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Complementary Metal Oxide Semiconductor-Compatible, High-Mobility, <111>-Oriented GaSb Nanowires Enabled by Vapor-Solid-Solid Chemical Vapor Deposition

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ABSTRACT – Using CMOS-compatible Pd catalysts, we demonstrate successfully the formation of high-mobility and <111>-oriented GaSb nanowires (NWs) *via* vapor-solid-solid (VSS) growth in surfactant-assisted chemical vapor deposition through a complementary experimental and theoretical approach. In contrast to NWs formed by conventional vapor-liquid-solid (VLS) mechanism, cylindrical-shaped Pd₅Ga₄ catalytic seeds are present in our Pd-catalyzed VSS-NWs. The stoichiometric composition is found to have the lowest crystal surface energy when constituted as solid catalysts, contributing to minimal surface diffusion and optimal in-plane interface orientation at the seed/NW interface for efficient epitaxial NW nucleation. All these VSS characteristics lead to the NW growth with slender diameter distribution (down to 26.9 ± 3.5 nm), uniform growth orientation (over 95 % NWs grown in <111> orientation for a wide diameter range of between 10 and 70 nm), and enhanced crystallinity. When back-gated field-effect transistors (FETs) are fabricated using these NWs, the Pd-catalyzed GaSb NW exhibits a superior peak hole mobility of $\sim 330 \text{ cm}^2\text{V}^{-1}\text{s}^{-1}$, and this value is close to the mobility limit for a NW channel diameter of ~ 30 nm with a free carrier concentration of $\sim 10^{18} \text{ cm}^{-3}$, indicating the excellent homogeneity of NW phase purity, growth orientation, surface morphology and electrical characteristics. Contact printing process is also used to fabricate large-scale assembly of Pd-catalyzed GaSb NW parallel arrays, confirming the potential constructions and applications of these high-performance electronic devices.

KEYWORDS: GaSb nanowires, growth orientation, high mobility, vapor-solid-solid, interface plane orientation, in-plane lattice mismatch

Due to their tunable bandgaps, composition, structural configurations and superior carrier mobilities, one-dimensional (1D) III-V semiconductor nanowires (NWs) are widely recognized as promising fundamental building blocks for next-generation electronics, photonics and *etc.*¹⁻¹² In particular, as a technologically important p-type semiconductor, GaSb NWs have drawn a lot of recent attention as they consist of an intermediate band gap of 0.726 eV, a high theoretical hole mobility of up to $1000 \text{ cm}^2\text{V}^{-1}\text{s}^{-1}$, and an impressively strong spin-orbit interaction, all of which are necessary for many advanced device utilizations.¹³⁻¹⁸ Although there remains a substantial challenge to obtain very thin and “non-tapered” GaSb NWs owing to the uncontrolled radial growth arising from the Gibbs-Thomson effect, high-quality and uniform GaSb NWs with diameters down to 20 nm have been successfully prepared using a sulfur-surfactant technique, which efficiently stabilizes the high-energy sidewalls for homogenous NW formation during the chemical vapor deposition (CVD) process.^{19, 20} However, the growth direction and corresponding carrier mobilities are still found to be heavily dependent on the NW diameters, wherein $\langle 111 \rangle$ -oriented GaSb NWs exhibit the highest hole mobility because of the optimized surface passivation and roughness resulting in minimized surface traps, thus indicating the potential applications of these phase-pure NWs for high-performance electronics.²⁰

On the other hand, in the previously reported surfactant-assisted growth technique, Au was typically utilized as the catalyst for the synthesis of high-quality GaSb NWs with excellent yields *via* vapor-liquid-solid (VLS) mode.^{19, 20} Unfortunately, Au is notorious for its complementary metal oxide semiconductor (CMOS) incompatibility; hence, the use of Au catalyst may restrict the deployment of these NWs for large-scale semiconductor device integration.²¹⁻²⁴ It is crucial to explore alternative means to synthesize CMOS-compatible NWs with well-controlled physical properties. For example, Robertson *et al.* employed the CMOS-compatible catalyst of Pd for the

CVD synthesis of Si NWs *via* a vapor-solid-solid (VSS) mechanism as confirmed by in-situ transmission electron microscopy (TEM).²⁵ Although both Au and Pd are transition metals, they behave dramatically different when they diffuse into the Si lattice.^{26, 27} Once Au is solid-diffused into Si, Au constitutes fatal defects quickly and deteriorates electrical properties of Si, being CMOS-incompatibility.²⁸ On the other hand, when Pd is diffused within the Si lattice, Pd would form the silicide compound of Pd₂Si efficiently without yielding any kind of defects, in which this silicide is commonly employed as the optimal electrical contact of Si devices; as a result, Pd is categorized as CMOS-compatible in the Si community.^{21, 29} The case of CMOS-compatible catalyst of Ni is similar to Pd, and Ni-catalyzed InAs NWs with electron mobilities up to 10,000 cm²V⁻¹s⁻¹ have also been successfully synthesized by Javey et al and others.^{30, 31} However, there are still limited reports of high-performance, uniform, phase-pure and, more importantly, CMOS-compatible GaSb NWs for large-scale practical device applications.

In this work, *via* a complementary experimental and theoretical approach, we propose and demonstrate the use of VSS technique to obtain high-mobility, phase-pure <111>-oriented and CMOS-compatible GaSb NWs. In contrast to Au-catalyzed GaSb NWs grown with mixed orientations and wide distributions of diameters *via* VLS mode,^{19, 20} our Pd-catalyzed GaSb NWs obtained are all grown along the <111> direction, with a diameter distribution of 26.9 ± 3.5 nm and an existence of cylindrical Pd₅Ga₄ catalytic seeds. When constituted as immobile solid catalysts, this specific CMOS-compatible catalytic composition is found to consist of the lowest crystal surface energy, contributing to the uniform nucleation of catalytic seeds with minimal surface diffusion and optimal seed/NW interface plane relationship, thereby resulting in minimal diameter variation and excellent phase purity. When these NWs are fabricated into back-gated field-effect transistors (FETs), the typical Pd-catalyzed GaSb NW exhibits a high peak hole

mobility of $\sim 330 \text{ cm}^2\text{V}^{-1}\text{s}^{-1}$. This mobility value is close to the mobility limit for a 30 nm NW channel diameter with a free carrier concentration of $\sim 10^{18} \text{ cm}^{-3}$,^{32, 33} which indicates the homogeneity of NW phase purity, growth orientation, surface morphology and electrical characteristics. By using the contact printing process, the versatility of these NWs is further illustrated with the configuration of NW parallel-arrayed transistors with impressive device performances, confirming the promise of these high-performance and CMOS-compatible GaSb NWs for a wide range of future high-tech applications.

Results and discussion

Figure 1. CMOS-compatible catalyst of Pd is used for the GaSb NW growth. (a) SEM image of the GaSb NWs grown by 1 nm Pd catalyst film, inset shows the corresponding cross-sectional SEM image. (b) TEM image of the Pd-catalyzed GaSb NWs with a thin diameter of 17 nm, inset is the corresponding SAED pattern indicating the NW grew along the $\langle 111 \rangle$ direction. (c) Growth orientation and (d) Diameter distribution of the NWs grown with 1 nm Pd catalyst film,

respectively. Compared with Au-catalyzed GaSb NWs (grown by 1nm Au film), the Pd-catalyzed NWs show single growth direction and thinner diameter with tighter distribution.

In order to achieve CMOS-compatible GaSb NWs using the sulfur-surfactant assisted CVD technique, various metal catalysts, such as Pd, Ni and Cu, which would form higher temperature eutectoids with Ga, are employed in this work to evaluate the VSS growth mechanism. The NW growths are always maintained at temperatures lower than the catalytic eutectic temperatures to ensure the VSS scheme.^{25, 34-38} A typical scanning electron microscope (SEM) image of the Pd-catalyzed GaSb NWs is shown in Figure 1a, and the SEM images of Ni- and Cu-catalyzed NWs can also be found in Supporting Information Figure S1. Using optimized growth conditions, the Pd catalyst yields GaSb NWs with the most uniform surface morphology and highest growth density among all catalyst choices. In general, Pd-catalyzed NWs are thin and long, up to 35 μm in length (Figure 1a inset). In the case of Au-catalyzed NWs, the length is less than 20 μm for the same growth duration (Supporting Information Figure S2a); therefore, the growth rate of Pd-catalyzed GaSb NWs (~ 100 $\mu\text{m/hr}$) is found much faster as compared with the one of Au-catalyzed NWs (~ 60 $\mu\text{m/hr}$). As shown in the TEM image in Figure 1b, the representative NW displays a smooth surface with a cylindrical-shaped catalytic tip and no tapering. This non-spherical tip is a clear indication and confirms the VSS growth mechanism happened here.^{37, 38} The thin Pd-catalyzed GaSb NW with a diameter of 17 nm grows along the $\langle 111 \rangle$ direction, and Au-catalyzed GaSb NWs in this diameter range would always grow along the $\langle 211 \rangle$ direction,²⁰ hence there is a fundamental difference in the growth mechanism between Pd and Au catalysts (*i.e.* VSS *versus* VLS modes). Indeed, although both Pd- and Au-catalyzed NWs are grown with the same cubic zinc-blende (ZB) crystal structure as demonstrated in the XRD patterns (Figure S2d, JCPDS Card No. 07-0215), the Pd-catalyzed NWs are found to have a dominant growth

orientation along $\langle 111 \rangle$ direction with an overwhelming proportion of 97% (*i.e.* 30 out of 31 NWs) within the diameter range demonstrated (Figure 1c and Supporting Information Figure S3). This finding is in contrast to that of Au-catalyzed NWs, where there are mixed growth orientations which are diameter dependent, similar to previously reported work.^{20, 39-41} Moreover, based on more than 100 NWs observed by TEM (Figure S2b and c), the Pd-catalyzed NWs (grown by 1 nm catalyst film) have smaller diameters and a much narrower distribution (26.9 ± 3.5 nm) compared with those of Au-catalyzed NWs (42.1 ± 11.7 nm) (Figure 1d). This diameter uniformity is more prominent when thicker catalyst films (*i.e.* 2 to 8 nm) are used, and thicker NWs are obtained (Figure S2e). These thinner diameter, narrower distribution and exceptional phase purity GaSb NWs grown using Pd catalysts are suitable for large-scale and high-performance advanced devices.

Apart from the NW diameter distribution and phase purity, it is also important to assess their corresponding electrical properties of Pd-catalyzed NWs for electronic device utilization. Back-gated NW FETs with channel lengths $>2 \mu\text{m}$ are used to ensure that the diffusive transport of carriers, rather than the ballistic or quasi-ballistic transport, are evaluated, and thus the intrinsic transport property of the NWs such as the carrier mobility can be deduced. As shown in the transfer and output characteristics of a representative NW FET device (Figure 2a and b), a typical p-type conducting behavior with a high ON/OFF current ratio up to 10^6 , as well as an ohmic-like contact formation with Ni source/drain electrodes, is confirmed. The corresponding field-effect hole mobility can be calculated using the well-accepted method based on the square law model.⁴² The low-bias (*i.e.* $V_{\text{DS}} = 0.1$ V) transconductance, g_m , can be extracted from the transfer characteristics, $g_m = (dI_{\text{DS}})/(dV_{\text{GS}})|_{V_{\text{DS}}}$ with $\mu = g_m(L^2/C_{\text{OX}})(1/V_{\text{DS}})$, where C_{OX} is the gate capacitance obtained from the finite element analysis software COMSOL using different

NW diameters. The transconductance, g_m , can then be used to calculate the field-effect mobility of individual NWs accurately once the NW channel length and the diameter are known. As given in Figure 2c, the peak hole mobility of the NW is found to be $\sim 330 \text{ cm}^2 \text{V}^{-1} \text{s}^{-1}$, with an estimated hole concentration n_h of $\sim 3.0 \times 10^{18} \text{ cm}^{-3}$. This hole concentration can be assessed using the equation $n_h = Q / (q\pi r^2 L) = (C_{\text{ox}} V_{\text{th}}) / (q\pi r^2 L)$, where r is the NW radius (15 nm) and V_{th} is the threshold voltage ($\sim 7 \text{ V}$). The $I_{\text{ON}}/I_{\text{OFF}}$ current ratio and V_{th} can be directly read off from the log-scale and square-root-scale of the transfer curves in Supporting Information Figure S4. When the NW diameters are greater than 40 nm, the peak hole mobility value of the Pd-catalyzed NWs is similar to that of Au-catalyzed NWs, approaching the theoretical mobility limit under a hole concentration of 10^{18} cm^{-3} . However, when the NW diameters are below 40 nm, the $\langle 111 \rangle$ -oriented Pd-catalyzed thin NWs out-perform their counterparts grown by Au catalysts with mixed $\langle 110 \rangle$ and $\langle 211 \rangle$ orientations. The mobility values of Pd-catalyzed NWs degrade at a much slower rate as the NW diameter decreases because the phase-pure $\langle 111 \rangle$ -oriented NWs would have minimized surface scattering effect along their atomically smooth $\{110\}$ surface.^{43, 44} Because of the negligible diameter dependence of the mobility values, these high-performance, small-diameter and CMOS-compatible GaSb NWs are excellent candidates for device miniaturization.

Figure 2. Electrical properties of the Pd-catalyzed GaSb NWs. (a, b) Transfer and output characteristics of the representative VSS grown GaSb NW configured in the back-gated field-effect transistors. The inset (panel a) gives the corresponding SEM image and device schematic of the device fabricated with Ni source/drain contacts. Based on the SEM image, the NW diameter, d , is found to be 30 nm and the channel length, L , is determined to be 3.7 μm . (c) Hole mobility calculation of the NW device. (d) Peak field-effect hole mobility statistics of both Pd- and Au-catalyzed NWs.

To shed light on the fundamental mechanistic difference facilitated by Pd catalysts as compared with Au catalysts for the GaSb NW synthesis, the crystallinity, the chemical stoichiometry as well as the growth characteristics of as-grown $\langle 111 \rangle$ -oriented Pd-catalyzed GaSb NWs, are studied using high-resolution TEM (HRTEM) and energy-dispersive X-ray spectroscopy (EDS). Figure 3a shows a representative TEM image of an individual NW, displaying a cylindrical catalytic tip with a length of 70 nm and a diameter of 40 nm, this is different from the spherical shape of NWs using Au catalysts grown in VLS mode. Moreover, as

shown in the HRTEM image of catalyst/body region (Figure 3b), both the catalyst tip and NW body grew along the direction of $\langle 111 \rangle$ and formed a cubic crystal structure. The stoichiometric compositions of the catalyst tip and NW body are confirmed by the EDS analysis (Supporting Information Figure S5), that is, the cylindrical tip is Pd_5Ga_4 crystal and NW body is stoichiometric GaSb. Also, the HRTEM images along the axial direction of the NW, from the catalytic tip to the NW root, are shown in Supporting Information Figure S6. The NWs display a smooth surface, an uniform growth direction of $\langle 111 \rangle$ and a diameter of 35 nm throughout the entire NW length ($>10 \mu\text{m}$ long). Furthermore, different cylindrical tip lengths are also observed in the HRTEM images (Figure 3c insets). Although the cylindrical tip length changes from ~ 26 nm to more than 100 nm, they are all oriented in the $\langle 111 \rangle$ direction with the tip having Pd_5Ga_4 stoichiometry as identified by the HRTEM images (Supporting Information Figure S7) and the EDS spectra. Pd_5Ga_4 crystal is known to have a relatively high eutectic temperature of $\sim 940^\circ\text{C}$ ⁴⁵ which is significantly higher than the NW growth temperature of 585°C employed in this work, thus confirming the VSS growth mode and the presence of non-spherical Pd catalysts. In the CVD process, when the NWs are grown without using any sulfur surfactant, the obtained NWs are severely tapered and coated with surface aggregates, but cylindrical shape catalytic tips still existed (Supporting Information Figure S8). This means that the formation effect of sulfur surfactant on the non-spherical tips can be excluded while the surfactants are only employed for the in-situ surface passivation of controlled NW radial growth. In this case, as catalyzed by the $\langle 111 \rangle$ -oriented Pd_5Ga_4 alloy seeds, all obtained GaSb NWs exhibit excellent crystallinity with uniform $\langle 111 \rangle$ growth orientation and without any noticeable amount of crystal defects, such as stacking faults or inversion domains.

Figure 3. Morphology, crystal structure and tip length statistics of representative Pd-catalyzed GaSb NWs. (a) TEM image of NW with cylindrical catalytic tip. (b) HRTEM image of the catalyst/body region of as-shown NW. Insets show the corresponding FFT images. (c) Pd₅Ga₄ catalyst tip length statistics, insets show the TEM images of NWs with tip lengths of 26 nm and 107 nm.

The cylindrical catalyst tips are also investigated in details in order to evaluate the comprehensive growth characteristics and their relationship to the obtained superior NW crystal quality, uniform diameter and phase purity. Firstly, the surface energies of Pd_xGa_y catalyst seeds with different compositions and different growth orientations are simulated, as shown in Figure 4a and b. The <111>-oriented catalyst tip with a Pd:Ga ratio of 5:4 has the lowest surface energy. The surface energy becomes larger for longer tip length, indicating that the thermodynamics

difficulty in forming very long catalyst tips. The surface energies of the solid-state Pd_5Ga_4 crystals (*i.e.* the catalytic seeds) with different crystal planes are listed in Table 1. The $\{110\}$ crystal plane has a much lower surface energy than those of $\{100\}$ and $\{111\}$, since the $\{110\}$ plane has the highest atomic density and thus the lowest surface energy due to the body-centered cubic structure of Pd_5Ga_4 crystal. As a result, all the observed Pd_5Ga_4 catalytic seeds are oriented in the $\langle 111 \rangle$ direction with most having a tip length of 60 nm, as shown in Figure 3 and Figure S7, since the $\langle 111 \rangle$ -oriented tips consist of all $\{110\}$ surface planes²⁰ with minimal crystal energies and are thermodynamically most favorable.

Table 1. Comparison of surface energy of Pd_5Ga_4 catalyst seed among different crystal planes

Pd_5Ga_4	Surface Energy (keV)
$\{001\}$	1.5322
$\{110\}$	1.1492
$\{111\}$	1.2582

It is worth to point out that the surface energy of $\text{Pd}_5\text{Ga}_4\langle 110 \rangle$ is not that much higher than the one of $\text{Pd}_5\text{Ga}_4\langle 111 \rangle$. However, in the epitaxial NW growth, the lattice mismatch between catalyst tips and NW bodies is also an important consideration which cannot be neglected. Once the $\langle 111 \rangle$ -oriented Pd_5Ga_4 seeds are obtained, the $\langle 111 \rangle$ -oriented GaSb NWs could be anticipated to form due to the epitaxial relationship.⁴⁶ As depicted in the catalyst seed/NW interface orientation relationship of the cubic $\text{Pd}_5\text{Ga}_4\{111\}|\text{cubic GaSb}\{111\}$ in Figure 4c, the atomic arrangement of Ga in the $\{111\}$ plane of both Pd_5Ga_4 and GaSb crystals exhibit excellent epitaxial characteristics with the smallest in-plane lattice mismatch of 2.7% among all the

possible interface configurations. As shown in Supporting Information Figure S9, the in-plane lattice mismatch between Pd_5Ga_4 $\{110\}$ and GaSb $\{111\}$ is around 16.1 % while the mismatch between Pd_5Ga_4 $\{110\}$ and GaSb $\{110\}$ is about 27.4 %. As a result, these relatively large lattice mismatches would make the $\langle 111 \rangle$ -oriented and $\langle 110 \rangle$ -orientated GaSb NWs difficult to be epitaxially grown from $\text{Pd}_5\text{Ga}_4\langle 110 \rangle$ tips. The lattice mismatch is calculated using the analytic equation $[(a_{\text{NW}} - a_{\text{Cat}}) / a_{\text{NW}} \times 100\%]$, where a_{NW} and a_{Cat} are the lattice constants of the NW and catalyst seeds involved, respectively. The superior lattice matching would enable the obtained GaSb NWs having an enhanced crystal quality and uniform growth orientation, minimizing the probability of NWs grown along other directions such as $\langle 110 \rangle$ and $\langle 211 \rangle$ with larger lattice mismatch and higher interfacial energy, as shown in Figure 4d and e.

Figure 4. Simulation of catalyst tip surface energy and epitaxy growth model of Pd-catalyzed GaSb NWs. (a) Simulated catalyst tip surface energy as a function of composition. (b) Simulated Pd_5Ga_4 catalyst tip surface energy as a function of growth orientation. (c), (d), (e) Schematic view of the in-plane orientation of catalytic seed/NW interface of $\text{Pd}_5\text{Ga}_4 \{111\} | \text{GaSb} \{111\}$, $\text{Pd}_5\text{Ga}_4 \{111\} | \text{GaSb} \{110\}$ and $\text{Pd}_5\text{Ga}_4 \{111\} | \text{GaSb} \{211\}$.

Although WZ GaSb {0001} planes have the same atomic alignment and the same lattice matching with Pd₅Ga₄ {111} planes and ZB GaSb {111} planes, no wurtzite (WZ) <0001>-oriented NW has been observed in this study. It is because as shown in the atomic model of the seed/NW interface plane orientation in Figure 5a and b, the difference between the atomic layer stacking sequence in ZB (*i.e.* “abcabc”) and WZ (*i.e.* “ababab”) would make the nucleation of WZ GaSb NWs from the Pd₅Ga₄ seeds not possible. By examining the closely-packed plane along both the ZB and the WZ NW growth directions, the first two atomic layer stacking of the ZB and the WZ NW structures are the same, namely “ab”; however, the third atomic layer of the WZ NW structure, namely “a”, cannot establish any consistent or stable crystal registration with the catalyst seed located above. Thus, the <111>-oriented Pd₅Ga₄ catalytic tip would catalyze predominantly the ZB GaSb NWs growing along the <111> direction with small and uniform diameter under the VSS growth mode, as well as improved crystal quality attributed to the superior in-plane lattice matching at the seed/NW interface.

After investigating the VSS growth mechanism of Pd-catalyzed GaSb NWs, the detailed comparison of the NW growth characteristic between VSS and VLS growth mode is shown in Figure 5c. In a typical CVD growth process, the catalyst film will first be annealed at the NW growth temperature, resulting in the formation of nanoscale metal nanoparticles. In the case of NWs grown with Au catalysts, the eutectic temperature of Au-Ga (<500 °C)⁴⁷ is lower than the required growth temperature of 545 °C, so the GaSb NWs could be anticipated to grow *via* the VLS mode, which is confirmed and indicated by the TEM observation of typical spherical catalytic tip. Based on the Gibbs–Thomson effect, these mobile liquid Au_xGa_y nanoparticles would further be aggregated and supersaturated with different precursor compositions, leading to the growth of NWs with different orientations that are determined mainly by the surface

energies.³⁹ In the case of VSS growth mode, the immobile solid catalyst tip is found to grow along close-packed plane with the lowest surface energy, and cylindrical shapes with different lengths are formed. The equivalent spherical diameter could be calculated if the cylinders are transformed into spheres, and after the transformation the Pd catalytic seed would have a similar equivalent diameter distribution as that of Au catalyst (Supporting Information Figure S10). This similar seed diameter distribution is also confirmed by the statistics of the AFM image of the annealed 1 nm thick Pd and Au catalyst, as shown in Supporting Information Figure S11. The spherical Pd catalytic seed is first transformed into cylinder at the initial stage of NW nucleation during the VSS growth mode, leaving only similar circular bottom for the thinner and uniform diameter growth of NWs. Hence, since the seed volume varies in length instead of diameter, it would become easier to control the NW diameter during the nucleation and the subsequent growth. The crystalline seed would dictate the epitaxial growth orientation of NWs *via* the established seed/NW interface plane relationship, which is independent of the NW diameter and growth condition. As a result, compared with a typical VLS-based NW growth mode, the VSS scheme has demonstrated significant advantages in attaining NWs with narrower diameter distributions as well as well-controlled crystal phase and growth orientation.

Figure 5. Proposed growth mechanism of Pd-catalyzed GaSb NWs. (a) Schematic view of the in-plane orientation of the catalytic seed/NW interface relationship of Pd₅Ga₄{111}|GaSb{111}. (b) Approximate atomic model demonstrating the epitaxial relationship between $\langle 111 \rangle$ -oriented Pd₅Ga₄ seed and $\langle 111 \rangle$ -oriented GaSb NW. (c) NW growth schematic showing the VLS and VSS growth modes catalyzed by the Au and Pd seeds, respectively.

To further understand the NW growth mechanism, the feasibility of large-scale device integration of the as-prepared CMOS-compatible GaSb NWs is demonstrated with the fabrication of NW parallel-arrayed FETs *via* the well-established NW contact printing technique.⁴⁸ The SEM image and device schematic of a typical Pd-catalyzed GaSb NW arrayed FET are shown in Figure 6a. The device channel length and width are designed to be 2.5 μm and 200 μm , respectively. Based on the SEM observation, there are about 400 NWs bridging the source/drain electrodes. As shown in the transfer and output characteristics in Figure 6b and c, the device has a current density of 1.35 $\mu\text{A}/\mu\text{m}$ under $V_{\text{DS}}=0.4\text{V}$ and $V_{\text{GS}}=-10\text{V}$ with a p-type conducting behavior and an ohmic-like contact characteristic. The corresponding field-effect mobility against the gate voltage, V_{GS} , is also evaluated and presented in Figure 6d. The mobility

is calculated based on the standard square law model similar to the operation of a single NW device. The capacitance is estimated using an upper-bound model that multiplies the capacitance of each nanowire with the number of nanowires in the channel. The calculated peak hole mobility is found to be $\sim 65 \text{ cm}^2 \text{ V}^{-1} \text{ s}^{-1}$, which is higher than that of state-of-the-art p-type thin-film transistors (TFTs) as compiled in Table 2.⁴⁹⁻⁵² Compared with single GaSb NW device (Figure 2), the average current density of each NW gets degraded in this arrayed configuration, which is probably caused by the ineffective NW-to-NW gate coupling owing to the misalignment and fragmented wires in the device channel leading to higher parasitic capacitances. These would then increase the total capacitance and reduce the output current. In the future, the performance of the NW arrayed device can be further enhanced through optimization of the NW print density, alignment, channel length scaling, as well as incorporation of high-k dielectrics for the top-gated configuration.

Figure 6. Electrical characterization of typical Pd-catalyzed GaSb NW parallel-arrayed FET. (a) (Top) SEM and (bottom) schematic of a back-gated Pd-GaSb NW arrayed FET. (b), (c) Transfer

and output characteristics of the device. (d) Corresponding field-effect hole mobility of the device as a function of the back-gate bias.

Table 2. Hole mobility comparison of p-type thin films.

Materials	Hole Mobility ($\text{cm}^2\text{V}^{-1}\text{s}^{-1}$)	Reference
GaSb parallel NW arrays	65	This work
Carbon nanotube parallel arrays	4.27±1.62	49
NiO_x thin film	25	50
Cu₂O thin film	3.9	51
SnO_x thin film	4.8	52

Conclusions

In conclusion, VSS growth of high-performance, CMOS-compatible and phase-pure <111>-oriented GaSb NWs is successfully demonstrated with the use of Pd catalysts. Experimental results and theoretical analyses show that this particular scheme can not only narrow the diameter distribution and make perfect the phase purity of obtained NWs, but also maintain their superior field-effect hole mobilities when fabricated into back-gated FETs, being insensitive to NW diameter variations. Compared with conventional Au-catalyzed VLS NWs, all these improvements can be attributed to the existence of immobile solid Pd₅Ga₄ catalytic seed for minimal surface diffusion and optimal in-plane orientation at the seed/NW interface during the NW nucleation. By exploiting a contact printing process, the versatility of these NWs can be further illustrated with the configuration of NW parallel-arrayed transistors having impressive

device performances, thus illustrating and confirming the promise of these high-performance and CMOS-compatible GaSb NWs for a wide range of device applications.

Methods

CMOS-compatible GaSb NW synthesis. CMOS-compatible metal catalysts of Pd, Ni and Cu are used for the growth of GaSb NWs by employing the surfactant-assisted solid-source CVD method as reported in our previous works.^{19, 20} In brief, the solid powders of GaSb (99.999% purity) and sulfur (99.99% purity) are used as the source materials. A dual-zone horizontal tube furnace, with one zone for the solid source (upstream) and another zone for the growth substrate (downstream), is used as the reactor while metal films of Pd, Ni and Cu are utilized as catalysts for the NW synthesis. Initially, catalyst films are pre-deposited onto Si/SiO₂ substrates (50 nm thick thermally grown), and the substrates are placed in the middle of the downstream zone. The solid sources, GaSb and sulfur powders, are next placed within two separate boron nitride crucibles, with distances of 15 cm and 9 cm away from the growth substrate, respectively. It is noted that the sulfur powder is actually placed in the middle of the two zones. During growth, the source and substrate are heated to the pre-set temperatures, accordingly. Hydrogen (99.9995% purity) is used as the carrier gas to transport the thermally vaporized materials to the downstream. Prior to heating, the pressure of the quartz tube is pumped down to 3×10^{-3} Torr and then purged with H₂ for 0.5 h. After growth, heating of the source and substrate are stopped together and the substrate is cooled down to room temperature under the hydrogen flow. At the same time, in order to confirm the VSS growth mechanism of Pd-catalyzed GaSb NWs, it is necessary to exclude any possible effect of sulfur surfactant on the NW growth mechanism; therefore, a

control experiment with the GaSb NWs grown without any sulfur surfactant is also performed. The optimal growth conditions of GaSb NWs are given in Table S1.

Material characterization. Surface morphologies of the grown NWs are examined using scanning electron microscope (SEM, FEI Company, Oregon, USA/Philips XL30, Philips Electronics, Amsterdam, Netherlands) and transmission electron microscope (TEM, Philips CM-20). Crystal structures are determined by collecting XRD pattern on a Philips powder diffractometer using Cu K α radiation ($\lambda = 1.5406 \text{ \AA}$) and imaging with a high resolution TEM (HRTEM, JEOL 2100F, JEOL Co., Ltd., Tokyo, Japan). Elemental mappings are performed using an energy dispersive X-ray (EDS) detector attached to a JEOL 2100F, and the chemical composition of the obtained NWs is measured. For the TEM studies, the NWs are first suspended in ethanol solution by ultrasonication and then drop-casted onto the grid for the corresponding characterization.

Single Nanowire FET fabrication and electrical measurements. NW FETs are fabricated by drop-casting the NW suspension onto highly doped p-type Si substrates with a 50 nm thick thermally grown gate oxide layer. Photolithography is employed to define the source/drain regions, and the 60-nm thick Ni film is thermally deposited as the contact electrodes followed by a lift-off process. The electrical performance of the fabricated back-gated FETs is then characterized with a standard electrical probe station and an Agilent 4155C semiconductor analyzer (Agilent Technologies, California, USA).

NW parallel-arrayed device fabrication and characterization. NW parallel arrays are printed on the same type of degenerately doped p-type SiO₂/Si substrates using the well-established contact printing method.⁴⁸ These printed substrates are then spin-coated with LOR and AZ5206 photoresists, which were patterned using standard UV lithography and development, followed by

Ni electrode deposition and lift-off. Electrical performance of the fabricated NW-array FETs is next characterized with a standard electrical probe station and Agilent 4155C semiconductor analyzer.

Simulation methods. The structural optimization of the Pd_xGa_y bulk alloys is obtained by calculating their corresponding total energies. In order to simplify the calculation, a 64-atom supercell with a diamond structure is considered. The Pd and Ga atoms randomly occupy the supercell sites until the composition matches with the experimental result. Using the Vienna *ab initio* simulation package,^{53, 54} the geometrical structure of all the supercells is optimized to attain the most stable structure. The surface structure, consisting of a 10 layer slab and a 15 Å thick vacuum layer, is modeled. The two layers on the bottom are fixed in order to mimic the bulk, and the surface reconstructions are carried out until all forces on the free ions converging to 0.01 eV/Å. The Monkhorst-Pack *k*-point meshes of $4 \times 4 \times 1$, $4 \times 4 \times 1$, $4 \times 5 \times 1$ and $4 \times 5 \times 1$ are used for (001), (111), (110) and (211) surfaces, which have been tested for the convergence. The kinetic energy cutoff for the plane-wave basis set is 500 eV. The formula for calculating surface energies of different surfaces can be written as:⁵⁵

$$\gamma_s = (E_s - E_b) / 2A$$

where E_s is the energy of the crystal with two surfaces, E_b is the energy of the bulk with the same amount of the atoms, and A is the area of the surface. The total surface free energy can be expressed in terms of the edge surface free energy of GaPd nanowires such that $E = \gamma_s S_s$, where γ_s and S_s are the surface energy and the surface area of different surfaces.

ASSOCIATED CONTENT

Supporting Information. Morphology of the as-prepared CMOS compatible GaSb NWs grown by 1 nm (nominal thickness) catalyst films of Ni and Cu; Structural characterization, diameter distributions and morphology of as-prepared GaSb NWs; Growth orientations of the Pd-catalyzed GaSb NWs; Transfer characteristics of a typical device fabricated with the Pd-catalyzed GaSb NW channel; Characterization of the chemical composition, morphology and crystal structure of a representative Pd-catalyzed GaSb NW; Electron microscopic characterization of the Pd-catalyzed GaSb NWs with different tip lengths; Epitaxial growth model of Pd-catalyzed GaSb NWs; Morphology of the Pd-catalyzed GaSb NWs prepared without using any surfactant in the growth process; Statistics and compilation of the Pd₅Ga₄ catalyst tips; AFM characterization of the annealed catalyst seeds; the optimal growth conditions of GaSb NWs performed in this study.

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Notes

The authors declare no competing financial interest.

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