Analyses of metalorganic chemical-vapor-deposition-grown $Al_xGa_{1-x}As/GaAs$ strained superlattice structures by backscattering spectrometry and x-ray rocking curves

A. H. Hamdi, V. S. Speriosu,[a] and M-A. Nicolet
California Institute of Technology, Pasadena, California 91125

J. L. Tandon and Y. C. M. Yeh
Applied Solar Energy Corporation, City of Industry, California 91749

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Backscattering spectrometry with channeling and x-ray rocking curves have been employed to analyze metalorganic chemical-vapor-deposition-grown $Al_xGa_{1-x}As/GaAs$ strained superlattice structures in significant detail. Both techniques complement each other in the precise determination of composition, thickness, and strain in the individual layers of the superlattices. In addition, the sensitivity of the two techniques allows quantitative measurements of transition regions at the interfaces of various layers. Such fine probing into thin layered superlattice structures provides essential feedback in controlling their growth.

In the past decade, strained thin layer $Al_xGa_{1-x}As/GaAs$ superlattice (SLS) structures have received considerable attention because of their unique electrical and optical properties.[1] Modern developments in molecular beam epitaxy (MBE) and metalorganic chemical vapor deposition (MOCVD) have made the growth of these structures possible with a claimed individual layer thickness of as low as ~60 Å.[2] However, to control the growth of these structures, accurate quantitative measurements of their composition, thickness, and uniformity are mandatory. Analytical techniques that have been used mainly to characterize SLS structures, e.g., Auger electron spectroscopy (AES), secondary ion mass spectrometry (SIMS), transmission electron microscopy (TEM), are all destructive and have not been able to provide precise details sufficiently. In addition, AES and SIMS require standards for absolute determination of composition and have limited depth resolution.

In this communication, backscattering spectrometry (BS) with channeling and x-ray rocking curves have been employed to analyze MOCVD-grown $Al_xGa_{1-x}As/GaAs$ SLS structures. The two techniques are essentially nondestructive, self-calibrative, and together provide precise determination of composition, strain, thickness, crystal quality, and uniformity of the SLS structure.

The $Al_xGa_{1-x}As/GaAs$ SLS structures used in this study were grown in a computer-controlled large-capacity MOCVD reactor. The reactor is production compatible with a handling capacity of 90 2×4 cm^2 wafers at a time. Two sets of SLS structures were grown on semi-insulating GaAs wafers oriented ~2° off ⟨100⟩ axis. Individual layers of $Al_xGa_{1-x}As$ and GaAs were grown at ~730 °C by switching on and off the Al source (trimethyl-aluminum) and modulating the mole fraction of the Ga source (trimethyl-gallium). Table I gives the time cycles involved in the growth of one period of the two SLS structures, along with the expected thicknesses calculated from growth rates determined from measurements made on thicker (~2000 Å) layers. These two SLS sets represent typical samples expected to be grown in a real production-type large-scale MOCVD reactor with a reasonable growth rate (~7.5 Å/sec).

BS measurements were made by a 2-MeV $^4He^+$ beam tilted at an angle of 80° with respect to the sample's surface normal, to obtain high depth resolution.[3] Channeling was carried out along ⟨100⟩ axis. Bragg case, double-crystal, x-ray rocking curves were obtained with the Fe $K_{\alpha 1}$ (200) reflection. The x-ray beam was collimated and rendered nearly monochromatic by (400) reflection in ⟨100⟩ GaAs. Experimental rocking curves were fitted using a kinematical model of x-ray diffraction in thin epitaxial layers.[4]

Backscattering and channeling spectra obtained from SLS1 (see Table I) and virgin GaAs samples are shown in Fig 1. The oscillatory behavior in the random spectrum is due to modulation in the Ga concentration in the alternating layers of $Al_xGa_{1-x}As$ and GaAs. The spectrum resolves only the first four periods of the sample. A 80° tilt angle was used for this measurement to enhance the depth resolution near the surface. Also, resolving deep layers becomes difficult due to the interfering signals from Al. Compared with the random spectrum from the virgin GaAs sample, the random spectrum from the SLS1 sample has a lower yield. This implies that in the growth of the SLS1 structure, pure GaAs layers were not achieved (the energy resolution of the measurement system used is five channels, which is enough to resolve at least the first GaAs peak in the SLS1 spectrum). Corresponding measurements carried out at a 45° tilted angle, where the analyzing beam probed to a greater depth into the samples, showed that both the virgin and the SLS1 samples had identical substrate yields. It is also interesting to observe a nonsymmetry in the signals of the individual periods. This reflects the existence of uneven composition transition regions at the two interfaces in a period of SLS1 structure. However, excellent crystalline quality of the SLS1 sample was verified by channeling along the ⟨100⟩ direction. The measured minimum yield of ~5% is comparable to that of the virgin GaAs sample.

[a] Present address: IBM Research Division, San Jose, California 95153.

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TABLE I. Growth parameters for SLS1 and SLS2 samples.

	Time schedule for growth of one period (s)			
	TMA on ($MF = 2.06 \times 10^{-4}$) TMG on ($MF = 1.44 \times 10^{-5}$)	TMA off TMG off	TMA off TMG on ($MF = 1.15 \times 10^{-4}$)	Expected period thickness (Å)
SLS1 (10 periods)	7	20	9	120
SLS2 (15 periods)	24	20	30	400

MF = Mole fraction.
TMA = Trimethyl-aluminum.
TMG = Trimethyl-gallium.

The measured (dashed line) and calculated (solid line) x-ray rocking curves obtained from the SLS1 sample are shown in Fig. 2. The angle $\Delta\theta$ is plotted relative to the Bragg angle of the substrate peak. The reflecting power is normalized with respect to the intensity of the incoming x-ray beam. Several peaks in the rocking curves are observed which are due to the periodicity in the sample. The substrate peak is at $\Delta\theta = 0$. The major peak P_0 (which overlaps with the substrate peak in this case) measures the average strain in the SLS structure. The magnitude of the average strain was determined using Fe $K_{\alpha 1}$ (400) reflection in another measurement where P_0 could well be separated from the substrate peak. The separation between the subsidiary peak (P_1, P_{-1}, etc.) in Fig. 2 corresponds to the average period thickness of the SLS structure. Details of the interpretation of the rocking curves for SLS structures are given in Ref. 5. The calculated curve was fitted to the experimental curve using a kinematical model of x-ray diffraction in thin epitaxial layers.[4] In the fitting, normal absorption coefficient and structure factor values were calculated from the tabulated atomic scattering factors.[6] Best fitting was accomplished by incorporating nonsymmetric transition regions at the two interfaces of each period in the SLS structure, without feedback from the BS data. The strain distribution as a function of thickness in one period of SLS1 structure which provided the best fit to the experimental curve is given in Fig. 3. Since a one-to-one correlation exists between strain and Al concentration in $Al_xGa_{1-x}As/GaAs$ structures,[7] an analysis of the rocking curve quantitatively determines the variation of Al concentration in the period. The result of this calculation is also reported in Fig. 3 (right-hand scale), and confirms the BS observations that Al undergoes uneven composition transition within one period. The sensitivity of the fitted rocking curve is such that the mismatch between the higher-order peaks in the measured and the calculated curves in Fig. 2 suggests a nonuniformity in the thickness of the various periods of < 50 Å.

The skewed strain (Al concentration) distribution determined by x-ray rocking curves in the period of SLS1 sample is compared with expected sharp distribution in Fig. 3. The average measured thickness of the period is 410 ± 15 Å (Ref. 8) when compared with expected thickness of 120 Å. The steps in the strain distribution are only suggestive of the true strain curve which one should expect to be continuous, as shown in Fig. 3 by the dotted curve. Qualitative features of this continuous strain (composition) curve are also evident from the BS measurements (see Fig. 1).

The skewed strain curve provides insight into the growth of $Al_xGa_{1-x}As$ and GaAs layers within the period of the SLS1 sample. The relatively sharp rise during the

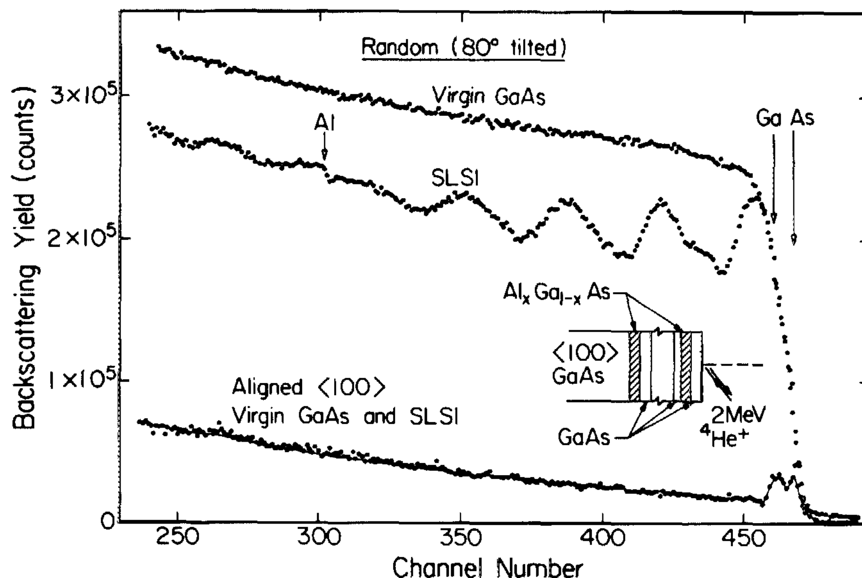

FIG. 1. Random (80° tilted) and ⟨100⟩ channeled backscattering spectra obtained from SLS1 and virgin GaAs samples. The lower yield of the random SLS1 spectrum, when compared with the random virgin GaAs spectrum, implies that pure GaAs layers were not achieved during the growth of the SLS1 structure.

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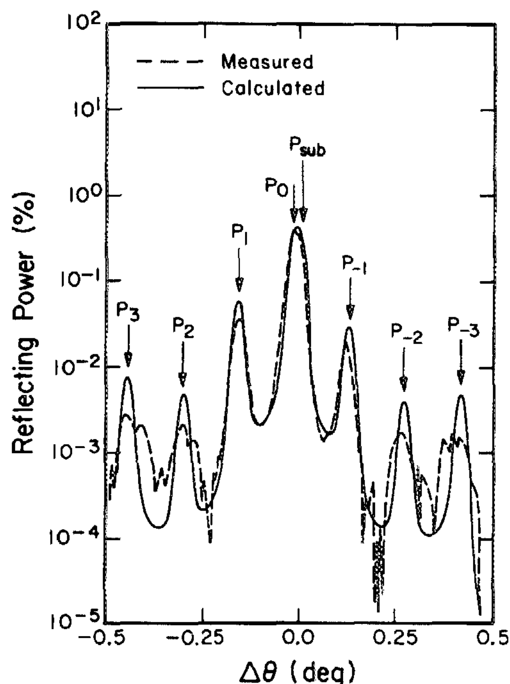

FIG. 2. Measured (dashed line) and calculated (solid line) x-ray rocking curves obtained from the SLS1 sample with Fe $K_{\alpha 1}$ (200) reflection.

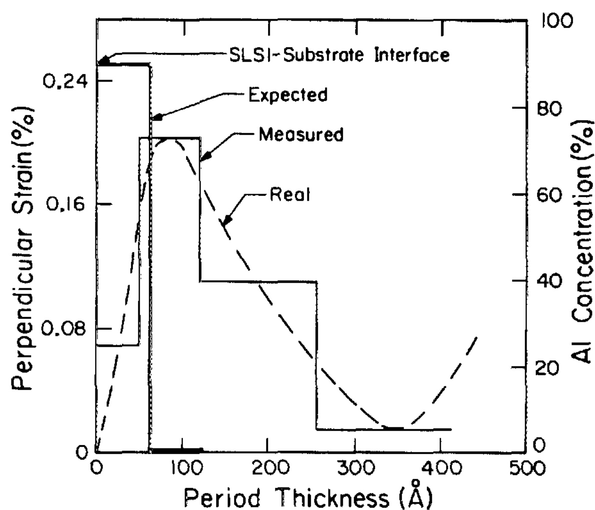

FIG. 3. Depth distributions of perpendicular strain and Al concentration in one period of the SLS1 sample. The expected distribution was estimated from growth rates determined by "α-step"-stylus-type measurements performed on ~2000-Å-thick layers. See text for the explanation of measured and real distributions.

growth of the $Al_xGa_{1-x}As$ layer is explained by a rapid injection of Al and Ga source gases into the reactor (see Table I). On the other hand, upon shutting off the gas supply, the residual gases in the reactor must be pumped out, which is slow and accounts for the extended tail in the Al composition and the strain curve. The nonzero strain beyond the tail suggests again that the growth of the pure GaAs layer was not achieved, as indicated by BS measurements (Fig. 1). Measurements conducted on the SLS2 samples (see Table I) revealed similar skewness in the strain (composition) distribution, confirming that the thicknesses of the transition regions were related to the reactor growth parameters.

In conclusion, the combined use of BS with channeling and x-ray rocking curves has provided detailed information about the depth distribution of composition, thickness, strain, crystal quality, and uniformity of $Al_xGa_{1-x}As$/GaAs SLS structures. Precise information such obtained proves useful in giving feedback in the controlled growth of these structures.

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