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Fabrication of quasi-one dimension silicon carbide nanorods prepared by RF sputtering

Dayin Xu, Zhiwei He, Yongping Guo, Yinyue Wang *

Department of Physics, Lanzhou University, Lanzhou 730000, PR China

Abstract

Anodic aluminum oxide (AAO) template was prepared by a two-step anodization method at low temperature (1 °C) and silicon carbide was deposited on the templates by non-reactive radio frequency sputtering method. Well-aligned quasi-one dimension silicon carbide nanorods with the average diameter about 80–90 nm and a mean length of 400 nm were obtained perpendicular to the substrate and observed by AFM and SEM after the aluminum substrates were striped off. Then some samples were annealing at flowing N_2 at 400, 500 and 600 °C and FTIR was performed on these samples to obtain the information of structure.

Keywords: Through-pore AAO; SiC nanorods

1. Introduction

One-dimensional silicon carbide (SiC) materials (i.e., nanowires and nanorods) have attracted great interest for many applications due to their excellent properties, such as high mechanical strength, high thermal stability, high thermal conductivity and large band gap and is a promising material for the development of solid-state electronics, transducers, field emission devices, etc. [1]. SiC nanowires and nanorods have been prepared recently by several different routes. The general method is converting carbon nanotubes into SiC nanowires [2,3]. SiC nanorods prepared by a chemical vapor deposition (CVD) method have been reported by many authors [4,5]. Jin et al. [6] reported a novel ternary sol–gel route for the synthesis of SiC nanowires. Recently, in situ chemical vapor growth (CVG) method for SiC nanowires on reaction-sintered SiC (RS-SiC) plates is reported by Yang et al. [7]. The methods mentioned above have two disadvantages, one is intertwist of SiC nanowires (nanorods) and the other is complicated preparing process and needing very high temperature. Nanowires of many compositions have been prepared using porous templates as molds. Anodic alumina oxide (AAO) has been used as templates to make many kinds of nanowires (nanorods) composed of metals, semiconductors, and polymers.

In this work, through-pore AAO templates were fabricated by a two-step anodization. Amorphous silicon carbide was deposited on them using a non-reactive radio frequency sputtering methods without any catalysts. Well-aligned silicon carbide nanorods perpendicular to the substrate were prepared using this simple method.

2. Experiment

AAO templates were fabricated by a two-step anodization at low temperature. High-purity aluminum sheets (99.99%, 0.1 mm) were employed in our experiment. Prior to anodization, the aluminum sheets were degreased in acetone, and then annealed in flowing N_2 at 400 °C for 4 h to remove the mechanical stresses. Then the aluminum sheets were electropolished under a constant-current condition of 0.5 A/cm^2 in a mixture of $HClO_4$ and C_2H_5OH with volume ratio 1:4 for 3 min. the first anodization step was performed in 0.3 M oxalic acid for 4 h at 40 V at 1 °C. The pieces were etched in 6% (w/w) phosphoric acid and 1.8% (w/w) chromic acid mixtures at 60 °C for 1 h to remove the products of first anodization. A second anodization was conducted

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* Corresponding author. Tel.: +86 931 891 2703; fax: +86 931 891 3554.
E-mail address: wangyy@lzu.edu.cn (Y. Wang).

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on these pieces in the same condition as first anodization for 6 h. After the second anodization, the pieces were anodically oxidized in the solution of $HClO_4$ and C_2H_5OH (V:V = 1:4) at 25 °C at 40 V for 50 s, then the porous anodic aluminum oxide films were separated from the aluminum substrate immediately. Then these transparent pieces were immersed in a 5-wt% phosphoric acid solution at 30 °C for 60 min to remove the barrier layer and to widen the pores as well as thinned the AAO templates. The final products were through-pore AAO templates.

The through-pore AAO films then were served as the templates of fabrication of SiC naorods by a non-reactive RF sputtering method. The working gas introduced into the sputtering chamber was pure argon (99.999%). The background pressure was 1×10^{-3} Pa. The argon pressure was set approximate 1.5 Pa. The rf power was 200 W. The sputtering time was 60 min.

SEM (JSM-5600), AFM (CSPM2000) and FTIR (NiCOLET NEXUS670) were performed on these samples to get the information about the morphology and the details of structure.

3. Results and discussion

Fig. 1(a) and (b) show the SEM images of the top and bottom surface of through-pore template, respectively. From Fig. 1(a), we can see that well ordered hexagonal pores with diameter ~100 nm have formed. Fig. 1(b) shows the aluminum substrate and the barrier layer have been stripped off from the AAO and ordered hexagonal pores with diameter ~100 nm can been seen clearly. It reveals that through-pore AAO template has formed. The thickness of the through-pore AAO template is about 1 μm.

Figs. 2(a) and 3 are the SEM and AFM images of a through-pore AAO template after sputtering silicon carbide. They show that well-aligned quasi-one dimension silicon carbide nanorods perpendicular to the substrate has grown on through-pore AAO and the mean diameter of these nanorods is about 80–90 nm. The diameter is less than that of AAO. Fig. 2(b) is the SEM image of the back surface of the through-pore AAO after sputtering. We can see that the morphology of AAO shown in Fig. 1(b) is still in existence but the definition and contrast of the pores decrease compared with Fig. 1(b), which indicates that the length of the pore has become shorter than that of before sputtering and it means that the pore has been partially filled with silicon carbide. It also proves that the silicon carbide nanorods have formed. The mean length of the silicon carbide nanorods is about 400 nm according the results of HR-TEM.

In general the growth of nanowires by CVD and PVD follows the vapor–liquid–solid (VLS) growth mechanism

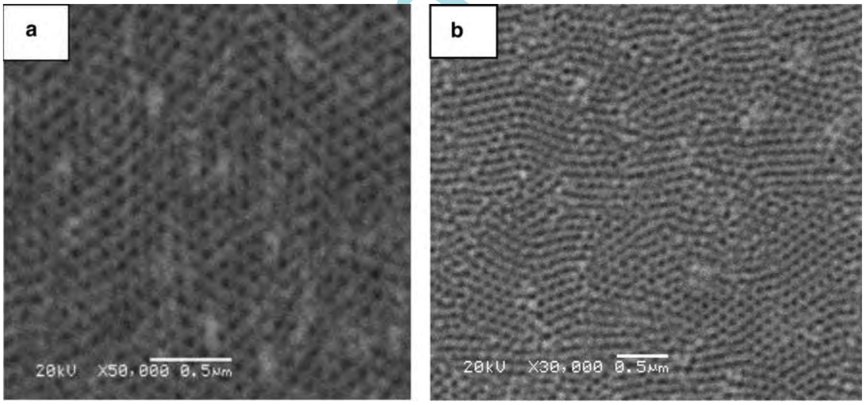

Fig. 1. SEM images of a through-hole AAO template: (a) image of top surface; (b) image of bottom surface.

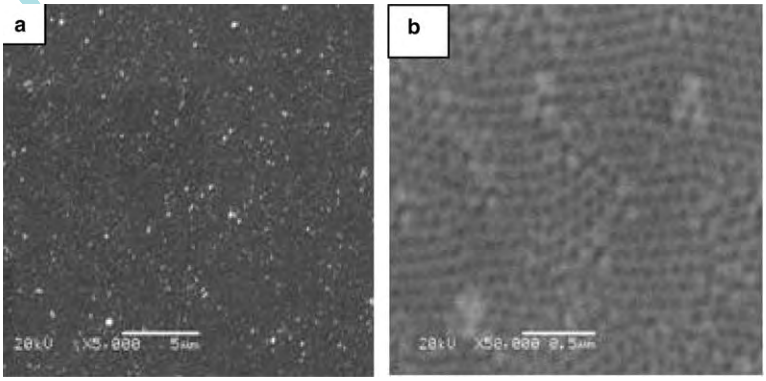

Fig. 2. SEM images of a through-hole AAO template after sputtering silicon carbide: (a) is the surface SiC deposited on; (b) is the opposite surface.

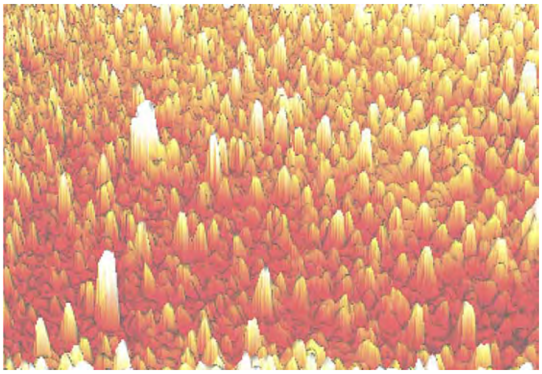

Fig. 3. AFM images of a through-hole AAO template after sputtering silicon carbide.

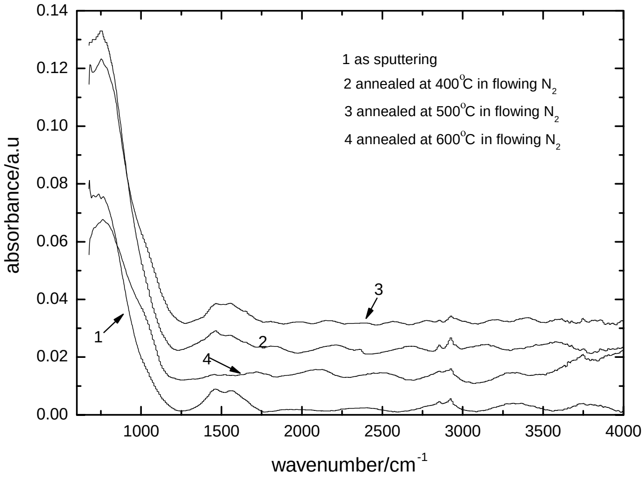

Fig. 4. FTIR spectra of as prepared and annealed samples.

[8], in which the forming of nanowires (naonorods) needs the assistant of catalyst. In our experiment, no catalyst is used so the mechanism may not be governed by VLS.

Fig. 4 is the FTIR spectra of SiC nanorods deposited on through-pore AAO templates. The peak at around 750 cm^{-1} is the vibration of Si–C bonds which becomes sharpness with increasing of the annealing temperature indicates that the structure is improved; the broadness of peak suggests that the network is still amorphous. The broad bands centered at 1500 cm^{-1} in curve 1, 2 and 3 origin from vibration of C═O, which disappear when annealed at 600 °C or higher annealing temperature. These results indicate that the main structure is Si–C bonds containing C═O bonds in as prepared samples.

4. Conclusions

Vertically well-aligned SiC nanorods with the average diameter about 80–90 nm and a mean length of 400 nm were fabricated on through-pore AAO templates by noreactive RF sputtering without catalysts.

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References

[1] X.T. Zhou, N. Wang, C.K. Au Frederick, H.L. Lai, H.Y. Peng, I. Bello, C.S. Lee, S.T. Lee, Mater. Sci. Eng. A 286 (2000) 119.
[2] H. Dai, E.W. Wong, Y.Z. Liu, S. Fan, C.M. Lieber, Nature 375 (1995) 769.
[3] C.C. Tang, S.S. Fan, H.Y. Dang, J.H. Zhao, C. Zhang, P. Li, Q. Gu, J. Cryst. Growth 210 (2000) 595.
[4] T. Seeger, P. Kohler-Redlich, M. Ruhle, Adv. Mater. 12 (2000) 279.
[5] S.Z. Deng, Z.S. Wu, J. Zhou, N.S. Xu, J. Chen, J. Chen, Chem. Phys. Lett. 356 (2002) 511.
[6] G.-Q. Jin, P. Liang, X.-Y. Guo, J. Mater. Sci. Lett. 22 (2003) 767.
[7] W. Yang et al., J. Cryst. Growth 264 (2004) 278–283.
[8] Clemens Burda et al., Chem. Rev. 105 (2005) 1025–1102.