

Three-dimensional electrostatic potential of a Si *p-n* junction revealed using tomographic electron holography

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Abstract: Off-axis electron holography and tomography have been combined to examine the 3-D electrostatic potential associated with a thin specimen containing a Si *p-n* junction. The device was prepared in a novel specimen geometry using focused ion beam milling, and a series of holograms was acquired over a tilt range of -70° to $+70^\circ$. Simultaneous iterative reconstruction was used to reconstruct the 3-D electrostatic potential in the specimen. The experimental results were compared with simulations of the potential variation. Quantitative results from the central, ‘bulk’ semiconducting regions and from the surface layers were extracted from the 3-D reconstruction.

1. Introduction

Off-axis electron holography has become widely used in recent years as a two-dimensional dopant profiling technique, with many examples of the successful visualisation of dopant-related electrostatic potentials in semiconductors (e.g., [1, 2]). Although electron holography promises to provide fully quantitative results, the measured potential is a 2-D projection along the electron beam direction through the semiconductor membrane thickness, thereby including all surface potential effects. Focused ion beam (FIB) milling is a highly site-specific sample preparation technique which is essential for semiconductor device examination. However, the surface modification of such membranes is significant. The technique is known to generate amorphous and electrically altered near-surface layers which have a considerable impact on the properties of thin membranes. In order to obtain a quantitative characterisation of the bulk and surface properties of a semiconductor membrane, a 3-D map of the electrostatic potential variation is required.

Electron tomography uses a series of images acquired over a large range of tilt angles to reconstruct the 3-D properties of a specimen. This technique has been applied successfully to a number of different problems in materials science, in particular to the examination of catalysts and other small inorganic particles [3]. However, its application in the fields of semiconductors and electron holography has been limited to date. In the absence of diffraction effects, the phase signal reconstructed from off-axis electron holograms satisfies the tomographic projection requirement in that the signal is a monotonic function of the sample thickness, and it should therefore be possible to reconstruct the 3-D phase (and therefore the related electrostatic potential) associated with a doped semiconductor device. This measurement is particularly important for the quantitative determination of the electrostatic potential at an FIB-modified semiconductor surface, but also has significant relevance to the examination of many nanoscale semiconductor structures.

2. Experimental details

2.1. Sample geometry and specimen holder

Samples for examination using off-axis electron holography must satisfy stringent geometrical requirements. There must be a vacuum region close to the area of interest, and the sample thickness must be close to the optimised membrane thickness for the material under examination [4], which is usually $\sim 200\text{-}300$ nm for silicon. These requirements can be satisfied by FIB-prepared samples. However, a standard trench-prepared FIB membrane is restricted in tilt to only $\sim \pm 10^\circ$ due to shadowing by the trench walls, therefore making it unsuitable for tomography. A modified sample geometry has been used, illustrated in figure 1a, in which a thin membrane is milled along the edge of a cleaved square of silicon that can be tilted through 360° without shadowing by the bulk specimen. This specimen was mounted in a Fischione two-contact electrical biasing tomography holder, which is illustrated in figure 1b, and is capable of tilts of $\pm 80^\circ$ in the electron microscope. A silicon p - n junction device with nominal dopant concentrations in excess of 10^{18}cm^{-3} in both p and n regions was prepared in this sample geometry for combined holography and tomography experiments.

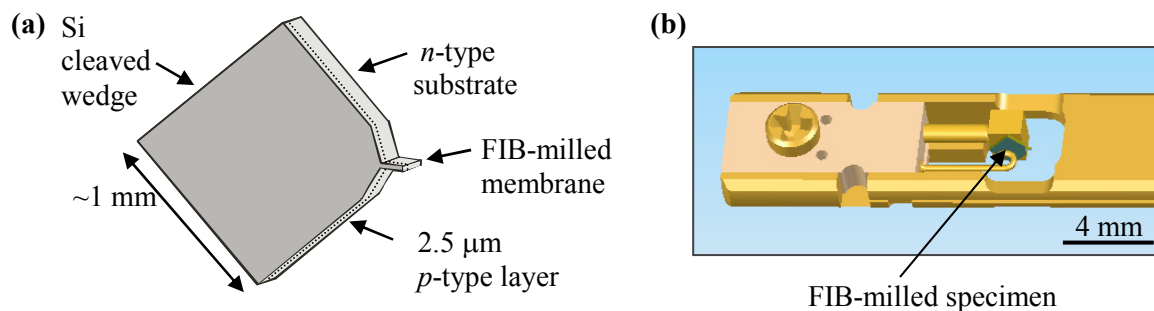


Figure 1. (a) Schematic diagram of the sample geometry used for combined electron holography and tomography of a silicon p - n junction. (b) Design drawing of the end of the Fischione TEM holder used for electrically biased electron tomography and holography.

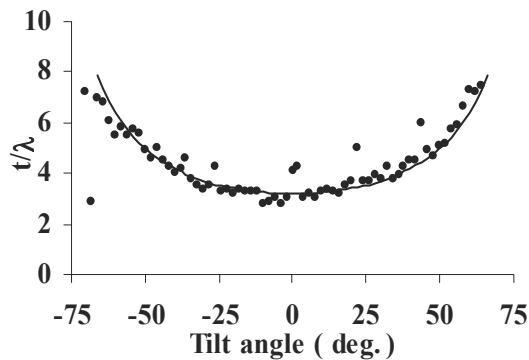
2.2 Experimental procedure

Off-axis electron holograms were acquired using a Philips CM300 field-emission TEM, operated in Lorentz mode and equipped with a Gatan imaging filter (GIF) 2000, using a biprism voltage of 100 V. Holograms were acquired over a tilt range of -70° to $+70^\circ$ at 2° intervals. Reference holograms were acquired every 10° in order to remove distortions associated with the imaging and recording system. Holograms were reconstructed immediately after acquisition using scripts written using Digital MicrographTM software to ensure that the p - n junction was positioned within the field of view, as no alignment features on the sample are visible in an unprocessed hologram. Convergent beam electron diffraction was used to determine the crystalline thickness of the FIB-prepared membrane. This thickness was determined to be 330 nm, giving a total membrane thickness of 380 nm including the thickness of amorphous surface layers generated by FIB milling.

2.3 Data analysis

Off-axis sample and reference holograms were reconstructed to obtain phase and amplitude images using library programs written in the Semper image processing language. Amplitude images were used to calculate normalised thickness (t/λ) maps of the specimen for each tilt angle in units of inelastic mean free path. Figure 2 shows the variation in t/λ over the entire tilt range, demonstrating that a number of points lie away from the expected thickness variation. The points that lie away from the expected inverse cosine variation may indicate that the specimen is in a strongly diffracting condition at those particular orientations, which will affect the reconstructed phase and amplitude such that they do not satisfy the projection requirement for tomography. Such images were therefore excluded from the tomographic dataset used for 3-D reconstruction. At the specimen edge, a number

of 2π phase ‘wraps’ is often present due to the abrupt thickness change present at the edge of the FIB-prepared specimen. These ‘wraps’ can lie directly on top of one another, preventing accurate phase unwrapping. In order to overcome this issue, the expected phase change was calculated (from the mean inner potential and thickness measurements) and used to determine the number of 2π wraps



present at the edge region. The phase in the specimen region in each reconstructed image was then adjusted to the corrected value.

Figure 2. Measured variation in specimen thickness (t/λ) as a function of tilt angle. The solid line indicates the expected variation in thickness with tilt angle. The points lying away from the line indicate that the image is significantly affected by diffraction contrast. The corresponding images are excluded from the tomographic reconstruction.

The original holograms do not contain any distinguishable features other than the interface between the specimen and the vacuum, and therefore the images were aligned using the interface to obtain a rotational and horizontal alignment, and using the junction position to align the images in the perpendicular direction. The simultaneous iterative reconstruction technique (SIRT) was used to reconstruct the 3-D electrostatic potential in the specimen. The specimen thickness was constrained in the reconstruction because the featureless membrane surfaces cannot be reconstructed accurately with the restricted tilt range due to the presence of a ‘missing wedge’ of information. The reconstructed volume contains only the region of varying potential within the total membrane; the amorphous and crystalline electrically-invariant surface regions have not been reconstructed.

3. Results and discussion

A schematic diagram showing the expected electrostatic potential variation is shown in figure 3a, illustrating the amorphous and crystalline electrically inactive surface layers deduced from previous studies [2]. The experimentally determined 3-D reconstructed electrostatic potential of the p - n junction, which is shown in figure 3b, can be observed qualitatively to show a comparable potential distribution to the expected variation. The electrically active membrane thickness is 260 nm with a voxel size in the reconstruction of 5.8 nm and a spatial resolution of 10 nm based on the Crowther equation [5]. The phase resolution is 0.1 rad. [6]. This 3-D data set can be used to extract information about the specimen, including line profiles across the p - n junction close to the centre and close to the surfaces of the specimen. The potential variation indicated in figure 3b is only the dopant-related electrostatic potential, although the absolute value of the potential (relative to vacuum) can be used to determine the value of the mean inner potential (V_0). Theoretical line profiles, taken from simulations described elsewhere [7] are shown in figure 3c, and corresponding experimental line profiles are shown in figure 3d. These line profiles show good correlation between simulations and experimental results in the centre of the membrane, but close to the membrane surfaces the profiles differ because the damage caused by FIB milling has not been incorporated into the simulations. Further simulations are required to model the point defects and amorphous layers present to provide a fully quantitative understanding of the potential at FIB-prepared sample surfaces. However, this preliminary result indicates that the near-surface layers of thin FIB-prepared membranes can make up a significant fraction of a TEM specimen, and must be considered carefully to provide a quantitative understanding of dopant potentials in semiconductor devices.

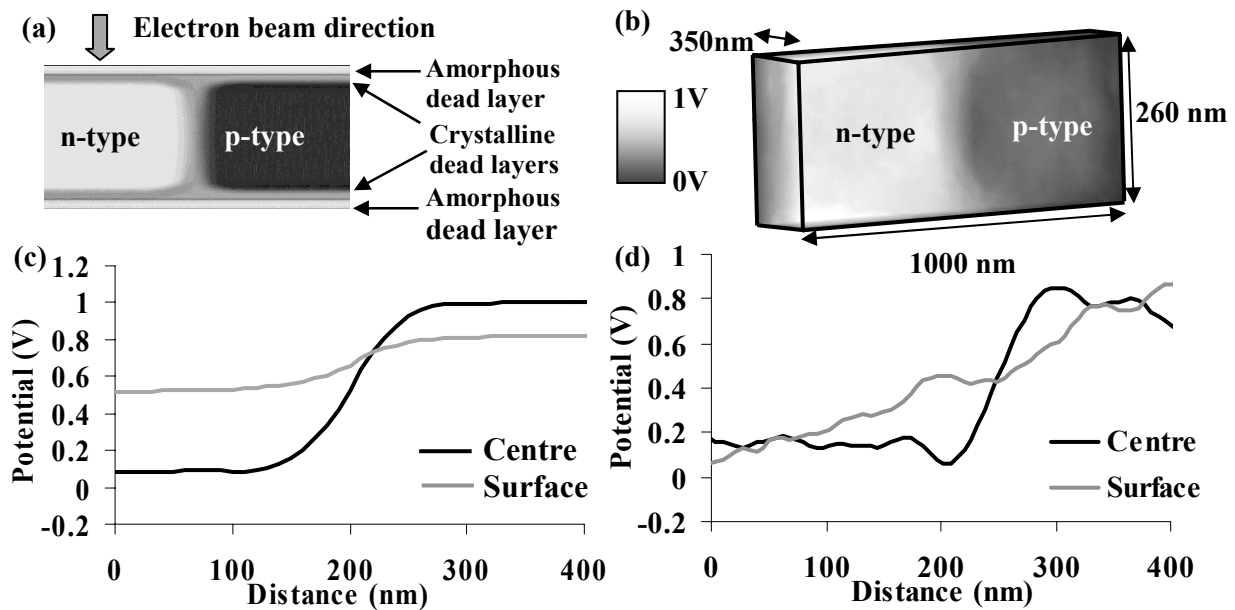


Figure 3. (a) Schematic diagram showing the expected electrostatic potential variation in an FIB-prepared membrane containing a p - n junction. (b) Corresponding experimental 3-D electrostatic potential obtained using electron tomography. (c) Line profiles from a simulation of the expected electrostatic potential. (d) Experimental line profiles extracted from the centre and the edge of the tomographic reconstruction, showing the variation in electrostatic potential across the p - n junction.

4. Conclusions

Off-axis electron holography and tomography have been combined successfully to reconstruct the 3-D potential in a silicon FIB-prepared p - n junction device. The 3-D reconstruction provides information about the mean inner potential and dopant-related potential at any position in a thin specimen. Further work is required to optimise the tomographic reconstruction procedure to ensure a fully quantitative 3-D potential. This information could also be combined with an iterative reconstruction approach to use *ab initio* simulations to deduce the electrical properties of the surface layers. Holographic tomography is a very promising technique for the quantitative examination of 3-D electrostatic potentials in semiconductor devices.

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