

Chapter 1

Active Dopant Profiling in the TEM by Off-Axis Electron Holography

1.1. Introduction

Electron holography is a powerful transmission electron microscopy (TEM)-based technique that can be used to measure the phase change of an electron wave that has passed through a region of interest compared to the phase of an electron wave that has passed through only a vacuum. As the phase of an electron is sensitive to the magnetic, electrostatic and strain fields that can be found in and around a specimen, electron holography is a unique method that can be used to recover all of these properties with nanometer-scale resolution. The electrostatic potential in semiconductor materials is modified by the presence of active dopants. At this time, when only a few dopant atoms can affect the properties of an electronic device, electron holography provides a unique opportunity to look inside these devices and to learn about the activity of the dopant atoms. Characterization techniques such as secondary ion mass spectrometry and atom probe tomography cannot differentiate between active and inactive dopants. Other techniques such as scanning capacitance microscopy and scanning spreading resistance microscopy, which are capable of measuring the active dopants at the surface of specimens, may well have problems adapting to the latest generations of semiconductor materials that can consist of doped nanowires and three-dimensional structures. Therefore, electron holography is unique in that it allows the position of active dopants to be measured inside a specimen with 1 nm spatial resolution today [COO 11], and potentially atomic resolution in the future.

It was Gabor who introduced electron holography in his paper “Microscopy by Reconstructed Wavefronts” in 1948 [GAB 48]. Gabor realized that the measurement of the phase of an electron beam would allow the aberrations of an optical system to be eliminated. These ideas have been used in what is now known as high-resolution electron holography that have provided the first examples of sub-Ångström imaging [ORC 95]. Today, electron holography is used to describe any method that allows both the amplitude and phase information that is contained in an electron wave to be reconstructed. There are many different methods for performing electron holography, notably in-line holography that has been successfully used for the characterization of strain, dopant and magnetic fields. However, it is off-axis electron holography that is the most widely used. For simplicity, from now on, it will be referred to as electron holography. Here, a Mollenstedt–Duker biprism is used; this is a charged wire, normally located in the selected area aperture plane in a microscope. The biprism is used to tilt a reference wave so that it interferes with an object wave to provide an interference pattern in the image plane. From this interference pattern, which is also known as the electron hologram, the phase of the electron wave can be reconstructed. It was not until the 1980s when groups led by Tonomura, Pozzi and Lichte began to successfully use electron holography to solve materials science problems. However, the invention of stable and coherent electron sources in the 1990s finally allowed electron holography to become more widespread. Indeed, using the latest, ultrastable electron microscopes in 2012, electron holography has become a much more user-friendly technique that provides the microscopist with wonderful opportunities to solve materials science problems that are not available elsewhere.

This chapter is designed to show the reader how to perform electron holography in a transmission electron microscope and then how to use electron holography for dopant profiling. There are many books and reviews that deal with the theory and background in detail that should be consulted for a more complete discussion of the aspects discussed here. This chapter is designed to provide a “hands-on” approach regarding electron holography that will allow the readers to be able to get the most out of their microscope and avoid many of the common and not-so-well-known problems that can be encountered when performing electron holography.

Experimental results have been used to illustrate everything that is discussed here. The experimental conditions have been kept as constant as possible. All examples shown here were acquired using an FEI Titan TEM operated at 200 kV. Unless otherwise discussed, the Lorentz lens was used with the conventional objective lens switched off. Although the microscope used here has a probe corrector, it was not used. The presence of the probe corrector meant that the third conventional lens was switched off in order to be able to achieve the astigmatism that is required for electron holography. For recording the electron holograms, a charge-coupled device (CCD) camera attached to a Gatan energy filter was used.

This provides convenience as the image is observed at a low magnification on the TEM viewing screen to allow the whole of the sample, beam and biprism to be observed at the same time with the additional magnification then provided in the energy filter. In addition, the energy filter can be used to improve the hologram contrast. Unless otherwise stated, a $2,048 \times 2,048$ pixel CCD camera was used in “double binning” mode to provide $1,024 \times 1,024$ pixel images. Although the examples shown were acquired using a Titan TEM, everything discussed in this chapter can be transferred to any other type of TEM that is equipped with an electron biprism in the selected area plane.

1.2. The Basics: from electron waves to phase images

1.2.1. *Electron holography for the measurement of electromagnetic fields*

The phase of an electron wave that has passed through a specimen will be changed by the electromagnetic field. This phase change is given by:

$$\phi(x) = C_E \int V(x, z) dz - \frac{e}{\hbar} \iint B_{\perp}(x, z) dx dz$$

where z is the direction of the incident electron beam, x is the direction in the plane of the specimen, V_0 is the electrostatic potential and $B_{\perp}$ is the component of the magnetic induction that is perpendicular to both x and z [TON 87]. When examining specimens containing dopants, it is assumed that there is no magnetic field present. For the measurement of electrostatic potentials, the interaction constant, C_E is given by:

$$C_E = \frac{2\pi}{\lambda} \frac{E_k + E_0}{E_k(E_k + 2E_0)}$$

where λ is the electron wavelength, E_0 is the rest energy of the electron and E_k is the kinetic energy of the electron. The interaction constant is 7.29×10^6 rads $V^{-1}m^{-1}$ for 200 kV electrons and 6.53×10^6 rads $V^{-1}m^{-1}$ for 300 kV electrons. Figure 1.1 shows C_E plotted for a range of microscope operating voltages revealing that the incident electrons interact more with the electrostatic potential at lower energies.

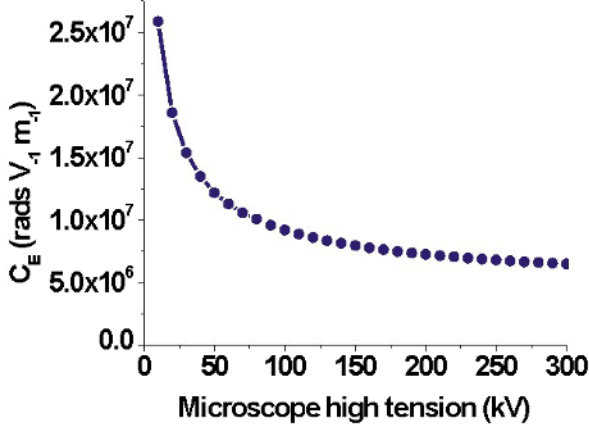


Figure 1.1. C_E as a function of the energy of the electron beam.

Following the notation of Hytch, when understanding the origin of the different phases that are measured by electron holography, we can write the phase as having four different components [HYT 11].

$$\phi_g(r) = \phi_g^G(r) + \phi_g^C(r) + \phi_g^M(r) + \phi_g^E(r)$$

where ϕ^G refers to the geometric phase that describes the distortion from the crystal lattice, ϕ^C refers to the crystalline phase resulting from the scattering of electrons from the crystal potential, ϕ^M is the magnetic contribution and ϕ^E is the contribution from the electrical fields in and around the specimen. For the purpose of this chapter, which concentrates on dopant profiling by electron holography, we will assume that the specimen is both non-magnetic and has been tilted to a weakly diffracting orientation and will only be concerned with the term $\phi_g^E(r)$. Within this term, the measured phase will have two components, the mean inner potential (MIP) V_0 and the dopant-related potential V_E .

$$V^E(r) = V_0(r) + V_E(r)$$

The MIP is defined as the volume average of the electrostatic potential in a specimen. The MIP can be calculated by using a non-binding approximation, which considers the sample as an array of neutral atoms and gives an upper limit, as it does not account for the distribution of valence electrons due to bonding. The electron

scattering factors, f_{el} for each atom in the volume, Ω can be summed over the unit cell [REI 89].

$$V_0 = \left(\frac{h^2}{2\pi me\Omega} \right) \sum_{\Omega} f_{el}(0)$$

For example, to calculate the MIP in GaAs, the scattering factors can be looked up and are 7.143 nm^{-1} for Ga and 7.3686 nm^{-1} for As [REZ 94]. Given that there are four of each type of atom in a unit cell with a lattice parameter 0.5653 nm , the MIP can easily be calculated.

$$\frac{6.626 \times 10^{-34}}{2\pi \times 9.109 \times 10^{-31} \times 1.602 \times 10^{-19}} \times \frac{(4 \times 7.143 \times 10^{-9}) + (4 \times 7.369 \times 10^{-9})}{(0.5653 \times 10^{-9})^3} = 15.4 \text{ V}$$

A lower limit for the MIP can also be calculated by adding the contribution of the valence elections to the non-binding approximation [BET 28, RAD 70].

The MIP can be measured directly by electron holography, and values that have been measured experimentally for well-known materials are 11.9 V for Si, 10.1 V for SiO_2 and 14.5 V for GaAs [KRU 06]. What is important for dopant profiling is that the values of mean inner potential present in most semiconductors are at least an order of magnitude larger than expected in an electrical junction. Thus, very small changes in specimen thickness can completely mask the potential measured due to the presence of active dopants.

The MIP is sensitive to the charge density distributions that are related to the bonding and ionicity in a crystal. As such, electron holography has been successfully used to visualize these polarization fields in a range of materials, for example for nitrides, such as GaN, InGaN and AlGaN, and also in ferroelectric perovskites. For more detail on this subject, the work of the groups in Arizona led by Molly McCartney [MCC 07] and in Dresden led by Hannes Lichte [LIC 07] should be referenced.

As well as the MIP, the active dopants present in the material will additionally contribute toward the measured phase change. Therefore, in a homogeneous material of constant thickness, if sufficient care is taken, the phase image will be directly related to the distribution of active dopant atoms.

1.2.2. The electron source

For performing off-axis electron holography, a stable electron microscope equipped with a field emission gun (FEG) is required. The FEG provides a bright, stable, coherent source of electrons to form an interference pattern. The spatial coherence is described by the size of the emitting area, and the smaller this area, the brighter the source. The brightness, β , is defined as the current density per unit solid angle of the source, and it can be measured if the emission current i_e , the diameter of the focused beam d_0 and the convergence semi-angle of the beam from the condenser aperture α_0 are known [ALL 99].

$$\beta = \frac{4i_e}{(\pi d_0 \alpha_0)^2}$$

The contrast, μ , of the interference fringes can be related to the brightness of the source where I_{coh} corresponds to the coherent current and λ , the wavelength of the incident electrons.

$$I_{\text{coh}} = \frac{-\beta \ln \mu}{2\pi / \lambda^2}$$

Thus, the brightness of the source is directly proportional to the coherent current. A brighter source will improve an electron microscope's ability to form higher contrast holograms, thus improving the signal to noise ratio for an identical hologram acquisition period.

Temporal coherence is dependent on the natural energy spread of the electron beam and on fluctuations in the beam energy caused by noise in the high-tension voltage supply and objective lens current. Temporal coherence can be improved using low-noise power supplies and a well-designed room environment. However, in most modern TEMs, for medium resolution electron holography, it is spatial coherence that dominates the hologram forming properties of the microscope. Thus, the interference fringes can be optimized by choosing beam settings that use electrons emitted from the very center of the electron source.

1.2.3. Forming electron holograms using an electron biprism

Figure 1.2 shows a schematic for electron holography. A coherent electron wave passes through a sample and is interfered with by an electron wave that has passed through only a vacuum by using an electron biprism. The biprism acts to create two

virtual sources, S1 and S2. These virtual sources can be thought of as being comparable to Young's slits experiment.

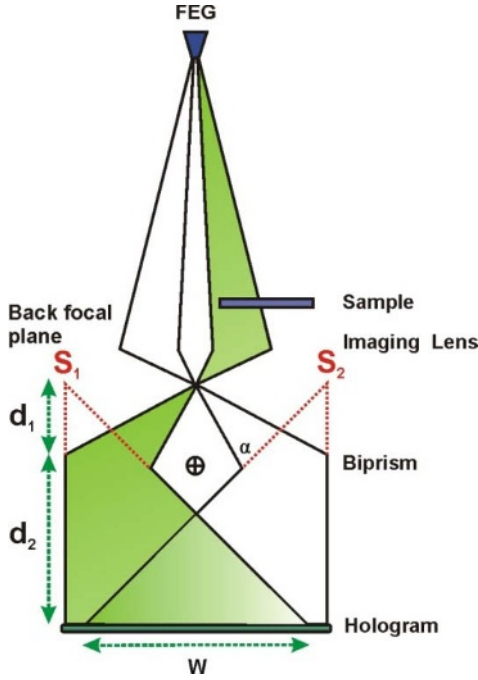


Figure 1.2. Schematic diagram showing the effect of the biprism in forming an electron hologram

Increasing the biprism voltage will increase the width of the interference pattern by moving the sources further apart. However, the limited coherence of the source places will affect the contrast of the interference fringes, as the electrons must then travel a greater distance to form the interference pattern. The width of the hologram, W , is:

$$W = 2 \left(\frac{d_1 + d_2}{d_1} \right) \left(\alpha \frac{d_1 d_2}{d_1 + d_2} - R \right)$$

where α is the deflection angle of the electron wave due to the voltage applied to the biprism and R is the radius of the biprism [MIS 81]. From the equation, it is clear that the biprism should be as narrow as possible. The voltage that must be applied to the wire to achieve the overlap between the two parts of the electron wave on either side of it is directly proportional to the diameter of the wire. However, the increasing

separation of the virtual sources required to push the biprism shadow out of the field of view will reduce the contrast quality in the hologram due to the limited coherence of the source. The biprism edge will also lead to Fresnel diffraction at the edges of the hologram, but this can be removed from the field of view by increasing the applied voltage, again at the expense of fringe contrast. Figure 1.3 shows the formation of an electron hologram at different biprism voltages. At zero volts, the biprism can be seen in the center of the pattern and Fresnel fringes are clearly observed coming from each side of the biprism. As the biprism voltage is increased, the waves begin to overlap and hologram fringes are formed. At higher voltages, finer fringes and wider patterns are observed. For the hologram formed using a biprism voltage of 50 V, several regular and fine fringes can be seen. The fringes in the center of the pattern can contain information about the amplitude and phase shift of the electron wave that has passed through the region of interest. In very simple terms, information about the phase change of the electrons can be determined from the shifts of the position of the interference fringes. The amplitude can be determined from the changes in the intensity of the fringes.

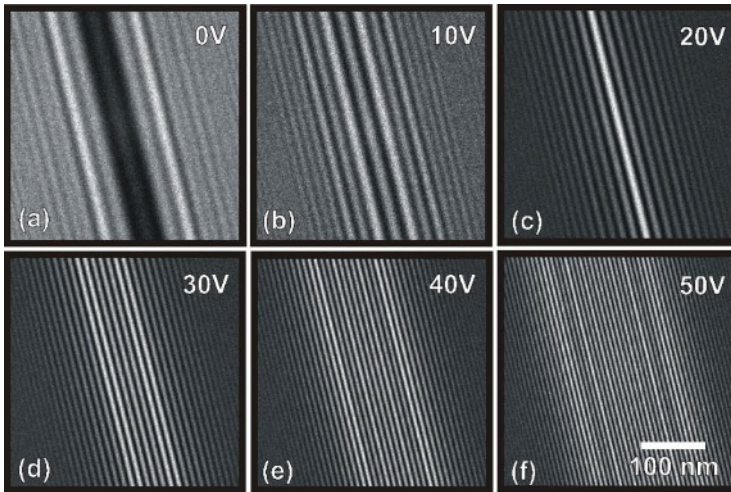


Figure 1.3. *Electron holograms acquired using different voltages applied to the biprism*

The interference fringe spacing, s , provides the spatial resolution obtainable from a hologram and can be calculated by

$$s = \lambda \left(\frac{d_1 + d_2}{2\alpha d_1} \right)$$

By increasing the voltage on the biprism, the width of the interference pattern will increase and the fringe spacing will decrease. Figures 1.4(a) and (b) show experimentally measured values of the width and fringe spacings for a range of biprism voltages that may be used in Lorentz mode electron holography. The hologram width is measured as the distance between the centers of the large Fresnel fringes at each side of the electron hologram. This is different to the field of view that describes the area of the electron hologram captured by the CCD camera. As will be shown, in practice, it is a good idea to match the size of the hologram to the CCD camera.

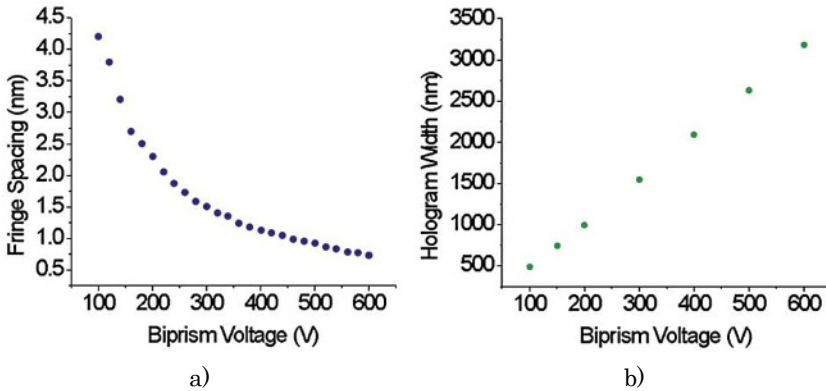


Figure 1.4. *Fringe spacing and field of view of the electron hologram for different biprism voltages typically used in Lorentz mode electron holography*

To perform electron holography, first, the beam alignments must be made as would be performed for high-resolution TEM. The principal difference is that holography is performed using an astigmatic beam. As the holographic fringes are parallel to the biprism wire, coherence is only required in one direction. By deliberately misadjusting the condenser stigmators, highly astigmatic illumination can be obtained that increases the coherent electron flux in the direction of interest. Figure 1.5(a) shows an image of a typical beam setting used for electron holography. Here, one of the stigmators has been set to 100% and the second stigmator has been used to align the major axis of the illumination at exactly 90° to the biprism to maximize the fringe contrast. It is important to take care at this step and make sure that the illumination is well aligned. Figures 1.5(b) and 1.5(c) show the beam focused using C_2 onto the electron biprism at 100% stigmation close to perfect alignment and at perfect alignment, respectively. For perfect alignment, it is clear that the fringes are continuous through the biprism. As the beam is kept astigmatic during the holography experiments, then care must be taken to ensure that the image of the sample is not distorted.

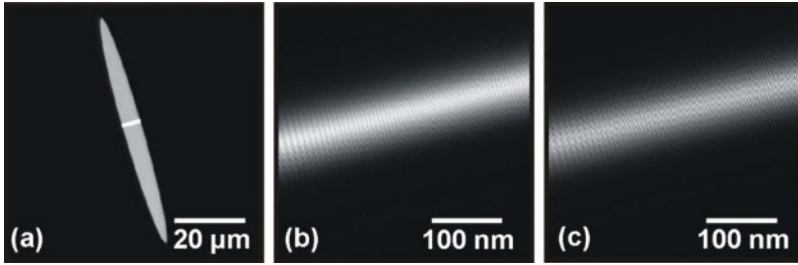


Figure 1.5. *a) An image of the typical stigmation used for electron holography experiments. b) The electron beam has been focused onto the biprism using C_2 , both close to perfect alignment and c) at perfect alignment*

The fringe contrast, μ , of the holograms is an important parameter that affects the ultimate phase sensitivity that can be achieved. The fringe contrast is measured using the formula shown below and for Lorentz mode holography; values of 20% or more should be obtained in a carefully aligned electron microscope.

$$\mu = \frac{I_{\max} - I_{\min}}{I_{\max} + I_{\min}}$$

1.2.4. Care of the electron biprism

The electron biprism can be easily damaged and it is important to take care of it. Never allow the electron beam to be incident on the biprism unless it is either grounded or connected to the power supply. Also, never focus the beam onto the biprism unless the beam is fully stigmated as this can cause damage. When changing beam settings or magnifications, it is a good idea to remove the biprism from the field of view as the electron beam can be focused down the column by changes in lens currents during these steps. If the biprism is stable and the microscope is performing well for electron holography, then it is a good idea to leave the biprism in the column and instead close the column valve when making changes to the beam settings. It is important to look after the biprism, as a clean and undamaged wire is much more stable and easy to use. For a modern, stable microscope, the biprism is often a weak link, typical biprisms are mechanical and over time the mechanism will become loose. Treat this mechanism with care. When making adjustments to the position of the biprism, if slight mechanical resistance is felt in the mechanism then this suggests that an orientation has been found that can be more stable. Lightly tapping the biprism mechanism will sometimes allow it to stabilize. In addition, the biprism will have some rotational positions that are much more stable than others. It

is well worth testing the stability of the biprism with different angles of rotation. Finding stability can be a very frustrating exercise and is certainly not an “exact science”, sometimes the microscope will be immediately stable despite changing from different modes and high tensions, and sometimes it can take a few hours to find the best stability.

1.2.5. Recording electron holograms

Originally, photographic film was used in electron holography to record the images, combined with an optical reconstruction process. Not only was this approach very time-consuming, but factors such as the nonlinear response of the photographic film and optical artifacts introduced during the reconstruction process introduced additional complications. The development of the CCD camera and powerful computers for hologram reconstruction allows electron holograms to be reconstructed quickly and easily.

The CCD camera has a linear response to incident electrons over a wide dynamic range, which makes it indispensable for quantitative TEM. CCD cameras are typically 1,024–4,096 pixels² in size. Cameras with a small number of pixels will limit the number of fringes that can be recorded for a given magnification, thus the field of view. Using a detector with more pixels to improve spatial resolution will be at the expense of intensity and therefore signal to noise. A perfect detector will record every electron in the correct position, but for real detectors, cross-talk between pixels is present. Incident electrons enter the scintillator to generate photons. These photons are then transmitted to the CCD pixels using fiber optics. As the light emission generated in the scintillator can be four times larger than an individual CCD pixel, which is typically 25 μm^2 , then a significant amount of cross-talk should be expected. The limitations placed on the spatial resolution from cross-talk should be considered when setting up the microscope. The modulation transfer function (MTF) describes the spatial transfer of information related to the cross-talk of the CCD camera and this can be measured for a given CCD system [DE 95].

The detection quantum efficiency (DQE) measures the efficiency of signal output for each incident electron. A perfect detector will have a DQE of 1. A typical CCD has a DQE of 0.8 at an electron dose of between 1 and 1,000 per pixel, making the CCD very effective at recording incident electrons. Figure 1.6 shows the effect of acquisition time on the formation of an electron hologram. The holograms have been acquired for (a) 0.01 s, (b) 0.1 s, (c) 1.0 s and (d) 10 s. Clearly for an electron microscope using a standard FEG, at least 1 s and preferably more, is required in order to build up an electron hologram on a standard CCD camera. Therefore, dynamic electron holography experiments are at this time limited to time periods of seconds.

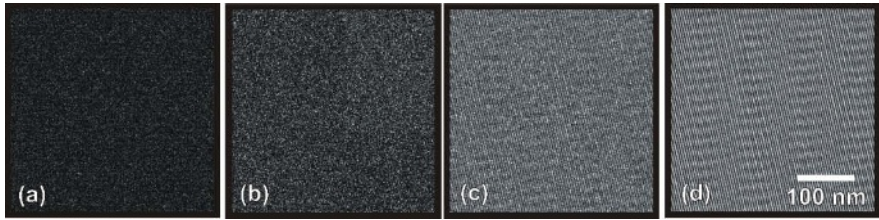


Figure 1.6. Electron holograms with a field of view of 360 nm and a fringe spacing of 2 nm using acquired for time periods of a) 0.01 s, b) 0.1 s, c) 1.0 s and d) 10 s

The CCD and imaging lenses in the microscope have distortions associated with them. Assuming they are stable, these distortions can be corrected from the hologram by carefully removing the sample from the field of view and obtaining a reference hologram. The complex division of the reconstructed sample and reference waves then reveals a distortion-free phase image. Figure 1.7(a) shows a phase image of an n-channel metal-oxide-semiconductor field effect transistor (nMOS) device specimen reconstructed using only the object hologram. Figure 1.7(b) shows the phase of only in vacuum with the specimen moved out of the field of view. This has been reconstructed from what is known as the reference hologram. In Figure 1.7(c), an nMOS device, reconstructed using both the reference holograms, can be seen and the distortions that are observed in (a) have been removed.

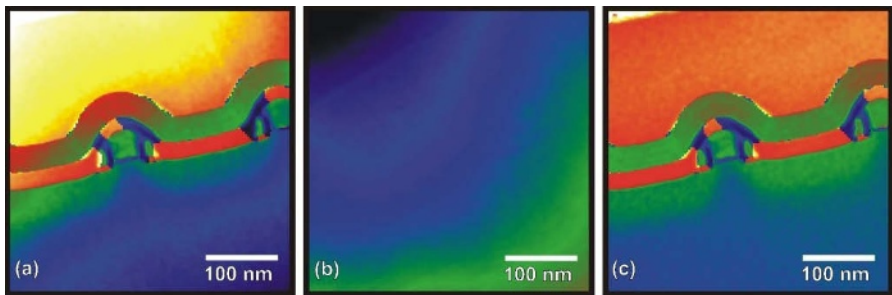


Figure 1.7. a) Phase image of an nMOS device reconstructed without a reference hologram. b) Phase image reconstructed using an empty reference hologram showing the distortions that are present in the imaging system and CCD camera. c) Phase image reconstructed with the use of the reference hologram

1.2.6. Hologram reconstruction

To recover the information about the amplitude, A , and phase, ϕ , of the electrons, a hologram reconstruction process is required. To reconstruct a hologram with fringe

spacing q_c , a Fourier transform is performed. The complex Fourier transform of the hologram is given by the expression:

$$\begin{aligned} FT[I_{hol}(\mathbf{r})] &= \delta(\mathbf{q}) + FT[A^2(\mathbf{r})] \\ &+ \delta(\mathbf{q} + \mathbf{q}_c) \otimes FT[A(\mathbf{r}) \exp[i\phi(\mathbf{r})]] \\ &+ \delta(\mathbf{q} - \mathbf{q}_c) \otimes FT[A(\mathbf{r}) \exp[-i\phi(\mathbf{r})]] \end{aligned}$$

This expression contains four terms, two peaks at the origin ($q = 0$) corresponding to the Fourier transform of the uniform intensity of the reference image and the Fourier transform of the intensity distribution of the normal TEM image, and two sidebands centered on ($q = -q_c$) and ($q = +q_c$) comprising the Fourier transform of the desired image wavefunction and the Fourier transform of the complex conjugate of the image wavefunction. The two sidebands contain identical information except for a change in the sign of the phase. It is also useful to multiply the hologram by a two-dimensional Hanning window to reduce the appearance of streaks in the Fourier transform arising from the mismatch between the intensity at each edge of the hologram. To obtain the phase image, a sideband is carefully selected and moved to the origin of Fourier space where an inverse Fourier transform is applied. Increasing the size of the mask used to select the sideband will give higher spatial resolution but will also introduce additional noise into the image. As the highest spatial frequencies are not always required, a reduction of the mask radius can be used to remove high-frequency noise. The edges of the mask should be diffuse to minimize any effects due to the abrupt loss of information near its edges. For a strongly scattering object, the maximum radius for the mask is one-third of the carrier frequency, as the radius of the center band is twice that of the sideband. Therefore, the spatial resolution in a reconstructed phase image is typically three times that of the fringe spacing [ALL 99].

If the center of the sideband is not accurately selected, then an artificial gradient can be introduced into the reconstructed phase image. However, the sideband center can be more accurately determined from the Fourier transform of a reference hologram, whose fringe pattern has not been perturbed by the specimen. The same position for the reference sideband and the object sideband can be used. The inverse Fourier transform of the sideband generates a complex image, from which amplitude and phase images can be obtained by using the equations below, where A is the amplitude, ϕ the phase, $\Im$ the imaginary component of the complex reconstructed image and $\Re$ the real component.

$$A(x, y) = \sqrt{\Re^2 + \Im^2}$$

$$\phi(x, y) = \tan^{-1} \left(\frac{\Im}{\Re} \right)$$

Figure 1.8 shows a schematic diagram of the hologram reconstruction process, for a hologram acquired from a symmetrical Si p-n junction containing dopant concentrations of $2 \times 10^{18} \text{ cm}^{-3}$. In the phase image, the n-type region appears brighter than the p-type region. As the dopant concentration is very low compared to the silicon atoms (0.0004%), no contrast is observed in the amplitude image due to the presence of the dopant atoms.

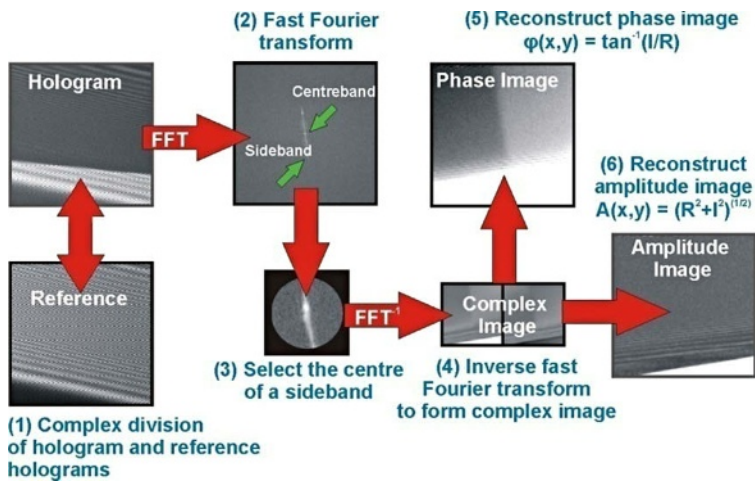


Figure 1.8. Schematic of the reconstruction procedure used for electron holography

There are many different software packages that are available for performing the reconstruction of electron holograms, these include Holoworks and HolagraFREE, which are both digital micrograph plug-ins. SEMPER is another software package that contains holography routines and can be used a Linux workstation. All of these packages contain methods to reconstruct and unwrap phase images from an electron hologram. Of course, it is relatively straightforward to perform the appropriate mathematical functions directly to the electron holograms.

One of the more tricky aspects of the reconstruction procedure can be finding the correct sideband to determine the correct sign for the phase images. By convention, the MIP is positive. Therefore, the phase measured in the specimen needs to be positive relative to the vacuum region. This can be complicated for focused ion beam (FIB)-prepared specimens as the rapid change in the thickness from the vacuum to the specimen leads to a phase change of many multiples of 2π in only a

few CCD pixels. This makes it difficult to link the measured phase in the sample to the vacuum region. Usually it is possible to find a feature on the sample edge to provide a clue as to which is the correct sideband to use.

1.2.7. Phase Jumps

A phase image is initially reconstructed with phase discontinuities that are unrelated to the specimen features when the phase changes by 2π . Phase jumps occur when the phase change in the region of interest is more than 2π and “phase unwrapping” is required. Figure 1.9(a) shows an electron hologram that has been acquired from a 40° wedge specimen prepared in an FIB containing a p-n junction with a concentration of $2 \times 10^{19} \text{ cm}^{-3}$. A low magnification of 1 k3 has been used to provide a large field of view and the fringe spacing is 4 nm, which leads to a hologram width of 500 nm. A $2,048 \times 2,048$ pixel CCD camera has been used to sample the fringes correctly and provide a useful holographic field of view of $500 \times 1,500$ nm. Figure 1.9(b) shows the reference hologram and (c) shows the reconstructed phase image, and the phase jumps of 2π can be clearly observed. In addition, the presence of the p-n junction can be observed in the thicker parts of the specimen. Phase unwrapping has been used to remove the phase jumps in Figure 1.9(d). Profiles extracted from across the indicated regions are shown in Figure 1.9(e) and the phase in multiples of 2π and continuous phase measurement are observed.

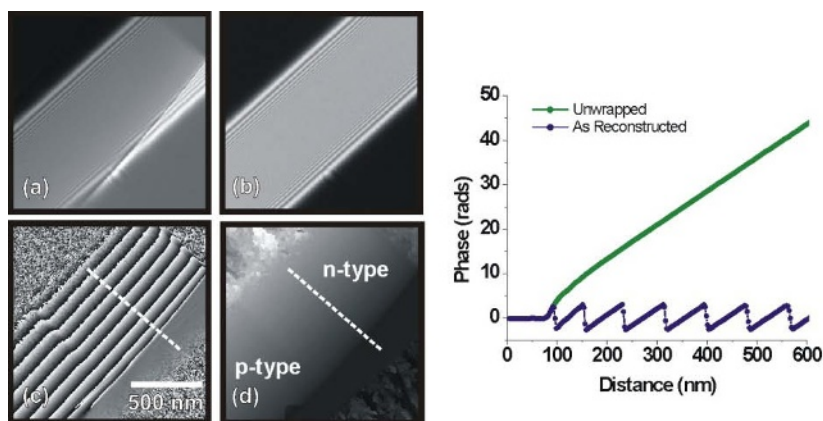


Figure 1.9. a) Electron hologram of a 40° wedge specimen containing a symmetrical p-n junction with a dopant concentration of $1 \times 10^{19} \text{ cm}^{-3}$. b) Reference hologram, c) reconstructed phase image and d) unwrapped phase image. e) Phase profiles taken from across the region indicated

1.3. Experimental electron holography

1.3.1. Fringe contrast, sampling and phase sensitivity

Experimental electron holography involves achieving a balance between fringe contrast and spatial resolution, hologram width and field of view and the number of electron counts that are recorded. Although the electron source is not perfectly coherent, it must be sufficiently coherent for an interference fringe pattern of sufficient contrast to be acquired within a time period, during which the drift of the specimen, biprism and beam, will not significantly degrade the results.

Considerations regarding the choice of biprism voltage, microscope magnification, electron source size and CCD exposure time will influence the phase information that can be reconstructed from the hologram. The sensitivity of the phase image is associated with the hologram fringe contrast. As has been seen, the ultimate contrast is selected by the choice of beam settings and this can then be maximized by carefully aligning a highly astigmatic electron beam exactly normal to the biprism.

The phase sensitivity is related to the number of electron counts that are recorded and the fringe contrast, μ , by the expression:

$$\Delta\phi_{\min} = \frac{\sqrt{2}}{\mu\sqrt{N_{el}}}$$

where N_{el} is the electron dose per pixel in the reconstructed image [LIC 08]. For a given electron beam setting, the fringe spacing can be decreased, and hence the spatial resolution increased by increasing the voltage on the electron biprism. However, by increasing the biprism voltage, two different effects will decrease the contrast. First, the ultimate limit on the achievable spatial resolution is that when higher biprism voltages are used, the virtual sources are pushed further apart and electrons must travel further to form the interference pattern that will reduce the hologram contrast. A second effect that will decrease the contrast is the sampling of the fringes by the CCD camera. When the number of pixels used to sample each fringe starts to fall below six, the recorded hologram contrast will decrease rapidly. Figure 1.10 shows the measured hologram contrast acquired using standard Lorentz lens settings at different biprism voltages to provide fringe spacings in the range 2.5–1 nm for different magnifications and an acquisition time of 16 s. The different magnifications are indicated by the field of view. At higher magnifications, the sampling of the fringes is improved. Using standard beam settings in the Lorentz mode, the smallest fringe spacing is limited to approximately 1 nm before the contrast becomes too low to be useful.

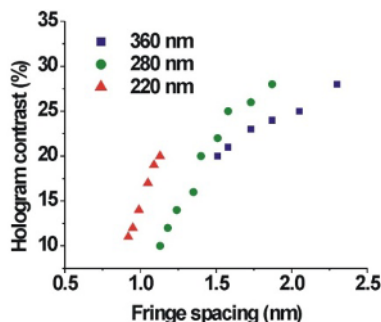


Figure 1.10. Hologram contrast measured as a function of fringe spacing for different magnifications that are indicated by the total field of view

Sampling of the electron holograms is a very important aspect of experimental holography and this links the achievable field of view to the spatial resolution. Figure 1.11 shows electron holograms that have been acquired using identical beam settings with a fringe spacing of 2 nm. For the acquisition, a nominal magnification of (a) 2k2 has been used to provide a field of view of 770 nm and (b) 4k7 to provide a field of view of 360 nm. For the higher magnification, each holographic fringe is sampled by six pixels using a $1,024 \times 1,024$ CCD camera and a contrast of 30% has been measured. For the lower magnification, only 3.4 pixels have been used to sample each fringe and a contrast of only 18% has been measured. Different intensities are observed for the different magnifications as the intensity, C_2 , was kept constant during the experiment and using a higher magnification has the effect of spreading the illumination relative to the CCD camera. Whenever possible at least six CCD pixels should be used to sample each holographic fringe, although clearly four pixels can be used at the expense of hologram contrast. Certainly, less than four pixels leads to a rapid decrease in hologram contrast and therefore signal to noise in the reconstructed phase images.

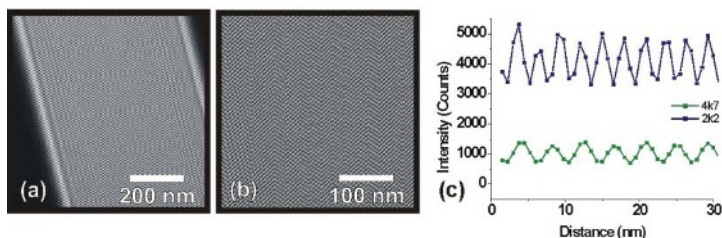


Figure 1.11. a) Electron hologram with a fringe spacing of 2.0 nm acquired using 3.4 CCD pixels per fringe and b) at higher magnification using six pixels per fringe. c) Profiles acquired from across the fringes showing the reduction in intensity and contrast of the holograms

To illustrate the difference between beam coherence and fringe sampling, Figure 1.12(a) shows a detail of an electron hologram acquired using a 1,024 pixel acquisition to provide a field of view of 360 nm. The fringe spacing of 2.2 nm is correctly sampled by 5.7 pixels per fringe, which allows a contrast of 30% to be preserved. Figure 1.12(b) shows the same acquisition settings except for a 1.6-nm fringe spacing. Now the fringe sampling is only 4.2 pixels and the contrast has been reduced to 15%, due to both the coherence of the beam and the sampling of the fringes. In Figure 1.12(c), the same hologram can be seen, except now acquired using a 2,048 pixel acquisition, the sampling has increased to 8.4 pixels per fringe and a contrast of 22% is now recorded. Finally, Figure 1.12(d) shows that when further increasing the biprism voltage to provide a 1.2-nm fringe spacing, although the fringes are still correctly sampled at 6.5 pixels per fringe, the contrast has dropped to 15% due to the limited coherence of the electron beam.

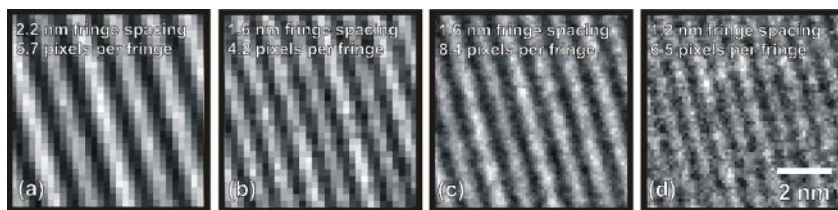


Figure 1.12. Detail of the center of an electron hologram with identical magnifications to provide a total field of view of 360 nm but with different fringe spacings and sampling densities

The sampling of the electron hologram fringes has important implications for electron holography when low magnifications are required. The hologram width and fringe spacing are linked and a fringe spacing of 4 nm is about the largest that can be obtained while still providing a hologram that is wide enough to be useful. If conventional Lorentz beam settings and a $1,024 \times 1,024$ CCD camera are used and four pixels are required to sample each fringe, then it is clear that one pixel is required per nanometer leading to a maximum field of view that can be achieved in Lorentz mode of approximately 1.0μ . This can be increased by using a $2,048 \times 2,048$ pixel CCD camera at the expense of electron counts.

To illustrate the sensitivity that can be expected by using different acquisition times and typical beam settings, two different empty holograms were recorded using identical settings for different times and used as the object and reference holograms. Figure 1.13(a)–(d) shows electron holograms that have been acquired in the Lorentz mode with a fringe spacing of 2 nm and a field of view of 360 nm using the acquisition times indicated. The reconstructed phase images are shown in Figure 1.13(e)–(h). To give an idea of the contrast, profiles extracted from across the

electron holograms are shown in Figure 1.13(i). In order to illustrate the noise in the phase images, profiles taken from across the center of the images are shown in Figure 1.13(j). As seen previously, for a perfectly stable electron microscope, the signal to noise is dependent on the hologram contrast and the number of electron counts that are recorded. In Figure 1.13(k), the experimentally measured sensitivity of the phase images is shown. The sensitivity has been assessed by taking the standard deviation of the center region of each of the phase images. The stability of the electron microscope has allowed the holograms to be acquired for time periods of between 1 and 64 s with a fringe contrast of approximately 40% irrespective of the acquisition times. These have been compared to the sensitivity calculated by theory and although the fit is not perfect, the link between the hologram contrast, the number of counts that are recorded in the hologram and the size of the mask that is used to reconstruct the hologram (required spatial resolution) is clear. As a result, it is possible to estimate the beam settings that will be required in order to provide the sensitivity to successfully perform a desired experiment.

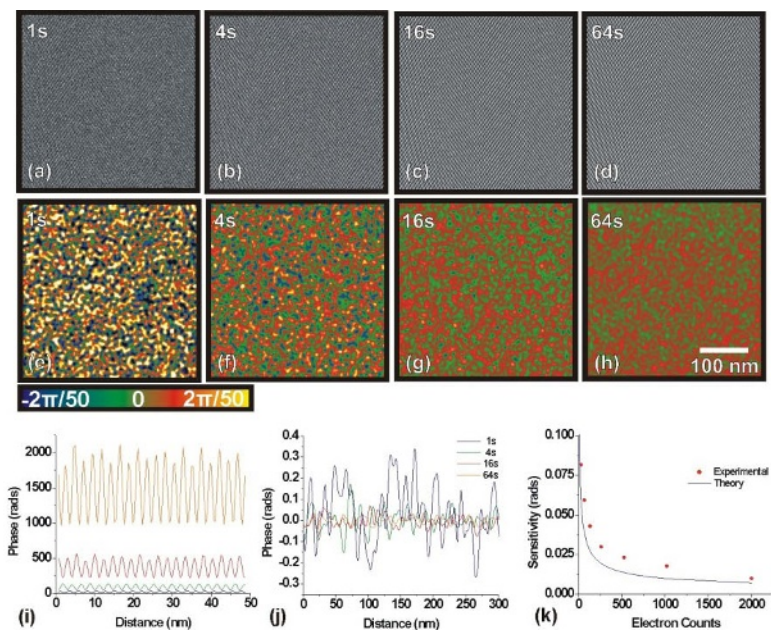


Figure 1.13. (a)–(d) Electron holograms that have been acquired for time periods in the range 1–64 s. (e)–(h) Phase images reconstructed from two electron holograms containing only the vacuums that have been acquired directly after one another, the first as an object hologram, the second as a reference. i) Profiles of electron holograms shown in (a)–(d) revealing the contrast. j) Profiles of phase images reconstructed using two empty holograms containing only vacuum shown in (e)–(h). k) The experimentally determined and calculated sensitivities plotted as a function of the average number of electron counts recorded on the CCD camera

The phase sensitivity measured experimentally is $2\pi/76$, $2\pi/150$, $2\pi/280$ and $2\pi/600$ for the holograms acquired for 1, 4, 16, and 64 s, respectively. Values of phase that can be expected when performing electron holography at 200 kV for different types of samples are as follows. In a perfect and undamaged 200-nm-thick specimen containing a p-n junction in a modern device structure, we would expect to see a step in potential of about 1 V, which corresponds to a step in phase of approximately $2\pi/4$ [RAU 99]. The phase change caused by single atoms approximately follows the relationship $\delta\phi = Z^{0.6}$, and for a single gold or silicon atom we would expect to measure a step in phase of around $2\pi/10$ and $2\pi/50$, respectively [LIC 08]. To measure the step in phase of a single ionized dopant atom would be significantly more difficult. Assuming that a screened potential of a phosphorus atom in silicon can have a potential of a few hundred millivolts across a radius of 1 or 2 nm, a phase change of $2\pi/1,000$ could be expected [KOH 55].

Using a modern TEM in the medium resolution Lorentz mode, we can expect to obtain a sensitivity of $2\pi/1,000$ relatively easily [LIN 12]. However, in high-resolution mode, a best sensitivity of around $2\pi/250$ has been recently demonstrated [COO 07]. Certainly, there is more work to be done for both instrumentation and specimen preparation before individual ionized dopant atoms can be detected with atomic scale resolution.

1.3.2. *Optimizing the beam settings for an electron holography experiment*

When planning an electron holography experiment, it is important to assess the spatial resolution and field of view that is required, as well as the minimum value of signal to noise needed in order to measure the changes in electrostatic potential. Using standard lens settings in the microscope where only the magnification and focus are adjusted, the spatial resolution and the field of view are fixed by the biprism voltage. The choice of beam settings (coherence) will also determine the achievable spatial resolution. Usually, in order to obtain a very good spatial resolution, a weak electron beam is required where the electrons that are collected are only emitted from the very center of the electron gun. This can be controlled by using combinations of a low extraction voltage, a low gun lens setting and a large spot size and a small C2 aperture. There is no “correct” setting for the holography mode, the author tends to prefer to use a very weak electron beam combined with longer acquisition times. For FEI microscopes, an extraction voltage of 3,810 V, a gun lens setting of 5, a spot size of 3 and a 150 μ C2 aperture are good settings to begin with, if more counts are required then the spot size can be reduced, which will double the number of electron counts available per step. Modern microscopes equipped with a monochromator are excellent for electron holography, as the intensity of the electron beam can be flexibly adjusted using the monochromator focus if more counts are needed. It is important to be aware that intense electron beams do not only lead to a reduction in the hologram contrast,

but can also cause the buildup of charge in the specimens, which can affect the phases that are measured in doped semiconductor specimens.

Most modern electron microscopes can be operated in the range 300–80 kV and sometimes even lower. The choice of operating high tension is generally dependent on the stability of the specimen under the electron beam. From Figure 1.1 it can be seen that the electrons interact more strongly with the specimen at low energies. For the examination of Si semiconductor specimens, an operating voltage of 200 kV provides a good compromise between reducing specimen damage, providing a large phase change in the electrons and also providing enough electron counts to be transmitted through the specimen. Electron holography can also be performed at lower high tensions; however, as thinner specimens can be required, this has implications for the dopant concentrations that can be measured. In addition, the gun can be operated using different settings to provide brighter electron beams or more coherent beams. Figure 1.14 shows the contrast measured across electron holograms acquired with a fringe spacing of 3 nm and a field of view of 750 nm as a function of the extraction voltage and operating high tension. Here, the gun lens and spot size have been kept constant. There is a large difference between the number of recorded counts and contrast for the different settings.

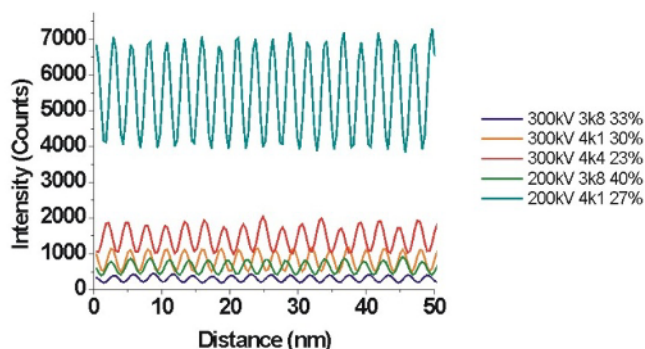


Figure 1.14. Profiles extracted from across different electron holograms for different beam settings and high tensions. All of the holograms were acquired for 16 s using a nominal magnification 2k9 to provide a field of view of 750 nm with a fringe spacing of 3 nm. The measured contrast is also indicated

1.3.3. Optimizing the field of view using free lens control

A different approach to improve the maximum spatial resolution that is achievable is to use free lens control. Although the biprism voltage can be increased to improve the spatial resolution, the hologram contrast will deteriorate and for many holography electron microscopes, it is difficult to have fringe spacings much

below 2 nm, which limits the spatial resolution to approximately 5 nm. Figure 1.15(a) shows an image of a standard beam configuration with the electron beam fully stigmated and 200 V applied to the biprism. The associated hologram in (b) has been acquired using a nominal magnification of 4k7 to provide a field of view of 360 nm and the each fringe is sampled by six pixels. If a better spatial resolution is required, then a higher voltage can be applied to the biprism. Figure 1.15(c) shows an image of the beam with 400 V applied to the biprism to provide a fringe spacing of 1 nm. However, now that the interference pattern is much wider, therefore even if the hologram is correctly sampled such as in (d), which shows an electron hologram that has been acquired using a nominal magnification of 8k0 and a field of view of 200 nm, the fringes will have lost contrast due to the limited coherence of the beam. Figure 1.15(e) shows an image of the beam configuration after selecting a high magnification in the Lorentz mode and then using the diffraction lens to reduce the width of the interference pattern. Here, there is still 400 V applied to the biprism; however, as the width of the pattern has been reduced, the contrast of the holograms is still high. Figure 1.15(f) shows an electron hologram acquired using this configuration with a field of view of 150 nm and a fringe spacing of 1 nm. Figure 1.15(g) shows profiles extracted from the holograms in Figures 1.15(b), (d) and (f). By using free lens control, it can be seen that 1-nm fringe spacing can be achieved with a good contrast level and with enough electron counts. When higher magnifications and spatial resolutions are required such that the electron hologram is larger than the field of view, the diffraction lens should always be used to reduced the width of the electron hologram so that the Fresnel fringes are just outside the field of view of the CCD camera in order to maximize the fringe contrast and hence the sensitivity.

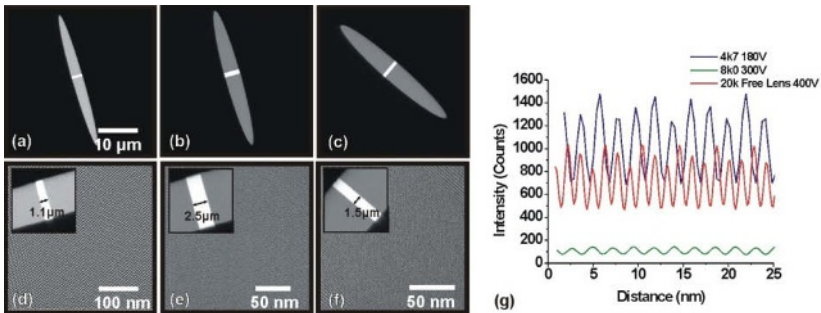


Figure 1.15. *a) An image of the electron beam used to acquire the electron hologram shown in b), here a standard Lorentz magnification is used with a biprism voltage of 200 V to provide a fringe spacing of 2.0 nm and an overlap width of 1.1 μ. c) An image of the electron beam used to acquire the hologram shown in d) with 400 V applied to the biprism to achieve a fringe spacing of 1.2 nm and an overlap with of 2.5 μ. e) By adjusting the diffraction lens settings, the interference width can be reduced while preserving the fringe spacing. f) An electron hologram with a fringe spacing of 1 nm and a total width of 1.5 μ. g) Profiles acquired from all of the holograms showing intensity and the fringe contrast*

There are many different methods that can be used to improve the spatial resolution when performing electron holography experiments. On both JEOL and FEI microscopes, a dual lens method can be used where the Lorentz and objective lenses are used simultaneously. This approach provides fringe spacings of approximately 1 nm [WAN 04]. An important consideration is that for high spatial resolutions, the field of view is limited, which means that a vacuum region must be provided very close to the region of interest. For very small samples, conventional high-resolution electron holography can be used to provide a field of view of a few tens of nanometers and atomic resolution.

An example of the benefits of using the diffraction lens is shown. Figure 1.16(a) shows an STEM image of an nMOS device with a 28-nm gate length in (b). The corresponding potential map that has been acquired using conventional beam settings to provide a field of view of 360 nm and a fringe spacing of 2 nm can be seen. However, it is difficult to determine the position of the active dopant atoms and the electrical junction under the gate. The potential map shown in Figure 1.16(c) has been acquired by adjusting the diffraction lens to provide a field of view of 150 nm and a fringe spacing of 1 nm. Now details such as the SiGe channel and the electrostatic potentials under the channel arising from the doped source and drain regions can be clearly seen. To achieve this spatial resolution, a vacuum reference region is needed near the region of interest, here the specimen was rotated by 90° to the ion beam in the FIB after lift out and the metal surface layers were removed to provide a vacuum reference 150 nm from the region of interest. If higher magnifications are required then fields of view in the range of 30–100 nm are also possible, by applying free lens control in objective lens mode. Objective mode holography is usually associated with atomic resolution imaging that provides fields of view below 30 nm. Therefore, the goal is the opposite as for the Lorentz mode and a wider interference pattern and larger fringe spacings are needed. By adjusting the diffraction lens and simultaneously increasing the projector lenses, the required hologram properties can be obtained [SIC 11]. In Figure 1.16(d), a potential map of an arsenic-doped pMOS device with a 40-nm gate length can be seen. To be able to accurately measure the electrical gate length, better spatial resolution is required as the region of interest is small compared to the total field of view. Figure 1.16(e) shows a potential map that has been acquired using the free lens objective mode and a field of view of 75 nm, and a fringe spacing of 0.33 nm has been achieved to provide a spatial resolution of 1 nm. The electrical overlap width, dl , has been compared to the arsenic distribution measured using electron energy loss spectroscopy (EELS) and the results are consistent within the spatial resolutions of the two techniques. However, to achieve these results, it was not possible to put a vacuum reference region close enough to the region of interest without damaging the specimen in the FIB. As a result, the lightly doped substrate has been used as a reference. Using a homogeneous region of the specimen instead of the vacuum reference is known as “differential” or “bright field” electron holography.

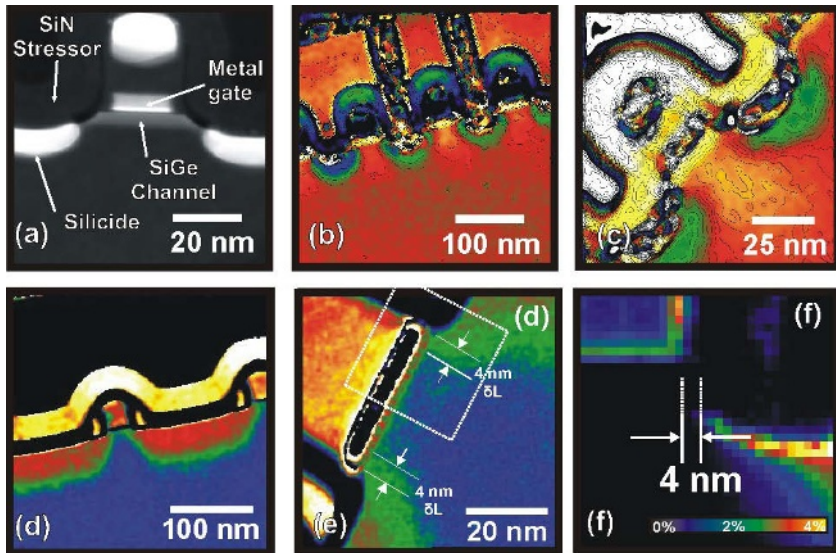


Figure 1.16. a) STEM image of an nMOS device with a gate length of 28 nm. b) Potential map showing the doped regions with a spatial resolution of 6 nm and a field of view of 360 nm. c) Potential map with a spatial resolution of 3 nm and a field of view of 150 nm provided in a free lens Lorentz mode. d) Potential map of a pMOS device with a gate length of 40 nm. The spatial resolution is 6 nm and the field of view 360 nm. e) Potential map of the pMOS device with a spatial resolution of 1 nm and a field of view of 75 nm. f) EELS map of the region indicated in e) showing the concentration of arsenic atoms

1.3.4. Energy filtering for electron holography

An energy filter can also be used to improve the contrast in the object hologram as this will remove the diffuse background scattering in the recorded image. Figure 1.17(a) shows an electron hologram containing an nMOS device acquired with a 10 eV slit inserted and (b) the potential map calculated from the reconstructed phase image. In Figure 1.17(c), an unfiltered electron hologram and (d) the potential map are also shown. Profiles acquired from the indicated regions in the electron holograms are shown in Figure 1.17(e). The hologram contrast in the silicon region of interest is improved from a value of 4% in the unfiltered hologram to 6% in the filtered hologram. For a comparison between the fringe contrasts between the region of interest and the vacuum region, a profile of the reference hologram is also shown. The improvement can clearly be seen in the phase images. The standard deviation has been measured in the regions indicated in Figures 1.17(b) and (d), and 0.10 rads is measured in the unfiltered phase image and 0.08 rads is measured in the filtered phase image. An energy filter will provide more significant improvements for

thicker TEM specimens and is also extremely valuable when using dark-field electron holography for strain mapping.

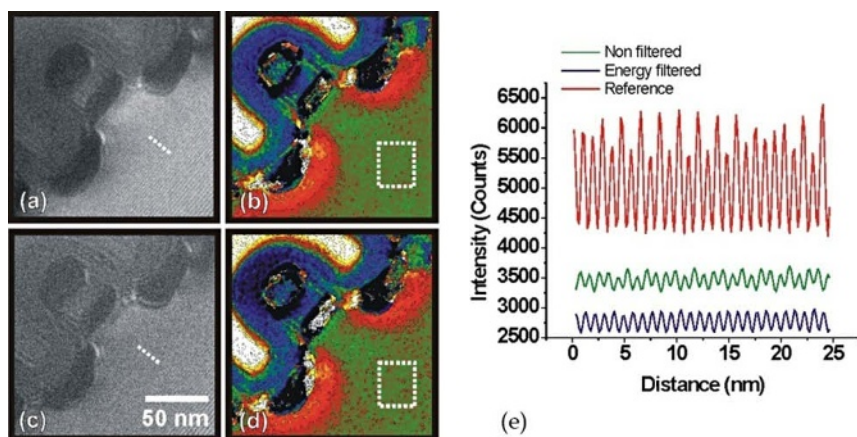


Figure 1.17. *a) Filtered electron hologram containing a 210-nm-thick nMOS device. b) Electrical potential map calculated from (a). c) Unfiltered electron hologram and d) electrical potential map. e) Profiles showing the fringe contrast acquired from the indicated regions in (a) and (b) and from the reference hologram*

1.3.5. Minimizing diffraction contrast

The measured phases in electron holography are extremely sensitive to the geometrical crystal potential, ϕ^G and as a result it is important to orient the specimen to a weakly diffracting condition. This has important consequences for electron holography as tilting the specimen from the zone axis can lead to a loss in the spatial resolution in the projection of the specimen. The specimen can be examined at an orientation of choice, which means that the spatial resolution imposed by the tilt can be eliminated for either the alpha or beta tilt direction. When performing electron holography on a specimen, it is important to find the zone axis and then tilt the specimen along one of the zone axes by 1° or 2° , or less if possible. Fine specimen tilts can then be applied to remove all traces of diffraction in the specimen and the contrast in the region of interest should be completely homogeneous and bright. In Figure 1.18(a), an electron hologram containing an nMOS device in a diffracting orientation can be seen. The diffraction contrast is also present in the reconstructed phase and amplitude images shown in Figures 1.18(b) and (c), which will lead to misleading measurements of the specimen potential. In Figure 1.18(d), an electron hologram of a different device, but from the same lamella tilted to a more weakly diffracting orientation, is shown. The diffraction contrast has been significantly

reduced from the hologram, and the phase and amplitude images in (e) and (f) have much more homogeneous contrast. It is not always possible to remove all the diffraction contrast from specimens during examination; however, this should be minimized and the effects of diffraction on the measured phase can be assessed by acquiring holograms with different amounts of contrast present.

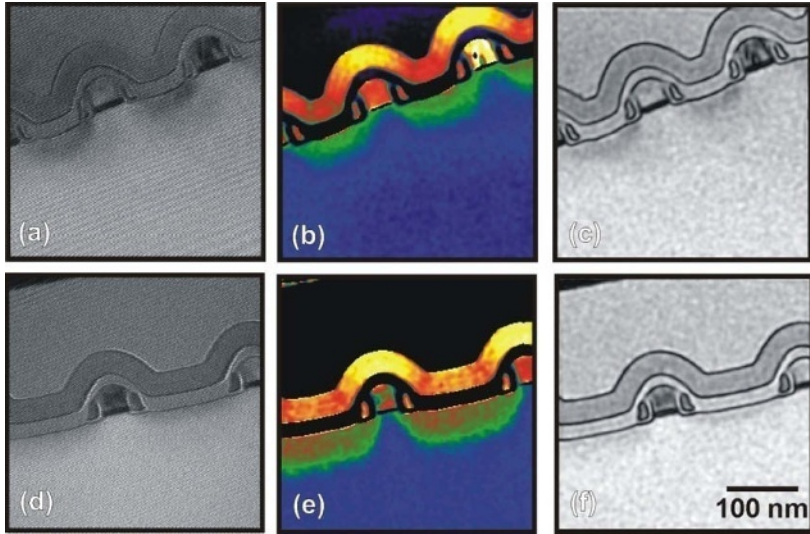


Figure 1.18. *a) Electron hologram containing an nMOS device with diffraction contrast in the source and drain regions. b) and c) Reconstructed phase and amplitude images showing evidence of diffraction contrast. d) Electron hologram containing an nMOS device tilted to a weakly diffracting orientation. e) and f) Reconstructed phase and amplitude images showing more homogeneous contrast in the region of interest*

1.3.6. Measurement of the specimen thickness

The phase change of an electron is determined by the integral of the potentials in and around the specimen. Therefore, in order to recover information about the potentials, it is important to know the thickness of the specimen. There are many different methods that can be used to measure specimen thickness; here, we focus on two that are most commonly used in electron holography. Convergent beam electron diffraction (CBED) can be used to determine the crystalline thickness of a specimen to approximately ± 5 nm [WIL 09]. Figure 1.19(a) shows a CBED pattern that has been acquired for a thick specimen (>150 nm), here a CBED pattern in a two beam condition will provide two discs from which the specimen thickness can be determined from the distances between the interference fringes. For thinner specimens, a single CBED pattern can be acquired at the zone axis and compared to

simulations to determine the correct thickness. If CBED is performed carefully, and the patterns are well focused, then this provides a robust and reproducible measure of the specimen thickness.

An alternative method of measuring the thickness is to use the reconstructed amplitude image. From electron energy loss spectroscopy, it is known that the image intensity is related to the inelastic mean free path, λ_{in} [EGE 11].

$$I_0 = I_{total} e^{-t/\lambda_{in}}$$

As the amplitude image is the square root of the intensity, an expression can be derived that relates the specimen thickness to the amplitude image [MCC 94].

$$\frac{t(x,y)}{\lambda_{in}} = -2\ln A(x,y)$$

Therefore, if the sample is homogeneous and the effects of dynamical diffraction are limited, then the thickness of the specimen can be calculated in units of λ_{in} . Figures 1.19(b), (c) and (d) show an electron hologram, amplitude image and thickness map, respectively, that has been calculated for a silicon lamella of thickness 400 nm. When calculating the thickness from the amplitude image, the image must first be normalized with respect to the vacuum region. Also, λ_{in} is dependent on microscope parameters such as the objective aperture size. To determine λ_{in} for Si using the FEI Titan, the thickness of five different lamellas prepared by FIB milling at 30 kV was evaluated by CBED and from the amplitude images. Figure 1.19(e) shows the relationship between the crystalline thickness of the specimen and its total thickness in units of λ_{in} . From the gradient, λ_{in} is calculated as 120 ± 10 nm for these microscope settings. From the x-intercept, the total thickness of the amorphous layer can be determined as 50 nm (25 nm on each face), which is consistent with what would be expected for an FIB operating voltage of 30 kV. Different values of λ_{in} have been given elsewhere that have been determined using different microscopes, operated at different high tensions using specimens prepared by different methods. Clearly, care needs to be taken when using this method of calculating the thickness of specimens. This is especially valid when thicker specimens are being examined where multiple scattering of the electrons is likely, which is often the case for off-axis electron holography.

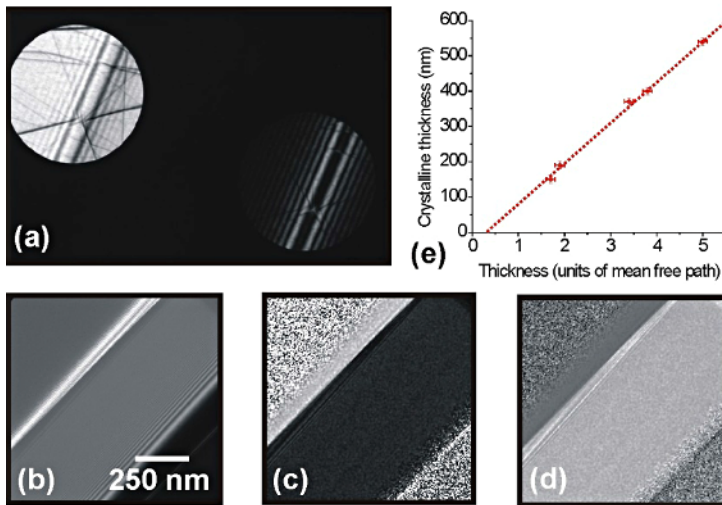


Figure 1.19. *a) CBED pattern of a 400-nm-thick specimen in a (004) two-beam condition. b)– d) An electron hologram, amplitude image and thickness map, respectively, of a 400-nm-thick silicon specimen. e) The crystalline thickness of a range of silicon specimens plotted against the thickness determined from the amplitude image*

1.3.7. Specimen preparation

Specimen preparation is the key to electron holography. The stability and computer control of modern TEMs make experimental electron holography relatively straightforward to perform. However, as has been discussed, changes in the MIP are typically in an order of magnitude higher than the electrical potential in a junction. As a result, excellent, flat specimens are required otherwise the data becomes much more difficult to interpret.

The process of using either Ar or Ga ions to thin a sample for observation by TEM will introduce many artifacts into the specimen that can change the measured potentials. Ideally, all specimens would be made by polishing as this provides specimens that have relatively undamaged surfaces. Examples of electron holography performed on wedge polished semiconductor devices do exist in the literature [TWI 02]; however, it is difficult and time consuming to find a specific semiconductor device with a gate length of 40 nm or less in a 300-mm wafer. In addition, a vacuum region is required to be close to the region of interest adding a further complication. In practice, most specimens that are made by polishing are also Ar milled to clean the region of interest, and even the use of low-energy ions can introduce significant artifacts when performing dopant profiling.

FIB milling allows parallel-sided specimens to be prepared from a region of interest with little difficulty. Modern dual-beam FIBs make it relatively easy to find an individual semiconductor device by using the scanning electron microscope. FIBs use a focused beam of Ga ions to cut around the region of interest. The region of interest is then extracted by *in situ* lift-out and then thinned to electron transparency. The strengths of the FIB are that it allows the thickness to be controlled and also allows parallel-sided specimens to be prepared, which do not have thickness variations across the region of interest. The problems with amorphization on specimen surfaces are well known; however, for electron holography this is not a great problem. In fact, the presence of the surface amorphous layers will create an isopotential surface on the specimen that will eliminate fringing fields in the vacuum regions near the specimen. The real problem of using Ga ion milling is that the ions penetrate the specimen and create what is known as the inactive thickness, t_{inactive} , which will be discussed later in this chapter.

Semiconductor specimens usually contain insulators between the silicon substrate and the metalized regions on the surface. Figure 1.20(a) shows a phase image of a pMOS device with the insulator region charging during observation in the TEM. The charging has completely dominated the dopant potentials in the specimen. Figure 1.20(b) shows a phase image of an nMOS device that has been carbon coated, but not prepared using backside milling. The device has no metallization on the top layers and the vertical stripes in the phase image are caused only by changes in the composition between the silicon, silicon oxide and silicon nitride. For a metallized device, the problems caused by differential milling would be significantly worse. Figure 1.20(c) shows the same device as seen in 1.20(a). The specimen has been prepared using backside milling and a thin layer of carbon has been applied onto one side of the specimen. The effects of charging have been significantly reduced and the changes in the potentials arising from the active dopant atoms can be clearly seen.

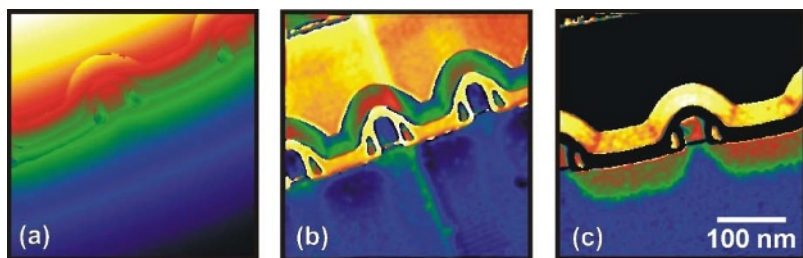


Figure 1.20. *a) Phase image of nMOS device without carbon coating. b) Phase image of a carbon-coated nMOS device that has been prepared using conventional FIB milling. c) Phase image of an nMOS device that has been prepared by back side FIB milling and then carbon coated*

1.3.8. *The electrically inactive thickness*

The relationship between the measured phase and the potential inside the semiconductor specimens is complicated due to the effects of specimen preparation and surface charging, which lead to what is known as the inactive thickness. There are many papers that discuss the problems encountered when using electron holography for dopant profiling; in this chapter, there is only space to introduce them briefly. The inactive thickness is used to describe a modified crystalline region in the specimen where the dopant atoms are not active [GRI 02]. From the equation below, it can be seen that in principle, it should be straightforward to recover the electrical potential across an electrical junction, where:

$$V_E = \frac{\Delta\phi}{C_E \cdot t_{\text{active}}}$$

however, the problem is the determination of the specimen thickness that contains active dopants, t_{active} . Clearly, the amorphous surface layers can be neglected from the calculation of the potential as these will not contain active dopants. Figure 1.21 shows a phase image of a Si specimen containing a p-n junction with a symmetrical dopant concentration of $2 \times 10^{18} \text{ cm}^{-3}$ that has been prepared by FIB milling at 30 kV. It is clear that the electrical junction does not extend all the way to the specimen surface, indeed the junction becomes visible at about 100 nm from the specimen edge. If a series of these p-n junctions with different thicknesses are examined and the step in phase across the electrical junction is measured such as in Figure 1.21(b) and then evaluated as a function of the crystalline thickness measured by CBED as in Figure 1.21(c), then the inactive thickness can be determined from the x-intercept. Here, the value of the inactive thickness is 140 nm. The inactive thickness will be equally distributed on each specimen surface; thus, 70 nm inactive thickness on each surface plus an amorphous layer of approximately 25 nm that corresponds well with the phase image in (a). Figure 1.21(d) shows a schematic of a TEM specimen containing a p-n junction.

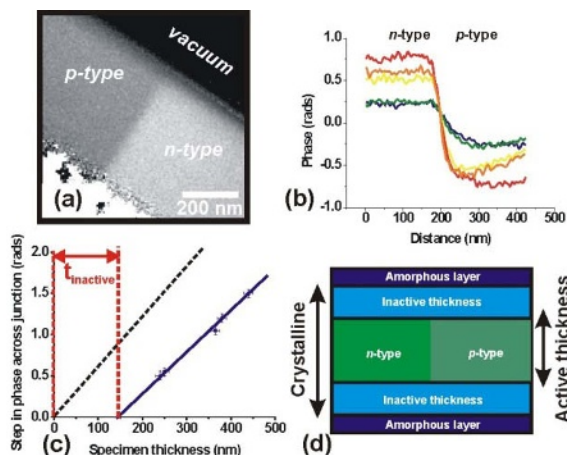


Figure 1.21. *a) Phase image of a specimen containing a p-n junction with a concentration of $2 \times 10^{18} \text{ cm}^{-3}$. b) Phase profiles extracted from across different specimens of a range of thicknesses. c) The step in phase measured across the p-n junctions as a function of the crystalline specimen thickness measured by CBED. d) Schematic of a TEM specimen containing a p-n junction*

If the inactive layer is known then the potential in a p-n junction can be calculated. The problem is that the inactive thickness is sensitive to the dopant concentration in the specimens, the amount of charge and also specimen preparation [COO 10]. Therefore, it is very difficult to predict the inactive thickness. Figures 1.22(a)–(c) show the experimentally measured phase across 400-nm-thick specimens containing p-n junctions with a range of dopant concentrations. For low dopant concentrations, there is a larger discrepancy between the experimentally measured values of phase and theory. This is demonstrated more clearly in Figure 1.22(d) where the step in phase across a series of junctions with different dopant concentrations is shown as a function of the crystalline specimen thickness. Here from the x-intercept it is clear that the inactive thickness varies strongly with the dopant concentration. The inactive thickness has been measured from the intercepts in Figure 1.22(d) and is shown as a function of the dopant concentration in Figure 1.22(e). Here, we see that the inactive thickness tends toward zero for higher dopant concentrations and can be reduced by using lower FIB operating voltages. The inactive thickness is caused by a combination of band bending caused by the presence of charge at the specimen surfaces and also from the effects of specimen preparation. The use of ions to prepare specimens for TEM analysis will create defects deep in the specimens and trap the dopants. These effects can be reduced by reducing the energy of the ions or by using larger ions that do not penetrate deep in the specimen. For example, Figure 1.22(e) also shows the inactive thickness measured in specimens prepared using an FIB operating voltage of 8 kV instead of 30 kV, and the inactive thickness has been reduced by a factor of two.

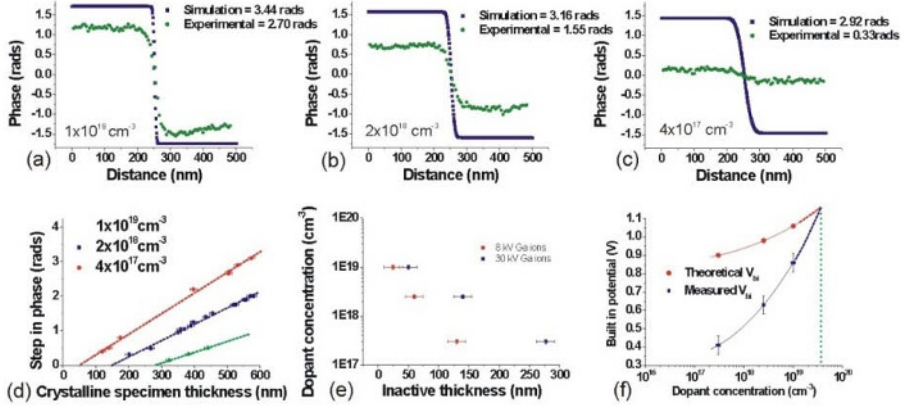


Figure 1.22. a), b) and c) The experimentally determined step in phase compared with simulations for specimens containing p-n junctions with different dopant concentrations. d) The step in phase across a range of p-n junctions as a function of the crystalline specimen thickness. e) The inactive thickness measured experimentally for specimens with different dopant concentrations that have been prepared using different FIB operating voltages. f) The measured built in potential across different p-n junctions compared to theory

Finally, the electrical potentials can be determined from the gradients in Figure 1.22(d), which uses a method that is in principal independent of the inactive thickness, where,

$$\phi = C_E V_E (t_{\text{cryst}} - t_{\text{inactive}})$$

The calculated values of the built in potential in the different p-n junctions are shown in Figure 1.22(f) and are compared to the theoretically expected values. Again, the experimental results suggest that for higher dopant concentrations the experimentally determined values tend toward theory. Again, suggesting that by examining samples with high dopant concentrations, the artifacts that are observed become less important.

If the detection of very low dopant concentrations is required, then thick specimens that are prepared extremely carefully using low-energy ions, or only polishing, are needed. Low dopant concentrations are very sensitive to surface charging and the whole thickness of the specimen can be depleted [COO 08]. In fact, the observation of dopant concentrations below $1 \times 10^{17} \text{ cm}^{-3}$ in silicon is very difficult. Because of the reduction in the size of modern semiconductor devices and the very high dopant concentrations that are present, it is becoming more straightforward to be able to determine information about the position of the electrical junctions in the devices.

The literature is full of approaches such as in situ annealing of Si specimens at low temperature (300°C) or the use of large ions such as Xe at low energy in order to significantly reduce the inactive thickness. In situ biasing can also be used to recover the correct built in potential. Although it is still difficult to obtain quantitative information about the dopant concentrations in semiconductor devices, approaches such as these may allow fully quantitative dopant profiling in the near future.

1.4. Conclusion

Off-axis electron holography provides the opportunity to measure the phase change of an electron wave to provide maps of the magnetic, strain and dopant potentials with nanometer-scale resolution. The stability of modern electron microscopes now makes off-axis electron holography a relatively straightforward technique to perform. The electron beam can be quickly and easily aligned and the biprism can be inserted to provide excellent contrast electron holograms that can be recorded with many electron counts. The key for electron holography is to have excellent quality, flat specimens. This is especially true for dopant profiling, as the MIP of most materials is an order of magnitude larger than the potentials expected from active dopants. Therefore, if the specimen is not flat, then it will be extremely difficult to obtain any useful information about the dopants in the specimens. The success or failure of an experiment will be determined before the specimen is put into the microscope. However, we hope that the examples of the dopant maps of real transistors that are shown here are enough to convince the reader of the possibilities of off-axis electron holography. It has not been possible to review all of the different examples of electron holography in the space available here. Groups based in Dresden, Arizona, Bologna and Japan have led the way, and now the technique is becoming widespread and centers can be found in Berlin, Jülich, Toulouse and Grenoble. The literature is now full of wonderful examples of electron holography being used to assess the electrical potentials in transistor devices, nanowires and quantum dots. Certainly, the prospects for off-axis electron holography for strain mapping, and for the visualization of magnetic fields and dopant potentials make it unique and it will surely become an indispensable tool for the characterization of nano-scaled materials.

1.5. Bibliography

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