Characterization of semiconducting nanowires by transmission electron microscopy

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CHARACTERIZATION OF SEMICONDUCTING NANOWIRES BY TRANSMISSION ELECTRON MICROSCOPY

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INTRODUCTION

1.1 General context

Recently many advances have been made in the field of physics, and many of these advances make use of nanometer sized objects. If a bulk material is made smaller and smaller in one (or more) direction(s), discrete energy levels arise due to quantum size effects, which gives rise to interesting optical and electrical properties. For example confinement in one direction (1D) can be obtained in a quantum well where carriers (electrons and holes) are confined to a layer of material with a thickness comparable to the de Broglie wagelength. 2D confinement can be obtained in nanowires, objects that are very small in two dimensions (i.e. 50 nm) and much longer in the third dimension (i.e. 50 μm) and therefore have a high aspect ratio. A third degree of confinement can be obtained in quantum dots. Furthermore these different geometries can be combined, an example is the growth of heterostructures in nanowires were the different material sections inside the wire behave as quantum dots and can be accessed electrically or optically using the wire geometry.

Nanowires have been at the center of active research now for around two decades, and the number of papers on NWs is still increasing. Two decades ago, semiconducting NWs were mostly regarded as potential replacements of components and functionalities in semiconductor device applications, for example CMOS (complementary metal-oxide semiconductor), where they would allow a smaller feature size no longer depending on the resolution of lithography techniques, and the use of the third dimension. However, a sufficient control of diameter and other properties is still difficult for NWs fabricated using a bottom-up approach (meaning grown using some synthesis procedure), and top-down fabricated NWs (fabricated using lithography and etching strategies) suffer from similar drawbacks as devices fabricated using conventional semiconductor technology, for example they depend on the resolution of the lithography technique used, and the patterning is relatively slow when electron beam lithography is used. Moreover, the feature sizes that are currently used in semiconductor devices, for example the gate length in current transistors, is already so small (around 15 nm), and the fabricated devices are already of such high quality, that it will be very challenging to improve the performance using bottom-up grown NWs. Therefore research in NWs has shifted to a focus on the more unique properties of NWs where they have definite advantages over bulk materials. For example, NWs are very interesting to grow heterostructures with a large lattice mismatch, as strain can be relaxed at the surface, allowing the growth of larger mismatch heterostructures without formation of dislocations than possible by 2D layer growth (Glas, 2006). Another interesting possibility is the growth of core shell structures, for example to form a radial field that can separate electron hole pairs after photo generation, for solar cell applications

(Krogstrup et al., 2013). Since the absorbing properties of a NW array can be equal to or maybe even outperform a 2D layer (Heiss et al., 2014), this is potentially very powerful to use less material for efficient solar cells.

Other potential applications involve use as composite materials (Ajayan et al., 2000; Allaoui et al., 2002) or meta-materials (Yao et al., 2008), as sensors (Agarwal et al., 2008; Verd et al., 2005; Penzaa et al., 2008) and as building blocks in electrical devices (Björk et al., 2008) (Lee et al., 2007), electro-optical devices (Lee et al., 2007) and as anodes in lithium batteries (Chan et al., 2008). Nanowires seem also very promising for thermoelectric applications (Hochbaum et al., 2008) since the surface roughness suppresses the thermal conductivity by increasing the phonon scattering (Moore et al., 2008). Surface roughness in silicon nanowires is a topic closely related to the faceting of the nanowire sidewall in the growth direction. It was shown by (Ross et al., 2005) that the sidewall of silicon nanowires exhibits a periodic faceting in the growth direction of the nanowire.

Recent findings making use of these nano-objects include manipulation of light at subwavelength length scales in the form of surface plasmons in thin metal stripes (Verhagen et al., 2009), observation of the Coulomb blockade in several nanowire systems such as silicon, silicon-germanium and gallium nitride (Huang et al., 2008; Kanjanachuchai et al., 2001; Songmuang et al., 2010), and the observation of single photon emission from nanowires that contain a quantum dot or luminescing defect (Aichele et al., 2009; Babinec et al., 2010). It has been shown for InP nanowires using correlated transmission electron microscopy (TEM) and photoluminescence (PL) measurements that the PL of single crystalline nanowires was very different from the PL of nanowires containing twin defects (Bao et al., 2009). Also it has been observed that sometimes only very few wires from the nanowire sample show good luminescent properties. It is assumed that this is due to the presence of defects, however other factors might contribute such as surface roughness and variations in the incorporation of the luminescing impurities. Correlated TEM and optics will clarify these effects and help to tune the growth parameters in order to optimize luminescent properties.

1.2 My personal evolution

I have performed my PhD degree on TEM characterization of silicon nanowires (NWs). After obtaining my degree in 2009, I have extended my work to semiconducting NWs based on compound semiconductors. First as a Postdoc at CNRS Institut Néel in the joint (CNRS-CEA) group ‘Nanophysics and Semiconductors‘ (NPSC), characterizing ZnSe NWs with CdSe quantum dot (QD) insertions. Then, since 2010 as a permanent CNRS researcher on the topic ’Correlation of transmission electron microscopy (TEM) with electrical and optical characterizations on semiconducting nanowires.’

In this general introduction I will first rapidly describe my characterization activities for material science, also related to my postdoctoral work, followed by an introduction on NWs with respect to my research project.

Since 2010 I have performed TEM characterization activities for material research. These are aimed at assisting the comprehension of NW growth and properties through structural characterization, mostly using high resolution (HR) high angle annular dark field (HAADF) scanning TEM, often combined with the Geometrical Phase analysis (Rouviere and Sarigiannidou, 2005; Hÿtch et al., 1998) or using bright field TEM characterization in combination with selected area diffraction. The NWs for these studies were grown in the NPSC group.

For these studies I mostly used the cleaving sample preparation method, this is my favorite sample preparation for NWs, that is without doubt an excellent tradeoff between preparation speed, sample quality and operation speed at the TEM, and can be applied to semiconducting NWs that are grown epitaxially on a crystalline substrate, and that do not grow too densely. I had developed this method during my PhD, based on the publication by (Walck and McCaffrey, 1997). This method was described in detail in my PhD thesis and the supporting information of (den Hertog, 2009; den Hertog et al., 2009a). In this method, a small wedge is cleaved manually from the NW growth substrate, inclined by 90°, and attached to a TEM grid. The NWs can now be oriented easily on a crystallographic zone axis (ZA) by orienting the substrate, and if the epitaxial relation with the substrate is of high quality, we can rapidly obtain high resolution images of many NWs. The large advantage of this method with respect to dispersion methods or a traditional cross section is that the NWs are not modified by anything, and therefore they are not bent (for example by capillary forces or by the embedding method used). It should be noted that orienting a single NW on a crystallographic ZA is increasingly challenging for decreasing NW diameters, since the intensity of the diffraction pattern decreases and the sample moves out of the beam when tilting the specimen. In Figure 1.1 optical microscope images and a TEM image are shown of the same cleaved sample, to show how well the nanowires are preserved on the substrate (den Hertog, 2009).

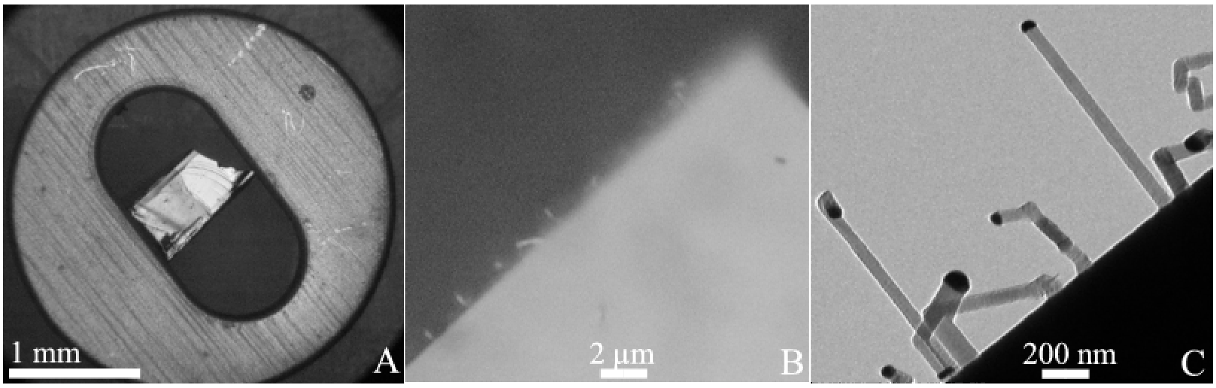

Figure 1.1: An example of a cleaved specimen (A) Optical microscope image of the nanowire stubstrate attached to the TEM grid (B) Optical microscope image showing the nanowires near the cleaved edge (C) TEM BF image (lorentz lens) showing the nanowires on the substrate.

For example, the cleaving method was used to prepare self catalyzed GaAs NWs (grown with a Ga catalyst) with the results shown in Figure 1.2. Two different NW samples were characterized by TEM, where the growth substrate was prepared with a different protocol. The SEM images of these two samples are already striking: a much better homogeneity is obtained with the improved substrate preparation (sample A). Moreover, the TEM images clearly show a very low defect density when the substrate is prepared with the improved method. This work was published in (Tan et al., 2017).

I performed my Postdoctoral research on TEM characterization of CdSe QD insertions in ZnSe NWs. These materials were really very challenging from a TEM perspective, as the NWs were thin (diameters below 10 nm) and beam sensitive (the crystal damages at all tested acceleration voltages, from 30 kV up to 400 kV, most likely due to heating effects that evaporate Se). Embedding the NWs in a traditional cross section protected them from beam damage; however, the embedding procedure does also bend these very thin NWs, making their study truly a challenge. I could obtain, with a large effort, some meaningful data, see (Bellet-Amalric et al., 2010; den Hertog et al., 2011c; den Hertog et al., 2011a; den Hertog et al., 2011b; Bounouar et al., 2012; Bellet-Amalric et al., 2013). During this period I also characterized the first attempts on growth of ZnTe NWs. The NW growth activity later shifted from a focus on ZnSe,

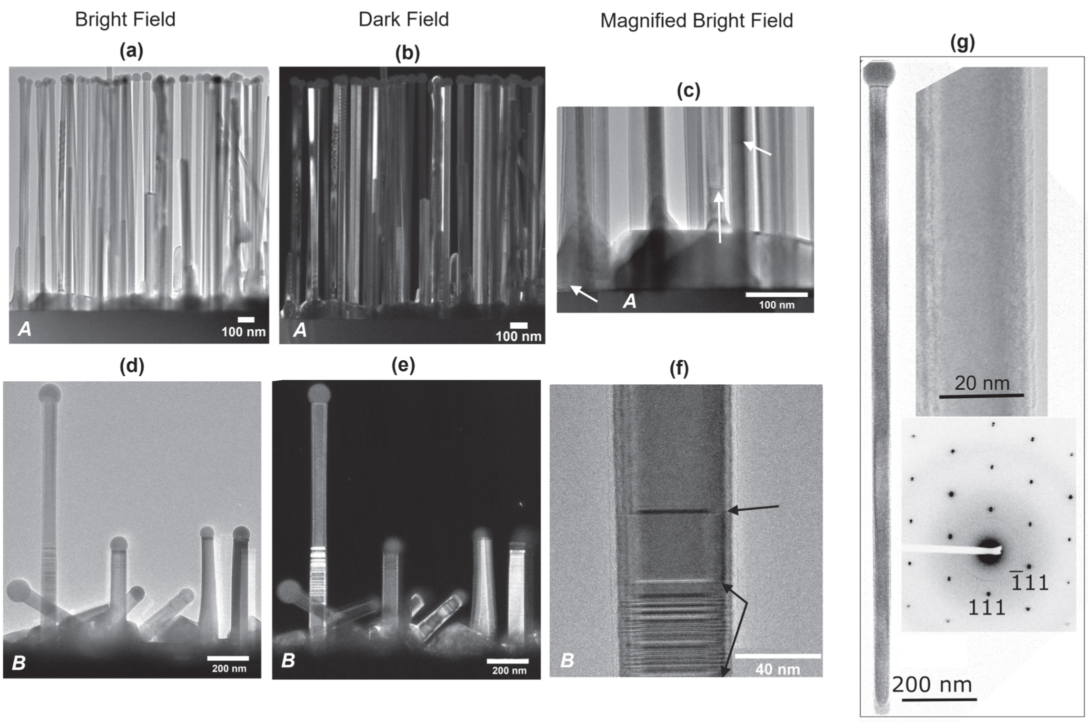

Figure 1.2: Bright-field, dark-field, and magnified bright-field TEM images of GaAs NWs oriented on the $[0\bar{1}1]$ zone axis, grown on (a), (b), (c), (g) chemical oxide-covered (sample A), and on (d), (e), (f) native oxide-covered (sample B) Si(111) substrates. The arrows in (c) and (f) indicate defects in the NW. A defect-free GaAs NW obtained from chemical oxide-covered sample is shown in (g) along with a magnified view of a NW segment and the corresponding diffraction pattern.

to ZnTe with CdTe QD insertions, and large progress was made in the growth of such NW structures during the PhDs of Pamela Rueda and Marta Orrù, both of which worked with me for TEM characterization. Apart from structural analysis (Artioli et al., 2013; Rueda-Fonseca et al., 2014; Rueda-Fonseca et al., 2016a), we could advance the understanding of the growth mechanism on this topic by using the CdTe insertions as a marker. The use of cleaved samples was very helpful in this respect (Orrù et al., 2017), as the entire NW can be studied and it is not broken off at some point (contrary to dispersed NW samples) (another publication is in preparation). Moreover, the possibility to study the same NW using several characterization techniques, including TEM, using an approach based on nitride membranes with metal markers, that I developed, was used to correlate detailed chemical analysis by Energy Dispersive X-ray spectroscopy (EDX) (Rueda-Fonseca et al., 2016b) with optical spectroscopy (Artioli, 2009).

Another topic that I adressed in the design of nanowire opto-electrical properties is doping. Transistors can be created in a nanowire if a p-n junction is present. Furthermore electrical access to optical functionalities (i.e. heterostructures or luminescing defects) present in the nanowire can be obtained using doping. Although doping is a fairly well understood phenomenon in bulk samples, simulation and experiments show that the efficiency of the doping can be modified by the nanostructure (M. Fernández-Serra and Blase, 2006), for example the ionization energy of dopants in nanostructures depends strongly on the dielectric environment (Niquet et al., 2006; Diarra et al., 2007; Björk et al., 2009). Also the presence of a surface can modify the potential in the nanowire since charges can be present at the surface and the surface to volume ratio increases for decreasing object size (Schmidt et al., 2007; Björk et al., 2009). The study of dopants in nanostructures is further complicated by the fact that it is difficult to measure the doping concentration quantitatively. Transport measurements can be used to estimate the doping concentration but they have to assume the mobility in the nanowire is equal to the bulk material (at a given doping concentration), which is not necessarily correct. Various TEM and STEM techniques (Voyles et al., 2002; Molina et al., 2007) are readily available and very successfully employed, but do not yet provide the sensitivity for dopant profiling. While secondary ion mass spectroscopy (Zelsacher et al., 2007) has a high detection sensitivity, it lacks the needed spatial resolution. Characterization methods based on field ionization effects, such as atom probe tomography (Perea et al., 2006; Hoummada et al., 2007; Blavette et al., 1999) can successfully map the 3D (dopant) atom distribution using a dedicated sample geometry. However, these methods are sensitive only to the chemistry of the structure, not the electronic properties. Therefore no information on the electrical activity of the dopant atoms is obtained, which becomes very important in nano-scaled devices (Diarra et al., 2007; M. Fernández-Serra and Blase, 2006) as the dopant distribution (M. Fernández-Serra and Blase, 2006) and activity (Diarra et al., 2007) can be influenced by the size of the nanostructure. We have shown that active dopants can be measured in a thin single nanowire using off-axis electron holography (den Hertog et al., 2009b; den Hertog, 2009), however these measurements are not quantitative. A contacted nanowire in the TEM would allow quantitative measurements of doping concentrations via in situ biasing of the specimen (Twitchett et al., 2004).

The metal contacts that are applied to nanowire structures are another topic of my research. Generally the difficulty consists of creating good Ohmic contacts with low resistance, which is possible by well controlled surface properties and the choice of contact metal (in contrast: for some device architectures Schottky contacts are preferred (Weber et al., 2006)). Furthermore, the channel length can be tuned in some nanowire systems by extending the metal-semiconductor phase (for example a silicide) into the nanowire (Weber et al., 2006). The crystal phase of the contacts, the dynamics of the contact formation, and the chemistry of the contacts are all parameters that could be studied in more detail using TEM based techniques.

1.3 Content of this document

The next two chapters will describe the two developments in my research I would like to highlight. The first topic is developing methods for opto-electrical experiments correlated with TEM, Chapter 2, to allow assessing the influence of the precise atomic structure, heterostructure or defects on the opto-electrical properties.

The second topic is electrical in-situ TEM, Chapter 3. This topic is divided in two parts: we have studied the propagation of metals, both Cu and Al, inside germanium NWs, with the aim to understand this contact formation (for instance the diffusion processes) and understand why a good electrical contact is created. In the second part of the chapter we use electrical contacts for dopant characterization, albeit on a different NW system: ZnO, in combination with off-axis electron holography to access the change in electrostatic potential when a Schottky contact is reverse biased.

In Chapter 4 I will describe the outlook for my research.

I have integrated my publications in the document by clickable links, that are printed in a **bold and blue font**.

1.4 Acknowledgements

Many people have helped me since the start of my research career. I would like to mention especially the help and influence of people at the Plate Forme de Nano Characterization at Minatec, CEA-Grenoble, where I have greatly benefitted from a strong collaboration with the INAC TEM group, LEMMA, with Jean-Luc Rouvière, Eric Robin, Hanako Okuno and Pascale Bayle-Guillemaud. Their support for my projects was extremely important for me. I'm always very happy to visit the PFNC, and meet everybody there. Since my postdoctoral research I maintain and enjoy a strong collaboration with the NPSC group (CNRS-CEA) and have learned a lot of physics there and enjoyed the collaborations with Eva Monroy, Bruno Gayral, Regis André, Edith Bellet-Amalric, Joel Cibert, Henri Mariette, Catherine Bougerol, Bruno Daudin, Moira Hocevar and all the students and postdocs especially Pamela Rueda, Marta Orru, Fernando González-Posada and Jonas Lähnemann. I would like to thank my students Khalil El Hajraoui, Akhil Ajay, Maria Spies and Minh Anh Luong for all their hard work and ideas. Institut Néel is a great laboratory for experimental physics, and I want to thank in particular Thierry Fournier, Bruno Fernandez and Jean-Francois Motte for their help with cleanroom fabrication, and for installing and maintaining such an excellent cleanroom facility. I thank Stephanie Kodjikian, Holger Klein and Christophe Lepoittevin for maintaining the TEM facility and environment at Néel, and their motivation and effort in the new TEM project. I greatly enjoyed the collaboration with Robert McLeod, Julien Pernot and Fabrice Donatini on the contacted ZnO NW. I would like to thank all people that helped me by discussions here and there, at workshops or conferences. I would also like to thank the ANR and EC for funding me generously (projects COSMOS and e-See).

2

Correlation of opto-electrical and TEM characterization on the same unique NW

In 2010 I started a project focused on the correlation of Transmission Electron Microscopy (TEM) based techniques with other physical characterization techniques in the field of optics and electronics on a unique nano object. The aim is to improve the understanding of physical processes in nano objects in the fields of electron transport and optics with the help of TEM analysis of the same unique object. The project is summarized in Figure 2.1.

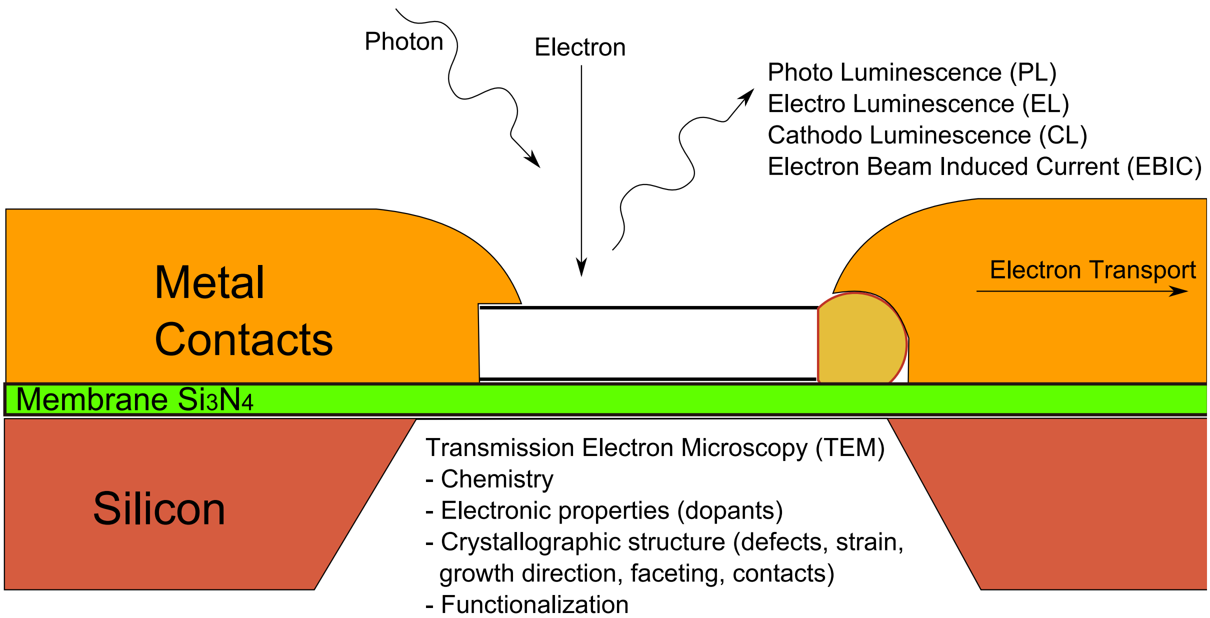

Figure 2.1: A connected nanowire on an electron transparent nitride (Si_3N_4) membrane. The sample geometry is adapted for TEM based techniques. The sample characteristics that can be studied by TEM are indicated as well as other characterization techniques.

In general only one physical property of the sample containing one or several nano objects is observed (i.e. electrical transport or photoluminesce) in combination with light or scanning electron microscopy (SEM) observations to see the global shape of the sample. Due to the limited spatial resolution of these techniques (~ 1 μm and a few nm respectively), it is difficult to estimate contributions to the physical properties of the sample due to fluctuations at atomic length scales. Such fluctuations could be for instance variations in the chemical composition. TEM can be used to measure these properties of a unique nano object, but this is generally done on a different nano object grown or fabricated under the same conditions. However, a common problem in the growth of nano-structures is that small differences between the objects can be present. An example is that some nanowires from the same growth will have crystallographic defects while others are single crystalline, or the crystallographic growth direction can vary from wire to wire (Schmidt et al., 2005).

The combination of TEM and optical or electrical measurements on a single nano object shows the influence of variations in the nano object present on an atomic length scale, that can be crystallographic defects or variations in the heterostructure, on the global response of the object to optical and electrical signals. This is very interesting from a fundamental point of view as these correlated measurements provide the coupling between microscopic effects caused by nanoscale variations. Furthermore, the understanding of the desired properties of a nano object for a given application will be increased, and therefore the objectives for the growth of this nanostructure can be better defined.

Nano-objects are ideal objects of study for correlated measurements for two reasons. First they are considered interesting objects that can be used as building blocks in electrical and optical devices. Furthermore, they do not need specific sample preparation for TEM observation and are therefore free of preparation artifacts. Therefore, the influence of for instance trapped charges at the surface is not due to the creation of more surfaces during the sample preparation, but is a real and interesting feature of the sample that will alter its physical properties. The detection, understanding and if necessary reduction of these effects at the surface is of great technological value if nano objects are to be integrated in devices.

First I will describe the sample fabrication strategies I have developed. Then I will illustrate such correlated experiments by several studies we have performed.

2.1 Sample fabrication

A first step in realizing the correlation of TEM and other characterization methods is the development of a versatile method that allows to contact different types of nano objects using the same technology.

A method to fabricate contacted nanostructures on a nitride membrane that is suitable for TEM analysis was developed. In collaboration with Bruno Fernandez and Thierry Fournier from Institut Néel (cleanroom Nanofab) and Jonas Lähnemann (Postdoctoral researcher NPSC) we developed an approach for membrane fabrication and subsequent NW contacting by electron beam lithography. We start with highly n-doped Si(100) 400 μm thick four inch wafers (the n-doping avoids artifacts for measurements in the UV and IR range of the spectrum) and have 200 nm of thermal SiO_2 followed by either 40 or 200 nm of stoichiometric Si_3N_4 deposited by LioniX International. The SiO_2 layer gives a better protection with respect to short circuits to the substrate, and may improve the nitride quality. The thin nitride layer (40 nm) is used to fabricate planar membranes, the thick nitride layer (200 nm) is used to fabricate membranes with holes. The respective membrane fabrication steps are described in Figure 2.2.

The first step in the fabrication is to protect the sample side of the wafer from scratches, by spinning a layer of resist, Figure 2.2a. Then resist is applied and UV laser lithography is performed on the other wafer side (the backside), Figure 2.2a. A reactive ion etching (RIE) step is used to etch the SiO_2 and Si_3N_4 layers, Figure 2.2b. Another laser lithography (or electron beam

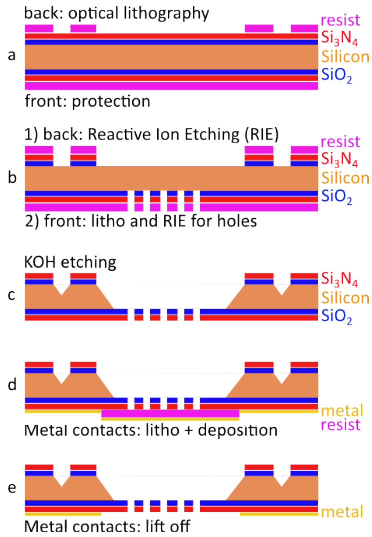

Figure 2.2: Steps for membrane fabrication. a) Application of resist on the front side and backside of the wafer, optical lithography and development on the backside. b) Reactive Ion Etching (RIE) on the backside transferring the pattern in the nitride and oxide layers. On the front side: application of resist followed by optical lithography, development and RIE to define holes. c) KOH etching. d) Fabrication of metal contacts by applying resist, optical lithography, development and metal deposition. e) Lift off step removing resist and metal deposited on the resist.

lithography for small holes) step followed again by RIE then defines the holes at the location where the membrane will be etched, Figure 2.2b. The front and backside lithography steps are aligned. Then a KOH bath at 80°C etches through the Si and the oxide on the other side of the wafer, leaving only the layer of Si_3N_4, which then constitutes the membrane, Figure 2.2c. Next, contact pads and markers are defined by UV laser lithography and electron beam deposition of Ti/Au (10 nm / 50 nm), Figure 2.2d, followed by a lift off step to remove the metal deposited on the resist, Figure 2.2e. On the wafer many chips each containing one membrane with markers, large contact pads, a calibration field for electron beam lithography and some more general markers are present. An image of such a chip is shown in Figure 2.3. These chips are organized on the wafer in arrays of four by four or two by two membranes. On the backside of the wafer cleaving lines are present, where the KOH has etched into the silicon without etching to the other side of the wafer, Figure 2.2c. On the front side of the wafer these lines are indicated by metal lines. The different fields of chips or single chips can be separated by pressing a diamond tip on a cleaving line defined on the backside. On the front side of the wafer the cleaving line is indicated with a metal line.

These chips fit a DENSSolution 6 contact double tilt holder, where small tips make contact with large metal pads defined on both sides of the membrane. This holder was financed in the framework of the ANR COSMOS. We also designed similar holders to use the same approach of NW contacting for making current-voltage, photocurrent, or micro PL experiments with biasing.

Then NWs can be dispersed on such membranes, located with respect to metal markers using SEM, and contacted using electron beam lithography and deposition of Ti/Al (10 nm / 120 nm) in the case of GaN NWs. A calibration field for electron beam lithography is patterned on each chip, to allow a semi-automatic procedure for electron beam lithography.

All steps are performed in the Nanofab cleanroom in Institut Néel, except for metal deposition on four inch wafers, as well as metal deposition for NW contacts, as a plasma is needed to

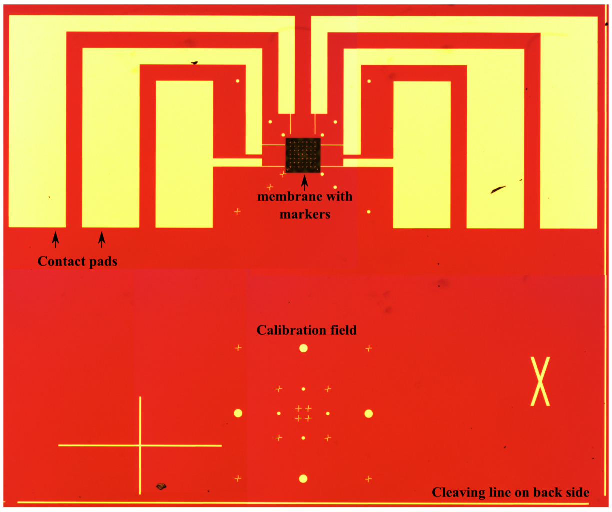

Figure 2.3: Optical microscope image of a single as fabricated chip, containing a flat nitride membrane in the center, with metal markers, contacts and calibration fields for electron beam lithography.

clean the NW surface prior to metal deposition to ensure good contact quality. These steps are performed in the PTA cleanroom in CEA-Grenoble. All studies in the following make use of this sample fabrication technology, unless otherwise indicated.

2.2 Electric fields in GaN/AlN NW heterostructures

Nitride GaN/AlN NW heterostructuctures are known to have strong polarization fields, due to the difference in spontaneous polarization, as well as piezoelectric effects. Since 2010 I was interested in studying the electric fields in these structures, and their influence on the opto-electrical properties, as a precise measurement of the internal electric fields and surface effects (Fermi level pinning and/or surface charges) will allow optimal engineering of these structures for applications. Within this framework we have conducted several studies, that I would like to highlight. For these studies I collaborated with different people, as indicated, and continuously enjoyed the collaboration with Eva Monroy, who is working on NW growth and opto-electrical characterization.

In the paper **Correlation of Polarity and Crystal Structure with Optoelectronic and Transport Properties of GaN/AlN/GaN Nanowire Sensors** (den Hertog et al., 2012) we studied GaN nanowires (NWs) with an AlN insertion by correlated optoelectronic and aberration-corrected scanning transmission electron microscopy (STEM) characterization on the same single NW. Using aberration-corrected annular bright field and high angle annular dark field STEM, we identified the NW growth axis to be the N-polar $[000\bar{1}]$ direction, as illustrated in Figure 2.4. The electrical transport characteristics of the NWs are explained by the polarization-induced asymmetric potential profile and by the presence of an AlN/GaN shell around the GaN base of the wire. In Figure 2.5 we show the comparison of three contacted NWs with their respective HAADF STEM image and characterization of the AlN insertion, combined with the current voltage (I-V) characteristics obtained on these NWs. A higher forward current is observed for increasing shell thickness, which supports the hypothesis that the conduction takes place via the surface pathway created by the GaN shell. The AlN insertion blocks the electron flow through the GaN core, confining the current to the radial GaN outer shell, close to the NW side-walls, which increases the sensitivity of the photocurrent to the environment and in particular to the presence of oxygen. The desorption of oxygen adatoms in vacuum leads to a reduction of the nonradiative surface trap density, increasing both dark current and photocurrent.

I greatly enjoyed the collaboration with Fernando González-Posada, who was a postdoc with Eva Monroy at that time and did the opto-electrical characterization of the contacted NWs. In this paper we could explain the asymmetry in current-voltage characteristics by the polarity. Moreover, we verified the polarity using a complementary method, off-axis electron holography, on a complementary sample, in the paper **Polarization fields in GaN/AlN nanowire heterostructures studied by off-axis holography** (den Hertog et al., 2010). For this sample, a specific growth was done on Si pillars, and prepared for TEM using the cleaving method I introduced in Chapter 1 Figure 1.1. In Figure 2.6 a comparison between experimental and simulated potential profiles is presented. The simulation was performed for the N-polar $[000\bar{1}]$ NW growth direction. Due to composition gradients at the interfaces, no further quantification was possible in this sample

In the paper **Bias-Controlled Spectral Response in GaN/AlN Single-Nanowire Ultraviolet Photodetectors** (Spies et al., 2017), we presented a study of GaN single-nanowire ultraviolet photodetectors with an embedded GaN/AlN superlattice. The heterostructure dimensions and doping profile were designed in such a way that the application of positive or negative bias leads to an enhancement of the collection of photogenerated carriers from the GaN/AlN superlattice or from the GaN base, respectively, as confirmed by electron beam-induced current measurements. The devices display enhanced response in the ultraviolet A (330-360 nm)/B (280-330 nm) spectral windows under positive/ negative bias. The result is explained by correlation of the photocurrent measurements with scanning transmission electron microscopy observations of the same single nanowire and semiclassical simulations of the strain and band

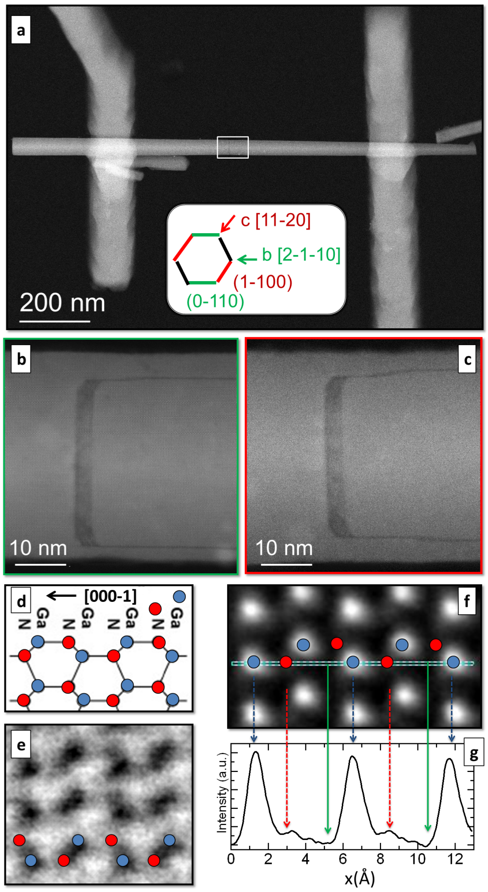

Figure 2.4: (a) HAADF STEM image of a contacted NW; the NW top is on the left. The inset schematically shows the two observation directions of the NW. (b, c) Zoom of the area around the AlN barrier indicated by the square shown in (a) viewed along $[2\bar{1}\bar{1}0]$ (b) and $[11\bar{2}0]$ (c). (d) Schematic representation of the GaN crystal structure viewed along $[11\bar{2}0]$. (e) Convoluted atomic resolution ABF and (f) averaged HAADF STEM image obtained on the same NW viewed along the $[11\bar{2}0]$ axis with a superposition of the GaN atomic structure. (g) Intensity profile along the superimposed line in image (f).

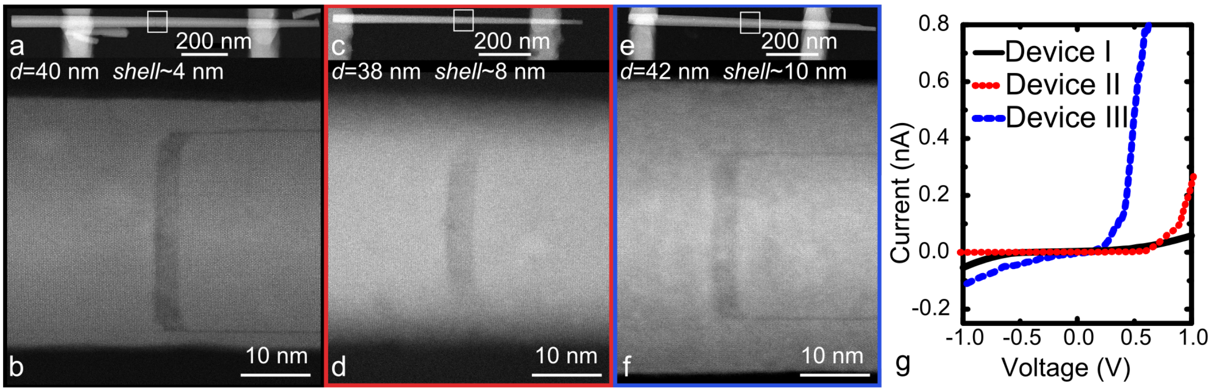

Figure 2.5: (a) HAADF STEM images (a, c, e) and corresponding zoom of the area around the AlN barrier (b, d, f) of NW devices I, II, and III, respectively. Devices I and III are viewed along $[2\bar{1}\bar{1}0]$ while device II is viewed along $[10\bar{1}0]$. The diameter d at the AlN barrier and the respective average GaN shell thickness (shell) are indicated in (b, d, f). (g) Smoothed I?V characteristics measured in the dark. The top of the NW (left side in images a, c, and e) was connected to ground.

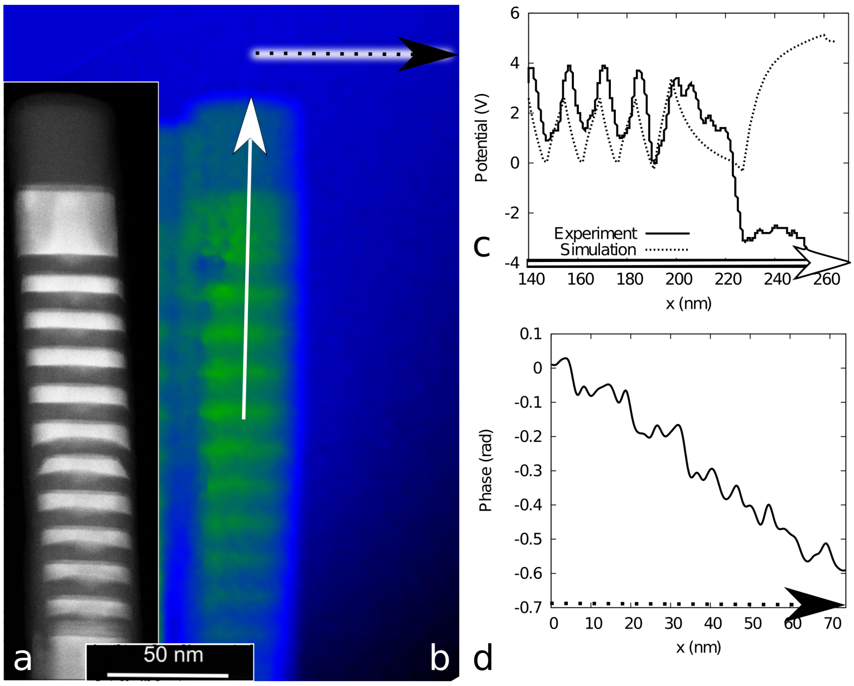

Figure 2.6: (a) HAADF STEM image of a GaN/AlN NW heterostructure. (b) Phase image of the same NW. (c) Potential profile obtained from the phase along the vertical white arrow compared with a simulated profile including a surface charge density of -2x10^{13} electron charges cm^{-2}. (d) Phase profile in the vacuum obtained along the horizontal black dotted arrow.

structure in one and three dimensions. This work nicely illustrates that the polarity can be used as a tool in device design. This work, as well as the next publication, was performed in collaboration with Jonas Lähnemann, a postdoc that recently started a permanent position at PDI Berlin, and Maria Spies, who started a PhD in the framework of an AGIR grant fall 2016 with Eva Monroy, Bruno Gayral and me.

Studying the illumination power dependence of the photocurrent of such structures, we observed that they present a sub-linear dependence of the illumination power (González-Posada et al., 2013; Lähnemann et al., 2016; Lähnemann et al., 2017), which is also the case for the structures presented in (den Hertog et al., 2012). However, looking carefully at the direction of the applied potential, we found that in fact such NW heterostructures can behave as linear detectors, depending on the direction of the applied bias and the NW diameter. This work, started during the internship of Jakub Polaczyński and finished during the PhD of Maria Spies, was presented in **Effect of the nanowire diameter on the linearity of the response of GaN-based heterostructured nanowire photodetectors** (Spies et al., 2018), demonstrating that the depletion of the nanowire due to surface effects can be beneficial for the photodetector performance, since it allows the fabrication of linear devices if the nanowire heterostructure is properly designed.

2.3 Optical signature of defects in GaN NWs

Like any crystal, NWs can contain crystal defects as a function of growth conditions. For the study of crystal defects, NWs have the advantage over bulk material that they are more easily dispersed. Correlated structure-properties studies are unambiguously interesting, as they reveal the influence of a few disordered atoms on, for example, the photoluminescence signal of the NW. I was involved in two studies relating specific defects to their optical properties. Both studies used my developed sample preparation, but I was involved in the STEM characterization only for the first publication.

In the paper **Cathodoluminescence of stacking fault bound excitons for local probing of the exciton diffusion length in single GaN nanowires** (Nogues et al., 2014) we performed correlated studies of individual GaN nanowires in scanning electron microscopy combined to low temperature cathodoluminescence, microphotoluminescence, and scanning transmission electron microscopy. We showed that some nanowires exhibit well localized regions emitting light at the energy of a stacking fault bound exciton (3.42 eV) and are able to observe the presence of a single stacking fault in these regions. Precise measurements of the cathodoluminescence signal in the vicinity of the stacking fault give access to the exciton diffusion length near this location.

Moreover, in the study presented in **Attribution of the 3.45 eV GaN nanowires luminescence to inversion domain boundaries** (Auzelle et al., 2015) we attributed the 3.45 eV luminescence of GaN NWs grown by plasma assisted molecular beam epitaxy (PA-MBE) to the presence of prismatic inversion domain boundaries (pIDBs) using correlated experiments on single nanowires (NWs) by microphotoluminescence (μ-PL) and high resolution scanning transmission electron microscopy.

2.4 Correlated PL and TEM in GaN/AlN NW heterostructures

With the internships of Arunima Sethi in 2015 and Jakub Polaczyński in 2016 we started working on correlated μphotoluminescence (PL) experiments on a single GaN quantum dot (QD) insertion between AlN barriers in a GaN NW with scanning TEM characterization. These studies were performed in collaboration with Eva Monroy, Bruno Gayral, Bruno Daudin and Benedikt Haas. Catalyst free grown GaN NWs are characterized by a very high density NW growth. Due to the high density, NWs can coalesce and bundle together, complicating correlated single NW studies. For these reasons it can be difficult to find a single isolated and luminescing NW. Arunima obtained some first results. She studied both AlGaN QDs, which proved not very suited for correlated studies as the shape of the QD appears difficult to define from the TEM projection image, and inhomogeneities in the alloy are likely to create localization centers for PL, but are difficult to assess by TEM. Multiple PL lines were observed in most of the NWs. An example of such a NW and its spectrum is shown in Figure 2.7. Two distinct peaks are observed for the QD transition at 4.35 and 4.29 eV respectively. Three different peaks are observed near the GaN band edge. The two lower energy ones can potentially be explained by an inversion domain boundary and stacking fault as shown above, Section 2.3, but the high energy peak is not explained. It may be somehow related to coalesced NWs (best visible at the base, Figure 2.7b).

She also studied pure GaN QD insertions, that have a more well-behaved shape and are therefore easier to characterize by a single projection image in STEM. For example she studied the NW shown in Figure 2.8, where near band edge luminescence at 3.47 eV and luminescence at higher energy, 4.31 eV that is attributed to the QD, was observed, Figure 2.8a. The STEM analysis showed that the height of the QD is around four GaN lattice planes or $\simeq 1$ nm in the center and one or two lattice planes towards the NW surface, as visible in an intensity profile made in Figure 2.9. To verify what emission energy would be expected for such a QD, Eva Monroy performed 3D finite element simulations using Nextnano3, taking into account the NW dimensions as extracted from the STEM images. The results of these simulations and the comparison

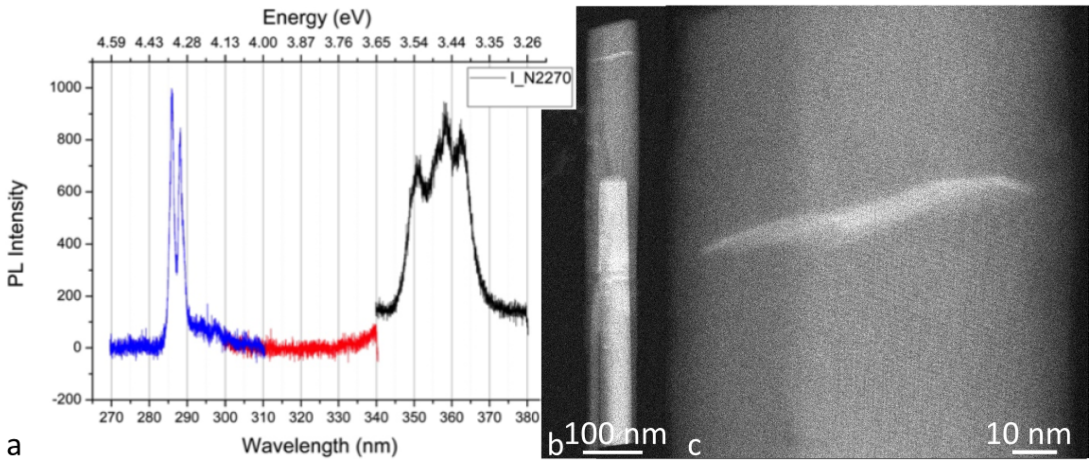

Figure 2.7: (a) μPL spectra measured at 10K. (b) Overview HAADF STEM image of the NW. (c) HAADF STEM image showing the AlGaN QD insertion surrounded by AlN.

with the experiment is presented in Figure 2.9c. In the figure the QD size for three and four monolayers of GaN are indicated by green boxes. The emission energy is very sensitive on the exact QD size in this region. Since the size of such crystals is quantized by the atomic lattice, we can't measure a QD size between 3 and 4 monolayers. Interestingly, placing our experimental point on the simulated trend-line, this could indicate that the QD size seen by the exciton is smaller than the measured size in atomic planes. Another potential explanation is that the emission energy is influenced by roughness at the AlN-GaN interface that is difficult to assess by TEM. We will need more statistics to see if any of these hypotheses could explain the emission of the QD. Moreover, we assessed the deformation of the QD, as shown in the geometrical phase analysis performed on the growth plane, the $(000\bar{2})$ reflection. While this analysis is noisy due to the small size of the QD, we find a lattice spacing in the QD of around 0.26 nm as shown in Figure 2.9b, but varying over the QD. The largest lattice spacing found was 0.263 nm and would correspond to a deformation of the GaN with respect to the relaxed GaN lattice of 0.014, agreeing well with the simulated deformation in the range of 0.012-0.013. However the precision of the current GPA analysis is not sufficient to draw definite conclusions.

Interestingly, we also observed the QD shown in Figure 2.10 The PL spectra for this NW can be seen in Figure 2.10a: there is a band edge luminescence at approximately 3.47 eV, characteristic of the GaN base. Unfortunately however, there is no peak at higher energy i.e. the QD does not luminesce. Observing the same NW in STEM we found a very original shape of the QD, that resembles a Stranski Krostanow grown QD on planar samples and is different from the usual planar insertion in a nanowire, Figure 2.10c,d. It seems that an inversion domain boundary (IDB) is present at the edges of the QD along the length of the nanowire, as can be best observed in the smoothed annular bright field image Figure 2.10d. Strangely however, the expected optical signature of the IDB mentioned earlier, Section 2.3, was not observed.

In conclusion, we could obtain some first correlated results on single GaN QD insertions in a NW, limited to four NWs. In three NWs a QD insertion was observed in HAADF STEM, however, no PL related to the QD was detected. Only one NW of the four showed QD related luminescence, shown in Figure 2.8. More correlated experiments are planned in the PhD of Maria Spies, were we also aim to combine in-situ biasing and μPL.

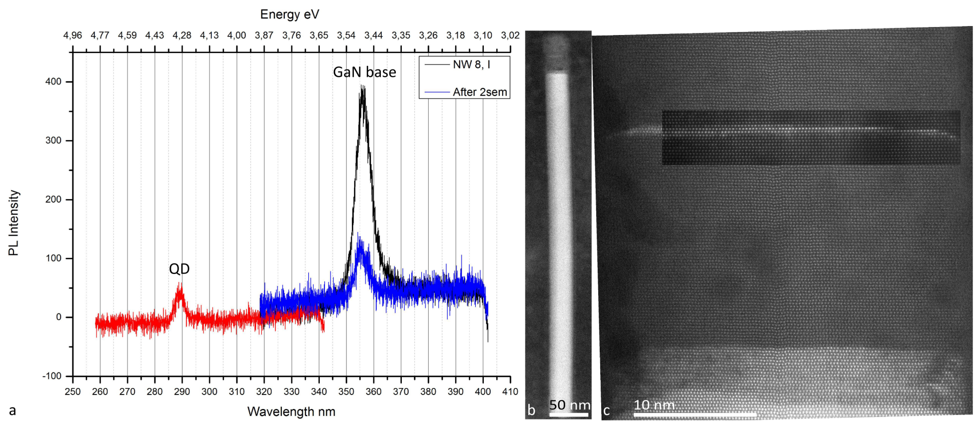

Figure 2.8: (a) μPL spectra taken at 10K showing the band edge luminescence of the GaN base and the QD insertion. (b) HAADF STEM image with the overview of the nanowire. (c) HAADF STEM image of the GaN QD inserted in a large AlN region. An averaged image with better signal to noise is overlaid on a normal (more noisy) image. The STEM characterization and image averaging were performed by Benedikt Haas on the Titan Ultimate.

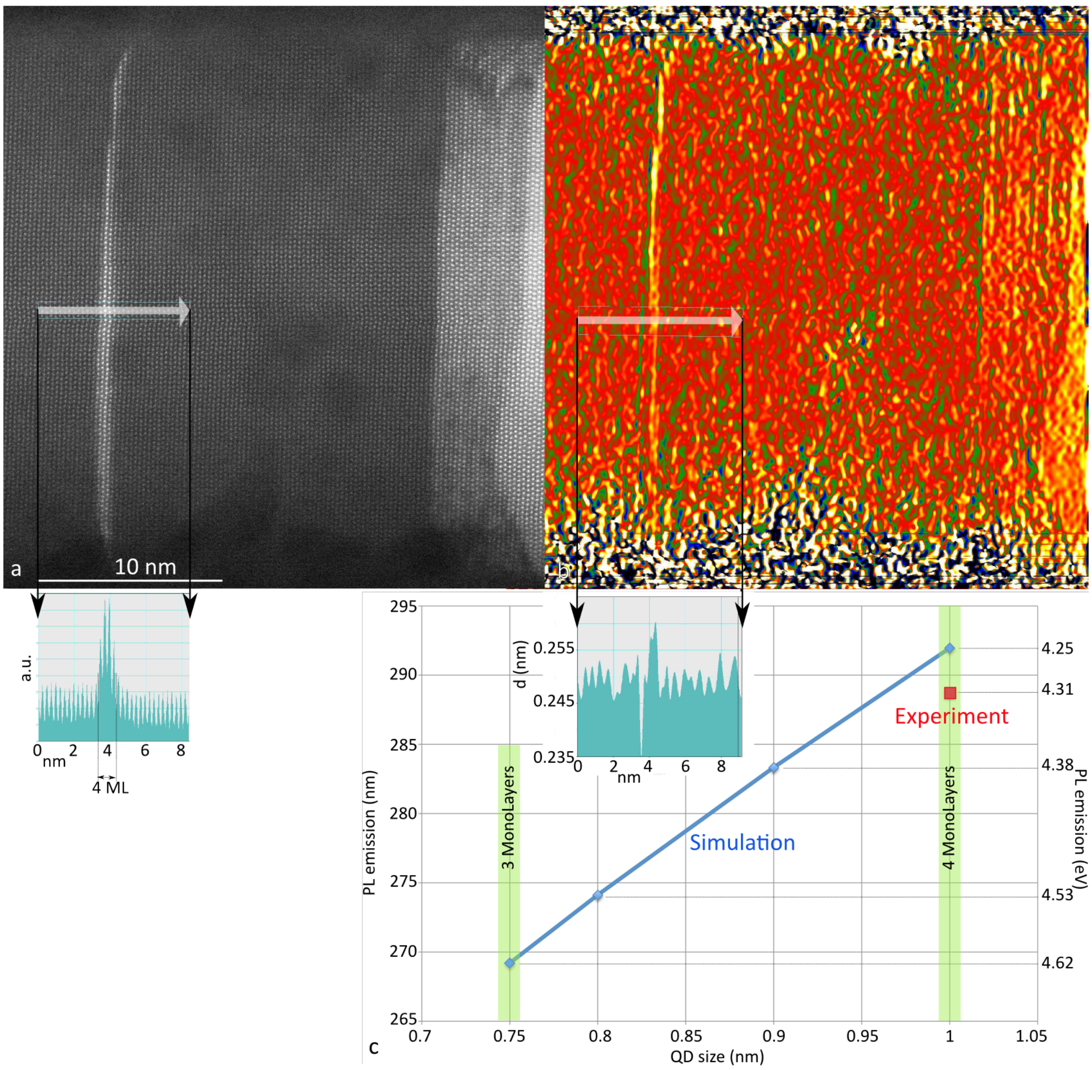

Figure 2.9: (a) HAADF STEM image of the QD insertions, similar to Figure 2.8b,c with an intensity profile below made at the thicker part of the QD. (b) Geometrical phase analysis of the same image, with a profile of the lattice spacing extracted at the thicker part of the QD. (c) Comparison of simulated and experimental photoluminescence of the QD.

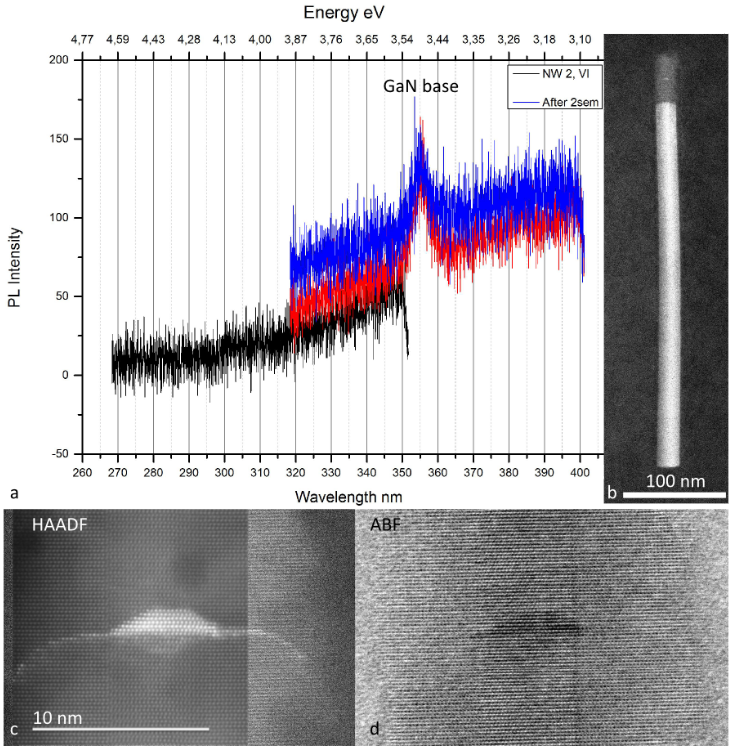

Figure 2.10: (a) μPL spectra taken at 10K showing the band edge luminescence of the GaN base. (b) HAADF STEM image with the overview of the nanowire. (c) HAADF STEM image of the GaN QD inserted in a large AlN region. An averaged image with better signal to noise is overlaid on a normal image. (d) Smoothed ABF STEM image showing two vertical lines that most likely indicate the presence of an IDB. The STEM characterization and image averaging were performed by Benedikt Haas on the Titan Ultimate.

2.5 Conclusion and Outlook

Several studies were done were structural properties could be correlated to electro-optical properties on the same unique NW (den Hertog et al., 2012; Spies et al., 2017; Lähnemann et al., 2017; Spies et al., 2018; Nogues et al., 2014; Auzelle et al., 2015). This allowed to define very precise input parameters regarding NW and heterostructure dimensions for simulations using Nextnano[3] 8-band k·p Schrödinger-Poisson equation solver (Birner et al., 2007) with the material parameters described in ref. (Kandaswamy et al., 2008). We have now well established fabrication methods and experimental protocols to perform such a correlation. Using electron holography we could obtain a qualitative view of the polarization fields in GaN/AlN NW heterostructures. Unfortunately, until now, we were not able to obtain a quantitative measurement of the electric field in such structures, which will be the object of future studies using in-situ biasing. The reason is that fabrication of samples for in-situ holography is more challenging than for a correlation of properties and structure only. For electron holography a vacuum region next to the sample is preferred. Therefore, to avoid interference of the nitride substrate (that also charges under the electron beam irradiation) suspended and contacted NW structures are needed, which are more difficult to fabricate.

Within the PhD of Maria Spies, entitled **Correlated electro-optical and TEM studies on single III-nitride nanowire heterostructures**, we plan μphotoluminescence experiments on a single GaN quantum dot (QD) insertion between AlN barriers in a GaN NW. Using the applied bias, the internal polarization field can be modified, and measured through the energy shift of the photoluminescence. We aim to correlate these experiments to the structural details of the insertion, such as size and strain. Moreover, to access the internal field by an alternative technique, off-axis electron holography, we have designed a relatively simple structure with a single large AlN insertion that we aim to electrically contact on membranes with holes, and study by holography in combination with in-situ biasing. Comparing the results with finite element simulations, we hope to obtain a more quantitative picture of the 3D potential distribution in such NW heterostructures.

3

IN-SITU ELECTRICAL BIASING EXPERIMENTS IN TEM

3.1 Metal propagation studies

Since 2010, I have tried to study the propagation of metals in NWs of group IV semiconductors: silicon and germanium. These studies were motivated by the work of Massimo Mongillo and Silvano De Franceschi (Mongillo et al., 2011) and my PhD work on dopant detection in silicon NWs using off-axis electron holography (den Hertog et al., 2009b). During my PhD we showed that doping contrast can be obtained in a NW, however, it was not possible to quantify the doping concentration. In planar p-n junctions, it has been shown that in-situ biasing can be used to extract the quantitative dopant concentration (Twitchett et al., 2004; Twitchett et al., 2002). In the work by (Mongillo et al., 2011) they show that a local Joule heating using a metal strip on the NW can be used to propagate a casi-metallic phase into the NW and is an interesting approach to obtain an atomically abrupt contact with low electrical resistance on NWs of group IV (silicon and germanium). Furthermore, I speculated that the thermally-induced propagation of the metal phase along the NW may lead to the modification of the doping concentration next to the contact due to the so-called doping pile-up or snowplow effect (Cojocaru-mirédin et al., 2007; Panciera et al., 2011), i.e. the dopant atoms are not integrated in the metallic phase but pushed forward into the semiconducting region. Moreover, the metal strip used for Joule heating can also be used as contact. Therefore, I thought this could be a perfect platform to simultaneously modify and measure the doping concentration quantitatively.

Initially, I worked on Ni contacts on Ge NWs in collaboration with Alois Lugstein's group from the Vienna University of Technology. However, since the NiGe phase diagram is very rich, we expected and saw different phases in the NiGe regions. Moreover, Ni is magnetic and therefore not suited for studies using electron holography to measure the electrostatic potential, as the magnetic signal could mask the electrostatic signals.

As an alternative approach, and in the framework of the ANR COSMOS, Khalil Elhajraoui studied both Cu and Al propagation in Ge NWs during his **PhD** (Hajraoui, 2017), focussing mostly on the diffusion mechanism of these metals, in collaboration with the Vienna University of Technology.

More recently, Minh Anh Luong started his PhD fall 2016 working specifically on **Doping engineering and characterization in germanium nanowires using in-situ transmission electron microscopy** using Al as contact material, in the framework of a LANEF PhD grant. Both PhD's rely heavily on model based quantitative and three dimensional EDX characterization, that proved very valuable. This was developed by Eric Robin (INAC, CEA-Grenoble).

In the following I give a brief description of these studies so far.

3.1.1 Cu diffusion in Ge NWs

In the paper in preparation **TEM analysis of copper - germanium nanowire solid-state reaction** we study the solid-state reaction between copper pads and Ge NWs using in-situ TEM observation and ex-situ characterization using Energy dispersive X-ray spectroscopy (EDX) and diffraction, with the aim to improve the quality of nanowire (NW) contacts. Two different methods of Joule heating to start the solid state reaction are studied and compared. Either a heating current is passed through the contact on the Ge NW, direct Joule heating H_a, leading to a very local heating, or a current is passed through a buried heating spiral, leading to a more conventional situation where the entire sample is heated, H_b. The location of the reaction interface is studied as a function of time, and interpreted using a diffusion model. The respective metal phase propagation experiments are presented in Figure 3.1, and EDX results are shown in Figure 3.2. Comparing the experimental data with a diffusion model, and supported by the EDX results, we could establish that at temperatures below 600°C, the reaction is limited by surface diffusion on the germanide and a Cu_3Ge phase is formed. At 600°C the reaction is limited by the metal-NW contact and a Cu_5Ge phase is formed. Novel model based EDX characterization reveal the 3D chemical cross section of the NW with a Cu_3Ge core, surrounded by thin layers of Cu (1 nm), Ge (0.35 nm) and GeO_2 (4.5 nm), an experimental observation of the surface diffusion layer. During the reaction, germanide crystals typically protrude from the reacted NW part. Their appearance can be avoided using a shell around the initial Ge NW.

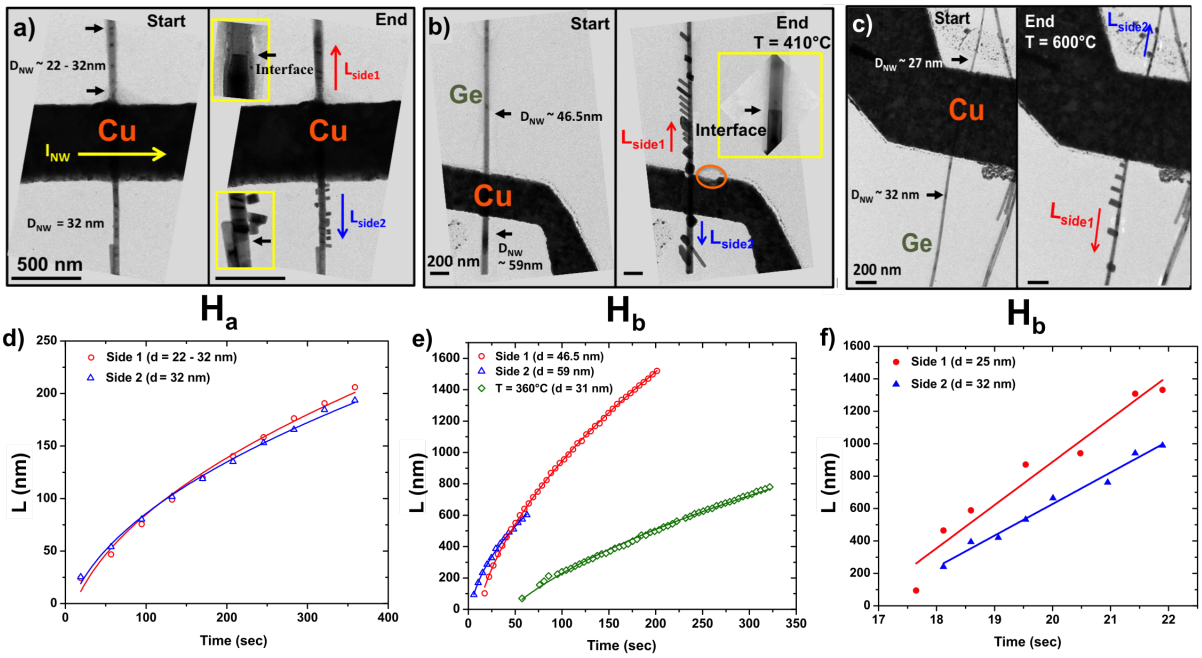

Figure 3.1: In-situ copper-germanium phase propagation experiments using both in-situ Joule heating (H_a) and membrane-substrate Joule annealing (H_b). a) TEM image showing the sample heated using in-situ H_a, both sides have a similar NW diameter (d_{NW} = 32 nm). b), c) TEM images of in-situ heating experiments H_b at two different temperatures 410°C and 600°C, respectively. The 2 sides of the NWs have different NW diameters. d) The length of germanide segment versus time at $\Delta V = 0,75V$ using in-situ H_a for both opposite propagation directions L_{side1} (○ ; fit (—)) and L_{side2}(△; fit (—)). e), f) The length of the germanide segment versus time at three different temperatures 360°C (◇ and fit (—))(TEM image not shown), 410°C (○ ; △ and fit (— ; —)) and 600°C (● ; ▲) and fit (— ; —). Profiles in *d* and *e* can be well fitted with a square root function (see eq (1)). Profiles in *f* fit well with a linear function. All the samples reveal an influence of the NW diameter on the reaction speed.

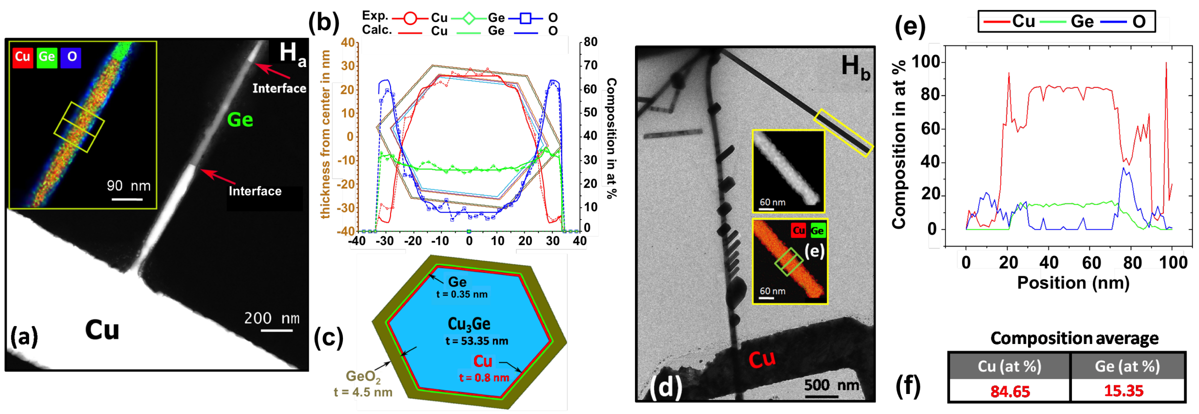

Figure 3.2: (S)TEM images and EDX quantification (line scan & hypermap) of different elements (O, Cu, Ge) in the NW after $H_a(V_{heat} = 0.775V)$ and H_b $(T = 600°C)$ experiments. (a) HAADF STEM image of a copper-germanium sample heated using H_a. The inset shows an EDX hypermap of the Cu_3Ge region. (b) Cu, Ge and O average concentration (at%) and local thickness profiles along the line shown in the inset in a. (c) schematic of the NW cross-section composed of different core-shell layers after heating. (d) TEM image of a heated sample at $(T = 600°C)$ using H_b. Inset images show a STEM and an EDX hypermap of the crystal formed after heating at $T = 600°C$. (e) Line scan of Cu, Ge and O concentration in atom % along the region defined in the inset image in d. (f) A local concentration in atom % of both Cu and Ge on the crystal core.

3.1.2 Al diffusion in Ge NWs

In the paper in preparation **TEM analysis of aluminum - germanium nanowire solid-state reaction** we present an in-situ transmission electron microscopy (TEM) study on Germanium nanowires contacted by aluminium metal allowing to form an axial metal - semiconductor - metal nanowire heterostructure using either a local (H_a) or a more conventional (H_b) Joule annealing process. In-situ TEM observations and ex-situ characterization using Energy dispersive X-ray spectroscopy (EDX) and conventional electron diffraction allowed a better understanding of the exchange mechanism between an Al pad and a Ge nanowire. The Ge of the nanowire is completely exchanged by the entering Al atoms. By tracking the reaction interface after the intrusion of Al in the Ge nanowire, two different propagation behaviors were observed. Using a local Joule heating technique or heating at temperatures below 400°C, the position of the interface as a function of time is well fitted by a square root function, indicating that the rate limiting step is a diffusion process. However, at temperatures above 400°C the position of the reaction interface appears to advance in tens of nm large steps. Model based EDX characterization reveals the 3D chemical cross-section of the transformed nanowire, with an Al core, surrounded by a thin pure Ge ($\sim$ 2 nm), Al_2O_3 ($\sim$ 3 nm) and Ge containing Al_2O_3 ($\sim$ 1 nm) layer respectively. In addition, the presence of Ge atoms in the Al metal line has been revealed by EDX analysis. Analysis of the kinetic data and comparison with a diffusion model indicate that Al volume diffusion is the rate limiting step in the exchange between the Al in the metal line and the Ge in the nanowire.

3.2 Doping quantification using electron holography and in-situ biasing

Quantitative characterization of electrically active dopants and surface charges in nano-objects is challenging, since most characterization techniques using electrons (Voyles et al., 2002; Wells and Goldberg, 1991; Batson, 1993), ions (Zelsacher et al., 2007) or field ionization effects (Blavette et al., 1999; Blavette et al., 1993; Moutanabbir et al., 2013) study the chemical presence of dopants, which are not necessarily electrically active. In the paper **In situ biasing and off-axis electron holography of a ZnO nanowire** (den Hertog et al., 2018) we present an holography in-situ biasing study of a contacted ZnO NW. We perform cathodoluminescence and voltage contrast experiments on a contacted and biased ZnO nanowire with a Schottky contact and measure the depletion length as a function of reverse bias, see Figure 3.3. We compare these results with state-of-the-art off-axis electron holography in combination with electrical in situ biasing on the same nanowire, presented in Figure 3.4.

The extension of the depletion length under bias observed in scanning electron microscopy based techniques is unusual as it follows a linear rather than square root dependence, see Figure 3.3f, and is therefore difficult to model by bulk equations or finite element simulations. In contrast, the analysis of the axial depletion length observed by holography may be compared with three-dimensional simulations.

The holography data in Figure 3.3f and Figure 3.5b are fit with an equation describing the variation of depletion width W with applied bias V_b in a bulk semiconductor system (Sze, 1985):

$$W = \sqrt{\frac{2\epsilon_s}{qN_D}(V_{bi} - V_b - \frac{kT}{q})} \qquad (3.1)$$

where V_{bi} is the built in potential, q is the electron charge, N_D is the doping concentration, ϵ_s is the relative permittivity (ϵ_s of ZnO was taken to be 8.7 (Yang et al., 2012)), k is the Boltzmann constant and T is the temperature in Kelvin. The same data are represented in Figure 3.3f and Figure 3.5b, both in linear and square y-axis scale, with the fit to Equation (3.1), to illustrate the bulk like nature of the NW core.

To extract the depletion width as a function of applied potential, the potential profiles extracted from treated holography data presented in Figure 3.5a are fit with a relation describing the potential $V(x)$ in a depleted semiconductor region near a metal-semiconductor interface in a bulk planar system (Sze, 1985) given by

$$V(x) = \frac{qN_D}{\epsilon_s}(Wx - \frac{1}{2}x^2) - V_{bn} \tag{3.2}$$

where V_{bn} is the barrier height. Figure 3.5b then compares the experimentally determined depletion width with the depletion width extracted from finite element simulations using different combinations of doping and surface charge.

The comparison of experiment and simulation allows estimating an n-doping level of $1\text{x}10^{18}$ cm^{-3} and negative sidewall surface charge of $2.5\text{x}10^{12}$ cm^{-2} of the nanowire, resulting in a radial surface depletion to a depth of 36 nm. We found excellent agreement between the simulated diameter of the undepleted core and the active thickness observed in the experimental data. By combining TEM holography experiments and finite element simulation of the NW electrostatics, the bulk-like character of the nanowire core is revealed.

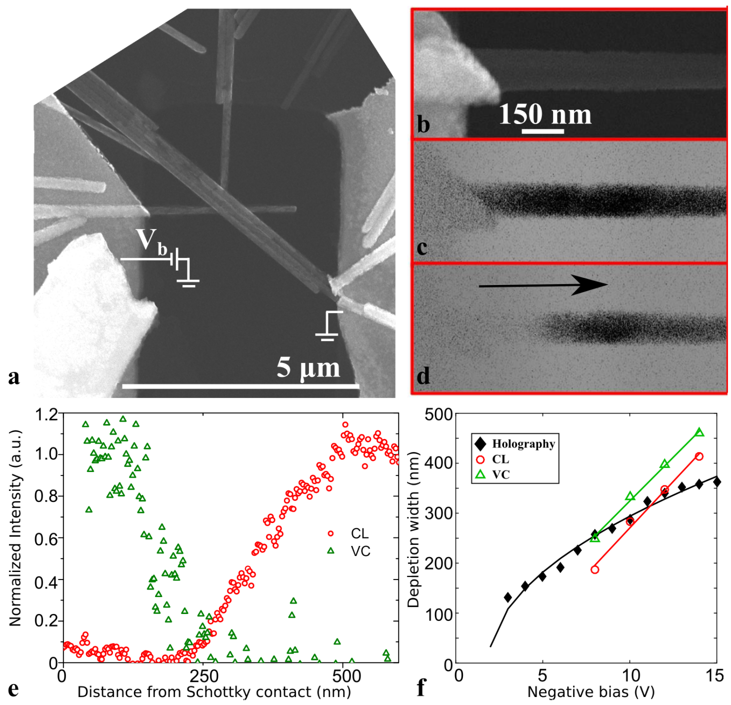

Figure 3.3: (a) SEM image of a contacted and suspended ZnO NW on a hole in a Si_3N_4 nitride membrane. (b) Zoomed SEM image at the biased contact. (c) CL map obtained at 0 V bias. Maximum intensity corresponds to dark contrast. (d) CL map obtained at 8V bias. (e) Profiles extracted from CL and VC measurements obtained by scanning from left to right, i.e. away from the Schottky contact biased at 8V, in the direction of the arrow in (d). (f) Depletion width as a function of the negative bias deduced from CL and VC, and fit with a linear function (solid lines), compared to the holography data with a fit to Equation (3.1) (solid line).

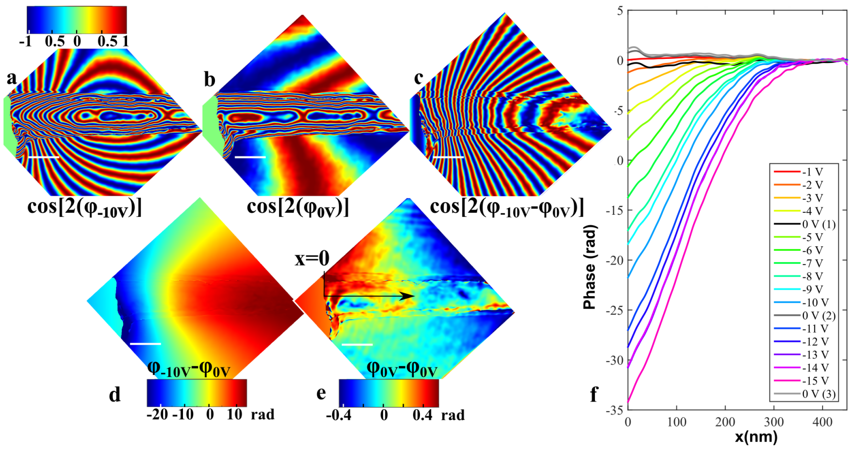

Figure 3.4: Description of data treatment where φ is the phase change of the electron wave that passed through the sample with respect to vacuum: (a)(c) $\cos[2(\varphi_{-10V})]$ (b) $\cos[2(\varphi_{0V})]$ and (c) $\cos[2(\varphi_{-10V} - \varphi_{0V})]$ of the averaged phase. The color scale is given in a for (a)(c). (d) Resulting phase image after subtraction of zero-bias (same data as in (c)). (e) Phase difference between two image sets at zero-bias, one made at the start and the other one made after the biasing experiment. Scale bars in (a)(e) are 150 nm. (f) Phase profiles obtained at the center of the NW (averaged over 43 nm) as a function of the reverse bias, obtained along the arrow as positioned in (e) (x=0 marks the edge of the Schottky contact). Three traces in zero-bias difference images (greyscale) are shown, acquired in between and after the biasing experiments, following the sequence as indicated in the legend. The profiles were normalized using the average value of the flat phase in the region 350-450 nm for each bias.

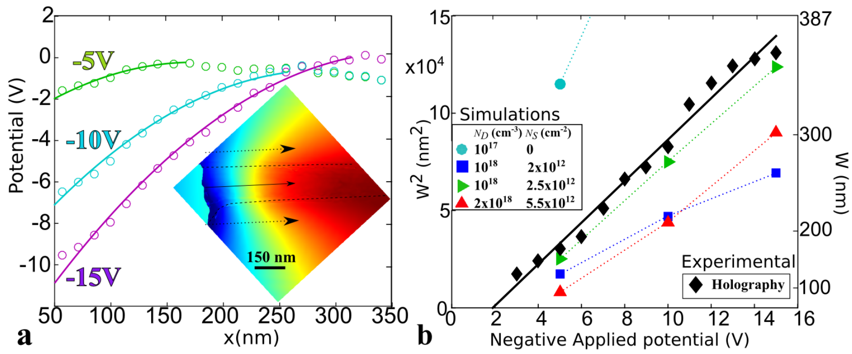

Figure 3.5: (a) Potential measurements extracted from phase profiles at 5V, 10V and 15V bias (o) with fits (solid lines, Equation (3.2)) to extract the depletion width. The spacing of symbols reflects the spatial resolution in the experiment (14 nm). The average signal in the vacuum was subtracted from the NW signal and the remaining phase signal was converted to potential using a thickness of 75 nm. The profile along x does not start at x=0 because the vacuum correction could not be extended to the contact. The inset shows the location of the phase profile in the NW core (solid arrow) and two symmetrically defined phase profiles (dotted arrows) obtained in the vacuum on either side of the NW (outline indicated by dashed lines). (b) Comparison of 3D calculations using the Nextnano3 software (symbols connected by dotted lines) including different doping (N_D) and negative surface charge (N_S) values in and on the NW, and experimental depletion widths obtained using electron holography from the data shown in a (diamond symbols) with fit (solid line, Equation (3.1)).

3.3 Conclusion and Outlook

We were able to perform Joule heating in-situ TEM studies, focussing on the formation of metal contacts using both Cu and Al in Ge NWs. Moreover, we have performed a first in-situ biasing and off-axis holography study on ZnO NWs, demonstrating that the depletion length can be visualized as function of bias in these structures. It will be interesting to see if in-situ biasing in combination with holography can also be applied to the Al-Ge system.

Minh Anh Luong started his PhD fall 2016 working specifically on **Doping engineering and characterization in germanium nanowires using in-situ transmission electron microscopy** using Al as contact material. He obtained interesting first results, that may indicate that no doping pile-up occurs in Phosphorus doped Ge NWs with Al contacts. However, it appears that dopants are mostly present in the near surface region of the NW, or in a shell deposited on the NW surface to protect the Ge from oxidation, given the low quality of the native GeO_2. Within his PhD we aim to contact Ge NWs containing a p-n junction suspended over a hole in the membrane. Using Al metal propagation, the metal contacts can then be advanced some 100's of nm's from the junction and used for in-situ biasing. Using in-situ biasing we hope to obtain quantitative information on the electrically active dopants in the NW. We then aim to correlate the quantitative dopant concentration found by holography with results from EDX, to establish the fraction of electrically active dopants. We have performed a first in-situ biasing experiment on such a structure, however, unfortunately no p-n junction was present between the metal contacts. Such a structure that represents an Ohmic current voltage characteristic can still be interesting as from the change in phase slope in the semiconductor region with applied bias it could be possible to measure the local NW resistivity (Yazdi et al., 2013), that also reflects its doping. Unfortunately, the quality of the current data are not sufficient, most likely due to limited fringe contrast in the NW under study due to its thickness.

4

OUTLOOK

4.1 Measuring charges in nanostructures

From the previous chapters we have seen that we have now the tools to make contacted NWs compatible with TEM characterization. I would like to focus in the coming years on studying charges/electric fields/potential in a NW under bias using off-axis electron holography, following on the work we did on the ZnO NW. I will mainly focus on GaN/AlN NW heterostructures, and Ge NWs with Al contacts. In the GaN/AlN samples we will aim to visualize and manipulate the strong polarization fields that are present in these structures. In Ge NWs we will try to measure and modify the potential drop at a p-n junction, and potentially visualize the extension of the depletion region at Schottky contacts.

I have proposed the ERC project **e-See**: *Single electron detection in transmission electron microscopy*, with the aim to spatially locate and electrically control a single or discrete number of charges in the device volume with atomic spatial resolution, to image the shape of the wave function and assess the influence of the local environment. The idea builds on the experience acquired with Ge NWs with Al contacts using it as a model system for state of the art transistors made of silicon. Using this system a small QD of Ge can be fabricated between Al contacts, and a Coulomb blockade can be observed if the sample is cooled to sufficiently low temperature and studied electrically. Roman Kramer, Cecile Naud and Olivier Buisson, researchers at Institut Neel, have already observed Coulomb Blockade in this NW system. We aim to fabricate samples compatible both with low temperature transport as well as TEM and holography: we need to add a gate and have a vacuum region next to or underneath the NW. A first step will be to perform transport prior to TEM on the same contacted NW sample. The next step is to perform the low temperature transport in the TEM. Using the Coulomb blockade effect we could block a well defined number of charges in the QD. Then we could make a TEM image, and hope the number of charges has not been modified by the electron beam. Then, defining a different number of charges in the QD using the electrical connections and making again an image, the difference image will show the location and extension of the wave function of one or multiple charges. The aim is that this technique can visualize the location of a single or multiple number of charges present on states due to a single dopant atom. Single dopant atoms located in the channel of state-of-the-art transistors are a growing problem for semiconductor industry since they generate differences between nominally identical devices and increase the off-current and transistor power consumption. Having a method to visualize these states at the nm or atomic scale can give more insight into these states and how to avoid them. The project will start on contacted NWs using them as model system, and can potentially extend to real transistors fabricated in LETI, and already studied by low temperature transport by several groups in Grenoble: SPSMS (CEA Grenoble) and Quantum Coherence (Institut Neel).

This project is challenging: the most critical points are the influence of the electron beam on the sample, and the development of an electrical cryogenic He sample holder.

Concerning the first challenge: The electron beam might destroy the Coulomb blockade. We will vary all accessible parameters, for instance acceleration voltage and electron dose, to see if a regime can be found where the Coulomb Blockade can be maintained under the electron beam. As we combine TEM and transport we will know if the state still exists by looking at a differential conduction map. If we can't maintain Coulomb blockade under the beam we have several mitigation options:

- Use dopants that are further below the conduction band, for example Se is 300 meV below the conduction band in Silicon. This will create a situation where the level is more stable to perturbation, and more difficult to perturb. We can still use Se atoms as a model system for more shallow dopants. However, its clear that 300 meV is a tiny amount with respect to 80 or 200 kV, which is the standard acceleration voltage in TEM. (For this idea a different sample geometry is proposed from the Ge NWs with Al contacts, as described in more detail in the complete project description inserted at the end of this section.)

- Mask the sample from the electron beam and look at fields in the vacuum. We probably need to put more than one charge to get enough signal, however, this idea should work since no beam will hit the sample.

- Another option is to use a focused small TEM probe and to do an electron beam induced current (EBIC) experiment, monitoring the current in the blocked situation. The current is zero except if the beam hits the level and delocalizes the trapped electron, giving current. This alternative method can potentially indicate the location of the level and wave function. This approach has the additional advantage that preliminary experiments could be carried out in an SEM at Neel, equipped with EBIC and a He cooled cryogenic stage, as well as electrical contacts.

The second challenge involves developing in-situ TEM at He temperatures. He holders have been developed by the Oxford company and can be bought commercially from the society GATAN (that acquired the IP). However, there are concerns about the mechanical stability and the accuracy of the measured temperature near the sample. With the cryogenics department at Institut Neel, we have started to fabricate a first prototype for a He holder compatible with an FEI TEM. In parallel we will also test the commercially available He holders for the different TEM types.

This ERC project will start in October 2018, and will involve the acquisition of a new transmission electron microscope at the Institut Néel. The short version of the project (B1) is enclosed below for a more detailed description.

ERC Starting Grant Research proposal (Part B section 1 (B1))

Single electron detection in Transmission Electron Microscopy

e-See

Proposal duration: 60 months

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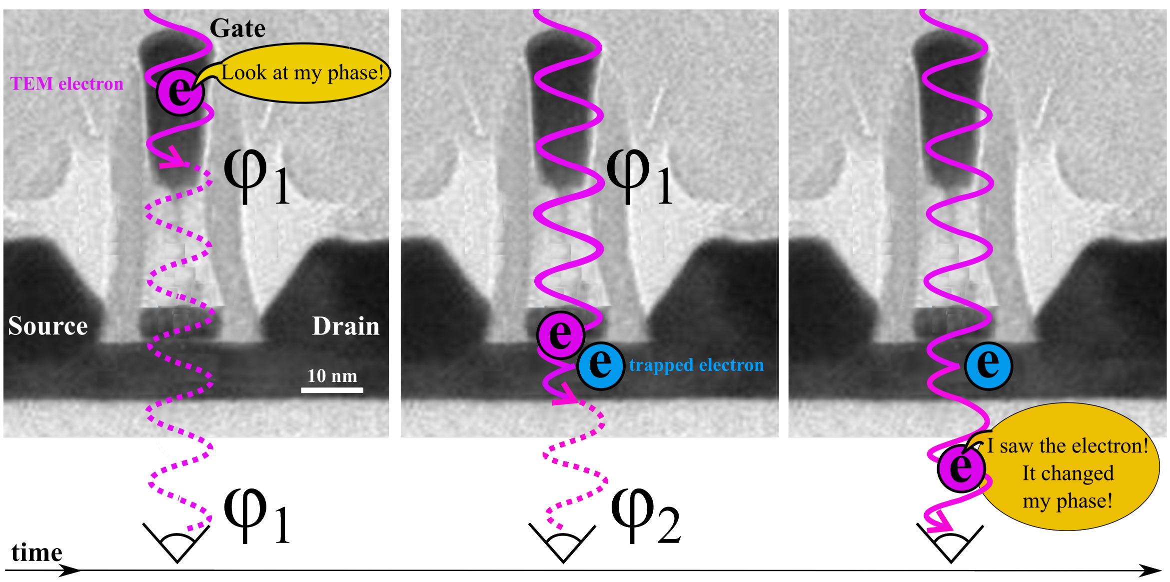
FRANCE

Summary:

The ultimate goal of device miniaturization is to rely on a single charge provided by a single dopant atom: solotronics. Currently the gate length in a transistor cannot be reduced beyond 10-12 nm, as variability between nominally identical devices reaches unacceptable levels. Elaborate quantum transport experiments can monitor the presence and spin state of a single charge, but do not provide information about location and distribution (wavefunction) of the charge or the local chemical and crystallographic environment. The latter, however, determine why the charge is present at a specific location with a particular distribution. Scanning probe techniques can measure charges but are restricted to the near surface region. In contrast, the phase of an electron in transmission electron microscopy (TEM) can probe the sample volume and is sensitive to charge. **The target of the e-See project is the first real time observation of the wavefunction associated to a single electron charge** in the volume of a device with atomic resolution. I aim to implement low temperature quantum transport experiments in a TEM to allow simultaneous electrical manipulation of this charge. Combined visualization and manipulation of a single charge trapped by Coulomb blockade in a transistor will (i) identify the origins of device variability, and (ii) show how the local properties of the sample affect localization of a single charge and its wavefunction. The project impact involves understanding of variability, improving device design and creation of a new research field on low temperature electrical *in situ* TEM experiments. It will provide the tool to visualize a single charge wavefunction in any device, **enabling ultimate device engineering**: deterministic 3D atomic scale control of the position of charge localization. To this end, I will use electron holography and scanning TEM, develop a low temperature electrical TEM sample holder, and novel sample preparation.



Section 1a: _Extended Synopsis of the project proposal_ (max 5 pages)

INTRODUCTION

Today's transistors are fabricated using a multitude of lithography, etching and metal deposition steps to fabricate a short and thin semiconducting nanowire (NW) contacted by metallic source and drain contacts underneath a gate. Due to the increased device miniaturization following Moore's law, increasing device variability is observed for decreasing gate lengths due to the random distribution of dopants under and at the edge of the gate[1,2]. This effect has prevented the semiconductor industry to decrease the gate length beyond around 10-12 nm as device variability reaches unacceptable levels. Understanding and reducing device variability is therefore a pressing concern that needs urgent solving.

Though scanning probe techniques such as scanning tunneling microscopy (STM) have a high spatial resolution combined with a high electrical sensitivity, they are inherently limited to the near surface regions of the sample[3]. From the ionization energy of a dopant and coupling to source and drain, an approximate location of the dopant atom can be inferred[2] by electrical characterization, however, these experiments do not yield any spatial information about the location and extension of the charge, nor do they present direct information on the local chemical or crystallographic environment. Transmission electron microscopy (TEM) is a technique capable of atomic scale spatial resolution through the sample volume. Furthermore the entire palette of TEM techniques gives access to all the relevant properties of the sample at the atomic length scale, for instance crystal structure, defects, strain and the chemical composition. The only drawback of TEM is that a sufficiently thin sample is required, and that the thinning procedure may modify the sample properties. In this project, I entirely eliminate this risk by using bottom-up grown NWs that are already thin as model system, and by using a backside wet etching technique I am patenting, allowing thinning of real devices without altering the electrically active regions. **As TEM uses electrons to 'see' the specimen, they are inherently sensitive to the charge in the sample: the phase of a transmitted electron will be modified by a single charge**, see Fig 1. My team will test off-axis and in-line holography as well as electron diffraction in scanning TEM mode for single charge detection at atomic length scales.

The absence of a technique to visualize single charge in the volume of a working device means that device engineering is done 'in the dark'. Obviously, as 'Seeing is believing', visualization of ultimately single charges in the volume of a device, and correlation with local properties, would greatly improve our understanding of devices and consequently lead to improved device design, reduced variability and improved performance. **The aim of the e-See project is to visualize or 'see' a single charge, a single electron generally denoted by '*e'*, in a working device, in real time, with atomic scale spatial resolution, and simultaneously manipulate this charge electrically in a Coulomb blockade experiment**. I will combine the atomic scale spatial resolution that can be achieved routinely in TEM with the sensitivity of low temperature transport experiments, to characterize the extension of the charge and its coupling to the environment and how the local properties of the sample (crystal structure, chemistry, interface quality) influence the localization of a single charge.

SCIENTIFIC APPROACH

My team will use three TEM based characterization techniques: i) Off-axis electron holography ii) In-line holography and iii) electron diffraction in scanning TEM (also referred to as differential phase contrast).

i) Off-axis electron holography is a transmission electron microscopy interference technique where an electron beam passing through the sample is interfered with an e-beam passing through vacuum (reference wave), see Fig. 1. From the interference pattern, the phase of the electron wave can be deduced, which is sensitive to the projected electrostatic potential, the strain and the magnetic field[4]. It has been shown that single charge sensitivity can be obtained with this technique[5] within defined contours. However, if a single charge, not induced by the electron beam, can be imaged with nm or atomic scale precision remains an open question in the field of TEM. Furthermore, doping contrast and polarization fields can be observed using this technique in semiconducting nanowires (NWs) [6] (several publications and a book chapter of my authorship). Recently, I succeeded in mapping the depletion width as a function of *in situ* applied bias to a Schottky contact on a ZnO NW (article submitted by PI). Comparison with finite element 3D potential simulations allows quantitative measurement of the doping concentration and surface charge in and on the NW, and demonstrates for the first time the bulk like character of the NW core. The required phase resolution to map a single electron with a lateral resolution of 0.1 nm has been numerically evaluated to be $2\pi/30$ at 200 kV electron acceleration[7], however this number is likely to be over-estimated as detection of light atoms like hydrogen is not state of the art. State of the art microscopes can attain a phase resolution of $2\pi/200$ at 200 kV[8]. Furthermore the phase resolution can be further improved by acquiring series of holograms and averaging[9] (done for my recent results on a reverse biased ZnO NW with a Schottky contact) and using next generation cameras with single electron sensitivity and an improved modulation transfer function. Using an improved biprism (this is a charged wire that interferes the two e-beams, see Fig. 1) design (startup M. Duchamp) and double biprisms could also potentially increase the phase resolution[10] and will give more flexibility concerning interference fringe spacing and pattern width.

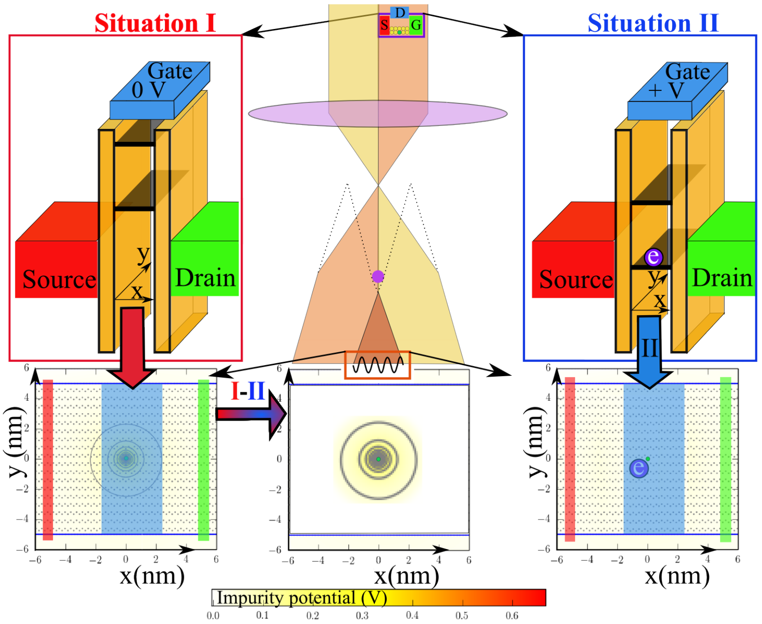


Fig. 1: ***Graphical outline of the project****. Center: A TEM technique, here off-axis holography, is used on an electrically connected sample with* ***source gate*** *and* ***drain****. Off axis electron holography is a technique where an electron wave traversing the sample is interfered with a wave traversing only vacuum with the help of a charged wire, the biprism. In the image plane an interference pattern is obtained, from which a phase image can be deduced. Using source, gate and drain we can either put zero (situation I, ionized impurity) or one electron (screened impurity, situation II) in the channel. A schematic of the chemical potential for both situations is shown. Then we obtain images of both situation I (zero charge) and II (1 electron charge). Taking the difference image will visualize the single charge and probability distribution with atomic scale spatial resolution*[11]*.*

ii) Potentially the measurement sensitivity can be improved using in-line electron holography, where the difference between two images at different defocus values can be related to the charge density using the transfer of intensity equation[12]. The difference of the charge densities of situation I and II (see Fig. 1) would reveal the location and extension of a single charge.

iii) Recently a **quantitative measurement of atomic electric fields** was demonstrated using a convergent probe in **Scanning TEM (STEM),** by looking at modifications in the intensity distribution in the transmitted disk, the so-called ronchigram, of the diffraction pattern[13]. The advantage of holography is that the image is obtained simultaneously, allowing faster acquisition and possibly reducing the influence of the e-beam, while in STEM mode the sample is scanned point by point. The advantage of in-line holography and STEM is that no vacuum region is required next to the region of interest, and atomic resolution is slightly more easily obtained. Therefore three techniques will be tested against the task of single charge detection.

Working at lower acceleration voltage is interesting, as both the electron–specimen interaction and the related phase signal are increased. Furthermore, it will avoid beam damage. The last, but most important point is that my team will use *in situ* **electrical biasing of the sample**. In the case of a Coulomb blockade experiment we will subtract the phase, charge density image or ronchigram with no electrons in the quantum dot (QD) from the image with an electrically defined number of electrons in the QD. Using this difference signal method all signals due to static contributions, for example chemical contrast, will be removed, see Fig. 1. Electrical biasing has been demonstrated for the simple case of a p-n junction[14], and electrical biasing and subtraction of the 0V image were used for experiments on a contacted ZnO NW.

Current electronic devices work at room temperature (RT); however at typical currents (~µA), gate lengths (~10 nm) and mobility (~1400 $cm^2\ V^{-1}s^{-1}$), on average less than one electron (0.05 e) is present in the channel of the device. Therefore it is challenging to measure current in a working device at RT, as the signal will be low and delocalized over the entire channel. I propose to use **Coulomb blockade experiments at low temperature as a tool to localize charge and visualize this charge**. Although in some systems Coulomb blockade can be observed at room temperature[15], many more systems can be studied if the temperature can be lowered. Furthermore the same QD can be studied at different temperatures. Therefore we aim to be able to work both at liquid nitrogen temperatures (77-85 K) and He temperatures (4-10 K). Single charges can be trapped and manipulated when the temperature becomes sufficiently low, depending on the capacitance of a QD separated by tunnel junctions from source and drain and controlled by a gate, leading to Coulomb blockade (see left and right schematics in Fig. 1). The quantum transport group SPSMS (CEA Grenoble) currently studies devices fabricated in CEA-LETI by low-temperature quantum transport. **To allow quantum transport experiments in the TEM my team will develop a mechanically stable low temperature (4-10 K) TEM sample holder with electrical contacts.** As indicated in Fig. 1, we will characterize the projected channel of the device both along x and y directions, which is entirely novel. We can even access the third dimension by acquiring images at various tilt angles, obtaining projections along different observation directions. We will be able to relate the exact shape of the Coulomb diamonds that reflect the capacitances of gate, source and drain and the charging energy of the QD with the observed 3D position of charge localization and its local environment. Furthermore, we can observe how the wave function is affected by local properties of the sample near the charge localization. For example: the presence of an interface, the quality of this interface (interface roughness) or the local strain distribution. We will first use bottom-up grown semiconducting nanowire (NW) structures as model system for top-down nanowires that are currently used in CMOS devices. I have already developed methods to fabricate NW devices on nitride membranes or suspended over a hole in the membrane (papers 1-3 from selected own publications). These membranes are transparent to the electrons used in transmission electron microscopy (TEM) and therefore compatible with the entire palette of TEM based techniques that will be used following the *in situ* experiments to obtain detailed structural (defects, strain, interface structure) and chemical information of the observed device. Structural and chemical data can then be correlated with the electrical characterization. Side or top gates can be defined on the NW using a thin layer of a light metal like Al or Ti, to avoid excessive scattering of the electron beam. We focus on three distinct nanowire devices:

i) NWs of group IV: I have already a large experience on *in situ* TEM metal phase propagation in Ge NWs,[16] with Ni, Cu or Al as metal. Using local Joule heating of a metal strip on one end of the NW[17], silicide or germanide phases can be propagated into the NW, see Fig. 2. By propagating the metal phase from both ends into the NW, a semiconducting QD can be fabricated between metallic leads. The size of the QD can be determined with atomic scale precision, and progressively decreased to measure size influence on the transport properties of the same device. Furthermore, the QD can be aligned perfectly with the gate. Using either non-intentionally doped, or low doped NWs, statistically one or few dopants will be present in the semiconducting regions that we will study with a combination of transport measurements and TEM.

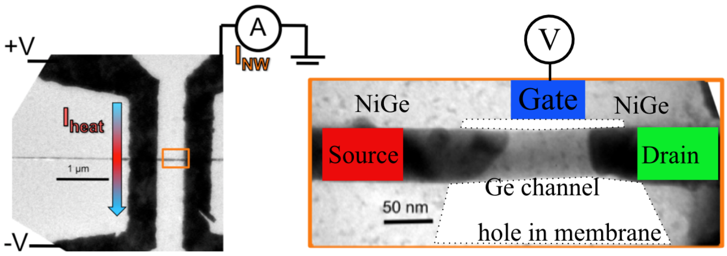


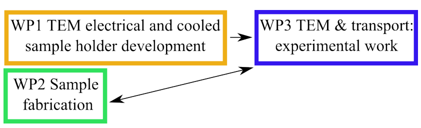
Fig. 2. Left: TEM image of a Ge NW connected by two Ni strips on a 50 nm Si_3N_4 membrane. A current is flown through the left strip, heating the strip due to Joule heating and starting a NiGe phase propagation. Flowing current through the right strip propagates the phase from the right. Right: TEM zoom of the final situation: a Ge region or quantum dot between two metallic leads A gate and hole in the membrane are drawn schematically to indicate the type of samples that will be fabricated. (1 article submitted by PI, 1 in preparation).

ii) InAs-InP NWs: To reduce the risk of the previously proposed structure, my team will fabricate NW devices of InAs-InP NW heterostructures to take advantage of the well-established fabrication procedures and of Coulomb blockade that is relatively easily observed.

Samples from group I and II are model systems that will allow a proof of principle experiment. Once a proof of principle experiment has been demonstrated, the method will be applied to:

iii) Transistors from CEA-LETI and IBM Zurich, fabricated by etching, deposition and implantation techniques, will be made TEM compatible by a local backside wet etching of the silicon wafer and buried oxide that I am currently patenting. Fig. 3 illustrates the nitride membrane fabrication procedure (3a-d), the local backside etching (3e) and the target sample structure both in top view (f) and cross section (g). My team will focus on devices pre-selected by electrical characterization and study devices from a group exhibiting the desired characteristic and a group exhibiting anomalous behavior.

WORK PLAN



The project contains 3 WPs: TEM holder design (*WP1*), sample fabrication technology (*WP2*) and experimental work (*WP3*), interrelated as represented in the schematic on the right. Besides central coordination, I will be involved in all WPs with 70% of my time, including experimental work, with a focus on WP2 and 3. A web site to show the project goals and achievements will be created for dissemination and I will organize a workshop.

WP0. Transmission electron microscopy context: The project needs an aberration corrected state of the art TEM suitable to perform electron holography and scanning TEM at low (80-120 kV) acceleration voltage with sufficient access to microscope time. Institut Neel will order a new TEM in 2017 that will be accepted in 2018. The new TEM will be installed in a dedicated room in a newly (2014) delivered low noise building designed for nanoscience. The ERC will use the new TEM for 25% of its time. Complementary experiments will be performed in collaboration at PFNC Grenoble, CEMES Toulouse and potentially in other leading groups (IWF Dresden (A. Lubk), Singapore (M. Duchamp) and EMAT Antwerp (J. Verbeeck)).

WP1. TEM electrical and cooled *in situ* sample holder design: The cryogenic part of the holder will be designed and developed by engineers of Institut Néel, in collaboration with the *in situ* TEM holder company DENSSolutions. At Institut Néel my team has already fabricated TEM sample holders and holders with electrical connections hosting TEM samples were fabricated to insert the same sample into a cryostat. My team and collaborators will combine the knowledge in cryogenic development at He temperatures with the holder fabrication know-how, including thermal shields and a cold trap in the design. DENSSolutions has a large experience in *in situ* TEM holders and will give feedback on the compatibility of a cryogenic solution with the required stability. Successful development will be jointly patented and commercialized by DENSSolutions. Additionally, we might modify and use the nitrogen cold trap, present in TEMs, with He.

WP2. Sample fabrication: Two different types of NWs, grown in collaborating groups, will be electrically connected using lithography techniques on nitride membranes. The new ingredient will be the addition of a gate electrode that should be either a side or a thin top gate, made of a light metal. Furthermore a vacuum region should be present near the structure to influence the reference wave as little as possible. My team has already fabricated membranes with holes and suspended and electrically connected NWs. Local backside KOH etching was already tested at the Institut Néel and will be further developed to allow *in situ* TEM observation on single electron transistor devices from the semiconductor industry fabricated in CEA-LETI. These devices will first be studied by transport in the SPSMS team, and transferred to the e-See project.

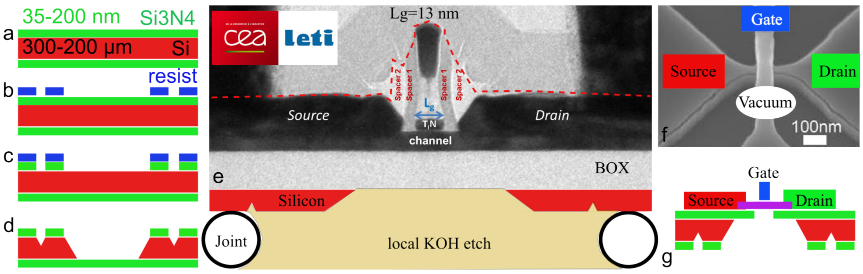


Fig. 3 Membrane fabrication procedure (a-d). e) TEM cross section image of a device[18] and schematic. All structure above the red dotted line will not be present in specimens aimed at in the project (backend). ***A local KOH back etch*** *(yellow) will allow creating a membrane of SiO_2 (BOX) supporting the device layer. f) Top[19] and g) Cross section of* ***target sample structure****. Schematics are not on scale.*

WP3. *In situ* TEM & transport: experimental work: This WP will deal with the actual experimental work. (3.1) Preliminary transport experiments in a TEM sample. (3.2) Coulomb blockade in TEM sample under the electron beam, assess influence on differential conductance map due to the electron beam. (3.3-3.4) Holography/STEM in combination with transport experiments to detect a single charge. (3.5) The characterization of the same device by other TEM techniques to provide an ultimate correlation between electrical properties and local structure. I am an expert in various TEM techniques including TEM, HAADF STEM, off-axis electron holography and EDX.

COMPETITORS: Other groups (CEMES, Toulouse; D. Cooper, CEA-Grenoble; Rafal Dunin-Borkowsky, Ernst Ruska-Centre Jülich; Hannes Lichte, Dresden; Martha McCartney, Arizona State University; H. Zandbergen, TU Delft) are working on related projects, combining electron holography, *in situ* biasing and/or low temperature. Globally three differences in approach with respect to **e-See** can be outlined: i) Often the focused ion beam (FIB) technique is used for thinning of the sample, this introduces the risk of electrically modifying the device, ii) The FIB can also be used for contact formation, or the contact can be established with a movable probe, in both cases its difficult to obtain high quality Ohmic contacts to the device. iii) There are projects to develop a dedicated cryogenic TEM with cold stage and electrical contacts.

In contrast I propose an approach based on home-fabricated nitride membranes. The membranes provide a substrate that is transparent to electrons in the TEM and compatible with the same lithography techniques on bottom-up grown NWs as used for fabrication of real devices, assuring measurement of intrinsic sample properties and good contact quality. I have been working on this type of sample fabrication for over five years and have established protocols for successful fabrication. To my knowledge no other groups have developed such methods. My team will work on bottom up grown NWs as a model system and use a local etching technique to work on real devices. The advantage of a robust cryogenic electrical TEM holder with respect to a cryo TEM (an alternative solution) is that it will be much more flexible than a dedicated TEM and could be fitted to any TEM, making the technique accessible to many more researchers.

MAIN RISKS AND FEASIBILITY*:* The cryo electrical *in situ* holder (WP1) has a risk, as both the cryogenic aspect and the TEM environment are challenging. At T18 there will be a decision moment: if the fabrication is not advanced sufficiently a commercial solution will be acquired: a Gatan electrical He cooled holder that can go to 6K and has 4 electrical contacts,[20] while continuing the holder development.

Unfortunately, on the Gatan holder the electrical connections to the sample are made in an impractical way, rendering the holder difficult to use[21], but allowing proof of principle experiments. The main risk is that Coulomb blockade can't be maintained under the electron beam due to beam-specimen interaction. The possibility of single charge detection represents an open question in the field of TEM. Mitigation plans involve studying Coulomb blockade without irradiating the object, using a mask, and looking at the electric fields in the vacuum due to single or multiple charges.

The host institution 'Institut Néel' is a CNRS institute. **Both Institut Néel and CNRS are fully dedicated to this ERC project**: assisting with complementary funding for a new state of the art TEM prior to the ERC starting date and the salaries of CNRS engineers working on the project. Though the e-See project is ambitious, high risk and high reward, it is feasible as all the necessary competences are gathered in the team: CNRS employed engineers that are highly dedicated to this project, specialized in development of cryogenic experimental setups for the cryogenic electrical biasing TEM sample holder, as well as engineers assisting with TEM maintenance and sample fabrication. Two postdoctoral fellows and a PhD student will work in the project, also interacting with researchers in CEMES Toulouse (A. Masseboeuf). Samples will be fabricated in the cleanroom Nanofab of Institut Néel. Transistors fabricated by CEA-LETI will be studied first in the SPSMS group (quantum transport) and transferred to the e-See project. One postdoctoctoral fellow will also interact with the SPSMS group. Y.M. Niquet (CEA-Grenoble) is working on non-equilibrium Green function simulations of the 3D electric field around a trapped charge in a single electron transistor as part of the MOSQUITO project (H2020, start 2016).

THIS PROPOSAL: TARGETS AND EXPECTED IMPACTS

This project aims to develop ***low temperature quantum transport experiments with atomic scale spatial resolution.*** The envisioned targets and open questions that will be answered include:

- The first observation of a single charge in the volume of a working device with atomic spatial resolution.
- The first imaging of the probability distribution of the wave function of this charge. How is the wave function affected by the local (di)electric environment, for example: the presence of an interface nearby?
- Study of charge related phenomena: point defects, impurities and interfaces. For example, Tunnel Field Effect Transistors (TFET) could decrease power consumption, as the subtreshold slope can be steeper than in Metal Oxide Semiconductor FETs, however charge traps degrade their performance.
- The first observation when more electrons are placed in the quantum dot and how the wave function is affected. Can we add a second charge to a single dopant? See Fig. 1.
- The first direct correlation of local structural and chemical properties with the location of trapped charge, wave function extension and shape of the Coulomb diamonds. Can we understand the device performance from the position of charge localization and is this position always related to a dopant atom?
- Validation of existing simulation codes of charges in nanostructures.
- The possibility to control the sample electrically gives the unique opportunity to define the number of charges in the QD, therefore the measured TEM signals can be interpreted quantitatively and compared with simulations, giving yet unknown information on beam-charge interaction.

Expected Impacts include:

- For transistors: **Improved understanding of device variability**. Providing the tool for ultimate device design: i.e. deterministic 3D atomic scale control of the position of charge localization. Once my team has acquired a certain statistics on a number of devices that show electrical characteristics as a function of where in the structure charge localization occurs, we will discuss with the SPSMS team, CEA-LETI and IBM Zurich which geometry demonstrates the target device performance, and which fabrication strategies could push towards this architecture. Iteration between fabrication and characterization will lead to improved fabrication strategies.
- Many subjects in physics related to charge transfer (devices, solar cells etc.), magnetism or superconductivity, could benefit as atomic scale, real time mapping of the sample volume at low temperature with electrical control is currently not possible. The cryogenic in-situ holder would allow observation of charge and magnetism related phenomena and the e-See project could inspire other researchers to use the same sample preparation approach. Patented successful holder development, WP1, can therefore create **a new research field**.
- Improved device design and observation methods and related patents will contribute to market share and benefits for European based semiconductor and electron microscopy companies and generate **jobs** in Europe. Semiconductor industries such as CEA-LETI and ST-Microélectronics are present on the same research campus or nearby, facilitating collaboration and interaction with the e-See project.

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[11] Simulation courtesy Dr. Y.M. Niquet. The situation of the screened impurity (right) is simplified: there will be a small remaining potential. The atoms are visible both for the ionized and screened impurity, but removed by the subtraction, to illustrate removal of all contrast not related to varying electrical phenomena.
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[21] The holder is electrically dangerous for the samples: I have often damaged samples in a Gatan biasing holder due to an electrical accident.
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Nomenclature

BF	Bright Field
EDX	Energy Dispersive X-ray Spectroscopy
GPA	Geometrical Phase Analysis
HAADF	High Angle Annular Dark Field
HR	High resolution
HRTEM	High Resolution TEM
ML	monolayer
NW	Nanowire
PL	Photo Luminescence
QD	Quantum Dot
RIE	Reactive Ion Etching
SEM	Scanning Electron Microscopy
STEM	Scanning Transmission Electron Microscopy
TEM	Transmission Electron Microscopy
ZA	Zone Axis

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EDUCATION, RESEARCH AND PROFESSIONAL EXPERIENCE

Since 2010 **Junior Researcher** (**current position**, permanent position at CNRS), group Materials Radiation and Structure at Institut Néel CNRS, Grenoble (France). Topic: *Correlation of transmission electron microscopy (TEM) with electrical and optical characterizations on semiconducting nanowires (NWs)*. Responsible of PhD's and intern students.

2009-2010 **Postdoctoral Fellowship**, ANR project, CNRS Institut Néel, Grenoble, France. Advisor: Dr. C. Bougerol. Topic: *Correlated TEM and optics on II-VI nanowires*.

2006-2009 **PhD Thesis** Physics (mention très bon) prepared at CEA-INAC, University Joseph Fourier, Grenoble, France. Advisor: Dr. J.L. Rouvière. Thesis: *Characterization of silicon nanowires by transmission electron microscopy*. FP7 fellowship.

2003-2005 **Master of Science** "Chemistry and Physics" (with honours**,** av. 8.1/10), University of Utrecht, the Netherlands. Advisor: Prof. Dr. A. Polman, Amolf. Thesis: *Pulsed CO_2 laser printing and smoothing of Si quantum dot solids (7.5/10)*. Advisor: Prof. Dr. P. van der Straten. Thesis: *Measuring the temperature of a Rb MOT using an oscillating magnetic field (9/10)*.

2000-2003 **Bachelor of Science** Chemistry (with honours, av. 7.5/10), University of Utrecht, the Netherlands. Advisors: Prof. Dr. A. Meijerink. Thesis: *Cooperative quantumsplitting in the $(Yb_xY_{0.99-x})_2O_3$:Tb1% system*.

TEACHING AND SUPERVISING EXPERIENCE

2015 Course 'Chemistry of Crystals', (20 hours) 36 bachelor students, Grenoble Alpes University.

2009-2015 Several TEM sample preparation trainings (25 hours), 30 permanent staff/students, Grenoble

2010 Supervision experimental courses (32 hours) 12 first years students PHELMA, Grenoble.

POSTDOC ADVISOR

2015- Dr. Jonas Lähnemann, Collaboration between Institut Néel (Dr. M. den Hertog) and CEA-Grenoble (Dr. E. Monroy). Now permanent researcher at PDI Berlin.

2011-2012 Dr. Fernando González-Posada. Now assistant professor at University of Montpellier. Collaboration between Institut Néel (Dr. M. den Hertog) and CEA-Grenoble (Dr. E. Monroy)

PhD SUPERVISOR

2016 - Minh Anh Luong, "*Doping engineering and characterization in germanium nanowires using in-situ transmission electron microscopy*". Co-supervised with E. Robin CEA-Grenoble.

2016 - Maria Spies, "*Correlated electro-optical and TEM studies III-N nanowire heterostructures*". Co-supervised with E. Monroy and B. Gayral CEA-Grenoble.

2015 - Akhil Ajay, "*GaN/AlGaN nanowires for quantum devices*". Co-supervised with

Dr. E. Monroy.

2015 - Daria Beznasyuk, "*High-mismatch Si/InGaAs nanowire heterostructures for quantum optics*". I supervise the TEM aspect of the PhD (Thesis supervised by M. Hocevar).

2013-2016 Khalil El–Hajraoui, "*Diffusion of Cu and Al in germanium nanowires studied by In situ TEM metal-semiconductor phase propagation*". I am the principal supervisor of this work.

2013-2015 Pamela Rueda, "*HRTEM characterization of ZnTe nanowires*". In the framework of her PhD on growth of ZnTe NWs I have supervised the TEM aspect of the PhD.

In addition, I also supervised four Master, two Bachelor and a visiting PhD student in the framework of an ERASMUS program (3 months).

ORGANIZATION OF SCIENTIFIC EVENTS

2010-2017 I have given several seminars on transmission electron microscopy in general for different groups in my institute. I have also organized user meetings on TEM.

2017 Organization of in-situ TEM workshop in Grenoble.

2017 Gave electrical in-situ TEM practical at the international Quantitative Electron Microscopy 2017 TEM school held in Balaruc les Bains.

CAREER BREAKS

2017 Four months maternity leave for the birth of Meije Verbeek, followed by working at 80%.

2013-2014 Four months maternity leave around the birth of Sam Verbeek, followed by working at 80% during 6 months, again full-time since September 2014.

MAJOR COLLABORATIONS

National collaborations:

- Different groups at CEA Grenoble, including the NPSC (Nanophysics and semiconductors) group (Dr. E. Monroy, Dr. B. Daudin, Dr. B. Gayral, Dr. E. Bellet-Amalric) for the synthesis and optical characterization of semiconductor nanowires since 2009 (27 publications), the electron microscopy group LEMMA since 2006 (Dr. J.L. Rouvière, Dr. E. Robin, 13 publications), the quantum transport group SPSMS since 2013 (Dr. S. De Franceschi, Dr. X. Jehl), SINAPS since 2006 (N. Pauc, P. Gentile, 11 publications) and LTM since 2006 (B. Salem, T. Baron, 9 publications): both groups working on group IV nanowires growth and device fabrication.
- CEMES Toulouse since 2010 (Aurelien Masseboeuf, Christophe Gatel) for the development of off-axis electron holography in combination with *in situ* biasing. Also interested in cryogenic in-situ.

International collaborations:

- IBM Zurich since 2008 (Dr. H. Riel, Heinz Schmid and Stephan Wirth, 2 publications).
- Institute for Solid state electronics, Vienna University of Technology since 2010 (Dr. Alois Lugstein, 1 publication) for *in situ* metal phase propagation in Ge NWs. PCH Campus France 'Amadeus' project.
- Johan Verbeeck, EMAT Antwerp: fabrication of devices for new phase electron microscopy.
- Justus-Liebig Universität Giessen since 2014 (Prof. Dr. M. Eickhoff) on the topic of nitride NW growth for correlated opto-electrical and TEM characterization.

– Universidad Politécnica de Madrid since 2015 (Dr. Z. Gacevic) for holography in nitride NWs.

COMISSIONS OF TRUST

– Elected member of the PLUM Department Council of Institut Neel since 2017
– Elected member of the Laboratory Council of Insitut Neel since 2016.
– Member of the French Microscopy Society (SFμ).
– Member of the evaluation committee for the PhD defense of Vidar Fauske, 29 June 2016, "*Electron microscopy based characterization of semiconductor nanowires*" and Jelena Todorovic, 3 december 2012, "*Correlated transmission electron microscopy and micro-photoluminescence studies of GaAs–based heterostructured semiconductor nanowires*" at the Norwegian University of Science and Technology in Trondheim.
– Referee of international journals (recently Journal of Applied Physics (1), Nanotechnology (1), NanoLetters (3), Journal of Luminescence (1), Ultramicroscopy (1), Nanoscale (1)).

Funding ID

On-going grants, *Past grants*

Project	Funding source	Amount (Euros)	Period	Role	Involved PhD students
TEMPO	*FMN (local)*	*20kE*	*2011-2012*	*PI*	
UVLAMP	*ANR*	*1199kE (363kE Néel)*	*2012-2014*	*Responsible TEM*	
MAGWIRE	*ANR*	*600 kE*	*2012-2015*	*Responsible TEM*	*Pamela Rueda*
COSMOS	*ANR*	*302kE*	*2013-2017*	*PI*	*Khalil Elhajraoui*
EMOUVAN	ANR	613 kE	2015-2019	TEM and devices	
ESPADON	ANR	600 kE	2015-2019	TEM	Marta Orru
PhD grant	GANEX + ERC TERAGAN	100 kE	2015-2018	GANEX + ERC TERAGAN	Akhil Ajay
AMADEUS	PHC (travel expenses)	15 kE	2016-2017	PI	Khalil Elhajraoui and Minh Anh Luong
PhD grant	LANEF (local)	100 kE	2016-2019	PI	Minh Anh Luong
PhD grant and budget	AGIR (local)	100 kE + 10 kE	2016-2019	PI	Maria Spies
e-See	ANR T-ERC	150 kE	2017-2018	PI	
e-See	ERC	2 ME	2018-2023	PI	*Starting Oktober 2018*

Scientific contributions of Martien den Hertog

Below are my scientific contributions, divided in first author publications, publications as contributing author, invited seminars/conference presentations and oral and poster presentations at conferences. Within the list of publications there are 8 publications in *Applied Physics Letters*, 8 publications in *Nanotechnology*, 4 publications in *Journal of Applied Physics*, 13 publications in *Nano Letters* and 3 publications in *Physical Review B*. The summary of my scientific work can be found here: `http://www.researcherid.com/rid/B-1912-2015`.

Publications as a first author

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   In-situ Biasing and Off-axis Electron Holography of a ZnO Nanowire, Nanotechnology **29** 025710 (2018).
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2. M. I. den Hertog, R. Songmuang and E. Monroy.
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3. M.I. den Hertog, R. Songmuang, F. Gonzalez-Posada, and E. Monroy.
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   `http://jjap.jsap.jp/link?JJAP/52/11NG01/`
4. M. I. den Hertog, F. González-Posada, R. Songmuang, J. L. Rouviere, T. Fournier, B. Fernandez, and E. Monroy.
   Correlation of Polarity and Crystal Structure with Optoelectronic and Transport Properties of GaN/AlN/GaN Nanowire Sensors,
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5. M.I. den Hertog, C. Cayron, P. Gentile, F. Dhalluin, F. Oehler, T. Baron, and J.L. Rouviere.
   Hidden defects in silicon nanowires,
   Nanotechnology **23** 025701 (2012).
   `http://stacks.iop.org/0957-4484/23/025701`
6. M.I. den Hertog, M. Elouneg-Jamroz, E. Bellet-Amalric, S. Bounouar, C. Bougerol, R. André, Y. Genuist, J.P. Poizat, K. Kheng and S. Tatarenko.
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7. M.I. den Hertog, M. Elouneg-Jamroz, E. Bellet-Amalric, S. Bounouar, C. Bougerol, R. André, Y. Genuist, J. P. Poizat, K. Kheng, and S. Tatarenko.
   Insertion of CdSe quantum dots in ZnSe nanowires: Correlation of structural and chemical characterization with photoluminescence,
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8. M.I. den Hertog, M. Elouneg-Jamroz, E. Bellet-Amalric, S. Bounouar, C. Bougerol, R. André, Y. Genuist, J.P. Poizat, K. Kheng and S. Tatarenko.
   Insertion of CdSe quantum dots in ZnSe Nanowires: MBE growth and microstructure analysis,
   Journal of Crystal Growth **323** 330 (2011).
   `http://www.sciencedirect.com/science/article/pii/S0022024810011413`
9. M.I. den Hertog, H. Schmid, D. Cooper, J.L. Rouviere, M.T. Björk, H. Riel, P. Rivallin, S. Karg and Walter Riess.
   Mapping active dopants in single silicon nanowires using off-axis electron holography,
   Nano Letters **9** 3837 (2009).
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10. M.I. den Hertog, J.L. Rouviere, H. Schmid, D. Cooper, M. T. Björk, H. Riel, F. Dhalluin, P. Gentile, P. Ferret, F. Oehler, T. Baron, P. Rivallin, S. Karg and W. Riess.
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11. M.I. den Hertog, PhD thesis.
    Caractérisation de nanofils de silicium par microscopie électronique en transmission, (2009).
    `http://tel.archives-ouvertes.fr/tel-00493934/en/`
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Book chapters

1. Chapter: *Electron Holography of Nanowires - part 2* in the book *Semiconductor nanowires: Materials, synthesis, characterization and applications*,
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   Demonstration of a 2 x 2 programmable phase plate for electrons,
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   `https://www.sciencedirect.com/science/article/pii/S0304399117305041?via%3Dihub`
2. M. Orrù, E. Robin, M. den Hertog, K. Moratis, Y. Genuist, R. André, D. Ferrand, J. Cibert, E. Bellet-Amalric.
   Nanowire growth and sublimation: CdTe quantum dots in ZnTe nanowires,
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Invited Seminars

- APMAS Conference, Istanbul, Turkey. *In-situ biasing of semiconducting NWs in transmission electron microscopy: doping quantification and contact formation*, June 1-3, 2016.
- EMN Nanowire conference, Amsterdam, the Netherlands. *In-situ biasing of semiconducting NWs in transmission electron microscopy: Doping quantification and contact formation*, Mai 16-19, 2016.
- DPG Frühjahrstagung Regensburg, Germany. *Fabrication and study of metal contacts on germanium nanowires using electrical biasing in a transmission electron microscope* , March 7-11, 2016.
- 14th Colloque de la Société Française des Microscopies , Nice, France. *Characterization of metal contacts on semiconducting Nanowires using electrical biasing in a transmission electron microscope*, 1 July 2015.
- 18th European Molecular Beam Epitaxy Workshop, Canazei, Italy. *Correlating Optoelectronic and Transport Properties of GaN/AlN Nanowires with Polarity and Crystal Structure*, 18 March 2015.
- International Microscopy Conference, Prague, Czech Republic. *Correlation of Optoelectronic and Transport Properties of GaN/AlN Nanowires with Polarity and Crystal Structure*, 9 September 2014.
- GDR NACRE Workshop on coupling between nano crystals and dopants, Villers sur mer, France, *Characterization of dopants and electric fields in semiconducting nanowires by off-axis electron holography*, 10 septembre 2013.
- 7th Nanowire Growth Workshop, Lausanne, Switzerland, *Transmission electron microscopy of semiconducting nanowire devices*, 11 june 2013.
- ImagineNano, Bilbao, Spain, *Correlation of polarity and crystal structure with optoelectronic and transport properties of GaN/AlN nanowire sensors*, 24 april 2013.
- Nanowire Workshop 10, Heraklion, Greece, *Transmission Electron Microscopy characterization of semiconducting nanowires*, 28 september 2010.
- Journée thématique: "Dopage dans les nanofils", Rouen, France, *Dopant mapping in silicon nanowires using Off-axis Electron Holography*, 12 march 2010.

Conferences - Oral presentation

1. Journées Nationales des Nanofils Semiconducteurs, Grenoble, France. *In-situ biasing and off-axis electron holography of a ZnO nanowire* , 14 November 2017.
2. European Microscopy Conference, Lyon, France. *Quantitative measurement of doping and surface charge in a ZnO nanowire using in-situ biasing and off-axis electron holography*, 29 August 2016.
3. International conference on one-dimensional nanomaterials, Annecy, France, *Correlation of polarity and crystal structure with optoelectronic and transport properties of GaN/AlN nanowire sensors*, 2013.
4. Microscopy of Semiconducting Materials, Oxford, U.K., *Correlation of polarity and crystal structure with optoelectronic and transport properties of GaN/AlN nanowire sensors*, 2013.
5. European Microscopy Conference, Manchester, United Kingdom, *Determination of polarization fields in single GaN/AlN nanowire heterostructures by direct correlation between high resolution electron microscopy and electronic transport*, 2012.

6. GDR nanofils, Porquerolles, France, *Correlation of structural, chemical and optical properties of CdSe quantum dot insertions in ZnSe nanowires*, 2011.
7. Microscopy of Semiconducting Materials, Cambridge, U.K., *Correlation of structural, chemical and optical characterization of luminescent CdSe quantum dots inserted in ZnSe nanowires*, 2011.
8. MBE 2010, 16 internation conference on molecular beam epitaxy, Berlin, Germany, *Correlation of structural, chemical and optical characterization of luminescent CdSe quantum dots inserted in ZnSe nanowires*, 24 august 2010.
9. Microscopy of Semiconducting Materials, Oxford, U.K., *Dopant mapping in silicon nanowires using Off-axis Electron Holography*, 2009.
10. Holography workshop, Dresden, Germany, *Electron Holography of silicon nanowires*, 2008.
11. Société Franąise des Microscopies Colloque 2007, Grenoble, France, *Structural properties of Gold Catalyzed Silicon Nanowires: Defects in the wires and Gold on the Wires*.
12. GDR nanofils, Lyon, France, *Defects in the wires and Gold on the Wires*, 2007.
13. Microscopy of Semiconducting Materials, Cambridge, U.K., *Gold Catalyzed Silicon Nanowires: Defects in the wires and Gold on the Wires*, 2007.

Conferences - Poster presentation

1. GDR nanofils, Porquerolles, France, *Hidden defects in silicon nanowires*, 2011.
2. Holography workshop, Toulouse, France, 2011.
3. European Microscopy Conference, Aachen, Germany, *Control of gold surface diffusion on silicon nanowires*, 2008.
4. Trends iN Nanotechnology 2006, Grenoble, France, *Evidence of gold on lateral surfaces of gold catalyzed silicon nanowires*, poster received a distinction.
5. JMC, Toulouse, France. GDR nanofils, Lille, France, 2006.

Other scientific contributions

- Organization committee member and lecturer of the practical 'in-situ biasing in TEM' at Quantitative Electron Microscopy 2017 school in Balaruc les Bains.
- Member of the evaluation committee for the PhD defense of Vidar Fauske, 29 June 2016, *Electron microscopy based characterization of semiconductor nanowires*, at the Norwegian University of Science and Technology in Trondheim.
- Member of the evaluation committee for the PhD defense of Jelena Todorovic, 3 december 2012, *Correlated transmission electron microscopy and micro-photoluminescence studies of GaAs–based heterostructured semiconductor nanowires*, at the Norwegian University of Science and Technology in Trondheim.