

The mean inner potential of GaN measured from nanowires using off-axis electron holography

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1. Introduction

Background on electric fields across InGaN/GaN quantum wells

Quantum wells in the InGaN/GaN system have attracted much attention because of their application to advanced wide-band-gap GaN-based optoelectronic devices such as light-emitting diodes and laser diodes [1]. Control of the indium concentration in the InGaN layers in principle enables production of device operating over a wide range of wavelengths. Since wurtzite InGaN has large piezoelectric coefficients, an in-plane compressive stress in the InGaN layer due to lattice mismatch at a hetero-interface causes strong piezoelectric fields across the quantum well [2].

Off-axis electron holography, which is an advanced transmission electron microscopy (TEM) technique, can be employed to measure the electric field across the quantum well. The basis of the technique is described elsewhere [3-4]. The phase change of a high-energy electron passing through the specimen relative to vacuum is proportional to the electrostatic potential of the specimen and to its thickness. A major contribution to the measured electrostatic potential is the mean inner potential of the specimen V_o , which is the volume-averaged crystal potential.

Theory of off-axis electron holography

According to Eq. (1), the phase shift between reference and image wave ϕ is determined by the potential $V(x,y,z)$ and sample thickness t :

$$\phi(x,y) = C_E \int V(x,y,z) dz \quad (1)$$

The constant C_E is given by $C_E = (2\pi e/\lambda)(E+E_o)/E(E+2E_o)$ is given by the kinetic energy of the electrons E , the wavelength λ , and the rest energy of the electron E_o . C_E is $6.53 \times 10^6 \text{ rad V}^{-1}\text{m}^{-1}$ for 300 keV electrons. In the absence of any internal magnetic and electrical fields, and where strong dynamical diffraction can be ignored, Eq. 1 can be simplified as follow:

$$\phi(x,y) = C_E V_o t(x,y) \quad (2)$$

where V_o is the mean inner potential and $t(x,y)$ is the local thickness of the specimen.

An accurate assessment of V_o is required to separate the phase change (relative to the vacuum) due to the V_o from the piezoelectric fields due to the quantum wells.

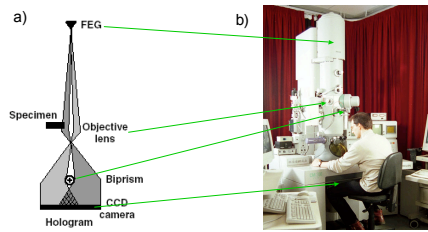


Figure 1: (a) Schematic diagram showing off-axis electron holography and (b) photograph of the Philips CM300 FEGTEM used in this study.

GaN nanowires for mean inner potential study

Measurements of mean inner potential V_o using off-axis electron holography require accurate correlation of measured phase change with thickness variation. GaN nanowires used in this study have a circular cross-section, which provides accurate data on the sample thickness. This accurate knowledge of the specimen shape, coupled with the absence of any possible damage that can be present in conventionally prepared TEM specimens (by ion milling) allows us to accurately assess the V_o of GaN.

2. Experimental

The GaN nanowires were synthesised in a chemical vapour deposition (CVD) reactor using a Ni catalyst. Growth was carried out on SiO₂/Si substrates held at 1050°C for 30 min. Metallic Ga is the group III source and ammonia the group V source. The nanowires grown using this method have a distribution of diameter from 5 to 20 nm from which nanowires of ca. 13 nm diameter were selected for holography studies. Electron energy loss spectroscopy (EELS) data (not presented here) confirms that the nanowires are composed of Ga and N.

GaN nanowires were transferred onto a holey carbon copper grid by tapping the copper grid on the substrate surface.

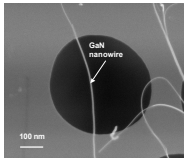


Figure 2: SEM image of GaN nanowires dispersed on a holey carbon copper grid.

Electron holography experiments were performed on a Philips CM300 FEG/ST microscope equipped with a Mollenstedt-Düker biprism. Holograms were acquired using a 2048 pixel charged-couple-detector (CCD) camera. The biprism voltage was set at 160 kV. The field of view in each hologram was 40 nm, with 12 pixels/holographic fringe and a fringe contrast of 30%. The magnification was calibrated accurately using graphitized carbon. Reference holograms acquired from vacuum were used to remove distortions associated with the imaging and recording systems of the microscope.

3. Analysis/Results

Object holograms Reference holograms

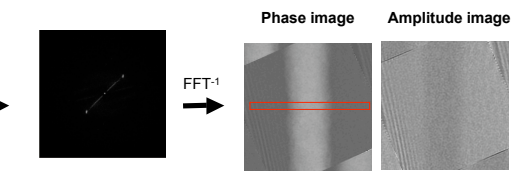
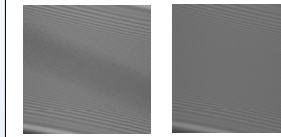


Figure 3: Steps to image reconstruction. FFT produces a spectrum of frequencies present. Sideband is selected and centred. FFT⁻¹ produce phase and amplitude images.

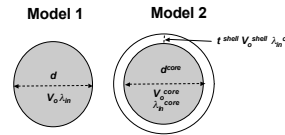


Figure 4: Schematic diagrams showing the cross-section of a nanowire for (a) Model 1 and (b) Model 2. V_o , d , t and λ_{in} denote mean inner potential, diameter, thickness and inelastic mean free path, respectively.

• A SEMPER program allows the fitting of this experimental phase profiles to extract the value of V_o using the 2 models.

• Model 1 assumes the nanowire to have a circular cross-section.

• Model 2 assumes a nanowire with circular cross-section and having a co-axial shell. This model takes into consideration the effects of carbon contamination.

• Eq 2 is used to fit the experimental data and the main parameters are d , V_o , V_o^{core} , d^{core} , t^{shell} and V_o^{shell} .

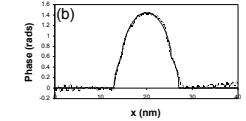
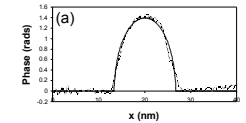


Figure 5: Experimental phase profiles obtained from the selected region in Figure 3 (highlighted in red). The fitted profiles using (a) Model 1 and (b) Model 2 are displayed along with the experimental profiles. Experimental and fitted profiles are dashed and solid lines, respectively.

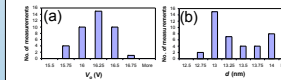


Figure 6: Histograms of (a) V_o and (b) d obtained by fitting experimental phase profiles using Model 1.

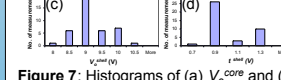
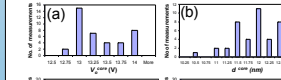


Figure 7: Histograms of (a) V_o^{core} and (b) d^{core} , (c) V_o^{shell} and (d) t^{shell} , obtained by fitting experimental phase profiles using Model 2.

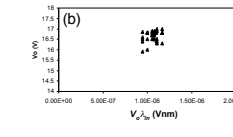
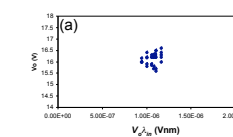


Figure 8: Fitted values of V_o plotted as a function of V_o^{core} for (a) Model 1 and (b) Model 2.

Table 1: Parameters fitted to experimental phase profiles.

| | Model 1 | | Model 2 | | | |
|----------------------------|-----------|----------|------------------|-----------------|-------------------|------------------|
| | V_o (V) | d (nm) | V_o^{core} (V) | d^{core} (nm) | V_o^{shell} (V) | t^{shell} (nm) |
| Mean | 16.12 | 13.24 | 16.67 | 11.78 | 9.01 | 0.94 |
| Standard deviation | 0.24 | 0.42 | 0.27 | 0.50 | 0.57 | 0.19 |
| Standard error in the mean | 0.037 | 0.066 | 0.042 | 0.080 | 0.090 | 0.030 |

4. Conclusions

15 different GaN nanowires with average diameter 13 nm have been characterized using off-axis electron holography. The mean inner potential of $(16.67 \pm 0.3) \text{ V}$ for GaN has been measured from these samples.

5. Reference

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4. E. Völkl, L.F. Allard and D.C. Joy (Eds.), Introduction to Electron Holography, Plenum, New York, (1998).

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