Off-axis electron holography of focused ion beam milled GaAs and Si p-n junctions

D Cooper, A C Twitchett, I Farrer[1], D A Ritchie[1], R E Dunin-Borkowski and P A Midgley

Department of Materials Science and Metallurgy, University of Cambridge, Pembroke Street, Cambridge CB2 3QZ, U.K.
[1] Semiconductor Physics Group, Cavendish Laboratory, Madingley Road, Cambridge CB3 0HE, U.K.

ABSTRACT: Si and GaAs p-n junctions have been characterised in the transmission electron microscope using off-axis electron holography. Focused ion beam milling was used to prepare parallel-sided membranes with thicknesses of 200-500 nm. Off-axis electron holograms were acquired at 200kV in order to assess the effect of specimen preparation on the electrostatic potentials measured across the junctions.

1. INTRODUCTION

Off-axis electron holography is a powerful transmission electron microscopy (TEM) technique that can be used to provide high spatial resolution two-dimensional maps of the phase shift of a high-energy electron wave that has passed through a specimen. The phase shift is proportional to the electrostatic potential integrated in the electron beam direction, suggesting that variations in potential arising from the presence of dopant atoms in a semiconductor can be measured. The technique promises to fulfil the requirement of the semiconductor industry for a dopant profiling technique that has sub-10nm spatial resolution.

Off-axis electron holography is also sensitive to variations in specimen thickness. A thickness variation of 10nm results in a phase change of just under one radian in Si, which can be as much as 50% of the phase change across a p-n junction in a 400-nm-thick specimen.

The study of semiconductor samples in the electron microscope requires a highly site-specific specimen preparation technique. Currently, only focused ion beam (FIB) milling can satisfy this requirement. FIB milling involves the use of a 30kV Ga^+ ion beam to sputter material from a bulk device to prepare an electron-transparent membrane containing the area of interest. Unfortunately, the Ga^+ ions damage the crystalline sample, resulting in the presence of amorphous layers on the membrane surfaces, and additional electrically-altered crystalline near-surface layers (Twitchett et al 2002). These damaged surfaces influence the phase shift across the a p-n junction measured using electron holography.

It is important to understand the effect of the artefacts introduced by different TEM sample preparation techniques on the measured phase shift in a doped semiconductor specimen. The results of electron holography experiments on FIB-prepared Si and GaAs p-n junctions are presented here.

2. EXPERIMENTAL DETAILS

TEM samples of Si and GaAs p-n junctions were prepared using an FEI 200 FIB Workstation operated at 30 kV. The Si p-n junction was grown using molecular beam epitaxy (MBE) and comprised a 2.5-µm-thick 4×10^{18} cm^{-3} B-doped (p-type) layer on a 4×10^{18} cm^{-3} Sb-doped (n-type) substrate. GaAs p-n and n-p junctions were also grown using MBE. Each sample contained a

1.0-µm-thick 1×10^{18}cm^{-3} Be-doped (n-type) layer and a 1.0-µm-thick 1×10^{18} cm^{-3} Si-doped (p-type) layer on an undoped GaAs substrate. Parallel-sided membranes were FIB-milled with total thicknesses ranging between 200 and 500 nm. Care was taken to minimise Ga^{+} implantation into the samples by exposing the region of interest at only a glancing angle to the beam. Final thinning was performed at a low beam current (150 pA) and care was taken to avoid re-deposition of sputtered material onto each sample.

Off-axis electron holograms were acquired using a Philips CM300-ST field-emission gun transmission electron microscope (FEGTEM) operated at a voltage of 200 kV. The holograms were formed using a Lorentz mini lens with the objective lens turned off and a rotatable Möllenstedt-Duker biprism located in the selected area aperture plane of the microscope, and were recorded on a 2048 pixel charge coupled device (CCD) camera. A schematic diagram of the experimental set up is shown in Fig. 1. A biprism voltage of 100V was used to obtain a holographic overlap width of approximately 750 nm with a fringe spacing of 5 nm. The samples were tilted a few degrees from <100> to minimise diffraction contrast across the specimen, whilst taking care to ensure that each junction was edge-on with respect to the electron beam.

Reference holograms were acquired after each hologram of a region of interest to remove any geometrical distortions associated with the imaging a recording system. Figure 1b shows a schematic diagram of the process used to reconstruct phase and amplitude images.

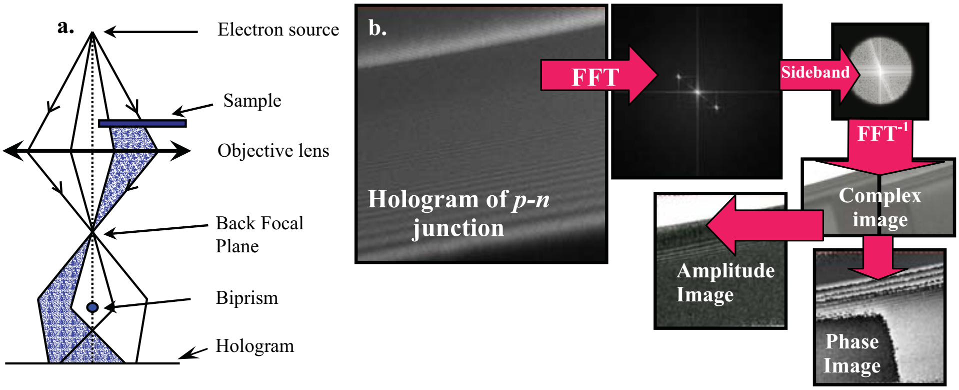

Fig. 1. (a) Schematic diagram illustrating the formation of an off-axis electron hologram. (b) Diagram illustrating the reconstruction of phase and amplitude images.

3. RESULTS AND DISCUSSION

Phase and amplitude images were reconstructed from off-axis electron holograms of all of the GaAs and Si FIB-prepared membranes. The total membrane thickness, t, of each sample was calculated in units of inelastic mean free path, λ from the measured normalised holographic amplitude, A (Gajdardziska-Josifovska and McCartney, 1994) by using the equation

$$t/\lambda = -2\ln A.$$

Crystalline membrane thicknesses were also measured using convergent beam electron diffraction (CBED).

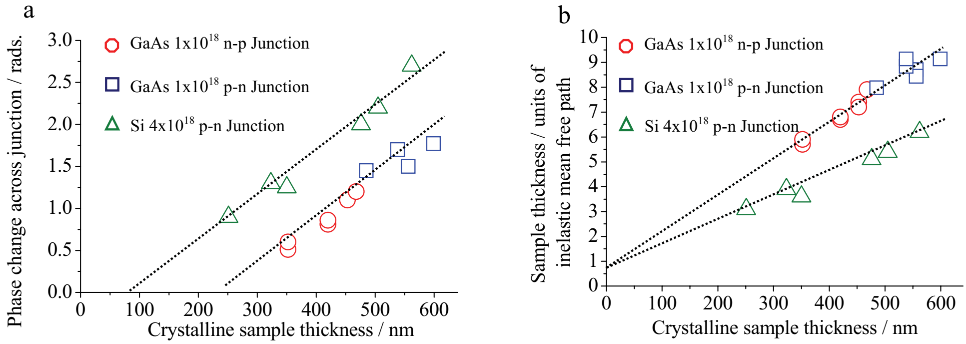

Fig. 2. (a) Phase change across junction, plotted against crystalline sample thickness determined by CBED. (b) Sample thickness measured in units of inelastic mean free path, plotted against crystalline sample thickness determined by CBED.

Figure 2a shows the measured phase changes, φ, across the junctions for all of the samples, plotted as a function of the crystalline thickness of each sample (measured using CBED). The built-in potential, V_{bi}, across the p-n junction is related to the measured phase change Δφ by the relation

$$\Delta\varphi = C_E V_{bi} t_{el}$$

where C_E is a microscope-dependent constant and t_{el} is the electrically active thickness of the sample. The built-in voltage,V_{bi}, was calculated from the gradient of the plot in Fig. 2a to be 0.77 +/- 0.1 V for GaAs and 0.78 +/- 0.06V for silicon. The non-zero x-intercept indicates that the electrically active thickness of the membrane is smaller than the crystalline thickness, and that part of each membrane is electrically inactive. FIB milling is known to create not only amorphous surface layers, but also point defects from knock-on damage to a significant depth in the membrane. These point defects can affect the electrical properties of the semiconductor, altering the electrically active dopant concentration in the near-surface region. The results in Fig. 2a indicate that the effects of such electrical damage extend further into GaAs than Si membranes, with crystalline, electrically inactive regions of 125 and 45 nm on each surface respectively. Their presence increases the membrane thickness required to detect a given phase change across a p-n junction in GaAs to a thickness at which inelastic scattering becomes significant, reducing the signal to noise ratio.

Figure 2b shows a plot of t/λ as a function of crystalline sample thickness for the Si and GaAs FIB-prepared membranes. The x-intercept reveals the thickness of the amorphous surface layers introduced by FIB-preparation. These are found to be 21 nm and 27 nm at each surface for GaAs and Si respectively, in agreement with other recent results (Yabuuchi et al 2005, Twitchett et al 2003). The gradient can be used to calculate the mean free path for inelastic scattering, λ, which is 66 +/- 2nm for GaAs and 99 +/- 4nm for Si. The empirically calculated mean free paths are 133 nm for GaAs and 125 nm for Si (Malis 1988). Previous experiments using electron holography have found λ_{Si} to be 88 nm (Chou and Libera 1998). The experimentally determined value of λ for GaAs may be significantly smaller than predicted due to the presence of relatively thick electrically altered layers near each membrane surface. These layers are expected to contain many point defects, which would act as additional scattering centres, thereby lowering the overall inelastic mean free path.

Figures 3a and b show the experimentally measured phase change across a p-n junction for both Si and GaAs. As indicated in the earlier results, the phase change is much smaller across the p-n junction in GaAs owing to the presence of much thicker electrically-altered surface layers. The signal to noise ratio observed in the GaAs experimental profile is also significantly lower than observed for Si, highlighting the problems associated with examining dopant potentials in GaAs devices. The membrane thicknesses required to measure any detectable phase change are significantly higher than in Si, and inelastic scattering generates significant background noise that reduces the quality of the phase signal measured.

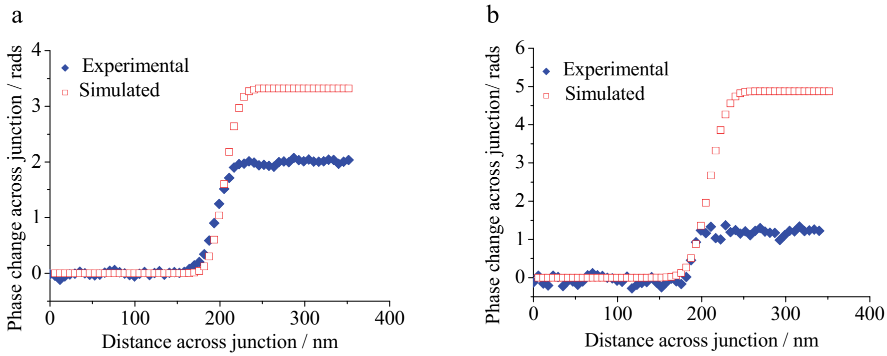

Fig. 3. Calculated and experimental phase variation across 470-nm–thick membranes containing p-n junctions in (a) Si and (b) GaAs.

This study indicates that alternative sample preparation methods must be investigated to reduce the depth of damaged surface layers in order to obtain better quality holograms of GaAs membranes, including additional methods that may reduce surface damage on FIB-prepared membranes such as low-energy Ar-ion milling, chemical etching and low temperature annealing.

4. CONCLUSIONS

Off-axis electron holograms have been acquired from Si and GaAs p-n and n-p junctions. This work has shown that for both GaAs and Si there are significant electrically altered surface layers. These layers may be associated with Ga^+ implantation and further cascade effects at the surfaces during FIB milling. The measured phase changes across the junctions are lower than anticipated, especially for the GaAs specimens. The mean free paths for inelastic scattering and the thicknesses of the amorphous surface layers have also been measured.

In order to successfully characterise the electrical properties of semiconducting devices using electron holography, a greater knowledge of the effects of sample preparation is required, as well as the development of more advanced sample preparation techniques.

ACKNOWLEDGEMENTS

We thank the EPSRC, Newnham College, Cambridge and the Royal Society for financial support.

REFERENCES

Chou T M and Libera M 2003 Ultramicroscopy **94**, 31
Gajdardziska-Josifovska M and McCartney M R 1994 Ultramicroscopy **53**, 283
Malis T et al 1988 J. Electron Microsc. Technol. **8**, 193
Twitchett A, Dunin-Borkowski R E and Midgley P A 2002 Phys. Rev. Lett. **88**, 238302
Twitchett A, Dunin-Borkowski R E, Hallifax R J, Broom R F and Midgley P A 2003 J. Microsc. **214**, 287
Yabuuchi Y, Tametou S, Okano T, Inazato S, Sadayama S, Yamamoto Y, Iwasaki K and Sugiyama Y 2004 J. Electron Microsc. **53**, 471