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MULTIPLE DIFFRACTION SIMULATION IN THE STUDY OF EPITAXIAL LAYERS

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A practical application of the X-ray Multiple Diffraction (XRMD) simulation in the study of semiconductor epitaxial layers (GaAs/Si) is described. A computer program was developed to simulate multiple diffraction patterns in Renninger geometry which proved to be highly versatile. The intensity is calculated by using the iterative method which allows for many beam interactions being simultaneously considered. Applications are made for some examples; in very good agreement with the experiments. Renninger scans for the epitaxial layer (GaAs) showing only surface secondary reflections due to the thickness effect (1 μm) were simulated by taking into account an appropriate beam path length calculation.

1. INTRODUCTION

The multiple diffraction (MD) phenomenon involves the interaction among several diffracted beams within a single crystal when rotated around the scattering vector of a so-called primary reflection in a usual Renninger scan[1]. During the rotation the incident beam can be scattered by other secondary planes asymmetric with respect to the crystal surface . In the primary beam intensity, monitored during the experiment, one can observe deeps and/ or peaks caused by those above mentioned interactions.

The number of applications of the MD phenomenon in the study of materials has increased in the last few years[2] which justifies an effort of obtaining a computer program for the simulation of MD patterns. Recently, two simulation programs[3] have been published based on the kinematical theory. In both, the effect of higher-order diffraction (n-beam interaction, n>3) is treated as the sum over the secondary reflections of the (n-2) pairs of three-beam cases.

A computer program based on the kinematical theory and primarily developed to simulate neutron MD patterns (MULTI)[4] was implemented for the X-ray case (MULTX). The primary intensity is calculated using the iterative method[5]. This approach allows for many beam interactions be simultaneously considered. The program can simulate the transmitted incident or the primary beam scans, the latter case for a Laue or Bragg reflection which proved to be highly versatile. The agreement between the experimental and theoretical patterns is very good as it will be seen in some examples. Furthermore, it is also presented a first practical application of the program for a thin GaAs epitaxial layer grown on top of a Si substrate. The MD pattern for the layer was reproduced in a very good agreement by taking into account the correct diffracted beam path lengths.

2. CALCULATIONS

2.1. Geometrical conditions

The program MULTX selects all the possible secondary reflections by determining the angle ϕ between a reference direction and the projections of the secondary scattering vectors on the plane normal to the primary scattering vector.

2.2. Intensity

The general expression, which describes the change in power of a beam i owing to absorption and to simultaneous reflections by n other reciprocal lattice points as it traverses a layer of thickness dx at depth x below the surface of a crystal plate, is given by[6]

$$S_i(dP_i/dx) = -(P_i/\gamma_i)\mu + \sum_j \bar{Q}_{ij}(P_j/\gamma_j - P_i/\gamma_i) \quad (1)$$

where γ_i and γ_j are the direction cosines of beams i and j relative to the normal to the plate surface, μ is the linear absorption coefficient, $\bar{Q}_{ij}$ is the interaction coefficient called effective reflectivity of the plane $(i-j)$[7], S_i is the sign which indicates the nature of the beams: positive for reflected beams (Bragg) and negative for transmitted beams (Laue). Using eq.(1) for the different beams involved in the simultaneous diffraction process, a set of coupled differential equations can be obtained. Nevertheless, when n-beam interactions (n>3) are considered the exact solution becomes to complex. Alternatively, an approximate solution as a Taylor series expansion of the primary beam around x=0 can be used[7].

The conditions $Q_{ij}\ell_i << 1$ and $\mu\ell_i << 1$, where ℓ_i is the diffracted beam path length, are enough to produce a rapid convergence of the series. However, if they are not fully satisfied it is necessary to consider several terms in the Taylor series expansion. Fortunately, the general term has already been developed which allows for the iterative intensity calculation, i.e., the n^{th} term of the series is calculated from the term $(n-1)$[5]. The n^{th} term is given by

$$P_i^{(n)}(0)x^n = \sum_k P_k(0)X_{ki}^{(n)}$$

where the coefficients $X_{ki}^{(n)}$ are taken from the recurrence formula

$$X_{ki}^{(n)} = \sum_j X_{kj}X_{ji}^{(n-1)}$$

where

$$X_{kj} = S_j\bar{Q}_{kj}x_k \qquad \text{for } k \neq j$$

$$X_{jj} = S_jA_jx_j \qquad \text{for } k = j.$$

A_j and x_k are given by:

$$A_j = \mu + \sum_{k\neq j} \bar{Q}_{jk}$$

$$x_k = x/\gamma_k$$

The x_k's are replaced by the diffracted beam path lengths which should be calculated taking into account the specific crystal shape.

2.3. Possible simulations

Figure 1 shows four distinct cases where MD patterns can be simulated by the program MULTX. They correspond to the monitoring of: a) an usual primary reflected beam, b) a primary transmitted beam, c) an incident beam when the primary is reflected and d) an incident beam when the primary beam is transmitted. In the cases a) and b) the patterns can have only deeps ('Aufhellung' type), if the primary reflection is strong, only peaks ('Umweg' type), if it is forbidden, and deeps and peaks (mixed type), if it is weak. In the cases c) and d) the patterns are only of the 'Aufhellung' type.

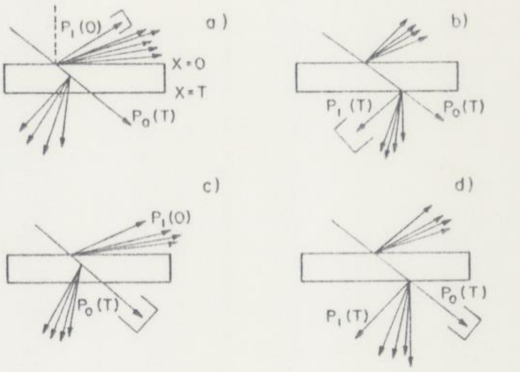

FIGURE 1
The four different simulation possibilities offered by the MULTX program where monitoring: a primary beam which is a) Bragg or b) Laue and an incident beam when the primary beam is c) Bragg or d) Laue.

2.4. Diffracted beam path lenghts

The diffracted beam path lenghts have been calculated for the normal cases in neutron or X-ray MD. In the neutron case of low secondary extinction and low absorption, for a large crystal plates of thickness T, the path length of a beam i is equal to[7]

$$\ell_i = T/\gamma_i$$

On the other hand in the X-ray case, for a highly absorbing crystal plate and a symmetric primary reflection,

$$\ell_i = 1/2\mu$$

which can be applied to all reflections[6]. In the X-ray MD study of thin epitaxial layers where the layer thickness should play an important role in the intensity calculations, this last expression is no longer applicable.

The secondary reflections scattered in extreme conditions of asymmetry, i.e. those diffracted beams parallel to the crystal surface (surface secondary reflections), will be very important. For this case, the plate dimensions play an important role in the interaction between the secondary and the primary beam. Besides, surfaces beams carry information about the crystal surface. Thus, the diffracted beam path lengths were calculated in an alternative way: in a finite crystal plate, the path length of a diffracted beam is averaged in a parallelogram normal to the crystal surface an parallel to the beam. Due to the dimensions of the plate and thickness of the layer, such parallelogram is several millimeters long and a few microns height. This implies that the path length of any surface beam is, at least, an order of magnitude bigger than the path length of an inclined beam. Thus, the contribution of a surface beam to the MD phenomenon is very important in an epitaxial layer.

2.5. Polarization factor

It has been used a general expression[6] for the polarization factor which takes into account the used or not of a monochromator. In the iterative method, the only way to include this factor in the calculations is to include it in the reflectivies[2]. This is the procedure adapted in this work.

3. EXPERIMENTAL

The MD experimental patterns were measured with a divergent X-ray beam (Microflex -Rigaku Denki) from a Cu target, with an effective focal size of about 50 μm x 50 μm. An angular divergence of about 3' was provided by a collimator (330 mm long) placed before the goniometer.

The samples had a retangular shape of about 10 mm long and a few mm wide. The thicknesses of the samples were 350 μm (GaAs), 1 μm (GaAs layer on top of Si) and 350 μm for the Si substrate. The GaAs/Si sample was grown by Vacuum Chemical Epitaxy (VCE). The GaAs layer was grown at 650°C after heating the substrate up to 900°C. Details of the sample preparation has been published elsewhere[8]. The other sample, i.e. the GaAs and Si, are pieces of commercial available waffers.

4. RESULTS AND DISCUSSION

The experimental MD patterns are shown in the upper part of the figures 2, 3 and 4 and some peaks are indexed. The corresponding simulations are in the lower parts for comparison purposes. The intensities in each simulation are normalized to the largest experimental ones. The three cases below are examples of the simulation of XRMD patterns by MULTX. The first two are general case whereas the last one is, as far the authors know, the first simulation of a XRMD pattern produced by a thin epitaxial layer.

4.1. GaAs

Figure 2.a shows the experimental ϕ scanning of the 002 primary reflection of GaAs. The simulation of this pattern, which is shown in figure 2.b, was calculated with a step $\Delta\phi = 0{,}045^{\circ}$, isotropic thermal parameters $B_{Ga} = B_{As} = 0.6$ Å^2 and mosaic spread $\eta = 0.05^{\circ}$ (9.10^{-4} rd). Although 30 terms in the expansion have been considered, the convergence was reached after just 6 terms. All diffracted beam path lengths were calculated as $\ell = 1/2\mu$.

The secondary contributions are positive since the intensity of the 002 reflection of GaAs is extremely weak, although not zero. The agreement is excellent and it can be observed that each peak in the simulation can be seen in the experimental pattern. Therefore, this agreement supports the validity of the treatment as well as the assumptions taken in this work. Concerning the positions of the several peaks in both figures, they also are in good agreement.

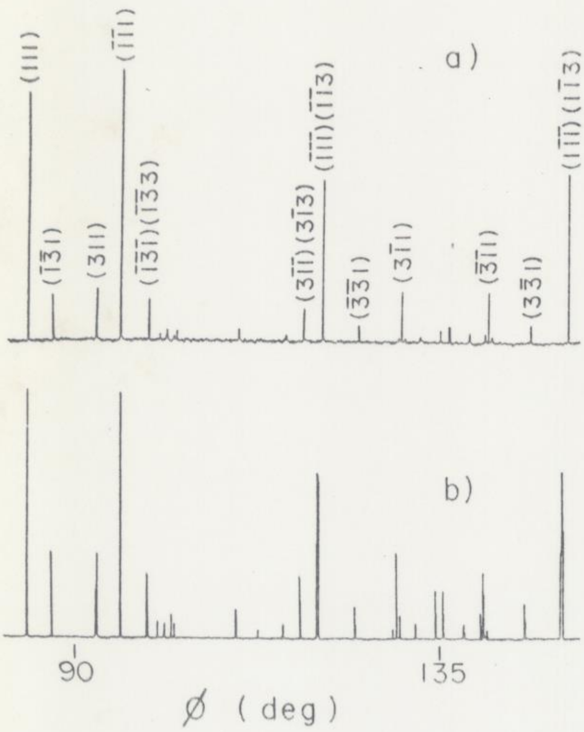

FIGURE 2
XRMD patterns for 002 of GaAs with Cuk_α: a) Experimental and b) Simulated.

4.2. Si

Figure 3.a shows the experimental MD pattern of the 002 primary reflection of Si. In figure 3.b the simulation was calculated with $\Delta\phi = 0.045^{\circ}$, $B_{Si} = 0.45$ Å^2 and $\eta = 0.05^{\circ}$ (9.10^{-4} rd). The comparison between the patterns shows again an excellent agreement.

4.3. GaAs/Si

Figure 4.a shows the experimental result of a XRMD pattern of the 002 reflection of bulk GaAs (350 μm). Figure 4.b shows the experimental XRMD pattern of the same reflection of a GaAs epitaxial layer grown on top of a Si substrate. It should be observed that only reflections with indice $\ell = 1$ are clearly seen. For a primary 002 reflection these are surface secondary reflections[2]. Then, the other secondary contributions almost disappeared. This effect can be explained based on the diffracted beam path lengths within the layer (1 μm) which is rather thin to allow for other reflections be present, as explained before. The two extra peaks around 111 and $\bar{1}\bar{1}1$ reflections are the subject of another work[9] and can be interpreted as contributions from the substrate surface reflections scattered by layer planes toward the detector window. In figure 4.c the simulation of the layer pattern is shown. The calculation is made for $\Delta\phi = 0.05^{\circ}$, $B_{Ga} = B_{As} = 0.6$ Å^2, $\eta = 0.068^{\circ}$ ($1.2.10^{-3}$ rd). As before, only 6 terms were enough for the convergence of the Taylor series. The diffracted beam path lengths were calculated as stated in sub-section 2.4. The peak broadening in figure 4.b is due to the large mismatch between the involved lattices.

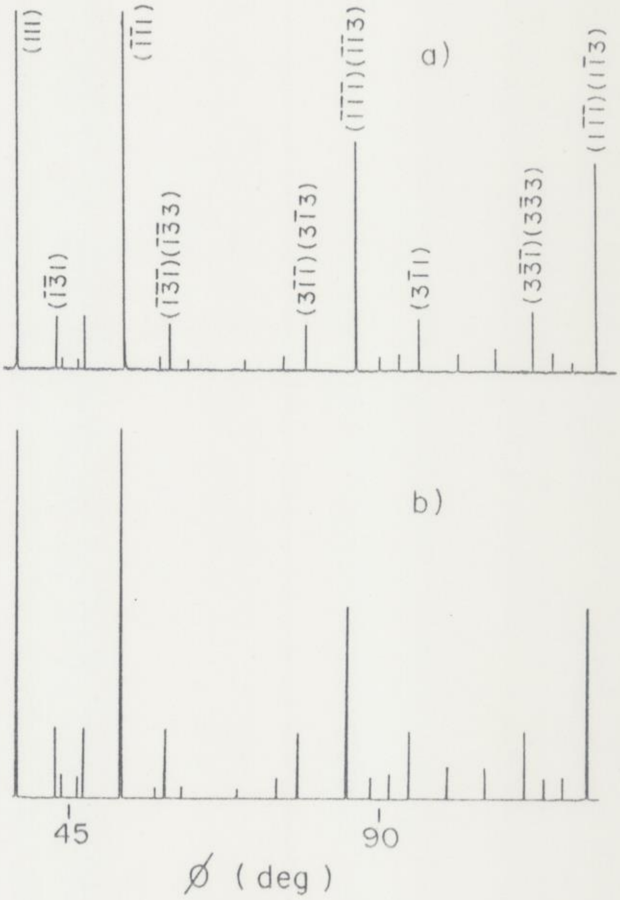

FIGURE 3
XRMD patterns for 002 of Si with Cuk_α: a) Experimental and b) Simulated.

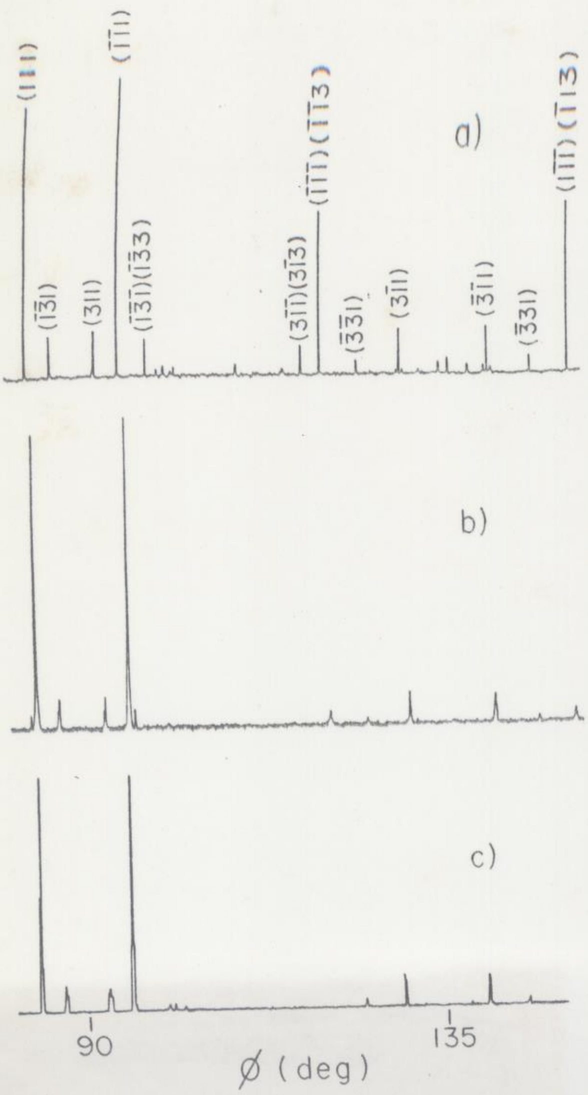

FIGURE 4

XRMD patterns with Cuk$_\alpha$ for 002 of: a) Experimental GaAs bulk, b) Experimental GaAs layer on top of Si and c) Simulated pattern corresponding to b).

It should be pointed out that the agreement between simulated and experimental MD patterns verified in figures 2, 3 and 4 shows the validity of the kinematical theory when applied to the calculation of MD intensities.

In this aplication of MULTX to the study of epitaxial layers it is emphasized that only surface secondary reflections are clearly observed in the layer MD pattern owing to the thin layer thickness. In addition, several different values for the layer thickness were tested and we conclude that increasing this thickness up to 10 μm the layer MD patterns just show surface secondary reflections confirming the striking surface effect. When this limit is overcome other than surface secondary reflections start to be visible. A normal GaAs bulk pattern is then reproduced. No further details are given here, concerning this thickness effect, since they will be subject of a future work.

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