Specimen preparation for off-axis electron holography using focused ions, energy filters and laser beams.

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Abstract. Focused ion beam milling is routinely used to prepare specimens with nm-scale site specificity for examination by transmission electron microscopy. Although low-energy milling techniques can be used to prepare excellent specimens for many TEM-based techniques, the case is more complicated for dopant profiling. Off-axis electron holography can in principle be used to provide 2D dopant maps of semiconductor devices. However, artefacts such as Ga implantation and the introduction of defects deep in the crystalline regions of the specimens significantly change the properties of the doped semiconductors. In this paper we discuss methods that can be used to remove the artefacts that are present in FIB-prepared semiconductors.

1. Focused ion beam milling for dopant profiling.
Focused ion beam (FIB) milling is the only technique that can be used to prepare specimens with nm-scale site specificity for examination by transmission electron microscopy. The FIB typically uses a beam of gallium ions that are accelerated to 30 kV to remove the material from around the region of interest to provide a thin lamella of between 50 and 500 nm, depending on the TEM technique and material. It is well known that the FIB introduces artefacts into the specimens in the form of amorphous surface layers (25 nm in Si for 30 kV Ga ions) and Ga implantation. Another artefact which is often not discussed, is the creation of defects deep in the crystalline regions of the specimen. These defects can cause a significant problem when measuring the active dopant concentrations using off-axis electron holography, by scanning electron microscopy and scanning probe microscopy as they can trap the dopants in the specimens. In this paper, the focus is on the development of specimen preparation for dopant profiling using off-axis electron holography, however, the ideas that are presented here are transferable to other techniques such as scanning electron microscopy and scanning spreading resistance microscopy.

Off-axis electron holography uses an electron biprism to interfere an object wave that has passed through a specimen with a reference wave that has passed through only vacuum. From the resulting interference pattern, or hologram, phase and amplitude images of the specimen can be reconstructed. As the phase of an electron is very sensitive to changes in electrostatic potential, such as from the presence of dopants, electron holography can in principle fulfil the requirements of the semiconductor industry for a technique that can provide 2D dopant maps with nm-scale resolution. As the phase of an electron is very sensitive to changes in specimen thickness, composition and strain, as well as from active dopants, specimens of constant thickness over an area of up to 1.0 µm² are required with nm-
scale site specificity. This is only possible with FIB milling, however the defects that are introduced deep in the specimen can trap the dopants which is one of the principle causes of what is known as the electrically inactive thickness. In addition, the electrically inactive thickness is strongly dependent on the FIB operating voltage and also the dopant concentration in the specimens [1]. Therefore in order to be able to extract quantitative information from phase images of semiconductors, this inactive layer must be better understood or removed.

2. The electrically inactive thickness and in situ annealing

In order to improve the understanding of the effects of FIB-milling on semiconductors, simple Si p-n junctions were grown using reduced-pressure vapour chemical deposition. The junctions examined comprised a 1.0-µm-thick, 2x10^{18} cm^{-3} phosphorus (n-doped) layer on a 1.0-µm-thick, 2x10^{18} cm^{-3} boron (p-doped) layer, on a lightly p-doped Si substrate. Two different specimens were prepared using a FIB operated at 30 kV using conventional milling procedures. Each specimen comprised lamellas of thicknesses in the range 300 to 600 nm. Electron holograms of the junctions were acquired using a FEI Titan TEM operated at 200 kV. In order to extend the field of view a Lorentz lens was used with the objective lens switched off. Figure 1(a) shows a reconstructed phase image of a 400-nm-thick specimen containing a p-n junction. The n-doped region appears brighter than the p-doped region as it is at a higher potential. It is also clear that the p-n junction does not extend to the edge of the specimen as the dopants in this region are not active. This is evidence of the damage introduced into the specimen during FIB milling. Figure 1(b) shows the step in phase extracted across the region indicated in (a). Figure 1(c) shows the step in phase measured across a series of junctions as a function of crystalline specimen thickness measured by convergent beam electron diffraction (CBED) for the two different specimens. From the x-intercept, the electrically inactive thickness can be determined. For both of the specimens it is approximately 140 nm, corresponding to 70 nm on each face of the specimen in addition to the 25 nm thick amorphous layer.

Figure 1. (a) Shows a phase image of a p-n junction. (b) Shows profiles extracted from a 400nm thick specimen before and after a 350 °C anneal. (c) Shows the step in phase as a function of the crystalline specimen thickness for two specimens with a range of different thickness lamellas both before and after annealing.

It is known that by annealing specimens in the TEM using an in situ heating holder at only 300 °C the damage in the specimens can be removed leading to an increase in the step in phase measured across the junctions [1]. Figure 1(b) shows the phase measured across a 400 nm thick junction before and after annealing. In Figure 1(c) it can be seen that the electrically inactive thickness is almost completely removed for both of the specimens, demonstrating that the technique is reproducible.

In situ annealing removes the defects that are present in the crystalline regions in the specimen that are introduced during specimen preparation. The gradient of the graph gives the built-in potential in the specimen; even after annealing it is clear that the measured potential is less than predicted by theory. In situ annealing has been successful here, as conventional FIB-milling has been used to prepare the specimens. In the case of specimens that have been prepared using lift out where a small lamella is stuck to a TEM grid, annealing is more problematic as the heat from the in situ holder
cannot be transferred to the specimen without destroying the Pt/W that is used to attach the specimen
to the grid. In order to apply the heat directly to the specimen, laser annealing has been developed.

3. Low energy excimer laser annealing
Lasers have been used to anneal ion implanted semiconductor devices for many years in order to re-
crystallise the amorphous specimens and reactivate the implanted dopants. Lasers are used as their
rapid pulse lengths can lead to very low distances of dopant diffusion. High laser powers (>800
mJcm\(^{-2}\)) are typically used in order to melt and re-crystallise bulk specimens, however, in order to
transfer only a small amount of energy to the specimen the laser has been operated at between 145 and
450 mJcm\(^{-2}\) [2]. Here, two different specimens have been annealed using 20 ns pulses with the laser
operated at two different powers. Figure 2(a) shows the step in phase measured across a series of
different lamella as a function of crystalline specimen thickness measured by CBED. Directly after
FIB preparation an electrically inactive thickness of around 140 nm is revealed from the intercept.
However, after the specimen has been annealed using a laser power of 235 mJcm\(^{-2}\), the inactive
thickness is reduced to around 5 nm. In this case, the laser power seems to be too high as two of the
thinner specimens melted during annealing and were not suitable for subsequent examination.
Although these results show that the Si \(p-n\) junctions can be repaired using either an \textit{in situ} biasing
holder or laser irradiation, the theoretical built in potential has not been recovered in either case. This
is due to the build-up of charge in the specimens during examination.

![Figure 2](image.png)

**Figure 2.** (a) Shows the step in phase as a function of the crystalline specimen thickness for a
specimen both before and after annealing using a XeCl laser with a power of 235 mJ cm\(^{-2}\). (b)
Shows two specimens, with and without electrical connections before and after annealing.

Figure 2(b) shows two different specimens before and after laser annealing using a power of 290
mJcm\(^{-2}\). One of the specimens has one of the sides of the \(p-n\) junction electrically isolated and the
other specimen has both sides of the junction connected to earth by using a thick layer of sputtered
platinum. For the electrically isolated specimen, even after the damage has been removed from the
specimen by the laser treatment, the phase measured across the \(p-n\) junction is much less than
expected from theory. However, after annealing the specimen with both sides of the junction
connected to earth, the theoretical phase across the junction is recovered. Therefore, in order to
recover the theoretical potential from a Si \(p-n\) junction, both the FIB-related damage and the build-up
of charge during examination must be removed from the region of interest.

4. Specimen preparation with silicon, gallium and gold
It has been shown that the electrically inactive thickness can be removed from Si semiconductor
specimens that have been prepared by FIB-milling. However, it is known that such specimens will be
heavily implanted with Ga. As Ga is a dopant in Si, it would be preferable to clean the surfaces of the
specimens before any annealing treatment.

Preliminary experiments have been performed using an energy filtered Orsay Physics EvB FIB
using an AuSi source [3]. Specimens were prepared to a thickness of 5 µm using conventional Ga
milling and then finished using the energy filter to select either 30 kV Au+ or Si++ ions for cleaning. Figure 3(a) shows the step in phase measured across the junction as a function of electrically inactive thickness measured using CBED for specimens prepared using Au+, S++ and Ga+ ions. The electrically inactive thickness which can be measured from the x-intercept is strongly dependent on the choice of ion and is 80, 140 and 410 nm for the specimens prepared using the Au+, Ga+ and Si++ ions respectively. Figure 3(b) shows both the inactive thickness plotted against the simulated range of the ion in Si calculated using SRIM. The graph shows that the electrically inactive thickness in the specimen is directly proportional to the simulated ion range. To test this relationship, a further specimen was prepared using an 8kV Ga ion beam and the inactive thickness was found to be 60 nm (indicated using the open symbol) which fits the general trend shown in Figure 3(b).

5. Discussion

The presence of an electrically inactive thickness has been shown to arise principally from the introduction of physical damage during FIB preparation. The inactive thickness can be almost entirely removed by annealing the specimens either in situ in the TEM or by using an excimer laser operated at low power. Even after the inactive thickness has been removed, charging of the specimen during examination will result in a measurement of the built-in potential that is less than predicted by theory. By using improved electrical connections to earth, the build-up of charge can be removed and the correct potential recovered from the junctions.

It has been shown that the Ga can be cleaned from the FIB-prepared specimens by using a Si ion beam. However, this beam introduces a large amount of damage into the specimens. In order to use Si ions for routine cleaning, it will be necessary to reduce the operating voltage of the FIB. In summary, a range of techniques have been used to improve semiconductor specimens prepared in the FIB which have improved our understanding of the artifacts in the specimens and have allowed the theoretical potential to be recovered from a Si p-n junction specimen.

References

[1] Cooper D, Twitchett AC, Somodi PK, Midgley PA, Dunin-Borkowski RE, Farrer I and Ritchie DA 2006 Appl. Phys. Lett. 88, 063510
[2] Cooper D, Hartmann JM, Aventurier B, Templier F and Chabli A 2008 Appl. Phys. Lett. 93, 183509
[3] Cooper D, Bertin F, Salles P and Benassayag G 2008 Appl. Phys. Lett. 93, 043510