Facet-selective group-III incorporation in InGaAs template assisted selective epitaxy

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Abstract

InGaAs is a potential candidate for Si replacement in upcoming advanced technological nodes because of its excellent electron transport properties and relatively low interface defect density in dielectric gate stacks. Therefore, integrating InGaAs devices with the established Si platforms is highly important. Using template-assisted selective epitaxy (TASE), InGaAs nanowires can be monolithically integrated with high crystal quality, although the mechanisms of group III incorporation in this ternary material have not been thoroughly investigated. Here we present a detailed study of the compositional variations of InGaAs nanostructures epitaxially grown on Si(111) and Silicon-on-insulator substrates by TASE. We present a combination of XRD data and detailed EELS maps and find that the final Ga/In chemical composition depends strongly on both growth parameters and the growth facet type, leading to complex compositional substructures throughout the crystals. We can further conclude that the composition is governed by the facet-dependent chemical reaction rates at low temperature and low V/III ratio, while at higher temperature and V/III ratio, the incorporation is transport limited. In this case we see indications that the transport is a competition between Knudsen flow and surface diffusion.

Supplementary material for this article is available online

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

1. Introduction

The ternary semiconducting alloy indium gallium arsenide (InGaAs) has gained significant technological importance in recent years. In particular, it is an excellent materials choice for photo-detection in the mid-infrared (up to 1.6 μm wavelength for In0.5Ga0.5As) with much lower dark-currents than Ge devices [1]. The composition In0.53Ga0.47As has gained...
particular interest for electronic device applications [2] because it can be grown lattice-matched to semi-insulating InP substrates, has a high electron mobility and low dark-currents in optoelectronic devices [3]. There is a tremendous interest in integrating InGaAs photodetectors, lasers and field-effect transistors with Si technology [4, 5], and many approaches for integration of InGaAs on Si have been reported [6, 7]. The major challenges for III–V integration on Si are the often-large crystal lattice-mismatch, the dissimilarity in crystal structure and the thermal expansion mismatch, which lead to the formation of detrimental crystal defects such as threading dislocations and antiphase boundary defects. Recently, high-quality InGaAs nanowires and InGaAs fin field-effect transistors on oxide-covered Si substrates were achieved through a technique called template-assisted selective epitaxy (TASE) [8, 9]. The key concept of TASE is that heteroepitaxy on the Si substrate starts from a single InGaAs nucleus for each individual device. This is achieved by limiting the exposed Si area to less than 100 × 100 nm², much smaller than the migration length of the growth species on the Si surface. By the utilization of a single nucleus per device, antiphase boundary defects are intrinsically avoided, and the small heterointerface area also prevents dislocation threading [10]. The full InGaAs crystal is formed by expanding the single nucleus into the desired crystal shape by continuing the epitaxy in a confined space within a pre-defined silicon oxide template. Although InGaAs TASE was recently demonstrated [8], detailed investigation into the chemical composition of the InGaAs crystals is still lacking. This information is crucial for the potential industrial adoption of this material. Here we present an investigation by x-ray diffraction (XRD) and transmission electron microscopy (TEM) of the chemical composition of InGaAs nanowires and platelets grown by TASE. We observe compositional variations that can be explained by a facet-type dependent incorporation of Ga and In at low temperature, and transport limited growth at higher temperature and V/III ratio.

2. Methods

Both vertical and horizontal InGaAs nanowires as well as horizontal platelets were investigated in this work. For XRD characterization, large areas of randomly arranged vertical templates were produced by the dispersion of 100 nm diameter polystyrene nanoparticles in solution onto a Si(111) wafer covered with 150 nm SiO₂. The colloids were used as a mask for subsequent pattern transfer into the underlying SiO₂ layer by reactive ion etching (RIE) and furthermore into the crystalline Si by HBr-based inductively coupled plasma RIE. In this way samples with sufficient InGaAs nanowire material for characterization in high-resolution XRD using a lab x-ray source were produced. Horizontal templates were fabricated on silicon-on-insulator wafers for the TEM studies. The processes for fabricating both vertical and horizontal oxide templates are detailed in previous works [8, 10].

InGaAs growth by TASE was conducted in a Veeco P150 metalorganic vapor phase epitaxy (MOVPE) system operated at 60 Torr with H₂ carrier gas. Trimethylgallium (TMGa), trimethylindium (TMIn) and tertiarybutylarsine (TBAs) were used as precursors for the InGaAs growth. Prior to loading into the MOVPE system, the substrates were briefly etched in diluted HF (1:20) to remove the native oxide on the Si seed surface within the templates. After loading, the reactor was heated to above 700 °C under flow of TBAs before ramping down to the growth temperature of 520 °C or 580 °C. Growth commenced upon the introduction of TMGa and TMIn at total group III molar flow of 2.8 μmol min⁻¹. This flow was kept constant throughout the experiments, however the vapor composition x_{vap} = [TMIn]/([TMIn] + [TMGa]) was varied throughout the composition range in the experiments on vertical nanowires before settling on x_{vap} = 0.51 for the horizontal nanowires. For the platelets the growth temperature was 560 °C, x_{vap} = 0.50 and total group III molar flow and V/III ratio 9 μmol min⁻¹ and 20, respectively.

Planar reference samples of InGaAs epitaxial thin films were grown on InP substrates with (100), (110) or (111)B orientation in a Aixtron 200/4 MOVPE system. TMGa, TMIn and AsH₃ were used as precursors and the growth temperature was 600 °C with total group III flow and V/III ratio of 70 μmol min⁻¹ and 30, respectively.

High resolution XRD (HRXRD) measurements were carried out using a Bruker D8 Discover diffractometer equipped with a rotating Cu anode. To enhance the measurement resolution, a Ge(022) monochromator and an analyzer have been installed on the primary and secondary side respectively.

TEM nanowire samples were fabricated using focused ion beam (FIB) preparation. Cross-section TEM samples were made in directions both parallel and perpendicular to the nanowire growth direction. For in-plane TEM, the substrate was etched away underneath the InGaAs material to allow for transmission imaging from the top. Though FIB sample preparation will incorporate Ga into the samples from the Ga ion beam, care was taken to reduce Ga contamination by lowering the ion energy and milling at high angle (~89° to sample surface) during final thinning.

Electron energy loss spectroscopy (EELS) data were recorded on a Thermo Fisher Scientific C₆ probe-corrected Titan TEM fitted with a Gatan Quantum energy filter. The spectrometer was typically set to have a 0.5 eV/channel dispersion. Running in Dual-EELS mode, the first low energy-range spectrum was recorded in the range from 80 to 1080 eV to capture the In M₄₅₆ (onset at 443 eV) and the O K (532 eV) ionization edges with an exposure time of 0.02 s per pixel and the second high energy-range spectrum was recorded in the range 800 to 1800 eV to capture the Ga L₂₃ (1115 eV) and As L₂₃ (1323 eV) edges with an exposure time of 0.2 s per pixel. Quantification was carried out using conventional power-law background fitting and integrating the counts within a 100 eV window with its lower energy offset to 20 eV the edge threshold. More details of how the quantification of the EELS data was done and is available in the
Energy-dispersive x-ray (EDX) mapping was carried out using a Thermo Fisher Scientific C$_s$ probe-corrected Titan Themis TEM operated at 300 kV and equipped with ChemiSTEM technology and on a JEOL C$_s$ probe-corrected ARM TEM operated at 200 kV. The spectrum images were recorded with a beam current of 3.44 nA, 1.65 nm/pixel spacing, and a dwell time of 2 $\mu$s pixel$^{-1}$. The As, In and Ga elemental maps were calculated from the EDX spectrum image by using the In–L$\alpha$, Ga–K$\alpha$, and As–K$\alpha$ lines, respectively.

3. Results and discussion

3.1. Compositional control

Randomly arranged vertical arrays InGaAs nanowires were grown at 580 °C and V/III ratio of 40 (figure 1(a)) with varying $x_{vap}$ and their average chemical composition in the solid, $x_{sol}$ was determined by HRXRD, using Vegard’s law and the relative position of the InGaAs (111) reflection peak to the Si (111) substrate peak. For this analysis we assume a fully relaxed lattice, which was confirmed by measurements of pure GaAs nanowires, for which the relative peak position accurately corresponded to the expected lattice parameter. The resulting compositions for InGaAs nanowires are plotted accurately corresponded to the expected lattice parameter.

$$k = \frac{x_{sol}(1 - x_{sol})}{x_{vap}(1 - x_{vap})}$$

(1)

is a measure of whether the growth conditions favor incorporation of In or Ga. Typically, in planar epitaxy of ternary III–V semiconductors with mixing on the cation lattice (group III), the growth conditions are such that the distribution coefficient is close to one throughout the composition range, thus giving a linearly changing $x_{sol}$ as a function of $x_{vap}$. This reflects that the group III species are incorporated rapidly as they adsorb to the growth surface, and that the transport rate to the surface is similar for both species. Instead, in figure 1(b), we see clearly a preferential incorporation of In compared to Ga leading to an In distribution coefficient much higher than one throughout the whole composition range. At low In concentration it reaches values as high as 2.6. Previous reports on InGaAs nanowires grown by selective area growth [11–14] also resulted in similar incorporation behavior as also shown in figure 1(b), highlighting the similarity between TASE and regular selective epitaxy. To double-check whether gas phase loading effects due to the selective nature of the growth influences the distribution coefficient we conducted reference growth experiments on planar InGaAs films on InP with (001), (110) and (111)B substrate orientation and otherwise similar growth conditions (see methods section). The chemical composition of the InGaAs film was then determined by the (224) peak position relative to the InP substrate peak in HRXRD reciprocal space maps. Insufficient signal was obtained in the asymmetric reflections for the (110) samples, which therefore are not included in the analysis. Interestingly, even for the planar films we observe a high In distribution coefficient in the case of (111)B oriented substrates, with a maximum value reaching 2.2. Contrary, for (001)-oriented samples the distribution coefficient is very close to one for all compositions. The same orientation dependence in chemical composition was previously observed by Elsner et al., who in addition provided a data point also for the (110) orientation [15]. This data matches our results very well, which is clear from figure 1(c), even

![Figure 1](image.png)

Figure 1. (a) SEM image taken at 65° tilt angle of randomly distributed vertical InGaAs nanowires grown on Si(111) still within an oxide template. The inset shows a colored close-up image of a single InGaAs nanowire. (b) In fraction ($x_{sol}$) in the nanowires as measured by HRXRD, charted as a function of In content in the vapor ($x_{vap}$). Included is also data for selective area growth from [11] for comparison. (c) $x_{sol}$ as function of $x_{vap}$ for planar InGaAs films grown on InP with (001) and (111)B orientation. The blue hexagon data points are from [15] and fits well to the experimental data shown here.
though a higher growth temperature of 660 °C was used in Elsner’s experiments.

Since the planar (111)B growth exhibits similarly high In distribution coefficient as for selective epitaxy we can conclude that the growth mask in selective epitaxy cannot be responsible for the high In distribution coefficient. The altered transport properties inside the oxide templates do not appear to change this behavior either. Instead, the consistently large In distribution coefficient of the (111)B surface compared to the (100) and (110) facets indicates that the surface chemical reactions on the (111)B surface strongly favors In incorporation.

3.2. Facet-selective incorporation in TASE

To more closely investigate the possibility of surface-selective incorporation in TASE-grown InGaAs nanocrystals we characterize horizontal InGaAs nanowires in HRTEM and EELS. These samples were grown along the [110] crystal direction at low temperature (520 °C, V/III = 160, \( x_{\text{vap}} = 0.51 \)) firmly in the kinetically limited growth regime where this effect should be strongest. Such a nanowire is shown in the TEM image of figure 2(a) where the Si seed is also visible in the inset to the right of the InGaAs nanowire and the final faceting at the end of the nanowire are visible in the inset to the left. The Si seed surface has a V-shaped profile originating from the anisotropic wet etch used to etch back the Si during the template fabrication process [8]. More information is available in the supplementary information. The growth front of this nanowire is a vertical (110) facet in the top half and a (111) facet in the bottom half. The (111) facet can be identified as (111)A because of the presence of a few twin plane defects near the Si/InGaAs interface in the otherwise twin-free nanowire. These defects are known to occur primarily on the (111)B growth plane, thus giving us the polarity of the structure. The faceting at the end of the InGaAs nanowire does not depend on the geometry of the initial Si seed, but rather corresponds to the equilibrium shape of a single crystalline InGaAs crystal at a given growth condition [16]. Elemental EELS maps are displayed in figures 2(b)–(d). The concentration of As is constant throughout the nanowire while there appears to be a clear step in the In and Ga concentration originating from the edge between the two growth facets. The crystal below this step has a slightly lower In content than the upper part of the nanowire, which can be understood if one assumes that the upper part originates from growth on the upper (110) surface, while the lower part of the nanowire originates from growth on the (111)A surface. This agrees with a report on GaInP liquid phase epitaxy, where the In distribution coefficient on the (111)A facets was found to be lower than on (111)B facets [17] and thus should also be lower than on (110). In

![Figure 2](image-url)
3.3.1. Two-dimensional structures. An even more complex situation arises when the one-dimensional growth of a nanowire fans out into two dimensions. In Figure 4 we show a plan-view annular dark-field Scanning TEM (STEM) image and the simultaneous EDX maps obtained from a horizontal InGaAs platelet grown at 560°C, $x_{\text{cap}} = 0.50$ and $V/\text{III} = 20$. Here we observe a relatively constant and high In content in the nanowire part of the structure near the Si/InGaAs junction, consistent with the previously presented results. But as the growth proceeds into the wider segment of the template two distinct compositional regions appear, with high In content in the center region (I) and higher Ga content in the side regions (II). The appearance of the regions labeled in the Si segment of the structure, and partly because the Ga enhancement is observed arbitrarily at the bottom surface or the top in different nanowires. Similarly, it is not believed that the Ga enhancement is a TEM electron beam-induced artifact because thorough studies on electron beam dose and scan direction showed no effect.

Instead we note that the Ga enrichment regions match the edge profile of the growth facet morphology observed for these growth parameters [16], which consists mainly of two intersecting $[110]$ facets, (101) and (011), bordered on the top or bottom with a small $(11\bar{1})B$ facet. A schematic image of this morphology is displayed in Figures 3(d), (e). Because the Si substrate has a non-polar diamond structure, while the InGaAs crystal structure is polar zinc-blende, either one of these two equivalent structural variants may arise in a template depending on the polarity of the initial InGaAs nucleus on the Si [10]. Judging from the similarity of the edge structure we conclude that Ga incorporation is enhanced on the edges in-between the low index facets at the growth front. It was previously reported that AlGaAs layers grown on structured substrates exhibit Al-rich regions where the convex curvature is the highest [18]. Similarly, AlGaAs and AlnP shells grown around GaAs nanowires also exhibit Al-rich regions at edges in-between [110] facets [19–21]. Biasiol and Kapon model this type of phase segregation as being the result of a non-uniform surface chemical potential; being higher at the edges compared to the low index facets. This causes diffusion of surface species towards the low index facets, but because of stronger Al–V bonds compared to Ga–V bonds the Al-content at the convex edges is enhanced [18]. Here, a sufficient explanation is that the incorporation on the stepped edge surface corresponds more to that on [001] surfaces on which the distribution coefficient is unity. In support of this a weak Ga-rich edge profile remain even when a very high $V/\text{III}$ ratio is used ($V/\text{III} = 200$) as is seen in Figure 3(c). At this $V/\text{III}$ ratio, the $(11\bar{1})B$ facet is dominant (compare Figure 3(f)), as is reflected by the changed Ga-rich edge profile. Notably, we do not see a significant difference in In content on the $(11\bar{1})B$ versus (101) and (011) facets for these growth conditions, indicating that at high temperatures and high $V/\text{III}$ ratios, at least on the low index facets, the composition is no longer governed by chemical reactions.
II can be explained by the appearance of vertical \{110\} growth facets as the nanowire crystal extends into the wider template. As discussed above, growth on these facets is expected to be more Ga rich than on the \{111\}B facet of region I. Furthermore, the overall composition of regions II becomes more Ga-rich towards the side walls of the template. This result hints at a mass transport related effect, which appears to favor Ga to reach and incorporate on the \{110\} facets as the space on the sides of the template becomes narrower. All in all, the platelets constitute a good example of the complex interplay between reaction limited and transport limited incorporation.

### 3.3.2. Mass transport into deep templates.

After the discussion of the kinetically limited growth regime taking place at lower temperatures, we explore the mass transport limited growth regime. Figure 5(a) shows a TEM image of a nanowire from the same sample set as figure 3(c) (7 = 580 °C, V/III = 200, x_{cap} = 0.51), that has just precisely grown out of the opening of an almost 700 nm deep template. The EELS map and line scan analysis of this nanowire is displayed in figures 5(b)–(d). HRTEM images of the nanowire are available in the supplementary information (figure S2). We suspect that at this growth temperature and high As overpressure, the surface chemical reactions relating to incorporation are fast, giving concentrations of group III adsorbed species close to zero. Under such conditions we can assume that the mass transport rates of the Ga and In-related species determine \(x_{\text{sol}}\) giving minimal distinction between growth on different facets. Indeed, here we do find a uniform composition in the cross-section throughout the entire nanowire. There is also no discernable compositional fluctuation occurring close to the Si/InGaAs interface as was observed at lower temperature (and lower V/III, not shown). Instead, we do observe a simple gradual change of the composition from the heterojunction towards the template opening, going from about \(x_{\text{sol}} = 30\%\) to \(x_{\text{sol}} = 60\%\) at the inner edge of the nanowire.
end of the nanowire. This straightforward situation gives the opportunity for learning what are the limiting mechanisms for mass transport into deep templates. In the following we assume that chemical reaction rates are not limiting incorporation under these conditions.

Initially after nucleation, the InGaAs growth front resides more than 700 nm within a high aspect ratio tube. In an earlier work we concluded that the mass transport in InAs TASE occurs through Knudsen diffusion of the In and As precursors, with an additional In surface diffusion contribution [16]. In stark contrast to the In-rich compositions observed for lower growth temperature or V/III ratio, here we observe an initial \( x_{\text{sol}} = 0.3 \), despite \( x_{\text{vap}} = 0.5 \). This would require that the transport of the Ga species is more than twice as effective as for the In species. The Knudsen diffusion flow for species \( i \) is expressed as

\[
\Phi_i^F = \frac{\hat{v}_i \Delta p_i d}{3LRT}
\]

where \( \Delta p_i \) is the gradient in the partial pressure of species \( i \), \( L \) and \( d \) are the length and diameter of the template, while \( \hat{v}_i = \sqrt{\frac{RT}{m_i}} \) is the thermal velocity of species \( i \). At 580°C it is likely that at least partial homogeneous decomposition is taking place in the vapor, thus we assume that the relevant diffusive species are either monomethyl-(In, Ga) (MM(In, Ga)) or the elemental species (In, Ga). The relative difference in Knudsen flow between In and Ga species depends solely on the difference in mass between molecules. This gives \( \Phi_{\text{MMGa}} / \Phi_{\text{MMIn}} = 1.25 \), and \( \Phi_{\text{Ga}} / \Phi_{\text{In}} = 1.28 \), which are both certainly significantly higher than unity, but not enough to explain \( x_{\text{sol}} = 0.30 \) at the initial growth interface.

However, in accordance with the results of reference [16], we should also compare the efficiency of the surface diffusion contribution on the SiO\(_2\) to the transport of the group III species. We can safely assume a negligible bonding strength of adatoms on the SiO\(_2\) surface at the growth temperatures of interest, since the growth is selective at these conditions. Thus, one can describe surface diffusion as Brownian motion of adatoms formulated by Fick’s diffusion:

\[
\Phi_F^i = -D^i \frac{\partial C^i}{\partial x},
\]

where \( D^i = \frac{i L}{m_i} \) in the first order approximation, and \( \frac{\partial C^i}{\partial x} \) is the gradient of concentration for species \( i \). Thus, under the assumption of the same concentration gradients (\( x_{\text{vap}} = 0.5 \) outside of the template and \( C^i = 0 \) at the growth interface) one gets \( \Phi_F^{\text{MMGa}} / \Phi_F^{\text{MMIn}} = 1.55 \) and \( \Phi_F^{\text{Ga}} / \Phi_F^{\text{In}} = 1.65 \), respectively. The highest of these numbers would result in \( x_{\text{sol}} = \frac{1}{1 + 1.65} = 0.38 \), which is close to the measured initial composition. Our hypothesis is therefore that apart from Knudsen diffusion there is a significant surface diffusion contribution to the material transport to the growth interface (figure 5(e)). This is similar to our finding for InAs TASE, where we concluded that the In species were transported via surface diffusion in addition to Knudsen diffusion [16].

Outside the template regular vapor phase diffusion should be the dominant transport mechanism, dwarfing any contribution from surface diffusion (figure 5(e)) [22–24]. In MOVPE we have diffusion of dilute species in an H\(_2\) carrier gas, which reduces the impact of differences between the individual dissolved molecular species. Thus, the difference between TMG and TMIn diffusivities outside the template will be less than 1% as obtained by the Sutherland-Einstein relation, leading to an expected \( x_{\text{sol}} = 0.5 \) in the InGaAs crystal at the template opening and outside. In addition, as the unconstrained transport outside template is much more efficient, we could expect to obtain similar conditions as in our planar calibration (figure 1(c)) with expected \( x_{\text{sol}} \) between 0.6–0.77. There must of course be a gradual change between the two extreme cases of surface diffusion limited incorporation deep within the template, and gas diffusion/reaction limited growth outside, which explains the gradual change of composition throughout the length of the nanowire in figure 5.

4. Conclusion and summary

We have presented an investigation into the compositional variations of InGaAs nanostructures grown by the TASE method in confined geometries. We can confirm preferential In over Ga incorporation on the (111)B facet, as has been observed also in planar epitaxy previously. Similarly, the incorporation on other facets appear to match the expectations from planar epitaxy. Secondly, at low growth temperature (520°C) or if the V/III ratio is low (< 40) we observe a facet-dependent incorporation, with higher Ga content on the edges between low index growth facets. At higher temperature (580°C) and higher V/III ratio (200) we find conditions for which incorporation appears mostly transport limited with only weak Ga enrichment on the edges between facets (see table 1). The gradual change from a very Ga-rich composition at the heterojunction to a balanced composition at the template opening indicates that mass transport of Ga and In species in deep templates occurs mainly via surface diffusion inside the templates.
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