

Determination of interfacial strain distribution in quantum-wire structures by synchrotron x-ray scattering

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High-resolution grating x-ray diffraction from a periodic quantum-wire structure is shown to be highly sensitive to strain-field variations near a surface or an interface. Information on two types of strain gradients can be obtained: a *longitudinal* gradient, which can produce asymmetric diffraction profiles, and a *transverse* gradient, which can generate additional diffuse intensity streaks in reciprocal space. These effects are demonstrated in a synchrotron x-ray experiment on an $\text{In}_{0.2}\text{Ga}_{0.8}\text{As}/\text{GaAs}$ quantum-wire array. Kinematical diffraction theory is used to describe the diffraction patterns and is found to agree very well with the experimental results. [S0163-1829(97)06924-5]

I. INTRODUCTION

Elastic strain field, due to lattice mismatch and surface relaxation, plays an important role in the physics of mesoscopic-scale crystalline materials of several to 100 nm in size. Although its effect on electronic band structures for bulk and two-dimensional thin-film materials has been known for quite a long time,¹ systematic studies of the strain effects in lateral low-dimensional nanostructures, such as quantum wires (QWR's) and quantum dots (QD's), have become available only recently.²⁻⁵ Recent experimental and theoretical developments on self-organized surface corrugations^{4,6,7} have further enhanced the scientific interests in studying strain and strain distributions near an interface of dissimilar materials during heteroepitaxial growths. In all these cases it is important to experimentally determine the strain fields in the corrugated surface structures or quantum-confinement structures and to correlate the measured strain with other physical properties such as optical luminescence and epitaxial growth modes.

In recent years high-resolution x-ray diffraction has been used as a convenient, nondestructive technique to characterize the geometric shape and the lattice strain in periodic nanostructures.^{5,8-13} The effect of coherent grating x-ray diffraction, i.e., the constructive interference among the periodic structures within the x-ray beam coherence width,¹⁴ can enhance the scattering signal from individual features, and thus significantly improve the strain detectability by x-ray diffraction. Based on this technique, average lattice relaxations have been studied for free-standing multiple-layer quantum wires,^{11,13} and more recently, in a synchrotron x-ray experiment, a lateral-size-dependent lattice distortion has been observed on a single-layer of 10-nm-thick quantum wires of $\text{In}_{0.2}\text{Ga}_{0.8}\text{As}$ buried in a GaAs substrate.⁵

There have been very few attempts, however, to study possible lattice constant variations in quantum-wire and quantum-dot structures using x-ray diffraction, primarily due to the diffuse weak signal from any strain-varying region of a few nanometers in size. In this paper, we show that by using an intense synchrotron x-ray beam and by taking advantage of the coherent-grating nature of a quantum wire or dot array, diffraction profile from a strain-varying region in the quantum wire or dots can indeed be observed in an ex-

periment. From such measurements, both the longitudinal and the transverse strain gradients can be determined along with the average strain. We demonstrate this capability of high-resolution x-ray diffraction with an experiment on the $\text{In}_{0.2}\text{Ga}_{0.8}\text{As}$ quantum-wire array. Our results indicate that a lattice parameter gradient as small as 10^{-5} Å/Å can be readily detected using the new x-ray analysis. Given its convenience and nondestructive nature, we believe that this x-ray technique should be a powerful alternative to electron diffraction methods such as high-resolution transmission electron microscopy.

II. UNIFORMLY STRAINED QWR's

A periodic lateral QWR array epitaxially fabricated on a crystalline substrate (Fig. 1) can be viewed as a coherent grating with a submicron period L . An x-ray diffraction pattern from such a grating structure consists of a set of superlattice peaks around each Bragg reflection, $\mathbf{G}$, of the internal QWR crystal lattice. The diffracted x-ray intensities of the superlattice peaks are given by¹²

$$I(\mathbf{q}) = |f_p(\mathbf{q})|^2 \left[\frac{\sin(Nq_x L/2)}{\sin(q_x L/2)} \right]^2, \quad (1)$$

where $f_p(\mathbf{q})$ is the scattering amplitude from a single period, NL the coherence length of the x-ray beam, and $\mathbf{q}$ is the momentum transfer measured from $\mathbf{G}$. The grating superlattice diffraction peaks occur at intervals $\Delta q_x = 2\pi/L$ and their intensities are determined by the geometric profile and the internal crystalline structure within a single period.

For a box-car-like QWR structure [Fig. 1(a)], the envelope function $|f_p(\mathbf{q})|^2$ gives rise to a single-slit Fraunhofer diffraction pattern in both the q_x and the q_z directions. If the wire is strained with respect to the substrate, then $|f_p(\mathbf{q})|^2$ would be centered around the reciprocal lattice point $\mathbf{G}'$ of the wire crystal structure rather than $\mathbf{G}$ of the substrate. However, as illustrated in Fig. 1(b), the grating superlattice peaks are still positioned commensurately with respect to the substrate $\mathbf{G}$, as pointed out by Holy *et al.*,¹³ because the grating period L is determined by an integral number of the lateral lattice parameter a_x of the substrate instead of that of the QWR lattice.

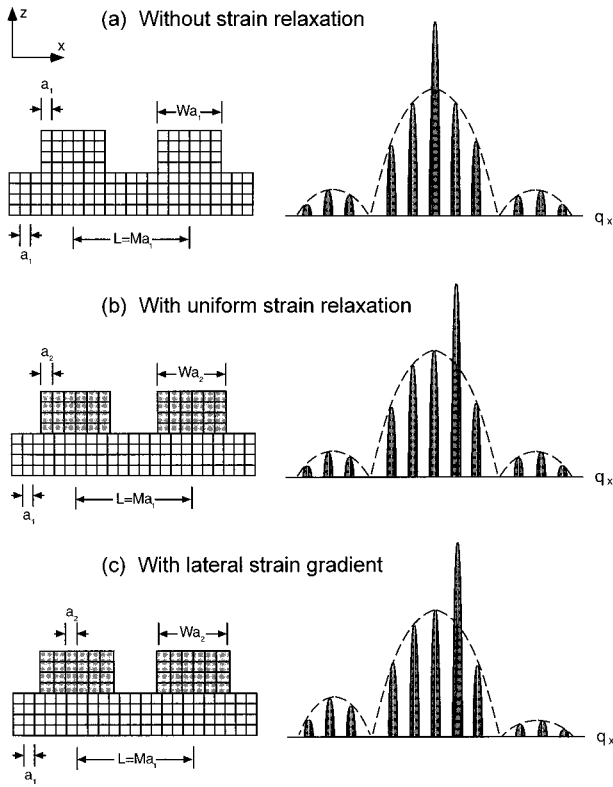


FIG. 1. Illustrations of grating x-ray diffraction profiles from quantum-wire arrays (a) without lateral strain relative to the substrate, (b) with uniform lateral strain relative to the substrate, and (c) with a lateral strain gradient toward the side walls of each quantum wire.

To show the above point more explicitly, we consider a row of atoms in the QWR [Fig. 1(b)] with a lateral lattice constant of a_2 , a width of $w = Wa_2$, and a period of $L = Ma_1$. The scattering amplitude from such a row of atoms is given by

$$F(q_x) = \sum_{n_1=0}^{N-1} e^{iq_x a_1 M n_1} \sum_{n_2=-W/2}^{W/2} e^{iq_x a_2 n_2}, \quad (2)$$

which leads to a scattering intensity

$$|F(q_x)|^2 = \left[\frac{\sin(q_x a_1 NM/2)}{\sin(q_x a_1 M/2)} \right]^2 \left[\frac{\sin(q_x a_2 W/2)}{\sin(q_x a_2/2)} \right]^2. \quad (3)$$

Equation (3) clearly indicates that while the intensity profile of the grating peaks has maxima at multiples of $2\pi/a_2$, the positions of the grating peaks remain locked at multiples of $2\pi/(Ma_1)$, which is commensurate with the substrate lattice a_1 .

In terms of strain-tensor components, the lateral strain described above represents ε_{xx} . Similarly, a shift in q_z of the envelope function $|f_p(\mathbf{q})|^2$ is related to component ε_{zz} . The off-diagonal components ε_{xz} and ε_{zx} may also exist, but these components represent an overall rigid-body rotation and can be set to zero by a proper choice of the coordinate system. A general description of strain-stress components in free-standing QWR heterostructures in relation to crystallographic axes has been given by De Caro and Tapfer¹⁵ and the readers are referred to their paper for further information.

III. EFFECT OF STRAIN GRADIENT

Because of their large surface-to-volume ratios, the physical properties of quantum wire and quantum dot structures can be significantly affected by the existence of a strain gradient near the surfaces or the interfaces. The gradient may result from a natural lattice relaxation, or from a lattice mismatch between the QWR or the QD and its surrounding materials.

A dynamical theory of x-ray diffraction involving lattice distortions in perfect semi-infinite bulk crystals was developed in the 1960(s) by Takagi¹⁶ and Taupin.¹⁷ Their theory takes into account multiple scattering and has been applied to relatively perfect multilayer system in which lattice properties may change as a function of depth.¹⁸ However, a general solution to the Takagi-Taupin equations for an arbitrary strain gradient is rather difficult. Since we are interested in nanostructures of only 1–100 nm in size, multiple scattering is negligible and the kinematic approximation can be applied instead, which is the approach that we will be using in this paper.

To describe a general strain gradient, we make use of an analogy to acoustic waves or phonons and categorize the strain gradients in crystal in to two types: *longitudinal gradients*, e.g., $\partial a_z / \partial z$ and $\partial a_x / \partial x$, involving a lattice constant and its variation along the same direction, and *transverse gradients*, e.g., $\partial a_z / \partial x$, involving a lattice constant and its variation along two orthogonal directions. For QWR's and QD's with well-defined geometric shapes, these two types of strain gradients can introduce distinctly different additional features in an x-ray grating diffraction pattern.

Although studies of longitudinal strain gradient or variation exist for multilayer structures^{18,19} and for surface relaxation of flat silicon wafers,²⁰ to our knowledge the present study is the first of its kind to apply the concept of longitudinal strain variations to lateral quantum structures such as QWR's and QD's. We show that in general, a longitudinal strain variation, which itself can be along either lateral or vertical direction, gives rise to an *asymmetric* intensity pattern [Fig. 1(c)] of the grating diffraction peaks along the corresponding reciprocal space direction.

To illustrate this point, we consider a free-standing, box-car-shaped QWR array with a lateral lattice relaxation. We assume that within each QWR of width w , the lattice constant a_x varies from the center outward according to a power law:

$$a_x(x) = a_0 \left[1 + \varepsilon_x^0 \left(\frac{2|x|}{w} \right)^p \right], \quad (4)$$

where a_0 is the lattice constant at the center and $a_0(1 + \varepsilon_x^0)$ is that at the edge. A similar quadratic dependence has been used by Steinfort *et al.* in the study of Ge hut clusters on a Si(001) surface.²¹ Following the derivation similar to Eq. (2), we can calculate the scattering intensity from such an array and the result for the envelope function $|f_p(q_x)|^2$ is shown in Fig. 2(a) for a lattice constant change of $\varepsilon_x^0 = 0.002$ and several values of p .

It is worth noting how the diffraction patterns in Fig. 2 change as the lattice distortion increases from more gentle ($p > 1$) to more abrupt ($p < 1$). First of all, the position of the central peak is essentially determined by the *average*

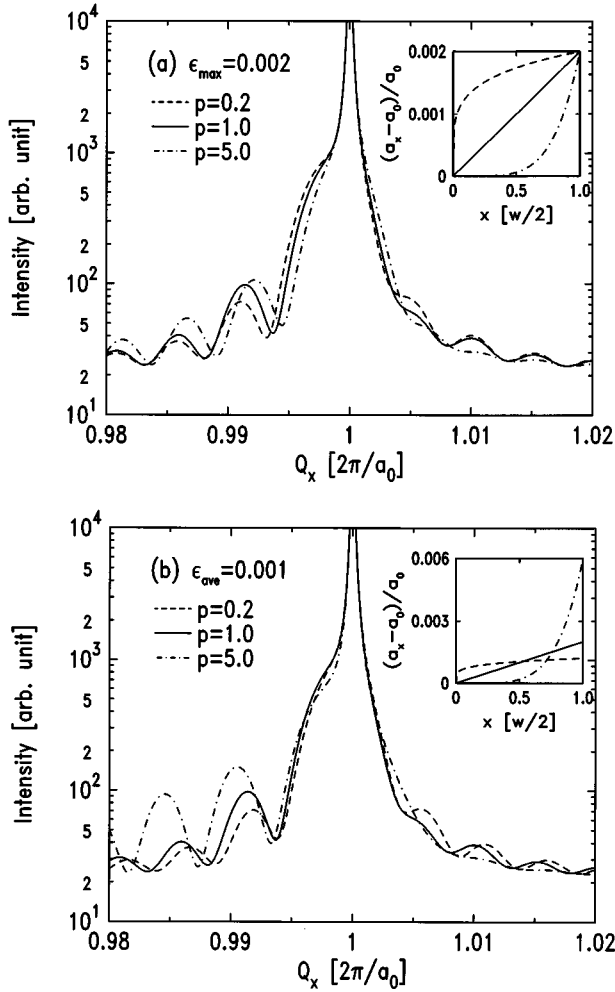


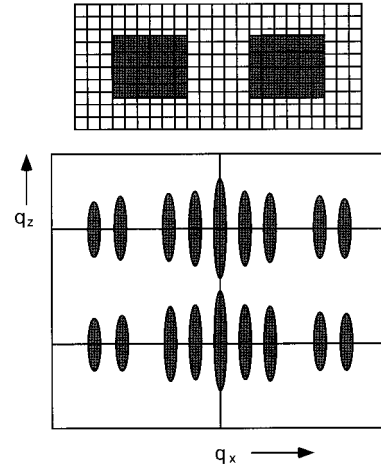
FIG. 2. Calculated grating peak intensity envelope profiles for quantum wires with a lateral strain variation according to the power-law model Eq. (4). (a) Maximum strain variation is kept constant, $\varepsilon_{\max}=0.002$. (b) Average strain is kept constant, $\varepsilon_{\text{ave}}=0.001$. In both cases, a stronger strain variation ($p=5$) near the side walls gives rise to a more asymmetric envelope profile

lattice parameter within the QWR: $\overline{a_x} = \int a_x(x) dx / w = a_0 \varepsilon_x^0 / (1+p)$. Second, the intensity asymmetry in the secondary modulations is very sensitive to the actual *strain distribution* in the QWR, especially the lattice variation near the side walls. For example, the case $p=5$ has the smallest shift due to $\overline{a_x}$, yet it gives rise to the largest asymmetry in the secondary peaks because of the strong variation near the side walls [Fig. 2(a) inset]. On the other hand, the case $p=0.1$ gives a more uniform lattice constant across the QWR and thus yields more symmetric secondary modulations, even though it has a larger shift in the central-peak position due to a larger $\overline{a_x}$.

For comparison, we also calculate the diffraction profiles for a constant *average* lateral strain, $\varepsilon_x^0=0.001$, but with different maximum ε_x^0 and exponent p . The results are shown in Fig. 2(b). In this case, the asymmetry in the secondary diffraction lobes is even more pronounced for $p=5$ since the maximum strain is now much larger than that in Fig. 2(a).

A transverse strain gradient, e.g., $\partial a_z / \partial x$, can produce a

(a) Without transverse gradient



(b) With $\frac{\partial a_z}{\partial x}$ gradient

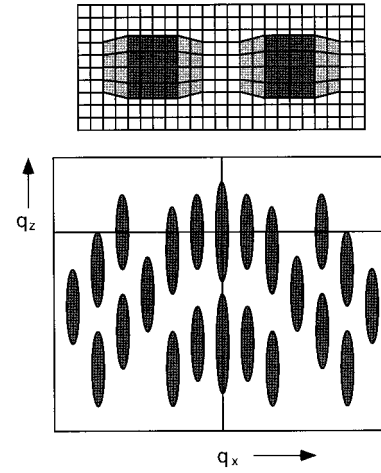


FIG. 3. Schematic illustration of grating diffraction patterns for a buried quantum wire array, (a) without transverse strain gradient $\partial a_z / \partial x$ and (b) with a $\partial a_z / \partial x$ gradient.

very different diffraction pattern that usually involves additional diffraction *streaks* in reciprocal space, as illustrated schematically in Fig. 3. Once again we demonstrate this phenomenon quantitatively with a box-car-shaped QWR array. We assume that the wire array, of a lattice parameter a_2 , is buried in a semi-infinite substrate of lattice constant a_1 , and located at a depth from the substrate surface. Using a power-law model similar to Eq. (4), we assume that the vertical lattice constant a_z varies with the lateral distance x in the following way:

$$a_z(x) = a_2 \left[1 + \varepsilon_z^0 \left(\frac{2|x|}{w} \right)^p \right], \quad (5)$$

where ε_z^0 is the maximum difference between the strains at the center and at the edge of the QWR.

In Fig. 4 we plot the calculated intensity contour images in the q_x - q_z plane around a $(0,0,l)$ Bragg reflection for three different transverse strain variations: (a) $p=1$, (b) $p=5$, and (c) $p=20$, all with $w=980$ Å, $L=4000$ Å, and $\varepsilon_z^0=$

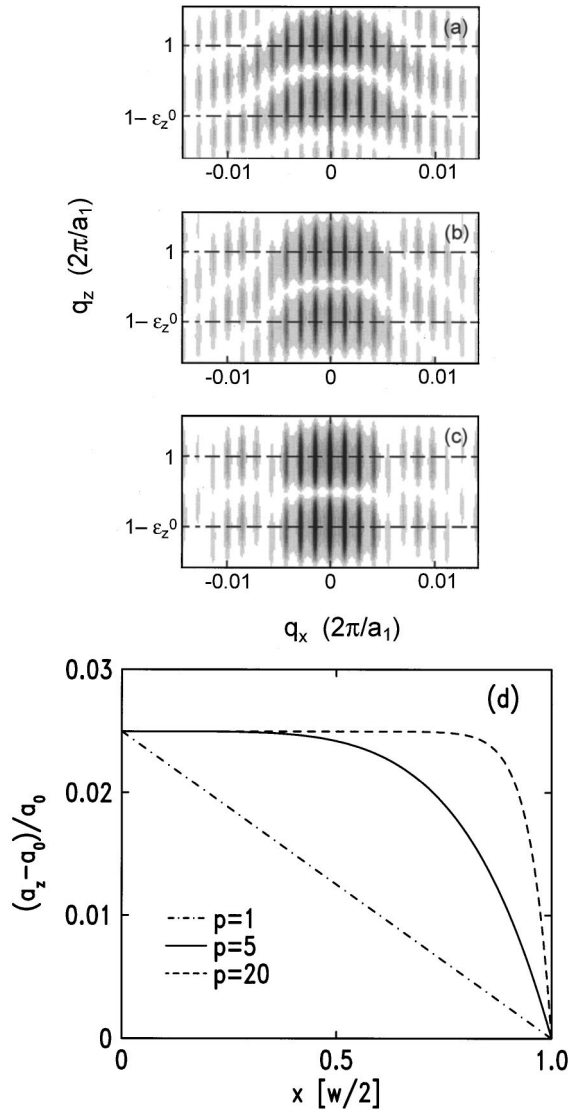


FIG. 4. Calculated grating diffraction patterns around a symmetric $(0,0,l)$ reflection for a buried quantum wire array with several different transverse strain variations, (a) $p=1$, (b) $p=5$, and (c) $p=20$, according to the power-law model Eq. (5). the maximum strain is assumed to be $\varepsilon_z^0 = -0.025$ and its variations are illustrated in (d).

-0.025 . Again, the diffraction patterns shown in these plots are very sensitive to the strain distribution in the quantum-wire structures. In both cases (a) and (b), there exist noticeably broad, tilted intensity streaks in the envelope function, which are characteristic of the diffraction pattern from a transverse strain gradient. By measuring the position and the direction of these streaks in reciprocal space, we can obtain the strain gradient directly from an x-ray diffraction experiment.

A plot of how the lattice constant a_z varies according to Eq. (5) is shown in Fig. 4(d). The case of $p=5$ is particularly interesting because it shows a central region with roughly a constant lattice parameter a_z and a transitional region near the side walls where a_z varies almost linearly. We will show in the next section that this case is very close to the true strain variation in a real quantum-wire structure.

IV. EXPERIMENT ON BURIED $\text{In}_{0.2}\text{Ga}_{0.8}\text{As}$ QWR's

We show in this section some recent experimental results obtained in a high-resolution synchrotron x-ray diffraction experiment on a buried $\text{In}_{0.2}\text{Ga}_{0.8}\text{As}$ QWR array. These QWR's, embedded in a GaAs substrate, were fabricated by a combination of electron beam lithography and molecular beam epitaxial growth techniques.² Each wire in this particular array (area of 0.5 mm^2) has a nominal width of 50 nm and a height of 10 nm and the period of the array is 400 nm. Because the GaAs cap layer on top of the $\text{In}_{0.2}\text{Ga}_{0.8}\text{As}$ QWR is about 380 nm thick, x-ray diffraction is one of the few nondestructive techniques that can be used to probe the structural properties of the QWR's.

The experiment was done at the A2 station at the Cornell high Energy Synchrotron Source (CHESS). The incident x-ray beam was monochromated by a pair of Si(111) crystals to an energy of 8.3 keV. Most of our measurements were concentrated around the symmetric (004) and the asymmetric (115) reflections of $\text{In}_{0.2}\text{Ga}_{0.8}\text{As}$ and GaAs. The QWR sample was mounted at the center of a standard four-circle diffractometer equipped with a postsample Si(111) analyzer. The incident beam, about 1 mm by 0.5 mm in size, covers a sample surface area about twice as large as the patterned QWR region at typical diffraction geometries. Bragg-reflection topographs were taken to ensure that the x-ray beam was centered on the patterned region. We show that several types of strain information, discussed in the last section, can indeed be obtained from the high-resolution x-ray diffraction measurements.

(1) *Average Strain.* In Fig. 5, we show the measured x-ray diffraction pattern around the (115) reflection of $\text{In}_{0.2}\text{Ga}_{0.8}\text{As}$: a two-dimensional reciprocal-space map in (a) and a line scan profile through the $\text{In}_{0.2}\text{Ga}_{0.8}\text{As}$ peak in (b). It can be clearly seen that the center of the grating peak intensity envelope is shifted with respect to the (115) peak from the unpatterned region, even though the grating peaks remain at positions that are commensurate to the unpatterned and substrate peaks. This result of the satellite-peak commensurality directly confirms the theoretical arguments presented in the last section. The amounts of the shifts in both the q_x and the q_z directions reveal an orthorhombic distortion of $\Delta\varepsilon_{xx} = 1.1 \times 10^{-3}$ and $\Delta\varepsilon_{zz} = -2.5 \times 10^{-3}$. It should be noted that this distortion in the QWR's is the strain relaxation relative to the tetragonal strain, $\varepsilon_{xx} = -0.014$ and $\varepsilon_{zz} = +0.013$ with respect to the bulk $\text{In}_{0.2}\text{Ga}_{0.8}\text{As}$, that already exists in the two-dimensional thin film of $\text{In}_{0.2}\text{Ga}_{0.8}\text{As}$.

(2) *Longitudinal strain gradient.* Besides the average strain components, the effect of a longitudinal strain gradient in the lateral direction $\partial a_x / \partial x$ can also be observed in Fig. 5. In particular, the grating peak intensities on the high- q_x side are substantially reduced compared to the corresponding peaks on the low- q_x side, just as illustrated in Fig. 1(c). Using the power-law strain distribution model Eq. (4), we can fit the intensity envelope of the experimental data in Fig. 5(b) by adjusting the exponent p and the effective wire width w , while keeping the average strain $\varepsilon_{xx} = 1 \times 10^{-3}$ constant. The best visual fit, shown as the dashed envelope curve in Fig. 5(b), is obtained with $p = 0.8 \pm 0.3$ and $w = 113 \pm 6 \text{ nm}$. The effective width w appears to be much larger than the true QWR width w , due to the effect of the transverse strain

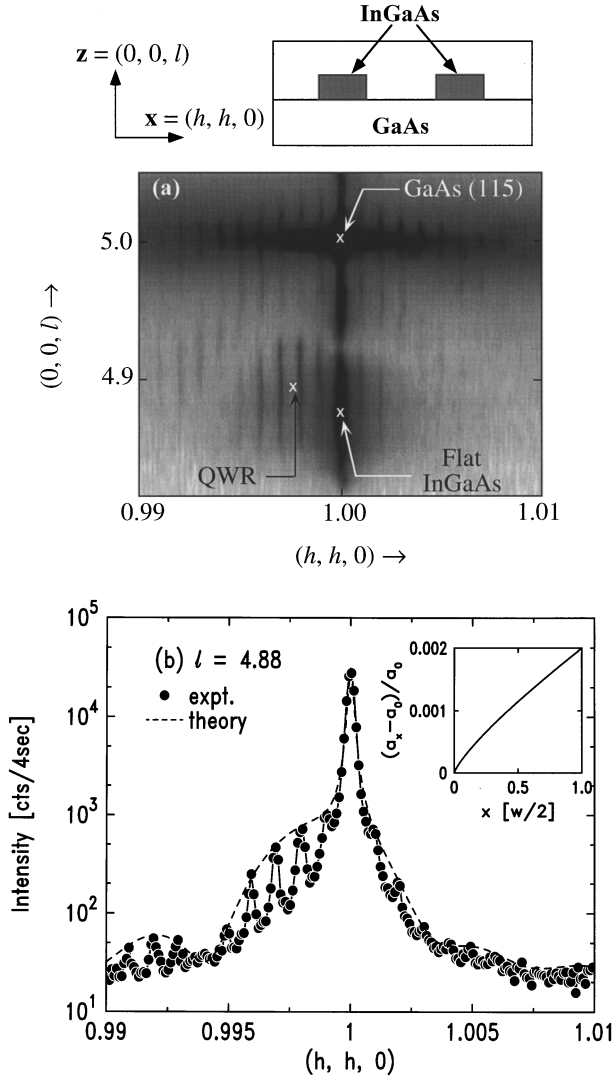


FIG. 5. (a) Measured grating x-ray diffraction pattern at (115) Bragg reflection from a buried $\text{In}_{0.2}\text{Ga}_{0.8}\text{As}/\text{GaAs}$ quantum-wire array. The wires are nominally 50 nm wide and 10 nm high with a period of 400 nm. Intensities are converted into a 32-level gray scale with uniform logarithmic intervals from 1 (white) to a cutoff intensity of 4×10^4 (black) counts. (b) A line scan in the $(h, h, 0)$ direction through the $\text{In}_{0.2}\text{Ga}_{0.8}\text{As}$ (115) peak, at $l = 4.88$. Experimental data are shown as filled circles connected by solid lines. The calculated best-fit intensity envelope function is shown as the dashed curve. Included in the calculation is a lateral strain variation as shown in the inset. This spatial distribution of strain gives rise to the enhanced intensities on the low- h side and the diminished intensity on the high- h side in the diffraction profile.

gradient $\partial a_z / \partial x$ as we will discuss in the next paragraph. The inset of Fig. 5(b) illustrates how the lateral lattice constant varies with lateral position x , according to the best fit values of the power-law model. This indicates a roughly constant strain gradient of $\partial a_x / \partial x = +1.0 \times 10^{-5}$, which shows a very gradual relaxation of the lateral lattice parameter a_x from the center to the edge of the QWR.

(3) *Transverse strain gradient.* The best way to observe the transverse strain gradient $\partial a_z / \partial x$ is around a symmetric Bragg reflection such as the (004) where the influence of lateral lattice distortions $\Delta a_x / a_x$ is minimal. In Fig. 6(a) we

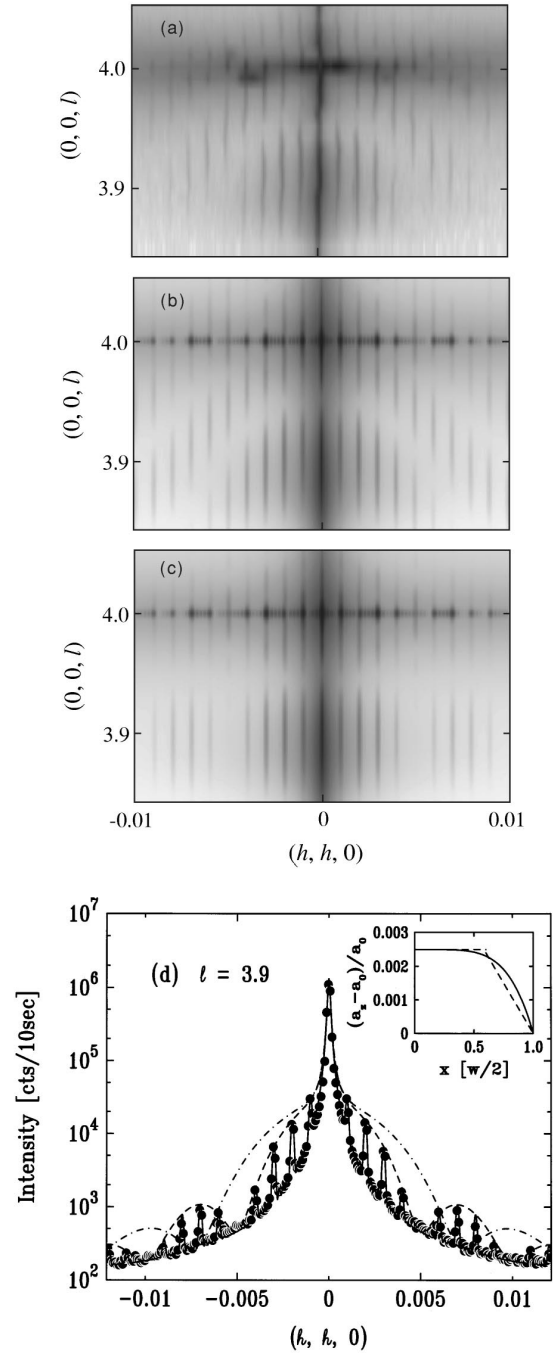


FIG. 6. X-ray diffraction patterns around the symmetric (004) reflection of the $\text{In}_{0.2}\text{Ga}_{0.8}\text{As}/\text{GaAs}$ QWR array. (a) Measured diffraction pattern in which intensities are converted into a 32-level gray scale with even logarithmic intervals from 1 (white) to a cutoff intensity of 1×10^5 (black) counts. (b) Best-fit calculation showing the tilted diffuse scattering streaks due to the existence of a transverse strain gradient $\partial a_z / \partial x$ near the QWR side walls. (c) Same calculation without the strain gradient. (d) A line scan in the $(h, h, 0)$ direction through the $\text{In}_{0.2}\text{Ga}_{0.8}\text{As}$ (004) peak. Experimental data are shown as filled circles and the best-fit calculation is shown as the solid curve with the dashed curve indicating the envelope function. The strain variation used in the calculation is shown in the inset as the solid curve, which can also be represented by a trapezoid model as indicated by the dashed curve in the inset. For comparison the envelope function calculated without the linearly strained region in the trapezoid model is shown as the dash-dotted curve in the main figure.

show the measured x-ray diffraction pattern around the symmetric (004) reflection of $\text{In}_{0.2}\text{Ga}_{0.8}\text{As}$. We see in this plot that there is no lateral shift in the $\text{In}_{0.2}\text{Ga}_{0.8}\text{As}$ (004) peak with respect to the substrate peak because of the null lateral momentum transfer. However, the tilted diffraction streaks as illustrated in Fig. 3 due to a transverse strain variation $\partial a_z/\partial x$, can be clearly observed. This strain variation in the QWR arises from the compressive pressure supplied by the surrounding GaAs substrate on its side [see Fig. 3(b)]. Using the power-law model Eq. (5), we can again fit the diffraction pattern by adjusting the exponent p and the wire width w . The maximum strain $\varepsilon_{zz}^0 = 0.025$, is kept constant in the fitting, which is obtained by the strain measurement around the (115) and is determined by the lattice mismatch between the $\text{In}_{0.2}\text{Ga}_{0.8}\text{As}$ and the GaAs substrate, the Poisson's ratio of the $\text{In}_{0.2}\text{Ga}_{0.8}\text{As}$, and the strain relaxation $\Delta\varepsilon_{zz}$. The best fit to the diffraction pattern is shown in Fig. 6(b) and is obtained with $p=5$ and $w=103\pm 5$ nm. A Q_x scan through the $\text{In}_{0.2}\text{Ga}_{0.8}\text{As}$ (004) peak at $Q_z=3.9(2\pi/a_0)$ is shown in Fig. 6(d) to demonstrate the excellent agreement between the fit and the experimental data.

The inset of Fig. 6(d) shows the vertical lattice constant a_z as a function of the lateral position x , according to the best-fit power-law model. It can also be approximated by a trapezoid model consisting of a strain-relaxed constant- a_z central core of about 55 nm in size and a linearly strained interfacial region of 24 nm on each side of the QWR, with a gradient of $\partial a_z/\partial x = -6.3\times 10^{-4}$, as indicated by the dashed line in the inset. In fact a fit to the data using the trapezoid model produced an almost identical result as the one shown in Fig. 6(b). We would like to point out that although the line scan in Fig. 6(d) could be fit by a single wire Fraunhofer diffraction profile with a wider wire width, it is impossible to describe the overall two-dimensional diffraction map in Fig. 6(a) without the linearly strained or the power-law-varying interfacial region near the QWR side walls, as shown clearly in Fig. 6(c).

V. CONCLUDING REMARKS

We have shown both theoretically and experimentally that high-resolution x-ray diffraction from quantum wire arrays is a sensitive technique for studying strain fields near the surfaces or the interfaces of the wire structures. The constructive grating interference among the periodic superstructures enhances the diffracted signal and allows a quantitative determination of the strain gradients and variations in the QWR's. Through an experiment on a real QWR sample, we have demonstrated that two types of strain gradients or variations can be distinguished unambiguously from the diffraction pattern. One is a longitudinal strain gradient such as

$\partial a_x/\partial x$, which produces an asymmetric profile in the grating peak intensity envelope, and the other is a transverse strain gradient such as $\partial a_z/\partial x$, which produces tilted diffraction streaks in reciprocal space. The diffraction patterns of both of these cases are qualitatively distinct from that of a uniformly strained QWR structure, which exhibits only an overall shift in the centroid position of the grating peak envelope profile.

From an x-ray crystallography point of view, the ability to determine strain gradients and strain variations in nanostructure arrays can be easily understood. The material within each period of the array, involving a single quantum wire or dot, can be viewed as a unit cell of the superlattice, and the grating peaks are simply the Bragg reflections from this superlattice and their intensities are uniquely determined by the positions of each atom in the unit cell. Thus, in principle, the information on strain as well as strain variations in the quantum wire or dot can be obtained completely by measuring the grating peak intensities and solving the crystallographic phase problem. What we have shown is that for most epitaxial quantum-wire or -dot systems the possibility of strain variation is constrained by lattice misfits, geometric shapes, and elastic properties of the wire or dot. Therefore the analysis can be greatly simplified by concentrating on the interfacial regions and by models that only involve monotonic variation in lattice constants.

The information on strain, especially on strain variation near the interfaces, of quantum-wire and -dot structures is very important to the fabrication and the performance of these quantum confinement devices. As we have shown, the strain may vary over a substantial fraction of the quantum-wire or -dot size, and thus may modify the confinement potential well in a significant way. On the other hand, one may be able to tune the strain variation to achieve a particular potential well shape. An example is the strain-induced confinement structures in which no geometric quantum confinement exists, and strain is the sole contributor to the confinement potential. This may be the case where the x-ray diffraction method described here can be applied to determine the quantum confinement potential experimentally.

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