Direct realizing the growth direction of epitaxial nanowires by electron microscopy

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The growth direction of nanowires is critically important for precisely controlling their physical and chemical properties and the performance of related nanowire-based electronic devices. Realizing the true orientations of nanowires is an important issue regarding designing and growth of nanowires with desired orientations and the subsequent applications. In this study, we demonstrated three electron microscopic techniques to determine the growth directions of epitaxial nanowires: mainly, correlating selected area electron diffraction pattern and bright-field imaging in transmission electron microscopes, tilting the nanowires until their growth directions parallel to the incident electron beams in scanning electron microscopes, and correlating the nature of cleavage planes of the nanowire substrates and nanowire projections in the side-view scanning electron microscopic investigations.

INTRODUCTION

Semiconductor nanowires, especially epitaxial III-V compound semiconductor nanowires are considered as the most versatile family of one-dimensional nanostructures since they provide an enormous wealth for novel fundamental research as well as unique building blocks for various interesting applications [1–4], due to their direct bandgaps, high carrier mobility, and high photoluminescence efficiency [5–7]. One of the challenges in realizing these unique nanowire properties in nanowire-based devices is to integrate nanowires on the nano-devices or chips with well-organized arrangement. To solve this issue, the epitaxial nanowire growth provides the uniqueness that well aligned nanowires can be grown on the chosen substrates, and by selecting substrates with particular orientations, specifically orientated nanowires can be grown. The epitaxial III-V semiconductor nanowires are generally grown in metal-organic chemical vapor deposition [8–10], molecular beam epitaxy [11–13], chemical vapor deposition [14,15], through the vapor-liquid-solid [16–19] or the vapor-solid-solid mechanisms [20–22].

Generally speaking, the epitaxial III-V nanowires are nucleated and grown in the $\langle 111 \rangle_B$ directions for zinc-blende structured nanowires or in the $\langle 000\bar{1} \rangle$ direction for wurtzite structured nanowires due to their lowest surface energy in the $\{111\}/\{0001\}$ atomic planes [23]. In most cases, nanowires grown along the $\langle 111 \rangle_B/\langle 000\bar{1} \rangle$ directions with the $\{111\}_B/\{000\bar{1}\}$ catalyst/nanowire interfaces tend to naturally contain uncontrolled mixture of polytypes and/or adopt planar defects [24–26], such as stacking faults or twins due to the small energetic differences for the stacking consequences in zinc-blende and wurtzite structures along their $\langle 111 \rangle_B/\langle 000\bar{1} \rangle$ directions [27]. To date, scientific findings have been reported that the growth of free-standing III-V nanowires are with non- $\langle 111 \rangle_B/\langle 000\bar{1} \rangle$ growth directions [22,27–33]. It is of interest to note that most of these non- $\langle 111 \rangle_B/\langle 000\bar{1} \rangle$ nanowires generally have high structural quality. Therefore, controlling the nanowire growth direction may provide a new approach to obtain III-V semiconductor nanowires with controlled crystal phase and high structural quality. On the other hand, from a fundamental scientific point of view, the optical, electrical and mechanical properties of nanowires are expected to differ for nanowires with different growth directions [34,35], and previous studies have shown that the growth directions of nanowires can affect the nanowire properties [36–38]. In this regard, in order to better understand the relationship between the nanowires growth directions and their properties and, in turn, to better fulfil nanowire applications, it is critically important to accurately determine the growth directions of grown epitaxial nanowires. In our previous investigations [29–32], different electron microscopic techniques were used to determine the nanowire growth directions.

In this study, we summarized three general electron microscopic techniques to determine the growth directions of epitaxial nanowires, including (1) the correlation of se-

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lected area electron diffraction patterns and transmission electron microscopy (TEM) images taken from individual nanowires in different orientations, (2) in scanning electron microscopes (SEM), tilting the target nanowires until their growth directions become parallel to the incidental electron beam, and (3) investigating nanowire projections in the side-view SEM images. In the latter two cases, the crystallographic information of the nanowire substrates and their cleavage nature must be known.

TECHNIQUE 1: ELECTRON DIFFRACTION TECHNIQUE IN TEM

It is well known that the electron diffraction pattern has been the most widely used technique in determining the crystallographic orientations and directions of TEM specimens. For a selected nanowire that has been orientated to a zone-axis (the zone-axis parallel to the electron beam), the correlation between the bright-field TEM image and the corresponding electron diffraction pattern allows the crystallographic orientation of the nanowire to be determined. Therefore, for determining the nanowire growth direction, it is important for the nanowire with its growth direction to be perpendicular to the incidental electron beam. In many studies, only one electron diffraction pattern was collected from individual nanowire to determine its growth direction. However, our extensive TEM investigations suggest that a single electron diffraction pattern is not sufficient to exclusively determine the growth direction of the nanowire. This is because, in many cases, when the nanowire was tilted to a crystallographic zone-axis in TEM, it is very likely for the tilted nanowire to be inclined to electron beam, as schematically illustrated in Fig. 1. As can be seen, for a

nanowire with a $\langle u_2, v_2, w_2 \rangle$ growth direction, the projection of the nanowire in the diffraction plane gives an electron diffraction pattern, from which a nanowire growth direction of $\langle u_1, v_1, w_1 \rangle$ can be indicated, according to the geometry outlined in Fig. 1. In this case, the nanowire growth direction will be wrongly determined.

In order to explicitly determine the nanowire growth direction, the nanowire must be perpendicular to the electron beam. To ensure this, it is necessary to tilt the nanowire to different zone-axes along its growth direction. Fig. 2 shows a typical example of growth direction determination of a $[\bar{1}\bar{1}0]$ nanowire, in which two sets of diffraction patterns were taken by tilting the nanowire along the $[\bar{1}\bar{1}0]$ direction. Fig. 2a is a bright-field TEM image taken from a $[\bar{1}\bar{1}0]$ nanowire. To determine its growth direction, the nanowire was tilted along its growth direction to a zone axis. The inset in Figs 2b and d is a schematic illustration showing the tilting process. Fig. 2b is the bright-field TEM image of the nanowire top and Fig. 2c is the corresponding diffraction pattern, taken along the $[\bar{1}\bar{1}0]$ zone axis. The correlation between the TEM image (Fig. 2b) and the corresponding electron diffraction pattern (Fig. 2c) suggests that the $\bar{1}\bar{1}0^*$ diffraction spot is parallel to the growth direction (note that orientations of the TEM images and their corresponding electron diffraction patterns have been carefully aligned). To confirm the determined nanowire growth direction, the nanowire was further tilted along $[\bar{1}\bar{1}0]$ direction (keep both $\pm\bar{1}\bar{1}0^*$ diffraction spots unchanged during the tilting process) until another zone axis is reached, as shown in Fig. 2d. Fig. 2e is the corresponding electron diffraction pattern and shows the $[\bar{1}\bar{1}\bar{2}]$ zone axis. Based on both electron diffraction patterns, the nanowire

Figure 1 Schematic illustration of wrongly determined growth direction of nanowire using one electron diffraction pattern if the nanowire is inclined to the electron beam.

Figure 2 Determination of nanowire growth direction in TEM. (a) Bright-field TEM image of a $\langle 110 \rangle$ nanowire. (b–e) TEM images and the corresponding electron diffraction patterns in different zone axes. The inset shows the tilting process along the nanowire axis.

growth direction can be unambiguously determined to be $[\bar{1}\bar{1}0]$. It should be noted that, if the nanowire is inclined to the electron beam when Fig. 2c is taken, as illustrated in Fig. 1, the $[\bar{1}\bar{1}0]$ growth direction suggested by Fig. 2c should be corresponded to $\langle u_1, v_1, w_1 \rangle$. As a consequence, when performing the further tilting to achieve Fig. 2e, the nanowire shown in the TEM (Fig. 2d) will no longer be parallel to the TEM image shown in Fig. 2b. Therefore, it is important to compare both TEM images after the tilting process. If they are parallel, as shown in Figs 2b and d, we can ensure that the growth directions determined by this approach are correct.

TECHNIQUE 2: TILTING PROCESS IN SEM

It is well known that the epitaxially grown nanostructures allow their crystallographic orientations to adopt from the orientations of their substrates. Because of this, it is possible to determine the growth directions of epitaxial nanowires if the orientations of the substrates are known. In a simple case, vertical epitaxial nanowires grown on the zinc-blende structured $\{111\}$ substrates must have their growth direc-

tions along either $\langle 111 \rangle$ or $\langle 0001 \rangle$ directions, depending on their crystal structures—zinc-blende or wurtzite. However, if the grown epitaxial nanowires are inclined, the situations become complicated, as it cannot be straightforward to determine their growth directions. In this case, the growth direction of the epitaxial nanowires may be determined by tilting them in an SEM until their growth directions become parallel to the electron beam, as illustrated in Fig. 3. Fig. 3a is a top-view SEM image and shows some nanowires (as white lines) orientated differently, although they are mainly orientated along some specific directions. This feature suggests that these nanowires are epitaxially grown nanowires and they are inclined to their substrate. Technically, by tilting the SEM sample along the direction perpendicular to the chosen nanowire, it is possible to align the nanowire growth direction to the electron beam. Fig. 3 illustrates the different stages during the tilting process, in which we choose a case of InAs nanowires epitaxially grown on the $(\bar{1}\bar{1}\bar{1})$ GaAs substrate. In this specific case, when the SEM sample is tilted for $\sim 35^\circ$, the nanowire becomes parallel aligned with the electron beam, as shown

Figure 3 Determination of the growth direction of epitaxial nanowires in SEM. (a) 0 degree tilted images of the GaAs{111}_B substrate. The inset in the SEM image of (a) is the sketch of all the $\langle 112 \rangle$ directions on the GaAs{111}_B substrate. (b) 20° tilted images. (c) 35° tilted images when the axis of inclined nanowire is parallel to the electron beam. The scale bar is 1 μm . The inset in the SEM image of (c) is an enlarged SEM image showing the hexagonal-shape cross section.

in the SEM image in Fig. 3c. However, to determine the growth direction of the epitaxial nanowire, it is necessary to clarify the crystallographic orientation of the substrate. So far, all we know is that the substrate is the $(\bar{1}\bar{1}\bar{1})$ GaAs, and single crystallographic information is not sufficient to determine the crystal orientation for a given crystal. For determining the absolute crystal orientation for a crystal, it is necessary to know two independent atomic planes and/or directions. In this regard, we note that semiconductors often have specific cleavage planes. For example, diamond structured Si have their cleavage planes of {111} [39], while zinc-blende structured GaAs have {110} cleavage planes [40]. Therefore, the information of the surface orientation and a cleaved side is often used to determine the crystallographic feature of a given semiconductor wafer in the semiconductor industry. In fact, we can use this approach to define the crystallographic orientation of nanowire substrates. Taking the case shown in Fig. 3a as example, a cleaved edge can be seen and we know for GaAs, the cleavage planes are {110}. For the $(\bar{1}\bar{1}\bar{1})$ GaAs substrate, there are three equivalent $\{1\bar{1}0\}$ planes that are perpendicular to the $(\bar{1}\bar{1}\bar{1})$ surface. Also, there are three equivalent $\langle 112 \rangle$ directions within the $(\bar{1}\bar{1}\bar{1})$ surface, as marked by arrows in Fig. 3a (which can be done only when the ori-

entations of {110} planes are clarified). It is of interest to note that, almost all inclined nanowires have their projections on the $(\bar{1}\bar{1}\bar{1})$ GaAs surface parallel to one of the three $\langle 112 \rangle$ directions. This suggests that these inclined nanowires must have their growth directions being along $\langle \bar{1}\bar{1}n \rangle$ or $\langle 00\bar{1} \rangle$ where n is an integer. Under the assumption that the cleaved plane is $(1\bar{1}0)$ in Fig. 3a, tilting must be along the $[1\bar{1}0]$ axis, as illustrated in Fig. 3. Based on the tilting angle measured from Fig. 3c and general crystallography geometry of the substrate, the growth direction of the inclined nanowire can be exclusively determined as $[\bar{1}\bar{1}0]$ for this specific inclined nanowire. It is of interest to note that, when the nanowire axis has been tilted to be parallel to the electron beam, the side-wall facets of the inclined nanowire can be clearly observed as shown in the inset of the SEM image in Fig. 3c. Using the substrate cleavage planes as reference ($(1\bar{1}0)$ in this case), the side-wall planes can be determined as schematically demonstrated in Fig. 3c. This SEM technique using the tilting process to determine the nanowire growth direction is a universal technique which is suitable for the epitaxial nanowires since the determined growth direction of epitaxial nanowire is based on the orientation of the substrate and its cleavage nature. It should be noted that, in this practice, we cannot determine the

crystal structure of the nanowires, so that the growth direction determination here is under the assumption that the epitaxial nanowires adopt the crystal structures of the nanowire substrates. Nevertheless, our extensive electron microscopic investigations suggest that non- $\langle 111 \rangle / \langle 0001 \rangle$ nanowires indeed often adopt the crystal structures of the substrates.

TECHNIQUE 3: CLEAVAGE PLANE SIDE-VIEW INVESTIGATION IN SEM

As mentioned above, when the crystallographic information of nanowire substrates is known, the nanowire substrates can be cleaved and viewed along the perpendicular direction of the cleaved plane in SEM (i.e., the cleavage edge is perpendicular to the electron beam and the electron beam is parallel to the substrate surface). From the side-view SEM image, the angle between the projection of nanowire and the substrate normal can be measured, from which, if the nanowire is parallel to the cleavage plane, the growth direction of epitaxial nanowires can be determined based on the crystallographic nature of the nanowire substrates. However, the following general factors must be considered. A crystal often has many equivalent directions. This is particularly true for high-symmetric crystals, such as zinc-blende structured GaAs, and for those low-index directions, such as $\langle 001 \rangle$, $\langle 110 \rangle$, $\langle 111 \rangle$ and $\langle 112 \rangle$ directions. The projections of these equivalent directions along the view direction (electron beam direction) in side-view SEM images are often different. Therefore, the angle between the projection of nanowire growth direction and the surface normal of the nanowire substrate may not be exactly the true angle between the nanowire growth direction and the substrate normal, so that care must be taken when using this technique to determine the epitaxial nanowire growth

direction. Taking the case of $\langle \bar{1}\bar{1}0 \rangle$ epitaxial nanowires grown on a $(\bar{1}\bar{1}\bar{1})$ substrate as an example, Fig. 4 illustrates the possible projections of nanowires when they are viewed along the $[1\bar{1}0]$ direction. Crystallographically, there are three equivalent $\langle \bar{1}\bar{1}0 \rangle$ directions that have acute angles with the substrate surface normal of $[\bar{1}\bar{1}\bar{1}]$, i.e., $[\bar{1}\bar{1}0]$, $[0\bar{1}\bar{1}]$ and $[\bar{1}0\bar{1}]$ directions (refer to Fig. 4a). As can be noted, only the $[\bar{1}\bar{1}0]$ nanowires are parallel to the $(1\bar{1}0)$ cleaved plane and the other two $[0\bar{1}\bar{1}]$ and $[\bar{1}0\bar{1}]$ nanowires are not. Therefore, when the substrate is viewed along the $[1\bar{1}0]$ direction (the side-view SEM image), the projection of $[\bar{1}\bar{1}0]$ nanowires on the $(1\bar{1}0)$ plane is $[\bar{1}\bar{1}0]$ (refer to the blue schematic nanowire in Fig. 4b). Since both $[0\bar{1}\bar{1}]$ and $[\bar{1}0\bar{1}]$ nanowires are not parallel to the $(1\bar{1}0)$ cleavage plane, their projections on the $(1\bar{1}0)$ plane is $[\bar{1}\bar{1}\bar{2}]$ (refer to the purple nanowire in Fig. 4b). In fact, due to the nature of equivalent directions of a specific type of epitaxial nanowires (refer to the SEM image shown in Fig. 3a), nanowires with different growth directions can result in different projected angles. In our specific case, the $\langle \bar{1}\bar{1}0 \rangle$ epitaxial nanowires can result in two different projected angles between the projected nanowire and substrate normal when they are viewed in SEM shown in Fig. 4c. As a consequence, care must be taken when using this approach to determine the growth directions of epitaxial nanowires. Similar to the previous technique, the crystal structure of the epitaxial nanowires cannot be determined, so that the determined nanowire growth direction is under the same assumption that the epitaxial nanowires adopt the crystal structures of the nanowire substrates. If the crystal structure and structural quality need to be clarified, TEM investigation must be performed. In this study, it is necessary to know the substrate orientation and this information should be known from the growth designers. If this information

Figure 4 Schematics and SEM image illustrating the growth of $\langle \bar{1}\bar{1}0 \rangle$ directions on the $(\bar{1}\bar{1}\bar{1})$ substrate. (a) Three equivalent $\langle \bar{1}\bar{1}0 \rangle$ directions on the $\{ \bar{1}\bar{1}\bar{1} \}_B$ substrate. (b) Side-view sketch viewed along the cleavage plane showing both real and projected $\langle \bar{1}\bar{1}0 \rangle$ nanowires on the cleavage plane. (c) The corresponding side-view SEM image.

Figure 5 (a) Side-view SEM image of nanowires grown on the $\{111\}_B$ substrate with different inclined nanowires. (b, c) TEM images of two type of nanowires picked up by FIB, and the insets are their corresponding diffraction patterns taken along $[1\bar{1}0]$ zone axis.

is not provided, convergent beam electron diffraction may be employed to determine the substrate orientations and, perhaps the polarity of the substrates.

It should be mentioned that, in the two SEM techniques outlined above, the determination of nanowire growth directions is relatively easy if all inclined epitaxial nanowires belong to one kind. However, if different kinds of inclined nanowires are simultaneously grown on a nanowire substrate [32], the determination of nanowire growth directions for different kinds of nanowires becomes complicated. In this case, in order to differentiate those different kinds of nanowires with the same inclined angle, the focused ion beam (FIB) equipment may be employed by picking up specific nanowires and mounting them on the specifically designed TEM grid for the TEM characterizations, as demonstrated in Fig. 5. Fig. 5a shows two inclined nanowires with identical inclined angle with respect to the surface normal of the nanowire substrate, Figs 5b and c are a pair of TEM images showing the two nanowires vertically mounted on the TEM grids. Their growth directions can then be determined by the TEM technique introduced early, and the diffraction patterns (as shown in the insets of Figs 5b and c) indicate that they grew along different growth directions.

CONCLUSION

In conclusion, we outline three electron microscopic techniques to exclusively determine the growth directions of epitaxial nanowires, including one TEM technique and

two SEM techniques. Since crystal structures of nanowires and their qualities cannot be determined by SEM, it is necessary to perform TEM investigation when the structural information is critical. These technical approaches provide a necessary guidance to determine the growth direction of epitaxial nanowires.

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Author contributions Zhang Z and Zou J conceived the study. Zhang Z wrote the manuscript, revised by Han X and Zou J, who also supervised this electron microscopic study. All the authors contributed to the general discussions.

Conflict of interest The authors declare that they have no conflict of interest.

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中文摘要 纳米线的生长方向对于精确控制纳米线电子器件的物理、化学以及电学性能至关重要. 准确确定纳米线的生长方向才能指导设计与制备特定生长方向的纳米线. 本文采用三种电子显微技术来准确确定外延纳米线的生长方向. 包括: 1) 通过关联选区电子衍射花样和相应的透射电镜明场像图片来确定纳米线生长方向; 2) 在扫描电镜中倾转纳米线直到纳米线的生长方向与电子束平行, 通过计算倾转角度来确定外延纳米线的生长方向; 3) 在扫描电镜侧视图中, 通过关联衬底的自然解理面与纳米线在侧视图上的投影的晶体学关系来确定外延纳米线的生长方向.