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Publication Date
1991

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LBL-30112

Lawrence Berkeley Laboratory

UNIVERSITY OF CALIFORNIA

Materials & Chemical Sciences Division

Presented at the Materials Research Society Conference,
Boston, MA, November 26 –December 1, 1990,
and to be published in the Proceedings

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This work was supported by AFOSR-ISSA-90-0009 and by
the Materials Science Division of the U.S. Department
of Energy under Contract No. DE-AC03-76SF00098.

MICROSCOPIC DETERMINATION OF STRESS DISTRIBUTION IN GaAs GROWN AT LOW TEMPERATURE ON GaAs (100)

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ABSTRACT

A microscopic strain distribution across commensurate interfaces between GaAs layers grown on semi-insulating GaAs substrates was observed by means of convergent beam electron diffraction (CBED) and large angle convergent beam methods (LACBED). Strain relaxation at a specific distance from the interface was observed in these layers without formation of misfit dislocations. It was proposed that specific point defects distributed close to the interface can explain the asymmetric broadening of high-order Laue zone (HOLZ) lines in the CBED patterns.

INTRODUCTION

Epitaxial semiconductor layers with different compositions and lattice parameters can now be grown to considerable thicknesses. These layers have received much attention lately because of their electronic properties. The strained layer growth of thin epitaxial films on substrates with different lattice parameters is sensitive to both the atomic misfit and the film thickness. Depending on the misfit between the layer and the substrate, such a layer can be grown with an unrelaxed strain field (commensurate) or with a relaxed strain field (incommensurate). In commensurate cubic systems grown on a (100) surface, a steady strain field gives rise to tetragonal distortion. Strain relaxation can be achieved by formation of misfit dislocations at the strained interface.

Originally it was suggested by Van der Merwe[1] that interfacial dislocations need to be formed in order to reduce the strain energy of the epilayer. This equilibrium argument did not consider either the dislocation introduction mechanism or any energy barrier to nucleation. A model for the introduction of misfit dislocations was proposed by Matthews and coworkers,[2-4] who considered the behavior of pre-existing threading dislocations under the influence of the epilayer stress. The critical transition occurs when either the epilayer stress or the thickness can lead to dislocation movement. This model implies to the existence of a critical layer thickness for a given composition, at which misfit dislocations can be formed by such a glide process. Using these models, one can determine the critical layer thickness at which an existing dislocation is in equilibrium, because the dislocation line tension is balanced by the misfit stress.

The approach of People and Bean[5] for critical layer thickness determination was to obtain the minimum energy that allowed the strain-energy density and the energy density required for dislocation generation to be the same. Another approach, which ascribed strain relaxation in semiconductor heterostructures to plastic flow, and which included dislocation multiplication, was given by Dodson and Tsao.[6] The values of the critical thickness determined from these models differs from model to model and although considerable theoretical work on this subject has been done, it still does not appear feasible to predict the critical layer thickness in all cases with the accuracy desired for device design. The reason for this inaccuracy is related to applying equilibrium calculations to non-equilibrium growth condition where kinetics factor play very important role.

Convergent beam electron diffraction (CBED), as demonstrated by the Bristol group[7] is a unique tool for studying the changes in local lattice parameter. Using this method, the strain distribution in

substrate. Splitting of this line into two can be noticed. At a layer thickness ~0.3 μm the $(0\bar{4}\,\overline{12})$ line recovers its sharpness, and its position shifts compared to the position of this line on the substrate. When LACBED patterns were recorded in the substrate or in the layer above the interface $(0\bar{4}\,\overline{12})$ line did not experience any broadening or shifting (Fig. 3). Similar broadening of HOLZ lines and recovery about 0.3 μm from the interface was observed for all four different samples tilted to different orientations, indicating that this phenomenon is characteristic of these interfaces.

DISCUSSION

The phenomena of broadening of HOLZ lines starting in the substrate close to the interface can be interpreted as the influence of the strain due to incorporation of As in the LT layer, as already observed by x-ray diffraction.[11,12] The fact that these lines are already affected when the electron beam is placed in the substrate indicates that the upper part of the substrate is strained by the epilayer with a larger lattice parameter. The fact that HOLZ lines smear only in the area close to the interface indicate that strain is responsible for this line broadening. The shift of the $(0\bar{4}\,\overline{12})$ line in the LACBED pattern clearly indicates a lattice parameter change across the layer. The broadening of HOLZ lines in the interfacial area can indicate bending of the planes near the interface. This bending can be caused by the surface relaxation in the thin TEM sample as it was discussed earlier[16,17], but the reason for different relaxation in specific areas of the sample must be related to the existing strain distribution in the bulk sample.

A distinct strain relaxation is observed for the top part of the layer more than ~ 0.3 μm from the interface. This relaxation is observed in commensurate layers without formation of misfit dislocations. The lattice parameter in this top part of the layers is larger than that in the substrate, but it is smaller than the value observed close to the interface. This phenomenon might be understood in the following way: Due to the presence of 0.75% excess As, the LT-GaAs layer has an intrinsically larger lattice constant than the GaAs substrate. Near the interface, lattice planes perpendicular to the interface are forced to assume the GaAs lattice spacing, creating a biaxial compressive strain with a hydrostatic component. At a certain distance from the interface, the covalent lattice relaxes the tetragonal component of the strain, so that the remaining layer is found with only hydrostatic strain with respect to the substrate. This corresponds to the result of the previous x-ray diffraction study using asymmetric reflections.[12] Using a layer of 2.8 μm total thickness the strain in the LT-GaAs layer was found to be cubic, probably because the first 0.3 μm near the interface could not be distinguished from the rest of the layer.

An asymmetry of the width of the lines (197) and $(19\bar{7})$ (as well (195) and $(19\bar{5})$) was observed in the layer close to the interface [Figs. 1(f-i)]. The respective planes with these indices are equally inclined to the interface, therefore the scattering angle should remain the same for the planes with the same indices numbers. This asymmetry cannot be explained by the surface relaxation effect in the thin TEM sample in the electron beam direction, because sharp (197) and (195) HOLZ lines are observed at the same beam position as smeared $(19\bar{7})$ and $(19\bar{5})$ lines. At the same time, all lines with even hkl are broad e.g. $(0\bar{4}\,\overline{12})$, $(0\bar{4}12)$, $(0\bar{6}\,\overline{14})$, $(0\bar{6}14)$, $(0\bar{8}12)$, and $(0\bar{8}\,\overline{12})$. Broadening of these lines gradually disappears when the electron beam is placed either in the substrate or in the upper part of the layer.

One can speculate that this asymmetry of odd hkl lines can be related to a specific point defect distribution in the area close to the interface. Electron paramagnetic resonance and optical absorption studies[11,12] in conjunction with PIXE analysis found in these layers up to $10^{20}cm^{-3}$ As_{Ga} antisite defects and at least the same concentration of additional excess As, probably in interstitial sites, and up to $10^{18}cm^{-3}$ of compensating acceptors.

thin film structures can be studied with high lateral resolution. This method can be applied easily to commensurate structures, because the absence of dislocations at the interface allows easier interpretation of the CBED pattern change due to the strain at the interface. Usually such experimental patterns are fitted by computer-simulated patterns in order to derive an existing strain. CBED has already been applied successfully for strain determination at the Ge-Si layers, grown on Si.[8,9] Another method called large-angle diffraction pattern (LACBED) developed by Tanaka[10] and coworkers, allows the illumination of large sample area. It can be used to observe the local change in lattice parameter distribution.

EXPERIMENTAL

In this paper the strain distribution in commensurate structures was studied by means of CBED and LACBED. These methods were applied to GaAs layers grown at low temperature (LT) (~200°C) on semi-insulating GaAs substrate by molecular beam epitaxy (MBE) using typical Ga and As fluxes for stable arsenic growth of 1 μm/h. First a buffer layer was grown at 600°C, followed by the respective LT layer.

Auger electron spectroscopy and analytical electron microscopy measurements reveal that these layers are very As rich, containing about 1 at % excess As. This excess of As leads to the change in the lattice parameter of the layer. A larger amount of As incorporated into the layer leads to a larger difference in the lattice parameter between the substrate and the layer.[11-15]

A recent TEM study[11-15] of layers grown at a nominal substrate temperature of 200°C showed that these layers are generally of very high crystal quality. Distinct change of contrast between the substrate and the layer was observed when micrographs were taken with (200) reflection. This is a direct evidence of the structure factor change due to different stoichiometry (different As concentration) in the layer and the substrate. No misfit dislocations were found in these structures. However, some samples showed the formation of pyramidal defects in the subsurface area. The difference in the thickness at which the LT-GaAs layer becomes polycrystalline can be related to the magnitude of the lattice mismatch and the consequent strain in the LT-GaAs.[11-15] A careful study, using CBED and LACBED, was performed on four samples, using a JEOL 2010 transmission electron microscope with an accelerating voltage of 200 keV. The excess of As on the LT-GaAs layers, determined by particle-induced x-ray emission (PIXE), was of 0.75 %. A lattice parameter change measured on these layers by x-ray rocking curves using double crystal x-ray spectrometer was 0.13%. An electron beam was focused on specific areas of the sample, starting from the substrate, followed by small steps to the interface and further to the top of the layer. CBED patterns were taken for each position, followed by a micrograph, with the beam position indicating the area from which the diffraction pattern was taken.

RESULTS

An example of the results of CBED studies is shown in Fig. 1. The CBED pattern from the substrate shows high order Laue zone (HOLZ) lines in the central disc. All lines are sharp [Fig. 1(d)]. When the electron beam approaches the interface, at the distance of 0.05 μm two lines ($0\bar{8}\,\bar{12}$) and ($0\bar{8}\,12$) are getting broader [Fig. 1 (e)]. At the interface [Fig. 1 (f)] all lines are smeared except for a line (197) and (195) where a striking asymmetry of the lines ($19\bar{7}$) and ($19\bar{5}$) can be seen. The half-width of these lines is 4×10^{-3} rad and 2.6×10^{-3} rad respectively, compared to 4×10^{-4} rad measured on the substrate. At the distance of 0.2 μm from the interface, a gradual recovery of the sharpness of these lines was observed, with full recovery at 0.3 μm from the interface.

LACBED patterns obtained on the same sample (Fig. 2) show the same broadening starting at the substrate close to the interface up to 0.3 μm from the interface with full recovery further than 0.3 μm. The broadening of the ($0\bar{4}\,\bar{12}$) line is not symmetric compared to the position of this line on the

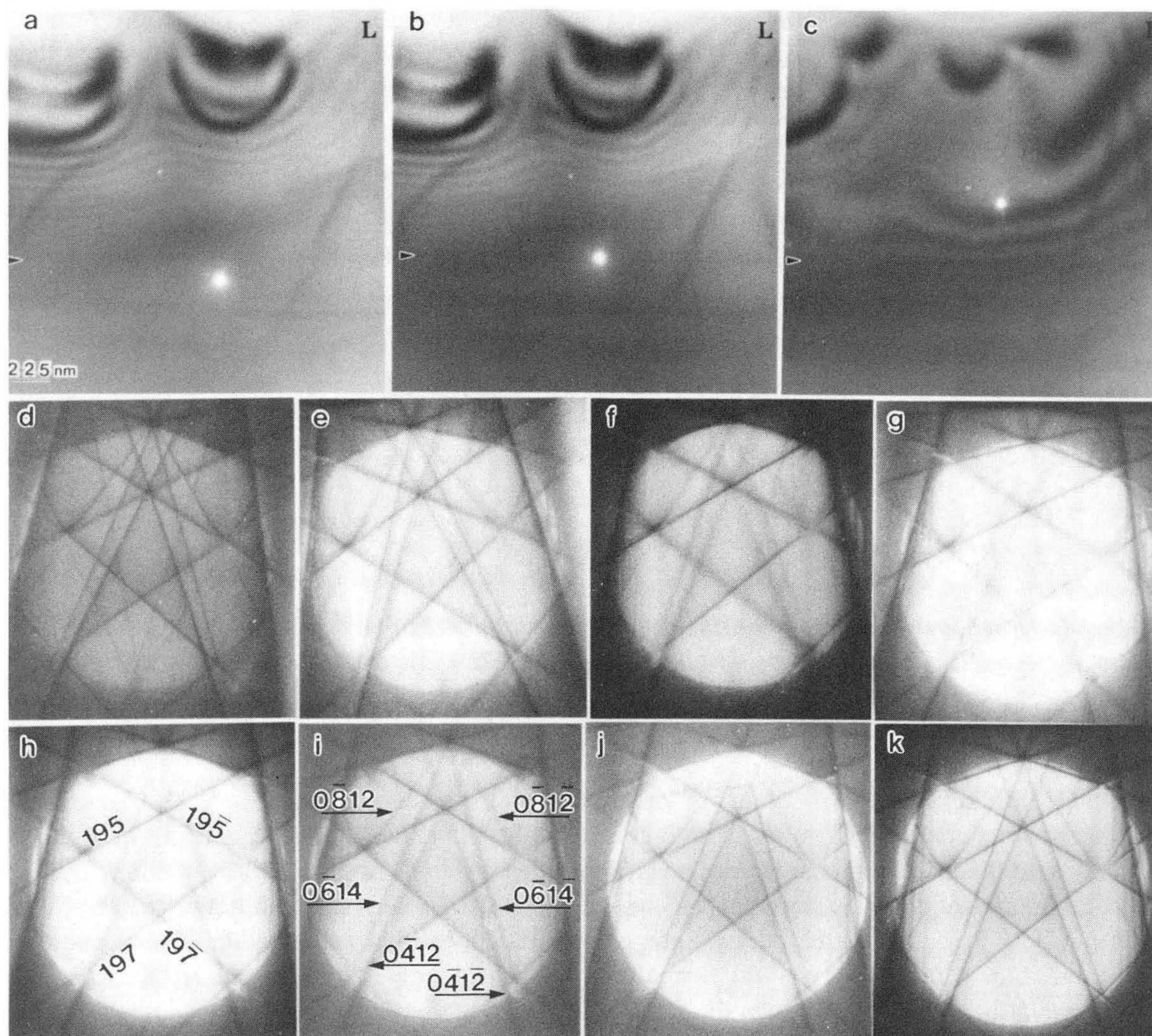

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Fig. 1: A set of micrographs showing beam position and CBED patterns in the cross-section LT-GaAs/ GaAs interface. The interface is marked by arrow: **(a)** beam position in the substrate 0.2 μm from the interface; **(b)** beam position at the interface; **(c)** beam position in the layer 0.3 μm from the interface; **(d)** CBED pattern from the substrate shown in (a); **(e)** CBED pattern from the substrate 0.05 μm from the interface shown in (b); **(f)** CBED pattern from the interface; **(g)** CBED pattern from the layer 0.03 μm from the interface; **(h)** CBED pattern from the layer 0.075 μm from the interface; **(i)** CBED pattern from the layer 0.2 μm from the interface; **(k)** CBED pattern from the layer 0.3 μm from the interface shown in (c); **(l)** CBED pattern from the layer 1 μm from the interface.

GaAs structure factor calculations assuming one As_{Ga} antisite defect per unit cell could not explain the observed asymmetry either of (197) and (19$\bar{7}$) or (195) and (19$\bar{5}$) lines. Similar calculations were done for interstitial As located in tetrahedral sites. Such an atom location could not produce asymmetry of these structure factors either. However, assuming lower symmetry positions such as a <111>-split interstitial (assuming that an As atom at 1/4 1/4 1/4 in the unit cell is shifted to a new position 1/8 1/8 1/8 and an interstitial is inserted at 3/8 3/8 3/8) would explain the observed asymmetry, because the values of a structure factor are different for (197) and (19$\bar{7}$) reflections and are the same for (0$\bar{4}$12) and (0$\bar{4}\bar{1}\bar{2}$) and for (0$\bar{6}$14) and (0$\bar{6}\bar{1}\bar{4}$) reflections. Therefore, it is possible to consider a specific arrangement of point defects to be responsible for the observed asymmetry of the lines with odd hkl numbers. This speculation would find support in PIXE channeling measurements[12] which show a narrowing of As <110> channels, which is a clear indication that interstitials are not located in the tetrahedral coordinations, but could well be explained by low-symmetry locations such as the <111>-split interstitial.

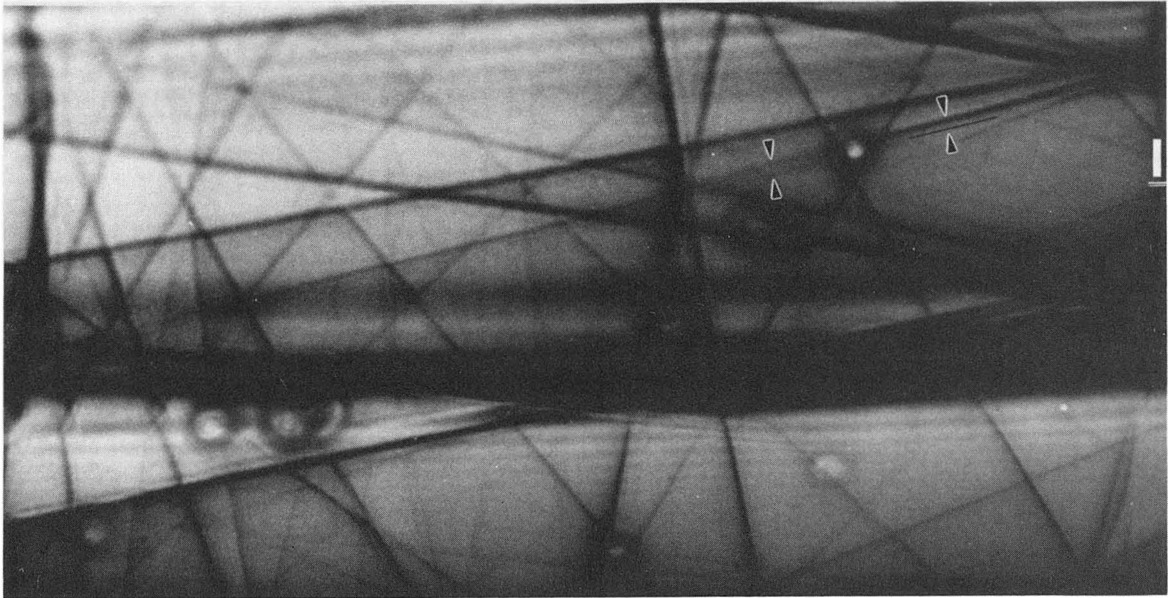

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Fig. 2: LACBED from the interfacial area showing (0$\bar{4}$1$\bar{2}$) HOLZ line shift in the layer indicating a change of the lattice parameter and a distinct broadening of this line (splitting into two lines) only for a specific distance from the interface. A position of the interface is marked by I.

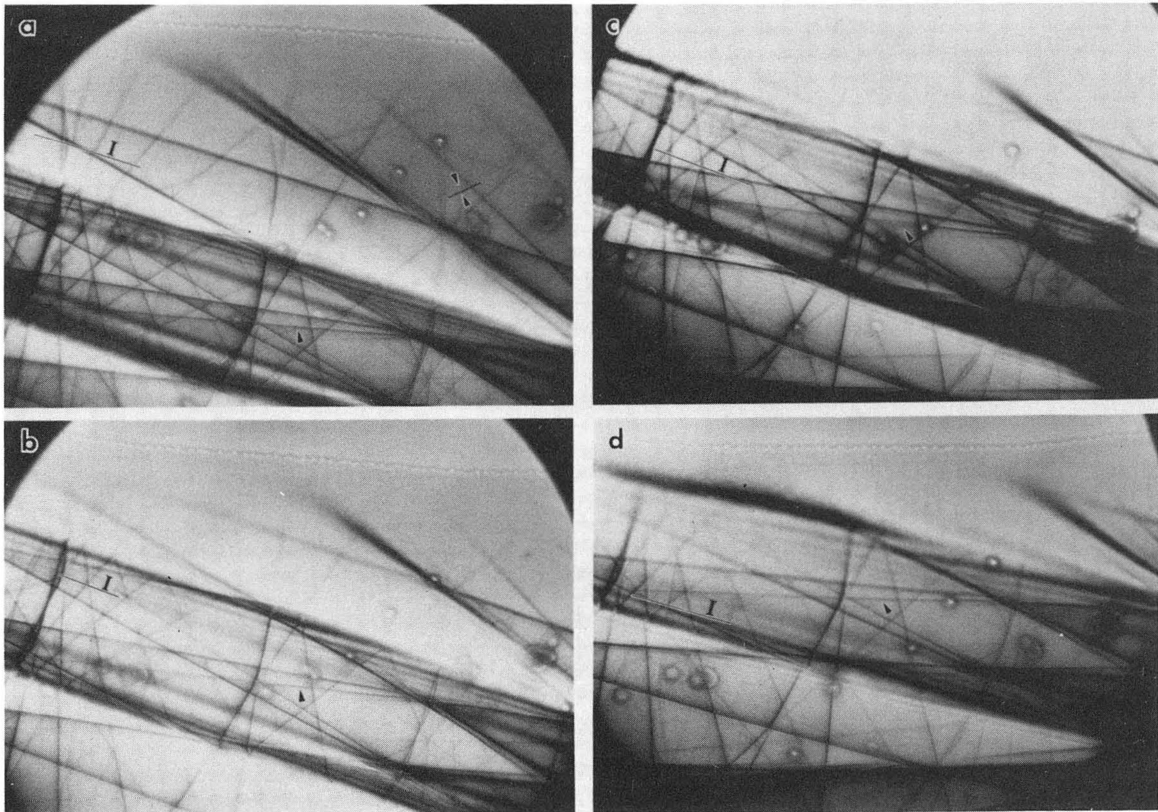

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Fig. 3: LACBED from different areas of the sample: (a) substrate far from the interface, (b) substrate close to the interface, (c) interfacial area, (d) the layer above the interface. A position of the interface is marked by I. A position of (0$\bar{4}$1$\bar{2}$) HOLZ line is marked by arrow. Note that broadening of (0$\bar{4}$1$\bar{2}$) HOLZ line shown in (d) is observed only for specific distance from the interface.

In summary, CBED and LACBED patterns were successfully applied for the spatially resolved determination of strain distribution across the commensurate interfaces between LT-GaAs layers and GaAs substrate. A distinct lattice relaxation without dislocation formation was observed for four interfaces about 0.3 μm from the interface. A possible explanation for the width asymmetry of the HOLZ lines near the interface was proposed by the presence of point defects in the biaxial compressive strain field.

ACKNOWLEDGMENT

This work was supported by AFOSR-ISSA-90-0009. Z.L.W. would like to thank Dr. R.A. Calawa and Dr. F. Smith for supplying samples, Dr. E.R. Weber and Dr. D.J. Eaglesham for fruitful discussions, Dr. K.M. Yu for PIXE measurements, and Mrs. W. Swider for TEM sample preparation. Use of the Facility of the Materials Science Division of the Department of Energy under Contract DE-AC03-76F00098 for completing this study is greatly appreciated. All CBED and LACBED patterns were taken during a stay of Z.L.W. in the Institute of Scientific Measurements at Tohoku University, Sendai, Japan.

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