Interfaces in nanoscale photovoltaics
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In this chapter we focus on the surface properties of the InP nanowires studied in Chapter 4. We show, that we can increase the carrier selectivity of the extended p-type part by HF etching and by applying the high work function interfacial layer MoO$_x$. HF etches the native oxide which is known to cause Fermi level pinning. We show, that by additionally applying MoO$_x$ the charge carrier selectivity of the extended p-part can be increased even further, resulting in an increase of the open-circuit voltage by up to 335 mV, from 500 mV to 835 mV. To electrically passivate the InP surface and protect it against oxidation, we study the influence of surface sulfurization. Preliminary results indicate that sulfurization can indeed increase the open-circuit voltage by removing the native oxide and potentially even passivate the surface against reoxidation. We lay out upcoming experiments and device schemes that have the potential to reach open-circuit voltages above 1V for InP nanowires.
5.1 Introduction

Carrier selective contacts are an essential component of solar cells. So far, nanowire solar cells as traditional silicon solar cells mainly rely on impurity doping to create two carrier selective regions.\[22\] While impurity doping is widely established in the wafer-based semiconductor industry, the control becomes increasingly difficult with decreasing dimensions. The precise doping control for nanowires in the sub-\(\mu\)m range is very challenging and strongly affected by the exact geometry and growth conditions. As we will also show in this Chapter, the doping profile of a nanowire can change unintentionally along the diameter or along the wire length, due to strain-effects of the crystal lattice and different dopant incorporation rates during the growth.\[22, 24, 25\]

However, carrier selectivity of a contact can also be obtained without the addition of impurity atoms into the host crystal (see also Chapter 2 and 3). If a material with a different work function than the main absorber is brought into close proximity, the charge carriers of the two materials will respond by equilibrating their free energies at the interface. As a result, the Fermi level of the charge carriers can change vastly compared to its value in the bulk. If the semiconductor work function is much larger (smaller) than the one of the adjacent material, the semiconductor can change its conductivity into quasi-n\(^-\) (p\(^+\)) type close to the surface. The semiconductor has thereby changed its carrier selectivity, even though no additional dopants have been incorporated. Metals or other high or low work function layers can be used, such as highly doped polycrystalline silicon or transparent conductive oxides.\[26, 28, 89–91, 116, 117\]

Besides providing carrier selectivity by impurity doping or the use of specific external layers, a high degree of surface passivation is crucial. In general, unpassivated interface defects can lead to Fermi level pinning. If those defects occur at the contact interfaces, often induced by direct contact with an adjacent metal, the efficient extraction of charge carriers with a photo-induced potential is inhibited. Importantly, the Fermi level pinning can even counteract the influence of a high or low work function material by fixing the Fermi level at a defect level, irrespective of the internal carrier concentrations.

In this chapter we show doping inhomogeneities of our studied nanowires by variations in the photoluminescence yield. Subsequently, we study the detrimental impact of the native oxide of InP on the carrier selectivity, especially for p-type InP. To that end, we conduct I-V measurements of single nanowire devices before and after HF treatment. In the next step, we apply the emerging carrier selective contact MoO\(_x\) which mitigates negative effects due to inhomogeneous dopant incorporation. Finally, we propose the use of a molecular passivation layer, sandwiched between the unoxidized InP surface and the high work function layer MoO\(_x\) for both decreased surface recombination and increased carrier selectivity.
5.1 Introduction

5.1.1 Doping inhomogeneities

One way to measure the doping concentration indirectly is via the collection of luminescence after initial photon absorption, that is photoluminescence. The absorption depends on the material geometry and the absorption coefficient and leads to the excitation of charge carriers. Subsequently, those carriers decay either radiatively or non-radiatively. Therefore, the photo-excited charge carrier density (determined by the absorption), the doping concentration and non-radiative recombination are essential. If structures are investigated that have the same geometric dimensions and are of the same material quality, photoluminescence can give detailed information about the doping density.\[158, 160\]

Figure 5.1: Variation in photoluminescence for randomly dropcast nanowires. (a) A map of PL intensity variations between different wires, all grown under the nominal same conditions. (b) SEM image of the marked region of Fig.5.1(a). The SEM allows for localization of the broken end part and hence the orientation of the wires (white arrows in (c),(d)). Together with the knowledge of the growth recipe (p-i-n, bottom-center-top), the bright end parts of the wires in (a) can be linked to the highly n\textsuperscript{-}doped region. Notably, for wires with similar diameters ($\Delta d \pm 10$ nm), the end parts as the rest of the wires do not show homogeneous brightness. (e) Strong PL variations along the length and between two different wires. (f) PL spectra of the two marked regions in (e). The shift in wavelengths and intensity due to the different doping concentration is clearly visible (explanation see text). The spectra have been averaged over the marked area from 800 nm - 900 nm to display the maps in (a) and (e).
Fig. 5.1(a) shows variations in photoluminescence (PL) for randomly dropcast nanowires that are indicative of variations in the doping profile. Fig. 5.1(b) shows an SEM image of the marked region of Fig. 5.1(a). The SEM allows for localization of the broken bottom part and hence the orientation of the wires (white arrows in Fig. 5.1(c) and (d)). Together with the knowledge of the doping profile (p-i-n, bottom-center-top), the bright end parts of the wires in Fig. 5.1(a) can be linked to the highly n-type-doped region. Furthermore, we conducted PL measurements before and after the contacting procedure for single nanowire devices. We employed specific contacts for the n-type (Ti/Au) and for the p-type part of the wire (Cr/Zn/Au) which allowed us to confirm our assumption about the orientation of the wire. For nanowires that have been oriented with the p-type (n-type) part towards the electron (hole) contact (connected backwards), high extraction barriers have been observed. In Fig. 4.3 in Chapter 4 we show a PL measurement of a wire after it has been contacted. Also this measurement confirms that the bright part is located next to the electron contact. As can be clearly seen in Fig. 5.1(a), the end parts, as the rest of the wires, do not show homogeneous PL brightness. Additionally, Figure 5.1(e) shows strong PL variations not only between but also along the length of two different wires. The PL variations in Fig. 5.1(a) and (e) are noteworthy. The wires have very similar geometric dimensions ($\Delta d \pm 10$ nm) and hence a very similar absorption. Furthermore, detailed TEM analysis, as in Section 4.3.13 of Chapter 4, shows a high crystal quality, except for a 500 nm short section of the highly doped end part, which exhibits pronounced stacking faults. Therefore, the absorption and non-radiative recombination due to defects can be assumed to be very similar across individual nanowires. Consequently, the PL variations that we observed can be related to a largely varying doping density, even though all nanowires have been grown under the same conditions. Figure 5.1(f) shows the PL spectra of two marked regions in Figure 5.1(e). It is worth noting, that not only the intensity changes, as described above, but also a shift and a change of the PL spectra occurs. The spectrum of the middle part of the wire (green) shows two distinct peaks at around 840 nm (~1.48 eV) and 860 nm (~1.44 eV) which is related to a splitting of the valence band for wurtzite InP nanowires by 40 meV (see also Fig. 4.2 in Chapter 4).[149, 161] The spectrum on the end part of the wire (red) shows only one peak at 840 nm. Considering, that the end parts of the wires are supposedly highly doped, we assume that a blue shift of the spectrum occurs because of band filling of the lower band. However, given that the intensity of the end part is strongly reduced compared to the middle part, the high doping must be accompanied by a strong non-radiative decay, such as Auger recombination. The spectra have been averaged over the marked area from 800 nm - 900 nm to display the maps in 5.1(a) and 5.1(e). The strongly varying doping profiles in Figure 5.1 clearly show the difficulties in controlling the exact doping profile and homogeneity during the growth. Those results underline the importance of finding alternatives to impurity doping and the need for single nanowire studies. Instead of relying on ensemble averages of arrays, which consist of millions of different wires, isolated studies of single nanowires enable better understanding of the intricate mechanisms.
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Figure 5.2: Studied single nanowire device schemes. (a) Shows schematically the single InP nanowire device as studied in Chapter 5 with the addition of a resist window, fabricated by e-beam lithography (layers not to scale). The current flows of the photo-induced charge carriers is shown below the scheme. As can be seen, the electrons not only flow through the n-type region and to the designated electron contact but also into the p-doped region, where they can recombine. (b) Shows the same device as in (a), however after the removal of the SiO$_2$ capping layer and the native oxide. As a result, the Fermi level pinning is reduced and the contact selectivity increases, decreasing the electron flow towards the hole contact. (c) Shows the device after the evaporation of the high work function interfacial layer MoO$_x$ and a capping layer of Au. Due to the high work function, the contact selectivity increases even further, as compared to (b). (d) Optical dark field microscope image of several devices after step (c).
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Fig. 5.2(a) schematically shows the single InP nanowire device as studied in Chapter 4 with the addition of a resist window, fabricated by e-beam lithography. As can be seen, the nanowire consists of a long extended p-doped and a shorter n-doped part. In Chapter 4, we explained the deviation from the intended, symmetric doping profile (p⁺ / p / i / n / n⁻, 1 μm / 3 μm / 4 μm / 3 μm / 1 μm) by the high diffusivity of the p-type dopant zinc, especially at high zinc concentrations and temperatures. Furthermore, we found that the limited contact selectivity causes a difference between the measured and the implied $V_{oc}$ of about 100 mV, even for the best performing devices, grown by SA-MOVPE. Figure 5.2 depicts in a schematic way how decreased contact selectivity in the p-doped part of the nanowire can lead to an electron current into the wrong direction, i.e. away from the electron contact and towards the hole contact. Eventually, those electrons can recombine radiatively or non-radiatively (depicted by stars), decrease the sustained carrier concentration and hence lower the measured open-circuit voltage. For an efficient hole-contact, the quasi-Fermi level of the holes has to be as close to the valence band as possible. However, the native oxide of InP (InO) leads to Fermi level pinning under the conduction band. As a result, for p-type InP the conduction type of the region close to the surface is inverted to quasi-n⁻. The minority carriers of the bulk, that is electrons, can now flow freely towards the surface, where they rapidly recombine. For the nanowire geometry with its large surface-to-volume ratio, the surface inversion can even extend into the bulk. Thereby, it strongly affects the effective doping concentration and hence reduces the contact selectivity of the extended p-doped part, together with the described non-ideal doping concentration.

Fig. 5.2(b) schematically depicts the same device after HF treatment of an extended region on the p-doped part. The SiO₂ capping layer and the native InO have been removed. As a result, the Fermi level pinning under the conduction band is reduced and the contact selectivity increased. Less electrons are flowing towards the p-doped region. Fig. 5.2(c) shows the device after evaporation of 20 nm MoOₓ and a capping layer of Au. Besides the removed Fermi level pinning by the HF treatment, the high work function interfacial layer MoOₓ induces hole accumulation in the p-type part and hence improves the contact selectivity substantially. As a result, electrons flow almost solely towards the electron contact, allowing for a higher carrier concentration and hence open-circuit voltage than in 5.2(a) and 5.2(b.)
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5.2.1 HF treatment

For the following, it is important to note that all the resist windows have been fabricated prior to the first measurements. This way any effects on the measurements by the fabrication can be excluded. Figure 5.3(a) shows the I-V curves of two devices before (dashed) and after (solid) HF treatment. The increase in $V_{oc}$ is clearly visible. Table 5.1 shows the increases in open-circuit voltage for different devices of the two growth batches. For the nanowires of batch 1 (S1B1 - S3B1), the HF treatment improves the open-circuit voltage by about 20 - 60 mV while the wires of batch 2 (S4B2 - S9B2) improve even by 16 - 130 mV. Clearly, the HF treatment and the removal of the native oxide improved the device performance substantially. The two different batches correspond to nanowire arrays grown by the same method, SA-MOPVE, however with different pitches (In 4.3.4, p1-d3 and p2-d1, for B1 and B2 respectively). While the exact influence of the different geometries is still under investigation we speculate that the tendency of sparse nanowire arrays to show higher $V_{oc}$’s might be related to better Zn incorporation from the gas precursor. For dense arrays depletion effects might take place, which reduce the overall concentration of the p-dopant and hence lower the carrier selectivity. Figure 5.3(b) shows the I-V curve for one wire before the HF etching and for subsequent times after the HF etching. As can be seen, after the initial improvement due to the HF etching the native oxide grows back on the exposed part. The $V_{oc}$ and the $I_{sc}$ decrease rapidly to the starting values. Interestingly, the response on the $I_{sc}$ and $FF$ is not consistent across different devices. As can be seen in Fig. 5.3(a), the $I_{sc}$ and $FF$ for devices S7B2 and S4B2 slightly decrease, while for S2B1 in Fig. 5.3(b) a pronounced increase, both in the $I_{sc}$ and $FF$ can be observed.

![Figure 5.3: I-V curves before and after HF treatment.](image)

(a) The effect on the contact selectivity and hence $V_{oc}$ is clearly visible. Dashed and solid lines show the IV curve before and after the HF etching, respectively. With the HF treatment the surface passivation and contact selectivity increase, because of the removal of the native oxide. (b) After the initial increase in performance (yellow), the native oxide slowly grows back (cyan, red, black) and thereby reduces the device performance. The vertical dashed lines are guides for the eye.
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<table>
<thead>
<tr>
<th>Sample</th>
<th>( V_{oc}^{start} )</th>
<th>( V_{oc}^{end} )</th>
<th>( \Delta )</th>
</tr>
</thead>
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<tr>
<td>S1B1</td>
<td>330</td>
<td>350</td>
<td>20</td>
</tr>
<tr>
<td>S2B1</td>
<td>430</td>
<td>485</td>
<td>55</td>
</tr>
<tr>
<td>S3B1</td>
<td>550</td>
<td>606</td>
<td>56</td>
</tr>
<tr>
<td>S4B2</td>
<td>820</td>
<td>836</td>
<td>16</td>
</tr>
<tr>
<td>S5B2</td>
<td>725</td>
<td>790</td>
<td>65</td>
</tr>
<tr>
<td>S6B2</td>
<td>680</td>
<td>780</td>
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</tr>
<tr>
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</tr>
<tr>
<td>S9B2</td>
<td>665</td>
<td>695</td>
<td>30</td>
</tr>
</tbody>
</table>

Table 5.1: HF treatment. All \( V_{oc} \)-values are in mV. As can be seen the \( V_{oc} \) increases for all devices directly after the HF treatment, between 20-60 mV for batch 1 (S1B1-S3B1) and between batch 2 (S4B2-S9B2).

5.2.2 MoO\(_x\) treatment

To prevent the regrowth of the native oxide and increase the contact selectivity, the high work function interfacial layer MoO\(_x\) has been evaporated on freshly HF etched devices. The work function of MoO\(_x\) has been determined to be between 5.7 eV - 6.6 eV, depending on the amount of carbon contamination (decreasing with increasing carbon content).[27] The valence band edge of zincblende InP (\( E_g = 1.34 \) eV, 300K) lies at a value of around 5.8 eV. For wurtzite InP ((\( E_g = 1.43 \) eV, 300K)) the valence band lies around 5.75 eV.[162] Therefore MoO\(_x\) should be able to completely invert the surface. While MoO\(_x\) has a very high work function it does suffer from relatively poor electrical transport and should therefore only be used as an interfacial thin layer.

Fig. 5.4 shows the I-V curves of two representative devices before and after the HF etching and MoO\(_x\) evaporation. The effect on the device performance is clearly visible, the open-circuit voltage increases by 334 mV for M2B1 from 454 mV to 788 mV, due to the improved contact selectivity. Furthermore, even though roughly half of the wire is covered with an opaque MoO\(_x\)/(20 nm)/Au (100 nm) double layer, the short-circuit current of M2B1 improves, as well. The later can be ascribed to a better charge carrier extraction than in the case without surface treatments. In Chapter 4 we observed a localized photocurrent collection close the the electron contact, while the extended p-part did not show any substantial photoactivity.

With the evaporation of MoO\(_x\) charge carriers in the vicinity can be extracted additionally via the MoO\(_x\) reducing the potential recombination in the extended p-type part of the wire. The second wire, M5B2, only improves the \( V_{oc} \) by about 35 mV from 757 mV to 792 mV. Furthermore, the \( I_{sc} \) decreases by a factor of two, which points to the fact that the MoO\(_x\)/Au double layer shadows roughly half of the active part of the wire. Together with the observation that the open-circuit voltage was already relatively high before the treatment this indicates that the wire
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Figure 5.4: I-V curves before and after HF+MoO$_x$ treatment. The effect on the contact selectivity and hence the open-circuit voltage is clearly visible. The selectivity increases substantially with HF etching and MoO$_x$ evaporation. The sample from batch 1 (M2B1) increases by 334 mV, while the sample from batch 2 (M5B2) increases by 35 mV. The vertical dashed lines are guides for the eye and indicate the open-circuit voltage.

did not strongly suffer from limited Zn incorporation and/or axial diffusion. In other words, it is likely that the $I_{sc}$ decreased for M5B2 because the wire performed already at a higher level than wire M2B1. This assumption is supported by the fact that the I-V curves for M2B1 (black lines) show a higher slope around the $I_{sc}$, which indicates insufficient charge carrier extraction. A similar behavior has been observed for M6B2 (see Table 5.2). However, more measurements, especially spatially resolved IQE and PLQY, are needed to understand better the mechanisms involved.

Some of the observed I-V curves, including the ones shown in Fig. 5.4, develop an s-shape after the treatment, even though they improve their $V_{oc}$. We explain this behavior by the electrical transport properties of the MoO$_x$ thin-film. It has been observed before, that the low mobilities in MoO$_x$ lead to the development of s-shaped I-V curves due to a strong increase of the series resistance for film thicknesses exceeding $\sim 10–15$ nm.[27]

Table 5.2 shows the open-circuit voltage enhancement of different devices before and after the HF and MoO$_x$ treatment. As can be seen, the $V_{oc}$ improves substantially by up to 335 mV up to a value of 835 mV, a value very close to the maximum $V_{oc}$ observed for the best performing device in Chapter 4 (850 mV). Interestingly, even though the average $V_{oc}$ increased by about 100 mV compared to Chapter 4, none of the devices exceeded the maximum value of 850 mV. PLQY measurements in Chapter 4 showed that even the maximum $V_{oc}$ of 850 mV is reduced due to
non-selective contacts by 100 mV, from around 950 mV. Therefore, the upper limit for our treatments in this chapter is 950 mV, not 850 mV. Hence we assume that another recombination mechanism inside the extended p-type part of the wire, which cannot be removed by the application of MoO$_x$, is ultimately limiting the contact selectivity. Currently, we are conducting experiments to replace the extended p-type part of the nanowire altogether.

5.2.3 Sulfur treatment

In the previous sections we showed how the native oxide reduces the open-circuit voltage by Fermi level pinning under the conduction band at the surface (schematically shown in Fig. 5.5(a)). However, we have also shown that the oxide grows back rather rapidly; a substantial reduction of the open-circuit voltage of 50 mV can already be observed after 30 min.

For the MoO$_x$ treatment we observe an increase in the open-circuit voltage of up to 334 mV (Fig. 5.5(b)). Even though the MoO$_x$ has been evaporated on freshly HF etched samples, it is likely that the native oxide grew back partially, especially because of the oxygen-rich MoO$_x$ film. We have not observed a pronounced degradation for the samples with the MoO$_x$ layer. Therefore we speculate that if the MoO$_x$ indeed leads to regrowth a thin native oxide layer, this growth must happen during or shortly after the evaporation and capping of the MoO$_x$ layer. Furthermore, because the samples are laying on a substrate, the HF is unlikely to etch the native oxide homogeneously and the MoO$_x$ capping does not extend around the whole wire.

To prevent the reoxidation and enhance the impact of the MoO$_x$ coating, we propose a contact scheme that makes use of an additional thin tunnel and passivation layer, very similar to the one employed for MIS solar cells. Recently a report appeared that utilized a similar contact scheme to improve the performance of AZO - InP nanopillar heterojunction solar cells.[163] In the following we will show preliminary results about the impact of surface sulfurization on our nanowires. However, in general many other possible passivation layers are available that could
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be studied, also those that can potentially be grown in the vapor phase, directly after the nanowire growth.

Fig. 5.6 shows preliminary results of the effect of surface sulfurization on a nanowire. The device (S2B1) is the same nanowire that was studied for the HF treatment in Fig. 5.3. After the native oxide grew back, the extended p-type part of the wire has been exposed to an ammonium sulfide ((NH₄)₂S) solution (for details see Methods section). The solution is known to etch the native oxide of InP and to passivate the surface. However, as can be seen in Fig. 5.6, the $V_{oc}$ increases to about 550 mV, substantially above the value of 485 mV that has been observed after the HF treatment (blue curve). We speculate that the sulfurized surface protects the wire against the rapid reoxidation during the rinsing and nitrogen blow-drying steps and therefore shows superior performance compared to the HF treatment. However, even though the open-circuit voltage increases substantially, the short-circuit current stays approximately at the value before the HF treatment. This is in contrast to the HF treatment, which not only increases the open-circuit voltage but also the short-circuit current. From experiments on InP bulk wafers (zinc blende)
and InP nanowires, it is known that the surface properties after sulfurization vary largely, depending on the exact condition (concentration, time, temperature, additional solvents).\[164–168\] Therefore, further experiments are needed to optimize those conditions also with respect to reducing non-radiative recombination due to surface defects and to increase the number of total measured devices. We found that for resist windows that were exposing not only the nanowire, but also partly the metal contacts, all of the HF and (NH$_4$)$_2$S treatments strongly degraded the metal contacts, which reduced the yield of the first run substantially.

Besides the optimization of the initial sulfur treatment, we are currently also exploring possibilities to stabilize the sulfurized surface against degradation by employing SiO$_2$ and other capping layers. It has been shown that sulfur atoms only form relatively weak covalent bonds and can easily degrade in ambient conditions over extended periods.\[169\] Therefore, a capping layer is of utmost importance to improve the stability of the sulfur layer.

Finally, we propose to enhance the effect of the MoO$_x$ by realizing a coating that wraps around the whole p-type part of the wire. This way, the MoO$_x$ can effectively invert the whole nanowire interior and create a highly selective contact (Fig.5.5(c)). While this wrap-around geometry is challenging to realize for horizontally aligned single nanowires, because of limited access to the nanowire-substrate interface, it can readily be achieved for nanowire arrays. In fact, as we will show in the following application chapter by proposing a novel nanowire fabrication process, the nanowire arrays can be embedded into a polymer or glass matrix which allows the selective sulfurization and application of MoO$_x$ to the nanowire tips.
5.3 Conclusion

In this chapter we have shown how the extended p-type part of the InP nanowires studied in Chapter 4 can be improved with respect to its carrier selectivity. We show how the removal of the native oxide can improve the open-circuit voltage by up to 100 mV. The addition of the high work function interfacial layer MoO$_x$ improves the open-circuit voltage up to 335 mV. By that we show that the reduced carrier selectivity is likely due to insufficient doping concentration of the extended p-type part of the wire, either due to limited dopant incorporation or post growth diffusion. Finally, we show preliminary results on the surface sulfurization of the extended p-part of the wire. We can show that the sulfur treatment improves the carrier selectivity of our studied device, but degrades the short-circuit current by a factor of roughly 1.5. We discuss ongoing experiments that focus on the optimization of the sulfur treatment, including encapsulation and direct application to the InP and MoO$_x$ interface. This way the growth and the negative impact of the native oxide can be prevented and at the same time the properties of the high work function layer MoO$_x$ fully exploited, especially when the extrinsic layers are wrapped around the tip of the nanowire.

The results presented here give new insights into the performance of InP nanowires and pave the way towards open-circuit-voltages $> 1\text{V}$. Eventually, we envision dopant free nanowire solar cells, unaffected by decreased mobilities, increased non-radiative recombination and inhomogeneous and insufficient dopant profiles.

As we will show in the following chapter, the removal of the material specific impurity doping extends its impact beyond improved device performance. By employing novel carrier selective contacts that rely on extrinsic surface layers e.g. MoO$_x$, their use is not limited to InP but can be extended to other materials, as well. This versatility does not only lead to a simplification but also enhances the flexibility of our proposed fabrication process.

5.4 Supplemental information

5.4.1 Fabrication and measurements

The samples were fabricated, contacted and measured as described in the supplemental information of Chapter 4, 4.3.1, 4.3.2 and 4.3.3, respectively. For the fabrication of the resist windows, a third electron beam lithography step was added. For the HF treatment, the exposed nanowire parts were etched in buffered HF (1:7, HF (49%): NH$_4$F (40%)) for 10 s to remove the protective SiO$_2$ shell ($\sim$50 nm) and the native oxide of the InP under the contact. For the MoO$_x$ treatment, first the HF etching has been repeated after which MoO$_x$ has been thermally evaporated at a rate of around 0.3-1 Å/s and a pressure of $2 \times 10^{-6}$ mbar. The target thickness was 15 nm, however, after a short but very high spike in the evaporation rate, the final
thickness was \( \sim 20 \text{ nm} \). For the sulfurization, the samples were immersed for 5 min in 5\% (NH\(_4\))\(_2\)S solution in water.

The photoluminescence spectra were obtained with a Witec confocal microscope (Alpha 300 SR). The samples were illuminated with a 532 nm laser, and the light was focused and collected via a 100X NA0.9 objective. A spectrometer with a 150 g/mm (BLZ= 500 nm) grating (center wavelength 760 nm) and a cooled CCD camera have been used. The integration time was 0.09 s and the scan speed 6.83 s/Line.