Guided Growth of Horizontal p-Type ZnTe Nanowires

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Supporting Information

ABSTRACT: A major challenge toward large-scale integration of nanowires is the control over their alignment and position. A possible solution to this challenge is the guided growth process, which enables the synthesis of well-aligned horizontal nanowires that grow according to specific epitaxial or graphoepitaxial relations with the substrate. However, the guided growth of horizontal nanowires was demonstrated for a limited number of materials, most of which exhibit unintentional n-type behavior. Here we demonstrate the vapor−liquid−solid growth of guided horizontal ZnTe nanowires and nanowalls displaying p-type behavior on four different planes of sapphire. The growth directions of the nanowires are determined by epitaxial relations between the nanowires and the substrate or by a graphoepitaxial effect that guides their growth along nanogrooves or nanosteps along the surface. We characterized the crystallographic orientations and elemental composition of the nanowires using transmission electron microscopy and photoluminescence. The optoelectronic and electronic properties of the nanowires were studied by fabricating photodetectors and top-gate thin film transistors. These measurements showed that the guided ZnTe nanowires are p-type semiconductors and are photoconductive in the visible range.

The guided growth of horizontal p-type nanowires opens up the possibility of parallel nanowire integration into functional systems with a variety of potential applications not available by other means.

INTRODUCTION

Semiconducting nanowires (NWs) with controlled size, structure, and orientation are promising building blocks for nanotechnology, and can be applied in the fabrication of a wide range of nanodevices, such as diodes, field-effect transistors (FETs), and memory elements, photodetectors (PDs), light emitting diodes (LEDs) and nanolasers, photovoltaic cells (PVs),† thermoelectric (TE) devices,‡ and chemical and biological sensors. However, one major challenge toward large-scale integration is the insufficient control over the alignment and position of the nanowires. To fulfill the technological advantages of nanowires and successfully integrate them into complex systems, a precise control over their dimensions, orientation, structure, and electrical properties must be achieved.⁷

Among several methods for nanowire growth, the vapor−liquid−solid (VLS) process, which was first presented in 1964 by Wagner and Ellis, is one of the most commonly used. However, nanowires grown by this method are usually perpendicular to the substrate or protruding from it at an angle; and therefore integration into planar devices requires two additional steps: “harvesting” the as-grown nanowires, and rearranging them horizontally on the desired substrate, using various methods, including Langmuir−Blodgett compression, liquid flow, and electric fields. However, the alignment and positioning of the nanowires achieved by these methods are usually not perfect, and the nanowires tend to break during the rearranging process, so that the positions of the two ends of each nanowire are not well controlled.

A promising alternative to postgrowth assembly is the guided growth approach, which enables the production of well-aligned horizontal nanowires that grow according to specific epitaxial or graphoepitaxial relations with the substrate.¹⁴ The origin of the nanowires can be determined by the controlled deposition of a catalyst, thus enabling massively parallel self-integration of nanowires into circuits.¹⁵ This guided growth process offers a high level of control over the position, alignment, direction, polarity, and crystallographic orientation of the nanowires. Such horizontal growth of nanowires has been demonstrated with a growing number of material including ZnO,¹⁶ In₃O₇,¹⁷ and GaAs.¹⁸ Recently, our group has mastered this novel process and grown horizontal GaN, ZnO, and ZnSe nanowires on different substrates including sapphire (α-Al₂O₃), quartz (SiO₂), silicon carbide (SiC), and spinel (MgAl₂O₄) substrates. Complex devices such as complementary logic circuits, LEDs, and photovoltaic cells require combined integration of n-type and p-type semiconductors. However, most of the horizontal nanowires reported so far presented n-type electronic behavior. Therefore, in order to exploit the full potential of the guided growth process, it is critical to extend the guided growth approach to nanowires with p-type behavior. Most II–VI semiconductors exhibit...
unintentional n-type behavior and p-doping is difficult to achieve in a reliable and reproducible manner. One notable exception among II–VI semiconductors is zinc telluride (ZnTe), which has been reported to display unintentional p-type behavior. Therefore, in this study we focused on demonstrating and characterizing the guided growth of ZnTe nanowires. We have chosen sapphire as a substrate for the variety of the flat and faceted surfaces that it can present when cut at different crystallographic planes.

ZnTe is an important II–VI semiconductor with a direct bandgap of 2.26 eV at room temperature, a Bohr exciton radius of 6.2 nm, and a high optical absorption coefficient in the visible range. ZnTe has a zincblende (ZB) crystal structure in bulk while the wurtzite (WZ) crystal phase can appear in nanostructures. Furthermore, with oxygen doping (ZnTe:O) ZnTe can possess intermediate band (IB) structure, which makes it a promising material for solar cells due to a theoretical efficiency as high as 63%. Potential applications of ZnTe nanowires are mostly optoelectronic devices such as green LEDs, photovoltaic cells, visible spectrum photodetectors, X-ray detectors, and THz emitters and detectors.

The p-type behavior of ZnTe has been attributed to the different vapor pressures of Zn and Te, which lead to stoichiometric deviation during the synthesis. As a result, the grown ZnTe nanowires tend to contain less Zn than Te. This stoichiometric deviation is equivalent to Zn vacancies, leading to a p-type behavior of the grown ZnTe nanowires. The electrical properties of ZnTe nanowires can be further enhanced by appropriate doping or postgrowth treatment. P-doping can be achieved with different dopants and methods for instance, replacing H2 with NH3 or adding solid Sb powder to the furnace during the synthesis will result in N and Sb p-doping, respectively. Also, postsynthesis immersion in Cu(NO3)2 solution result in Cu p-doping. Vacuum annealing of as grown pristine nanowires was shown to reduce their resistivity significantly, and to enhance their carrier concentration and mobility, producing electrical properties comparable to those obtained by p-doping. These attributes make ZnTe a promising candidate for the fabrication of p-type nanowires and their integration into functional systems.

Despite this interest, relatively few studies of ZnTe nanowires have been reported compared with other II–VI semiconductors. Polytypism, twinning, and polarity considerations in ZnTe nanostructures have been the focus for some structural studies, but works regarding doping of ZnTe nanowires are rare and n-type doping of ZnTe nanowires has not yet been reported. Therefore, the controlled doping of ZnTe nanowires remains a challenge. Although ZnTe nanowires are promising building blocks for many applications, practical use is not yet achieved due to some major obstacles, including the need for uniform, controllable, and reproducible doping processes; and the insufficient control over the alignment and positioning of the nanowires, required for their integration into functional electrical devices.

In this article, we demonstrate the VLS guided growth of horizontal p-type ZnTe nanowires on four different substrates including flat and faceted planes of sapphire: flat planes M (1010) and C (0001), and faceted planes annealed M (1010) and annealed miscut C (0001). The nanowires also show photocurrent under visible illumination. The guided ZnTe nanowires display three modes of guided growth: (i) epitaxial growth along specific lattice directions, (ii) graphoepitaxial growth along nanosteps, and (iii) graphoepitaxial growth along nanogrooves (Figure 1). This is the first demonstration of guided growth of horizontal ZnTe nanowires, thus expanding the generality and flexibility of the guided growth phenomenon. We characterized the crystallographic orientations of the nanowires by preparing thin cross-sectional lamellas using focused-ion beam (FIB) for high-resolution transmission electron microscope (HRTEM) imaging. The best alignment of the nanowires was obtained on annealed M-plane (1010) sapphire, but these nanowires grew with no preferred crystallographic orientation while presenting mainly ZB crystal structure along with few WZ exceptions. On nonannealed M-plane the ZB nanowires grew along the [110] axis, and on C-plane and annealed miscut C-plane (0001) sapphire the nanowalls and nanowires grew along the [112] axis. This indicates that the crystallographic orientation of the nanowires can be controlled by the selection of the substrate orientation. By fabricating photodetectors and top gate thin film transistors (TFTs), we have shown that the guided ZnTe nanowires grown on annealed M-plane are p-type, and are photo-conductive optically active in the visible spectrum. Although the uniformity, morphology, roughness, and guidance could be further optimized; this proof of concept may pave the way for the massively parallel integration of p-type and n-type nanowires into functional systems.
MATERIALS AND METHODS

Substrate Preparation. Sapphire ($\alpha$-Al$_2$O$_3$) wafers with five different orientations were used in this research (Roditi International, Inc.). The different planes that were used as substrates for guided growth of ZnTe nanowires are R-plane ($\{1102\}$), M-plane ($\{10\bar{1}0\}$), C-plane ($\{0001\}$), and miscut C-plane (C-plane wafers with a few degrees miscut). The M-plane and the miscut C-plane wafers were annealed at 1400–1600 °C in air for 10 h using a high-temperature tube furnace. All substrates were sonicated for 10 min in acetone and then rinsed minutes under the same gases the furnace was turned off and was then taken out.

Catalyst Patterning and Deposition. To achieve a long order control over the position of the nanowires, the areas for the catalyst deposition were defined using a standard photolithography process (MA/Ba6 Karl-Suss contact mask aligner) with negative photoresist (NR-9 1000PY) and suitable masks. After patterning and developing (RD-6), thin films (4 Å) of Au (Holland Moran, 99.99%) were deposited using an electron-beam physical vapor deposition system (e-beam PVD, Telemark), followed by lift-off in acetone and cleaning procedure: rinse in acetone and IPA and immediately blow-dried in N$_2$. A sample was positioned 33 cm downstream to the crucible. As a last step prior to the synthesis process, the substrates were heated to 550 °C for 7 min. This process burns organic residues and dewets the Au films, turning them into clusters of nanoparticles (NPs) that serve as the catalyst during the VLS growth of the nanowires.

Nanowires Synthesis. The nanowires were grown in a home-built hot-wall chemical vapor deposition (CVD) system with fast heating capability. ZnTe powder (99.99%, Sigma-Aldrich) served as the ZnTe nanowires source. H$_2$ and N$_2$ served as carrier gases (all gases 99.999%, Gordon Gas). In a typical synthesis, a sample (sapphire wafer patterned with Au films) was placed on a fused-silica carrier plate and loaded onto a 25 mm diameter fused-silica tube, which serves as the reactor. A fused-silica crucible containing the ZnTe powder (0.2–0.25 g) was positioned 33 cm from the tube entrance, while the sample was positioned 33–42 cm downstream to the crucible. The tube was inserted into a 2-zones home-modified furnace (two Lindberg/Blue M one-Zone Tube Furnaces that were connected), in a way that the ZnTe crucible was placed at the middle of the first zone and the sample was placed at the middle of the second zone. The system was then sealed and purged of O$_2$ by six cycles of pumping to 5 mbar and purging with N$_2$ and H$_2$ at elevated temperature. After the first three cycles, the furnace was moved such that the crucible was out of the heating zone, and the sample was at the upstream side of the first heating zone. Once the tube was purged, N$_2$ gas (190–480 sccm) and H$_2$ gas (10–20 sccm) were streamed into the tube, and a pressure of 300–400 mbar was maintained. Once the desired temperatures were achieved (800–950 °C for the first zone and 570–640 °C for the second zone), the furnace was moved to its original position, and remained there for the entire synthesis time (25–35 min). At the end of the synthesis, the furnace was turned off; the sample was cooled for a few minutes under the same gases flow and was then taken out.

Microscopic Characterization. Initial evaluation of the grown nanowires was done under an optical microscope (Olympus BX-51). Detailed examination of the grown nanowires and of the fabricated devices was done using field-emission scanning electron microscope (SEM, LEO Supra 55 VP Zeiss) at a low working voltage of 3–4 kV. Thin cross-section lamellas were prepared using focused-ion beam (FIB, FEI Helios DualBeam). High-resolution TEM (HRTEM, FEI Tecnai F-30) was used at a working voltage of 300 kV to acquire detailed information regarding the dimensions, morphology, and crystal structure of the nanowires. To determine the crystallographic orientation of the guided nanowires, and calculate their epitaxial relations with the sapphire substrates, the HRTEM images were analyzed using Fourier transform (FFT) from selected areas in the nanowires cross-section and according to crystallographic tables for bulk ZnTe. The longitudinal and transversal mismatch between the nanowires and the substrate were calculated using eq 1.

$$\text{mismatch (°)} = \frac{\text{la}(\text{Al}_2\text{O}_3) - \text{a}(\text{ZnTe})}{\text{a}(\text{Al}_2\text{O}_3)}$$

An energy-dispersive X-ray spectroscopy (EDS) detector installed within a transmission electron microscope (TEM, Philips Tecnai F-20) was used at a working voltage of 300 kV for elemental characterization of the nanowires. Energy-filtered TEM (EFTEM, Philips Tecnai F-20) was used for elemental mapping.

Optical Characterization. Photoluminescence (PL) spectra measurements of as-grown ZnTe nanowires on annealed M-plane ($\{10\bar{1}0\}$), were performed at room temperature using argon-ion laser with 2 lines, 457 and 514 nm (Renishaw inVia Reflex Spectrometer System), as the excitation source. The excitation power was 0.1–50 mW. The measurements were taken with ×50 objective and laser spot size of 2 μm. Photoluminescence spectra were measured from 470 to 740 nm for (1) single guided nanowires and (2) several guided nanowires.

Electrical Characterization. Thin film transistors (TFTs) were fabricated with a top gate on an array of guided ZnTe nanowires. The source–drain electrodes (5–10 μm gap) were defined using a standard photolithography process (MA/Ba6 Karl-Suss contact mask aligner) with negative photoresist (NR-9 1000PY) and suitable masks. After patterning and developing (RD-6), 130 nm of Ni or 25 nm of Ti and 130 nm of Au were deposited using an e-beam PVD system (Telemark), followed by lift-off in acetone and cleaning procedure: rinse in acetone and IPA, and blow-dry in N$_2$. The thermal annealing process was done in Ar atmosphere at 280 °C for 30 min in order to lower the contact resistance of the nanowires–electrodes junction. For gate dielectric, 50 nm of Al$_2$O$_3$ was deposited by low temperature (100 °C) atomic layer deposition system (ALD, Fiji F200, Cambridge NanoTech), using trimethylaluminiun and water as precursors. The top-gate electrodes were defined using a standard photolithography process (MA/Ba6 Karl-Suss contact mask aligner) with negative photoresist (NR-9 1000PY) and suitable masks. After patterning and developing (RD-6), 50 nm of Cr and 150 nm of Au were deposited using an e-beam PVD system (Telemark), followed by lift-off in acetone and a cleaning procedure: rinse in acetone and IPA and blow-dry in N$_2$. To remove the dielectric layer that masks the source–drain electrodes, a standard photolithography process with a suitable mask followed by 1 min HF etching was conducted.

Two-terminal electrical measurements were performed by applying a source-drain DC bias and recording the $I–V$ curves for different gate voltages using a homemade probe station. To amplify the electrical current, the devices were also measured under a 532 nm laser excitation. The charge carrier mobility, $\mu$,
was extracted from the transconductance, \( g_m \), which is defined as the slope of the \( I_{sd} - V_g \) curve in the linear region, and is given by eq 2, where \( I_{sd} \) and \( V_{sd} \) are the source–drain current and voltage respectively, \( V_g \) is the gate voltage, \( L \) is the nanowires channel length, and \( C \) is the capacitance. The latter was calculated using coaxial capacitor approximation, and is given by eq 3, where \( \varepsilon_0 \) is the vacuum permittivity, \( \varepsilon \) is the relative permittivity, \( h \) is the dielectric layer thickness, and \( r \) is the ZnTe nanowire radius, which is assumed to equal half of the nanowires average width. The hole concentration, \( n_h \), is calculated from the threshold voltage, \( V_{th} \), using eq 4, where \( e \) is the electron charge.

\[
g_m = \left. \frac{dI_{sd}}{dV_g} \right|_{V_{sd}=\text{const}} = \frac{\mu C}{L^2} V_{sd}
\]

\[
C \approx \frac{2\pi\varepsilon_0 L}{\ln[(h + r)/r]}
\]

\[
n_h \approx \frac{CV_{th}}{e\varepsilon \pi r^2 L}
\]

**Optoelectronic Characterization.** ZnTe nanowire-based photodetectors were fabricated by defining source and drain electrodes separated by 5–10 \( \mu \)m from each other on nanowire arrays. The areas for the electrode deposition were defined using a standard photolithography process (MA/BA6 Karl-Suss contact mask aligner) with negative photoresist (NR-9 1000PY) and suitable masks. After patterning and developing (RD-6), 25 nm of Ti and 130 nm of Au were deposited using an e-beam PVD system (Telemark), followed by lift-off in acetone and cleaning procedure: rinse in acetone and IPA and blow-dry in \( \text{N}_2 \). The measurements were performed at room conditions.
temperature using 532 nm laser as the illumination source. Two-terminal electrical measurements were performed by applying a source−drain DC bias and recording: (1) $I-V$ curves for different excitation power and (2) $I-t$ curves for a fixed excitation power while using an optical chopper. The on/off ratio is defined as the current under illumination divided by the current under dark conditions, when both are measured under a maximal source−drain bias.

**RESULTS AND DISCUSSION**

Horizontal ZnTe nanowires were grown by the VLS mechanism, using a home-built CVD system, consisting of a fused silica tube that was placed inside a 2-zone tube furnace. ZnTe powder served as the source material, $N_2$ and $H_2$ were used as carrier gases, and the Au patterned samples (photolithography and e-beam physical vapor deposition of Au thin films) were held downstream from the ZnTe powder on a fused-silica carrier plate. The Au dewets upon heating, forming nanoparticles, which act as VLS catalyst.

The ZnTe nanowire growth was carried out on five different sapphire planes: the flat planes R (1102), M (1010), and C (0001), and the faceted planes annealed M (1010) and annealed miscut C (0001) (2° toward [1102]). For each substrate the growth conditions were optimized in order to achieve guided growth. Out of these five substrates, guided growth of well-aligned nanowires was achieved on the annealed M-plane sapphire (Figure 2A−F). On the C-plane sapphire, guided growth of well-aligned nanowalls was achieved (Figure 5A−G). Some degree of alignment of the nanowires was observed on M-plane sapphire (Figure 4A−E) and annealed miscut C-plane sapphire (Figure 6A−D). Horizontal growth of ZnTe was demonstrated on R-plane sapphire, but these nanowires were not guided (Figure 7A). Table 1 summarizes the growth directions and crystallographic orientations of the guided nanowires/nanowalls grown on the different substrates. Comprehensive data regarding the crystallographic orientations and mismatch of the ZnTe nanowires grown on the different substrates can be found in Tables 2 and 3 in the Supporting Information.

It was observed that syntheses with a high yield of horizontal nanowires had a low yield of vertical nanowires, and vice versa. This indicates that the horizontal and vertical growth of nanowires is thermodynamically or kinetically favored under different sets of synthesis conditions. The different parameters (i.e., source/sample, temperature/position, carrier gases flow rates, ambient pressure, etc.) can also have significant impact on the synthesis outcome in terms of nanowire morphology and alignment. It is therefore reasonable to assume that by optimizing the synthesis conditions, better alignment of the ZnTe nanowires could be achieved on M-plane and annealed miscut C-plane sapphire (planes that demonstrated limited alignment), and that a higher yield of fine-structured nanowires
could be achieved on many of the presented substrates. In the next paragraphs we describe the guided growth of ZnTe nanowires on the different substrates.

**Guided Growth of ZnTe Nanowires on Annealed M-Plane (1010) Sapphire.** M-plane (1010) sapphire is an unstable plane, that is, with a relatively high surface energy. When M-plane is annealed in air at high temperatures (1400–1600 °C) for a number of hours, the surface undergoes restructuring to present the more stable S (101̅1) and R (11̅02) facets (Figure 1B). As a result, the substrate surface becomes faceted with well-structured and periodic V-shaped nanogrooves in the ±[1̅2̅1̅0] directions. Upon different annealing conditions, the pitch of the nanogrooves can vary between 40 and 200 nm. These periodically faceted nanogrooves guide the growth of the horizontal ZnTe nanowires by a graphoepitaxial effect (Figure 2A–D). As seen in previous works done by our group, the interaction of nanowires with nanometric geometrical features on the surface is strong, and the graphoepitaxial guidance dominates over epitaxial guidance. This explains the observation that nanowires grown on annealed M-plane sapphire show a much higher yield of guided nanowires in comparison to nanowires grown on M-plane sapphire (as will be discussed in the next section). The most successful syntheses resulted in well-aligned nanowires, but many of these nanowires had a rough shape (nonuniform thickness), usually with a bulky part near their origin. These nanowires also demonstrated a wide variation in thickness and length within a single sample (Figure 2A,B). The bulkiness of the ZnTe nanowires grown on annealed M-plane sapphire may be attributed to a nonuniform radial vapor–solid growth in addition to the axial VLS growth. As the nanowires grow, the catalyst is being pushed away from its original position, and parts of the nanowires that precipitated earlier, that is, closer to the nanowires origin, have more time to undergo radial vapor–solid growth. Therefore, nanowires tend to be bulky near their origin and wire-like near their tip (the metal droplet). To determine the crystallographic structure and orientation of the guided nanowires and calculate their epitaxial relation with the sapphire substrate, three thin cross-section lamellas (50–100 nm thick) with a total number of 10 nanowires were prepared using a focused ion beam (FIB), and were observed under HRTEM. The HRTEM images were analyzed using FFT from selected areas in the nanowires cross-section. The longitudinal and transversal minimal mismatch between the ZnTe nanowires and different planes of sapphire are summarized in Table S2, while Table 1 summarizes the crystallographic structure and orientations of the nanowires that were examined by HRTEM. The ZnTe nanowires grown on annealed M-plane sapphire can have either zincblende or wurtzite crystal phase; they also demonstrate a variety of crystallographic orientations, cross-section geometries and dimensions (Figure 3). Out of nine nanowires that were examined, six different crystallographic orientations were detected, namely, [100], [110], ±[1̅1̅2], ±[30̅3], and ±[00̅01] (the ± sign indicates that the absolute polarity is not known because the cross-sectional TEM does not enable its determination). This lack of specificity in the crystallographic orientation and crystal phase of the nanowires indicates that for ZnTe nanowires that grow within the sapphire nanogrooves the graphoepitaxial effect is much stronger than the epitaxial effect, and therefore it dominates the energetic considerations that rule the nanowires growth. The epitaxial relations between the substrate and the nanowires play only a minor role, which allows a variety in the crystal...
phase and the crystallographic orientations. It is possible that some orientations are slightly preferred over others and that the crystallographic orientations of the nanowires are not completely random. To validate this issue, more samples should be examined, so that better statistics could be gained.

The wide variation in the cross-section geometry (i.e., hexagonal, rectangular, round, etc.) of the nanowires may be related to the variation in the crystallographic orientations and phases of the nanowires. According to the HRTEM images, the widths of the nanowires range from 33 to 185 nm, and the height of the nanowires range from 17 to 105 nm. Overall, the mean width and height are $80 \pm 50$ nm and $56 \pm 24$ nm, respectively. This relatively large variation in dimensions may be attributed to the variation in catalyst nanoparticle size and the radial vapor–solid growth phenomenon, in which Zn and Te atoms are added directly to the ZnTe nanowires during the synthesis. Therefore, the cross sectional dimensions of the nanowires are mainly a function of distance from the tip of the nanowires (the metal droplet), and different nanowires were cut at different positions along their length.

Guided Growth of ZnTe Nanowires on Nonannealed M-plane (1010) Sapphire. On nonannealed M-plane (1010)
sapphire, a high-density of guided nanowires was not yet achieved. Most horizontal ZnTe nanowires seem to grow in all directions, and in many cases the nanowires change their direction as they grow. Nonetheless, it seems that a significant number of nanowires grow precisely along the \( \pm [12 \bar{1} 0] \) directions (Figure 4A−C). The ZnTe nanowires on the M-plane wafers were synthesized with the same parameters used to grow the nanowires on the annealed M-plane wafers. It is reasonable to assume that with further optimization of the synthesis process, a larger percentage of the ZnTe nanowires will be guided along the \( \pm [12 \bar{1} 0] \) directions.

Cross sectional HRTEM analysis was done on one lamella with three nanowires. All three nanowires had a zincblende crystal structure and [110] crystallographic orientation (Figure 4D,E). This crystallographic orientation was observed in only two out of the nine examined nanowires that grew on the annealed M-plane wafers. Unlike the case of the ZnTe nanowires on annealed M-plane, where no crystallographic orientation was energetically preferred, on M-plane sapphire nanowires that grow in the [110] direction and with the \( \pm [12 \bar{1} 0] \) orientation seem to be energetically favorable as the epitaxy between the nanowires and the substrate governs both the guidance and the orientation of the nanowires. The nanowires widths range between 180 and 200 nm, and their heights range between 70 and 130 nm. Overall, the mean width and height are 187 ± 12 nm and 93 ± 32 nm, respectively. The variation in these dimensions of the nanowires can be explained by the tapering phenomenon and by variations in the catalyst nanoparticles size.

**Guided Growth of ZnTe Nanowalls on C-Plane (0001) Sapphire.** On C-plane (0001) sapphire, horizontal ZnTe nanowalls grow along six isoperiodic directions \( \langle 10 \bar{1} 0 \rangle \) defined by the 3-fold symmetry of the C-plane. This symmetry causes the guided nanowalls to create a triangular network (Figure 5A−C). Unexpectedly, on C-plane sapphire, the syntheses resulted in the formation of tall and narrow nanowalls, instead of nanowires (Figure 5D−G). On C-plane, the syntheses were conducted at higher temperature than on the other substrates: source temperature of 950 °C compared to 800 °C and sample temperature of 630−640 °C compared to 560−590 °C.
resulted nanowalls have a rectangular geometry with a large aspect ratio (height divided by width). It is postulated, that instead of vapor−solid radial growth, the case of guided ZnTe nanowires on annealed M-plane sapphire, ZnTe nanowires on C-plane experience facet-selective vapor−solid growth in addition to the axial vapor−solid growth; during the synthesis, Zn and Te atoms are added to the upper facet of the nanowires, converting them to nanowalls. This phenomenon might be related to the higher temperatures in these syntheses. The high temperatures allow the Zn and Te atoms to diffuse to energetically favored planes, leading to growth in a preferred direction. The facet selective vapor−solid growth can also explain the tapered shape of the nanowalls (the height of the nanowalls is decreasing toward their tip).

Cross-sectional HRTEM analysis was conducted on two lamellas with a total number of nine nanowalls. All the nanowalls had a rectangular cross-section and the same crystallographic orientation. The diffraction pattern does not allow to differentiate between the ZB \([1\bar{1}2]\) and the WZ \([10\bar{1}0]\) orientation of ZnTe. Since ZnTe usually has a ZB crystal phase, we consider the ZB \([1\bar{1}2]\) to be the most likely growth axis of the nanowires with the \([111]\) planes as the upper and lower facets and the \([110]\) facets as the side walls (Figure 5F). The nanowalls width range between 95 and 200 nm, their height range between 175 and 2200 nm, and the aspect ratio range between 1.75 and 13.7. Overall, the mean width and height are 140 ± 40 nm and 1380 ± 730 nm, respectively. The relatively large variation in nanowalls height is due to the tapering phenomenon—nanowalls that were cut near their tip had lower height compared to nanowalls that were cut near their origin. The variation in the nanowalls width can be explained by a limited horizontal vapor−solid growth during the synthesis or by variation in the metal droplets dimensions.

Besides being scientifically interesting, the formation of nanowalls holds applicable potential due to its high surface-area to volume ratio and its high aspect ratio. An example of a possible application is the trigate transistors, in which the gate electrode covers a rectangular channel.40

**Guided Growth of ZnTe Nanowires on Annealed Miscut C-Plane (0001) Sapphire.** When C-plane sapphire is miscut within a plane that is slightly tilted from the C plane (miscut C), it results in a vicinal surface with a high surface energy. When this unstable substrate is annealed in air at high temperatures (1400−1600 °C) for a number of hours, the surface undergoes restructuring to present a periodic array of L-shaped nanosteps spaced by C-plane terraces (Figure 1A). Depending on the miscut orientation and annealing conditions, nanosteps with different morphologies and orientations can be created.26 We used miscut C plane sapphire by 2° toward \([1\bar{1}20]\). The periodically faceted steps guide the growth of the horizontal ZnTe nanowires by a graphoepitaxial effect (Figure 6A−B), and only two opposite growth directions remain out of the six isoperiodic \([10\bar{1}0]\) directions. Unlike the nanowires grown on an annealed M-plane, on annealed miscut C-plane the nanowires show a much lower yield of guidance and many horizontal nanowires are misoriented. Syntheses on annealed miscut C-plane that were conducted with parameters matching the syntheses on M-plane and annealed M-plane resulted in guided nanowires. Syntheses that were conducted with parameters matching the syntheses on C-plane failed to produce nanowires at all. The low yield of guided nanowires on annealed miscut C-plane may be attributed to the fact that these nanosteps are smaller in dimensions than the nano-
grooves formed on annealed M-plane, and thus have a lower interaction with the growing nanowires. It is reasonable to assume that with further optimization of the synthesis process, a larger percentage of the ZnTe nanowires will be guided along the nanosteps.

Cross sectional HRTEM analysis was done on two lamellas with a total number of three nanowires. All the nanowires had aspect ratio smaller than 1, zincblende crystal structure and a [112] growth axis, with the {111} planes as the upper and lower facets and the {110} facets as the side walls (Figure 6D). This crystallographic orientation is exactly the same as in the case of ZnTe nanowalls on C-plane sapphire, indicating that both the graphoepitaxial and the epitaxial effects play a significant role in the guidance of the nanowires along the nanosteps (unlike guided ZnTe nanowires grown on annealed M-plane). The growth direction of the nanowires is mainly dictated by the nanowires—nanosteps interactions while the crystallographic orientation of the nanowires is derived from the nanowires—substrate epitaxial mismatch. The nanowires widths range between 39 and 90 nm, their heights range between 37 and 44 nm and the aspect ratios range between 0.67 and 0.95. Overall, the mean width, height, and aspect ratio are 60 ± 27 nm, 47 ± 12 nm, and 0.83 ± 0.15, respectively. The observation that the aspect ratio is smaller than 1, strengthens the postulation that the formation of nanowalls on the C-plane is related to the relatively high temperatures during the synthesis. In the annealed miscut C-plane case the source and sample temperatures were 800 °C and 570 to 580 °C respectively, while in the C-plane case the source and sample temperatures were 950 °C and 630 to 640 °C, respectively. For nanowires grown on annealed C-plane C-plane, the temperature was not high enough for atoms to diffuse to the favorable upper facet, and as result the nanowires experience radial vapor—solid growth instead of facet selective vapor—solid growth. The variation in the dimensions of the nanowires can be explained by the tapering phenomenon and by variations in the metal droplets dimensions.

Horizontal Growth of ZnTe Nanowires on R-Plane (1102) Sapphire. On R-plane (1102) sapphire, horizontal growth of ZnTe nanowires was achieved, but no guidance was observed (Figure 7A). In addition, vertical nanowires grown on R-plane tend to present a finer shape than horizontal nanowires, and many vertically grown nanowires lose their directionality and fine structure when coming in contact with the surface during growth. This observation indicates that the growth of horizontal nanowires is not energetically favorable under the current conditions. Surprisingly, at elevated temperatures (source at 950 °C and samples at 640 to 680 °C) instead of nanowires or nanowalls, the syntheses resulted in flat zigzag crystals (width range of 1–4 μm, thickness range of 100–150 nm, Figure 7B). This phenomenon was not studied because it is beyond the scope of this work. Perhaps with a different set of synthesis parameters, guided growth of ZnTe nanowires on R-plane sapphire could be achieved.

The demonstration of guided ZnTe nanowires, in addition to the previously demonstrated guided growth of GaN, ZnO, and ZnSe nanowires, strengthens the generality of the growth phenomenon and indicates that it can be applied on a variety of different compounds. The syntheses outcomes indicate that the graphoepitaxial effect dominates the growth direction of the nanowires: the yield of guided nanowires on M-plane sapphire improved significantly upon the formation of nanogrooves along the surface and the six isomorphic growth directions of nanowalls on C-plane sapphire were reduced to only two directions upon the formation of nanosteps along the surface. The crystallographic orientation uniformity observed on guided nanowires and nanowalls grown on M-plane, C-plane, and miscut C-plane substrates implies that the epitaxial relations between the nanowires/nanowalls and the substrate govern the crystallographic orientation of nanowires/nanowalls. Most of the syntheses, including the more successful ones, resulted in rough and tapered nanowires, indicating that a vapor—solid growth occurred along with a VLS growth. Further optimization of the synthesis process might reduce this undesired vapor—solid growth, and produce finer nanowires.

**Compositional Analysis of Guided ZnTe Nanowires.**

The elemental composition of the ZnTe nanowires was analyzed using energy-dispersive X-ray spectroscopy (EDS), energy filtered TEM (EFTEM), and photoluminescence (PL) techniques. The EDS and EFTEM were carried out on nanowires grown on annealed M-plane sapphire due to the high yield of guided nanowires achieved on this particular substrate. To obtain quantitative data regarding the exact stoichiometry of the nanowires, a thin cross-section lamella was prepared using FIB, and was analyzed with an EDS detector installed within a TEM. Quantitative EDS measurements that were done on two different nanowires revealed a 1:1.10 ± 0.02 atomic ratio of Zn and Te, respectively (Table S3 and Figure S1). The deviation from the 1:1 ratio can be explained by the fact that the vapor pressure of Zn is higher than that of Te. Therefore, the as-grown ZnTe nanowires are expected to contain less Zn than Te. For II–VI semiconductors, metal vacancies or nonmetal interstitials usually result in p-type behavior. This correlation between stoichiometry and electronic behavior will allow us to corroborate the EDS measurements upon fabricating and measuring TFTs. EFTEM was used for elemental mapping. The resulting images revealed that the guided nanowires were indeed ZnTe; but their quality is insufficient to accurately determine whether Al or O diffused into the nanowires or whether Zn or Te diffused into the sapphire substrate.
Optical Characterization of the Guided ZnTe Nanowires and Nanowalls. PL spectra of guided ZnTe nanowires grown on annealed M-plane and guided ZnTe nanowalls grown on C-plane sapphire were measured using the 457 nm and the 514 nm lines of an argon-ion laser (Figure 8. The PL spectra of guided nanowires grown on annealed M-plane is consistent with previous optical characterizations done on ZnTe nanostructures. The maximum emission at 547 nm represents a small blue shift of 2 nm with respect to the 549 nm for bulk ZnTe. This slight blue shift may be attributed to compressive strain induced in the nanowires, or to a change in the dielectric constant of the substrate with respect to vacuum. A blue shift resulting from quantum confinement is not plausible because the Bohr radius of the exciton for ZnTe (6.2 nm) is significantly smaller than the diameter of most nanowires. Intrinsic point defects (IPD) tend to result in a broad deep level (DL) emission peak due to in-gap optical transitions. ZnTe with oxygen doping (ZnTe:O) possesses intermediate band structure, which results in a broad emission peak of red light. The observation that the measured spectra consist of a single peak indicates that guided ZnTe nanowires on annealed M-plane sapphire are relatively free of defects and oxygen doping. The PL spectra of guided nanowalls grown on C-plane sapphire were typical for ZnTe, although with a maximum emission at 547 nm and a broad emission peak of red light. This red emission may be the result of defect caused by the vapor–solid growth mechanism that turned the nanowires into nanowalls.

Electronic Properties of Guided ZnTe Nanowires. The electronic properties of the guided ZnTe nanowires were studied by fabricating top-gate field-effect transistors and conducting two-terminal electrical measurements by applying a source–drain DC bias and recording the $I−V$ curves for different gate voltages, as shown in Figure 9. These measurements were carried out on arrays of nanowires grown on annealed M-plane sapphire due to the high yield of guided nanowires achieved on this particular substrate.

Initial $I−V_{sd}$ measurements indicated a large contact resistance between the source–drain electrodes and the ZnTe nanowires. Annealing the devices in Ar atmosphere at 280 °C for 30 min was found to lower the contact resistance. No significant difference in the measured resistance was observed between devices with Ni or with Ti/Au contact electrodes. Measurements were made on nine arrays containing between 2 and 25 nanowires. In some of the devices, the signal was weak and noisy. $I−V_{sd}$ curves often show varying degrees of asymmetry, which could be attributed to different effects (e.g., tapering, presence of catalyst layer on one end, etc.). To amplify the signal, these field-effect transistors were also measured under a 532 nm laser excitation (these devices were not included in the hole mobility and hole density calculations). For all the working devices, $I−V_{sd}$ curves for different gate voltages, $V_g$, showed that the conductivity increases by 1–2 orders of magnitude when the gate voltage is decreased from positive to negative values. These results indicate that the guided ZnTe nanowires are p-type with a significant response to gating. This observed p-type behavior is consistent with the EDS measurements that revealed a 1:1 stoichiometric ratio between Zn and Te. The calculated hole mobility values range from $\mu_h = 3.3 \times 10^{-5}$ to $1.2 \times 10^{-4} \text{cm}^2/\text{V s}$, which are relatively low compared to reported values for nonhorizontal ZnTe nanowires. The hole density was found to be $1.2 \times 10^{17}$ to $6.5 \times 10^{17} \text{cm}^{-3}$, which is comparable to reported values for nonhorizontal ZnTe nanowires. The influence of the different crystallographic orientations on the electronic properties of the guided ZnTe nanowires is not in the scope of this work, but will be evaluated in following research.

Photoconductivity of Guided ZnTe Nanowires. To conduct photocurrent measurements, two-terminal devices were built on an array of between 1 and 15 nanowires, by defining source and drain electrodes. The measurements were...
performed using a 532 nm laser as the excitation source, while applying a source-drain DC bias and recording \( I-V \) curves for different excitation power and \( I-t \) curves for a fixed excitation power while using an optical chopper (Figure 10A). Measurements were made on 30 working devices. For all the measured devices, the conductivity increased upon illumination, indicating that the guided nanowires are photoconductive in the visible range (Figure 10C,D). The calculated on/off ratio ranges between 10 to 150, with an average of 80 \( \pm \) 50. This relatively large range may reflect a variation between the different devices including the quality of the crystal of the nanowires in each photodetector and the quality of the contacts between the nanowires and the source/drain electrodes. Without illumination, the current in many devices was in the order of pA, which is near the detection limit of our measurement system, making the signal noisy and the on/off ratio calculation inaccurate. These devices were therefore discarded from the on/off ratio calculation. Time-dependent measurements (Figure 10D) show response and recovery times as fast as the measuring system resolution, indicating that \( \tau_{\text{on}} \approx \tau_{\text{off}} \leq 5 \text{ ms} \). These response and recovery times are comparable to reported values of nonhorizontal ZnTe nanowires.\(^{31,34}\) The measured on/off ratios and response and recovery times indicate that ZnTe nanowires are promising building blocks for visible spectrum photodetectors.

**CONCLUSIONS**

We have demonstrated the VLS guided growth of horizontal p-type ZnTe nanowires on four different substrates including flat and faceted planes of sapphire. The nanowires also proved to be photoconductive in the visible range. We showed that ZnTe nanowires can be guided by three different modes: (a) epitaxial growth along specific lattice directions, (b) graphoepitaxial growth along nanogrooves, and (c) graphoepitaxial growth along nanosteps. The demonstrated growth morphologies include six or two growth directions, according to the symmetry of the substrate or the geometry of the surface perturbations. The synthesis of nanowires on a variety of substrates by different guiding modes demonstrates the generality and flexibility of the guided growth phenomenon. The crystallographic orientations and directionality of the nanowires results from the interplay between the epitaxy and graphoepitaxy effects, which are different for each substrate and synthetic conditions. The graphoepitaxial effect was found to be dominant in dictating the nanowire’s directionality, while the epitaxial effect seemed to be dominant in dictating the crystallographic orientation of the nanowires. The best alignment of the nanowires was obtained on annealed M-plane sapphire, but these nanowires grew in up to six different crystallographic orientations without any clear preference. On nonannealed M-plane, only a limited number of nanowires were guided, but these nanowires had the same [11\( \overline{2} \)] crystallographic orientation. On C-plane and annealed miscut C-plane sapphire the nanowalls/nanowires grew with a [1\( \overline{1} \)2] crystallographic orientation. These observations indicate that both the growth direction and the crystallographic orientation of guided nanowires can be controlled upon the selection of the substrate. This could be useful for future technological applications since the crystallographic orientation of semiconductors can affect their electronic properties.\(^{44}\) By fabricating photodetectors and top-gate TFTs, we have shown that the guided ZnTe nanowires grown on annealed

Figure 9. Electronic properties of guided ZnTe nanowires grown on annealed M-plane (10\( \overline{1} \)0) sapphire: (A) Illustration of a top gate TFT and the measurement setup. (B) SEM image of the displayed TFT. Contrast variability is due to the presence of the dielectric layer. (C) Source–drain current (\( I_{sd} \)) vs source–drain voltage (\( V_{sd} \)) are displayed for different gate voltages (\( V_{g} \)). (D) Source–drain current (\( I_{sd} \)) vs gate voltage (\( V_{g} \)) is displayed for a different source–drain voltages (\( V_{sd} \)).
M-plane are p-type and photoconductive in the visible range. The grown ZnTe nanowires are still not yet optimal in terms of degree of alignment, morphology, uniformity and the nanowire density. Nonetheless, they do serve as a proof-of-concept for the generality of the guided growth approach, and may pave the way for the integration of p-type nanowires into functional systems by a parallel assembly method that was not available previously. Producing ordered arrays of ZnTe nanowires with controlled position, structure, and properties have a great technological potential, and will enable large-scale integration of complex devices with a wide range of applications, such as LEDs, photodetectors, photovoltaic cells, sensors, nonlinear optical devices, and more.

■ ASSOCIATED CONTENT

The Supporting Information is available free of charge on the ACS Publications website at DOI: 10.1021/acs.jpcc.6b05191.

Added crystallographic data and mismatch analysis; elemental composition data and elemental mapping of ZnTe guided nanowires (PDF)

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Notes
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