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Darhuber, A.A.; Bauer, G.; Wang, P.D.; Sotomayor Torres, C.M.

Published in:
Journal of Applied Physics

DOI:
10.1063/1.366709

Published: 01/01/1998

Document Version
Publisher's PDF, also known as Version of Record (includes final page, issue and volume numbers)

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Download date: 01. Nov. 2018
Shear strains in dry etched GaAs/AlAs wires studied by high resolution x-ray reciprocal space mapping

A. A. Darhuber and G. Bauer
Institute for Semiconductor Physics, University of Linz, Altenbergerstr. 69, A-4040 Linz, Austria

P. D. Wang and C. M. Sotomayor Torres
Department of Electronics and Electrical Engineering, Nanoelectronics Research Center, University of Glasgow, G128LT Glasgow, United Kingdom

(Received 16 July 1997; accepted for publication 30 September 1997)

We have fabricated GaAs/AlAs quantum wires and quantum dots by means of molecular beam epitaxy, electron beam lithography, and subsequent reactive ion etching using SiCl$_4$ and O$_2$. The nominal periods are 300 nm and 350 nm for both wire and dot samples. High resolution x-ray reciprocal space maps of the 350 nm samples exhibit not only satellites corresponding to a periodicity of 350 nm but also additional satellites corresponding to a period of three times 350 nm, whereas there are no such extra peaks in the maps of the 300 nm samples. These secondary satellites are shown to be associated with a discretization effect in electron beam writing. Moreover, we found, that the shear strain in the wires has a distinct influence on the intensities of these weak extra satellites. Hence, they provide a sensitive means for the assessment of shear strains in elastically relaxed quantum wires. © 1998 American Institute of Physics. [S0021-8979(98)07101-1]

I. INTRODUCTION

In polar crystals like most of the III–V and II–VI semiconductors, shear strains induce a piezoelectric field, which modifies locally the electronic and optical properties. Since shear strain distributions often average to zero over the whole quantum structure, they are difficult to determine. We will demonstrate, that shear strains distinctly influence extra x-ray diffraction peaks, which are due to a weak superperiodicity in the wire/dot arrays. Hence, they serve as a sensitive means for the determination of a shear strain distribution in semiconductor nanostructures.

Electron beam lithography (EBL) is an indispensable tool not only for the industrial development and production of semiconductor devices but also for the preparation of nanostructured quantum systems (quantum wires and quantum dots). Research laboratories are exploring the sub-10 nm—regime. With the exception of projection lithographic systems, patterns are written through a series of pixel exposures to delineate the desired pattern. In this article we make use of the effect of an incompatibility between pattern dimensions and the spacing of the exposure pixels.

II. SAMPLE PREPARATION

The GaAs/AlAs quantum wires and quantum dots have been fabricated by nanostructuring a 30 period AlAs/GaAs multiquantum well (MQW) deposited on a 1 µm GaAs buffer. The 8.2-nm-thick GaAs wells are separated by 13.1 nm AlAs barriers resulting in a total thickness of 639 nm. The MQW was capped by a 20 nm GaAs layer. The whole structure was grown on a [001]-oriented GaAs substrate on top of which 25 periods of a 5 ML/5 ML short period GaAs/AlAs superlattice have been deposited.

The lithography was done with a Leica Cambridge EBPG5-HR electron beam pattern generator using a pixel size of 30 nm. The samples have been prepared by magnetically confined plasma reactive ion etching (MCP RIE), using TiAu metal masks for the wire and NiCr for the dot samples. The flow rates of the etching gases SiCl$_4$ and O$_2$ were 13.5 and 1.5 sccm, respectively. The operating pressure was 0.5 mTorr, the microwave power was 54 W, the rf power 35 W and the resulting dc bias was ~230 V. Scanning electron micrographs (SEMs) revealed vertical sidewalls of both wires and dots.

The etch depth was ~900 nm, consequently about $H_2=250$ nm of the GaAs buffer have been structured into dots as well. The TiAu mask did not totally withstand the etching procedure and hence, the top part of the wires has been etched as well. The short period superlattice below the GaAs buffer was not etched and will not be considered further. The wires and dots with periods of 300 and 350 nm are oriented along the [110] direction. We have prepared the dot samples with a rectangular symmetry, i.e., in the [110] direction the dot period $d$ was nominally 300 nm, in the perpendicular direction 350 nm, which is no integer multiple of the pixel spacing (30 nm). The total structured areas vary between 8 and 26 mm$^2$. Such comparatively large areas are necessary for an acceptable signal-to-noise ratio, if the x-ray experiments are performed with standard laboratory equipment. If a more powerful source of x-rays is available (like synchrotron radiation) the patterned area can be reduced to about one square millimeter.

III. EXPERIMENT

We used a Philips MRD diffractometer with a four crystal Ge(220) monochromator and a channel-cut two crystal Ge(220) analyzer with a horizontal and vertical divergence.
of 12 arc s and \(-1.65^\circ\), respectively, and a wavelength dispersion of \(\Delta\lambda/\lambda \approx 1.1 \times 10^{-4}\). A conventional fixed copper anode with a power consumption of 1.6 kW and Cu K\(\alpha_1\)-radiation (\(\lambda = 1.54 \ \text{Å}\)) was used. The divergence of 12 arc s corresponds to a lateral coherence length in the order of 5 \(\mu\text{m}\) at (004), which is sufficient to investigate the spatial phase coherence of periodic sub-0.5 \(\mu\text{m}\)-structures.

The independent variation of the two diffraction angles \(\omega\) (between incident x-rays and sample surface) and 2\(\theta\) (between incident and scattered x-rays) provides the possibility of reciprocal space mapping, i.e., the acquisition of two-dimensional projections in the three-dimensional reciprocal space.6,7

The lateral macroperiodicity of the wire and dot arrays gives rise to lateral intensity maxima (wire satellites \(W_i\) and dot satellites \(D_i\)) in the diffraction pattern. In principle, the full information about the geometrical shape (height, width, inclination of the sidewalls, period) as well as about the structural quality (strain and crystalline damage) can be obtained from a two-dimensional map of reciprocal space.8–11

Figure 1(a) shows a reciprocal space map around the (004) reciprocal lattice point (RLP) of an unstructured (as grown) GaAs/AlAs reference sample from the same wafer. ‘‘S’’ denotes the GaAs substrate peak, ‘‘SL\(_0\)’’ and ‘‘SL\(_1\)’’ the zero and first-order MQW peak, respectively. ‘‘A’’ is a symbol for an artefact, the so-called analyzer streak. RLPs with high intensity, in the present case the substrate reflection \(S\) and the SL\(_0\) peak, have slowly decaying tails along the Ewald sphere intersecting the growth direction with the Bragg angle \(\theta_B\). Thickness fringes in-between the MQW peaks SL\(_0\) and SL\(_1\) indicate the excellent crystalline quality of the system. Their spacing (see Fig. 1) corresponds to the total thickness of the superlattice of \(\sim 640 \ \text{nm}\).

In Fig. 1(b), the diffraction pattern of the 300 nm periodic wire array is shown. Wire satellites accompanying the SL\(_0\) peak and the first-order MQW-peak SL\(_1\) are observed. The wire period determined from the spacing of the satellites along the \(q_z\)-direction is 303 nm in excellent agreement with the nominal value. The inset in Fig. 1(b) defines the diffraction geometry, the arrow is the normal to the diffraction plane, which is defined by the incident and diffracted (004) x-ray wave vectors.

For comparison, we have also plotted isointensity contours of the 350 nm periodic wire sample in Fig. 2(a). In-between the ordinary wire satellites labelled \(W_i\), additional satellites are visible in Fig. 2(b), where part of the region of Fig. 2(a) has been measured with a smaller stepwidth and longer detector integration times. Their spacing corresponds to a periodicity of 1.05 \(\mu\text{m}\), i.e., three times the nominal wire period. We have also prepared another sample of quantum dots with a period of 300 nm in the [110]-direction and 350 nm in the perpendicular direction. Figure 3(a) shows the reciprocal space map of this dot array with rectangular symmetry for the orientation, where the 300 nm period is in diffraction. Narrow satellites appear plus a broad peak of
diffusely scattered intensity. The latter allows to quantify the influence of defects introduced in the remaining crystal lattice by the etching process. When the sample is rotated by 90°, the 350 nm periodicity comes into diffraction. The corresponding reciprocal space map can be seen in Fig. 3(b). As in the case of the 350 nm wire sample, extra-peaks between the dot satellites appear which again reflect a period of 1.05 μm. For a further improvement of the signal-to-noise ratio, we have also plotted the integrated intensity of the lower part of the reciprocal space maps in Fig. 3(c).

IV. ELECTRON BEAM LITHOGRAPHY

As explained above, the x-ray measurements indicate the occurrence of a second “super”-period in the 350 nm samples of three times the nominal period, which is due to a discretization effect of the EBL system. Prior to every lithography process a figure called pixel size has to be specified, which is the distance between two neighboring exposure points. If the pattern to be exposed is inconsistent with this pixel spacing, that means, if the pattern contains dimensions, which are no integer multiples of the pixel spacing, a discretization effect will occur [see the sketch in Fig. 4(a)]. The lateral coordinates of the desired quantum wires/dot positions are rounded up or down to the nearest multiple of the pixel spacing. The pixel spacing has been chosen as 30 nm and the period of the quantum wires/dots is 350 nm with a wire/dot diameter of 175 nm. The width remains constantly 180 nm but the pitch between two wires/dots is periodically varying between 150 and 180 nm. The superperiodicity of three times 350 nm, hence stems from the periodic variation of the 150 and 180 nm pitches.

In order to check our propositions, we have recorded scanning electron micrographs of the 350 nm wire array [Fig. 4(b)]. The periodically varying pitch between two adjacent wires is clearly visible. Every third pitch is smaller than the following two, whereas the width of the wires is constant as it has been sketched in Fig. 4(a).

Scanning electron microscopy (SEM) has the disadvantage, that it is detrimental to the electrical and optical prop-
properties of nanostructured semiconductor layers, because the high energy electrons (typically 25 keV) of a SEM induce defects near the surface.\textsuperscript{12,13} Moreover, a high probability for a hydrocarbon contamination of the surface exists, and carbon is known to act as an (unwanted) \textit{p}-type dopant for GaAs and related materials.

\textbf{V. X-RAY DIFFRACTION}

In order to extract information about the strain status, we have extended the kinematical diffraction model by Tolan \textit{et al.},\textsuperscript{12} which was developed for the description of unstrained periodic surface profiles of bulk crystals.

If a heteroepitaxial layer structure is etched, the lattice mismatch between the individual constituents causes an elastic relaxation in the vicinity of the sidewalls of the wires and dots. In the coordinate system parallel to the wires (the \textit{y}-direction is along the wires and the \textit{z}-direction is parallel to the growth direction), the strain tensor has the following structure:

\[ \varepsilon = \begin{pmatrix} \varepsilon_{xx} & 0 & \varepsilon_{xz} \\ 0 & 0 & 0 \\ \varepsilon_{xz} & 0 & \varepsilon_{zz} \end{pmatrix}. \]  

(1)

In the present case, the wires are oriented along the [1 \textit{1} 1 \textit{0}] direction (\textit{y}-direction) and the base vectors [100], [010], and [001] of the Bravais crystal lattice have the coordinates

\[ \mathbf{a}_0 = \frac{a_0}{\sqrt{2}} \begin{pmatrix} 1 \\ -1 \\ 0 \end{pmatrix}, \quad \mathbf{b}_0 = \frac{a_0}{\sqrt{2}} \begin{pmatrix} 1 \\ 0 \\ 1 \end{pmatrix}, \quad \mathbf{c}_0 = a_0 \begin{pmatrix} 0 \\ 0 \\ 1 \end{pmatrix}. \]  

(2)

in the wire coordinate system. \(a_0=5.653 \) Å is the (relaxed) GaAs lattice constant. If a unit cell is subjected to a strain field according to Eq. (1), the base vectors are elongated by

\[ \delta \mathbf{a} = \frac{a_0}{\sqrt{2}} \begin{pmatrix} \varepsilon_{xx} \\ 0 \\ \varepsilon_{xz} \end{pmatrix}, \quad \delta \mathbf{b} = \frac{a_0}{\sqrt{2}} \begin{pmatrix} \varepsilon_{xx} \\ 0 \\ \varepsilon_{xz} \end{pmatrix}, \quad \delta \mathbf{c} = a_0 \begin{pmatrix} \varepsilon_{zz} \\ 0 \\ \varepsilon_{zz} \end{pmatrix}. \]  

(3)

The volume of the unit cell \( V \) is thus changed to

\[ V = (\mathbf{a}_0 + \delta \mathbf{a}) \cdot (\mathbf{b}_0 + \delta \mathbf{b}) \cdot (\mathbf{c}_0 + \delta \mathbf{c}) = V_0 \left( (1 + \varepsilon_{xx})(1 + \varepsilon_{zz}) - \varepsilon_{xz}^2 \right), \]  

(4)

where \( V_0 = a_0^3 \) is the volume of the undistorted unit cell. In the case of small strain, all quadratic strain terms can be neglected and the familiar formula

\[ \frac{\Delta V}{V} = \varepsilon_{xx} + \varepsilon_{zz} = \text{trace}(\varepsilon) \]  

(5)

results. Consequently, the reciprocal lattice vectors \( \mathbf{u} \), \( \mathbf{v} \), and \( \mathbf{w} \) change as well, e.g.,

\[ \mathbf{u} = \frac{1}{V} (\mathbf{b} \times \mathbf{c}) = \frac{a_0^2}{V \sqrt{2}} \begin{pmatrix} 1 + \varepsilon_{zz} \\ -(1 + \varepsilon_{xx})(1 + \varepsilon_{zz}) + \varepsilon_{xz}^2 \\ -\varepsilon_{xz} \end{pmatrix}, \]  

(6)

where \( V \) is given by Eq. (4). Thus, the corresponding reciprocal lattice vector of a reflection (hkl) is shifted by

\[ \Delta G = -\frac{1}{a_0} \frac{1}{(1 + \varepsilon_{xx})(1 + \varepsilon_{zz}) - \varepsilon_{xz}^2} \left( \begin{array}{c} \frac{1}{\sqrt{2}} (h+k)(1 + \varepsilon_{zz})e_{xx} - e_{xx}^2 + l e_{xz} \\ 0 \\ \frac{1}{\sqrt{2}} (h+k)e_{xz} + l e_{xz} \end{array} \right) \]  

\[ \approx -\frac{1}{a_0} \left( \begin{array}{c} \frac{1}{\sqrt{2}} (h+k)e_{xx} + l e_{xz} \\ 0 \\ \frac{1}{\sqrt{2}} (h+k)e_{xz} + l e_{xz} \end{array} \right). \]  

(7)

The last approximation is valid for small strain (\( e^2 \ll 1 \)). Thus, for a symmetrical reflection (h=0=k), the shear strain only enters into \( \Delta G_y \).

In our case, 60 layers of AlAs and GaAs are present in the superlattice, thus we have 60 regions of positive and negative shear strain on each side of the wire. Since the TiAu mask was affected during the etching, we have assumed that 20 periods of totally 30 remained undamaged and we assumed a total height of 400 nm. In Fig. 5(a), we have plotted the normalized intensity distribution for an array of unstrained rectangular quantum wires with a height of 400 nm, a width of 170 nm and a lateral periodicity of 350 nm (integrated in \( q_z \) direction). A series of satellites evolves symmetrically around \( q_z = 0 \). Since the ratio of the wire width and the period is near 1/2, the even order satellites are weak. If the periodical variation of the pitch between the wires is taken into account, additional satellites occur [Fig. 5(b)]. The intensities of these extra peaks are between two and three orders of magnitude below the central peak. For comparison, we present the integrated intensity of the reciprocal space maps of Figs. 2(a) (full line) and 2(b) (dots) in Fig. 5(d). All curves are normalized to one. The most pronounced difference is that the intensities of the extra peaks is about one order of magnitude larger than in the simulation for an unstrained wire. We attribute this discrepancy to the presence of strain fields in the structure. The slight asymmetry of the curve is due to the integration of part of the analyzer streak, which runs across the left part of the wire satellites in Fig. 2.

As evident from Eq. (7), the lateral strain \( \varepsilon_{xx} \) does not affect the diffraction around (004). For asymmetric reflections (h,k \( \neq 0 \)), a lateral strain \( \varepsilon_{zz} \) induces a shift of the envelope of the wire satellites.\textsuperscript{11} The vertical strain \( \varepsilon_{zz} \) influences only the \( q_z \) direction for a rectangular wire, which is eliminated by integration in \( q_z \) direction to gain a higher signal-to-noise ratio in the measured curve. Hence, it is only the shear strain \( \varepsilon_{xz} \) which remains to be considered.

An analytical calculation of the elastic relaxation of dry etched quantum wires is involved and possible only for special geometries like rectangular profiles.\textsuperscript{15} Treacy and
Gibson have developed an analytical solution of the Airy stress equation using a Fourier series method and assuming equal elastic constants in the wells and barriers and the presence of thick caplayers. Faux and Haigh have used this method for the calculation of the stress and strain fields in etched single and multiple quantum wells. These calculations show, that the shear strain has its maximum at the interface between well and barrier material very close to the sidewall surface of the wires. If several interfaces are present, $\varepsilon_{xz}$ is zero in the center of the layers for reasons of symmetry. It has a significant strength only in a region close to the sidewall with an extension inside the structure of about twice the layer thickness. The maximum value is roughly equal to the lattice mismatch between well and barrier material, which is $1.2 \times 10^{-3}$ for AlAs on GaAs.

If the shear strain is averaged vertically over one period of the multiple quantum well or laterally over the total width, the net result is zero. Therefore, we have averaged the shear strain distribution in growth direction over one half of the MQW period, and laterally in regions of 20 nm width close to the sidewalls $|\varepsilon_{xz,ave}| = 1.75 \times 10^{-4}$ and the rest of the structure $|\varepsilon_{xz,ave}| = 0.175 \times 10^{-4}$. The resulting distribution is sketched in the inset of Fig. 5, where the corresponding diffraction pattern is plotted. In the inset, the position $x=0$ corresponds to the center of the wire and $x=85$ nm to position of the right sidewall.

It can be clearly seen, that the intensity of the extra peaks has increased by one order of magnitude relative to the intensity of the central peak. If the experimental background and some diffuse scattering [see e.g., Fig. 3(a)] is taken into account, it compares well with the experimental intensity ratio. Moreover, the intensity ratio of the pair of extra satellites between every pair of “ordinary” satellites is qualitatively correct in the simulation. The quantitative comparison of the entire distribution along $q_x$ with the experimental one is not perfect. This is attributed on one hand to the crude discretization of the shear strain distribution and on the other hand to the uncertainties in the wire structure, due to the degradation of the mask and the damage to the material underneath.

VI. CONCLUSIONS

We have demonstrated that the discretization of a pattern, which is to be transferred onto a semiconductor layer by electron beam lithography, potentially leads to superperiodicities, which manifest themselves in additional satellite peaks in high resolution x-ray diffraction measurements. Periodically occurring pitch variations as small as 10% of the period can easily be detected. The presence and the distribution of shear strains has a distinct influence on the intensities of these generally weak peaks. In turn, they provide a highly sensitive means for the assessment of shear strain in quantum wires.

ACKNOWLEDGMENTS

This work was supported by the Bundesministerium für Wissenschaft und Verkehr, Vienna (Project No. GZ 601.571/1-IV/6/97), by the Fonds zur Förderung der wissenschaftlichen Forschung, Vienna (Project 11557), by the Gesellschaft für Mikroelektronik (Project No. P VI/96) and by the UK EPSRC (Grant No. ER/J90718). The authors are grateful to Steven Thoms, Hubert Straub, and Florian Kaesen for fruitful discussions and to Alexander Ross for assistance in the laboratory.