Strain determination in heterostructures by TEM in selected area electron diffraction mode

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Abstract. The technique for determination of strain and elemental composition distribution across the graded layer in heterostructures by means of selected area electron diffraction in transmission electron microscope (TEM) is proposed. The approach accounts for the modification in the residual elastic strain due to sample thinning during TEM specimen preparation. The technique is approved using In₀.₇Ga₀.₃As/InₓAl₁₋ₓAs/GaAs(001) and Al₀.₇₅Ga₀.₂₅N/AlN/Al₂O₃ heterostructures, and the results are in a good agreement with those obtained by alternative ways.

1. Introduction
Understanding of the strain distribution and elemental composition in semiconductor heterostructures is of great importance due to their strong influence on electronic properties. Transmission electron microscopy (TEM) is considered as a powerful characterization tool enabling strain measurements at the nanoscale by convergent-beam electron diffraction [1-3], geometrical phase analysis [4-5], electron nanodiffraction [6-7]. The most conventional and simple TEM technique which yields information on interplanar spacings is selected area electron diffraction (SAED). It has been utilized for the mismatch behavior monitoring across a gradient InGaAs buffer layer grown on GaAs substrate [8-9]. To get the mismatch profile, the stepwise shifting of the cross-sectional TEM sample along the growth direction was applied as it schematically reproduced in Figure 1. The procedure starts from the substrate which serves as a reference. From the subsequent set of obtained SAED patterns, information on the layer-substrate lattice mismatch and misorientation are extracted. The spatial resolution of the technique, determined in this case by the aperture size, is several dozens of nanometers that is worse as compared with the most TEM methods used for strain measurements. Instead, it is less sensitive to the local fluctuations in structural defect distribution, involves simplified data processing and does not require any modification of the standard electron microscope.
As well as any TEM method, the SAED-based strain measurement technique provides data related to thin, electron transparent specimen of which the strain state is different from that in the bulk sample. In this paper, we consider correlation between both states and use it to apply correction to the SAED-based strain measurement data.

2. Theoretical consideration
Heteroepitaxial layers of which the lattice constants differ from those of the substrate are known to be acted on by biaxial stress developed by the lattice mismatch. As a result, a heterolayer becomes elastically strained. During the (110) cross-section TEM specimen preparation the sample is thinned to an electron transparent thickness (10 – 100 nm) which often is much less than the thickness of the
heteroepitaxial layer. Because of thinning, the biaxial stress state of the initial bulk sample transforms to a new state which approaches uniaxial stress condition with the distance from the layer-substrate interface. To understand the strain behavior in the thinned sample, we modeled the stress distribution in a thin GaAs lamella of which the bottom plane is stressed accordingly to a strain of 0,003. An example of calculation by finite element method is presented in Figure 2. Modeling showed the in-plane stress component to quickly decrease with the distance from the plane of acting stress, the transition region being comparable to half of the lamella thickness for any scale of the problem. In the case when the layer thickness is much larger than the thickness of the lamella, transient stress region may be neglected. It allows one to consider the strained layer in the TEM specimen as being completely under uniaxial stress.

![Figure 1](image)

**Figure 1.** The use of methods for determining mismatch on a crystalline sample in a cross section

Knowing the Poisson's ratio of the studied material, it is possible to restore the initial strain in the sample. Strain for biaxially and uniaxially stressed layers can be deduced from the generalized Hooke law as:

\[
\begin{align*}
\varepsilon_x^{(2)} &= \frac{1}{E} [1 - \nu_x] \sigma_x, \\
\varepsilon_z^{(2)} &= -\frac{2\nu_x \sigma_z}{E}
\end{align*}
\]

\[
\begin{align*}
\varepsilon_x^{(1)} &= \frac{1}{E} \sigma_x, \\
\varepsilon_z^{(1)} &= -\frac{\nu_x \sigma_z}{E}
\end{align*}
\]  

(1)

where \( \varepsilon_x, \varepsilon_z \) are in-plane and out-of-plane strain components, respectively, \( E \) is Young's modulus, \( \nu \) is Poisson's ratio, \( \sigma_x \) is stress. Consequently, for uniaxial stress

\[
\varepsilon_z^{(1)} = -\nu_x \varepsilon_x^{(1)}
\]

(2)

By definition, the strain is
\[ \epsilon = \frac{a - a_0}{a_0} \]  

(3)

If attributed to the crystal lattice, \( a \) is the lattice constant of the strained layer, \( a_0 \) is the lattice constant of the strain-free layer. Substitution in (2) provides:

\[ \frac{a_z}{a_{z0}} + \nu_x \frac{a_x}{a_{x0}} = 1 + \nu_x \]  

(4)

In the general case, the lattice constants along different axes are not equal, but their ratio is equal to a certain number \( k \). Eventually

\[ a_{z0} = ka_{z0} \]

\[ \frac{a_z}{a_{z0}} + \nu_x \frac{a_x}{a_{x0}} = \frac{k + \nu_x}{1 + \nu_x} \]  

(5)

Knowing the Poisson’s ratio and coefficient \( k \), the lattice constants of the strain-free layer can be found. Value of \( a_{z0} \) can be determined from diffraction pattern. Knowing the lattice constants of the strain-free layer from (3), strain in uniaxially stressed layer can be found. From the system of equations (1) initial strain components can be determined as

\[ \epsilon_{z}^2 = 2\epsilon_{z}^1 \]

\[ \epsilon_{x}^2 = (1-\nu_x)\epsilon_{x}^1 \]  

(6)

If mole fraction of solid solution components is known, the lattice constant for ternary solution can be defined through Vegard’s law

\[ a_{AB} = x a_A + (1-x) a_B \]  

(7)

Knowing the components of the solid solution and considering (5) the mole fraction of the components can be found as

**Figure 2.** Calculated distribution of stress along the axis parallel to the interface (in-plane stress) in 100 nm thick GaAs lamella biaxially stressed at the bottom plane (pseudocolored)
3. Results and Discussion
The approach described above was applied for composition behavior determination in $\text{In}_{0.7}\text{Ga}_{0.3}\text{As}/\text{In} \text{Al}_{1.7}\text{As}/\text{GaAs}(001)$ structure grown by molecular beam epitaxy. The structure contained a 1250 nm thick graded composition $\text{In} \text{Al}_{1.7}\text{As}$ layer with In fraction $x$ increasing to 0.8 with layer thickness. The study was conducted in JEM 2100F transmission electron microscope using TEM specimen prepared in cross-section geometry. Figure 3 represents the dependence of the indium mole fraction in the graded $\text{In} \text{Al}_{1.7}\text{As}$ layer on the distance from the substrate/layer interface. The error bar was determined by the aperture size used in SAED and the random measurement error. Data on random measurement error were extracted from a series of diffraction patterns taken from the GaAs substrate, which served as the reference.

As it can be seen, the In content stepwise drops from 0.8 down to 0.7 at a distance of about 1300 nm. The observed values correspond well to the transition from the graded layer with the nominal In content 0.8 at its top to the overlapping $\text{In} \text{Ga}_{0.8}\text{As}$ layer with the constant In content 0.7.

Also, the proposed approach was used to measure strain in $\text{Al}_{0.75}\text{Ga}_{0.25}\text{N}/\text{AlN}$ sample with a layer thickness of 1500/500 nm, respectively. The sample was grown by molecular beam epitaxy on $\text{Al}_{2}\text{O}_{3}(0001)$ substrate. The results for in-plain and out-of-plain strain components are presented in Figure 4 and Figure 5, respectively. Blue and yellow colors indicate two series of measurements, the red dotted line shows the calculated strain caused by different thermal expansion coefficients of the substrate and layer materials, the red bold line corresponds to the average of both measurement series.

![Figure 3](image)

**Figure 3.** Dependence of In mole fraction in graded $\text{In} \text{Al}_{1.7}\text{As}$ layer on the distance from the substrate/layer interface

The results represented on the graphs in Figure 4 and Figure 5 show that along with the strain caused by the difference in thermal expansion coefficients of the constituent materials an additional residual strain presents in the layers.
The observed strain behavior correctly reflects the presence of two epitaxial layers with different average level of the strain. The stress magnitude determined close to the surface of $\text{Al}_{0.75}\text{Ga}_{0.25}\text{N}$ layer by the proposed approach without thermal expansion impact yielded 1.2 GPa that is in a very good agreement with the value 1.19 GPa deduced from optical measurements of the substrate curvature \textit{in situ} just after the sample growth completion.

4. Conclusions
The approach for local determination of in-plane and out-of-plane strain components as well as elemental composition by TEM operating in SAED mode has been demonstrated. The approach accounts for the modification in the residual elastic strain due to sample thinning during TEM specimen preparation. The proposed technique possess spatial resolution better than 100 nm, is of little sensitivity to the local fluctuations in structural defect distribution, involves simplified data processing and does not require any modification of the standard electron microscope.

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