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High-resolution x-ray analysis of strain in low-temperature GaAs

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X-ray-diffraction measurements have been used to characterize GaAs layers grown at low temperature on GaAs substrates by molecular-beam epitaxy. Three ranges of low-temperature growth are defined, labeled as "low range," (less than 260 °C), "midrange," (between 260 and 450 °C), and "high range," (above 450 °C), as measured by a growth-chamber thermocouple. Films grown in the low range are amorphous, those in the midrange are fully strained and lattice matched to the substrate, and those grown above 450 °C are similar to ordinary GaAs. A notable property of the midrange layers is the expansion and contraction of the lattice parameter with thermal anneals up to 900 °C. From x-ray rocking-curve measurements on more than 200 anneal conditions in this group, a growth model based on arsenic antisite defects is proposed.

INTRODUCTION

The molecular-beam-epitaxy (MBE) growth of arsenic-rich GaAs layers at low temperatures (LT's) on GaAs (LT GaAs) was reported recently.[1-3] The interesting physical and electronic properties of these and similar III-V compounds make them useful for a wide variety of solid-state devices. Among these are reduced recombination times of about 400 fs, suitable for integrated subpicosecond optoelectronic switches, and their high breakdown voltage, useful for power field-effect transitors (FET's).[4,5] The layer can also be used to help eliminate backgating and sidegating in FET's due to their high resistivity, to help enhance intentional interdiffusion, provide a cap layer for nonalloyed Ohmic contacts, and be employed in Fermi-level unpinning in GaAs for surface passivation.[1,6,7] Transmission-electron microscopy (TEM) results reported in Ref. 2 showed the layers to be highly perfect. Double-crystal x-ray rocking curves for the particular growth temperature of 200 °C were also reported, showing a second peak at an angle slightly lower than that of the substrate. This peak was interpreted as arising from a relaxed cubic lattice with a lattice parameter slightly larger than, and supported by, the GaAs substrate, which is also cubic. However, an interfacial dislocation network which is nearly always present in mismatched systems was not reported. This concept of a relaxed cubic lattice, free from tetragonal distortion, appeared to be in conflict with our usual understanding of misfit and threading dislocations. Studies of the electrical properties of LT GaAs along with the observation of tetragonal distortion in LT GaAs have also been reported.[8,9] We have already reported a brief study of the midrange materials, and have suggested the formation of antisite defects as the responsible mechanism for the growth of strained-layer LT GaAs.[10] In the present work, these results are examined in greater detail, and the connection among the three possible crystal structures in the low-temperature (LT) growth is discussed with the aid of depth profile x-ray rocking-curve analysis.

EXPERIMENT

Epitaxial layers of low-temperature GaAs, nominally 0.5-μm thick, were grown on semi-insultating GaAs substrates in a VG V80H MBE system. The substrates were prepared for growth by the standard procedures of degreasing and etching in 7 H_2SO_4:$1H_2O$:$1H_2O_2$. The As-to-Ga beam equivalent pressure, using As_4, was fixed at 18. Following oxide desorption from the substrate surface in the growth chamber at $T_s = 580\,^\circ\mathrm{C}$, the temperature was adjusted to the desired value and stabilized. Within experimental error, all growth parameters other than the temperature were kept the same in different runs. Growth temperatures were monitored by means of a thermocouple located about 12 mm from the sample block, which held the substrates without indium bonding. Temperatures ranging from 150 °C to 500 °C, as measured by the thermocouple, were investigated. The thermocouple readings were generally higher than the actual temperature and represented the heater temperature more closely. A calibration of these temperatures, described elsewhere,[11] showed corrections of about −35 °C for a reading of 250 °C, to about −90 °C for 500 °C.

In order to better understand the mechanism of growth and defect structures in LT GaAs, its annealing effects were extensively investigated. Furnace annealing was done at temperatures ranging from 300 °C to 900 °C in step of 50 °C in a forming gas atmosphere (90% N_2, 10% H_2). The accuracy in the annealing temperatures was ±5 °C. The anneal time at temperature was 10 min. To maintain consistency in the heat treatment, as many as 17 samples corresponding to different growth conditions were furnace annealed together in the same graphite crucible. They were sandwiched between two GaAs wafers to minimize the escape of As during the anneal. The crucible was placed in a quartz tube preheated to the desired temperature. However, the furnace temperature usually dropped by several tens of degrees Celsius after the insertion of the samples, but reached the equilibrium temperature in about 15–20 min. X-ray measurements were per-formed with a Blake double-crystal diffractometer, equipped with Si(100) as the first crystal, adjusted for the (004) reflection of Cu $K\alpha_1$ radiation. The incident beam was 0.4 mm wide × 0.5 mm high which, depending on the angle of incidence, covered a width of approximately 0.4 to 3 mm.

RESULTS AND DISCUSSION

The analysis of x-ray rocking curves from samples grown at *thermocouple* temperatures between 150 °C and 500 °C identified three ranges of low-temperature growth. These were labeled for reference as the "low-range," $T(\mathrm{TC}) < 260\,^\circ\mathrm{C}$, the "midrange," $260\,^\circ\mathrm{C} < T(\mathrm{TC}) < 450\,^\circ\mathrm{C}$, and the "high range," with $T(\mathrm{TC}) > 450\,^\circ\mathrm{C}$. Samples in both the low- and the high range showed only one diffraction peak, corresponding to the substrate lattice parameter alone, while those in the midrange gave two peaks, corresponding to both the substrate and the epilayer. Although, in principle, any GaAs layer grown at less than 500 °C should be called LT GaAs, this term is now applied only to the midrange group, the only one showing strained layers.

X-ray-diffraction analysis of strain was based on one (004) and two (224) rocking curves, the latter in both "glancing incidence" and "glancing exit" configurations (Fig. 1). Initially, four (004) rocking curves were measured, to ascertain that the layers were not tilted with respect to the substrate. Thereafter, only one (004) rocking curve was used in calculating the perpendicular lattice parameter.[12] Nearly 1000 rocking curves on about 40 as-grown and 250 annealed samples were examined. The in-plane lattice parameter and the equivalent bulk (relaxed) lattice parameter were also evaluated assuming that the elastic constants for these layers are nearly the same as for pure GaAs. While in most cases the epilayer was found to be fully strained, meaning in-plane lattice matching of the epilayer with the substrate, a slight relaxation in the epilayer not exceeding 5% was occasionally observed. The initial as-grown vertical strains $(a_\perp - a_{\mathrm{sub}})/a_{\mathrm{sub}}$ were positive for all of the layers, indicating a larger bulk lattice parameter for the epilayer compared to the substrate. We must point out that the expression "vertical strain" is used only to compare the vertical dimensions of the epilayer and the substrate, and does not represent the magnitude of elastic strain. The reader is referred to Ref. 12 for a definition of elastic strain and its measurement in epitaxial systems. Using that method, the maximum bulk lattice parameter of the "free-standing," as-grown LT GaAs was found to be 0.08% greater than ordinary GaAs. This number was obtained from the maximum rocking-curve peak separation of nearly 200 arc sec, or a vertical strain of 1.5×10^{-3} (0.15%), for full, in-plane lattice matching, or a true strain of 100%. Material deposition was generally uniform over the entire surface of the wafers, but the as-grown samples often gave varying and unpredictable vertical strains for the same system temperature (Fig. 2). Although this variation seemed to be random with the first few measurements, a well-defined clustering of the data into two bands was noted as more measurements were taken. Additional data on the effect of the growth parameters and their interaction on the final strain are being collected to understand this phenomenon.

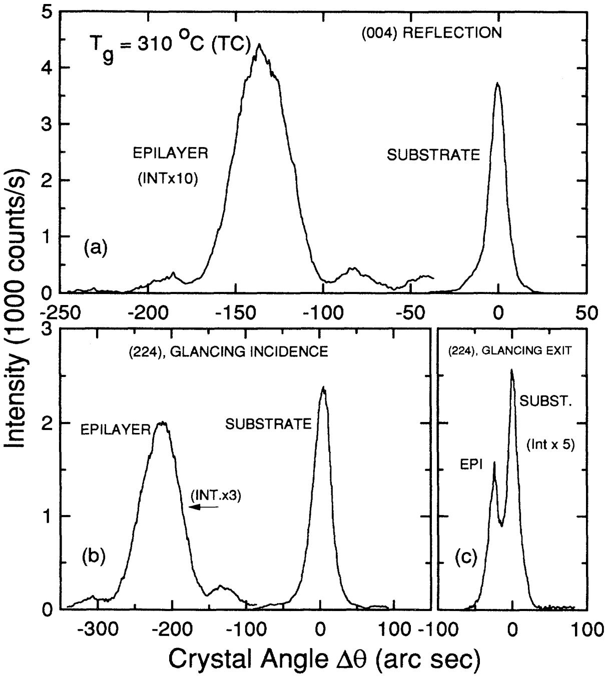

FIG. 1. X-ray rocking curves of LT GaAs on GaAs. (a) symmetric (004) reflection, (b) asymmetric, glancing incidence (224) reflection, and (c) asymmetric, glancing exit (224) reflection.

ANNEALING EFFECTS

For annealing studies in the midrange group, a large number of samples with different initial strains was chosen from those plotted in Fig. 2. X-ray rocking-curve measurements showed a complex pattern of expansion

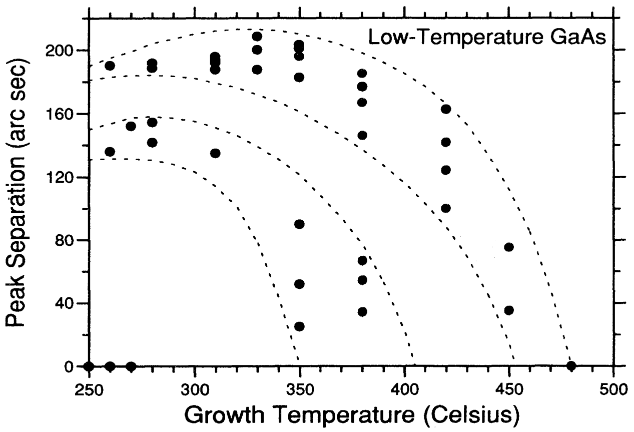

FIG. 2. Variation of the rocking-curve peak separation for different growth temperatures, recorded by thermocouple, in as-grown LT GaAs.

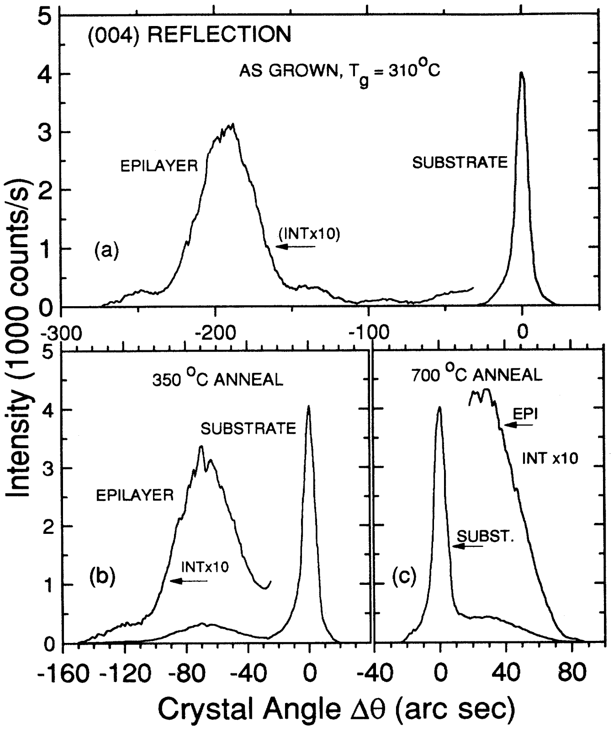

FIG. 3. Shift of the expilayer diffraction peak with anneal in LT GaAs; (004) rocking curves. (a) As-grown material; (b) furnace annealed at 350 °C for 10 min; (c) furnace annealed at 700 °C. The growth temperature is the value recorded through the MBE chamber thermocouple.

and contraction of the epilayer lattice parameter relative to the substrate. Figures 3(a)–3(c) show the rocking curves for a sample grown at 310 °C (TC) in the as-grown condition and after furnace annealing at 350 °C and 700 °C, respectively. Typical strain plots (equivalent to rocking-curve peak separations) for these and several other anneal temperatures are shown in Fig. 4. Most samples annealed at temperatures between 400 °C and 600 °C eventually reached a zero vertical strain with respect to the substrate. However, some samples, when annealed at higher temperatures such as 700 °C, showed lattice contractions up to 0.04%, whereas other layers showed no contraction at all. Layers which did show an initial contraction with anneal, later showed again a zero vertical strain near 750 °C–800 °C. Finally, when these samples were further annealed at temperatures near 900 °C, they showed an asymmetrically higher intensity on the low-angle side of their rocking curves. This asymmetry could not be correlated with a single, distinctly different lattice parameter, but was interpreted as a continuous distribution of slightly larger lattice parameters. It is, therefore, important to keep in mind that the zero vertical strain observed after annealing at about 600 °C does not necessarily imply lattice relaxation, even though it may appear so to the x rays, since annealing at a higher temperature may still alter the lattice parameter.

An interesting feature is noted in the strain anneal data of Fig. 4. The vertical strain begins to change at an anneal temperature T_a which is less than the growth temperature as measured by the thermocouple. For example, sample no. 5 (filled circles), grown at a thermocouple temperature of 330 °C shows a transition in the vertical strain at $T_a \approx 260$ °C. Similarly, the sample grown at 450 °C (TC) (filled triangles) shows sign of strain reduction at below 370 °C anneal. A related effect is that the observed maximum vertical strains appear to be unrelated to the thermocouple readings. The same curves, however, show a systematic order when rearranged in terms of the transition temperatures. It can thus be shown that the latter are more closely tied to the actual growth temperatures.[11]

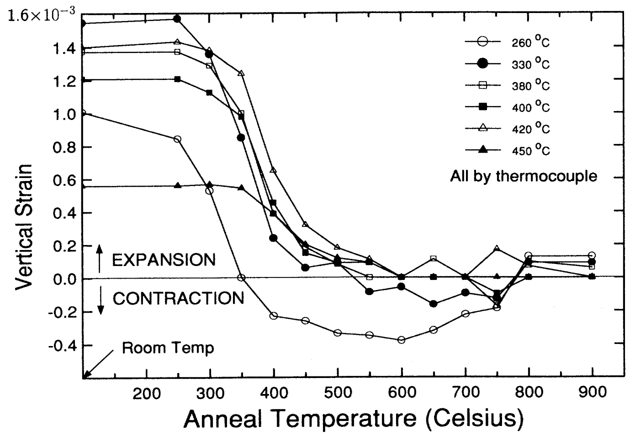

FIG. 4. Typical strain plots as a function of furnace anneal temperature for several samples grown between 260 °C and 450 °C as recorded through the MBE chamber thermocouple (TC).

From the x-ray-diffraction point of view, one may interpret the observed changes in the strain in terms of interstitial and substitutional defects, precipitates, and antisite defects, which influence the evolution of the rocking curves in different ways. For example, interstitial and substitutional defects are known to produce measurable changes in the lattice parameter in the as-grown state. This is a familiar phenomenon in ion implantation and epitaxial growth. However, these defects cannot account for the reduction in the lattice parameter after anneal relative to the host lattice. On the other hand, the effect of the precipitates is to increase the full width half maximum of the rocking curves without shifting the average peak position, i.e., without changing the average lattice parameter. It should be noted that arsenic precipitates have been observed in LT GaAs by many researchers using transmission-electron microscopy. On the other hand, our TEM studies on samples grown at the U.S. Naval Research Laboratory (NRL) have revealed no significant levels of precipitates in the as-grown samples, although these did appear in the annealed materials. The difference in the precipitate content among samples grown at various laboratories suggests that these defects are not "universally" essential for the growth of LT GaAs.

Figures 5 and 6 show high-resolution electron micro-

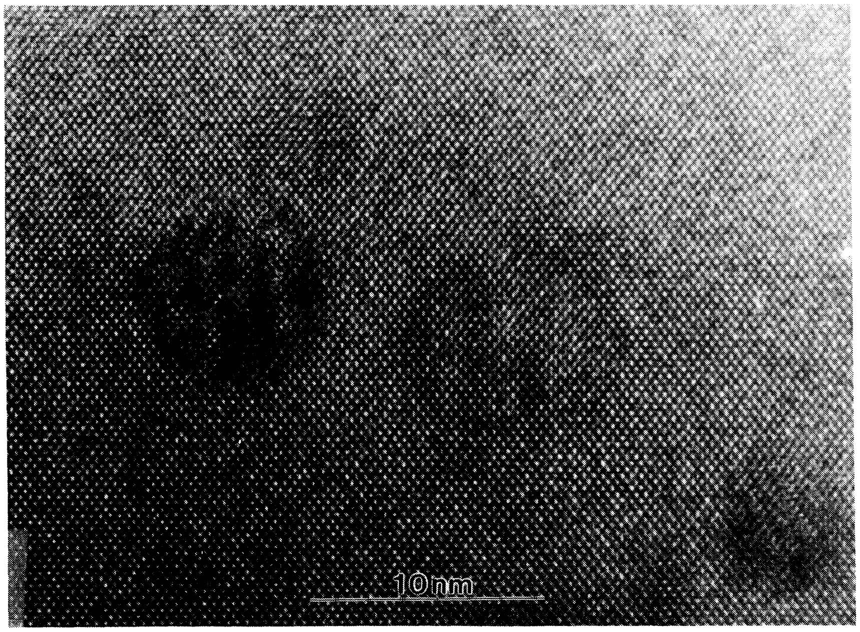

FIG. 5. High-resolution transmission-electron micrograph of an LT GaAs layer grown at thermocouple temperature of 310 °C measured by MBE thermocouple (TC) and annealed at 650 °C. Small precipitates 2–10 nm are seen, with moiré fringes indicating a region of different lattice parameter.

graphs from a sample grown at 310 °C (TC) and annealed at 650 °C, and from the same sample annealed at 800 °C. While no dislocations or precpitates were visible in the as-grown condition, small precipitates about 2–10 nm in diameter were seen in the sample annealed at 650 °C, and larger precipitates, nearly 30 nm, at the higher anneal temperature. High-resolution transmission-electron-microscopy (HRTEM) estimates also showed that the order of magntidue of the total arsenic precipitate volume fraction did not change with the anneal.

Further consideration of these facts leads us to believe that arsenic antisite defects play an important role in lattice strain in our LT GaAs materials, since they can account for both expansion in the as-grown state electrostatically, as well as for contraction after anneal by forming vacancies.

We now examine the connection between LT GaAs (midrange) materials and each of the two remaining temperature ranges. As seen from Fig. 2, the extent of strain over which these materials are grown narrows considerably at both extremes of 260 °C (TC) and 450 °C (TC), compared to the materials belonging to the "core" temperatures such as 310 °C (TC). We also note that the samples grown at the extreme temperatures have peak x-

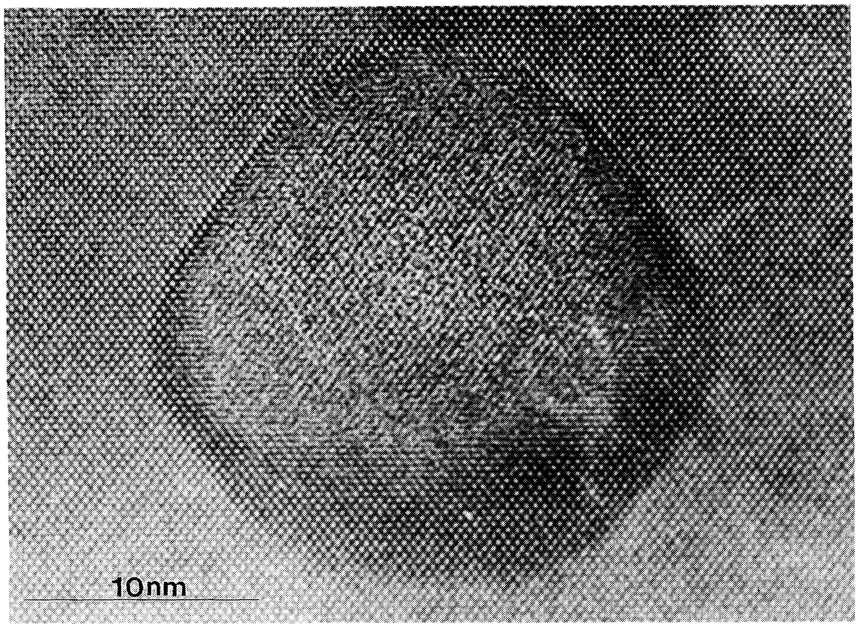

FIG. 6. High-resolution transmission-electron micrograph of a sample grown at 310 °C (TC) and annealed at 800 °C, showing a faceted precipitate approximately 30 nm in diameter. The structure within the precipitate region appears amorphous.

ray efficiencies about one-third that of the remaining ones (compare the as-grown conditions in Figs. 8, 9, and 11). In order to understand the source of this difference, samples grown at the three temperatures mentioned above were chemically etched in steps of approximately 400 to 800 Å, and their rocking curves measured. A mixture of 1 part NH_4OH (30% solution): 2 parts concentrated H_2O_2 (30% solution): 1 part H_2O by weight, diluted 1:100, was used as the etchant, giving an etch rate of about 900 Å (90 nm) per minute. The etching process was repeated as long as any diffracted intensity from the epilayer could be measured. Figures 7–10 show the variation of the rocking-curve intensity as a function of depth for the three samples.

For reference, we consider the sample grown at 310 °C (TC), Fig. 7. Sustained crystalline perfection throughout the growth is apparent from diffraction fringes typical of thin, perfect crystals. The relative peak intensities are nearly proportional to the thickness of the remaining epilayer after the etch. A subtle shift of the peak toward the substrate peak is noted as the layer thickness increases, indicating a lower true strain. The fact that the measured bulk lattice parameter for the layer remains constant indicates that the quality of the layer may deteriorate only slightly with growth.

In contrast, the "borderline" sample grown at 260 °C (TC) retains the same peak intensity when a layer of approximately 50 nm has been removed, but it also shows a slight reduction in half-breadth [Fig. 8(a), filled circles]. Beginning with the 100-nm etch, the peak intensity first rises unexpectedly by nearly 20%, accompanied by a further reduction in the half-breadth [Fig. 8(a), filled squares]. The peak intensity then decreases with further etching to a level equal to that of the as-grown one, at the same half-breadth. The crystal quality thus remains good to a depth of 300 nm. Below this point, lattice perfection initially worsens [filled circles, Fig. 8(b)], but improves to a depth of 550 nm. The profile at 600 nm consists only of the substrate peak. It is interesting that aside from a thin layer marked by the 350-nm etch, the peak separation between the substrate and the epilayer decreases systematically with decreasing layer thickness. This effect is due both to a reduced strain and the dynamical x-ray diffraction in thin films, which has been observed previously.[13,14]

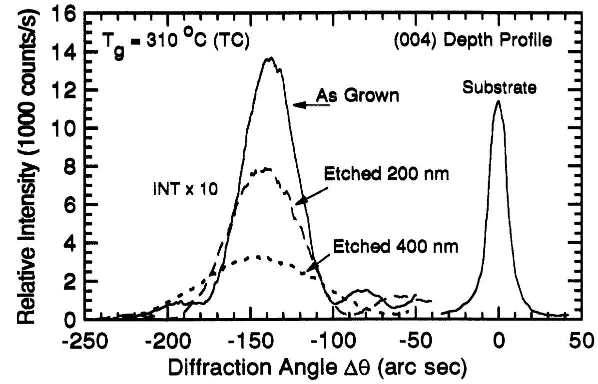

FIG. 7. X-ray rocking-curve profiles from an epitaxial LT GaAs layer grown at 310 °C (TC), and etched to 100- and 300-nm thicknesses. The high perfection of the layers is seen from the fringes due to perfect crystal diffraction from thin films.

Changes in the shape and intensity of the substrate rocking curve add further insight into the defect configuration in this sample. Figure 9 shows the progression of the substrate rocking curves for several stages of etching among those shown in Figs. 8(a) and 8(b). An interesting aspect is the enhanced tail of the rocking curve for the as-grown condition compared to the perfect substrate. Clearly, if the only effect of the epilayer were to reduce the diffracted intensity from the substrate through absorption, no such broadening would have been observed. The reduced rocking-curve peak intensity before the etch, together with a higher tail intensity, suggests the presence of a thin layer of GaAs containing point defects and amorphous material near the outer surface of the epilayer. Since the growth temperature in this sample is at or near the low range, the formation of these imperfect layers should not be surprising.

A more systematic strain configuration governs the growth of LT GaAs at the high-temperature extreme of the midrange group, 420 °C (TC), Fig. 10. The LT GaAs intensity is seen to diminish immediately after removing 40 nm of the material. The intensity continues to decrease with etching and vanishes at a depth of 240 nm. Here, as in Fig. 8, the peak separation between the epilayer and the substrate decreases with decreasing film thickness. The rocking-curve half-breadth, while seem-

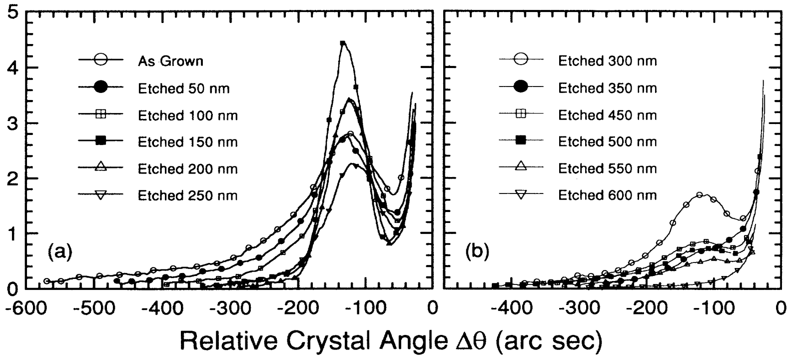

FIG. 8. X-ray rocking-curve data from an LT GaAs sample grown at 260 °C measured by the thermocouple (TC). (a) Sample etched from 0 to 250 nm, (b) sample etched from 300 to 600 nm.

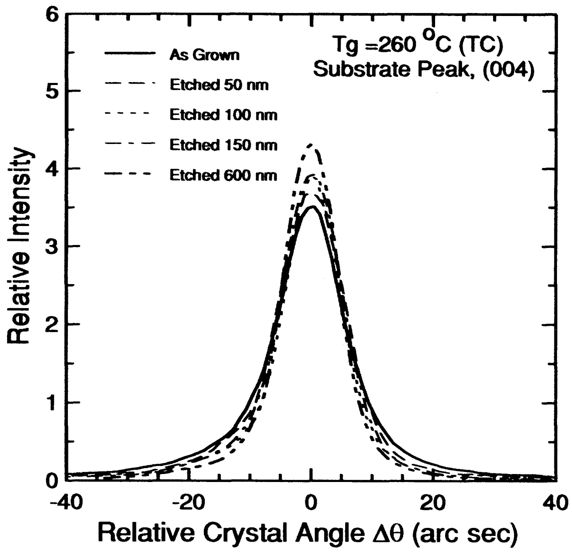

FIG. 9. Substrate (004) rocking-curve peaks from the etched sample of Fig. 8 showing the broader profile near the surface compared to the interior of the LT GaAs layer.

ingly increasing as more material is removed, nonetheless corresponds to perfect crystal values for films in the range of 50–200-nm thickness.

The rocking curves of Figs. 7 and 8 indicate that near the low-range limit [260 °C (TC)] only part of the layer has the highest perfection, in the form of a strained layer. This reflects the sensitive state of transition from the amorphous to the fully strained stage. On the other hand, Fig. 10 indicates that near the high-range limit (420 °C) the crystal quality can improve with growth. It is seen that the LT GaAs layer is bounded by two imperfect regions, one at each extreme of the epilayer. The growth of a strained-layer LT GaAs following the growth of an imperfect layer near the interface occurs at both the upper and the lower boundaries of the midrange. We suspect that this phenomenon is connected with the "bimodal" randomness shown in Fig. 2.

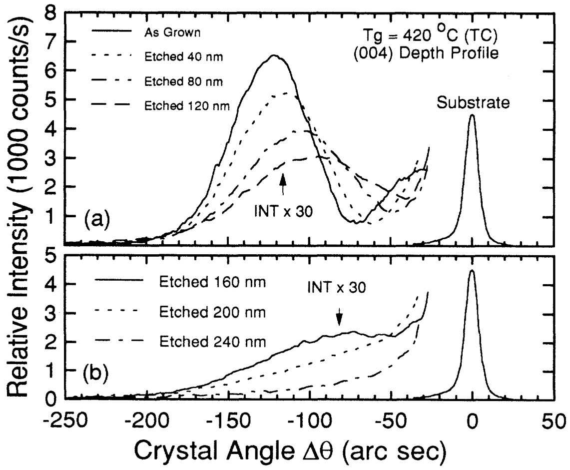

FIG. 10. (004) rocking curves of a sample grown at the high-temperature limit of the midrange group, 420 °C (TC). (a) Etched from 0 to 120 nm; (b) etched from 160 to 240 nm. Although the nominal thickness of the layer was 0.5 μm, the diffracted intensity disappears at about 250 nm. Hence, only the top half of the layer is strained.

The distinction between the lower and upper growth temperature limits generally arises from the difference in conditions that produce the amorphous and the ordinary GaAs structures, respectively. This is also seen in the relative volume and location of the strained and the imperfect layers. For example, at the low-temperature end of the midrange group, the strained layers are affected by the growth conditions similar to those of the amorphous regime. Fluctuations in these conditions could thus result in layers of poor crystalline quality, reducing the substrate x-ray intensity by absorption. On the other hand, in the high-temperature limit, the formation of the strained layer near the top surface is related to the conditions appropriate to the growth of ordinary GaAs. The epilayer may thus consist of both ordinary GaAs and strained layers, the mixture resulting from fluctuations in the prevailing system conditions at that limit.

Based on the foregoing discussion of the x-ray results, we propose the following model (subject to further research and verification by other techniques) for the growth and the defect structure in the strained-layer LT GaAs, i.e., the material grown at estimated actual temperatures of 210 °C to 340 °C. During low-temperature deposition, antisite As_{Ga} defects are produced in the arsenic-rich environment, causing the epilayer unit cell to expand. Other defects, such as precipitates, may also be formed depending on the particular growth system and the extent of arsenic overpressure. Subsequent annealing removes the antisite defects and gives rise to gallium vacancies, causing the lattice to contract. At even higher anneal temperatures, the Ga vacancies are also removed, and a relaxed lattice is formed which contains line defects (dislocations and stacking faults), as well as precipitates and interstitial arsenic. Using a simple calculation involving electrostatic forces between the antisitie defect and its neighboring atoms in the GaAs lattice, the concentration of the As_{Ga} defects can be estimated to be about 5×10^{18} cm^{-3} or a defect volume fraction of 0.001.[11] This number which, fortuitously, is of the same magnitude as the measured strain, is nevertheless an order of magnitude less than the measured stoichiometric imbalance.

In this connection, we also measured the arsenic level in several of our samples before and after anneal using energy dispersive x-ray spectroscopy (EDXS). Groups of repeated measurements on each sample indicated the presence of excess As, at a level in the order of 1%, agreeing generally with the results reported by earlier investigators.[2] An attempt was also made to measure the level of As_{Ga} defects by electron paramagnetic resonance (EPR). With the total volume of the LT GaAs material much smaller than that of the substrate, the minimum detectable limit for these defects was calculated to be about 10^{18} cm^{-3}. The fact that no EPR signal was detected is consistent with the level of antisite defects estimated by x-ray diffraction. Results of further measurements using extremely thin substrates to increase the detection limit in EPR will be presented in a later publication.

CONCLUSIONS

In this paper, several aspects of the growth of LT GaAs layers on GaAs substrates were investigated through the analysis of strain by high-resolution x-ray diffraction. A summary of the results follows.

(1) Strained layers of LT GaAs can be grown on GaAs substrates at temperatures ranging from 260 °C to 420 °C, as measured by a thermocouple in the growth chamber. This range is considerably wider than those reported by others.

(2) The bulk lattice parameter of the as-grown LT GaAs is larger than that of the substrate by varying amounts up to 0.08%.

(3) An antisite defect model for LT GaAs based on x-ray data is proposed to account for the modulation in the lattice parameter following thermal anneal. The strain level in the as-grown lattice is consistent with a concentration of $\mathrm{As_{Ga}}$ antisite defects of approximately 5×10^{18} cm^{-3}. The volume fraction of the antisite defects is an order of magnitude less than the total stoichiometric excess As in the growth system. However, in contrast to the x-ray technique, both EPR and high-resolution TEM techniques are either limited in sensitivity or difficult to use in characterizing antisite defects.

(4) The apparent lattice matching at temperatures near 500 °C–600 °C may, in some cases, correspond to a nonequilibrium state, and hence should not be taken as equivalent to complete strain relaxation. Subsequent lattice contraction observed in some samples for anneal temperatures above 600 °C is significant, since it suggests the presence of unrelieved internal microscopic stresses which influence the electrical properties of LT GaAs.

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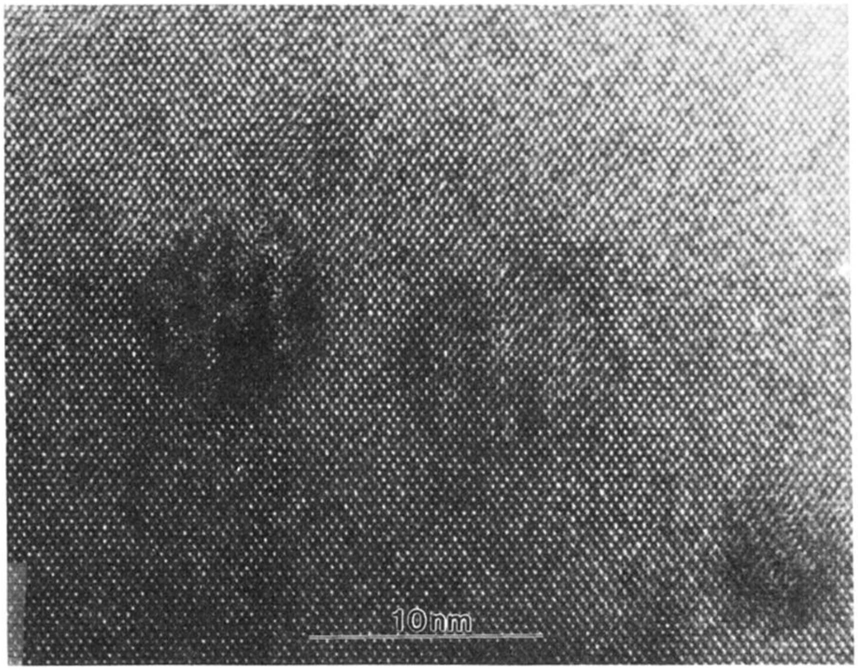

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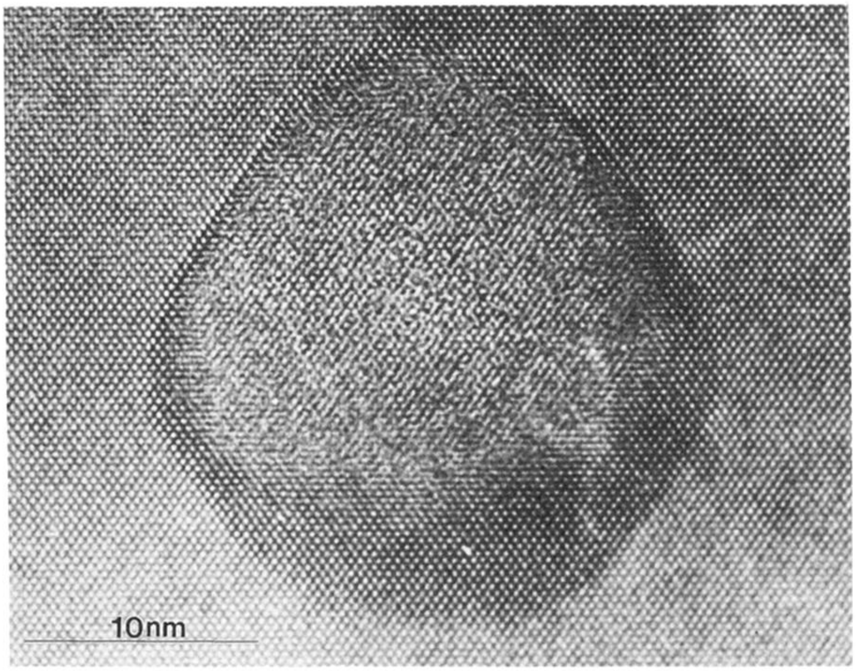

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