Photoluminescence and microwave absorbing properties of SiC nanowires with different morphology

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Periodically twinned SiC nanowires and smooth SiC nanowires were largely synthesized by the sol-gel and the carbothermal reduction method, in which tetraethoxysilane and biphenyl were employed as silica and carbon precursors, iron nitrate and cobalt nitrate used respectively as additives. XRD, SEM, PL and VNA were used to characterize the SiC samples. The results show that the samples are silicon carbide nanowires with different morphology. Periodically twinned SiC nanowires and smooth SiC nanowires have different photoluminescence properties. Periodically twinned SiC nanowires exhibited excellent microwave absorption properties.

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1. Introduction

Microwave absorbing materials have been extensively studied due to the rapid development of wireless communications and high-frequency circuit devices in commercial, industrial, and military applications [1,2]. Nanomaterials are better microwave absorbing materials than conventional ferrites due to its excellent properties of wide absorption frequency, high absorption capability, low weight, good thermal stability, and antioxidation capability [3-7]. Microwave absorption capability depends on a nanomaterial's nature, shape, and size [8]. However, some nanomaterials were not effective microwave absorbing materials because of high costs or complex production methods [9]. Meanwhile, a large amount of microwave absorbing materials is not used at the higher temperature because of the low curie temperature [10].

Silicon carbide nanowires have many excellent properties, such as high thermal conductivity, high mechanical strength, high chemical stability and good field-emitting performance. On the other hand, SiC nanowiress have an adjustable and wide range of electrical resistivity from 10^{-3} to $10^4 \Omega$ m [11], which have been considered to be one of the important microwave absorbing materials used in the higher temperature environments [12]. As a result, SiC has recently drawn interest for applications in photonics and electromagnetics.

In this paper, periodically twinned SiC nanowires and smooth SiC nanowires were largely synthesized by the sol-gel and the carbothermal reduction method. The surface morphologies of SiC nanowires are analyzed. The microwave absorption properties and photoluminescence properties are also studied.

2. Experimental

2.1 SiC preparation

All the chemical agents were used as received without further purification. Biphenyl, tetraethoxysilane (TEOS, AR), acetone (AR), iron nitrate and cobalt nitrate were provided by Tianjin chemical corporation.

According to our previous work [13], the synthesis process is briefly described as follows. Firstly, 20 g of biphenyl and 1.5 g of iron nitrate were dissolved in 80 ml of acetone and then mixed with 50 ml of tetraethoxysilane under stirring. Then, 10 ml of oxalic acid (3.4 wt. %) was added into the mixture under stirring to enhance the hydrolysis of TEOS. After 24 h, 10 ml of hexamethylenetetramine aqueous (35.8 wt. %) solution was added into the above mixture for rapid gelation. Finally, the gel was dried at 100 °C for 12 h to obtain the xerogel. The carbothermal reduction was carried out in a horizontally tubular reactor. The xerogel was heated at 1300 °C in argon flow and maintained at the temperature for 7 h. The raw product was collected after the furnace was cooled down to room temperature and then purified at 700 °C for 2h to remove unreacted carbon followed by acid treatment with HF and HCl for 48h to eliminate the impurities. The purified samples were denoted as SiC-Fe. According to the same procedure, by employing 1.5 g of cobalt nitrate in the sol-gel process, we can obtain purified samples, denoted as SiC-Co.

2.2 Characterization

The crystallographic data were collected by a Rigaku D-Max/RB X-Ray diffractometer (XRD) with CuKa The microstructure of the samples was radiation. examined by LEO-438VP scanning electron microscope (SEM). The photoluminescence (PL) spectrum measurement was performed in a SLM48000SCF spectrometer with IK-series KIMMON He-Cd laser as an excitation source at room temperature. The measurements of microwave loss property for the specimens were carried out using Agilent-N5230A vector network analyzer in the 2-18GHz ranges. In this study, because silicon carbide is the dielectric loss material, the real part μ' and imaginary part μ'' of permeability are1 and 0, respectively.

3. Result and discussion

All samples are greenish powders. The XRD patterns of the samples are shown in Fig. 1. From the patterns, all of the strong diffraction peaks can be indexed to β -SiC, no other crystalline phases such as silica, carbon or other impurities were detected. The small peak marked with SF is due to stacking faults. The XRD results demonstrate that all the samples are pure β -SiC. The intense diffraction peaks indicate that the SiC samples have good crystalline structure.



Fig. 1. XRD patterns of the SiC samples.

The general morphology of the samples is shown in Fig. 2. Form Fig. 2a image, the SiC-Fe sample mainly consists of periodically twinned SiC nanowires with a diameter of 50-300 nm and a length of tens to hundreds of micrometers [13]. Fig. 2b shows that the SiC-Co sample mainly consists of smooth nanowires with a diameter of 80-300 nm and a length of tens to hundreds of micrometers.



Fig. 2. SEM image of SiC-Fe (a), SEM image of SiC-Co (b).

The photoluminescence (PL) spectrum of the SiC nanowires is depicted in Fig. 3. The results show that the PL features of periodically twinned SiC nanowires are strikingly different from smooth nanowires. From the Fig. 3a, three broad PL emission peaks of SiC-Fe are clearly observed at the center wavelength of 404 nm, 434 nm, and 547 nm respectively. Fig. 3b displays the PL spectrum of SiC-Co. A strong and broad emission band centered at 461 nm is observed. The peak at 404 nm may result from size-confinement effect [14]. The peak at 434 nm and

461nm are close to that of SiC nanowires [15]. The obvious different may be the combined results of quantum confinement, micro-defect and morphology effect. The peak around 547 nm is due to the presence of defect centers within the band gap [16]. Different emission wavelengths indicate that the luminescence characteristic depends strongly on the size, surface structure and morphology of the SiC nanowires.



Fig. 3. PL spectrum of SiC-Fe (a), PL spectrum of SiC-Co (b).

The complex permittivity reveals the ability of a material to store electric energy under an alternative electric field. Fig. 4 shows the real part (ϵ'), imaginary part (ϵ'') of permittivity of periodically twinned SiC nanowires and smooth SiC nanowires as a function of frequency in the frequency range of 2–18 GHz. As shown in Fig. 4a, the ϵ' and ϵ'' of periodically twinned SiC nanowires are

16.8-24.3 and 3.9-9.5. For smooth SiC nanowires, the ε' and ε'' are 4.96-6.25 and 1.36-1.69 (Fig. 4b). The results show that periodically twinned SiC nanowires have relatively high ε'' . The higher ε'' suggests periodically twinned SiC nanowires have a better capacity of dielectric loss than smooth nanowires in the microwave range.



Fig. 4. Permittivity spectra of SiC-Fe (a), Permittivity spectra of SiC-Co (b).

Fig. 5 shows the the reflection loss (RL) of the different morphology SiC nanowires for various thicknesses (1-3 mm) at 2-18 GHz. The minimum RL value shifts to lower frequencies with increasing thickness of SiC nanowires. Fig. 5(a) shows the RL of periodically twinned SiC nanowires. The 1 mm thick SiC nanowires did not exhibit minimum RL values. The values of minimum RL for the 1.5mm and 3mm thick SiC nanowires are -11.53 dB and -16.15 dB at 10 GHz and 4.56 GHz, respectively. The RL values are lower than -10

dB (90% absorption) at 4.2-5.0 GHz. Fig. 5(b) shows the RL of smooth SiC nanowires. The 1 mm and 1.5 mm thick SiC nanowires did not exhibit minimum RL values. The values of minimum RL for the 3mm thick SiC nanowires are -8.13 dB at 13.32 GHz. The periodically twinned SiC nanowires have the better microwave absorbing abilities than smooth nanowires result from a large amount of interfacial electric polarization at the the twin boundary.



Fig. 5. Microwave absorption properties of SiC-Fe (a), Microwave absorption properties of SiC-Co (b).

4. Conclusion

Periodically twinned SiC nanowires and smooth SiC nanowires were largely synthesized by the sol-gel and the carbothermal reduction method, in which tetraethoxysilane and biphenyl were employed as silica and carbon precursors, iron nitrate and cobalt nitrate used respectively as additives. They have different PL properties due to the size, surface structure and morphology of the SiC nanowires. Besides, the periodically twinned SiC nanowires have excellent microwave absorbing abilities because of a large amount of interfacial electric polarization at the the twin boundary.

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