Effect of Sn Addition on Epitaxial GaAs Nanowire Grown at Different Temperatures in Metal–Organic Chemical Vapor Deposition

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ABSTRACT: In this study, we investigated the growth behaviors of GaAs nanowires with tetraethyl-tin (Sn) as addition grown at different temperatures in a metal–organic chemical vapor deposition system. It was found that the nanowire axial growth rate can be influenced by the addition of Sn in opposite ways at different growth temperatures. The growth rate of nanowires is higher because of the enhanced decomposition of trimethyl gallium (TMGa) with increasing the Sn addition at 390 °C while lower because of the lower catalyst supersaturation level with increasing the Sn addition at 450 °C. With the Sn addition, nanowire quality can be maintained at 390 °C because the lower temperature benefits stabilizing the structure but further degraded at 450 °C when compared with intrinsic nanowires. This study provides an insight into the effect of the Sn addition on GaAs nanowire growth, which will be useful for the design of nanowire-based devices.

INTRODUCTION

III–V semiconductor nanowires have been considered as promising one-dimensional nanoscale materials in nano-electronics and optoelectronic applications due to their high aspect ratio and unique electrical and optical properties.^{1–3} For practical device applications in using nanowires as building blocks, it is necessary to understand their growth mechanism so that the required structural, physiochemical, electrical, and optical properties can be controlled. As a kind of representative III–V semiconductor nanowires, GaAs nanowires have received great attention due to their promising applications in solar cells,^{4,5} nanolasers,⁶ and the next-generation transistors.⁷ Indeed, their epitaxial growth using the metal-catalyzed approach has been well developed.⁸ In many applications, however, the electrical properties of the nanowires need to be controlled by means of doping.⁹ So far, less effort has been devoted to the epitaxial growth of doped III–V semiconductor nanowires.^{10–14} Although examples such as p-type doping in GaAs nanowires using C¹⁵/Zn^{16,17}/Be,¹⁸ n-type doping in GaAs nanowires using Si¹⁹/Te²⁰/Sn^{21,22} dopants have been reported, the growth behaviors of doped nanowires are not fully understood as these impurities may cause undesirable effects. For instance, using dimethyl-zinc as the Zn dopant precursor, axial growth of InP nanowires can be significantly enhanced²⁴ while the axial growth of InAs nanowires is impeded with the Si addition.²³ In addition, impurities may also change the crystal structure^{25–27} and structural quality^{28,29} of the nanowires. The Zn addition has modified the catalyst–nanowire interfacial energy and hence

the contact angles, leading to the switch of the InP nanowires from the zinc blende (ZB) structure to a wurtzite (WZ) structure.²⁵ Thus the properties of doped nanowires are not only affected by the incorporated dopants but can also be strongly influenced by the change in their morphology and crystal structure.^{30–32} Therefore, it is necessary to clarify the mechanism of how impurities influence nanowire growth.

In this study, we investigate the effect of tetraethyl-tin (TESn) on the epitaxial growth of GaAs nanowires grown via the metal-catalyzed approach at different growth temperatures in a metal–organic chemical vapor deposition (MOCVD) system. It is found that the TESn addition has an opposite effect on the axial growth rate of GaAs nanowires grown at different temperatures. Specifically, with increasing the TESn addition, the axial growth rate increases only marginally when GaAs nanowires grown at 390 °C but decreases significantly at 450 °C when compared with the growth of undoped GaAs nanowires. The structural quality of nanowires grown at 450 °C is also severely degraded with the TESn addition. Through detailed electron microscopy characterization, the effects of the TESn addition on the growth mechanism of GaAs nanowire are clarified and discussed.

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Figure 1. SEM side views of as-grown nanowires on substrates. (a–c) Nanowires grown at 450 °C with TESn addition of (a) 0 (reference), (b) TESn/TMGa = 0.13, and (c) TESn/TMGa = 0.26. (d–f) Nanowires grown at 390 °C with TESn of (d) 0 (reference), (e) TESn/TMGa = 0.13, and (f) TESn/TMGa = 0.26. Insets show the top views on substrate and nanowires. Scale bars in insets are 200 nm.

Figure 2. TEM images of typical reference GaAs nanowire (without TESn, a) and the GaAs nanowire with TESn addition (TESn/TMGa = 0.26, b) grown at 390 °C. (c,d and e,f) HRTEM images and corresponding SAED patterns of the marked regions in (a) and (b), respectively. Typical EDS results on nanowires compositions without TESn (g–h) and with TESn addition (i–j). Insets in (g) and (i) show the 10× magnified EDS spectra between 3.2–3.6 keV.

EXPERIMENTAL SECTION

Epitaxial nanowire growths were performed in an AIXTRON 200/4 horizontal flow MOCVD system. The chamber pressure was maintained at 100 mbar with total gas flow rate at 15 standard liters per minute. Trimethyl-gallium (TMGa), arsine (AsH_3), and TESn were used as group III, V, and IV precursors, respectively, carried by

ultrahigh-purity hydrogen gas. Prior to nanowire growth, GaAs (111) substrates were cleaned by deionized water and pretreated by poly-L-lysine (PLL). Then 50 nm diameter colloidal Au particles (British Biocell International—Ted Pella, Inc.) were dispersed and adhered onto substrate surfaces. The substrates were then annealing at 600 °C under AsH_3 ambient for 10 min in the MOCVD reaction chamber to

Figure 3. TEM investigation on a typical reference GaAs nanowire and GaAs nanowire with high TESn addition grown at 450 °C. (a–b) BF-TEM images for individual nanowires. (c–d and i–j) Enlarged areas in (a) and (b). (e–f and k–l) HRTEM images and corresponding SAED patterns with index of the marked areas in (c) and (j) respectively. (g–h and m–n) Typical EDS results show the composition of catalysts and nanowires. Insets in (g) and (m) show the 10X magnified EDS spectra between 3.2–3.6 keV.

remove oxide contaminations from the substrate surfaces. After cooling to 450 °C, nanowire nucleation was performed for 1 min by switching on TMGa. The reaction chamber was then set to the nanowire growth temperature of 450 and 390 °C, followed by ~30 min nanowire growth with switched on the TESn addition. During nanowire growth, flux rates of TMGa and AsH₃ were set at 1.157×10^{-5} and 5.357×10^{-4} mol/min, respectively, which gave a V/III ratio of 46 for all nanowire growths. Three different TESn flow rates were used, in the range from 0 to 2.993×10^{-6} mol/min, giving a nominal Sn/Ga ratio as 0, 0.13 and 0.26, respectively. Growth was terminated by switching off the TMGa and TESn sources simultaneously and cooled down in the presence of AsH₃ until 350 °C.

The nanowire morphology was determined by scanning electron microscopy (SEM, JEOL Field-Emission 7800, operated at 5 kV), and their structural and compositional characteristics were investigated by transmission electron microscopy (TEM, FEI Tecnai F20, 200 kV, equipped with energy dispersive X-ray spectroscopy, EDS). SEM specimens were prepared by cleaving the GaAs substrates along the {110} atomic planes (note that nanowires still attach on the substrates). Individual nanowires for TEM investigations were prepared through ultrasonating the substrates in ethanol and dispersing the suspended nanowires onto holey carbon-lined TEM copper grids.

RESULTS AND DISCUSSION

Figure 1 is a set of SEM images showing the side view of the nanowires grown with different TESn additions at 450 °C Figure 1a–c and 390 °C Figure 1d–f, respectively. The insets on the top-right corners of Figure 1 are the corresponding plan view of as-grown nanowires, confirming that nanowires grew perpendicularly on the substrate. Therefore, the nanowire lengths can be determined from their side views, from which nanowires are much longer when they grew at 450 °C (note the difference of scale bars in parts a–c and d–f of Figure 1), which can be explained by the lower energy barrier and the

enhanced surface diffusivity of group III adatoms at higher temperature.^{33,34} The differences in nanowire lengths grown at the same temperature can be attributed to the TESn addition. For nanowires grown at 450 °C, the nanowire length is reduced with the TESn addition (comparing parts a and b of Figure 1). With further increasing the TESn flow rate, the nanowire length reduces further (comparing parts b and c of Figures 1). Interestingly, an opposite trend is observed at the growth temperature of 390 °C, in which the TESn addition promotes axial growth rate and therefore longer nanowires are seen (comparing parts d and e of Figure 1). With further increasing the TESn addition, the average length of nanowires remains similar, indicating that after a certain amount of the TESn addition, further increment in TESn does not promote the growth rate.

To understand the structural characteristics of as-grown nanowires, TEM characterization was performed. Parts a and b of Figure 2 show two typical nanowires grown at 390 °C without and with the highest TESn addition, respectively. Parts c and d of Figure 2 are high-resolution (HR) TEM images taken from the two marked regions in Figure 2a, viewed along the $\langle 110 \rangle$ direction. The nanowire grown without TESn is defect-free and has a ZB crystal structure as shown by the HRTEM images with corresponding SAED patterns in the insets. Parts e and f of Figure 2 with insets are HRTEM images and SAED patterns taken from the marked regions in Figure 2b, viewed along the $\langle 110 \rangle$ zone axis. Similarly, when the TESn addition is used, the grown nanowires are maintained defect-free with the ZB structure. This indicates that the TESn addition does not destabilize the nanowire structural quality. Parts g and h of Figure 2 show respectively the EDS spectra taken from the catalyst and the nanowire segment for the nanowires grown without the TESn addition, indicating that

Figure 4. Schematic of GaAs nanowire growth with TESn at (a) 390 °C and (b) 450 °C.

the catalyst is an Au–Ga alloy and the nanowire is GaAs (note that Cu peaks are caused by the Cu grids). On the other hand, parts i and j of Figure 2 are the corresponding EDS spectra taken from the nanowire grown with the TESn addition, in which a small amount of Sn (~2 at. %) can be detected in the catalyst but was not found in the nanowire, indicating that, if there exists a small amount of Sn in nanowires, it should be beyond the detectability of the EDS technique.

Similar TEM characterization was performed for the nanowires grown at 450 °C. Parts a and b of Figure 3 are BF-TEM images of typical nanowires grown at 450 °C without and with the highest TESn addition, respectively. Parts c and d of Figures 3 and parts i and j of Figure 3 are magnified BF-TEM images of the marked regions in parts a and b of Figure 3, respectively. On the basis of the diffraction contrast in BF-TEM images, both nanowires contain defects, but a higher density of defects can be witnessed in nanowires grown with the TESn addition. Parts k and l of Figure 3 are the HRTEM image and SAED pattern taken from marked area in Figure 3j, in which a ZB structure with twins can be seen. The side facets shown in Figure 3k can be indexed as the {111} planes, attributed to the rotational twins.^{29,35} On the basis of our extensive TEM investigations of over a dozen nanowires from each specimen grown at 450 °C, the average defect density of nanowires with the highest TESn addition can be estimated to be 24 μm^{-1} , approximately 6 times higher than that of nanowires grown without the TESn addition. Parts g and h and parts m and n of Figure 3 are EDS spectra taken, respectively, from nanowires grown without and with the TESn addition. As can be seen, like the case of nanowires grown at 390 °C, Sn cannot be detected in the nanowire grown at 450 °C, suggesting that the Sn concentration must be at a very low level below the detectable limitation of the EDS (~1000 ppm) in our nanowires. However, the Sn concentration in the catalyst can be estimated as ~3.5 at. %, nearly double the Sn concentration found in the catalysts of nanowires grown at 390 °C.

On the basis of our SEM investigations, it is found that, with the TESn addition, the nanowire length increases when grown at 390 °C but decreases at 450 °C when compared with their intrinsic counterparts. Furthermore, for nanowires grown at 390 °C, the growth rate increases with the TESn addition but saturated when increasing the TESn addition. To understand this phenomenon, we note that the decomposition rates of

reaction precursors can influence the epitaxial growth of nanostructures in MOCVD,^{8,36} and temperature plays a crucial role in the decomposition of precursors. At 390 °C, TMGa is not fully pyrolyzed,³⁷ but the pyrolysis of TESn is nearly complete.³⁸ On the other hand, it has been reported that ethyl radicals (from dopant precursors) may react with H_2 (the carrier gas) and create H-radicals, which increases the pyrolysis rate of TMGa^{26,39,40} so that the growth rate of nanowires is enhanced by the enriched Ga source because of the TESn addition. On the other hand, further increase of the TESn addition may hinder the Ga incorporation into catalyst^{41,42} and hence causes the growth rate saturating at the higher TESn addition, evidence shown in Figure 1f. Interestingly, for nanowires grown at 450 °C, the TESn addition reduces the growth rate. To understand this, we note that the catalyst plays a crucial role in nanowires growth,^{25,43,44} and the chemical potential difference between the catalyst and underlying nanowire (the supersaturation in the catalyst) is the key driving force for axial growth.⁴⁵ In our previous study,⁴⁶ we found that when Sn is incorporated into the Au catalysts, it competes with Ga to form Au–Ga–Sn alloy droplets, resulting in a reduced Ga concentration in the catalysts and in turn lowers the catalyst supersaturation level, causing lowered nanowire growth rate. The growth rate reduction increases with increasing the TESn. In addition, it has been reported that Sn adatoms may act as surfactant and hinder Ga diffusion along the nanowire sidewalls, limiting the Ga source for the nanowire axial growth.^{41,47–49}

On the basis of our TEM investigations, we found that the GaAs nanowires grown at 450 °C contain more defects with the TESn addition. In fact, such a phenomenon has been observed and explained in our previous study.⁴⁶ Interestingly, we found that the high crystal quality of as-grown GaAs nanowires at 390 °C is still maintained, irrespective of the TESn addition, although 390 °C has been confirmed as the temperature to grow defect-free GaAs nanowires.⁵⁰ To understand why the TESn addition does not influence the structural quality of nanowires grown at 390 °C, we note that the low-temperature nanowire growth may benefit for stable formation of ZB against the instability caused by TESn addition in this study.^{51,52} In addition, a low growth temperature of 390 °C leads to a lower Sn concentration in catalysts compared with 450 °C, suggesting the weaker impact of Sn addition on the nanowire growth front at 390 °C.^{53,54}

Consequently, the nanowire quality is maintained when the nanowire growth is at 390 °C even with the TESn addition.

To summarize the impacts of the TESn addition on GaAs nanowire growth, Figure 4 illustrates how the different functions of TESn performs on nanowire growth respectively at 390 and 450 °C. Figure 4a shows the nanowire growth at 390 °C, in which the extra H-radicals caused by the TESn addition enhance the bond breaking between Ga and ethyl. Therefore, the enhanced TMGa decomposition promotes the nanowire axial growth under the III-limited ambient. Figure 4b illustrates the nanowire growth at 450 °C, denoting that Sn atoms incorporate into catalyst as well as act as surfactant on side walls, which results in slowly grown nanowires. Also, the TESn addition induces higher density of defects in the nanowires when they are grown at high temperature, indicated by red dashed lines. Note that As is not taken into this schematic illustration because the growth is proceeded in As-rich ambient, and hence As is not the limiting factor in the nanowire growth.

CONCLUSIONS

In this study, we investigate the influence of the TESn addition on GaAs nanowire growth at two temperatures (namely 390 and 450 °C) in MOCVD. Through detailed electron microscopy characterization, it has been found that, when comparing with intrinsic GaAs nanowire growth, the TESn addition causes nanowires grown longer at 390 °C but shorter at 450 °C. We demonstrate that the TESn addition can promote the TMGa decomposition during the nanowire growth at 390 °C, while Sn atoms lower the catalyst supersaturation level and may act as surfactants on the nanowire sidewalls when grown at 450 °C. In addition, by benefitting from the low growth temperature, the nanowire quality can be maintained even with the Sn addition at 390 °C. This study verifies the temperature-dependent effect of impurity addition on the epitaxial growth of GaAs nanowires, which will benefit for the design of nanowire-based device with impurity addition.

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Notes

The authors declare no competing financial interest.

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