X-ray interference measurement of ultrathin semiconductor layers

C. R. Wie,[a] J. C. Chen,[b] H. M. Kim, P. L. Liu,[a] and Y.-W. Choi
State University of New York at Buffalo, Department of Electrical and Computer Engineering and Center for Electronic and Electro-optic Materials, Bonner Hall, Amherst, New York 14260

D. M. Hwang
Bell Communication Research, Redbank, New Jersey 07701

(Received 26 May 1989; accepted for publication 14 August 1989)

We have characterized, using the interference structure in x-ray rocking curves, a single or double strained GaInP layer grown on an InP substrate. The measured GaInP layer thicknesses are 9 ± 3 Å and 107 ± 3 Å for the single strained layer samples and 7 Å/50 Å and 32 Å/32 Å for the double strained layer samples. The rocking curve results for the 107 Å single-barrier sample and the 7 Å/50 Å double-barrier sample agreed well with the cross-section transmission electron microscopy data and the secondary-ion mass spectrometry data. The x-ray interference structure for the single strained barrier samples indicates the existence of many half-monolayer steps within the 1×1 mm^2 x-ray beam spot at each GaInP/InP interface.

X-ray interference structure in rocking curves is very useful in characterizing strained quantum well and quantum barrier layers.[1] Common use of the x-ray rocking curve (XRC) technique involves measuring the separation of Bragg peaks[2] or fitting the experimental curves with a calculated curve using either the dynamical[3] or the kinematical[4] diffraction theory. The lattice mismatch and layer composition are easily obtained from the Bragg peak separation. However, this simple method fails when the layer is too thin.[5] The minimum thickness for application of the peak separation method was found to depend only on the lattice mismatch, independent of the substrate material, x-ray wavelength, and diffraction angle.[6] Since the rocking curve profile for a heteroepitaxial multilayer sample is rather complicated, the understanding of a few characteristics of the rocking curve can make the layer parameter determination more easy and accurate. Using a dynamical theory simulation and a kinematical diffraction modeling, it was shown in a previous paper[1] that the x-ray interference phenomenon in rocking curves can be used to determine the active layer thickness in a strained quantum well laser structure with a submonolayer resolution. In this letter, we present experimental data and use the diffraction fringe and interference fringe structures to determine the layer thicknesses for samples containing a single or double buried pseudomorphic strained layer.

Samples containing one or two ultrathin strained layer(s) were grown on an Fe-doped semi-insulating (SI) or an n^+-type InP(001) substrate in an atmospheric-pressure metalorganic chemical vapor deposition (MOCVD) system. The single strained layer samples (262 and 264) were grown on a chemically polished SI InP substrate in order of a 2000–4000 Å InP buffer layer, a 10–150 Å GaInP pseudomorphic strained layer, and a 3000–5000 Å InP cap layer. For the double strained layer samples (263 and 303), the epitaxial layers were composed of an InP buffer layer, a GaInP layer, an InP layer, a GaInP layer, and an InP cap layer. The growth technique has been described elsewhere.[7] Figure 1 shows the schematic sample structures. Table I shows the layer thickness and layer composition for each sample. The thickness values were determined from the x-ray rocking curves and were verified from the cross-section transmission electron micrographs (XTEM) and secondary-ion mass spectrometry (SIMS). XRCs were measured using a double-crystal diffractometer equipped with a conventional-anode Fe $K\alpha$ x-ray tube. The Fe $K\alpha_2$ line was removed by placing a slit in the beam path after reflection from the GaAs(004) first crystal. Through this slit, the Fe $K\alpha_1$ beam of $\sim 1 \times 1$ mm^2 cross section was incident on the sample. Rocking curves were analyzed using a personal computer program developed based on a dynamical diffraction theory.[3]

We now discuss the single strained layer data. Figure 2 shows the experimental (dotted) and theoretical (solid) 004 rocking curves for the single strained layer samples [also see Fig. 1(a)]. The main peak in the middle is the InP 004 Bragg peak. Secondary peaks marked by *D* are the diffraction fringes of the InP cap layer. Since the diffraction fringe period is dependent only on the layer thickness but not on the composition,[8,9] the fringe peak spacing determines the InP cap layer thickness. The structure on the shoulder of the main peak, indicated by *I*, is an interference fringe structure which is formed by the interference between the wave dif-

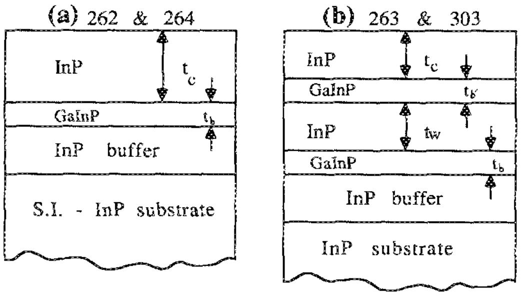

FIG. 1. Schematic sample structure: (a) Single strained layer, (b) double strained layer.

---

[a] National Science Foundation Presidential Young Investigator.

[b] Present address: Varian Research Center, Palo Alto, CA 94303.

TABLE I. X-ray analysis results for the strained GaInP barrier samples. The sixth column is the x value in $Ga_xIn_{1-x}P$.

Sample No.	t_b (Å)	t_w (Å)	$t_{b'}$ (Å)	t_c (Å)	x	Substrate
262	107	0	0	3100	0.2	SI InP
264	9	0	0	4700	0.2	SI InP
263	50	not determined	7	5000	0.2	SI InP
303	32	not determined	32	4000	0.24	n^+-InP

fracted by the InP cap layer and the wave diffracted by the InP buffer and substrate. The interference arises due to a finite phase difference between the two x-ray waves.[1] The phase difference is proportional to the product of the sandwiched GaInP layer thickness and the lattice mismatch of the sandwiched layer with the layers which produce the Bragg peak (InP in this case).[1] The interference fringe position is sensitive to a submonolayer thickness variation, as shown in Fig. 2. The XTEM and SIMS results for the sample 263, shown in Fig. 3(a), agree well with the x-ray rocking curve results for the thicknesses of the InP cap layer (from the diffraction fringe spacing) and the strained GaInP layer thickness (from the interference fringe position with an assumed phosphorus composition of 0.2). In Fig. 2, comparing the theoretical and experimental rocking curves in the region around the interference fringe (indicated by I), the experimental curves are smoother and will be better fitted if a weighted average is used for the theoretical curves. A weighted average[12] is shown in Fig. 2(a). The averaged curve (with an average t_b of 107 Å) is clearly a better fit than the $t_b = 107$ Å curve in the interference fringe region. In Fig. 2(b), the smoother experimental interference fringe probably indicates many half-monolayer steps (with a step height of about 3 Å) at both heterointerfaces within the approximately 1×1 mm^2 x-ray beam spot.

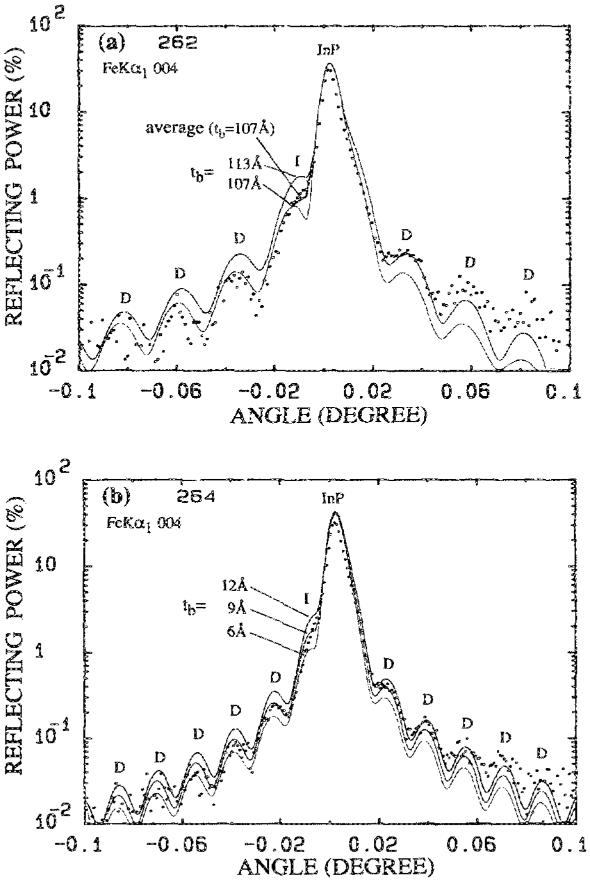

FIG. 2. 004 rocking curves for the single strained barrier samples: (a) 262 and (b) 264 [see Fig. 1 (a)]. Experimental (dotted) and theoretical (solid) curves are shown. Different theoretical curves correspond to different thicknesses for the strained GaInP layer with all other parameters kept the same as given in Table I.

We now discuss the double strained layer data. Figure 4 shows the experimental rocking curve and the best fit theoretical rocking curve [also see Fig. 1(b)]. Sample 263 has two strained GaInP layers which are very different in thickness (7 and 50 Å, see Table I). Sample 303 is a resonant tunneling diode structure with a double GaInP barrier and a single InP well structure. The two GaInP layers have the same thickness of 32 Å. For the rocking curves in Fig. 4, the thickness of the InP cap layer [see Fig. 1(b)] determines the diffraction fringe period which appears around the InP Bragg peak. Lattice mismatch and thickness of the two strained GaInP layers determine the position of the interference fringe structure within the InP Bragg peak, as indicated by an arrow in the figure. For the sample 263, the XRC-determined layer thicknesses of the InP cap layer and the GaInP layers are in good agreement with the SIMS result which is shown in Fig. 3(b).

We now discuss some aspects of this interference method. The position of the interference fringe structure within the InP Bragg peak is insensitive to the middle InP layer thickness for the sample structure shown in Fig. 1(b). This is because the middle InP layer has a zero lattice mismatch with the top and bottom InP layers and therefore, this layer produces no additional phase difference between the two x-ray waves diffracted by the top and bottom InP. The same rocking curve is obtained with 2500 Å or with 0 Å for the middle InP layer thickness of the sample 263, which is shown as a solid curve in Fig. 4(a). The SIMS data indicate that the middle InP layer thickness is close to 2500 Å for the sample 263. Another important point for the sample structure shown in Fig. 1(b) is that, if the sum of mismatch times thickness products is kept the same, the interference kink position is insensitive to how the total GaInP strained layer is divided into the two layers. Therefore, for a multiple ultrathin strained layer sample, the interference structure in the rocking curve will determine the sum of mismatch times thickness products for all the strained layers.

$$\sum_i (\text{mismatch})_i \times (\text{thickness})_i = \text{determined by interference}, \qquad (1)$$

where the summation is over the layers in between the cladding layers which produce the Bragg peak within which the interference structure is formed, and the mismatch is between these layers and the cladding layer.

In summary, the x-ray interference structure in rocking curves for samples containing ultrathin buried strained lay-

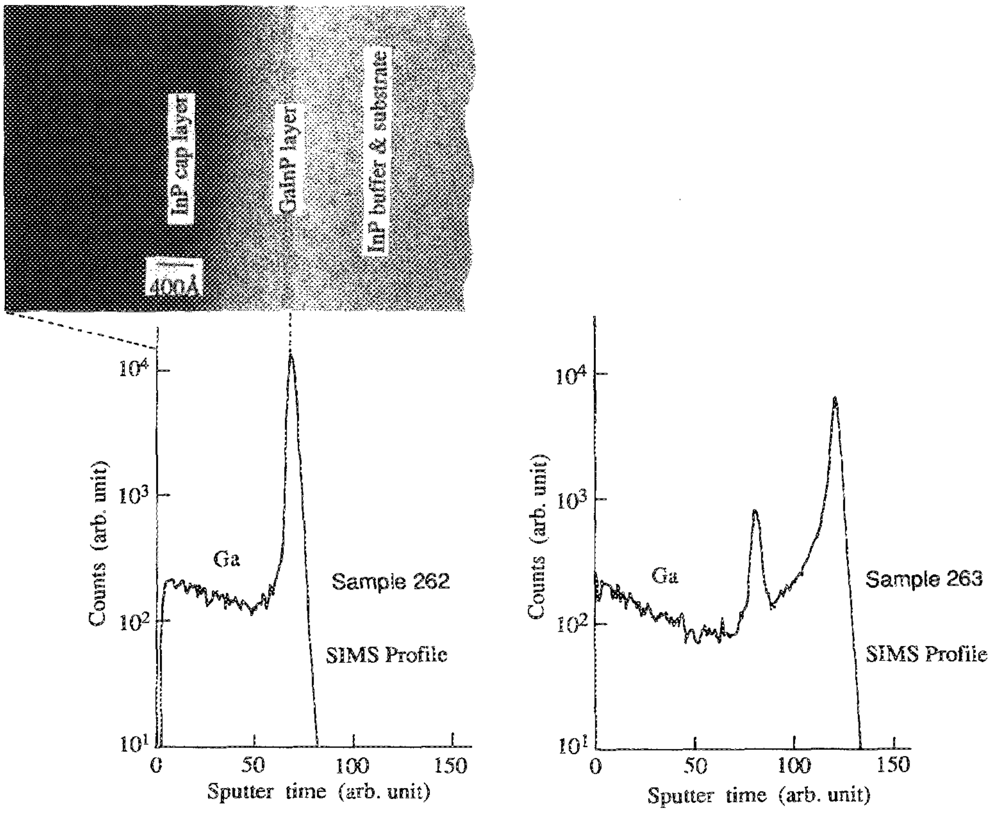

FIG. 3. XTEM and SIMS data for samples 262 and 263.

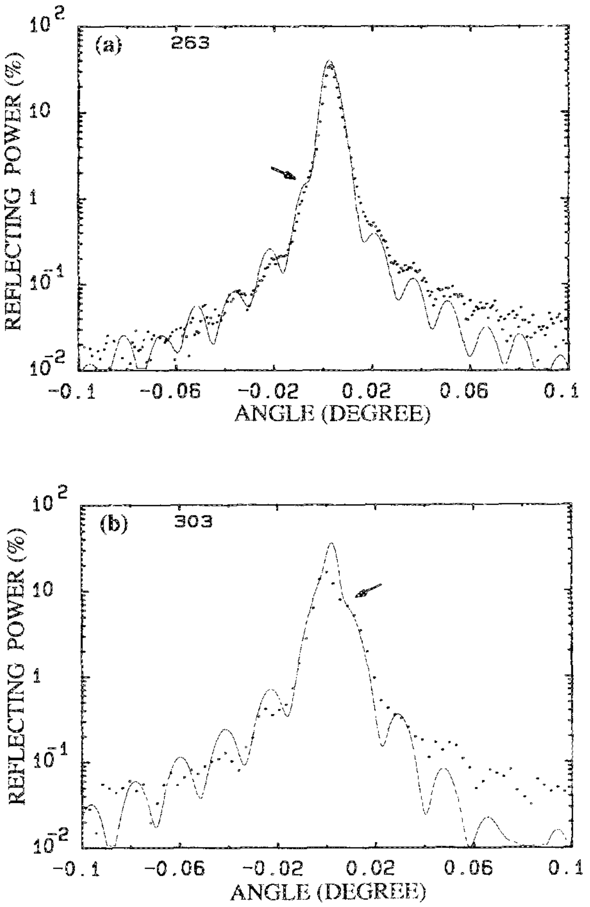

FIG. 4. 004 rocking curves for the double strained barrier samples: (a) 263 and (b) 303 [see Fig. 1 (b)]. The best fit theoretical curve is shown as a solid curve.

ers is a useful nondestructive tool for structural determination. For a single buried strained layer, the x-ray interference can determine the layer thickness with a submonolayer resolution. For a resonant tunneling diode with a double strained barrier and a single strained well, the x-ray interference can determine an additional structural parameter which can be used in combination with other nondestructive measurement techniques such as the photoluminescence and photoreflectance measurements. The interference method is useful for determining the active layer thickness in the double heterojunction laser structure.[1,9-11]

The authors would like to acknowledge Dr. S. A. Schwarz for the SIMS measurement. This work was supported in part by the National Science Foundation and the Eastman Kodak Company.

[1] C. R. Wie, J. Appl. Phys. **65**, 1036 (1989).

[2] C. R. Wie, H. M. Kim, and K. M. Lau, SPIE Proc. **877**, 41 (1988).

[3] C. R. Wie, T. A. Tombrello, and T. Vreeland, Jr., J. Appl. Phys. **59**, 3743 (1986).

[4] V. S. Speriosu, J. Appl. Phys. **52**, 6094 (1981).

[5] P. F. Fewster and C. J. Curling, J. Appl. Phys. **62**, 4154 (1987).

[6] C. R. Wie, J. Appl. Phys. **65**, 985 (1989).

[7] J. C. Chen, W. K. Chen, P. L. Liu, J. Maloney, and O. T. Beachley, SPIE Proc. **877**, 21 (1988).

[8] W. J. Bartels and W. Nijman, J. Cryst. Growth. **44**, 518 (1978).

[9] For various rocking curve methods and diffraction theories, see, for example, C. R. Wie (unpublished).

[10] X. Chu and B. K. Tanner, Appl. Phys. Lett. **49**, 1733 (1986).

[11] G. T. Baumbach, H. Rhan, and U. Pietsch, Phys. Status Solidi A **109**, K7 (1988).

[12] The averaged curve in Fig. 2(a) is $R(\text{average}) = 0.273R(107\ \text{Å}) + 0.216[R(104\ \text{Å}) + R(110\ \text{Å})] + 0.108[R(101\ \text{Å}) + R(113\ \text{Å})] + 0.039[R(98\ \text{Å}) + R(116\ \text{Å})]$. Here, the coefficient is the weight factor calculated from a Gaussian distribution of the 3 Å steps with $\sigma = 4.4$ Å, and R is the reflecting power at each t_n.