Report on Grant No. GR/R97986/01:

Electron Holography of Semiconductor Junctions:
Developing a Reliable Methodology for the Microelectronics Industry

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Background and Context

For many years, the microelectronics industry has published the semiconductor roadmap, which shows how key feature sizes (e.g. DRAM pitch length) will continue to to shrink in the next few years and are predicted to be below 50 nm in 2010. As one of its priorities, the industry has identified the development of characterisation techniques that can map dopants in two and three dimensions with high spatial resolution and with high sensitivity. This proposal set out to address these issues and to develop a reliable methodology for the microelectronics industry using electron holography. In particular, we stated that our aim was to develop off-axis electron holography in the field emission gun transmission electron microscope (FEGTEM) to allow electrically active dopant distributions in semiconductor devices to be measured and interpreted a) to a spatial resolution of 5 nm, b) to an energy resolution of 50 meV, c) in both two and three dimensions, d) in working devices in the TEM and e) to do so reproducibly and quantitatively.

Electron holography is an interferometric technique that allows the phase shift of an electron wave to be encoded in the form of changes in the positions of interference fringes. There are a number of ways to form these fringes, but the two that are used predominantly are off-axis electron holography and in-line holography (or Fresnel imaging). The former uses a positively charged electron biprism to bring together two halves of the electron wave and the latter involves defocusing the image to form Fresnel fringes whose intensity and spacing depend on the phase of the wave and the defocus value. The techniques are complementary: off-axis holography is excellent at measuring gradual changes in the potential quantitatively over large distances (the phase is proportional to the electrostatic potential), while the Fresnel technique is excellent at revealing the form of rapid changes in potential such as those found at boundaries.

Objectives. The aim of this project was to develop electron holography to allow the accurate, reproducible and quantitative characterisation of electrostatic dopant potentials in working semiconductor devices at high spatial resolution, in both two and three dimensions. The specific objectives were:

(i) To design, test and commission a new electrical stage for use in a Philips CM300 FEGTEM to allow bias voltages to be applied to different semiconductor device geometries and to enable ultra-high specimen tilts to be achieved for the three-dimensional tomographic reconstruction of dopant potentials in TEM specimens.

(ii) To determine the effect of the presence of the surfaces of a TEM specimen on the electrostatic potential associated with a semiconductor *p-n* junction, and to establish the degree to which electron holographic phase images can be acquired reproducibly and interpreted quantitatively.

(iii) To develop a dedicated software package for the simulation of semiconductor dopant potentials in thin TEM specimens, and to compare the results of these simulations with experimental electron holography measurements acquired from a number of semiconductor systems.

(iv) To characterise devices in the TEM under conditions appropriate to their normal working environment: by recording holograms of semiconductor junctions that are biased electrically *in situ*.

These objectives were achieved within the proposed timescale. The significance of the success of this project is noteworthy because, at the time that the grant was submitted, only one research group had published a detailed study of the use of electron holography to form two-dimensional maps of dopant potentials in transistors. Hence, the stated objectives of this project were highly ambitious and prescient. The actual expenditure matched that specified in the proposal very closely.

Associated Grants. Experimental work for the present project was carried out on a Philips CM300 field emission gun TEM (EPSRC grant GR/L22164), with data analysis carried out in part using a Silicon Graphics Origin 200 workstation (EPSRC grant GR/L54837). Site-specific TEM specimens were prepared using an FEI focused ion beam (FIB) workstation (EPSRC grant GR/L21044). The tomographic work was supported by personnel employed under EPSRC grant GR/R30457.

Key Advances and Supporting Methodology

The proposal made a number of key advances in the subject. We were aided through links with external collaborators, specifically: (a) advice on TEM specimen preparation and novel specimen geometries for electron holography was provided by Professor Simon B Newcomb (Ireland) and by Drs Arpad Barna and Bela Pécz (Hungarian Academy of Sciences); (b) the Semiconductor Physics Group in the Cavendish Laboratory supplied MBE-grown doped GaAs samples; (c), Philips Research (Eindhoven) provided doped Si semiconductor devices. Specific advances were in the areas of:

1. Development of an Ultra-High Tilt Biasing Holder. A novel ultra-high-tilt cartridge-based side-entry two-contact electrical biasing TEM specimen holder was designed, commisoned, built and tested, as a collaborative development project with E. A. Fischione Instruments, Inc. of Export, PA, USA (see Fig.1). This specimen holder allows electrostatic potentials in semiconductor devices to be characterised using electron holography with an electrical bias applied to them *in situ* in the electron microscope. The holder incorporates two primary electrical contacts to the sample, and allows specimen tilts in excess of ±70° to be achieved for the three-dimensional characterisation of electrostatic potentials, by combining electron holography with electron tomography. A further advantage of this holder is that the specimen is held in a universal cartridge, which can be transferred between the TEM specimen holder, the SEM and the FIB workstation, allowing different specimens to be stored in separate cartridges during preparation and examination, as well as for specimen transfer to collaborators. During this project, the specimen holder was used successfully for the characterisation of Si and GaAs *p-n* junctions with an electrical bias applied to them *in situ* in the TEM. These experiments, which are described further below, also required the development of a new specimen geometry, in which FIB milling was used to micro-machine an electron-transparent membrane of uniform thickness at one corner of a cleaved square of the semiconductor wafer, which was placed between the two electrical contacts in the cartridge.

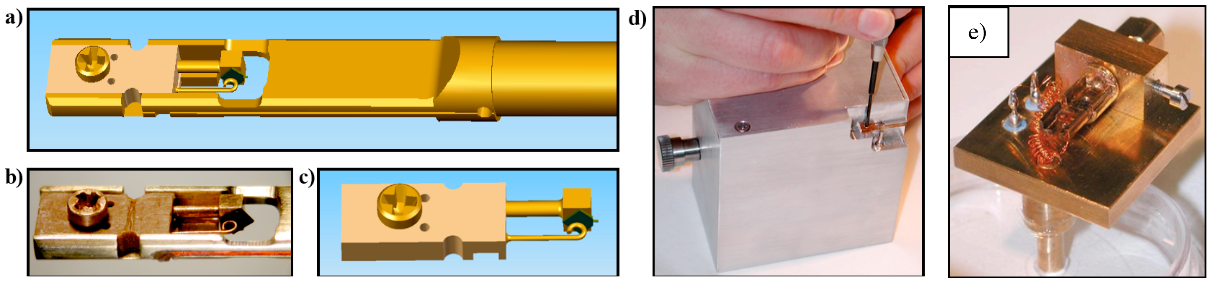
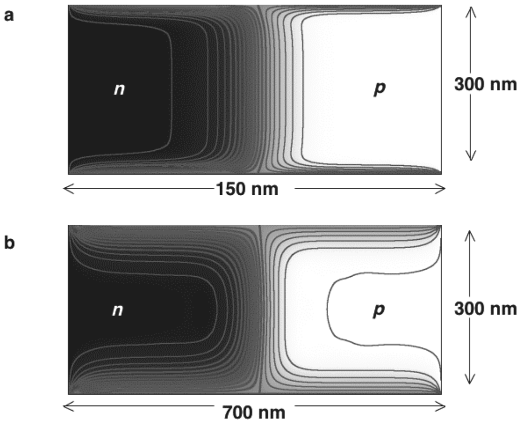


Figure 1 a) Design drawing of the end of the ultra-high-tilt two-contact cartridge-based electrical biasing holder with a sample in place in the cartridge. b) Photograph of the tip of the holder with a cleaved FIB-milled Si sample in the cartridge. c) Design drawing of the cartridge and sample alone. d) The loading station used to pull the spring contact back to load the sample into the cartridge. e) Stub used to mount the cartridge in the FIB workstation or the SEM at any desired angle about the cartridge axis.

2. Simulation Software. Dedicated software was written to simulate electrostatic potentials in parallel-sided thin specimens of doped semiconductors containing *p-n* junctions. These simulations were then compared with experimental electron holographic phase images acquired from FIB-milled Si and GaAs specimens. As electrostatic fringing fields have never been observed experimentally in vacuum outside the positions of *p-n* junctions in FIB-milled specimens, the majority of the simulations incorporated surface states that modelled equipotential sample surfaces. Reassuringly, in the simulations the step in the projected potential across a *p-n* junction was *always* found to be lower than would be predicted from the properties of the bulk device, as observed experimentally. The magnitude of the reduction in phase shift resulting from the presence of the specimen surfaces was found to be insensitive to the surface state energy for dopant concentrations in excess of 10^{17} cm^{-3}. The significance of this observation is that this parameter is not known experimentally, and is likely to depend on the TEM specimen preparation approach used. For dopant concentrations of below 10^{16} cm^{-3}, the simulations showed that not even the central part of a TEM specimen that has a thickness of up to 500 nm retains the electrical properties of the bulk device. The simulations indicated that the reduction in the step in potential across a *p-n* junction observed experimentally in FIB-milled specimens cannot be reproduced by surface depletion alone, and may be accounted for partially by the effects of Ga doping close to the specimen surfaces and defect states in the band-gap of the semiconductor. Accordingly, the removal of these effects of TEM specimen preparation was assessed experimentally, as described below.



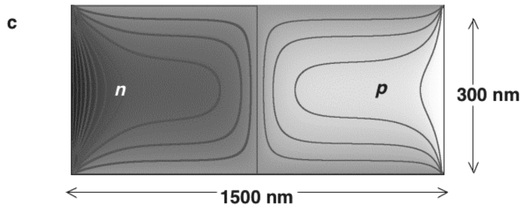


Figure 2 Simulations of electrostatic potential distributions in parallel-sided slabs of thickness 300 nm containing abrupt, symmetrical Si *p-n* junctions formed from a) 10^{19} , b) 10^{18} and c) 10^{17} cm^{-3} of Sb (*n*-type) and B (*p*-type) dopants. The potential at the specimen surfaces was set to be 0.7 eV above the Fermi level. Contours of spacing 0.05 V are shown. The horizontal scale is different in each figure to show the variation in potential close to the position of the junction.

3. Low-Temperature Annealing and Quantification. A systematic examination of electron holographic phase images of unbiased and electrically-biased FIB-milled Si and GaAs *p-n* junctions was carried out, for a range of dopant concentrations. Experimental results indicated that a small fraction of the thickness of each specimen retained the electrical properties of a *p-n* junction, and that both crystalline and amorphous regions close to each specimen surface were electrically inactive, and did not contribute to the measured phase shift across the *p-n* junction. These effects were much more severe for GaAs than for Si specimens. Reassuringly, phase shifts measured from unbiased semiconductors were found to be consistent with those acquired from samples to which electrical contacts were made. Most significantly, it was found that *in situ* low temperature (200-600 °C) annealing removes noise and increases the step in phase shift across a FIB-milled *p-n* junction in both Si and GaAs (see Fig. 3). In GaAs, electrically-inactive surface layer thicknesses are then reduced from 80 to 5 nm on each specimen surface after annealing at 500 °C, while in Si the improvement is from 25 to 5 nm. Although the resulting phase profiles still do not match simulations perfectly, this observation provides a very important step towards the quantitative measurement and interpretation of dopant potentials using electron holography, and the full understanding of the nature of the defects in these specimens.

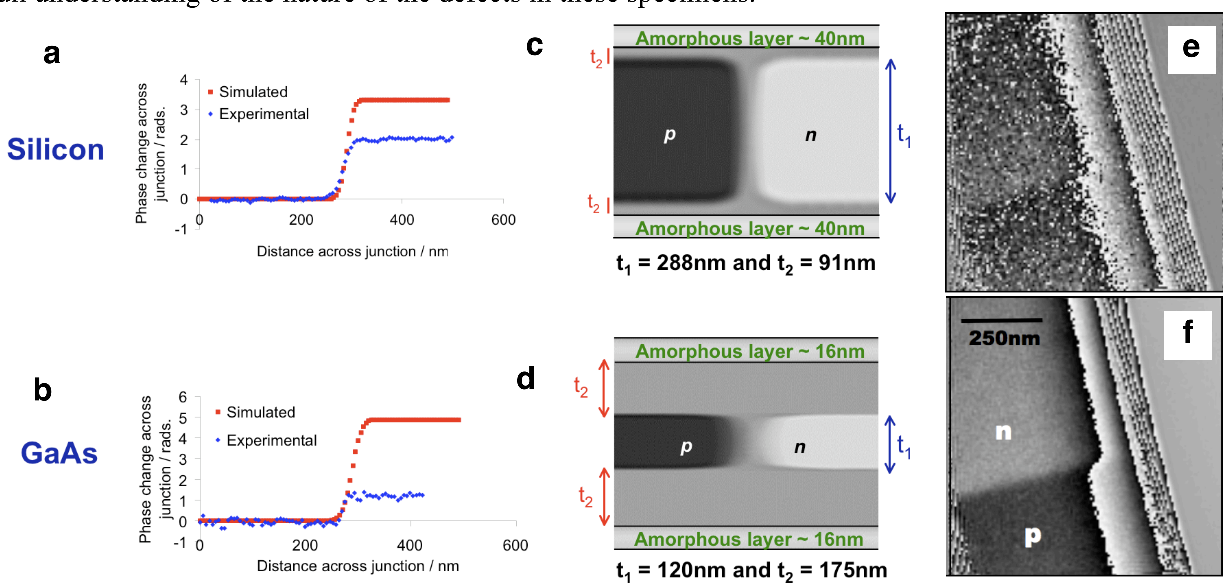


Figure 3 Representative experimental and predicted phase profiles across a *p-n* junction in FIB-milled specimens of a) Si and b) GaAs. c) and d) Schematic diagrams showing, in cross-section, the physical and electrical structures of FIB-milled TEM specimens inferred from the experimental profiles shown in a) and b), respectively. e) and f) show wrapped electron holographic phase images of a FIB-milled GaAs *p-n* junction specimen of crystalline thickness 240 nm, recorded at room temperature e) before and f) after *in situ* annealing at 500 °C.

4. Electron Tomographic Holography. The ultra-high tilt capability of the new specimen holder was used to provide the first reconstruction of the three-dimensional electrostatic potential of a Si *p-n* junction in a TEM specimen. This work involved the analysis of electron holograms of the junction acquired over a specimen tilt range of ±70° in 2° steps. The measured three-dimensional potential matched that expected from simulations closely, and displayed both the electrically active layer and the increase in depletion width close to each specimen surface directly, see Fig. 4.

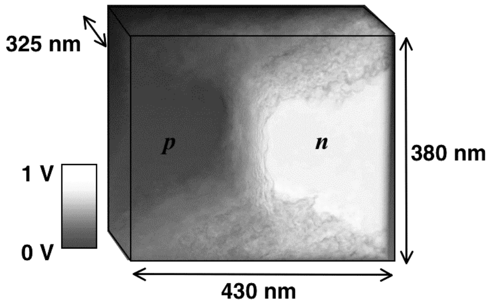


Figure 4 The three-dimensional electrostatic potential of a Si *p-n* junction in a FIB-milled specimen, reconstructed from a tilt series of electron holograms acquired over ±70° in 2° steps. The voxel size is 5.8 nm^3.

5. Fitting Procedures. As a result of noise in experimental phase profiles across *p-n* junctions, Poisson's equation cannot usually be used to infer electric fields or charge densities from phase profiles directly. Hence, computer programs were written to allow empirical simulations to be fitted to experimental phase profiles. The best-fitting simulated profiles, rather than the experimental profiles, were then differentiated to provide charge density profiles, which were used to provide an indication of how specimen preparation had affected the electrical properties of the junction (Fig. 5). Three different empirical models for the electrostatic potential in the specimen were used to make quantitative measurements of depletion widths and electrically active charge densities in specimens containing Si and GaAs *p-n* junctions. The best-fitting model always corresponded to an increased depletion width close to the specimen surfaces, as observed using electron holographic tomography, and in agreement with the first principles computer simulations described above.

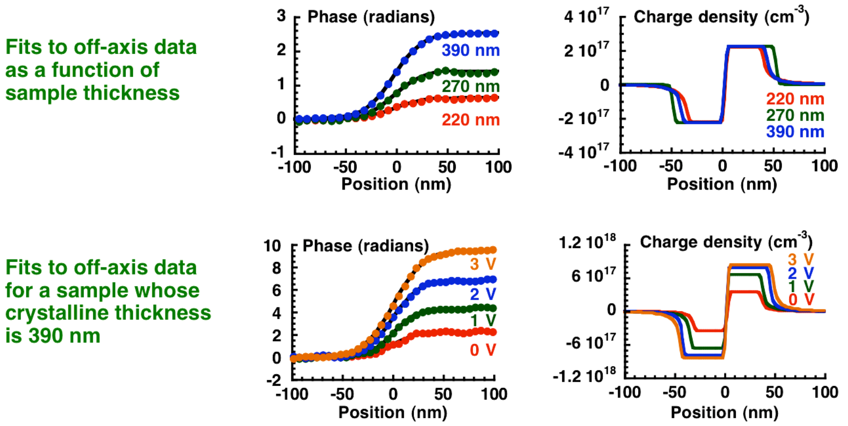


Figure 5 Experimental phase profiles acquired from Si *p-n* junctions in focused ion beam milled specimens, for three unbiased FIB-milled specimens of crystalline thickness 220, 270 and 390 nm (top), and as a function of reverse bias voltage from a single focused ion beam milled specimen whose crystalline thickness was measured to be 390 nm (bottom). The right hand column shows charge density profiles derived from best-fitting simulations to the experimental phase profiles.

6. Specimen Charging. Specimen charging resulting from secondary electron emission during electron irradiation, resulting in the presence of an electrostatic fringing field outside the specimen edge, was studied in FIB-milled semiconductor devices. The charging effects were shown to be removed after the specimen was coated on one side with 20 nm of carbon (Fig. 6). During charging, the electric field in the specimen was shown to be almost equal to its breakdown electric field. The identification and removal of such charging effects is important as dopant potentials in such specimens are undetectable before carbon coating.

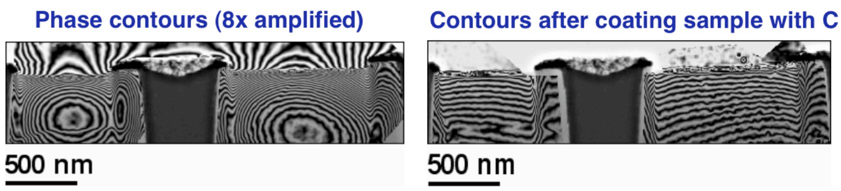


Figure 6 Eight-times-amplified phase contours obtained from a FIB-milled Si transistor specimen of nominal thickness 400 nm, showing W plugs and Si oxide. Specimen charging results in the presence of electrostatic fields outside the specimen edge (left), as well as elliptical phase contours in the oxide layers between the W contacts. After coating the specimen on one side with approximately 20 nm of carbon (right), the effects of charging are removed.

7. In-Line Holography. Experimental energy-filtered in-line electron holography (Fresnel imaging) was carried out to obtain intensity profiles for a range of defoci across *p-n* junctions in Si. Unlike off-axis electron holography, simulations are required to interpret the phase information encoded in the in-line holograms. Through comparison with simulations of in-line holograms and off-axis electron holography results of the same specimens, it was found that a uniform background intensity was present in each defocused image, which had to be removed before a good correlation could be achieved with the simulated profiles for each experimental sample thickness (see Fig. 7). This uniform background is thought to arise from diffuse scattering associated with phonons and point defects, and constitutes a medium resolution equivalent of the 'Stobbs factor' used to explain the contrast mismatch observed in experimental high-resolution electron microscopy. Despite requiring off-axis electron holography results to obtain a good match to the step height of the electrostatic potential variation, this study has shown that in-line holography is an excellent technique for revealing the *shape* of the dopant-related electrostatic potential.

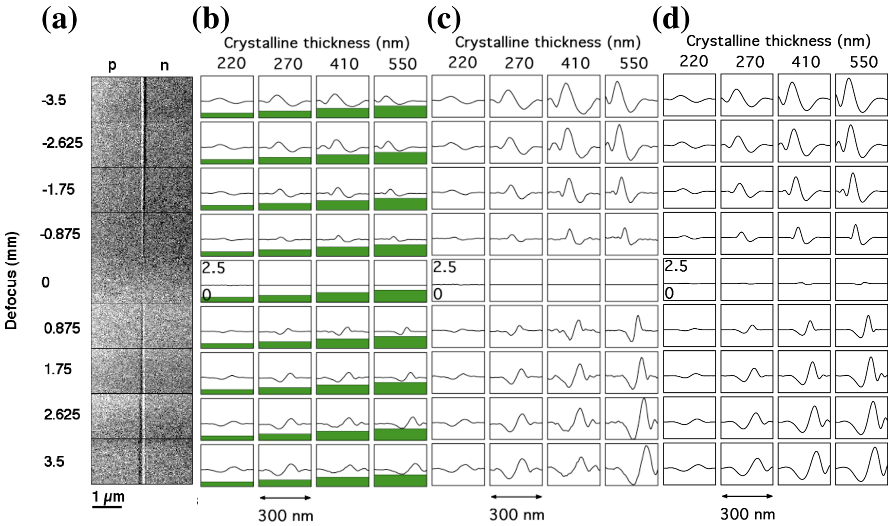


Figure 7 a) A montage of regions extracted from an experimental series of in-line holograms of a FIB prepared Si *p-n* junction. (b) Normalised intensity line profiles obtained by projecting the contrast parallel to the junction for a range of sample thicknesses. The height of the uniform background that needs to be subtracted from each image for the experimental contrast to be consistent with electron holography data from the same samples is shown. c) Experimental line profiles after subtracting the uniform backgrounds shown in b), which show excellent agreement with simulated profiles for the experimental sample thicknesses, shown in (d).

Project Plan Review

This project began on 1 October 2002 and finished on 30 September 2005. In that time, one post-doctoral researcher and one PhD student were employed on the project, as originally planned. During that period the Philips CM300 FEGTEM, the only microscope capable of electron holography in Cambridge, had about 15% downtime (i.e. about 6 months) which is not unusual for microscopes of this type. Nevertheless, we were able to tackle all of the facets of the project proposed originally.

The personnel employed under the grant proposal were augmented by a college Junior Research Fellow and a further student, whose appointments resulted directly from the award of the grant proposal and the success of the initial results. The appointments, together with personnel employed under a separate EPSRC grant (GR/R30457) to develop electron tomography techniques, gave the team a 'critical mass' that enabled the proposed research to be completed on schedule and led to a number of advances beyond those envisaged in the original 3 year proposal, as illustrated by the success of tomographic holography.

Research Impact and Benefits to Society

In order to keep pace with the proposed roadmap for semiconductor device development, the microelectronics industry requires a method to map dopants (or electrostatic potentials which, are sensitive only to *active* dopants) in two, or preferably three, dimensions accurately and reproducibly. We have addressed this pressing need in this proposal and for the most part feel that we have progressed towards meeting many of the industry's demands in this respect. Given the industry's requirement for the site-specific analysis of devices prepared using FIB milling, we have made significant progress towards understanding the effect of such preparation on the electronic properties of the sample. Indeed, we have shown that the modification of the surface has a remarkably long range influence. This observation is clearly of importance not only for understanding the properties of TEM samples but may also govern the properties of next-generation (nanotechnology-driven) 3-dimensional semiconductor devices, fashioned using nano-lithographic methods, in which near-surface properties may dominate device performance.

We have developed new TEM holders that allow the *in situ* electrical biasing of FIB-prepared devices and the examination of devices at high tilts to enable tomographic reconstructions of electrostatic potentials. *In situ* biasing of *p-n* junctions has been key to the interpretation of the properties of the modified device, and has provided a significant step towards the examination of more complex working devices in the electron microscope. We have discovered that *in situ* low temperature annealing can change the properties of FIB-prepared devices dramatically, indicating that such treatment reactivates dopants. In such specimens, dopant atoms may be rendered inactive through being bound to silicon vacancies that are created by cascade events generated by the high-energy gallium ions. Careful simulations have confirmed that surface properties play a crucial role in determining the details of the overall electrostatic potential, at a distance well beyond that envisaged.

We feel that all of the pieces are now in place to generate a reliable methodology for the semiconductor industry to provide quantitative dopant profiles in two and three dimensions across a range of device structures. The research carried out here has centred on relatively simple devices to allow a thorough understanding of their fundamental electronic properties. It is our intention to study more complex structures in the future.

We emphasise again that great progress has been made, and we believe that the ability to interpret and quantify electron holograms will enable progress along the semiconductor roadmap.

Explanation of Expenditure

The majority of the grant was spent as indicated on the proposal. The expenditure can be broken down as below:

Expenditure Category	Budget	Expenditure	Variance
EQUIPMENT	59,165.00	48,036.07	11,128.93
EXCEPTIONAL ITEMS	14,510.00	14,538.00	-28
OTHER COSTS	12,985.00	35,705.03	-22,720.03
OVERHEADS	54,014.00	34,567.51	19,446.49
STAFF COSTS	117,422.00	104,646.68	12,775.32
TRAVEL & SUBSISTENCE	5,246.00	6,402.22	-1,156.22
Project Totals	**263,342.00**	**243,895.51**	**19,446.49**

The major variance from the proposed budget was the extra cost incurred under 'Other Costs', which resulted from an increase in electron microscopy-related expenditure, contributions to service contracts, microscope running costs and software costs, excess conference costs, page charges, etc. Equipment costs (e.g. the purchase of a biasing holder and computer) were broadly in line with the budgeted costs. The travel budget was exceeded by about £1k as the project's success led to many invitations to speak at conferences in the UK and overseas. Some of the Staff costs (about 10%) were vired to other headings. There was an overall underspend of £19.5k.

Further Research and Dissemination Activities

Under the direction of the PI, the Co-investigators and Dr Twitchett, the project will continue to finalise the methodology demonstrated throughout this project, to apply it to more complex CMOS structures and to pursue the application of electron tomographic holography to three-dimensional electronic devices in general. Collaboration with Fischione has continued, and we are discussing the development of new holders and sample preparation equipment (e.g. the Fischione Nanomill) to minimise and mitigate device damage.

Many routes have been used to disseminate our work. We have published, and will continue to publish, in conference proceedings and scientific journals and on the WWW. Invitations to give talks are frequent and this allows further dissemination of our research activities to a more general audience than is found in some journal readerships. The project led directly to 6 published journal papers, a further 2 submitted and at present another 4 in preparation. There were over 20 conference papers, 8 invited and 5 contributed talks at international conferences and 1 award.