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Long indium-rich InGaAs nanowires by SAG-HVPE

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Abstract

We demonstrate the selective area growth (SAG) of InGaAs nanowires (NWs) on GaAs (111)B substrates using hydride vapor phase epitaxy (HVPE). A high growth rate of more than 50 $\mu\text{m}/\text{h}$ and high aspect ratio NWs were obtained. Composition along the NWs was investigated by energy dispersive x-ray spectroscopy (EDS) giving an average indium composition of 84%. This is consistent with the composition of 78% estimated from the photoluminescence (PL) spectrum of the NWs. Crystal structure analysis of the NWs by transmission electron microscopy indicated random stacking faults related to zinc-blende/wurtzite (ZB/WZ) polytypism. This work demonstrates the ability of HVPE for growing high aspect ratio InGaAs NW arrays.

Introduction

III-V semiconductor nanowires (NWs) are currently considered as the most versatile family of one-dimensional (1-D) nanostructures due to their unique physical properties and potential applications in nanoelectronics and optoelectronics¹⁻³. Among III-V semiconductors, a particular interest lies in the growth of InGaAs material because of its widely tunable bandgap wavelength (from 880 to

3500 nm) and high electron mobility. The composition of planar InGaAs heterostructures is restricted to nearly lattice-matched substrates, such as $\text{In}_{0.53}\text{Ga}_{0.47}\text{As}$ on InP^4 , to inhibit strain induced defects. In contrast, the more efficient strain relaxation of NWs allows the integration of InGaAs NWs on diverse substrates with much larger composition tunability. Thus, a wide variety of applications have been realized using InGaAs NWs such as infrared photodetectors⁵, lasers⁶, field-effect transistors^{7,8}, and photovoltaics^{9,10}.

Several methods have been developed to grow InGaAs NWs. The most common one is the vapor-liquid-solid (VLS) method¹¹ where a metal catalyst is used to nucleate NWs. For example, a wide range of InGaAs composition has been obtained by Au-assisted growth^{12,13}. However, Au may incorporate into NWs and cause deep level centers^{14,15}. Consequently, much effort has been invested in growing Au-free InGaAs NWs in the self-catalyzed VLS approach, where the Au catalyst is replaced by a group III element droplet. Nevertheless, some issues exist with self-assisted growth, such as an incubation time for In precipitation and a limitation in In concentration (less than 5%)^{16,17}. Therefore, efforts have been made to use a catalyst-free vapour-solid (VS) method to grow InGaAs NWs. In addition, to achieve homogeneous NW growth¹⁸, selective area growth (SAG) is widely used. In SAG, the growth occurs in defined openings on a patterned dielectric mask. This leads to highly regular, large-scale NW arrays. SAG of InGaAs NWs has been demonstrated by MBE and MOVPE^{19,20}.

In this paper, we used hydride vapour phase epitaxy (HVPE) to grow InGaAs NWs. Using III-chloride and V-hydride precursors, HVPE has been proven to be a powerful tool to grow III-V materials. Thanks to the low sticking coefficient of the III-chloride precursors on dielectric masks²¹, SAG with high selectivity has successfully used to grow binary GaAs and InAs on both GaAs (111)B and Si (111) substrates²²⁻²⁴. Here, we study the features of SAG of InGaAs NWs using SiO_x patterned GaAs (111)B substrate by HVPE. A remarkably high axial growth rate (more than 50 $\mu\text{m/h}$) leads to NWs with a high aspect ratio of 23. NW composition is investigated by energy dispersive X-ray spectroscopy (EDS) along the NWs. Crystal structures and optical properties of these NWs are also discussed.

Experimental

SAG of InGaAs NWs was carried out on GaAs (111)B substrates. The SiO_x dielectric mask was deposited by plasma-enhanced chemical vapor deposition (PECVD) and patterned by electron beam lithography into an array of holes. The hole diameter was 60 nm and the pitch was 2 μm. The sample was transferred in a hot-wall HVPE operating at atmospheric pressure. The reactor is divided into three zones (source zone, mixing zone and deposit zone) and is heated by a six zone split tube furnace. H₂ is used as a carrier gas. The GaCl and InCl precursors were formed in the source zone by reacting gaseous HCl with liquid gallium and indium at 760 °C. The AsH₃ gas was introduced in the mixing zone which is heated at a higher temperature to guarantee homogeneous mixing of the gas phase. When AsH₃ is introduced in the reactor, it is completely decomposed in As₂/As₄ gaseous species with equilibrium concentrations. The substrate was deposited in the deposit zone which is maintained at a lower temperature than the mixing zone. In this study, no intentional catalyst was used for growth. The vapor phase above the substrate is composed of GaCl, InCl, As₂/As₄, H₂ and residual HCl species. At the growth temperature used in this study, the partial pressure of As₄ is 100 times greater than that of As₂. The partial pressure of As₄, GaCl, and InCl are noted as P_{As4}, P_{GaCl}, and P_{InCl}, respectively. In this study, P_{As4}=7.5×10⁻⁴ atm, P_{GaCl}=6.6×10⁻⁴ atm, and P_{InCl}=1×10⁻² atm leading to a III_{In}/(III_{In}+III_{Ga}) ratio of 0.94 and to a (III_{In}+III_{Ga})/V ratio of 3.55. The substrate was stabilized under As₄ during the heating phase. The growth starts when InCl and GaCl fluxes are turned on simultaneously. The sample was heated at 715 °C for InGaAs deposition and the growth time was 10 min. At the end of the growth, both InCl and GaCl are turned off simultaneously. The substrate was then cooled under As₄ until the temperature of 560 °C.

The morphology of as-grown NWs was investigated using a scanning electron microscope (SEM) with an acceleration voltage of 3 kV and the crystal structure was studied using a FEI-TECNAI high resolution transmission electron microscopy (HRTEM) operated at an acceleration voltage of 200 kV. EDS was used in a SEM to estimate the chemical composition of the NWs based on K-alpha In and Ga lines. Photoluminescence (PL) spectroscopy was performed using a Bruker-Vertex 70 FTIR spectrometer equipped with a KBr beamsplitter and a cooled InSb detector. PL was excited by a 780 nm laser diode. The spot diameter was 200 μm and the power was 5 mW. The sample was cooled using a closed cycle He flow cryostat.

Results and discussion

Figure 1 shows SEM images of as-grown InGaAs NWs and three EDS profiles along a single NW. The SEM images in Figure 1a reveal pencil-like shaped NWs grown in the [111]B direction with a diameter of 370 nm at the top of the NW and a length of 8.7 μm , corresponding to a remarkable axial growth rate of more than 50 $\mu\text{m}/\text{h}$. This value is notable in SAG compared to the growth rate below 10 $\mu\text{m}/\text{h}$ observed in MOVPE^{25,26} and MBE^{20,27}. The NW morphology is pencil-like because the intrinsic growth rate of the (111)B facet is greatly superior to the lateral facets assumed to be $\{-110\}$. The radial growth rate on the $\{-110\}$ side facets, which consist of Ga-As rows, is generally very low over a wide range of HVPE conditions because of the low adsorption of GaCl on the $\{-110\}$ surface²⁸. However, it cannot be excluded that this morphology could also be interpreted by the formation of element III droplets at the top of InGaAs NWs which shrink at the end of growth.

Figure 1b-d shows EDS profiles along the length of a single NW. The EDS results show an average In composition of 84%, highlighting that these NWs are In-rich InGaAs. The difference between the In composition in the vapor phase (93%) and the In composition in the solid (84%) is a well-known phenomenon for the growth of ternary NWs. This is mainly explained by the different transport of Ga and In atoms into the NW, due to the different diffusion lengths on the NW sidewalls and different desorption rates²⁹. The Ga and In concentrations were nearly homogeneous along the length of the three different NWs analyzed. Such composition has already been demonstrated by MBE and MOVPE but on shorter NWs^{20,30}. Therefore, HVPE seems to be a powerful tool, able to achieve ternary composition with good homogeneity over nearly 10 μm in NW length.

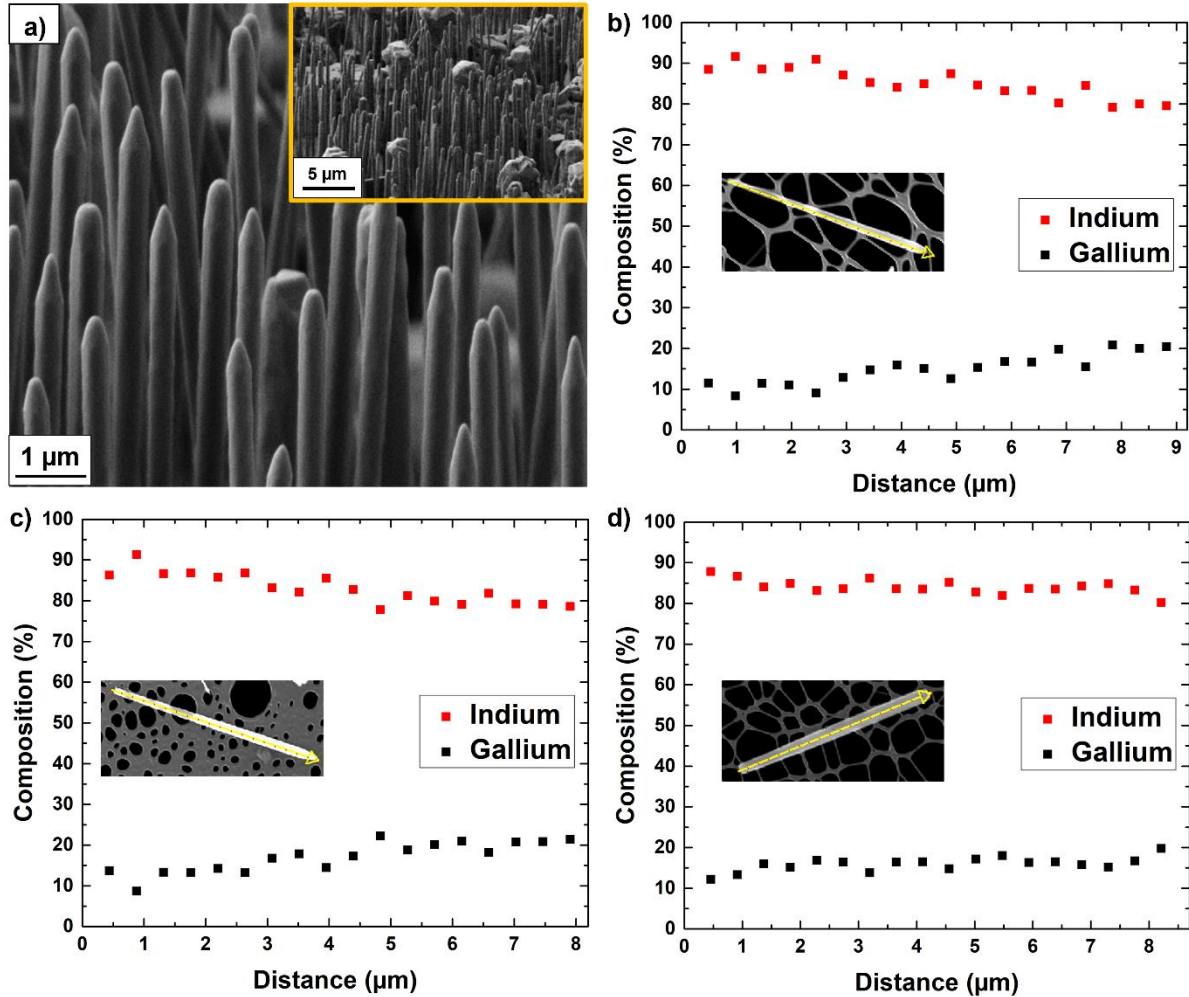


Figure 1: a) 20° tilted-view SEM image of InGaAs NWs with an inset that shows a wide field SEM image. EDS profiles along the [111]B direction of single InGaAs NWs showing an average indium composition of b) 85%, c) 83% and d) 84%. Points run from base to top of the NW.

The crystal structure of the NWs was characterized by HRTEM as shown in Figure 2. Random stacking faults related to the zinc-blende/wurtzite (ZB/WZ) polytypism are observed. This polytypism is commonly observed during the growth of III-As NWs along the [111]B direction^{20,31,32} and is mainly due to the small difference in the internal formation energies of ZB and WZ phases^{33–35}.

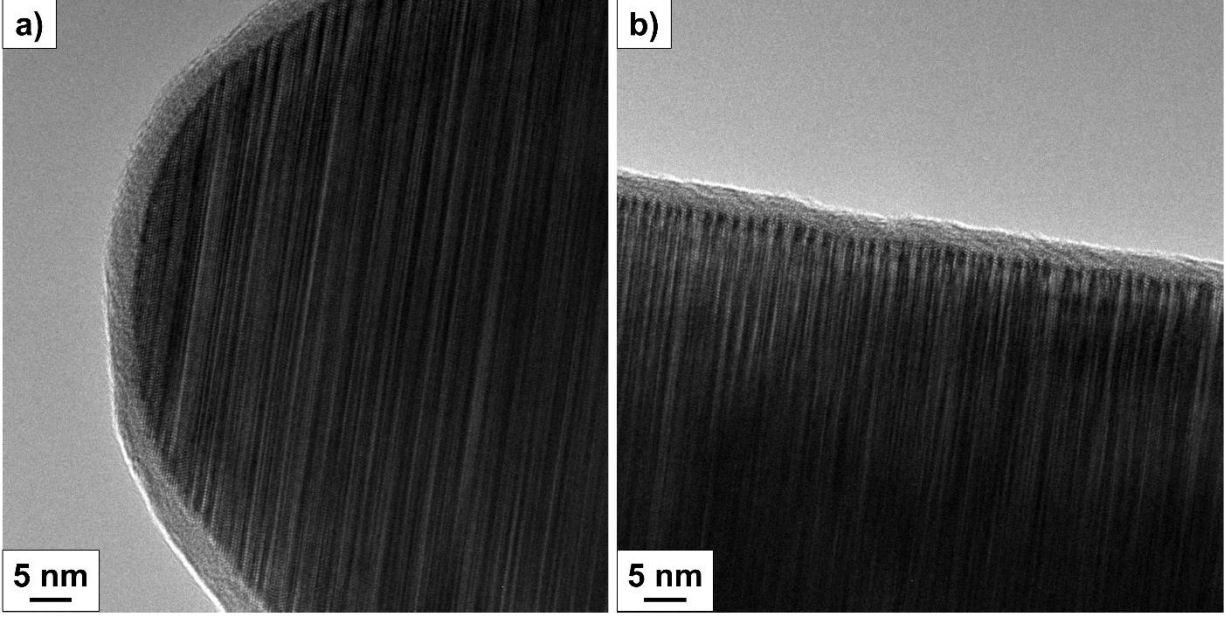


Figure 2: HRTEM images of InGaAs NW at a) the top and b) the bottom of the NW. The shell present visible on the NW is an oxide layer. The incident electron beam was along the $\langle 1-10 \rangle$ direction.

In order to confirm the composition, photoluminescence (PL) characterization was performed at low temperature (14 K) on as-grown NW array. The spectrum in Figure 3 shows a main broad peak at 0.58 eV with a secondary peak at 0.50 eV. The main peak is likely due to either a band-to-band or exciton-related transition and the low energy peak may be due to unintentional impurity-related transitions. The full width at half maximum (FWHM) of 0.042 eV of the main peak is similar to observations made on InGaAs NWs grown by MOVPE and MBE^{18,19,36}. Two hypotheses could explain the width of the main emission peak. As TEM measurements have shown, the as-grown NWs exhibit a high density of stacking faults related to the ZB/WZ polytypism. Consequently, the presence of both ZB/WZ phases could result in an increase of the emission peak width due to difference in the bandgap energy of these two phases^{18,37}. The second reason concerns the homogeneity of composition between the NWs. A variation of the composition leads to different energy emissions. To determine the composition from the main peak of the PL spectrum, Eq. (1) taken from Ref.³⁸ is used:

$$Eg(x, T) = 0.42 + 0.625x - \left[\frac{5.8}{(T+300)} - \frac{4.19}{(T+271)} \right] \cdot 10^{-4} T^2 x - \frac{4.19 \cdot 10^{-4} T^2}{(T+271)} + 0.475x^2 \quad (1)$$

where E_g is the InGaAs bandgap in eV (taken as the peak energy of the PL main peak), x is the Ga composition and T is the absolute temperature. From Eq. (1), the indium composition is estimated at 78%. This is close to the indium composition of 84% measured by EDS. However, we note that Eq. (1) was verified for unstrained cubic ZB material. The NWs are expected to be unstrained due to their high aspect ratio but they exhibit a ZB/WZ polytypism. As a consequence, the WZ phase of InGaAs NWs may affect the position of the PL peak and therefore Eq. (1) does not provide the precise value of the composition.

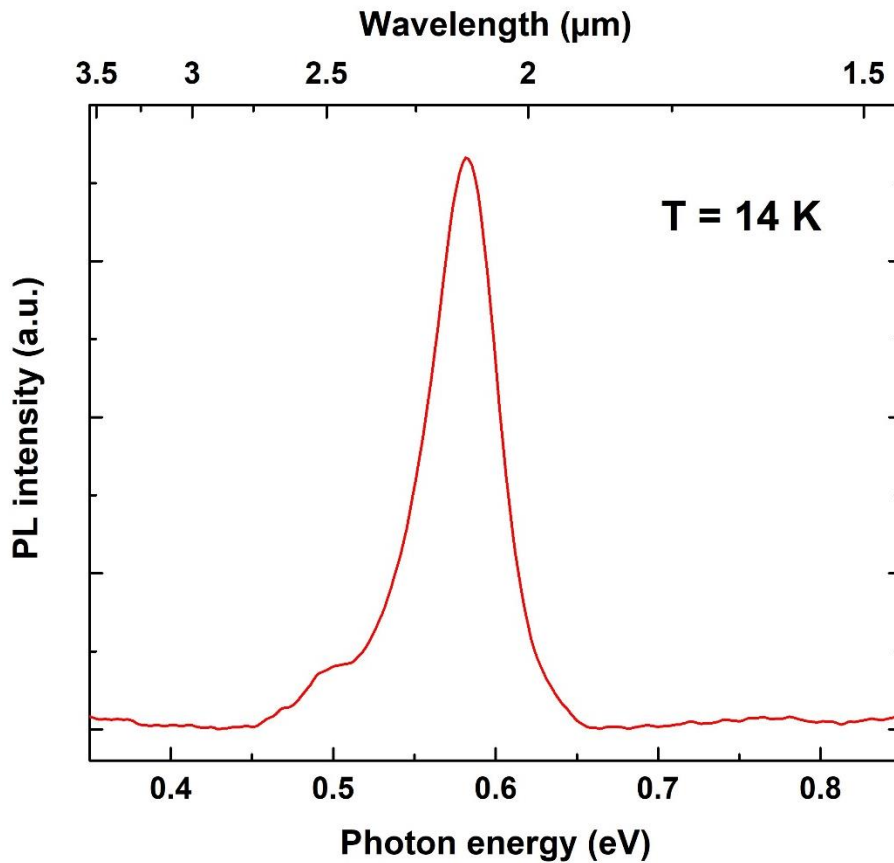


Figure 3: 14 K PL spectrum of InGaAs NW array.

Conclusion

In conclusion, we demonstrated the SAG of InGaAs NWs on GaAs (111)B substrate with good selectivity using the HVPE technique. A high growth rate was observed of more than 50 μm/h leading to high aspect ratio NWs. The TEM measurements confirmed the presence of stacking

faults in the NWs. The composition was investigated by EDS profiles along a single NW, resulting in an average indium composition of 84% with nearly homogeneous concentrations along the NW length. The estimated composition by PL of 78% is consistent with the EDS results. Overall, SAG of InGaAs NWs by HVPE provides a new platform for the fabrication of homogeneous InGaAs NW arrays with high aspect ratios.

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