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Periodically twinned 6H-SiC nanowires with fluctuating stems

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Abstract

Periodically twinned 6H-SiC nanowires with fluctuating stems were successfully synthesized on SiC substrate by chemical vapor deposition with ferrocene as the catalyst. The morphology and structures of the products were systematically characterized by X-ray diffraction, scanning electron microscopy, and transmission electron microscopy. The results showed that the nanowires consisted of periodically twinned segments and presented stem-fluctuating morphology, whose diameters fluctuated in the range of 10–150 nm along the axial direction. A model based on vapor–liquid–solid mechanism was proposed to explain the growth of periodically twinned SiC nanowires with fluctuating stems, which revealed that the alternating high-density stacking faults facilitated the formation of stem-fluctuating morphology.

Keywords: 6H-SiC nanowire; Microstructure; Growth mechanism

1. Introduction

SiC nanowires have attracted significant interest due to their outstanding physical and chemical properties, such as large band gap, high chemical stability, high thermal conductivity, and excellent mechanical strength [1]. As a result, they can be widely used in functional nanodevices and nanocomposites, such as stress sensors [2], field-emitters [3], and reinforcing and toughening agents [4,5]. As is well-known, some factors from nanowires, such as structures and morphology, might affect the performance of the nanowires [6,7]. Therefore, many efforts have been devoted to fabricating and characterizing SiC nanowires with different structures and morphology, including twinned [6–8], diameter-fluctuating (such as beaded and bamboo-like) [9–11], and needle-shaped [12]. Up to now, SiC nanowires with special structure and morphology have shown great potential in many fields, especially in functional and structural materials. In the field of functional materials, periodically twinned SiC nanowires exhibited a distinctive photoluminescence spectrum due to the collective influence of size effects and twin defects, which indicated the potential applications in optical devices [8]; the needle- and bamboo-shaped SiC nanowires possessed significantly enhanced field-emitting properties, which were ideal candidates for field-emitters [13]. In the field of structural materials, the necklace- and bamboo-shaped SiC nanowires have been used as reinforcements in the composites and showed a better strengthening effect than the common SiC nanowires, owing to the mechanical interlocking between the contoured surface and the matrix [9,11–14].

On the other hand, SiC has many polymorphs, such as 3C, 4H and 6H-SiC, which presents different property. Most of the reported SiC nanowires are 3C-SiC because it is more favorable in energy. But compared with 3C-SiC, 6H-SiC might have a more promising application in harsh environment due to its larger band-gap and higher breakdown electric field strength [15,16]. In our previous work, 6H-SiC nanowires have been successfully *in-situ* grown on SiC coatings to improve the oxidation inhibition ability of carbon materials [17,18]. However, to date, there are limited papers reporting the synthesis of 6H-SiC nanowires with special structures and morphology and it is still a challenge to synthesize 6H-SiC nanowires with special structures and morphology, such as periodically twinned 6H-SiC nanowires with fluctuating stems.

In the present work, novel periodically twinned 6H-SiC nanowires with fluctuating stems were synthesized via a simple and economical chemical vapor deposition (CVD) technique

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with ferrocene as the catalyst. The microstructures were investigated systematically and a growth mechanism of the as-received nanowires was proposed.

2. Experimental

Small graphite specimens (10 mm × 10 mm × 10 mm) were cleaned ultrasonically with ethanol and then dried at 100 °C for 2 h. SiC coating was prepared on these specimens by pack cementation. Details for preparing SiC coatings on the graphite specimens were reported elsewhere [19]. The preparation process of SiC nanowires on SiC coated graphite samples was described as follows: A mixture composed of 50–70 wt% SiO_2 (purity: 99.7%), 10–20 wt% Si (purity: 99.0%), 15–30 wt % graphite (purity: 99.5%), and 5–15 wt% ferrocene (purity: 99.0%), was milled for more than 2 h followed by being placed in a graphite crucible. The SiC coated samples were suspended above the mixture in the graphite crucible with a graphite lid. Then, the graphite crucible was put into an electrical furnace and heated to 1500–1600 °C and maintained at this temperature for 2 h in an argon atmosphere. When the furnace was cooled to ambient temperature naturally, the SiC substrate was covered by a light-yellow fluffy layer.

The phase composition of products was characterized by X-ray diffraction (XRD, PANalytical X'Pert PRO, Netherlands). The morphology and structures of products were examined by a field emission scanning electron microscopy (FE-SEM, ZEISS Supra 55, Germany) and transmission electron microscopy (TEM, FEI Tecnai F30 G^2, USA). The bright-field (BF) images, selected area electron diffraction (SAED) patterns, high-resolution TEM (HRTEM) images, and energy-dispersive X-ray spectrometer (EDS) data were acquired using the TEM.

3. Results and discussion

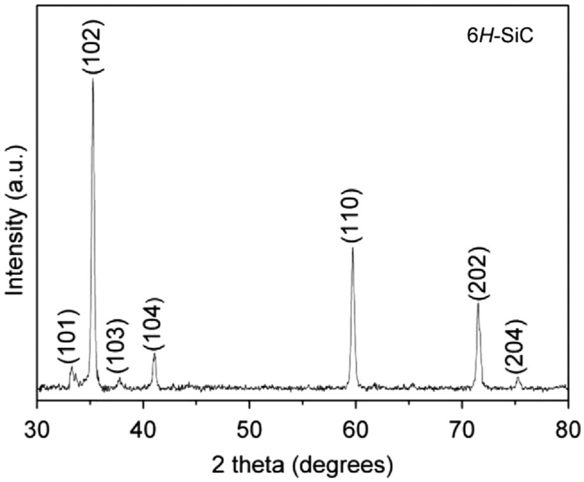

Fig. 1. A representative XRD pattern of the products.

Fig. 1 is the XRD result of the light-yellow products collected from the SiC substrate at room temperature, which suggests that 6H-SiC is the only crystalline phase. The values of diffraction peaks are well agreed with 6H-SiC (JCPDS Card no. 29-1128) with the lattice constants of $a = 0.308$ nm and $c = 1.509$ nm. The major diffraction peaks are assigned to the (101), (102), (103), (104), (110), (202), and (204) crystalline planes of 6H-SiC.

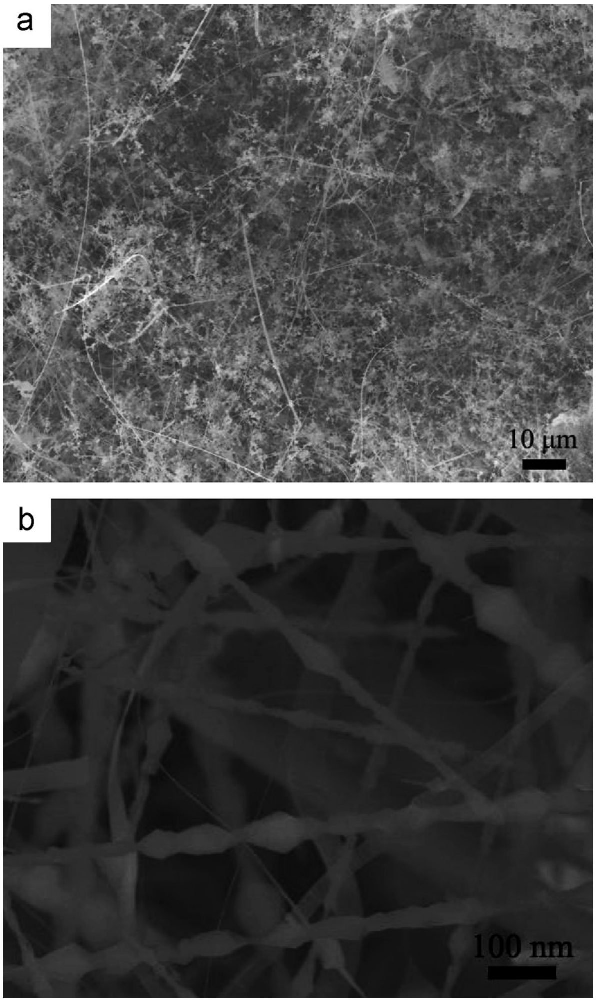

Fig. 2. General morphology of the as-prepared SiC nanowires. (a) SEM image at low-magnification and (b) SEM image at high-magnification.

Fig. 2(a) presents a typical SEM image of the product at low magnification, which reveals that the light-yellow fluffy product is nanowires. Their lengths are ranged from tens to over 100 µm. A closer examination (Fig. 2(b)) shows that the surface of most nanowires is rough rather than smooth because of their non-periodically fluctuating diameters. It can be estimated that the diameters of nanowires fluctuate between 10 and 150 nm along their axial directions.

Further characterizations for the structures and compositions of as-synthesized nanowires were carried out by TEM and EDS. Fig. 3(a–c) exhibit the representative structures of the nanowires with different diameter-fluctuating ranges. According to the BF images in Fig. 3(a–c), obvious striped structures can be found in the nanowires, which suggests the presence of stacking faults (SFs). The nanowires present a regular zigzag appearance, indicating the formation of periodically twins [8]. Fig. 3(d) clearly shows a droplet attached at the nanowire tip. The corresponding EDS result (Fig. 3(e)) indicates that

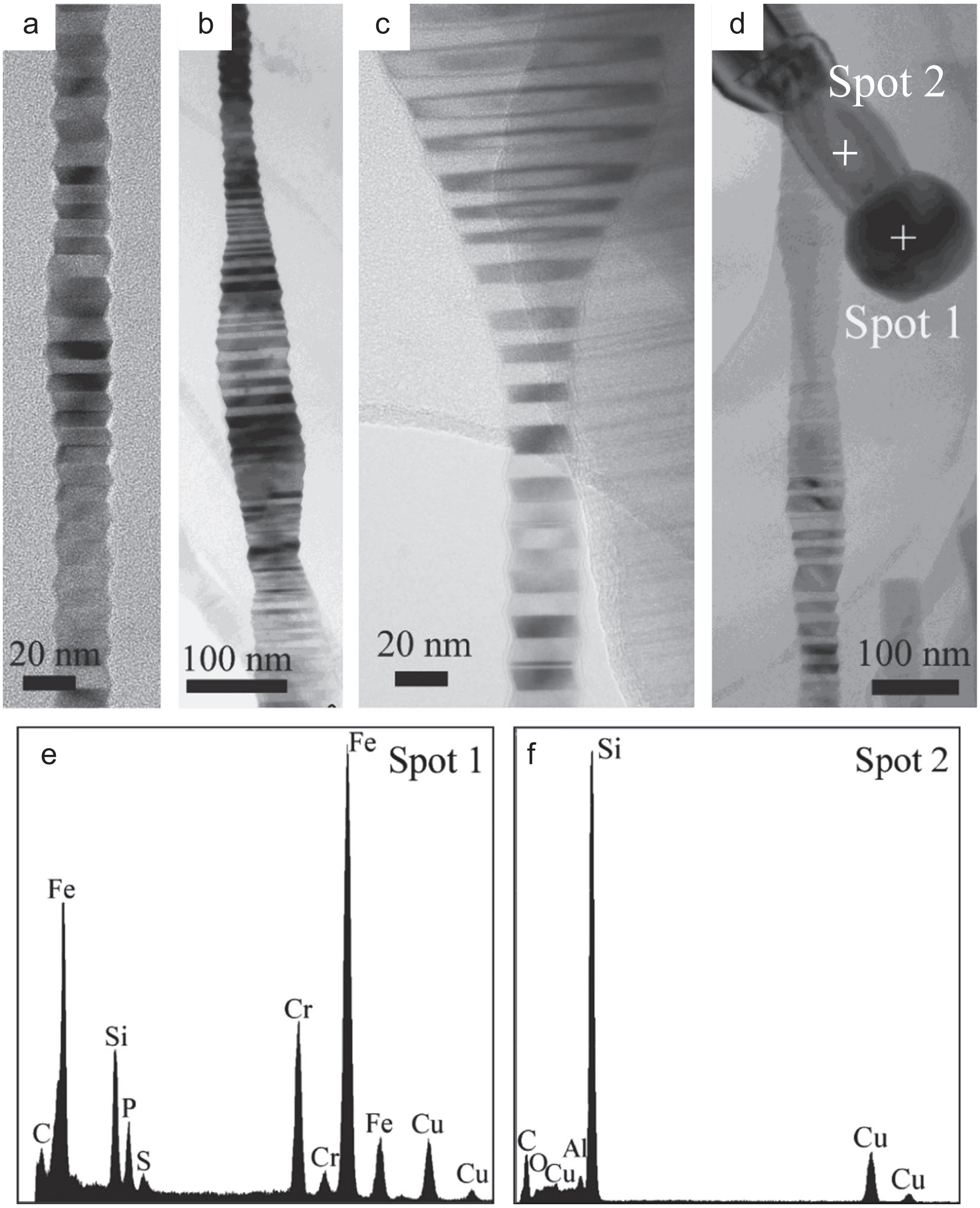

Fig. 3. (a) BF image of a nanowire with uniform diameter; (b) BF image of a nanowire with small diameter fluctuation; (c) BF image of a nanowire with large diameter fluctuation; (d) BF image of a catalyst droplet attached at the nanowire tip; (e) EDS spectrum of the catalyst droplet; and (f) EDS spectrum of the as-synthesized SiC nanowire.

the droplet contains Fe, Si and C. Therefore, it is inferred that the growth of nanowires is governed by vapor–liquid–solid (VLS) mechanism [20]. A typical EDS spectrum recorded from the stem of nanowire is presented in Fig. 3(f), which reveals that the nanowire consists of Si, C and Al, as well as a small amount of O (Cu is from the copper grid). The EDS spectra recorded from different areas are identical, suggesting that the as-grown nanowires are Al-doping 6H-SiC [21]. It is worth noting that no Al element was introduced during the synthesis of nanowires. However, Al_2O_3 was added during the preparation of SiC coating. It is inferred that the Al element in the nanowires might come from Al_2O_3. Moreover, Al was not detected in the droplet (Fig. 3(e)), which suggests that the Al element was not precipitated from the catalyst droplet. Taking into account these evidences, we propose that the Al in the nanowires is derived from the reaction of SiC and a small amount of Al_2O_3 [22].

HRTEM and SAED were employed for investigating the microstructures of nanowires in detail. Fig. 4(a) is a representative BF image of a single 6H-SiC nanowire with fluctuating stem. It is confirmed that the nanowire has a feature of zigzag faceting over the wire surface. From the HRTEM image (Fig. 4(b)), it is found that the nanowire is wrapped by an amorphous SiO_2 layer of ~2 nm, which can account for the O detected in

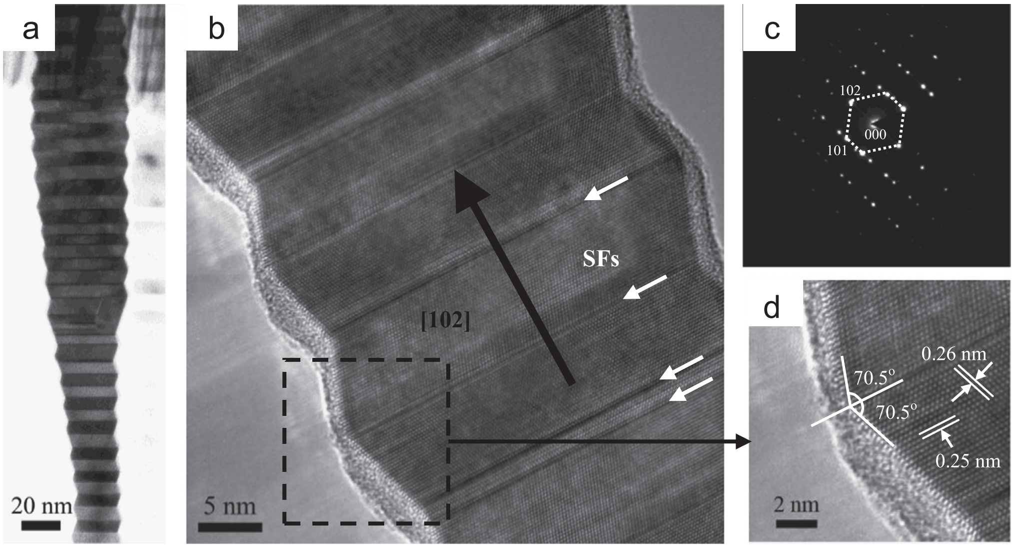

Fig. 4. (a) BF image of a typical twinned nanowire with zigzag appearance; (b) HRTEM image of the twinned nanowire; (c) Corresponding SAED pattern recorded from the nanowire; and (d) Magnified HRTEM image of the square area in (b).

Fig. 3(f) [23]. Furthermore, it is also found that the stacking of crystal planes is not consecutive and is usually separated by some SFs (indicated by white arrows in Fig. 4(b)). Generally speaking, from the structural view, 6H-SiC has a hexagonal structure with ABCACBABCACB stacking sequence and twins can be formed in the nanowires by periodically inserting SFs into the normal stacking sequence [24,25]. The corresponding SAED pattern (Fig. 4(c)) also exhibits obvious twinning features. Fig. 4(d) is the magnified HRTEM image of marked area in Fig. 4(b), which further confirms the periodically alternating twins and the zigzag angle is exactly 141° (70.5° + 70.5°). The lattice fringe spacings are measured to be 0.25 nm and 0.26 nm, consistent with the (102) and (101) plane space of bulk 6H-SiC, respectively. Both the SAED pattern and HRTEM image suggest that the nanowire grows along [102] direction (indicated by black arrow in Fig. 4(b)).

As catalyst droplets can be often observed at the tips of nanowires (Fig. 3(d)), the obtained nanowires grow via the typical VLS mechanism. The detailed growth process is demonstrated as follows: Firstly, mixed gas of SiO and CO was mainly generated via reaction (1) and (2). Simultaneously, ferrocene thermally decomposed into active C and Fe (reaction (3)) [26]. The active C could be used as reactant in reaction (1), which would promote the reaction. The Fe particles reacted with the mixed gas of SiO and CO to form Fe–C–Si catalyst droplets, followed by nucleating on the SiC substrate. With the continuous dissolving of SiO and CO, SiC generated and diffused to the liquid–solid interface. After the SiC concentration in a droplet reached supersaturation, 6H-SiC precipitated from the liquid–solid interface. Subsequently, 6H-SiC nanowires nucleated on the SiC substrate and grown along the preferential orientation. Gaseous Al_2O was generated accompany by the reaction (4) occurring at elevated temperature (above 1327 °C) [22]. Then, Al atoms were incorporated into the nanowires by forming substitution solid solution (reaction (5)) and Al-doped 6H-SiC nanowires were formed eventually [21]. The SiO_2 shells wrapped in the nanowires were formed during the cooling stage. It could be explained by reaction (6), which occurred at temperature below 900 °C [27].

$$\mathrm{SiO_2(s)+C(s)\rightarrow SiO}(g)+\mathrm{CO}(g) \tag{1}$$

$$\mathrm{SiO_2}(s)+\mathrm{Si}(s)\rightarrow \mathrm{2SiO}(g) \tag{2}$$

$$(C_5\mathrm{H_5})_2\mathrm{Fe}(s)\rightarrow \mathrm{10C}(s)+\mathrm{Fe}(s)+\mathrm{5H_2}(g) \tag{3}$$

$$\mathrm{SiC}(s)+\mathrm{Al_2O_3}(s)\rightarrow \mathrm{SiO}(g)+\mathrm{Al_2}O(g)+\mathrm{CO}(g) \tag{4}$$

$$\mathrm{Al_2O}\overset{\mathrm{SiC}}{\Longrightarrow}\mathrm{2Al_{Si}}''' + \mathrm{O_C^{\bullet\bullet}} + \mathrm{V_C^{\bullet\bullet\bullet\bullet}} \tag{5}$$

$$\mathrm{3SiO}(g)+\mathrm{CO}(g)\rightarrow \mathrm{SiC}(s)+\mathrm{2SiO_2}(s) \tag{6}$$

Fig. 5 is a HRTEM image of nanowire node position. It is observed clearly that some SFs exist in the node (indicated by white arrows), whose density is much higher than that in the stem position. It is notable that high-density SFs are usually found in the nodes of nanowires.

The formation of twinned structure can be explained by total energy minimization during the growth process, including contributions from solid surface, liquid surface, solid–liquid interface, twin boundary and edge at twin boundary [28]. Combined with the above microstructure analysis results of periodical twins and fluctuating stems, we propose a growth mechanism based on the model described the formation of periodically twinned ZnS nanowires [29], to explain the formation of periodically twinned SiC nanowires with fluctuating stems. The mechanism is shown schematically in Fig. 6.

Firstly, the source gas of SiO and CO reacted with Fe to form a Fe–C–Si catalyst droplet and the VLS system was established. In a VLS system, the catalyst droplet was the key to drive the growth of nanowire. Two factors might determinate the nanowire ultimate growth under the steady supply of source gas: the precipitation of SiC at the liquid–solid interface and its diffusion inside the droplet. In our case, we assumed that the precipitation

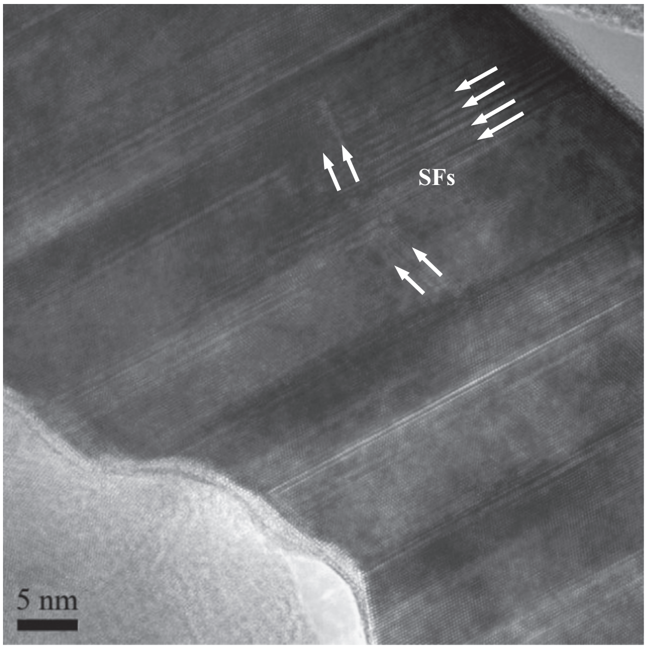

Fig. 5. HRTEM image of the nanowire node position.

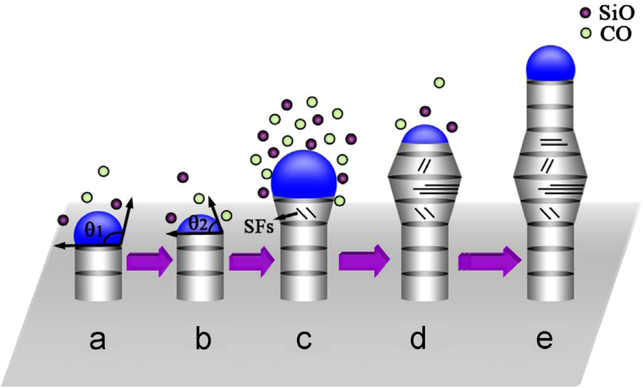

Fig. 6. Schematic model for the formation of periodically twinned SiC nanowires with fluctuating stems (twin boundaries are indicated by dark gray slices): (a–b) The wetting angle changed periodically, followed by successive release of interface energy. So the periodically twinned SiC nanowire was formed; (c) With the increase of local gas concentration, SFs began to generate and the nanowire diameter increased; (d) The diameter of nanowire decreased followed by the decrease of local gas concentration; and (e) Finally the periodically twinned SiC nanowires with fluctuating stems were formed.

rate of SiC was faster than its diffusion rate in the droplet. Therefore, SiC species in the droplet was consumed largely after a fast growth of the nanowire, which depressed the SiC precipitation and leaded a larger wetting angle θ_1 due to the reduced wettability at the liquid–solid interface (Fig. 6(a)). As source gas was supplied constantly, the SiC concentration in the droplet increased gradually, followed by the increase of growth rate. Meanwhile, the wettability was enhanced and the wetting angle θ_2 became smaller (Fig. 6(b)). Accompanied by the increase of nanowire growth rate, the next segment of nanowire growth was initiated and leaded to the consumption of SiC species in the droplet once more. During the whole process, the concentration of SiC species in the catalyst droplet varied periodically, inducing the periodically-varied wettability of liquid–solid interface. Meanwhile, the wetting angle changed periodically, followed by successive release of interface energy. The SFs were produced periodically to release the stored elastic energy and the periodically twinned structure was formed [30]. The change of wetting angle has been observed in the growth of carbon nanofiber [31].

In real condition, the local concentration of source gas was not permanent. In fact, it was non-periodically fluctuant and could be affected by many factors, such as the consumption in forming large-diameter stem and the activities of reactions (1) and (2). The relationship between the nanowire energy and the nanowire diameter can be described using a simple formula by assuming a cylindrical configuration of the nanowire [30]:

$$E = 4\pi C/D \qquad (7)$$

where E is the elastic energy per unit length of nanowire, C is a constant describing the elastic property and the surface energy of special material, and D is the nanowire diameter.

Furthermore, it was considered that the droplet size dictated the nanowire diameter in a VLS system [32]. Hence, when the source gas remained at a low concentration, the droplet was small, leading to the small diameter of periodically twinned nanowire (Fig. 6(a and b)). With the reaction proceeding, the local source gas concentration around the nanowire increased gradually, which might be attributed to the sealing of graphite lid and the high reactivities of SiO_2, C and Si, resulting in the energy of VLS system increasing. And the VLS system was in a metastable state. To reduce the system energy E, the nanowire diameter D should increased. According to Fig. 5, it can be inferred that high-density SFs played an important role in increasing the nanowire diameter. When the VLS system remained a metastable state, high-density SFs started to generate. The defects in the nanowire (high-density SFs) were beneficial for the nucleation of SiC and boosted the epitaxial growth of nanowire [33]. As a result, the droplet size and the nanowire diameter increased (Fig. 6(c)). Meanwhile, the local source gas concentration decreased because of the depletion in forming large-diameter stem. Thus, the VLS system recovered to a stable state and high-density SFs disappeared, followed by the decrease of droplet size and nanowire diameter (Fig. 6(d)). With the varying concentration of local source gas, high-density SFs alternately appeared and the periodically twinned SiC nanowire with stem-fluctuating morphology was formed eventually (Fig. 6(e)).

4. Conclusions

Periodically twinned 6H-SiC nanowires with fluctuating stems were synthesized on SiC substrate by ferrocene-assisted CVD. Their growth was dominated by typical VLS mechanism. The formation of periodically twinned structure could be ascribed to the periodically-varied wettability of liquid–solid interface at the nanowire tip. And the stem-fluctuating morphology was mainly resulted from the inevitable fluctuation of local source gas concentration. It is worth noting that the alternating high-density SFs play an important role in forming fluctuating stems.

It is believed that SiC nanowires with this special structure and morphology will find a wide range of applications in nanodevice.

Acknowledgments

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