Off axis holography of doped and intrinsic silicon nanowires: interpretation and influence of fields in the vacuum

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Abstract. Intrinsic and axially modulated doped silicon nanowires (NWs) were studied by off-axis electron holography. Phase gradients in the vacuum were observed and compared to simulations of NWs with a varying charge density at the wire-oxide interface. It seems that intrinsic NWs are positively charged with a charge density around $2 \times 10^{10}$ e.c. cm$^{-2}$ and axially modulated doped NWs (with n-doped regions) are negatively charged with a charge density around $-1 \times 10^{12}$ e.c. cm$^{-2}$. Expected fringing fields around the doped regions are incidentally observed but smaller than predicted by simulations. The effect of the surface charge on the reference wave is evaluated, and should not modify the obtained phase image.

1. Introduction
The scaling of transistors and integrated circuits for future technology nodes not only poses huge challenges on modelling, design and fabrication but increasingly also on the available tools required for structural and compositional analysis of individual devices. Many techniques exist that can probe the doping concentration such as Scanning Transmission Electron Microscopy [1], Atom Probe Tomography [2], Secondary Ion Mass Spectroscopy [3], Scanning Spreading Resistance Microscopy [4], Scanning Capacitance Microscopy [5], or Scanning Tunneling Microscopy [6], however, off-axis electron holography is the only technique that can measure active dopant atoms in the volume of the sample.

Electron holography has been successfully used to map active dopant distributions in semiconductor devices that are several hundreds of nanometers thick [7] and in a ~300 nm diameter core-shell nanowire [8]. To date, however, off-axis electron holography was not applied to sub-100 nm structures with modulated doping because it was generally accepted that the signal would be far too small. Off-axis electron holography is a TEM-based technique that uses a biprism (charged wire) to interfere an object wave that has passed through the sample and a reference wave that has traversed through only vacuum. From the interference pattern (known as the electron hologram) an amplitude and phase image can be reconstructed. In the absence of magnetic fields and diffraction contrast, the
phase difference $\Delta \varphi$ between electrons that have passed through vacuum and electrons that have traversed the specimen is related to the crystal potential, or mean inner potential (MIP) $V(x, y, z)$, by

$$\Delta \varphi = C_E \int_0^t V(x, y, z) \, dz$$

where $C_E$ is an interaction constant ($7.29 \times 10^6$ rad V$^{-1}$ m$^{-1}$ at 200 kV) that depends on acceleration voltage and $t$ is the thickness of the sample along the beam direction $z$ [7].

Holography was performed on Vapour Liquid Solid grown silicon nanowires with axially modulated doping. For the first time doping contrast was observed in thin (60 nm) nanowires. Comparison of the experimental potential variations in the NWs with simulated potential maps reveals that a charge density of $-1 \times 10^{12}$ e.c. cm$^{-2}$ is present at the wire-oxide interface (so-called sheet charge), which is in excellent agreement with the value obtained by transport measurements [9]. These results will be published elsewhere [10]. The aim of this paper is to evaluate the phase signal in the vacuum at the side of the NW, and compare this signal with simulations. Both NWs with modulated doping and homogeneous intrinsic NWs will be considered. This is both of fundamental and applied interest, as a correct interpretation of holography data for microelectronics purposes is possible only if the beam-specimen interaction and its effect on the measured potential is understood. Furthermore the potential variations around the specimen will influence the measurement [11]. A problem in electron holography is that according to potential simulations so-called fringing fields would be expected in the vacuum, for example at the side of a p-n junction [11-13]. In practice such fringing fields are only observed if the junction is reversed biased [12,13]. It was proposed by Fazzini et al. [13] that this is due to positive surface charges in the oxide created by the electron beam and they estimated a charge density around $1.5-3 \times 10^{13}$ e.c. cm$^{-2}$. It should be noted that simulations including such high positive charge densities on the surfaces of the studied NWs [10] would result in hardly any potential variations at the junctions, since the complete NW volume is depleted.

![Figure 1. Phase image of an intrinsic silicon nanowire (left) and phase profile along the dotted line.](image)

2. Experimental results

The nanowires were grown epitaxially on a lightly doped ($10^{15}$ at. cm$^{-3}$ p-type) (111) silicon wafer and contained three 150 nm long n-doped (phosphorus) regions with $N_D$ of $10^{20}$, $10^{19}$ and $10^{18}$ at. cm$^{-3}$ separated by intrinsic segments. All doping concentrations were determined from electrical transport measurements on individual homogenously doped wires for each carrier concentration [9]. Growth conditions are given in [9,10]. The oxide shell on the nanowire surface was approximately 3 nm and grown under ambient conditions (native oxide).

Holography was performed on intrinsic NWs. Phase maps were extracted either by using the holoworks software or a home made Digital Micrograph script. A phase profile made perpendicular to the nanowire axis is shown in Fig. 1. In the vacuum the phase gradually decreases, indicating a positive charging of the NW. The phase gradient in the vacuum was compared to potential
simulations, where an intrinsic NW was simulated with different charge quantities at the wire oxide interface, as shown in Fig. 2A. Simulations were performed with a TCAD simulation tool by Silvaco™ [14]. The potential simulations were converted to a phase signal as follows. A radial potential profile was made in the simulated potential map perpendicular to the NW axis. The radial profile was imported in Digital Micrograph (DM), the 2D cylindrical profile was reconstructed from this radial profile. A projection of the potential along the electron beam (y-axis) was realized using DM. The obtained projected potential (in V nm) was converted to radians (Equation (1)). A charge density of $2 \times 10^{10}$ e.c. cm$^{-2}$ at the wire-oxide interface seems to correspond well to the experimental phase gradient (Fig. 2A). The shape of a phase profile made perpendicular to an intrinsic NW was always similar to Fig. 1, however, the gradient in the vacuum was sometimes larger, indicating a higher positive charge density at the wire surface (Fig. 2A).

**Figure 2.** A: Phase gradient in the vacuum at the right of an intrinsic silicon NW (see Fig. 1) compared with simulations including positive sheet charge values. B: Phase gradient at the right side of an axially modulated doped NW compared with simulations including negative sheet charge values.

A perpendicular profile made on NWs with axially modulated doping showed an increasing phase gradient in the vacuum going away from the wire, indicating negative charging of the wire. Such a phase gradient is compared to simulations in Fig. 2B. The simulations were performed for an entirely intrinsic NW and for an entirely $10^{19}$ cm$^{-3}$ n-doped NW, with different negative sheet charge values. The experimental profile corresponds well to the simulation including a sheet charge of $-1 \times 10^{12}$ e.c. cm$^{-2}$, which is in excellent agreement with [9,10]. From Fig. 2B it can be seen that phase modulations are expected around the doped regions, since the gradient depends also on doping concentration. In the case of a p-n junction even larger phase variations, of approximately 0.3-1 rad are expected in the vacuum around the doped regions, due to the space charge regions. However, phase variations around the doped regions were only incidentally observed and were around 0.1 rad, as shown in Fig. 3, smaller than predicted by simulations.

As shown in the foregoing the NWs are charged, which creates a linear phase gradient in the vacuum. If a charged NW creates a potential gradient in the vacuum, this will perturb the reference wave, as is shown schematically in Fig. 4. The potential simulations were used to describe the influence of the field in the vacuum. The potential $V(x)$ was converted to phase $\phi(x)$, which was used in the object wave $\psi_{obj}$ and the reference wave $\psi_{ref}$ (Fig. 4). The intensity of the hologram is then given by [11,15]:

$$I_{holography}(x) = \left| \exp \left\{ i \phi(x + \frac{W}{2}) \exp \left( i \gamma \frac{2\pi}{\lambda} \right) + \exp \{ -i \phi(x - \frac{W}{2}) \exp \left( -i \gamma \frac{2\pi}{\lambda} \right) \} \right| $$

where $W$ is the width of the hologram, and $\gamma$ is the change in direction of the electron beam due to the biriprism. The $W/2$ shift introduced in $\phi$ is a way to describe mathematically the interferences.
between the object and reference waves. In most experiments the biprism was almost parallel to the NW axis, therefore the influence of the field on the reference wave can be easily compared with simulations. The hologram simulations show that the influence of the potential on the reference wave is negligible. We speculate that this is due both to the potential variation in the vacuum that is linear in first approximation (see Fig 1,2) and the treatment of the hologram. The phase image is made symmetrical with respect to the NW axis by defining two reference regions symmetrically at both sides of the NW. A linear relation is fitted to the reference regions and the obtained gradients are subtracted from the phase image. Therefore the effect of any gradient that is not symmetric with respect to the NW axis is removed.

**Figure 3.** A: Phase image with adjusted grey scale to show phase variations in the vacuum at the side of an axially modulated doped nanowire, as indicated in the schematic (top). B: Phase profiles made on the wire and in the vacuum 60 nm at the side of the NW centre.

3. **Conclusion**

We have shown that electron holography can give valuable information on doping profiles in nanostructures [10] and phase gradients in the vacuum could be attributed to the sheet charge at the wire-oxide interface. However, phase variations in the vacuum around the i-n junction were only incidentally observed, and smaller than predicted by simulation. We speculate that this is due to an influence of the electron beam on the charge distribution. For this reason the effect of the electron beam on the charge distribution in the sample should be included in future potential simulations.

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