Measuring two dimensional strain state of AlN quantum dots in GaN nanowires by nanobeam electron diffraction

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Abstract. Nanobeam electron diffraction (NBED or NBD) is a transmission electron microscope (TEM) based technique able to measure strain with nanometre spatial resolution. This technique is especially well designed for nanostructures as reference diffraction pattern can be acquired anywhere on the sample. NBED is here applied on AlN/GaN superlattices grown on SiC and results compared to finite element simulations in order to assess the accuracy of the method. Strain profiles were then acquired with success on GaN nanowires presenting 3 nm GaN inclusions separated by 3 nm thick AlN.

1. Introduction

Measuring strain at the nanometer scale became of heavy need these last ten to fifteen years due to the introduction of strain in devices on the ten nanometers scale in the microelectronic industry [1]. Presently, the only instrument able to achieve strain measurement at this scale with sufficient strain sensitivity is the transmission electron microscope (TEM). This versatile instrument offers different ways of measuring strain, in real space (high resolution TEM [2,3], dark field electron holography (DFEH) [3,4]) or reciprocal space (convergent beam electron diffraction [5], nanobeam electron diffraction [6]).

Nanobeam electron diffraction (NBED or NBD) consists of forming an almost parallel sided electron probe with nanometer probe diameter that will be scanned over the sample surface. Compared to the other techniques, NBED is well designed for measuring strain on nano-objects as it can be applied to thin samples, in contrast to CBED, it does not require a reference close to the region of interest unlikely DFEH and offers a very large field of view compared to HRTEM. Moreover, if the structure is aligned along a zone axis, strain can be measured in the two directions normal to the electron beam.

These abilities make NBED an excellent technique for measuring strain in nano-objects and it is applied to GaN/AlN nanowires in the present study. In order to realize reliable strain measurement, NBED profiles were first measured for an AlN/GaN superlattice grown on SiC and compared to finite element simulations.

2. Experimental conditions

NBED strain profiles were realized on an FEI Titan microscope equipped with a Cs probe correction module which was acting as pure transfer lens during the experiment. The microscope high tension was set to 200kV and a high angle annular dark field detector (HAADF) was used to record an image
of the sample and precisely locate the probe on the region of interest. Diffraction patterns were acquired on a 2k x 2k Gatan Ultrascan charge-coupled device (CCD) camera located before the Gatan Imaging Filter.

As in most TEM based techniques, strain measurement was made relative to a reference region in a relaxed strain state. In the following, the strain $\varepsilon$ will then be defined using the Lagrange formalism $\varepsilon = (a_{roi} - a_{ref})/a_{ref}$ where $a_{ref}$ is the lattice parameter of the reference region and $a_{roi}$ the lattice parameter of the region of interest. Moreover, all axes directions have been defined such that $x$ is the growth direction, $y$ the in-plane direction and $z$ the direction of electron propagation. The processing of the diffraction patterns was done using a specially designed program based on an automated diffraction spot fitting algorithm.

The first sample is a superlattice consisting of an arrangement of 6 layers of GaN(16 nm)/AlN(13 nm) deposited on top of a 60 nm thick AlN layer grown on a (1-100) SiC substrate. NBED was mainly used on this sample to determine if relaxation was occurring in one specific direction. Two samples were prepared in two different crystallographic orientations, [0001] and [11-20], by FIB milling at low energy with extra care to prevent modification of the strain field.

The second sample consists of GaN nanowires of 50 nm in diameter grown on <111> Si substrates. The top parts are composed of 11 periods of 3 nm GaN separated by 3 nm AlN barriers. The sample, made by simple cleavage of the substrate and the wires, was studied in the cross-section direction.

3. AlN/GaN superlattice on SiC

The first NBED experiment was conducted on the AlN/GaN superlattice and results compared to finite element simulations. This comparison allows checking the reliability of strain measurement on such materials.

For this experiment, an electron probe of 6 nm in diameter was created using a 20 µm C2 aperture, resulting in 0.4 mrad semi-convergence angle. These conditions were specifically chosen for this sample as they allowed us to obtain good spatial resolution with respect to the geometry and excellent strain sensitivity. A picture of the probe and a corresponding diffraction pattern taken through a crystal oriented along the [11-20] zone axis are displayed in Figure 1.

Figure 2a displays a HAADF image of the sample acquired with the previously cited NBED probe. The first profile in Figure 2b, acquired along the [11-20] direction, reveals almost perfect epitaxy between the substrate and the superlattice, the strain in the y direction being equal to zero. In the x direction, the difference in strain between AlN and GaN layers is clearly visible and each layer can be precisely defined. The first 60 nm thick AlN layer appears in a strain state of ~1% compared to the SiC substrate. The GaN layers are also in tensile strain, but with much higher strain value: 4.3%. It is interesting to notice that the noise in the unstrained region of the sample is very low (±0.05%).
Figure 2. (a) HAADF image of the sample using the probe displayed in Fig. 1a. (b) Strain profiles acquired along the [11-20] zone axis. (c) Strain profiles acquired along the [0001] zone axis.

The same experiment was then realized on a sample oriented along the [0001] zone axis, as displayed in Figure 2c. In the x direction, a behaviour similar to the one observed in the [11-20] direction occurred: the first AlN layer is 1% tensely strained and the GaN layers on top are 4.3% strained. However, the y direction now also reveals a strain relaxation of the thick AlN layer as its strain state is just under 1%. This reveals the presence of defects (no preservation of the epitaxial conditions) in the in-plane direction (11-20), the zone axis direction in the previous sample. The GaN/AlN superlattice presents an even higher relaxation with 2% difference in lattice parameter compared to the SiC substrate.

Figure 3. (a) Comparison between experimental and simulated strain profile for the [11-20] direction. (b) Same comparison for the [0001] direction.

Numerical modelling of the AlN/GaN superlattice sample has been performed on both previous samples in order to assess the reliability of the NBED measurement. The Comsol Multiphysics software was used to model the strain relaxation which appears in the thin TEM lamella. Figures 3a and 3b display the comparison between the experimental and the simulated strain profiles in the x direction for both [11-20] and [0001] orientations. The relaxation effect in the y direction for the second sample in the (11-20) orientation was taken into account, as well as the convolution imposed by the probe size [6].

Figure 4. (a) Image of the electron probe through a (11-20) GaN crystal using a 50 µm C2 aperture. (b) Diffraction pattern in the (11-20) zone axis acquired using the probe displayed in (a). The difference in convergence compared to Figure 1b is clearly visible in the fainter diffraction spots.
The agreement between experimental and simulated curves is really impressive and the small differences can be attributed to the lack of homogeneity of the layer thicknesses and orientations. These results prove that NBED is a reliable technique for measuring strain that can be used on more complicated devices.

4. GaN nanowires with AlN/GaN superlattice
As the thickness of the layers deposited on top of the GaN nanowires are much smaller than the one described in the previous section, the same NBED settings cannot be used. Indeed, a 6 nm probe diameter would have led to a blurring of the AlN/GaN layers and a lack of details in the strain profiles. In order to decrease the probe diameter, a bigger C2 aperture was selected (50 µm) which results in the formation of a 2 nm diameter probe (Figure 4a). As the probe diameter reduces, the Rayleigh criterion implies an increase of the semi-convergence angle to 1 mrad. The diffraction pattern, displayed in Figure 4b, reveals wider diffraction spots which will result in a decrease of the strain sensitivity of the NBED measurement.

This NBED probe was then employed on the nanowires to obtain the image displayed in Figure 5a. A strain profile was then acquired along the dashed line and is reported in Figure 5b. For this measurement, the nanowire was oriented along the (11-20) zone axis and the first GaN part taken as a reference. The noise level in the unstrained part of the wire (± 0.15%) is much higher than in section 3 due to the increase in semi-convergence angle. The relaxation of a part of the strain (-1.5 to -2%) in the y direction is clearly visible in the superlattice but does not correspond to a fully relaxed state (-4%), contrary to the result for the x direction. This relaxation in the growth direction was expected as this upper surface is a free surface.

5. Conclusion
The evolution of the transmission electron microscopes these last five years allows obtaining nanometer scaled probe with almost parallel beams and strain profiles with very good strain sensitivity \((5 \times 10^{-4})\). The nanobeam electron diffraction technique (NBED or NBD) is then a well suited method for measuring strain at the nanometre scale in nano-objects such as nanowires for several reasons: it does not required a reference region close to the region of interest, it can be applied on a sample with a wide range of thicknesses, and it is easy to process.

References
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