fig. 4(a) and (b). Three satellite peaks are evident, in addition to the zero-order peak. Their spacing indicates that the period of the superlattice is $(163 \pm 10)\text{\AA}$. Theoretical calculations of rocking curves require a knowledge of the parallel strain in the superlattice layers. The values deduced from the zero-order peaks in the symmetric and asymmetric reflections are consistent within the combined uncertainties. However the discrepancy in the principal values suggests that there may be some small variation of parallel strain in the superlattice or substrate with depth: because of the substantial absorption of x-rays in the epitaxial layers, the different diffraction conditions sample different depths and hence yield different average strains. The variation must be small, however, to account for the strong structure observed in the rocking curves. Also, its small magnitude means that it does not have a strong effect on the observed rocking curves. Therefore the best approach for modelling the structure is to assume small uniform ϵ^\parallel . At first a perfectly periodic structure of HgTe and CdTe, with abrupt interfaces, was assumed. Guesses were made for ϵ^\parallel , which given $\langle\epsilon^\perp\rangle$, the period of the superlattice, and lattice and elasticity constants for CdTe and HgTe, completely define the individual layer thicknesses and perpendicular strains. The best fit, which was obtained with $\epsilon^\parallel = -0.28\%$, is shown as a solid line in Fig. 4(a). This magnitude of the parallel strain is larger than that found from the (440) and (422) reflections because the x-rays penetrate considerably deeper and therefore sense a wider variation of ϵ^\parallel . Thicknesses of the HgTe and CdTe layers were $(133 \pm 10)\text{\AA}$, and $(30 \pm 8)\text{\AA}$, while their perpendicular strains were $-(0.39 \pm 0.02)\%$ and $+(0.39 \pm 0.02)\%$ respectively. The fact that these strains have equal magnitudes is coincidental: it is not an equilibrium configuration for the superlattice because the different layer thicknesses imply different magnitudes for the net forces in the HgTe and CdTe layers.

The calculated curve of Fig. 4(a) fits the major structure well, but there are some discrepancies for the small (more than 10 times lower intensity) -2 and +1 peaks. The higher data at negative $\Delta\theta$ is caused by the positive strain in the underlying CdTe layer that was noted earlier. The strain profile obtained by fitting rocking curves from samples with only the CdTe epitaxial layer was introduced in the calculation for the superlattice. The result is good agreement with the data, evident at $\Delta\theta < 0$ in Fig. 4(b). Finally, a reasonable fit to the -2 peak is obtained by introducing

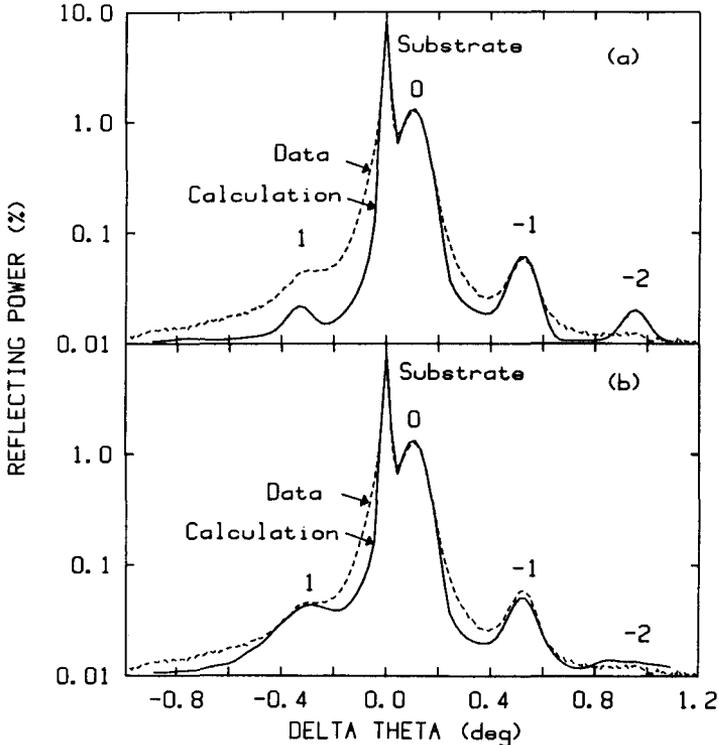


Fig. 4. Rocking curves for a HgTe/CdTe superlattice on (100) CdTe. The dashed curves are experimental data; the solid curves are calculated. In (a) the curve is calculated for a periodic structure with abrupt interfaces. In (b) the calculation includes a narrow strained region in the CdTe substrate, and 10% variations in the thicknesses of the HgTe and CdTe layers.

variations of the layer thicknesses with an average magnitude of 10%; these were also included in the calculation of Fig. 4(b). Alternatively, a similar fit could be obtained by introducing small interdiffusion of adjacent layers (of the order of 10% of their thicknesses), resulting in slight smoothing of the transition in strain from one layer to the next.

CONCLUSION

We have applied x-ray double crystal diffractometry, in conjunction with He ion backscattering spectrometry, to the challenging problem of characterizing HgTe/CdTe superlattice systems. DCD is very useful because it is non-destructive, highly sensitive to strain in both the superlattice and underlying layers, and gives accurate determinations of the thicknesses of the superlattice layers. BSS gives complementary information about the configuration of the sample and the thicknesses of other layers. The latter technique is destructive, but once the basic configuration of samples is learned from a small number of analyses, need not be applied further. Rather, double crystal diffractometry then becomes the tool of choice for routine high-precision characterization of samples as they are produced. Indeed the technique may also find important application in the future for checking wafers before deposition, and monitoring for possible changes in

the epitaxial layers due to processing steps such as formation of electrical contacts.

ACKNOWLEDGEMENTS

This work was supported financially by the Office of Naval Research (Contract No. N00014-83-C-0736).

REFERENCES

1. See for example the Proceedings of the 1985 U.S. Workshop on the Physics and Chemistry of Mercury Cadmium Telluride, to be published, J. Vac. Sci. Technol. A.
2. J. N. Schulman and T. C. McGill, Appl. Phys. Lett. 34, 663 (1979).
3. D. L. Smith, T. C. McGill and J. N. Schulman, Appl. Phys. Lett. 43, 180 (1983).
4. B. M. Paine, these proceedings.
5. J. T. Cheung, G. Niizawa, J. Moyle, N. P. Ong, B. M. Paine and T. Vreeland, Jr., in Ref. 1.
6. V. S. Speriosu and T. Vreeland, Jr., J. Appl. Phys. 56, 1591 (1984).
7. H. Nagai, J. Appl. Phys. 45, 3789 (1974).
8. D. A. Neumann, H. Zabel and H. Morkoc, Mat. Res. Soc. Symp. Proc. 37, 47 (1985).
9. V. S. Speriosu, J. Appl. Phys. 52, 6094 (1981).