INTRODUCTION

Minatec is a multidisciplinary research centre based in Grenoble, by the French Alps. Within Minatec there are research activities focused on the development of the future generations of semiconductor devices, on renewable energy, magnetic storage as well as fundamental research. In addition, we work closely on research and development with larger semiconductor companies such as ST Microelectronics and IBM. In order to support all of these activities, a characterisation platform has been created in order to both provide information on the structure and properties of today’s nanoscaled materials and to ensure that the future needs for materials characterisation are anticipated.

The transmission electron microscope (TEM) is a versatile tool that is indispensable in the semiconductor industry. In conventional TEM, elastically or inelastically scattered electrons are imaged, from which information about the morphology and composition of the specimen can be determined. However, information about the phase of the transmitted electrons is lost. Off-axis electron holography is a powerful technique that can be used to recover the phase of these electrons. As the phase of electrons is sensitive to electrostatic, magnetic and strain fields, then electron holography can be used to provide quantitative maps of these properties with nm-scale resolution [1].

In this article we show how off-axis electron holography has been used to investigate the properties of a range of specimens including doped CMOS devices, doped nanowires, magnetic storage devices and strained SiGe devices.

METHODS

The experiments shown here were performed using the Minatec probe Cs-corrected FEI Titan field-emission gun (FEG) TEM operated at 200 kV with the objective lens switched off and a Lorentz lens used to provide a large field of view. The electron holograms were recorded using a 2048\texttimes\texttimes 2048 pixel Gatan Tridium CCD camera and analysed using Gatan Digital Micrograph.

The CMOS and strained SiGe device samples were prepared by in-situ lift out using a dual beam FEI Strata 400 focused ion beam (FIB) system. The ferromagnetic sample was prepared by wedge polishing. The nanowire samples were prepared by cleaving the wafer to provide high-resolution TEM samples.

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BIOGRAPHY

David Cooper completed his BSc in physics at the University of Liverpool in 1997 and then spent five years in commercial research and development focused on optical and electronic engineering. In 2002 he moved to the University of Cambridge to study for a PhD focused on off-axis electron holography for dopant profiling jointly supervised by Paul Midgley and Rafal Dunin-Borkowski. David then moved to CEA LETI in 2006 where he has worked on developing new techniques for the characterisation of semiconductors using one of the first FEI Titan TEMs.
vide a sample with a large quantity of different wires that could be examined which were all aligned with one another.

**THE BASICS OF OFF-AXIS ELECTRON HOLOGRAPHY**

Off-axis electron holography uses a charged biprism to interfere an object electron wave with a reference wave to form an interference pattern known as a hologram. The electron biprism, usually located in the selected area aperture plane of the TEM, is typically a one-micrometre-thick gold-coated quartz wire.

Figure 1a shows the experimental arrangement for off-axis electron holography. A field-emission gun is required to provide a coherent source of electrons. By applying a voltage to the biprism, two virtual electron sources are formed in the back focal plane to form the electron hologram. By increasing the voltage on the biprism, the virtual sources are pushed further apart which will increase the width of the hologram and decrease the fringe spacing. For medium-resolution electron holography, a Lorentz lens is used instead of a conventional objective lens in order to provide a useful field of view in the range 200 nm to 2 µm.

The holograms are typically recorded on a CCD camera, taking care to preserve the fringe contrast. The holograms are then reconstructed by taking a Fourier transform of the hologram, and selecting a sideband containing the phase information that is carried by the interference fringes. An inverse Fourier transform of this sideband will then provide a complex image from which phase and amplitude images of the specimen can be calculated. From this reconstruction procedure, the spatial resolution of the phase image is usually three times the holographic fringe spacing [2].

By taking advantage of the exceptional electrical and mechanical stability of the latest generation TEMs, electron holography has become much more straightforward. Using these new microscopes it is possible to introduce a sample, load a set of alignments and then perform an experiment with the required stability within an hour or less. Electron holograms can be recorded for time periods of one minute or more to fill the CCD camera while preserving the contrast of the holographic fringes to provide phase images with excellent signal-to-noise ratios [3].

Figure 1b shows an electron hologram with a fringe spacing of 2.0 nm acquired for 64 seconds. Figure 1c shows the measured hologram contrast as a function of the fringe spacing for electron holograms recorded using different magnifications, indicated as the field of view (FOV). In all cases the holograms were recorded for 64 seconds using a 2048 × 2048 pixel CCD camera with an average of 1,000 electron counts in each hologram. A hologram contrast of 20% is considered to be reasonable. These long acquisition times are used routinely on our TEM improving the trade off between spatial resolution and signal-to-noise.

If the specimen of interest is thin and diffraction effects can be minimised, then the phase change of an electron that has passed through the specimen in the direction of the

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**Figure 1:**
Figure 1a: Experimental arrangement for off-axis electron holography. Figure 1b: An electron hologram with a fringe spacing of 2.0 nm acquired for 64 seconds. Figure 1c: Measured hologram contrast as a function of the fringe spacing for electron holograms recorded using different magnifications, indicated as the field of view (FOV).

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**Figure 2:**
A potential map (a) and amplitude image (b) of a 450-nm-thick specimen containing 45-nm gate length CMOS devices. The specimen has been prepared using back-side milling using an FIB operated at 30 kV. The spatial resolution in the potential map is 7.5 nm, the contours show steps of 0.10 V and the noise standard deviation in the Si substrate in the potential map is 0.05 V. Although a physical gate length of 45 nm is measured in the amplitude image, the potential map shows that the doped extension regions have provided an active gate length of 33 nm.

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**Figure 3:**
A TEM image (a) and a phase map (b) of a 60-nm-thick doped Si nanowire. In the phase map, the colour scale has been adjusted to highlight the central part of the nanowire. The approximate edges of the nanowire are shown by the fine dashed lines. The electrical junctions can clearly be seen in the phase images and quantitative information about the dopants can be obtained by comparing the experimental results to simulations.

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**Figure 4:**
Phase image showing the magnetic component of a FePd data storage device revealing the leakage fields in vacuum that interact with the reading head. Here the contours represent steps of ¼ radians.
beam \( z \), is given by:

\[
\phi(x) = C_z \int V(x,z)\,dz - \frac{e}{\hbar} \int A_z(x,z)\,dz
\]

where \( C_z \) is a constant dependent on the operating voltage of the TEM, \( V \) the electrostatic potential and \( A_z \) the component of the magnetic vector potential in the direction of the electron beam. If neither \( V \) nor \( A_z \) varies in the beam direction then this can be simplified to:

\[
\phi(x) = C_z V(x) - \frac{e}{\hbar} B_z(x)
\]

where \( B_z \) is the in-plane component of the magnetic induction.

**DOPANT PROFILING BY OFF-AXIS ELECTRON HOLOGRAPHY**

The principle component of the electrostatic potential is known as the mean inner potential \( V_0 \), which has the value of \(-12 \text{ V} \) for crystalline silicon. In doped semiconductors, there will also be a built-in potential component \( V_b \) across electrical junctions that is typically around \( 1.0 \text{ V} \). As \( V_0 \), is an order of magnitude smaller than the mean inner potential in Si, it is extremely important to be able to have a flat TEM specimen to be able to extract the information that relates only to the dopants. If the thickness of the specimen is known, then a quantitative potential map of the specimen can in principle be calculated directly from the phase image. However, in practice this is more complicated because of artifacts that are introduced during TEM specimen preparation [4].

Requirements for dopant profiling are for an electron transparent, parallel-sided specimen, with a region of vacuum near the region of interest for the reference wave. However, for many specimens in the microelectronics industry, nm-scale site specificity is also required. Focused ion-beam (FIB) milling fulfills all of these requirements. However, this approach also introduces artifacts, such as the inactive thickness, which reduces the effective crystalline thickness of the specimen making quantitative dopant profiling complicated.

The inactive thickness, which arises from physical damage in the crystalline regions of the specimen, is strongly dependent on the dopant concentration in the semiconductors as well as the type and energy of the ions used during preparation [5]. Therefore if ion milling is used then the measured electrical potential will be less than expected.

Figures 2a and 2b show a potential map and an amplitude image of a specimen containing 45-nm gate length CMOS devices. The specimen was prepared using 30 kV backside FIB milling to prevent thickness changes in the region of interest due to differential ion milling under the surface metallisation. In the potential map, the doped regions are visible and the distance between the extension regions in the source and the drains can be measured. No contrast can be seen in the amplitude image indicating that the effects of strain have been minimised by tilting the specimen to a weakly-diffracting orientation as close to the zone axis as possible. To minimise the effects of the inactive thickness, a thick TEM specimen of 450 nm has been examined, leading to an error of 0.05 V in the potential map. The contours indicate steps of 0.1 V and the spatial resolution here is around 7.5 nm. By taking advantage of the latest developments of specimen preparation including low-energy FIB milling and subsequent cleaning using low-energy inert ions, specimens can be prepared with significantly reduced damage. This permits thinner specimens to be examined with fewer projection effects during TEM examination, better signal-to-noise in the potential maps and the possibility to use finer hologram fringe spacings to improve the spatial resolution. It should also be noted that the research groups at Arizona State University and IBM have used only wedge polishing to prepare specimens leading to fully quantitative dopant maps with spatial resolution of between 2 and 3 nm [6].

Doped nanowires are used in many applications from solar cells to lasers, however it has proved difficult to directly characterise the dopants. In principle, the study of these nanosstructures is straightforward as ion milling is required. However, due to their small dimensions, only a small step in phase is expected across the doped regions. By taking advantage of the stability of our TEM, electron holograms are acquired for 64 seconds in order to provide phase images with an adequate signal-to-noise ratio in order to successfully locate not only the position and abruptness of the electrical junctions, but also quantitative measurements of the dopant concentration. Figures 3a and 3b show a brightfield TEM image and a phase map of a Si nanowire respectively, the dopant concentrations are indicated. Although the thickness of the nanowire changes in the differently doped regions, it was shown that the \( V_c \) and \( V_0 \) components could be separated and that the electrical junctions could be delineated for a range of dopant concentrations [7].

**ELECTRON HOLOGRAPHY FOR MAGNETIC IMAGING**

Off-axis electron holography has been used to measure the magnetic fields in nanostuctures since the pioneering work of Tonomura in the early 1980s and the literature is full of reports of successful characterisation of magnetic samples. There is an increasing demand for characterisation of nanoscaled magnetic structures for applications such as data storage. Inside a hard disk, the information is stored using the magnetisation of a material to create a leakage field that is read by the reading head. Figure 4 shows the magnetic component of the phase for a cross-section of a ferromagnetic sample comprising a hard FePd alloy that is chemically ordered, giving rise to an out-of-plane magnetisation, and a soft FePd alloy, with in-plane magnetisation, deposited on Pd on a MgO substrate. The contours show that the field lines and information about the leakage field in the vacuum can be determined.
quantiatively with nm-scale resolution from the phase image [8].

**STRAIN MAPPING WITH NANO-METRE-SCALE RESOLUTION**

Strain is now routinely applied to semiconductor devices in order to increase the mobility of carriers in the conduction channel. However until recently, no suitable technique existed to measure strain maps in these devices with the required spatial resolution.

Darkfield electron holography is a technique that was recently developed by Martin Hytch and co-workers at CEMES [9]. For darkfield electron holography, the electron biprism is used to interfere an electron wave that has passed through a region of interest with a wave that has passed through an unstrained reference region such as the substrate. An objective aperture is then used to select a diffracted beam corresponding to the strained lattice planes of interest. A strain map is then calculated from the reconstructed phase image using Geometrical Phase Analysis. The Titan is well adapted for this task as the dark field holograms are of low intensity. It has been shown that by using long acquisition times to record the holograms, fully quantitative strain maps can be measured with a sensitivity of $\pm 0.02\%$ [10]. However, for dark holography, specimen preparation is the most critical step and perfect parallel-sided lamellas with no bending are required in order to provide the strain maps.

Figure 5 shows how darkfield electron holography has been applied to two SiGe device structures with different gate lengths and Ge concentrations in order to assess how the values of compressive strain under the gate vary. From the high-resolution images the gate lengths of 38 and 27 nm can be measured. The strain maps for the $\langle 220 \rangle$ direction are shown and profiles can be extracted from directly under the gate into the substrate and the compressive strain in the channel can be determined. The different strain profiles that are shown for the two devices were acquired at different times, from different devices in different TEM specimens giving an idea of the reproducibility of the technique. In addition, these profiles are averaged over only 3 nm. Although it is relatively straightforward to simulate the strain in simple device structures it is much more difficult to assess how device processing affects the strain. Due to the excellent reproducibility of the results in darkfield holography, it has been possible to track the changes in strained device structures during the silicidation process.

Figure 6 shows high-resolution images, strain maps and profiles for a SiGe device specimen during the silicidation process. Different samples with the same geometry as seen in Figure 5a were prepared including one with a 9-nm-thick layer of Ni deposited onto the specimen surface and another with a Ni layer which was then annealed at 420°C in order to form a mono-phase NiSi region and provide good quality, low resistance electrical contact regions on the SiGe devices. It can be seen from the high-resolution images that the SiGe regions have reacted with the Ni during the annealing process. However from the $\langle 220 \rangle$ strain maps, it can be seen directly that the annealing process also has a large effect on the strain in the conduction channel. Just the deposition of the Ni layer reduces the strain directly under the gate from 1.6 to 1.4% and annealing at 420°C reduces the strain to 0.7%. Darkfield electron holography is extremely well suited for the measurement of strain for the semiconductor industry as quantitative maps with nm-scale resolution with a large field of view can be calculated from a single strain map. Importantly, once the effects of specimen relaxation are accounted for by using finite element simulations, this technique is fully quantitative and compatible with FIB milling. Already this new technique is being applied to a range of specimens in the microelectronics industry to solve materials problems for both conventional CMOS technologies and silicon on insulator (SOI) devices.

**CONCLUSIONS**

Off-axis electron holography is now used routinely to map the electrostatic, magnetic and strain fields in semiconductor specimens with nm-scale resolution. Advances in the stability of state-of-the-art TEM’s as well as improvements in specimen preparation by ion milling have improved both the accuracy and precision of the technique. In addition, an improvement in the spatial resolution is expected with the next generation of high-coherence electron sources that are just coming into use. The future is bright for electron holography!

**REFERENCES**