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Catalyst-free selective-area epitaxy of GaAs nanowires by metal-organic chemical vapor deposition using triethylgallium

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Catalyst-free selective-area epitaxy of GaAs nanowires by metal-organic chemical vapor deposition using triethylgallium

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ABSTRACT

We demonstrate catalyst-free growth of GaAs nanowires by selective-area metal-organic chemical vapor deposition (MOCVD) on GaAs and silicon substrates using a triethylgallium (TEGa) precursor. Two-temperature growth of GaAs nanowires – nucleation at low temperature followed by nanowire elongation at high temperature – almost completely suppresses the radial overgrowth of nanowires on GaAs substrates while exhibiting a vertical growth yield of almost 100 %. A 100 % growth yield is also achieved on silicon substrates by terminating Si(111) surfaces by arsenic prior to the nanowire growth and optimizing the growth temperature. Compared with trimethylgallium (TMGa) which has been exclusively employed in the vapor-solid phase growth of GaAs nanowires by MOCVD, the proposed growth technique using TEGa is advantageous because of lower growth temperature and fully suppressed radial overgrowth. It is also known that GaAs grown by TEGa induce less impurity incorporation compared with TMGa, and therefore the proposed method could be a building block for GaAs nanowire-based high-performance optoelectronic and nanoelectronice devices on both III–V and silicon platforms.

Keywords

Nanowire, GaAs, MOCVD, catalyst-free, selective-area, TEGa

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1. INTRODUCTION

Freestanding GaAs nanowires have emerged as promising building blocks for nanoelectronic and nanophotonic devices, such as light-emitting diodes [1], solar cells [2], lasers [3], field-effect transistors [4], and photodetectors [5]. Various growth techniques have been proposed to realize the growth of nanowires with high material quality. Vapor-liquid-solid (VLS) method is the most common method to grow freestanding nanowires, which typically employs Au catalysts as seed particles. However, Au is incorporated in nanowires and substrates during the growth [6], and this is an especially critical problem when grown on Si substrates since deep-level traps are formed in Si by Au impurities [7]. Vapor-solid (VS) method, on the other hand, is a catalyst-free growth technique which does not employ any foreign or self-formed catalysts. Nanowires are grown by the adsorption of vapor-phase atoms and their diffusion on the surfaces in the VS method [8]. This VS growth can arise either on specified positions on the substrates by selective-area epitaxy (SAE) [9, 10] or on random positions of the substrates [11]. The VS SAE technique, which controls the nanowire growth sites by depositing a dielectric film and exposing nanoholes, is particularly useful in the applications requiring precise arrangement of nanowires such as photonic crystal devices [12] and plasmonic devices [13].

Growth of GaAs nanowires by VS SAE has been mostly achieved using metal-organic chemical vapor deposition (MOCVD). Trimethylgallium (TMGa) and triethylgallium (TEGa) are two typical gallium (Ga) sources used for the epitaxy of GaAs thin films; however, for nanowires, TMGa has been a commonly used precursor [9, 14-16] and the use of TEGa is limited [17]. Compared with TMGa, TEGa is known to have much lower decomposition temperature as well as much lower vapor pressure than TMGa, which make TEGa to be more suitable than TMGa when low growth temperature or slow growth rate is required [18, 19]. More importantly, it has been demonstrated that the concentration of carbon impurities in GaAs can be significantly reduced when TEGa is used instead of TMGa, regardless of the group-V precursors [19, 20]. Despite these advantages, there has been no study on GaAs nanowires grown by a SAE technique using TEGa, to the best of our knowledge.

Here, we report on the VS SAE growth of GaAs nanowires by MOCVD using TEGa. The growth on GaAs(111)B substrates shows that the growth temperature and the molar flow rate are two critical factors to achieve high uniformity and high vertical growth yields. A two-temperature growth

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method – nucleation at a lower temperature followed by nanowire elongation at a higher temperature – is proposed to completely eliminate radial overgrowth and achieve a growth yield of 100 %. The growth on Si(111) substrates is also demonstrated to verify the compatibility with silicon-based nanoelectronic and photonic platforms.

2. EXPERIMENTAL SECTION

GaAs nanowires are grown on undoped GaAs(111)B wafers and lightly p-doped (Boron, 10−20 Ω-cm) Si(111) wafers. First, 20 nm-thick SiO_2 films are deposited on GaAs wafers by electron-beam evaporation, whereas 20 nm-thick Si_3N_4 films are deposited on Si wafers by low-pressure chemical vapor deposition (LPCVD) as growth masks. Then, nanoholes are patterned by electron-beam lithography, followed by reactive-ion etching (RIE) to expose nanoholes for SAE. CHF_3 and Ar plasma are used for GaAs substrates, while CHF_3 and O_2 plasma are used for Si substrates in the RIE process to expose nanoholes. Finally, the wafers are diced into square-shaped 7 × 7 mm^2 size pieces, and the resist is fully removed by wet etching.

The nanowire epitaxy is performed using a low-pressure (60 Torr) vertical MOCVD reactor (Emcore D-75) which uses hydrogen (H_2) as a carrier gas. Si samples are cleaned by a 6:1 buffered oxide etch (BOE) solution for 30 s followed by water rinsing and drying to remove native oxide right before loading the sample into the reactor. TEGa and tertiarybutylarsine (TBAs) are used as precursors for all experiments. For the growth on GaAs substrates, the reactor is directly ramped up to 720 °C and held for 10 min under a TBAs molar flow rate of 5.33×10^{-5} mol/min to perform thermal deoxidation. Similarly, Si substrates are held at 860 °C for 10 min under H_2 ambient to completely remove native oxide. Prior to the growth of nanowires on Si substrates, TBAs is supplied with a molar flow rate of 1.58×10^{-4} mol/min for 10 min to form a (111)B-like surface [21]. Detailed growth conditions and corresponding growth results are discussed below. All growths are terminated by shutting off TEGa and cooling the rector down to 300 °C under a TBAs molar flow rate of 7.40×10^{-5} mol/min to prevent the desorption of nanowires.

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Optical properties of nanowires are characterized by microphotoluminescence (μPL) measurements. The nanowires are optically pumped using a continuous-wave He-Ne laser operating at 632 nm wavelength. The pump laser is focused using a 50× objective lens from the direction normal to the substrate, where the diameter of the laser beam spot on the substrate is approximately 3 μm. The emission from nanowires is spectrally resolved by a spectrometer (Acton SP-2500i, Princeton instruments) and a commercial Si photodetector (Model 2151, Newport corporation).

3. RESULTS AND DISCUSSION

3.1. GaAs nanowire growth on GaAs(111)B

GaAs nanowires are grown on nanohole arrays with a lattice period of 500 nm and an array size of 50 × 50 μm^2 (100 × 100 nanoholes). The diameters of nanoholes are varied from 80 nm to 110 nm to verify the effect of opening sizes on the growth. The samples are grown under various growth temperatures and molar flow rates to study optimized conditions for the nanowire growth. The V/III ratio of TBAs to TEGa is fixed at 30±5 by adjusting the flow rate of TBAs accordingly.

The nanowire growth is first conducted at 720 °C, which is a typical growth temperature in a VS growth mode when TMGa is used [10, 14, 22]. Figure 1 shows GaAs nanowires grown on nanoholes with different diameters. The growth period and the TEGa molar flow rate are 10 min and 2.51×10^{-6} mol/min, respectively. Although the vertical growth yield is higher than 70 % for all arrays, a non-negligible portion of irregular growths – fat and short nanowires or very short stubs – are clearly observed. The yield is improved at larger mask openings, which can be attributed to the diffusion of adatoms on nanoholes which affects the nucleation inside nanoholes [22, 23].

Since the growth temperature is one of the critical parameters governing the nanowire growth in SAE, GaAs nanowires are grown under various temperatures to compare the growth yield and nanowire morphology. The growth temperatures are varied from 720 °C to 620 °C, as shown in Figure 2. The growth yield is increased by decreasing the growth temperature from 720 °C to 680 °C, and reaches 100 % by further decreasing the temperature to 640 °C (Figure 2(c)). On the other hand, non-

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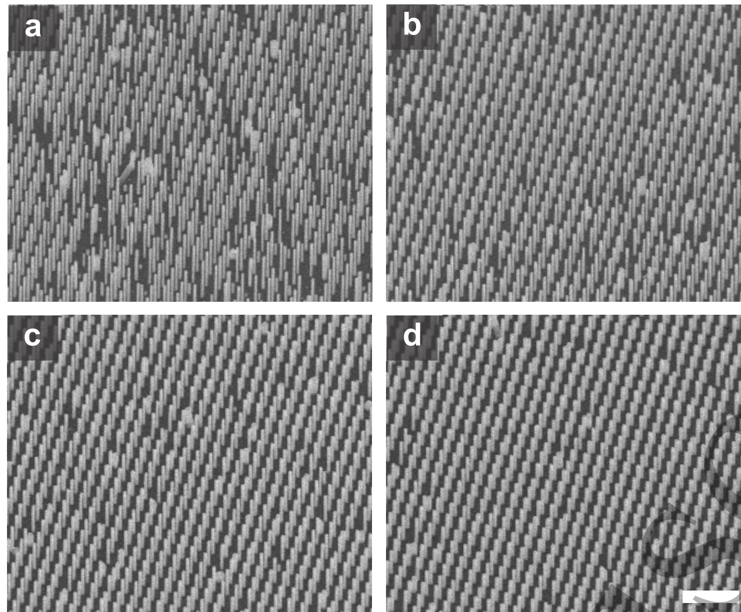

Figure 1. 30° tilted SEM images of GaAs nanowires grown at 720 °C for various nanohole opening diameters. (a) 80 nm, (b) 90 nm, (c) 100 nm, (d) 110 nm. The growth yield improves for larger mask openings. Scale bar, 2 μm.

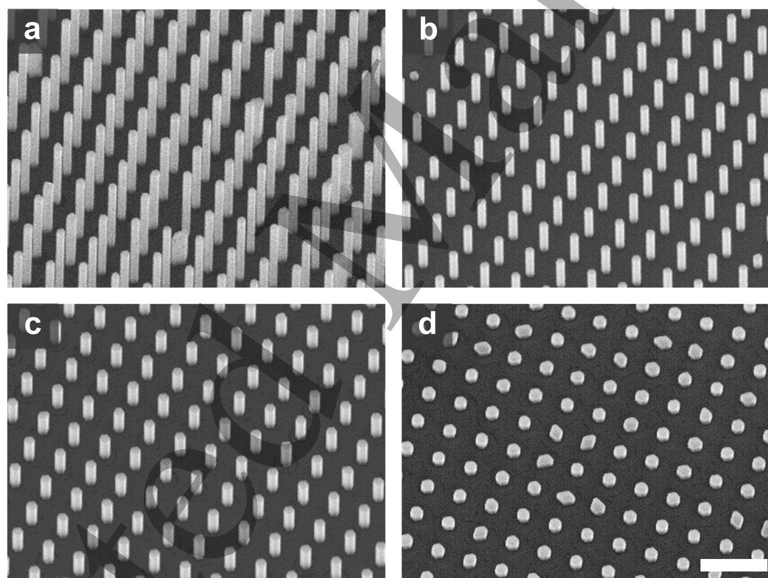

Figure 2. 30° tilted SEM images of GaAs nanowires grown under various temperatures. The temperatures are: (a) 720 °C, (b) 680 °C, (c) 640 °C, (d) 620 °C. The diameter of nanohole openings is 90 nm for all arrays. Scale bar, 1 μm.

hexagonal nanowires are observed when the temperature is further decreased to 620 °C. In addition, the nanowires grown at 620 °C show a lower aspect ratio (the ratio of nanowire height to diameter), since nanowires tend to grow radially rather than axially when the growth temperature is decreased [8]. We note that the molar flow rate of TEGa is reduced when the growth temperature is decreased for the nanowires shown in Figure 2. The TEGa molar flow rates are 2.51×10^{-6} mol/min, 8.03×10^{-7}

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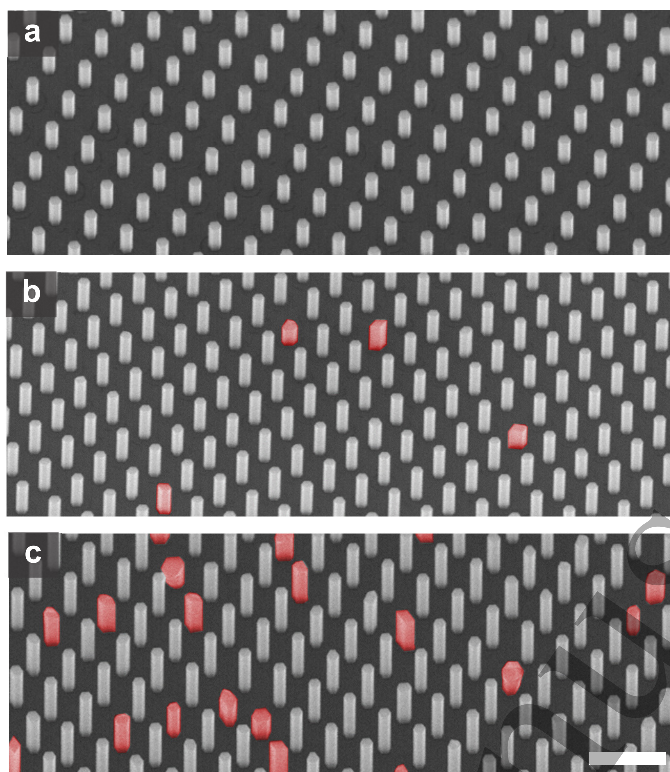

Figure 3. 30° tilted SEM images of nanowires grown 640 °C by changing a TEGa molar flow rate. The TEGa flow rate is (a) 4.01×10^{-7} mol/min, (b) 6.02×10^{-7} mol/min, (c) 8.03×10^{-7} mol/min. An increase in non-hexagonal growth is observed as the TEGa molar flow rate is increased. Scale bar, 1 μm.

mol/min1, 4.01×10^{-7} mol/min, and 4.01×10^{-7} mol/min, corresponding to the temperatures at 720 °C, 680 °C, 640 °C, and 620 °C, respectively. This is because the desorption and evaporation of adatoms are more significant at higher temperatures, and thus the flow of precursors need to be increased to compensate the loss. For instance, the TEGa flow is more than fivefold lower at 640 °C (Figure 2(c)) compared with the flow rate at 720 °C (Figure 2(a)) to realize the growth yield of 100 %. On the other hand, when the TEGa supply is increased by 50 % (6.02×10^{-7} mol/min) and 100 % (8.03×10^{-7} mol/min) at 640 °C, an increase in non-hexagonal and polycrystalline growth is observed as shown in Figure 3. It is interesting to note that the optimum growth temperature of 640 °C is significantly lower than the growth temperature of GaAs nanowires using a TMGa source, which is normally around 720 – 750 °C [10, 14, 22]. We ascribe this to the different organic byproducts generated from the cracking of TEGa and TMGa [18, 24], which are adsorbed on the nanowire sidewalls and affect their surface morphology as well as the optimum growth temperature [25]. The differences in the activation energy of TMGa and TEGa could also be one of the reasons for this result [26]. It should be highlighted that

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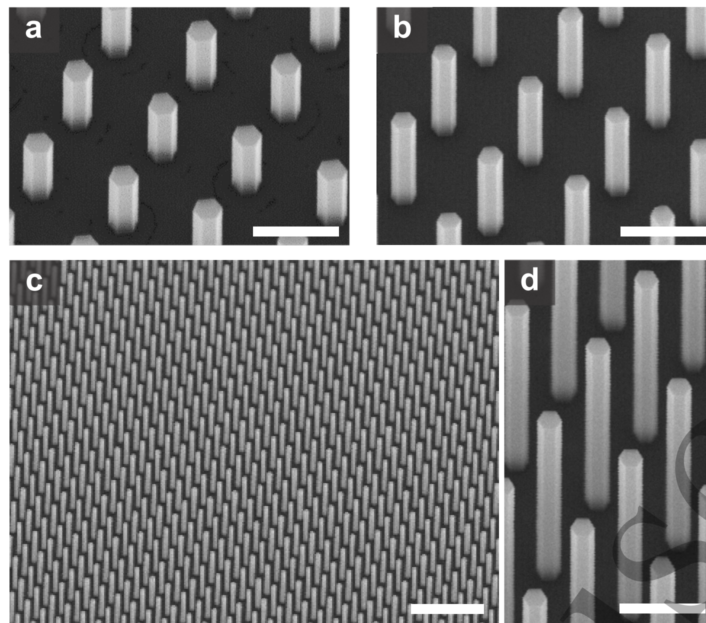

Figure 4. Two-temperature growth for high yield and high aspect ratio nanowires. (a) Nanowire growth at 640 °C gives high yield, but low aspect ratio. (b) Growth at 680 °C gives high aspect ratio, but low yield. (c) Nucleation at 640 °C followed by elongation at 680 °C gives high yield and high aspect ratio nanowires. (d) Close-up view of nanowires in (c) All SEM images are taken by tilting the stage by 30°. Scale bars, 500 nm (a, b), 2 µm (c), and 500 nm (d).

such a low growth temperature significantly reduces the diffusion length of atoms in lattices, suggesting that employing TEGa source could enable sharp interfaces when heterostructures are grown in nanowires [10].

Although the growth yield of ~100 % is achieved at 640 °C, as-grown nanowires exhibit non-negligible growth along the radial direction due to the low growth temperature. High-magnification SEM images in Figure 4(a) and Figure 4(b) show that the nanowires grown at 640 °C are fatter and shorter than the nanowires grown at 680 °C. The residual growth along the radial direction could be detrimental for photonic and plasmonic applications requiring a precise control of nanowire dimensions [27, 28]. Furthermore, the lateral overgrowth could lead to the formation of thin shells in nanowire heterostructures, which can act as parasitic paths for carriers and degrade the device performance [29]. To overcome this issue, we implement a two-temperature growth technique. In the two-temperature growth, the nucleation of nanowires is first conducted at 640 °C to ensure a high growth yield, followed by nanowire elongation at higher temperature (680 °C) to suppress radial overgrowth. The nucleation at 640 °C is conducted for 20 min with a TEGa supply of 4.01×10^{-7} mol/min, and the elongation at

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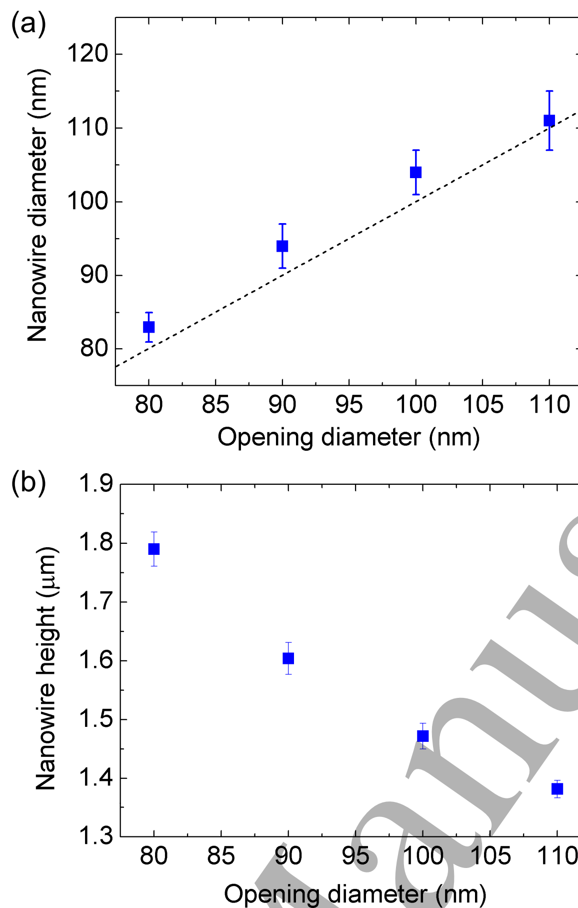

Figure 5. (a) Nanowire diameter for various mask opening diameters. Dotted line represents equal opening and nanowire diameter. (b) Nanowire height for various mask opening diameters. Error bars are standard deviation of 50 measured nanowires.

680 °C is conducted for 26 min with a TEGa supply of 8.03×10^{-7} mol/min. As shown in the SEM images in Figure 4(c) and 4(d), the nanowires exhibit 100 % growth yield with an extremely high aspect ratio. The height and diameter of as-grown nanowires are measured to quantitatively analyze the growth rates along the axial and radial directions. It is observed that as the nanohole diameters are increased from 80 nm to 110 nm, the diameters of nanowires are increased from 83 nm to 111 nm as well, as shown in Figure 5(a). The average height of nanowires is decreased from 1.79 μm to 1.38 μm by increasing the nanohole diameter, which is a well-known phenomenon in III–V nanowires grown by SAE techniques [10, 30]. Remarkably, the radial overgrowth is almost negligible (less than 5 nm regardless of the nanohole diameter), and the average vertical to radial growth rate ratio (nanowire height / radial overgrowth) is ~700, which is higher than GaAs nanowires grown by SAE using TMGa [10, 16, 22, 31]. We therefore believe that the proposed two-temperature growth of GaAs nanowires

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using a TEGa source is a promising approach to enable high-performance nanowire-based devices in photonic, optoelectronic and electronic applications.

3.2. GaAs nanowire growth on Si(111)

The growth of GaAs nanowires on Si(111) substrates is also demonstrated using a TEGa source. Nanohole arrays with a lattice period of 500 nm and an opening diameter of 70 nm is patterned on Si(111) substrates as a growth template. Compared with III-V substrates, growing vertical nanowires on Si substrates is not straightforward because GaAs nanowires tend to grow along <111>B directions whereas Si does not exhibit polarity. Nanowires can therefore grow along any of the four exposed <111> directions, which is composed of one vertical <111> direction and three angled <111> directions. To control the growth orientation, TBAs is supplied before initiating the nanowire growth to terminate the exposed Si(111) surface by arsenic and form a (111)B-like surface [21].

The GaAs nanowires are grown on Si under various growth temperatures, as shown in Figure 6. TBAs is first supplied for 10 min at the growth temperature, followed by the initiation of nanowire growth by turning on TEGa. The TEGa flow rate is fixed to 8.03×10^{-7} mol/min regardless of the growth temperature. As shown in Figure 6(a), the height of nanowires is non-uniform when the growth temperature is higher than 710 °C. In addition, a significant portion grows as a short stub, which is also observed from the GaAs nanowires grown on GaAs substrates at high temperatures (Figure 2(a) and 2(b)). The uniformity of nanowires increases by decreasing the temperature, and the vertical growth yield of nanowires reaches close to 100 % at the growth temperature of 700 °C, as depicted in Figure 6(d). However, at temperatures lower than 700 °C, the growth along angled <111> directions starts to evolve. The portion of angled nanowires and polycrystalline structures is further increased by decreasing the temperature down to 680 °C. This is attributed to the non-polar nature of Si, whereas angled nanowires are not observed on GaAs substrates regardless of the growth temperatures.

The height and diameter of GaAs nanowires grown at 700 °C is 740 nm and 105 nm, respectively, which correspond to a vertical to radial growth rate ratio of 21 given that the nanohole diameter is 70 nm. This is a significantly smaller value than the ratio obtained from GaAs nanowires grown on GaAs substrates. This could be due to a strain at the Si/nanowire interfaces, difference in

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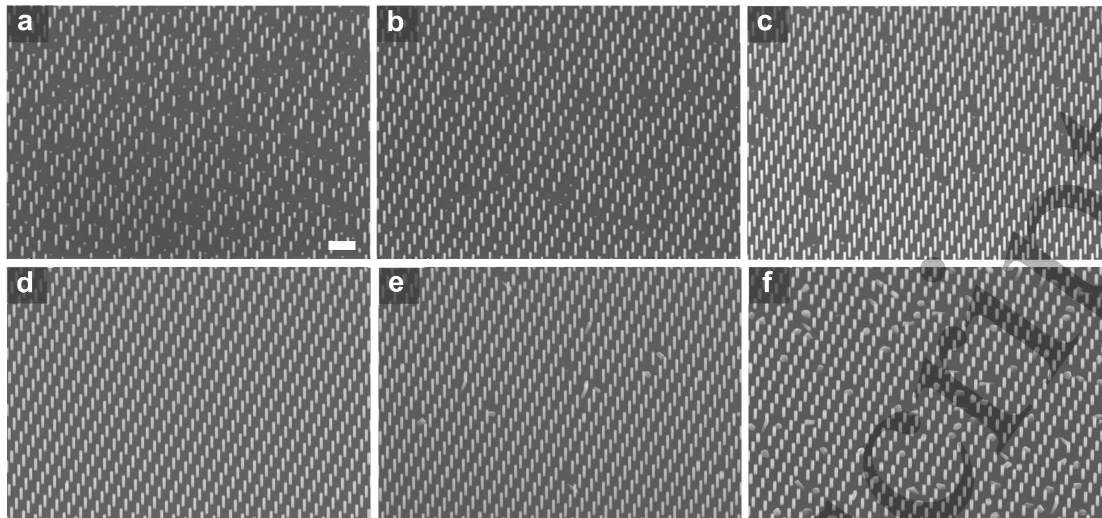

Figure 6. 30° tilted SEM images of GaAs nanowires grown on Si under various temperatures. The temperatures are (a) 740 °C, (b) 725 °C, (c) 710 °C, (d) 700 °C, (e) 690 °C, (f) 680 °C. The diameter of nanohole openings is 70 nm for all arrays. Scale bar, 1 μm.

growth temperatures, or dielectric materials of growth masks – Si_3N_4 on Si and SiO_2 on GaAs – which affect the diffusion characteristics of adatoms [32, 33]. However, further study is required to explore this discrepancy.

The optical properties of GaAs nanowires grown on Si are characterized by μPL measurements at room temperature. For comparison, supplementary nanowire samples are grown on silicon using a TMGa precursor instead of TEGa. The growth temperature is 730 °C when a TMGa source is used, and the nanowires with similar dimensions are chosen for μPL measurements to properly compare the radiative efficiency. Figure 7 shows the μPL spectra of GaAs nanowires grown on silicon using TEGa and TMGa at a pump power of 424 W/cm^2. Both samples show a dominant peak at 880 nm which correponds to the bandgap energy of GaAs at room temperature. A relatively long tail is observed at longer wavelengths in these samples, which is typically observed from GaAs nanowires with zinc-blende/wurtzite polytypism [34, 35]. Although the emission from TEGa-grown nanowires is around 20 % stronger than TMGa-grown nanowires, it is unclear whether the difference in the radiative efficiency is stemming from the concentration of carbon impurities. Effects of nanowire surface states, nanowire/substrate interface states, and scattering of carriers from stacking disorders in nanowires need to be considered together with the impurities to evaluate the efficiency. Therefore, further

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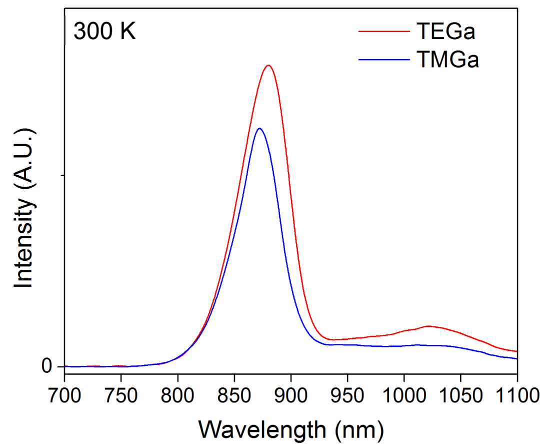

Figure 7. μPL spectra of GaAs nanowires on silicon substrates grown using TEGa and TMGa measured at room temperature.

characterization is necessary to elucidate the material properties of GaAs nanowires grown using TEGa and TMGa.

4. CONCLUSION

We have demonstrated a catalyst-free selective-area growth of GaAs nanowires using TEGa. Compared with a commonly-used TMGa source, the TEGa source could be advantageous for controlling the growth rate and lowering the impurity incorporation. A vertical growth yield close to 100 % was achieved on both GaAs and Si substrates by optimizing the growth conditions. A two-temperature growth technique is proposed to fully suppress the radial overgrowth of GaAs nanowires on GaAs substrates while maintaining a high growth yield. The growth of GaAs nanowires on Si implies that the proposed approach can be applied for a monolithic integration of nanowire-based devices on Si platforms.

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