

***In-situ* electrical and structural characterization of individual GaAs nanowires**

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Abstract. A method for probing the electrical and structural characteristics of individual as-grown III-V nanowires was studied. *In-situ* electrical characterization was performed in a focused ion beam / scanning electron microscopy system by using a fine nano-manipulator and ion beam assisted deposition. Transmission electron microscopy specimens of probed nanowires are prepared afterwards. This method would potentially allow the correlation of electrical and structural characteristics (e.g. crystal faults such as twinning) of the nanowire-substrate system. The challenge is in contacting the nanowires so that the electrical characteristics of the nanowire-substrate system can be extracted correctly.

1. Introduction

Several interesting optoelectronic applications are envisaged for III-V nanowires (NWs), amongst others for solar-cells and light emitting diodes [1]. To move towards devices, precise, homogenous positioning of high quality NWs on low-cost substrates is desired. This requires optimization of both the positioned growth, and the NW crystal structure. To use the growth substrate as an electrode for devices, it is also necessary to optimize the electrical properties of the combined NW-substrate system.

There exist several methods to synthesize NWs, but the most commonly used are based on the vapour-liquid-solid mechanism [2]. Here, a liquid catalyst droplet adsorbs precursor vapour, and facilitates the growth of a solid, crystalline NW. If grown on an appropriate substrate, the crystal growth will nucleate in such a manner that epitaxial growth is achieved. For GaAs NWs, as studied here, common catalysts include Au and Ga, the latter of which is termed self-catalyzed growth. For reproducibility and implementation into devices, positioned growth is required, by either controlling where the catalyst droplets are, or where the crystal can nucleate. Specifically, one way of doing this is by patterning a silicon oxide mask on the substrate [3]. The Ga droplets only form in the holes of the oxide mask, and thereby a position controlled NW growth is achieved.

In general, to measure the electrical properties of a system, it is important to be able to achieve good ohmic contacts. Particularly for GaAs, making ohmic contacts is challenging due to its high level of surface states, requiring proper pre-surface treatment and choice of best contact materials [4].

Here we studied a method to measure the electrical properties of as-grown GaAs NWs in a focused ion beam (FIB) system, and to subsequently characterize the same as-grown NW by (scanning) transmission electron microscopy ((S)TEM) for different NW-substrate systems. This should allow for



the correlation of electrical and structural data of individual NWs, and to verify any effects of e.g. crystal defects, on device performance [5].

2. Materials and methods

The NWs used in this study were grown using the self-catalysed growth technique in a solid source Varian Gen II Modular molecular beam epitaxy system. The substrates used in this study were p-type Si(111) and Kish graphite. In total, four GaAs NW samples were studied. In sample A, intrinsic GaAs NWs were grown on a Si substrate for a duration of 25 min with an As flux of 5.6×10^{-6} Torr, and at a substrate temperature of 640 °C. The nominal planar growth rate was 2 Å/s. Sample B was grown with the same growth conditions as sample A, but the NWs were Be-doped (p-doped). The temperature of the Be effusion cell was set to yield a nominal Be concentration of 3.5×10^{18} cm⁻³. Sample C was grown as sample B, but on a patterned Si substrate (figure 1(a)). The substrate pattern was prepared by depositing a thin layer of SiO₂ (thickness < 60 nm) on the substrate by plasma-enhanced chemical vapour deposition method. Nano-hole array patterning (hole diameter < 200 nm) was done using standard e-beam lithography. Sample D had Be-doped GaAs NWs grown on Kish graphite, by a two-temperature technique [6].

After growth, the samples were mounted on aluminium scanning electron microscopy (SEM) stubs with conductive silver paint. The NWs were characterized in a FEI DualBeam FIB/SEM system (Helios NanoLab 600), equipped with an Omniprobe tungsten (W) nanomanipulator (AutoProbe 200), hereafter called W-probe, and a gas injection system with C-, Pt- and W-sources. The FIB has a Ga source and was operated at 30 kV. Electrical measurements were performed by connecting a Keithley 2636A source-measurement unit to the sample stage (ground) and W-probe. Before each electrical measurement, the W-probe was cleaned by ion sputtering to remove any contaminants and oxides that might interfere with the measurement.

TEM specimen preparation of as-grown NWs was performed *in-situ* in the same FIB/SEM. The as-grown NWs were extracted using the W-probe as a lift-out finger, before the specimen was thinned to electron transparency by ion sputtering. The final sputtering was done with a 5 keV ion beam. Before extraction, the NW was surrounded by carbon deposited by electron beam assisted deposition (EBAD) at 10 kV, to protect the area of interest against ion beam damage. The TEM characterization was performed on a JEOL 2010F operated at 200kV.

3. Results and discussion

To measure the electrical characteristics of the as-grown NW and substrate, current-voltage (I-V) measurements were performed, using the substrate as one electrode, and the W-probe as the other (figure 1). Different approaches to contact the Be-doped NW samples were studied. Initially, the W-probe was simply put in contact with the NW or the Ga droplet on the top, but such a contact was mechanically unstable due to drift and vibrations, leading to fluctuating and irreproducible results. The measurements also indicated a diode like behaviour of the system. A local deposition of W or Pt on the contact area between the probe and the NW by EBAD (a W or Pt weld) gave sufficient mechanical stability, but the signal was again diode-like. As EBAD deposited materials can have high carbon incorporation (from the precursor gas), ion beam assisted deposition (IBAD) was considered as a way to improve the contact. By attaching the W-probe to the NW tip using a local deposition of W by IBAD, (semi-) linear I-V characteristics could reliably be obtained (see figure 2(a)), which implies ohmic contacts. However, while the contact is ohmic, the system still has a significant resistance (~20-30 MΩ). A control measurement was performed in which intrinsic NWs were used (sample A). Intrinsic GaAs wires have a much higher resistivity (~10 GΩ by two-probe measurements [6]), which should give a very low current. However, these control measurements consistently showed similar behaviour as the doped NWs (compare figure 2(a) and (b)). This indicates that the electrical characteristics do not originate from the NW itself. Most likely, W has been deposited along the length of the NW by deposition overspray, which is caused by the wide tails of the ion-beam profile. Consequently, the deposition method would need to be optimized with regards to spatial precision.

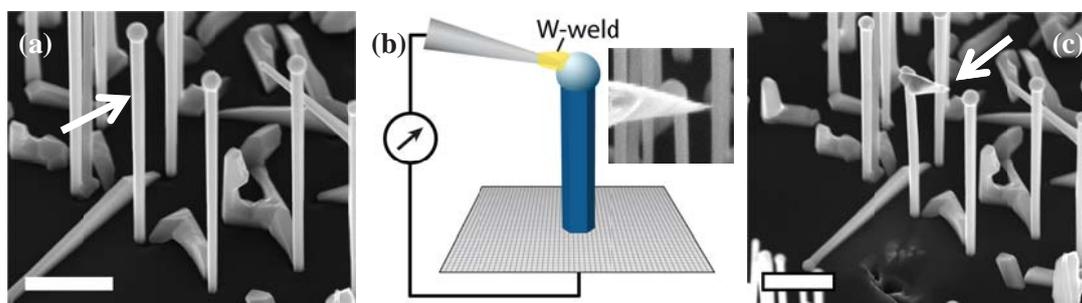


Figure 1. Principle of NW-contacting. (a) NW grown on Si with patterned oxide mask (sample C). NW to be contacted indicated by arrow. (b) Illustration showing how the NW is contacted (inset: SEM of attached probe to NW). (c) NW with part of the W-probe (indicated by arrow) still attached after the probe was cut free by ion sputtering prior to TEM preparation. Scalebar 1 μm in all images.

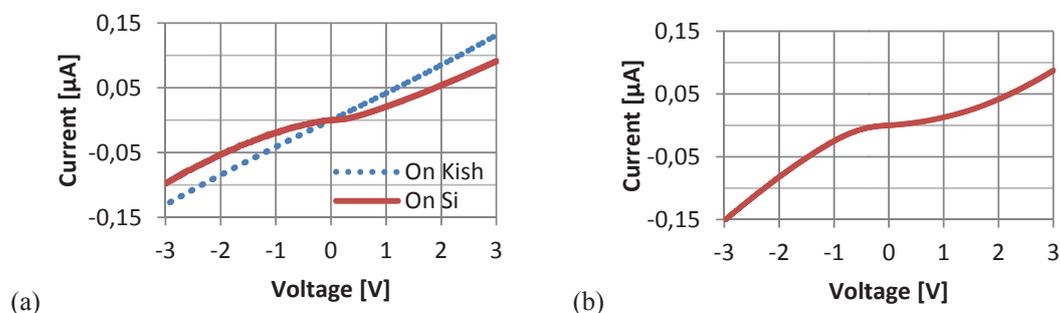


Figure 2. I-V measurements of NWs. (a) p-doped NW on p++ (highly p-doped) Si substrate (solid line) and on Kish graphite (dotted line), samples B and D respectively. (b) Intrinsic NW on p++ Si substrate (sample A). The W-probe was welded to the NW tip by W-IBAD.

Even if it is the case that material is deposited along the exterior of the NWs, the doped NWs are expected to have a low enough resistivity that the electrons would preferentially pass through it, giving a lower resistance than what was measured. As this is not the case, it is likely that the contact to the NW is blocked by the high surface states of the GaAs. To fix this, the contact quality would need to be improved, e.g. by annealing it *in-situ* with a heating sample holder, or the contact would need to be made with a different material. The preliminary conclusion of the present study is that contacting W-probe to GaAs NWs by FIB is limited to Schottky-like contacts using EBAD.

To correlate the SEM and I-V data with the NW crystal structure, TEM specimens were made from the probed NWs within the same set-up. To do this, the tip of the W-probe is cut off by ion sputtering, and then the TEM specimen is prepared, as outlined in the methods section. This allows the detailed study of the NW, the NW-substrate interface, and the contact between probe and NW.

STEM of the same NW (sample C) as depicted by SEM in figure 1, is shown in figure 3. The high angle annular dark field (HAADF) overview image (figure 3(a)) shows that the NW is about 3.6 μm long, and both the interface and the tip with W-probe remnant have been conserved. Diffraction patterns from the centre of the NW (not shown) confirm zinc blende GaAs with growth direction along the [111] crystal direction. Bright field TEM imaging (not shown) shows that the NW is mostly stacking fault free, except near the substrate interface and near the Ga-droplet. In figure 3(b), a HAADF image of the tip of the NW is shown. From energy dispersive x-ray (EDX) measurements it was confirmed that the W-deposition continues down the NW, approximately down 1/3 of its length, as can be seen as bright areas on NW in figure 3(a) and (b). This W-deposition was also visible by SEM (not shown).

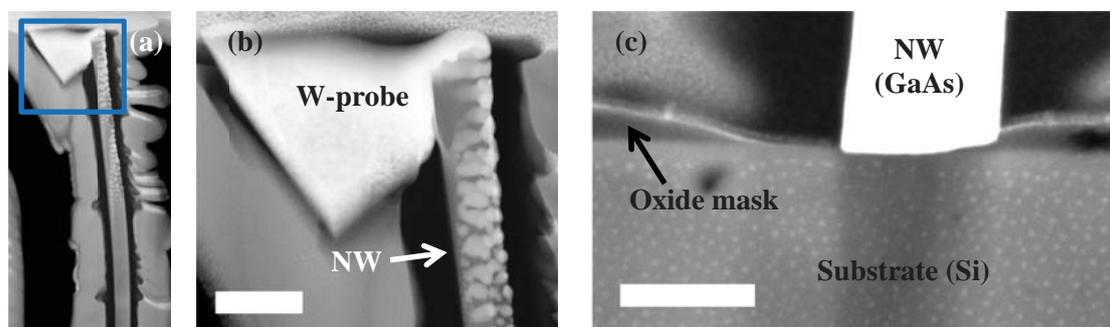


Figure 3. STEM HAADF images of as-grown NW on patterned Si substrate. **(a)** Overview of the entire NW. Boxes indicate the position of figures **(b)** and **(c)**. **(b)** HAADF of NW tip with the free-cut W-probe. **(c)** HAADF showing the interface region of the NW. The profile of the NW and oxide mask can be seen clearly. Scalebar 500 nm in **(a)** and **(b)**, and 100 nm in **(c)**.

Finally, in figure 3(c), a higher magnification HAADF image of the NW-substrate interface is shown. The image shows the position of the NW base in relation to the oxide mask, and it can be seen that the NW is growing over the oxide mask in one direction, along the etch profile. This effectively reduces the NW-substrate contact area, which might be detrimental to its function as a contact. A way to avoid this would be to achieve straighter etch profiles, possibly by reactive ion etching. In figure 3(c), a bright layer can be seen on top of the oxide mask. EDX analysis reveals this to be W, further corroborating the earlier suspicion that W overspray covers the length of the NW.

4. Conclusions

Electrical characterization of individual, as-grown GaAs NWs was performed *in-situ* in a FIB-SEM system by using a fine W-probe. The observed I-V curves depend on the way the W-probe is contacted to the NW. For reproducibility, welding the W-probe to the NW is required. Contacting by EBAD is limited to Schottky-like contacts. W-IBAD gives apparent ohmic contacts; however, a conducting layer might have been deposited along the NW, forming a short circuit. Future work should focus on improving the *in-situ* contacting of the probe to the NW, either by improving the deposition process, changing the deposited material and/or the probe material.

The method combines well with TEM, since a TEM specimen can be made directly of the probed, as-grown NWs. The TEM data acquired shows that the NWs grow epitaxially on the Si substrate, and that the NWs are mostly defect free zinc blende, but with a concentration of stacking faults near the substrate interface and NW tip. The NW base is found to expand over the oxide mask. If a low resistance, ohmic contact can be made, the combination of TEM and probing should allow the correlation of electrical and structural data.

References

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