Improved accuracy in nano beam electron diffraction

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Abstract. Nano beam electron diffraction (NBD or NBED) is applied on a well controlled sample in order to evaluate the limit of the technique to measure strain. Measurements are realised on a 27nm thick Si_{0.7}Ge_{0.3} layer embedded in a silicon matrix, with a TITAN microscope working at 300kV. Using a standard condenser aperture of 50µm, a probe size diameter of 2.7 nm is obtained and a strain accuracy of 6×10^{-4} (mean root square, rms) is achieved. NBED patterns are acquired along a [110] direction and the bidimensionnal strain in the (110) plane is measured. Finite element simulations are carried out to check experimental results and reveal that strain relaxation and probe averaging in a 170nm thick TEM lamella reduces strain by 15%.

1. Introduction

Supported by the microelectronic industry to increase performance of devices [1], perfect control of strain fields at very high spatial resolution is required to characterize the latest generation of transistors [2]. At the present day only transmission electron microscopy (TEM) can get quantitative strain measurement with sufficient spatial resolution and sensitivity. Several TEM techniques are now available, each with their own resolution, sensitivity and experimental drawbacks.

High resolution imaging has a very good spatial resolution (1-2 nm) and a sensitivity of 1×10^{-3}, but its field of view is very narrow (100 × 100 nm²) and specimens need to be very thin and homogeneous [3, 4]. Convergent Beam Electron Diffraction (CBED) is probably the most accurate TEM technique (sensitivity of 1×10^{-4}) due to information from high indices diffracting planes and has a very fine spatial resolution (2-3 nm) However, the specimen must be tilted slightly off axis (9 to 11°) which leads to structure shadowing and must be rather thick (more than 200nm). Moreover, strains in TEM lamellae relax leading to splitting effects and heavy numerical simulations are then needed to get quantitative strain information [5,6]. A new technique, dark field holography, seems very promising as its spatial resolution is quite good (around 5 nm) combined to a high sensitivity (around 1×10^{-3}) and a large field of view of 250×1000 nm² [7]. However, this method will be limited to specimens having a large defect free unstrained region in epitaxy (i.e. same orientation) with the deformed structure. Compared to these techniques, nano beam electron diffraction (NBD or NBED) investigated here, and quite recently applied to strain measurement, presents some advantages [8, 9, 10, 11].

NBED consists of illuminating the sample with a small almost parallel probe to obtain diffraction patterns with quite sharp spots. The spot positions of diffraction patterns from a perfectly known
unstrained region are compared to the ones from regions of interest. As diffraction patterns are recorded point by point, a reference picture can be taken anywhere in the sample, or the reference can even be taken on a different sample. Moreover, the complete 2D distortion matrix can be retrieved using main diffraction axis although no particular orientation is required to get strain information.

2. Experimental considerations

Measurements were made using a probe corrected FEI Titan microscope working at 300 kV. The microscope was setup to μProbe Scan Mode, probe corrector on with a 50 μm condenser aperture. In these conditions a probe of 2.7 nm in diameter (full width at half maximum) was formed in the image plane. The probe size was evaluated by imaging the probe after its propagation through the [110] Si crystal (fig. 1a and 1b). [110] high resolution lattice fringes can be seen in this probe image. The convergence angle of 0.5 mrad was measured directly in the diffraction pattern (fig. 1c). High angle annular dark field (HAADF) images allowed quickly visualizing the structure and accurately positioning the probe on the sample (fig. 1d). Line scans were realized with the TIA software of the microscope Diffraction patterns were recorded on 2k × 2k Gatan UltraScan cameras using either the Tridiem Gatan image filter (GIF) or not.

A sample composed of a 27 nm silicon-germanium (30% of germanium) layer epitaxially grown on silicon and covered with a 325 nm silicon cap was realized by Reduced Pressure-Chemical Vapour Deposition (RPCVD) [12]. This type of sample, represented in fig. 1d, is perfect for evaluating the NBED technique as the amount of strain, in biaxial condition, is perfectly known from epitaxy and has been measured by different techniques. Moreover, finite element modelling can be easily realized in order to obtain the relaxed structure after thinning. [110] cross-section samples were prepared using an FEI Strata 400 Focus Ion Beam.

Figure 1. (a). Image of the electron probe in a silicon crystal on [110] zone axis. {111} high resolution fringes are visible within the probe. (b). Profile of the probe as represented by the white line in a. (c). Diffraction pattern in silicon obtained along the [110] axis at 300kV with the same probe as in (a). Semi convergence angle is 0.5 mrad. (d) HAADF image of the sample and definition of geometrical axis.

3. Strain measurement

Diffraction patterns recorded during the experiments were treated by a home written program. The algorithm consists in first searching the maxima positions in the image and improves their positions using a 2D Gaussian fit. This last step allows sub-pixel precision and increases the precision. Two spots are then chosen in the fitted diffraction pattern (presently (004) and (220)) which define two vectors (x and y respectively, in figure 1d). These two vectors define a matrix in reciprocal space which gives access to the complete 2D distortion matrix in the diffraction plane [13, 14].

$$\mathbf{D} = (\mathbf{G}_d^T)^{-1} \mathbf{G}_0^d - \mathbf{I}d$$

$$\mathbf{E} = \frac{1}{2} (\mathbf{D} + \mathbf{D}^T)$$

$$\mathbf{\Omega} = \frac{1}{2} (\mathbf{D} - \mathbf{D}^T)$$

Here \( \mathbf{G}_0 \) and \( \mathbf{G} \) are the matrices composed by the two reciprocal vectors (004) and (220) respectively obtained in the reference and deformed Si crystal, \( \mathbf{D} \) is the 2D distortion matrix, \( \mathbf{E} \) and \( \mathbf{\Omega} \) are
respectively the strain and rotation matrices and \( I \) is the identity matrix. With this type of algorithm, the strain state is given regarding to the material used as reference.

### 4. Experimental and numerical results

NBED patterns were acquired along the [110] axis at different points located along a line in the \( x = 0, 0, 1 \) direction. From these patterns strain profiles, such as the one displayed in figures 2 and 3, were extracted. Strain is measured with respect to the unstrained Si crystal. So strain in the SiGe layer is not evaluated with respect to the unstrained equivalent SiGe crystal. With this definition of strain, the strain is concentrated in the silicon germanium layer in the \( x \) direction, as expected from epitaxy. Presence of strain in the \( y \) direction is attributed to strain relaxation phenomena, typical for the sample thickness considered here (170 nm). In the \( x \) direction, sensitivity of the method was investigated by measuring the standard deviation (rms = root mean square) of the measure in the Si crystal far from the SiGe/Si interface. An rms value of \( 6 \times 10^{-4} \) is typically found (this is the value calculated for the points taken in the bold part of fig. 2). This value should be compared to the \( 10^{-3} \) value generally reported in recent publications [9, 11].

In order to validate nano beam diffraction as a quantitative method for strain measurement, finite element calculations were performed using the Comsol\textsuperscript{®} program [15]. Strain curves obtained numerically were convoluted with a Gaussian probe using the experimental probe FWHM and compared with a biaxial strain state and experimental results as shown in figure 3. There is a very good agreement between the simulated and experimental curves within the SiGe layer. The presence of a reverse strain state in the silicon close to the SiGe layer is also reproduced in the simulation but is less strong than in the experimental measurement. This effect can be due to 3D relaxation effects caused by buckling of the lamella. Further investigation is necessary to validate this hypothesis.

Finite Element simulation allows a comparison of the strain state in the thin lamella and in the bulk wafer. For instance, \( \varepsilon_{xx} \) is equal to 2.3% in bulk but equal to only 2% in the thin lamella (thickness taken equal to 170nm). Neglecting strain relaxation in the thin lamella would then lead to a relative error of about 15% in strain measurement. Depending on the application, such an error can be admissible.

![Figure 2](image2.png)  **Figure 2.** Strain profiles taken across the SiGe layer. The different two dimensional strain components \( (\varepsilon_{xx}, \varepsilon_{xy}, \varepsilon_{yy}) \) are drawn.

![Figure 3](image3.png)  **Figure 3.** Comparison between experimental, numerical and bulk strain states in \( x \) direction.

### 5. Experimental considerations

Comparison between strain curves extracted from different spots in the diffraction patterns reveals different behaviours as visible in figure 4. If the general shape of strain curves is very similar, small discrepancies appear in the final strain state in the SiGe layer. These differences can be attributed in the present work to double diffraction phenomena which give rise to the (002) and (006) spots.

Energy filtered diffraction patterns exhibit a very high signal to noise ratio and an increased contrast in recorded the image, as effects due to absorption phenomena are removed. However, figure 5 reveals that energy filtered diffraction patterns using the GIF display much noisier strain data than the one recorded without any filtering. This effect is due to image distortions introduced by the imaging system of the GIF which, even though very small, influences the spot positions [16].
6. Conclusion
Nano beam electron diffraction is a very powerful method, easy to use and fast to process. Its quite high sensitivity ($\Delta \varepsilon = 6 \times 10^{-4}$) and fine resolution (2.7 nm) make it really valuable for quantitative strain measurement at the nanoscale. Simulations have well reproduced the experimental strain profile. In the near future, better lateral resolution and better sensitivity can still be expected by using a smaller condenser aperture and by accurately tuning the probe Cs-corrector of the microscope.

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