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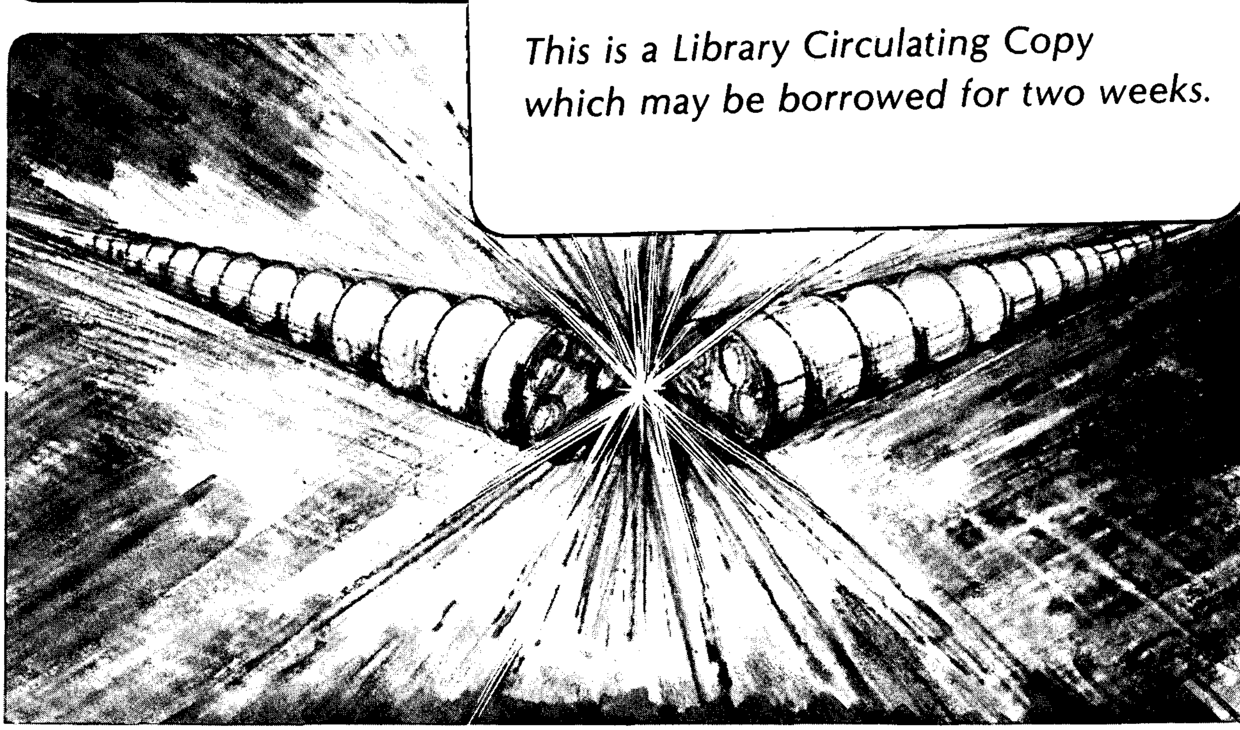

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# GRAZING INCIDENCE X-RAY SCATTERING STUDY OF DEFECT STRUCTURES IN MBE GaAs/Si USING SYNCHROTRON RADIATION

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## ABSTRACT

Defect structures in thin, MBE grown, GaAs/Si(001) heteroepitaxial films were studied with grazing incidence x-ray scattering techniques. The diffuse scattering associated with GaAs having the predominant epitaxial orientation varied strongly with the penetration depth of the x-rays. Well directed streaks corresponding to planar faults were observed and easily distinguished from a broad and more randomly oriented background. Bragg peaks corresponding to twinned GaAs were also studied. The probability of twinning was seen to be much higher about the {111} planes whose normals, when projected onto the sample surface, lay parallel to the off-orientation direction than about the {111} planes whose normals, when projected onto the sample surface, lay perpendicular to the off-orientation direction. The intensity and breadth of the twin reflections were analyzed to estimate the corresponding defect density. The depth dependence of the twinning probability was seen to vary with growth parameters.

## INTRODUCTION

Growth procedures used to produce high quality GaAs films on Si substrates are significantly more complex than those typically used in homoepitaxial growths or in the growth of heteroepitaxial films of small lattice mismatch. Empirically optimized growth procedures for the production of device quality GaAs films on Si substrates currently employ at least a two-step growth process to achieve acceptably smooth surface morphologies without unduly sacrificing crystal quality. Recently, observation of a marked reduction in structural defect density with in-situ or post growth high temperature annealing suggests that this additional step may soon become standard [1,2]. The reduction of crystalline defect densities after high temperature anneals proves that the as-grown defect densities are not at an intrinsic minimum and shows that commonly occuring defects are kinetically frozen structures which may be altered with thermally activated processes in a beneficial way. It appears likely that continued detailed characterization of structural defects and their dependence on process variables could lead to efficient improvements in material quality. Improved structural quality would likely bring improved device performance.

Transmission electron microscopy studies have shown that extended structural defects in GaAs/Si include three generic forms: dislocations, stacking faults and twins [3,4]. The densities of these defects were seen to vary strongly with depth in the GaAs films. It is not clearly understood, however, how the relative proportions of these defect types are determined nor what fraction of any one type is due to growth faults as compared to deformation or strain relief. It is therefore also not clearly understood to what degree the defect density gradient is determined by the large lattice mismatch at the substrate interface as compared to the two-step growth process. Due to these uncertainties, it is not clear what options there are for inhibiting the appearance of structural defects nor which stage of growth or anneal is the optimum point to attempt to remove them. To answer questions like these and to monitor progress in the growth technology, it is extremely useful to be able to measure the densities and crystallographic orientations of the structural defects in a type specific way and as a function of depth over a large concentration range and in films whose thicknesses are comparable to device quality films.

We performed grazing incidence x-ray diffraction experiments on GaAs/Si samples in order to obtain basic defect characterization data from a few particular GaAs/Si films and to obtain data demonstrating the degree to which such experiments might generally be expected to fill this need in the study of the GaAs/Si system.

## EXPERIMENTAL

Data from two films, sample A and sample B, will be described. Both samples were grown by MBE in two steps: 100 nm of GaAs was grown at a temperature close to 400 C and 200 nm of GaAs was grown at about 600 C.

The crystallographic orientation of the substrate surfaces differed between samples A and B. As determined by Laue back reflection, sample A had a surface normal inclined 3.7 degrees from the [001] towards the [110] while sample B had a surface normal inclined 1.1 degrees from the [001] towards a direction between the [110] and the [010].

The pre-growth surface treatments were also different between samples A and B. Sample A was cleaned in a manner very similar to that of Shiraki as has been reported [5]. The substrate was heated to 1000 C before growth. Sample B was cleaned with solvents followed by an HF dip and loaded into the MBE chamber under nitrogen. The substrate was heated to 750 C before growth. The preheat temperature used for sample B was considered to be less optimum than that used for sample A.

The full width at half maximum of the (004) GaAs reflection using Cu K-alpha-1 radiation in a double crystal x-ray diffractometer was found to be 550 arc sec for sample A and 1500 arc sec for sample B. By this measure, the aggregate structural defect density of sample A was found to be comparable to, although slightly larger than, the best attained in typical MBE growths of films with the same thickness. The aggregate structural defect density in sample B is many times larger than in conventionally grown material. In the discussion below, sample B will be used as a point of reference to strengthen certain conclusions regarding sample A. Complementary characterizations of sample A have been reported [3,4,5].

X-ray data for the grazing incidence experiments were collected at the Stanford Synchrotron Radiation Laboratory during parasitic and during dedicated operation at beamlines VI and VII. The data described here were obtained in two distinct geometrical arrangements. In one, scattering vectors within a few degrees of the sample surface were studied and the incident x-ray energy was 10 keV. In the other geometry, scattering vectors making large angles with respect to the sample surface were studied and the incident x-ray energy was 7 keV. In both cases, the angular divergence of the incident x-ray beam in the scattering plane was about 0.2 milliradians while the angular divergence perpendicular to the scattering plane was about 5 milliradians. Further details regarding the experimental geometry, resolution, count rates and scan strategies will be reported elsewhere [6].

## RESULTS

The principal advantage of the grazing incidence geometry for studying GaAs/Si films derives from the ability to closely control the penetration depth of the incident beam. Figure 1 shows the penetration depth in GaAs as a function of incident angle for angles close to the critical angle for total external reflection and x-ray energy of 7 keV. With the high collimation of the synchrotron source, it has been possible to clearly limit the incident beam to the near surface region, to the near surface plus high temperature growth layer and to the total layer in turn. Note that a very large volume of reciprocal space can be explored in the grazing incidence mode and it is not necessary to change reflections to change the penetration depth.

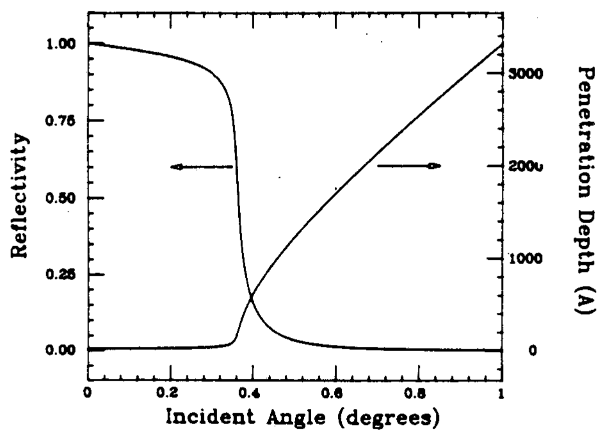



Fig. 1. Reflectivity and penetration depth in GaAs of 7 keV x-rays.

Figure 2a displays the scattered signal as a function of momentum transfer parallel to the sample surface close to the (220) reflection from sample B at a mean incident angle above and below the critical angle. From these data it is seen that when the low temperature layer is included in the sampled volume, there is a nearly two orders of magnitude increase in diffuse scattering close to the Bragg reflection. This is direct evidence of the large defect density gradient in the sample. Further inspection of the diffuse scattering reveals local maxima displaced from the center of the Bragg peak. Figure 2b shows that the displacements of the maxima increase with incident angle. Each scan is primarily along the [220]. Increased incident angle above 0.61 is not increasing the penetration in these thin samples so much as moving the scattering vector further out from the (001) plane. Standard analysis shows that the maxima correspond to streaks emanating from the [220] reciprocal lattice spot. We can consistently identify these streaks with well directed diffuse scattering corresonding to planar faults lying along {111} planes [7]. Note that scans which pass through the (220) reflection center in a direction different from [111] or [-1-11] would miss this planar fault component. To study these signals as a

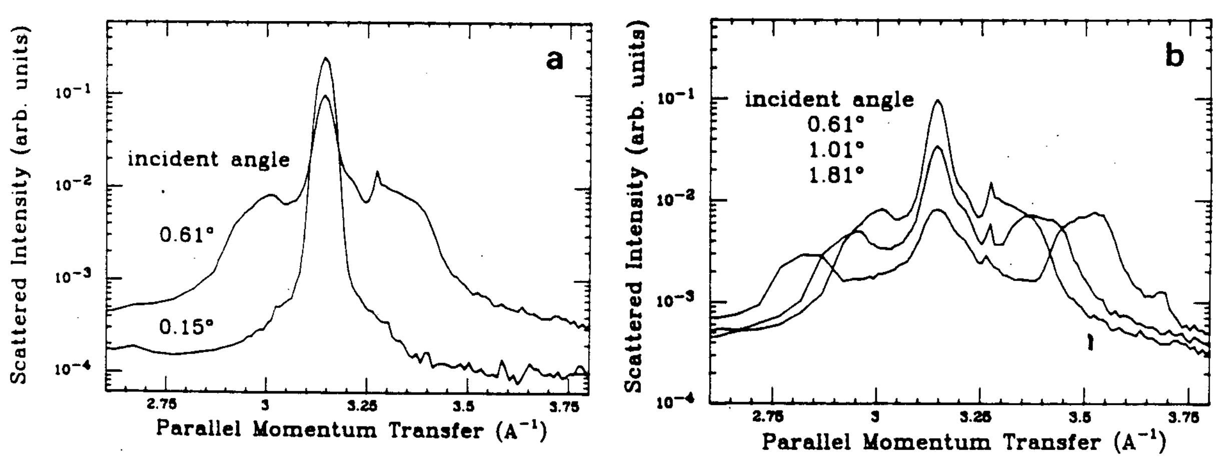
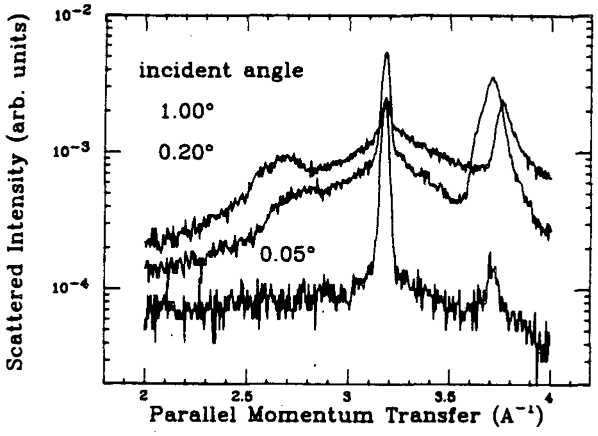



Fig. 2. Scattering from sample B close to the (220) Bragg reflection.



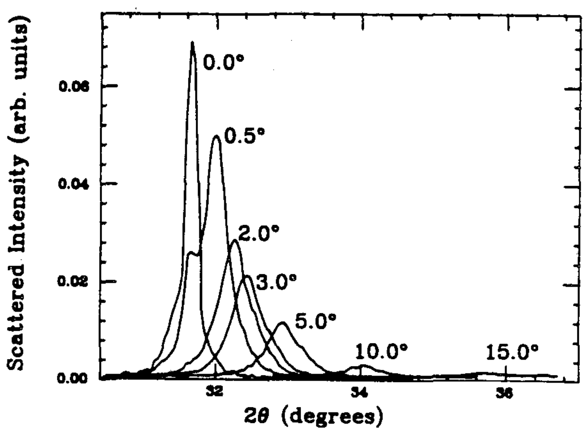




Fig. 3. Scattering from sample A close to the (220) Bragg reflection.

Fig. 4. Scattering from sample B close to the (111) twin reflection at various incident angles.

function of depth, grazing incidence methods coupled with complete crystallographic capability is essential.

Figure 3 shows that qualitatively, the same features were found for sample A. The overall intensity of the diffuse scattering was less than seen for sample B, as was consistent with the lower overall defect density in sample A. We note, too, that similar diffuse scattering, including local maxima, were seen for both samples close to the (-220) reflection. From data like these, quantitative comparisons between samples of the signal strengths for each defect structure type is fully justifiable. We will report on detailed comparisons of this sort elsewhere [6]. Still considering Figure 3, a strong peak is shown at momentum transfers near 3.7 inverse Angstroms. This peak corresponds to a (-1-1-3) reflection from GaAs twinned with a (111) twin plane. Similar twin reflections were seen in sample B. To gain further insight into the twin structrures, we consisdered data from the (111) reflections from the twins.

Figure 4 shows a series of scans of the (111) reflection from twinned GaAs. The scan direction is predominantly perpendicular to [-1-11] and again, increased incident angle shifts the scan in reciprocal space. This method displays the diffuse nature of the twin reflection. The data show it to be streaked in the [-1-11] direction, that is, perpendicular to the twin plane. One interpretation of this diffuseness is that a significant fraction of the GaAs contains discrete twins having a narrow width. So we used the Scherrer equation to estimate a characterisitic size representative of the actual distribution [8]. The result was 9 nm. Combining the diffuse intensity distribution as shown in Figure 4 with the observations of the (-1-1-3) reflection, we estimated that close to one percent of sample B was twinned with a (111) twin plane. To do so, we used the measured intensity of the (220) reflection truncation rod and the theory of Robinson [9] to fix the intensity scale. The data in Figure 4 can be analyzed from an alternative point of view. Following Paterson [7], the intensity shown in Figure 4 could be due to a very large density of the class of stacking fault which, if present on every plane, would yield the twin of the GaAs structure. Using the observed integral breadth, we derived 0.95 as the approximate fraction of planes which would be faulted in this way. We interpret the result of this alternative analysis to imply that discrete, nearly unfaulted twinned regions are a good model for the source of scattering shown in Figure 4.

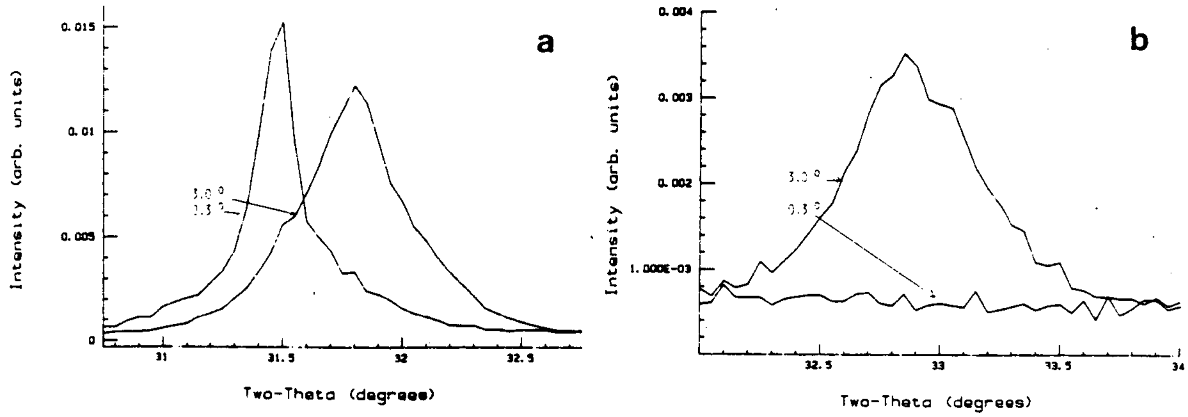



Fig. 5. Scattering from (a) sample B and (b) sample A close to the (111) twin reflection at two incident angles.

Figure 5 shows the difference in depth distribution of twinned material between samples A and B. Unlike sample B, no twins were observed in sample A when the beam was confined to the high temperature layer. Sample B showed twin reflections at orientations corresponding to twins with (111), (-1-11) and (-111) twin planes. Sample A showed twins corresponding to (111) and (-1-11) twin planes. When we looked in the region which would have included scattering corresponding to a (-111) twin plane, no peak was found. The twin formation probability was therefore seen to not follow the four-fold symmetry about [001] which a perfect GaAs film would display. Instead, the pattern was found to follow the symmetry of the sample surface when the surface orientation was taken into account. Twin planes whose projections onto the sample surface lay parallel to the off-orientation direction were found to have a much higher twinning probability than twin planes whose projections onto the sample surface lay perpendicular to the off-orientation direction. This is new, although not the first, evidence of a growth or deformation asymmetry coupled to the off-orientation direction. Sample B did show some asymmetry too, but to a much smaller degree. This may have been due to the skewed off-orientation direction or to the surface preparation procedure.

## SUMMARY

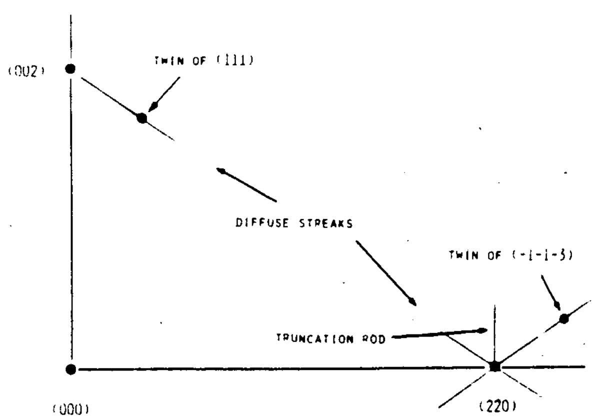



Fig. 6. Schematic projection of reciprocal space showing main features discussed in the text.

Defect structures in thin, MBE grown, GaAs/Si heteroepitaxial films were studied with grazing incidence x-ray scattering techniques. Twins were observed and their densities were seen to be strongly correlated to the off-orientation direction. The density of twin related defects was estimated for one sample and found to be about 1 volume percent. The twins scattering was consistent with a distribution of twin widths characterized by a mean size of 9 nm. The depth dependence of diffuse scattering associated with normally oriented material was also studied. Well directed streaks corresponding to planar faults were observed and easily distinguished from broad and more randomly oriented background. Some of these features have been schematically summarized in Figure 6. Grazing incidence x-ray scattering techniques can be used to quantify, in a type specific way, the overall magnitude and relative probabilities of different forms of structural disorder as a function of depth and orientation in these heteroepitaxial films.

These studies can be readily extended to samples many microns thick. Analogous work would be successful at much lower defect densities. The data shown here were collected in scans with just a few minutes total duration and the angular resolutions were not all optimized for each defect type. It appears that continuation of this work would be helpful in improving and monitoring the GaAs/Si growth technology and in understanding correlations between device performance and structural defect densities.

## ACKNOWLEDGEMENTS

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