JOURNAL OF APPLIED PHYSICS **101**, 094508 (2007)

The influence of electron irradiation on electron holography of focused ion beam milled GaAs *p*-*n* junctions

David Cooper,[a] Alison C. Twitchett-Harrison, Paul A. Midgley, and
Rafal E. Dunin-Borkowski
*Department of Materials Science and Metallurgy, University of Cambridge, Pembroke Street,
Cambridge, CB2 3QZ, United Kingdom*

(Received 16 November 2006; accepted 10 March 2007; published online 14 May 2007)

Electron beam irradiation is shown to significantly influence phase images recorded from focused ion beam milled GaAs *p*-*n* junction specimens examined using off-axis electron holography in the transmission electron microscope. Our results show that the use of improved electrical connections to the specimen overcomes this problem, and may allow the correct built in potential across the junction to be recovered. © *2007 American Institute of Physics*. [DOI: 10.1063/1.2730557]

I. INTRODUCTION

There is a requirement from the semiconductor industry for the development of a characterization technique that can be used to provide quantitative information about electrically "active" dopants in nanoscale device structures.[1] Off-axis electron holography is a transmission electron microscopy (TEM) technique that involves the use of an electron biprism to interfere a coherent, high-energy electron wave that has passed through a thin specimen with a reference wave that has passed only through vacuum. The resulting interference pattern (or hologram) can be used to recover both the phase shift and the amplitude of the transmitted wave. In the absence of magnetic fields and diffraction contrast, the phase shift of the electron wave is given by the expression

$$\varphi(x,y) = C_E \int_{-\infty}^{\infty} V(x,y,z)dz, \tag{1}$$

where C_E is a constant dependent on the energy of the electron wave, V is the electrostatic potential, and z is the electron beam direction perpendicular to the (x,y) plane.[2] In a semiconductor TEM specimen of uniform thickness, the measured phase shift is expected to provide a direct quantitative measure of the variation in potential associated with the presence of dopant atoms.

Focused ion beam (FIB) milling is used routinely to prepare semiconductor devices for TEM examination due to its unprecedented site-specificity, as well as the ease with which a specimen of near-uniform thickness can be prepared. However, physical damage and the presence of defects resulting from specimen preparation using an energetic ion beam can introduce significant artifacts in the phase shifts measured from doped semiconductors.[3] The electrostatic potential in the specimens can be affected by the implantation of Ga ions during sample preparation,[4] by irradiation using high-energy electrons in the TEM,[5] as well as by surface depletion resulting from the presence of the specimen surfaces.

Previous studies of FIB-prepared Si and GaAs *p*-*n* junctions have shown the presence of an electrically modified layer, usually termed as an electrically "inactive" layer, at each specimen surface that does not contribute to the total phase shift measured across the junction.[6] The presence of such surface layers results in the built-in potential across the junction, V_{bi}, that is calculated directly from a phase image, being lower than predicted by theory. By plotting the step in phase measured across a *p*-*n* junction as a function of crystalline specimen thickness measured using convergent beam electron diffraction (CBED), a value for V_{bi} in the electrically "active" part of the specimen can be determined on the assumption that the thickness of the electrically "inactive" layer is the same in each specimen examined.[7] However, this approach also provides values for V_{bi} that are significantly lower than expected, for reasons that are not yet understood.[8]

Previously, we showed that by annealing Si and GaAs specimens *in situ* in the TEM, the electrically "inactive" layer thickness could be reduced significantly, while at the same time providing an increase in the measured step in phase across the junction and an improved signal-to-noise ratio in the phase image.[9] We suggested that annealing removes defects from the specimens which are introduced during FIB preparation and can pin the Fermi level in the band gap.[10] Although measured values of V_{bi} also improved following annealing, they were still lower than expected, suggesting that the presence of such electrically "inactive" regions and defects does not alone account for the discrepancy between theoretical and experimentally-determined values of V_{bi}. Here, we show for FIB-prepared GaAs *p*-*n* junction specimens that a significant additional contribution to this discrepancy arises from the effect of electron irradiation on the specimen during examination in the TEM.

GaAs *p*-*n* junction specimens were prepared for electron holography using an FEI 200 FIB workstation operated at 30 kV. The junctions were grown by using molecular beam epitaxy, and in all cases comprised a 1.0-μm-thick 1×10^{18} cm^{-3} Be-doped (*n*-type) layer on a 1.0-μm-thick 1×10^{18} cm^{-3} Si-doped (*p*-type) layer on a GaAs (001) substrate. Some specimens were grown on a *p*-doped substrate, while others were grown on nominally undoped substrates.

---

[a] Presently at CEA LETI, Minatec, 17 rue des Martyrs, Cedex 9, 38054 Grenoble, France; electronic mail: david.cooper@cea.fr

The value of V_{bi} for these GaAs *p-n* junctions is expected to be 1.34 V after the effects of degeneracy are taken into account.[11] Parallel-sided electron-transparent membranes were FIB-milled from each wafer in "trench" geometry,[12] with final crystalline thicknesses, measured using CBED, of between 200 and 550 nm. A Pt layer was deposited over each region of interest to protect the specimen surface from Ga^+ implantation and damage during FIB milling. An additional cut was made in each membrane to provide a region close to the junction for the reference wave required for electron holography. Care was taken to minimize Ga^+ implantation by exposing the region of interest only at a glancing angle to the ion beam. Final thinning was performed using a low beam current of 150 pA. Additional specimens were FIB-prepared in an alternative geometry based on FIB milling that allowed them to be biased electrically *in situ* in the TEM using a dedicated electrical biasing TEM specimen holder.[7]

II. RESULTS

Off-axis electron holograms were acquired at 200 kV in a Philips CM300-ST field-emission gun TEM, equipped with a rotatable biprism and a 2048 pixel charge-coupled device (CCD) camera. A Lorentz mini-lens was used as the imaging lens, with the conventional TEM objective lens switched off. A biprism voltage of 100 V was used to generate a holographic interference width of 750 nm and an interference fringe spacing of 5 nm. Specimens were tilted by a few degrees from ⟨110⟩ to minimize diffraction contrast, while ensuring that the *p-n* junctions were kept edge-on with respect to the electron beam. Reference holograms were acquired from vacuum after each hologram of the specimen, and used to remove geometrical distortions associated with the imaging and recording system of the microscope.

Figure 1(a) shows a hologram containing a FIB-prepared GaAs *p-n* junction and Fig. 1(b) shows a reference hologram containing only vacuum. The resulting phase and amplitude images are shown in Figs. 1(c) and 1(d), respectively. The junction is clearly visible in the phase image whereas no contrast variation is present in the amplitude image. In order to obtain quantitative information, phase profiles were extracted from each phase image. Although care was taken to prepare parallel-sided specimens, a phase ramp was present on each phase profiles. The origin of the phase ramp was found to be due to small thickness variations across the specimens, measured by using thickness maps calculated from the reconstructed amplitude images.[13] GaAs has a mean inner potential of approximately 15.3 V,[14] therefore an inaccuracy of the FIB milling angle of less than 0.4° on each specimen surface results in the phase ramp shown in Fig. 1(e) which can be removed by "flattening" the profile using the *p*-type region.

Figure 2(a) shows the height of the step in phase measured across GaAs *p-n* junctions in several specimens, plotted as a function of crystalline specimen thickness, t_{cryst} measured using CBED. Thickness maps were used to verify the flatness of the specimens as a large thickness variation across the membranes can introduce an inaccuracy to the CBED measurement. In addition, the thickness variation of a single membrane, prepared using an identical FIB-milling procedure to all of the other specimens described here, was assessed by using scanning transmission electron microscopy (STEM) CBED. By positioning the electron beam onto a calibrated high-angle annular dark field (HAADF) image, a CBED pattern can be obtained from a desired region. The thickness variation was found to be less than 10 nm, from the specimen surface to a depth of 2 μm. The *x*-intercept of each series in Fig. 2(a) provides an approximate measure of the crystalline contribution to the total electrically "inactive" specimen thickness, t_{inactive}. The slightly larger "inactive" thickness for the specimen with the doped substrate may have resulted from small changes in FIB milling conditions. The gradient of each graph can be used to determine values for V_{bi} within the electrically "active" part of each specimen. These values are measured to be 0.70±0.10 V for the specimen with the undoped substrate and 0.95±0.10 V for the specimen with the doped substrate. Figure 2(a) also shows that specimens prepared from the wafer with the doped substrate exhibit a larger step in phase across the *p-n* junction when they are examined in an electrical biasing specimen holder, even with no voltage applied across the junction. These initial results suggest that the quality of the conduction path to earth, which is required to remove charging of the specimen due to the generation of electron-hole pairs by the

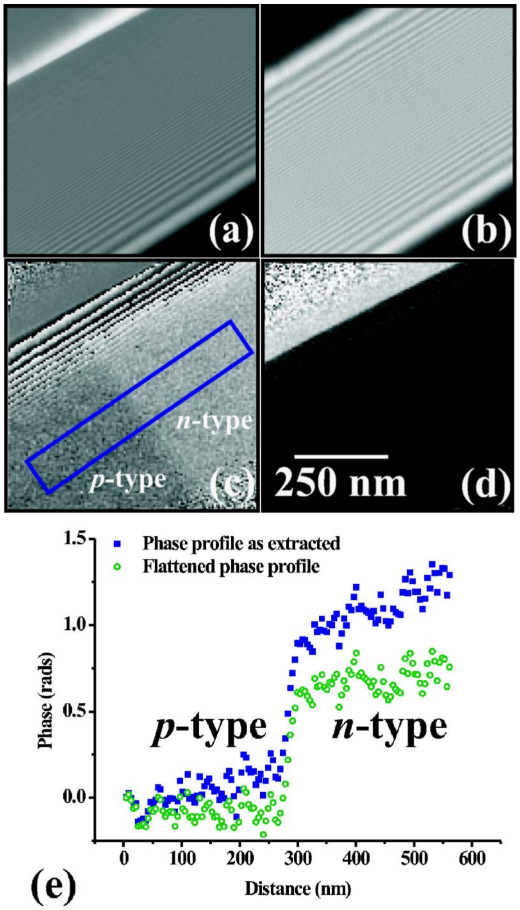

FIG. 1. (a) Electron hologram of FIB-prepared GaAs specimen and (b) reference hologram acquired from vacuum. (c) Phase image derived from (a) and (b). (d) Corresponding amplitude image. (e) Phase profile as extracted from (c), and artificially 'flattened' profile.

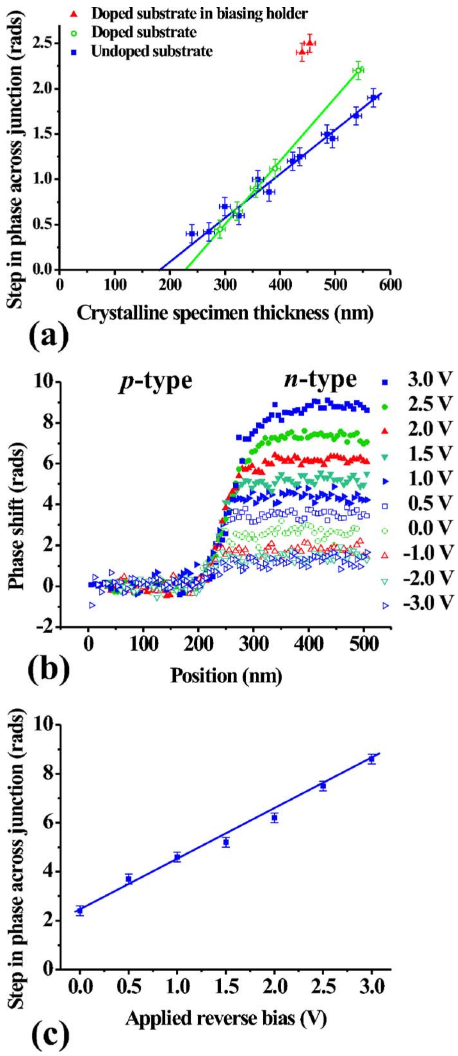

FIG. 2. (a) Step in phase measured across *p*-*n* junctions in several different FIB-milled GaAs specimens, plotted as a function of crystalline specimen thickness measured using CBED. (b) Phase profiles measured across a 440-nm-thick FIB-milled GaAs *p*-*n* junction on a doped substrate for different applied bias voltages. (c) The step in phase across the *p*-*n* junction plotted as a function of applied reverse bias voltage. In each instance, a relatively high electron irradiation intensity was used (spot size 2).

incident electron beam, may influence the electrical properties of FIB-prepared TEM specimens measured using electron holography.

By measuring the gradient of the step in phase, $\Delta\phi$ across the junction plotted as a function of applied reverse bias voltage, V_{app}, the total thickness of the electrically "active" layer, t_{active} can be determined for an electrically-biased specimen by using the equation

$$\Delta\phi = C_E V_{\mathrm{bi}} t_{\mathrm{active}} + C_E V_{\mathrm{app}} t_{\mathrm{active}}, \tag{2}$$

where $t_{\mathrm{active}} = t_{\mathrm{cryst}} - t_{\mathrm{inactive}}$. Figure 2(b) shows phase profiles measured from a 440-nm-thick specimen with a doped substrate, and Fig. 2(c) the corresponding step in phase across the junction plotted as a function of applied bias. From the gradient of this graph, the electrically "active" specimen thickness was determined to be 266±15 nm, and from the intercept of the graph the value for V_{bi} across the *p*-*n* junction was determined to be 1.30±0.10 V. Significantly, this measured value is comparable to the value of V_{bi} expected for this specimen to within experimental error, and is considerably higher than the value measured from a specimen prepared from the same wafer using conventional "trench" geometry, as shown in Fig. 2(a).

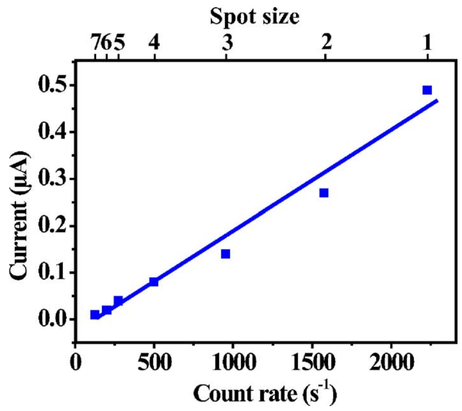

FIG. 3. Current measured across a GaAs *p*-*n* junction during examination in an electrical biasing TEM specimen holder, plotted as a function of the intensity of the electron illumination. The intensity of the electron beam was varied by using different spot sizes and quantified by using the count rate recorded on the CCD camera from the center of a reference hologram. The current was measured using a sourcemeter connected in series with the biasing holder and for each measurement the area of the electron illumination was kept constant. The spot size associated with each measurement is indicated on the upper *x*-axis.

During the electrical biasing experiment described in Figs. 2(b) and 2(c), it was noted that a current was measured on a sourcemeter connected to the junction, even when no reverse bias was applied. The measured current was investigated as a function of electron beam intensity by varying the spot size while keeping the area of the illumination constant (the spot size determines the flux of electrons collected by the aperture before the second condenser lens). Figure 3 shows measurements of the current flowing across the junction, plotted as a function of the count rate recorded on the CCD camera in a reference hologram. The relationship between these parameters are approximately linear. The spot size used for each measurement is shown on the upper *x*-axis.

In order to assess the effects of electron beam irradiation, further electron holograms of the GaAs *p*-*n* junction with the conducting substrate, prepared using "trench" geometry, were acquired using a range of illumination conditions. Figure 4(a) shows flattened phase profiles from the junction measured using different spot sizes. The electron beam is seen to have a significant influence on the profiles on only the *n*-type side of the junction and no change in the phase ramp is observed in the *p*-type region, even before "flattening" the profiles. The step in phase measured across the junction is reduced when the specimen is illuminated using a higher current. It has been reported that the effects of charging can be reduced by carbon-coating the specimen.[15] Figure 4(b) shows results obtained from the same specimen after coating it on one side with approximately 20 nm of carbon. Although the effect of electron irradiation on the step in phase across the junction is indeed reduced, carbon-coating does not remove the effects of specimen charging

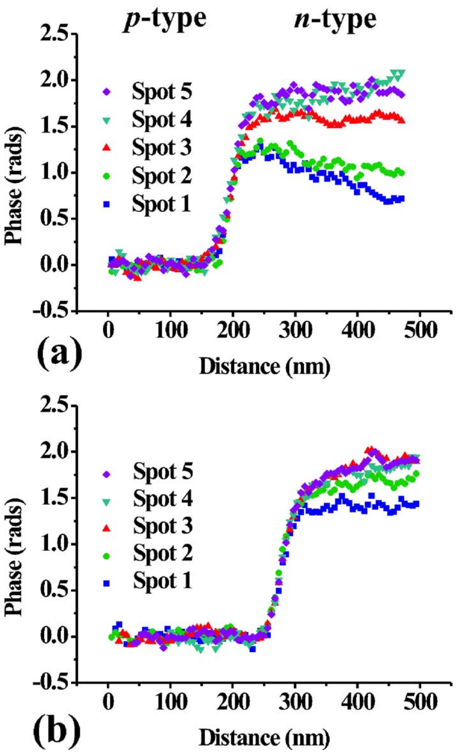

FIG. 4. (a) Phase profiles measured across an unbiased GaAs *p*-*n* junction on a doped substrate acquired using different spot sizes. (b) Phase profiles measured from the same specimen after carbon coating.

completely. In addition the asymmetry of the phase profiles shown in Figs. 4(a) and particularly in Fig. 4(b) suggest that the effects of carbon-coating and electron irradiation on charging are different on each side of the junction. This behavior can be observed in the relatively gradual change in phase on the *n*-side of the depletion region in Fig. 4(b), suggesting that the active dopant concentration on this side of the junction when measured in projection is lower than on the *p*-side. Even after carbon-coating the *p*-*n* junction specimen, the gradient of the step in phase plotted as a function of crystalline specimen thickness, which is shown in Fig. 5(a) for a constant area of illumination, provides values for V_{bi} of only 0.85±0.10 V and 0.95±0.10 V when using spot sizes 2 and 4, respectively. In contrast, for the *p*-*n* junction specimen examined in the electrical biasing specimen holder, Fig. 5(b) shows that electron irradiation does not influence the step in phase across the junction, presumably because of the presence of an improved electrical contact between the region of interest and the microscope ground. The intercept of the graph shown in Fig. 5(b) provides a value for V_{bi} of 1.30±0.1 V, which is again consistent with the value that would be predicted for this junction.

During TEM examination, electron-hole pairs are generated in the specimen, particularly as GaAs is a direct bandgap semiconductor.[16] The electric field in the *p*-*n* junction will cause the electrons and holes to separate, with the holes drifting toward the *n*-doped regions and the electrons toward the *p*-doped regions. Although no variation of the phase is seen in the *p*-type layer, presumably as the excess carriers can easily flow from the region of interest into the substrate, the results shown here suggest that the holes generated in the junction are trapped in the *n*-doped layer due to the poor conduction path to earth and that their presence results in a reduction of the measured potential. This problem can be solved by attaching electrical connections onto each side of the *p*-*n* junction.

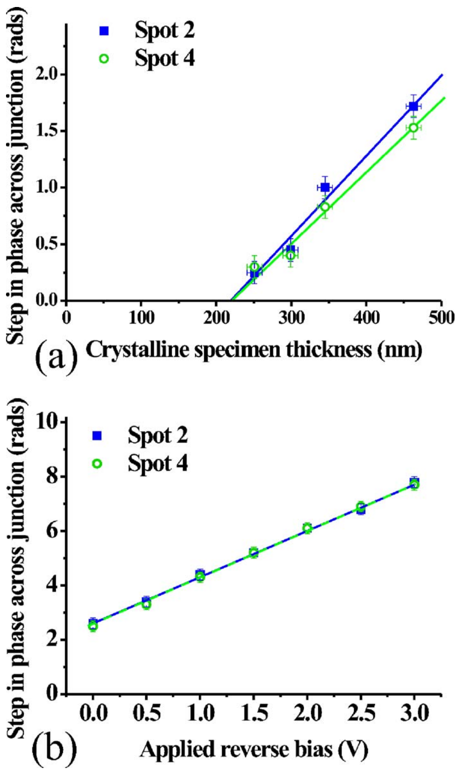

FIG. 5. (a) Step in phase, plotted as a function of crystalline specimen thickness across a carbon-coated FIB-prepared GaAs *p*-*n* junction on a doped substrate measured using spot sizes 2 and 4 for a constant area of illumination. (b) Step in phase measured across a 440-nm-thick GaAs *p*-*n* junction on a doped substrate, plotted as a function of applied reverse bias voltage for spot sizes 2 and 4 and for a constant area of illumination.

III. DISCUSSION AND CONCLUSIONS

In a previous study, we showed that *in situ* annealing can be used to significantly reduce the thicknesses of electrically "inactive" surface layers on FIB-prepared *p*-*n* junction specimens, but that the measured built-in potential was too low for both Si and GaAs specimens. Here, we have shown that phase shifts measured across identically-doped FIB-prepared GaAs *p*-*n* junctions depend on the efficiency with which charge resulting from electron-hole pair generation and secondary electron emission during electron irradiation can be removed from the regions of interest, even after carbon-coating the specimens. In specimens that have poor electrical contacts, the measured step in phase across the junctions increases as the incident electron current is reduced. This problem can apparently be eliminated by the use of improved electrical connections to the specimen, for example by using an electrical biasing TEM specimen holder. The step in phase across the junction is then not influenced significantly by the electron beam current, and a value for V_{bi} that is both consistent with theory and independent of the incident electron beam current can be obtained. The signal-to-noise ratio in phase images acquired from specimens examined in the biasing holder can then be improved by reducing the thickness of the damaged surface layers, either by annealing the specimen or by using low voltage Ga^+ ions during FIB preparation to reduce the thickness of both the amorphous[17] and electrically "inactive" regions.[18] Further work is now required to assess whether the present conclusions are equally applicable to specimens with different doping concentrations, to *p*-*n* junctions in semiconductors other than GaAs and to *p*-*n* junctions that are formed from *p*-doped layers grown on *n*-doped substrates, as well as to the *n*-doped layers on *p*-doped substrates that have been examined here.

ACKNOWLEDGMENTS

We would like to thank Ian Farrer and David A. Richie at the Cavendish Laboratory, Cambridge for supplying the samples. For financial support we thank the EPSRC and the Royal Society.

[1] International Technology Roadmap for Semiconductors, 2005 ed., Semiconductor Industry Association, San Jose, CA, 2005, http://public.itrs.net
[2] P. A. Midgley, Micron **32**, 167 (2001).
[3] A. C. Twitchett, R. E. Dunin-Borkowski, R. J. Hallifax, R. F. Broom, and P. A. Midgley, J. Microsc. **214**, 287 (2004).
[4] D. Sutton, S. M. Parle, and S. B. Newcomb, Inst. Phys. Conf. Ser. **168**, 377 (2001).
[5] L. Houben, M. Luysberg, and T. Brammer, Phys. Rev. B **70**, 165313 (2004).
[6] W. D. Rau, P. Schwander, F. H. Baumann, W. Hoppner, and A. Ourmazd, Phys. Rev. Lett. **82**, 2614 (1999).
[7] A. C. Twitchett, R. E. Dunin-Borkowski, R. J. Hallifax, R. F. Broom, and P. A. Midgley, Phys. Rev. Lett. **88**, 238302 (2002).
[8] D. Cooper, A. C. Twitchett, I. Farrer, D. A. Ritchie, R. E. Dunin-Borkowski, and P. A. Midgley, Inst. Phys. Conf. Ser. **107**, 221 (2005).
[9] D. Cooper, A. C. Twitchett, P. K. Somodi, I. Farrer, D. A. Ritchie, P. A. Midgley, and R. E. Dunin-Borkowski, Appl. Phys. Lett. **88**, 063510 (2006).
[10] D. Pons and J.C. Bourgoin, J. Phys. C **18**, 3839 (1985).
[11] J. F. Gibbons and R. E. Tremain, Appl. Phys. Lett. **26**, 199 (1975).
[12] K. H. Park, Mater. Res. Soc. Symp. Proc. **199**, 271 (1990).
[13] M. R. McCartney and M. Gajdardziska-Josifovska, Ultramicroscopy **53**, 283 (1994).
[14] P. A. Doyle and P. S. Turner, Acta Crystallogr. **A24**, 390 (1968).
[15] M. R. McCartney, M. A. Gribelyuk, J. Li, P. Ronsheim, J. S. Murray, and D. J. Smith, Appl. Phys. Lett. **80**, 3213 (2002).
[16] D. B. Williams and C. B. Carter, *Transmission Electron Microscopy* (Plenum Press, New York, 1996).
[17] N. I. Kato, Y. Kohno, and H. Saka, J. Vac. Sci. Technol. A **17**, 1201 (1999).
[18] D. Cooper (unpublished, 2006).