X-RAY REFLECTIVITY OF InAs/GaAs HETEROSTRUCTURES WITH SURFACE AND INTERFACIAL ROUGHNESS

S. Woronick, Bin Yang, A. Krol, Y. Kao, H. Munekata, L. Chang, J. Phillips

► To cite this version:

S. Woronick, Bin Yang, A. Krol, Y. Kao, H. Munekata, et al.. X-RAY REFLECTIVITY OF InAs/GaAs HETEROSTRUCTURES WITH SURFACE AND INTERFACIAL ROUGHNESS. Journal de Physique Colloques, 1987, 48 (C5), pp.C5-51-C5-56. 10.1051/jphyscol:1987508 . jpa-00226679

HAL Id: jpa-00226679

<https://hal.science/jpa-00226679>

Submitted on 1 Jan 1987

HAL is a multi-disciplinary open access archive for the deposit and dissemination of scientific research documents, whether they are published or not. The documents may come from teaching and research institutions in France or abroad, or from public or private research centers.

L'archive ouverte pluridisciplinaire **HAL**, est destinée au dépôt et à la diffusion de documents scientifiques de niveau recherche, publiés ou non, émanant des établissements d'enseignement et de recherche français ou étrangers, des laboratoires publics ou privés.

X-RAY REFLECTIVITY OF InAs/GaAs HETEROSTRUCTURES WITH SURFACE AND INTERFACIAL ROUGHNESS

S.C. WORONICK, B.X. YANG, A. KROL, Y.H. KAO, H. MUNEKATA*,
L.L. CHANG* and J.C. PHILLIPS**

*Department of Physics, State University of New York at Stony
Brook, Stony Brook, NY 11794, U.S.A.*

**IBM Thomas J. Watson Research Center, P.O. Box 218, Yorktown
Heights, NY 10598, U.S.A.*

***Department of Chemistry, State University of New York at
Buffalo, Buffalo, NY 14214, U.S.A.*

The reflection of monochromatic x-rays by heterostructures shows regular oscillations in the reflectivity as a function of the grazing angle. These oscillations contain information about the layer thickness and interfacial roughness. Our results indicate that an overgrowth of an InAs layer on GaAs by molecular beam epitaxy has started with a rough surface and ended with a smooth finish. We thus demonstrate a practical technique for nondestructive evaluation of the interfacial roughness in semiconductor heterostructures.

Many important physical properties of quantum-well structures are strongly related to the material structure at the boundary between different layers. Yet, little is known at the present time about the interfacial roughness. The understanding and control of the interface appears to be essential for further development and application of the superlattices and heterostructures, but very few techniques are capable of obtaining direct information on the structural quality of the interface, especially for characterization of interfaces beneath the top surface of as-made samples. The purpose of this paper is two-fold. First, it points out that the root-mean-square roughness of the interface, defined in the long-wavelength regime of a scalar scattering theory^{1,2}, can be determined by measuring the oscillations in the x-ray reflectivity. Second, recent data obtained at a variety of x-ray wavelengths with InAs/GaAs heterostructures grown by molecular beam epitaxy indicate that the oscillations found in the reflectivity are in reasonable agreement with our model. Two roughness parameters, characteristic of the top surface and the sub-surface interface, have been determined. This experimental method can be employed as a practical technique for probing other multilayer systems.

Consider a thin layer of material (InAs) deposited on a thick substrate (GaAs). If the interfaces are perfectly flat, specular x-ray reflection by this heterostructure can be described by the well-known Fresnel equations.³ Following a recursion formula derived by Parratt⁴ which is valid for thin layers, the x-ray reflectivity as a function of the grazing angle θ , $R(\theta) = |R_{12}|^2$ can be obtained from the following expression:

$$R_{12} = \frac{F_{12} + F_{23}e^{-i\gamma}}{1 + F_{12}F_{23}e^{-i\gamma}}, \quad (1)$$

where F_{ij} is the Fresnel coefficient generally defined for the reflection amplitude between the i th and j th semi-infinite media. For the case of σ -polarization corresponding to the present experiment, we have $F_{ij} = (p_i - p_j)/(p_i + p_j)$ with $p_j = n_j \sin \theta_j$, and where $n_j = 1 - \delta_j - i\beta_j$ is the refractive index of the j th medium. The factor $\exp(-i\gamma)$ accounts for the interference and attenuation due to the presence of the thin layer (InAs) of thickness d , with $\gamma = 4\pi d p_2 / \lambda$, and where λ is the x-ray wavelength. Writing $p_2 = A - iB$, where

$$\begin{aligned} A &= \frac{1}{\sqrt{2}} [\sqrt{(\theta^2 - 2\delta_2)^2 + 4\beta_2^2} + (\theta^2 - 2\delta_2)]^{1/2}, \\ B &= \frac{1}{\sqrt{2}} [\sqrt{(\theta^2 - 2\delta_2)^2 + 4\beta_2^2} - (\theta^2 - 2\delta_2)]^{1/2}, \end{aligned} \quad (2)$$

the variation of A and B as a function of θ gives rise to the observed oscillations and attenuation in $R(\theta)$.

The effect of random interfacial roughness causes scattering in different directions, thus it appears as a Debye-Waller factor in the expression for specular reflection. In our model, we assume that the roughness of each interface is uncorrelated because the x-ray beam covers a large in-plane area ($\sim 1 \text{ cm}^2$) at grazing angles. We also assume that the deviation of the interface from its average ideal plane is described by a normal distribution, and introduce two parameters σ_1 and σ_2 on the basis of scalar scattering theory under the condition that $\sigma_j \sin \theta_j \ll \lambda$. Here σ_1 and σ_2 represent the root-mean-square roughness at the air- or vacuum-InAs and InAs-GaAs interfaces, respectively. Under these assumptions, the Fresnel coefficients F_{ij} in (1)

should be replaced by F'_{ij} to account for the random roughness viz.

$$\begin{aligned} F'_{12} &= F_{12} e^{-\frac{1}{2} \left(\frac{4\pi}{\lambda} \sigma_1 \sin \theta_1 \right)^2}, \\ F'_{23} &= F_{23} e^{-\frac{1}{2} \left(\frac{4\pi}{\lambda} \sigma_2 \sin \theta_2 \right)^2}, \end{aligned} \quad (3)$$

Although x-ray reflection from single surfaces with random roughness has been extensively studied in the past,⁵⁻⁸ we are not aware of any quantitative analysis of the sub-surface interfacial roughness based on Eq. (3).

We have carried out numerical calculations to investigate the effects of roughness according to Eq. (3). It was found that the influence of σ_1 is most pronounced in the neighborhood of $\theta \approx \theta_c$, where $\theta_c \approx (2\delta)^{1/2}$ is the critical angle. On the other hand, the effect of σ_2 is most important in the region $\theta > \theta_c$ where the oscillations in $R(\theta)$ are present. This result is physically clear because the x-rays are totally reflected at small grazing angles $\theta < \theta_c$. It also indicates that the values of the two roughness parameters σ_1 and σ_2 as determined by a curve-fitting process in the present case are only weakly coupled. Consequently, each roughness parameter can be determined almost independently with reasonably high accuracy.

The experiment was performed at the National Synchrotron Light Source (NSLS) using the U-15 beamline for the longer wavelengths and the X-21 beamline for the shorter wavelengths. The U-15 beamline uses a toroidal-grating monochromator (TGM) in the 300-1000 eV range. Near the oxygen K edge, the TGM's calibration is good to about 0.05Å and stable to well within 0.02Å during a typical spectral scan; the wavelength resolution $\Delta\lambda/\lambda$ was about 0.004. The X-21 beamline used a Si(111) double-crystal monochromator, followed by a 1:1 gold-coated, doubly-focusing, bent toroidal quartz mirror, plus a Huber four-circle diffractometer for angular manipulation of the sample. A schematic diagram is shown in Fig. 1.

The two samples studied in the present work were InAs/GaAs heterostructures made by an overgrowth of an InAs thin layer on a GaAs (100) surface at 420°C with an intermediate GaAs buffer layer using molecular beam

epitaxy. The nominal thickness of the InAs thin layer was approximately 250Å for each sample, estimated from the deposition time. In each case, the GaAs (100) substrate was less than 0.5° off. The sample R921 was grown under In-stabilized conditions with a flux ratio of $As_4/In = 2.5$, while sample R922 was grown under As-stabilized conditions with a flux ratio of $As_4/In = 5.3$. Optical microscopy revealed that R921 had In droplets on its surface, while R922 showed no In droplets but had an oval defect on its surface.

Typical experimental curves of $R(\theta)$ are shown in Fig. 2 along with theoretical calculations obtained from Eq.(1) and (3). It can be seen that the model used here seems to fit the experimental data very well. The fit of the theory to the data was achieved by using simplex minimization to adjust certain parameters in the theory so as to minimize the root-mean-square difference between the function $\log(R(\theta))$ calculated from the data and the same quantity calculated from the theory, essentially a least-squares fit. Known parameters, such as the x-ray wavelength, offset angle, and normalization were held fixed, while parameters such as σ_1 , σ_2 , and d were adjusted. The fits also provided an estimate of the accuracy with which these quantities were determined: $\lesssim 1\text{\AA}$ for σ_1 and σ_2 ; $\lesssim 5\text{\AA}$ for d .

Results of our determination of d , σ_1 , and σ_2 from the $R(\theta)$ curves are summarized in Table 1. As the table indicates, there is good agreement between the values of d , σ_1 , and σ_2 found for sample R921 using both long and short wavelengths. Table 1 also indicates that sample R922 (As-stabilized growth) has greater interfacial roughness than sample R921 (In-stabilized growth). Indeed, this difference can also be observed in the $R(\theta)$ curves shown in Fig. 2; the oscillations decay faster with angle for R922 than for R921.

The determination of the interfacial roughness by a nondestructive technique should provide useful information on the growth of epilayers. The large difference between σ_1 and σ_2 found in the present work for both In- and As-stabilized growth offers experimental support to the notion that an overgrowth of the epilayer has started from a rough surface and ended with a smooth finish. Using this method, it now appears possible to investigate the changes in the surface condition as a function of the layer thickness and to

correlate it with the substrate roughness. Other effects such as inverted heterostructures and interrupted growth can also be studied.

In conclusion, it has been demonstrated that x-ray reflectivity of an InAs/GaAs heterostructure exhibits strong oscillations as a function of the grazing angle due to interference effects. The experimental data are in good agreement with theoretical calculations using a model based on a combination of the Fresnel equations and a scalar scattering theory in the long wavelength regime. This result provides evidence for the applicability of a technique which can be useful in nondestructive evaluation of subsurface interfacial roughness in heterostructures.

We would like to thank the U-15 and X-21 staff for the use of the beamlines. The present work is supported by the Office of Naval Research. The NSLS is supported by the Department of Energy under contract No. DE-AC02-76-CH00016.

References

1. P. Beckmann and A. Spizzichino, The Scattering of Electromagnetic Waves from Rough Surfaces (Pergamon Press, New York, 1963)
2. C. K. Carniglia, Opt. Eng. 18, 104 (1979); and references cited therein.
3. See for example, A. H. Compton and S. K. Allison, X-rays in Theory and Experiment (D. Van Nostrand Co., New York, 1935).
4. L. G. Parratt, Phys. Rev. 95, 359 (1954).
5. For a recent review, see T.W. Barbee, Jr., Opt. Eng. 25, 895 (1986); E. Spiller and A.E. Rosenbluth, Opt. Eng. 25, 954 (1986).
6. H. Kiessig, Annalen der Physik 10, 769 (1931).
7. D.H. Bilderback, SPIE 315, 90 (1981).
8. A. Braslau, M. Deutsch, P.S. Pershan, A.H. Weiss, J. Als-Nielsen, and J. Bohr, Phys. Rev. Lett. 54, 114 (1985); and references therein

TABLE 1. Summary of results obtained from least-squares curve-fitting of Eqs. (1) and (3) to the reflectivity R as a function of the glancing angle θ . The long-wavelength result is a summary for measurements at seven different wavelengths in the range indicated.

Sample	σ_1 (Å)	σ_2 (Å)	d (Å)	Wavelength(Å)	Growth
R921	< 1	17.9	248	25.56 - 31.00	In-stabilized
R921	< 1	17.5	258	1.196	" "
R922	< 1	19.5	254	1.196	As-stabilized

Fig. 1. -- Schematic of Experiment. A: Traveling slit. B: Sample (mounted on an angular manipulator). C: Beam Stop. I_0 and I : Detectors for measuring x-ray intensity before and after reflection.

Fig. 2. -- Normalized Reflectivity as a Function of θ at $\lambda = 1.196$ Å.
 (a) Sample R922 grown under As-stabilized conditions on GaAs(100) less than 0.5° off.
 (b) Sample R921 grown under In-stabilized conditions on GaAs(100) less than 0.5° off. (Curve is displaced by a factor of 6.3 from curve (a))