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The detection of strain within InP-InGaAs single quantum well structures using large angle convergent beam electron diffraction

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ABSTRACT: The large angle convergent beam electron diffraction technique has been used to obtain rocking curves from MOVPE and MBE grown InP-$In_{.53}Ga_{.47}As$ single quantum well samples. Reflections sensitive to strain within the well layer were studied. From analysis of the curves an estimate of the mean strain present within the well in the samples has been made. Strains as low as $1.2 \cdot 10^{-3}$ have been measured. Plan-view specimens were used, reducing the problem of surface relaxation effects often encountered in thin cross-sectioned foils.

1. INTRODUCTION

The Bristol group has used the method of large angle convergent beam electron diffraction (LACBED) to study composition profiles in a number of single and multiple quantum well systems (Vincent et al (1987), Cherns et al (1988), Jordan et al (1989a) and Cherns et al (1991)). Here we extend the method to detect the low level of strain present in nominally lattice matched InP-InGaAs SQW (Single Quantum Well) samples. Structures grown using both atmospheric pressure MOVPE and gas source MBE were studied, (001) oriented substrates were used in all cases.

The LACBED method enables the selection of a single convergent beam disc. The maximum convergence angle is limited only by the electron optics (typically 6° in our case) (Vincent 1989). A single disc is selected using the selected area aperture, this aperture also acts as a transverse momentum filter and significantly reduces the level of thermal diffuse scattering within the pattern (Jordan et al 1991). The filtering enables the detection of detail within a LACBED disc at higher values of deviation parameter than are possible in comparable conventional CBED discs, or dark field (DF) images.

The technique differs from conventional CBED in that an image of the illuminated area (typically 2 to 3 μm in diameter) maps onto the diffraction disc, with spatial resolution usually limited by the probe size. For the information within a LACBED disc to be readily interpretable the specimen must be reasonably flat over the illuminated region of sample. This is easily achieved when preparing plan-view specimens of InP-InGaAs samples as excellent selective etches exist. Specimens with up to $2mm^2$ of flat electron transparent areas have been produced. Electron transparent areas comprise a tricrystal formed from a thin InGaAs layer sandwiched between InP layers of suitable thickness (500 to 3000Å). To allow the use of selective etches a thin InGaAs buffer layer must be grown to isolate the tricrystal from the substrate.

2. LACBED FROM InP-InGaAs SQWs

To successfully model all the features within a rocking curve from a multilayer structure dynamical diffraction theory must be used (Jordan et al 1989b, Rossouw et al 1991). However, at large values of deviation parameter, s, typically greater than 0.003Å^{-1}, the main features in the scattered amplitude in a rocking curve can be calculated using a kinematic approach. The amplitude A is given by:

$$A \propto \int_0^t F_{hkl}(z)\exp\{-2\pi i(sz+\mathbf{g}.\mathbf{R}(z))\}dz \qquad (1)$$

F_{hkl} is determined by the phased sum of the scattering from the two fcc sublattices, these add in phase for reflections such as 400 and 220 but in antiphase for 200. **R**(z) is a measure of the displacement within the crystal due to strain. In the structures studied here the layers are constrained to the same lattice parameter in the plane of the foil. Any lattice mismatch in the SQW results in a tetragonal distortion involving elongation or compression parallel to the growth direction.

In previous studies we concentrated on reflections with **g** in the plane of the foil. The term in **g.R** is then zero, and eqn. 1 can be considered as the Fourier transform of the structure factor. Analysis of rocking curves from compositionally sensitive reflections enables the measurement of mean well thickness to an accuracy of ±5%. For well layers greater than approximately 25Å the thickness may be obtained by simply measuring the value of deviation parameter, s_{min}, at which the modulation present in the rocking curve falls to a minimum (see, for example, Fig. 1, Jordan et al (1989)). The thickness, t, of the well layer is given by $t=s_{min}^{-1}$. In thin wells the position of modulation loss cannot be detected directly, as it occurs at high values of deviation parameter where the diffracted intensity is very low. Computer matching of the intensity profiles of the experimental and theoretical rocking curves must then be used (Jordan 1991).

Similar modelling can be performed on rocking curves formed in reflections with a component of **g** out of the plane of the foil. An estimate of the mean strain level within the well layer of the sample can then be made.

3. MEASUREMENT OF THE MEAN STRAINS WITHIN SQW GROWN USING MOVPE AND MBE.

3.1. 32Å well layer

Fig 1 shows a montage of 2 202 DF LACBED images obtained from a sample of 1550Å total thickness, with a well layer of nominally 32Å. The specimen was grown using MBE. Due to the low intensity of the LACBED discs only a limited range of s may be obtained (exposures of 2 to 3 minutes were used to capture the discs shown in fig. 1). The deviation parameter ranges from -0.025Å^{-1} at the left of the montage to $+0.025\text{Å}^{-1}$ at the right. A similar montage obtained from a sample with the same structure, but grown using MOVPE, is shown in fig. 2. All patterns were obtained with a specimen tilt, θ_t, of approximately 55°. The rocking curves show fringes with a spacing of s_f equal to t_{proj}^{-1}, where t_{proj} is the projected thickness of the sample (i.e. 1550Å/cos 55° in this case).

A strong modulation in the fringe intensity may also be seen. This effect is predominantly due to strain within the InGaAs well layer, the 202 reflection being relatively insensitive to variation in composition. An asymmetry in the modulation about s=0 may also be observed. The modulation decays to a minimum in the two areas of the pattern arrowed. As for the case of rocking curves formed in unstrained reflections the mean well thickness, t_{mw}, may be calculated from the spacing between consecutive losses of modulation, and is given by:

$$t_w = 2 \cdot (s_r - s_l)^{-1} \cdot \cos\theta_t \tag{2}$$

where s_r and s_l are the positions of the first modulation loss to the right and left respectively of the Bragg peak. The substitution into eqn. 2 of values obtained from fig. 1 give a well thickness of 35Å, in good agreement with that specified.

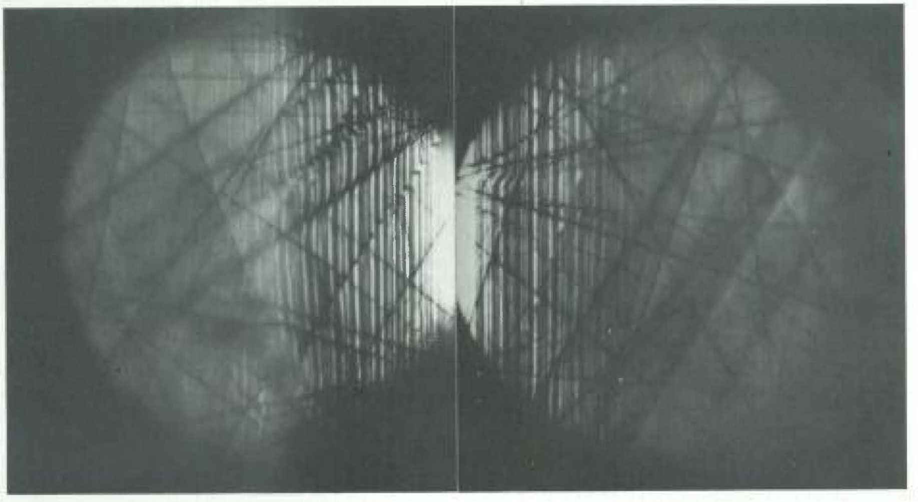

Fig. 1. A montage of 2 202 DF LACBED discs obtained from SQW samples containing a nominal 32Å well grown using MBE.

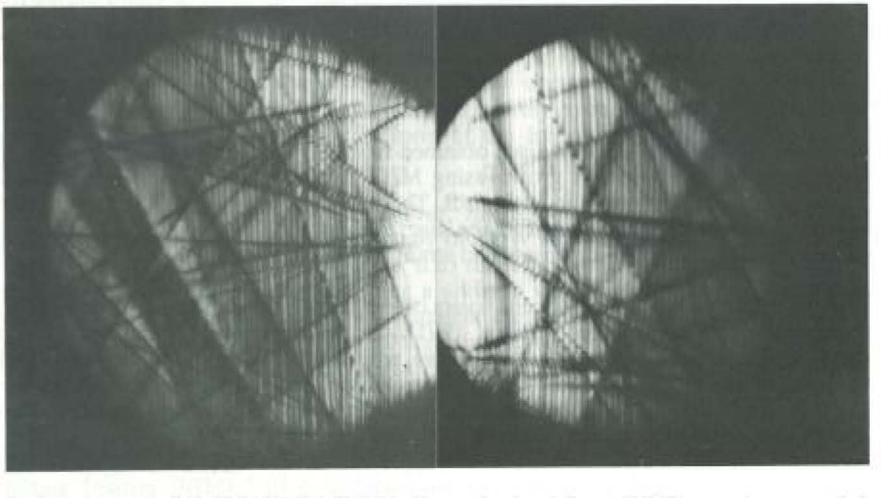

Fig. 2. A montage of 2 202 DF LACBED discs obtained from SQW samples containing a nominal 32Å well grown using MOVPE.

An estimate of the degree of strain within the well layer may be obtained from the level of asymmetry in the positions of s_r and s_l. To ensure consistency all values of deviation parameter must be measured relative to the Bragg position of the unstrained InP layers. This may be found by study of the position of high order deficiency lines, which cross the 202 LACBED disc.

A montage of 202 type rocking curves, calculated using eqn. 1, for a structure similar to that which generated figs. 1 and 2 is shown in fig. 3. The level of strain included in the calculation varies from 0 at the top to $1.0 \cdot 10^{-2}$ at the bottom, a 55° tilt angle was assumed. The curves best matching the patterns in fig. 1 and 2 are arrowed; they indicate a mean strain within the well layer of $2.4 \cdot 10^{-3}$ (MBE) and $1.2 \cdot 10^{-3}$ (MOVPE), with an accuracy of $\approx \pm 10\%$. (All strains are quoted relative to the relaxed cubic unit cell).

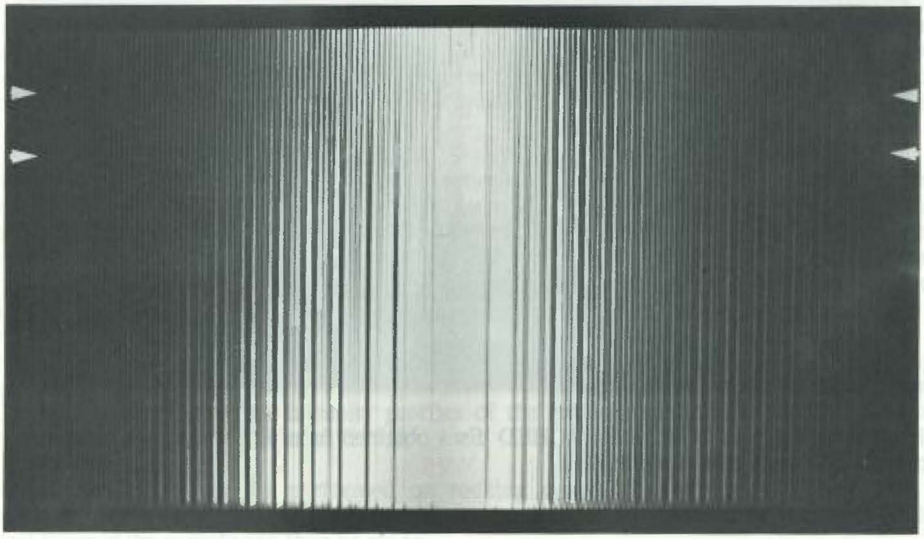

Fig. 3. A montage of simulated 202 type rocking curves, The level of strain varies from 0 (top) to $1.0 \cdot 10^{-2}$ (bottom). A 55° tilt angle was assumed.

3.2. 9Å well layer

Fig. 4 shows 2 202 DF LACBED discs obtained from SQW samples containing a nominal 9Å well layer. One sample was grown using MBE, the other using MOVPE. Modulation due to strain can be clearly seen in both discs. The well layer was too thin in the samples for a loss of modulation to be visible within the disc; therefore in this case it was not possible to obtain an estimate of the well thickness and strain without computer simulation.

Initially the thickness of the constituent layers of the sample are measured by comparison of rocking curves from 200 images (Jordan 1991). As the structure is then known the strain can be found by comparison of the 202 curve with theoretical rocking curves calculated with varying strain. At present these comparisons are performed by eye, limiting the level of accuracy possible, but in the future it is intended that the process will be computerised, using a routine similar to that developed to analyse 200 LACBED discs.

The mean strains present in fig. 4 were measured at $5 \cdot 10^{-3}$ (MBE grown) and $4 \cdot 10^{-3}$ (MOVPE grown). The accuracy of the measurements is at present only estimated at $\approx \pm 30\%$.

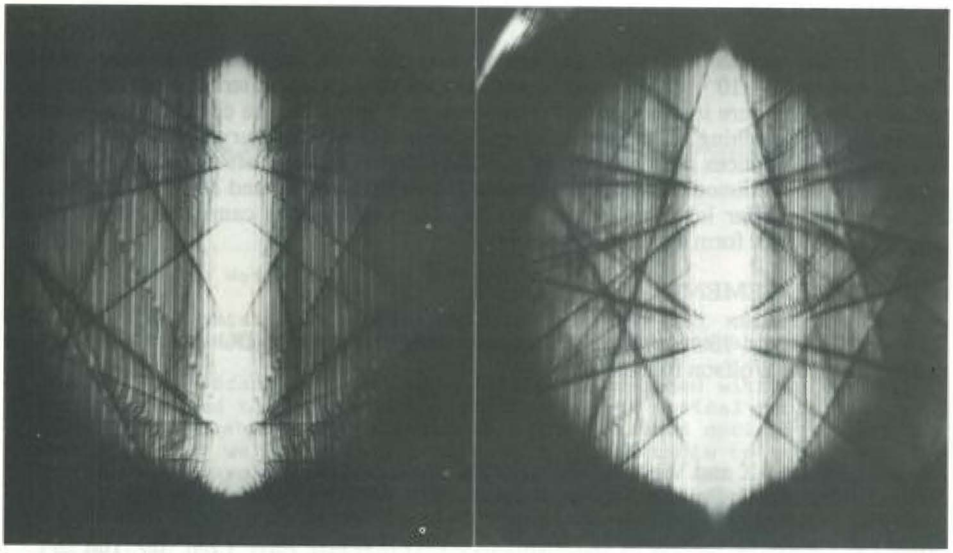

Fig. 4. 2 202 DF LACBED discs obtained from SQW samples grown using MBE (left) and MOVPE (right). The samples contained a nominal 9Å well layer.

4. DISCUSSION

The results indicate that small strains may be detected in SQW samples using this method. At present conventional x-ray double crystal diffractometry from SQW samples is typically limited to wells thicker than 100Å due to the low diffracted intensity levels involved. As the samples are viewed in plan-view the problem of further strain relaxation during specimen preparation does not arise.

In matching the experimental rocking curves with those predicted from kinematic theory it was assumed that the strain was equally distributed throughout the InGaAs well layer, with no strain present in the bulk InP. Clearly this is an over-simplification. Lyons (1989) has shown that thin layers of relatively high strain may occur due to gas flow switching at interfaces. Interfacial strain cannot be eliminated entirely even for ideal growth conditions as monolayers of InGaP and InAs will form at the InP to InGaAs and InGaAs to InP interfaces respectively.

Strain localisation at interfaces will, however, mainly effect the low frequency Fourier components of the rocking curve. In the study of SQW samples we are necessarily limited to analysis of the high order components (i.e. relatively low deviation parameter), It is therefore valid to use a model incorporating a uniform strain field to match theoretical and experimental rocking curves. Further experiments are to be performed to study this. It is also possible to determine the sign of the strain in the well layer from the direction of the asymmetry within rocking curve, but this has not been performed on the samples described here.

In the future it should be possible to extend the technique to the study of more highly strained systems. Analysis of rocking curves formed in out of plane reflections, incorporating strain effects, may also produce a way of measuring mean well thicknesses in the AlGaAs-GaAs system. Here study of in plane 200 type rocking curves has so far not proved satisfactory, due to the small structure factor fluctuation present.

5. CONCLUSIONS

The preliminary results presented here indicate that it is possible to detect an average level of strain as low as $4{\cdot}10^{-3}$ within a well layer only 9Å thick, using a structure of appropriate geometry. In the future it is hoped that the accuracy of the technique could be improved by automating the matching of experimental and theoretical rocking curves. The use of plan-view specimens reduces the problems of surface strain relaxation often encountered when preparing cross-sectioned TEM foils. Samples grown using MBE and MOVPE were found to incorporate similar levels of strain, but detailed comparisons cannot be made as the samples used do not form a systematic set.

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