Embedded sacrificial AlAs segments in GaAs nanowires for substrate reuse

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Abstract
We report on the use of a sacrificial AlAs segment to enable substrate reuse for nanowire synthesis. A silicon nitride template was deposited on a p-type GaAs substrate. Then a pattern was transferred to the substrate by nanoimprint lithography and reactive ion etching. Thermal evaporation was used to define Au seed particles. Metalorganic vapour phase epitaxy was used to grow AlAs–GaAs NWs in the vapour–liquid–solid growth mode. The yield of synthesised nanowires, compared to the number expected from the patterned template, was more than 80%. After growth, the nanowires were embedded in a polymer and mechanically removed from the parent substrate. The parent substrate was then immersed in an HCl:H2O (1:1) mixture to dissolve the remaining stub of the sacrificial AlAs segment. The pattern fidelity was preserved after peeling off the nanowires and cleaning, and the semiconductor surface was flat and ready for reuse. Au seed particles were then deposited on the substrate by use of pulse electrodeposition, which was selective to the openings in the growth template, and then nanowires were regrown. The yield of regrowth was less optimal compared to the first growth but the pattern was preserved. Our results show a promising approach to reduce the final cost of III–V nanowire based solar cells.

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(Some figures may appear in colour only in the online journal)

1. Introduction

III–V semiconductor nanowires (NWs) have important applications in different fields such as electronics and photonics [1–7]. Nanowires recently received a lot of attention for their promise in photovoltaics (PV) [8–15]. NWs have the potential to exceed the current world record for solar cell efficiency due to geometrically enhanced light absorption and the possibility of combining materials with different band gaps optimised to the solar spectrum due to reduced lattice matching requirements in these low dimensional structures. Efficiencies as high as 17.8% and 15.3% have been achieved for NW-based InP and GaAs solar cells, respectively [11, 12]. Nanowire solar cells use less material than their thin film counterparts because of enhanced light absorption [16] which provides a significant cost reduction. However, the fabrication cost remains high because of the expensive substrate. One possibility to reduce substrate costs is to use Si as a substrate, and the successful growth of III–V NWs on (111)Si has been reported [17, 18]. However, growing high quality material on Si is limited to a relatively small parameter space. In addition, in-diffusion of dopants from the III–V materials into Si and Si in-diffusion into the III–V materials poses challenges to control the electrostatic potential profile across the junction [19].
One way to reduce the cost related to material synthesis, and thus the final device cost, would be to transfer the as-grown NWs to another substrate or membrane for device fabrication to enable reuse of the substrate for growth. Epitaxial lift-off (ELO) is a well-known method in the GaAs thin film industry for substrate reuse [20, 21]. In ELO a sacrificial layer of, e.g., AlAs, AlGaAs or GaNp, is grown between the parent substrate and actual device. The sacrificial layer is subsequently dissolved in a wet etchant such as hydrofluoric acid (HF) or hydrochloric acid (HCl), whereafter the device layer is separated from the parent substrate [22–25]. It is important that the resulting substrate surface after processing is ready for epitaxy, free from any residues, and has a low surface roughness.

To date, peeling off an array of NWs is the common way of transferring them from the parent substrate [26–28]. A growth template can be used in combination with selective area electrodeposition and NW transfer to enable multiple substrate reuse and impede costs of iterative patterning steps for seed particle definition. Seedless selective area (SA) epitaxy [29] has been used to grow InP NWs on (111)A InP substrates and enable substrate reuse. The substrate was flattened by wet chemical etching after peeling, due to the fact that the etch rate of (111)A InP in HCl is relatively slow as compared to other facets [30].

Particle-assisted vapour–liquid–solid (VLS) [31] NW growth offers advantages with respect to SA by advanced bottom-up synthesis using complex heterostructure design [32–34]. Substrate reuse of VLS-grown NWs is however challenging with respect to maintaining stability of the growth template during post transfer substrate treatments. Nanowires are usually grown on a native substrate and using a wet etch process to remove the stubs could etch particular facets of the substrate. This could result in formation of surface pits which complicates subsequent nucleation and synthesis. It has been shown that the template lost its initial pattern after four cycles of substrate reuse for VLS growth of Si microwires, because of post transfer wet etching of the substrate [26].

In this study we investigate the use of a sacrificial AlAs NW segment to flatten the growth surface and reduce post transfer defects after transferring GaAs NWs from a (111)B native substrate. The GaAs substrate was first covered with a silicon nitride (SiN$_x$) growth template. A hexagonal pattern of holes was defined in this template using nanoimprint lithography (NIL) and reactive ion etching. Gold (Au) seed particles were deposited using metal vaporization and lift-off. The GaAs/AlAs/GaAs NWs were then grown using MOVPE in the VLS growth mode. The NWs were subsequently embedded in polydimethylsiloxane (PDMS) and transferred from the substrate. In order to dissolve any remaining AlAs, the substrate was immersed in HCl:H$_2$O (1:1) resulting in a clean, flat surface suitable for reuse. Gold was subsequently deposited selectively in the growth template openings by use of pulse electrodeposition (PED) [35, 36]. GaAs and AlAs–GaAs NWs were then grown and the pattern fidelity was evaluated after reuse. The pattern was preserved and the grown NWs had a high yield indicating that this method is promising for enabling substrate reuse.

![Figure 1. Schematic of the substrate reuse process showing the different steps. (a) Sample after NIL patterning. Blue, red and orange colours represent SiN$_x$, LOR 0.7A and TU7-120, respectively. (b) Sample after Au deposition and lift-off. (c) Grown NW heterostructures with GaAs, AlAs and Au shown in green, dark red and yellow, respectively. (d) An oblique profile of the AlAs stubs after peel-off is illustrated. (e) Sample after etching the remaining AlAs stubs and (f) sample after gold electrodeposition, ready for regrowth. The drawings are not in scale with the real sizes.](image)

2. Experimental methods

2.1. Substrate preparation

To deposit the growth template, a p$^{++}$ (111)B GaAs substrate was coated with 105 nm of SiN$_x$ using plasma-enhanced chemical vapour deposition (PECVD) (all experimental details are available in the supplementary information, SI, available online at stacks.iop.org/NANO/31/204002/mmedia). This template not only impedes parasitic growth on the GaAs substrate surface, but is important for substrate reuse because the pattern already defined in the growth template can be used for selective placement of Au particles using electrodeposition. The lithography steps for patterning need only to be performed once, and the growth template can be used repeatedly. The substrate was covered with resists (see SI) and patterned using nanoimprint lithography (NIL). Hexagonally arranged 200 nm openings with 500 nm pitch were formed in the SiN$_x$ template using reactive ion etching (RIE). Figure 1(a) shows a schematic of the sample after NIL patterning. The substrate was then placed in the thermal evaporator and 65 nm Au was deposited. Thermal evaporation was used to eliminate any speculation on the effect of seed definition method on the growth results. Based on our previously reported comparison of NWs grown from evaporated and electrodeposited Au seed particles [36], we strongly believe that the seed definition method does not have any significant effect on the growth result. The excess Au and polymer were subsequently removed in a lift-off process using hot Remover 1165. Figure 1(b) shows a schematic of the sample at this stage. The substrate was then diced to 1 × 1 cm$^2$ pieces for subsequent growth.

2.2. Nanowire growth

The NWs were grown in a low pressure (100 mbar) MOVPE system (Epiquip) with a total flow of 61 min$^{-1}$. Trimethylgallium (TMGa), arsine (AsH$_3$), trimethylaluminum
(TMAl) and hydrogen bromide (HBr) were used as precursors while H₂ was used as carrier gas. The sample was first annealed at 450 °C for 10 min and subsequently a GaAs nucleation step of 40 s with precursor molar fractions $\chi_{\text{TMGa}} = 2.53 \times 10^{-5}$ and $\chi_{\text{AsH}_3} = 1.67 \times 10^{-3}$ was initiated. AlAs was then grown with precursor molar fractions of $\chi_{\text{TMAl}} = 3.69 \times 10^{-6}$ and $\chi_{\text{AsH}_3} = 1.67 \times 10^{-3}$ and HBr molar fractions of $\chi_{\text{HBr}} = 4.1 \times 10^{-6}$. HBr was used to hinder tapering [37]. After 60 min of AlAs growth, GaAs NW growth was initiated using precursor molar fractions of $\chi_{\text{TMGa}} = 1.77 \times 10^{-6}$ and $\chi_{\text{AsH}_3} = 1.67 \times 10^{-3}$ and growth proceeded for 15 min. Figure 1(c) depicts the grown NW heterostructures.

2.3. Peel-off and post transfer treatments

For peel-off, the NWs were embedded in PDMS and removed from the substrate, figure 1(d). In order to clean the sample from any remaining residues of PDMS, the parent substrate was cleaned in Dynasolve 220 for 2 h. A final 90 min ozone cleaning step was performed before etching. To remove any remaining stubs of AlAs, the substrate was immersed in a mixture of HCl: H₂O (1:1) for 5 h, followed by a rinse step in flowing water for 3 min, and drying with flowing N₂. An illustration of the sample at this stage is shown in figure 1(e).

2.4. Gold electrodeposition

In order to selectively deposit the Au seed particles, an electrodeposition system from Yamamoto-MS was filled with 160 ml of 24 K Pure gold solution [38] and heated to 35 °C. The substrate was then placed on a 1 x 1 cm² custom made cathode and immersed in the Au solution together with a platinized titanium anode. Pulse electrodeposition was implemented by applying a 20 Hz pulse with a peak current density of 5 mA cm⁻², a 20% duty cycle and a duration of 3800 cycles. The sample was illuminated by a series of LEDs, emitting at 405 nm, embedded in a paddle agitator moving back and forth in front of the cathode. The illumination is required to generate electron-hole pairs during electrodeposition on p-type substrates [39]. The sample was subsequently rinsed in cold and then hot water and cleaned for 90 min using ozone cleaning to make sure that the surface is free from any organic residues of the Au solution. The desired sample profile at this step is illustrated in figure 1(f).

2.5. Regrowth

The sample was subsequently moved to the MOVPE reactor to grow NWs on the processed substrate. The molar fraction of the precursors during growth was the same as for the first growth run.

3. Results and discussions

A common way to transfer NWs from substrates is to embed the NWs in a polymer such as PDMS and mechanically peel them off. We started by peeling the InP NWs grown on (111)B InP substrates using the VLS method. Nanowires were embedded in the polymer and removed from the parent substrate. As can be seen in figure 2(a) there are oblique stubs remaining at the bases of the NWs after peel-off. These stubs remain regardless of the NW material, figure 2(b).

Deposition of Au on this substrate is challenging because if the stubs are not removed, electrodeposition will occur on the sidewalls of the stub in addition to on the top of it, figure 3(a). As the top of the stubs stick out of the growth template the seed particles could move during annealing and growth, which degrades the yield of the regrowth, figure 3(b). The remaining stubs need to be removed before substrate reuse. Because the etch rate of (111)B InP in an HCl:H₂O mixture is relatively high, a precisely controlled etching is hard to achieve. The oblique profile adds another degree of complexity: the shorter part of the stub will be etched down to the substrate while the other side still remains. Over-etching is required to remove the stubs, which leads to etching of the substrate. In this case, Au will be deposited on exposed conductive substrate material underneath the template during electrodeposition, figure 3(c), which affects the growth condition and may cause delamination of the SiNₓ template and failed growth, figure 3(d). In both scenarios a stub remaining at the bottom of the NWs presents challenges for deposition of the Au particles on the reused substrate and regrowth.
We conclude that the use of a sacrificial layer in the NWs could be beneficial in order to avoid problems related to the oblique stubs. The remaining stub could then be selectively etched with minimal effects on the substrate and growth template. Inspired by ELO, AlAs was chosen as the sacrificial layer in combination with lattice-matched GaAs NWs.

Growth of AlAs NWs on GaAs substrates using molecular beam epitaxy (MBE) has previously been reported. Li et al [40] obtained an average growth rate of 40 nm min⁻¹ at temperatures between 650 °C and 660 °C. To remove any oxide from the Au-substrate interface, it is quite common to conduct an annealing step at high temperature under Group-V overpressure. However, there was a poor adhesion between the deposited SiNx template and the GaAs substrate, which led to template delamination during the high temperature annealing, so we avoided this step and the Au-GaAs substrate was annealed at 450 °C instead. This limits the growth temperature, an issue for AlAs in particular which is preferably grown at temperatures above 480 °C [41].

Initial results showed that the SiNx template had a negative effect on the growth yield of AlAs. Better results were achieved after the substrate had been etched in diluted HF: in water (H₂O) (1:100) for 60 s. We speculate that if the melted seed particle touches the template it may affect the wetting angle, and hence synthesis. The HF etching widened the template and reduced this effect. Hydroflouric acid could also change the surface properties of the template and affect the migration of growth species on the template surface.

It was not possible to directly nucleate AlAs on the GaAs substrate at 450 °C. This is most probably because the Al–Au system at this temperature is in the solid phase [42] wherefore the growth is expected to proceed via the vapour–solid–solid (VSS) growth mechanism [43], not VLS. In order to improve the nucleation of AlAs, a short stub of GaAs was grown at 450 °C, figure 4(a). This ensures that the particle is in the liquid state when the AlAs growth starts. Interestingly, on the same substrate we found particles with different wetting angles, figure 4(a), which can significantly affect the growth.

It is obvious that the two stubs have different lengths. It can be seen that the template is delaminated at the edges of the pattern, an observation that will be further addressed in the regrowth discussion below. This nucleation stub is also the reason why a template thickness of 105 nm is used; this stub height in addition to the thickness of the electrodeposited Au seeds will still be shorter than the template thickness.

The growth rate of AlAs was very low, around 10 nm min⁻¹, especially at TMAI molar fractions below $\chi_{\text{TMAI}} = 3.69 \times 10^{-6}$, for which no growth of AlAs was observed under scanning electron microscope (SEM) inspection. The low growth rate could indicate VSS growth [44]. This would imply that during switching from Ga to Al the Au particle was initially in a liquid state but solidified as an effect of increased Al content. The lateral growth rate of AlAs was as high as half of the axial growth rate, so a large tapering was observed, figure 4(b). This observation could also indicate VSS growth. If the supply of material is higher than the axial growth rate, which for VSS could be limited by the diffusion through the solid particle, the material should either desorb or be deposited on the sides of the NWs. At this low growth temperature, desorption might not be significant so a large fraction of the supplied material contributes to sidewall growth (tapering).

To prevent shell growth during the growth of the upper GaAs section, a HBr molar fraction of $\chi_{\text{HBr}} = 4.1 \times 10^{-6}$ was used. Hydrogen bromide was observed to improve the growth yield of AlAs. We found that the growth rate is very sensitive to the HBr molar fraction used: increasing the HBr molar fraction from $\chi_{\text{HBr}} = 4.1 \times 10^{-6}$ to $\chi_{\text{HBr}} = 5.43 \times 10^{-6}$ reduced the axial growth rate by a factor of 2. At some positions on the sample the growth yield of AlAs was not high when compared to the pattern. The effect of the V/III ratio was studied, and high V/III ratios seem to improve the yield. However, at high V/III ratios the growth rate drops to almost zero. After 60 min of growth, a 400 nm AlAs segment was observed after synthesis, figure 4(c).

The upper GaAs section of the NWs had a length of around 2.5 μm, figure 4(d). It has been shown that growth of GaAs on AlAs in a MOVPE system is challenging [41]. However, in our study the growth yield simply followed the yield of the AlAs segments; a high AlAs yield led to a high GaAs yield. We speculate that this is related to different effective V/III ratios when using ordered array patterning as compared to random seed definition due to aerosol particle deposition [45]. Also, the SiNx template changes the pyrolysis of the growth precursors and could improve the growth by localising the Au particles [46].

Figure 5(a) shows the state of the sample after peel-off. The etch rate of GaAs in HCl is very low so the surface of the GaAs NW stubs remains stable and becomes suitable for subsequent reuse [25]. As is evident in figure 5(b), the template pattern is preserved after etching, which is a promising
result compared to a previous study in which an SiO$_2$ template lost its initial pattern during post transfer treatments [26]. A benefit of using AlAs as a sacrificial layer is that the transferred NWs will have a flat surface (see SI) which is crucial for further device fabrication.

Although the height of the nucleation stub is shorter than the template thickness, its diameter is also smaller than the diameter of the holes so Au can deposit on unprotected sidewalls. Figure 6(a) shows an SEM image of the sample after Au deposition. The deposited Au seed is not uniform. Gold is deposited on unprotected facets of the nucleation stub and not on the top facet of the stub only. Another important observation is that Au creeps under the template which leads to an underplating effect. It is well-known that cyanide-based solutions can dissolve the organic polymer masks and cause underplating [47]. However, it is less likely that the inorganic SiN$_x$ template is etched away during electrodeposition, especially when no measurable change on the template surface was observed. Note that we did not observe any underplating when electrodeposition was used to define the seed particles directly on GaAs substrate, figure 6(b). The main reason for this underplating is delamination of the template during the first growth as depicted in figure 4(a). Delamination is a result of thermal stress caused by different thermal expansion coefficients of the template and substrate.

In order to simplify synthesis and evaluate the success of regrowth without issues relating to heterostructure formation, pure GaAs NWs, rather than AlAs–GaAs NWs, were first grown on the electrodeposited reused substrate. As depicted in figures 7(a) and (b), the pattern is preserved but parasitic growth on the sample surface occurs. The Au deposited on the sidewalls of the nucleation stub caused this parasitic growth. Template delamination was also observed on part of the substrate. Gold underplating due to poor adhesion between the SiN$_x$ template and the substrate makes delamination more severe. In order to evaluate the possibility of continued substrate reuse, AlAs–GaAs NWs were grown as shown in figures 7(c) and (d). The length and diameter of the regrown NWs were the same as in the first growth run. The pattern is preserved but parasitic growth is observed on the surface. In this case, the parasitic growth results from both Au deposited on the sidewalls of the nucleation stubs and inherent difficulties of growing heterostructure GaAs/AlAs/GaAs NWs.

It is important to preserve the quality of the growth template to enable substrate reuse. Higher quality SiN$_x$ deposited with low stress and with a better adhesion to the substrate could significantly improve the results. It is possible to grow AlAs in an Al-rich particle provided that the template can withstand higher growth temperatures without delamination [40, 41]. At present, we need to start the growth with a short GaAs stub which adds to the height of the stub from previous growth runs. After a few reuse cycles, the GaAs nucleation stub will be long enough to stick out of the template after NW transfer. This makes it challenging to deposit Au seed particles since electrodeposition will occur on the sidewalls of the stub sticking out of the growth template.

4. Conclusions

In conclusion, AlAs was used as a sacrificial layer in a GaAs-based NW system to preserve pattern fidelity after post transfer treatment of the parent substrate for substrate reuse. A SiN$_x$ growth template was patterned using NIL and RIE on a GaAs substrate. Gold seed particles were defined using thermal evaporation and GaAs–AlAs–GaAs heterostructure NWs with high yield were grown in a low pressure MOVPE system. We found that a short GaAs stub for nucleation improved heterostructure synthesis, as opposed to direct
growth of AlAs on the GaAs substrate. As-grown NWs were embedded in PDMS and removed from the parent substrate. In order to dissolve the remaining AlAs segment, the parent substrate was immersed in HCl: H₂O (1:1) leaving the substrate ready for subsequent selective Au deposition in the holes of the preserved growth template. GaAs and AlAs–GaAs NWs were then grown using the reused patterned substrate. Results showed that the pattern fidelity was preserved. However, parasitic growth due to Au deposition on the sidewalls of GaAs nucleation stubs was also observed. Because of poor adhesion between template and substrate, template delamination was observed on parts of the samples. As the success of AlAs synthesis is expected to be higher at higher growth temperature, a low stress template with better adhesion to the substrate is the key to improving this. We believe this approach can lead to multiple substrate reuse and diminish substrate costs for the realisation of economically viable flexible NW based devices, such as NW solar cells and light emitting diodes.

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