Synthesis SiC nanoparticles from foamed plastic wastes

DONG-HUA WANG*, XIN FU
College of Chemistry and Life Science, Weinan Teachers University, Weinan 714000, PR China

A simple method is proposed to synthesize small SiC nanoparticles , in which foam plastic wastes and tetraethoxysilane (TEOS) were respectively employed as carbon and silica precursors. X-ray powder diffraction patterns (XRD) indicates that the product is 3C-SiC. Transmission electron microscopy (TEM) images show that the SiC samples were nanoparticles. Dynamic light scattering (DLS) indicates that The SiC nanoparticles have dimensions in the range of 10-40 nm. The SiC nanoparticles with smaller size have better reinforcement effect in epoxy resin and exhibit different photoluminescence properties.

1. Introduction

SiC as a very useful functional semiconductor due to its wide energy band gap, high physical and chemical stability, high thermal conductivity, low induced activity and so on[1, 2]. Due to various technological applications, nanoparticles of silicon carbide have attracted the interest of scientists and engineers [3]. Such as SiC nanoparticles allow the creation of composite materials with better mechanical properties [4]. In addition, the PL of SiC nanoparticles can be easily pushed in to the blue/violet range due to their small sizes and their PL emission intensities at room temperature are much stronger than bulk SiC [5, 6]. Thus, various methods were proposed to produce SiC nanoparticles. For example, Yu et al. produced β-SiC particles by plasma [7]. Buschmann et al. used chemical vapor to synthesis SiC nanoparticles with dimensions around 3-10nm [8]. Herlin et al synthesized small SiC particles with diameter about 10nm from C_2H_2 and SiH_4 mixture by laser pyrolysis [9]. However, sophisticated equipments or expensive reactants were frequently employed in these methods. So, it is a challenge to develop an efficient and economical route to prepare SiC nanoparticles.

As we all know, huge amounts of foamed plastic are consumed every year all over the world, and it has also caused serious environmental problems. This case has prompted the development of different techniques to eliminate from foamed plastic [10, 11].

Using foam plastic wastes as carbon source can provide an economical way to produce SiC and reduce their environmental pollution to some extent. Ju et al. adopted plastic bags to synthesize SiC nanomaterials [12]. SiC platelets and nanowires were produced via a sulfur-assisted approach at 350–500 ∘C in a stainless steel autoclave when plastic bags (main components: polyethene) and Si powder were used as carbon and silicon source. In this study, we synthesize SiC nanoparticles by foam plastic wastes and TEOS via the carbothermal reduction method. The SiC nanoparticles with smaller size (10-40 nm) have better reinforcement effect in epoxy resin and exhibit different photoluminescence properties.

2. Experimental

2.1. SiC preparation

The foam plastic wastes were washed with water to remove the impurity and dried, they were dried at 140 °C for 1h. Then they were cooled down to room temperature and comminuted.

The solution preparation is briefly described as follows. First, 1.5 g of cobalt nitrate was dissolved in 100 ml of ethyl alcohol (anhydrous, AR) and then mixed with 50 ml of tetraethoxysilane (TEOS, AR) 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, 16 g of from foamed plastic particle was added into the above mixture under for 48 h. Finally, the mixture was dried at 110 ◦C for 3 h to obtain the sample. The sample were put into the a horizontally tubular reactor and heated to 1000 °C at a rate of 5 °C/min in Ar flow, then to 1300 °C at a rate of 2 °C /min and maintained at this temperature for 7h. The raw product was collected after the furnace was cooled down to room temperature and then purified by air calcination and acid treatment (HF + HCl) to eliminate the impurities.

2.2 Composites preparation

Fabrication of the SiC-reinforced composites was similar to that described in the literature [13]. Firstly, 20 g epoxy resin mixed with 5 ml of acetone (AR) under strong stirring for about 10 min, and then 0.1 g of milled SiC powder (0.5 wt% of the resin) by mechanical grinding was dispersed in the mixture. The above mixture was infused into a silicone and vacuumized to eliminate air bubbles. Finally, the composite was dried at 130 °C for 4 h, the sample was collected for measurement, and therefore it was denoted as EP-SiC. Dumbbell-like and cuboid-like moulds were used for measuring tensile and bending strengths, respectively. The dumbbell-like mould has a length of 60 mm, with a central narrow part of 30 mm. The two ends of the mould have a width of 15 mm, while the central narrow part of 4 mm. The entire mould has a thickness of 3 mm. The cuboid-like mould has a length, width and thickness of 60 mm, 9 mm and 3 mm respectively. For comparison, epoxy resin without any filler was also prepared and it was denoted as EP.

2.3 Characterization

The crystalline structures of the samples were characterized by using a Rigaku D/max rA X-Ray diffractometer (XRD) with CuKα radiation. The structural features of the samples were observed by a JEOL-1011 transmission electron microscope (TEM). Dynamic light scattering (DLS) measurements of average sample sizes were obtained using a Zeta Potential and Particle Size Analyzer (Brookhaven Instruments, Holtsville, NY). The

mechanical properties of the composite materials were performed on a materials testing machine (SANS, Shenzhen, China). The photoluminescence (PL) spectrum measurement was performed in an F-7000 FL Spectrophotometer with a Xe lamp as an excitation source at room temperature.

3. Results and discussion

3.1 SiC characterization

The sample was greenish powders. The XRD pattern of the SiC sample is 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 XRD results indicate that the sample consists of pure β-SiC with good crystalline structure. Besides, the average size calculated by Scherrer formula is about 17 nm.
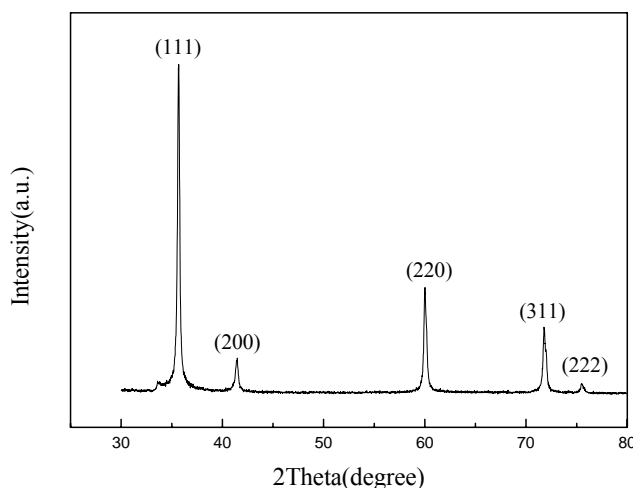

Fig.1. XRD pattern of SiC nanoparticles

The structure and morphology of the SiC sample is shown in Fig.2 (a) and (b). From the TEM image, it can be seen that the sample mainly consists of sphere-like nanoparticles with narrow size distributions, which the diameters lie between 10 nm and 40 nm.
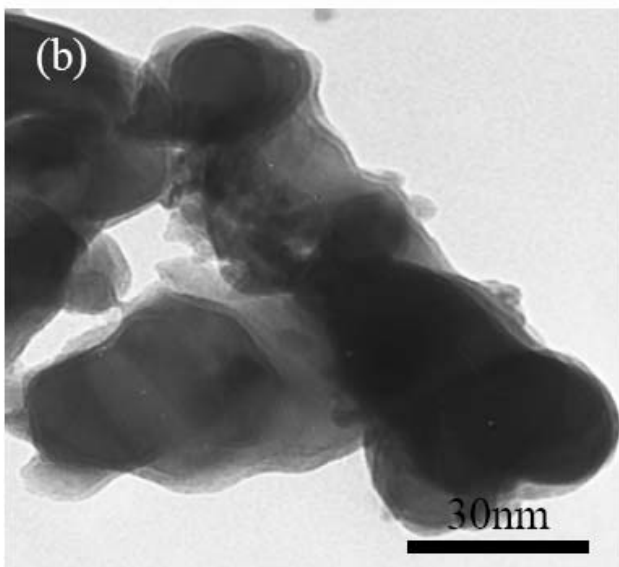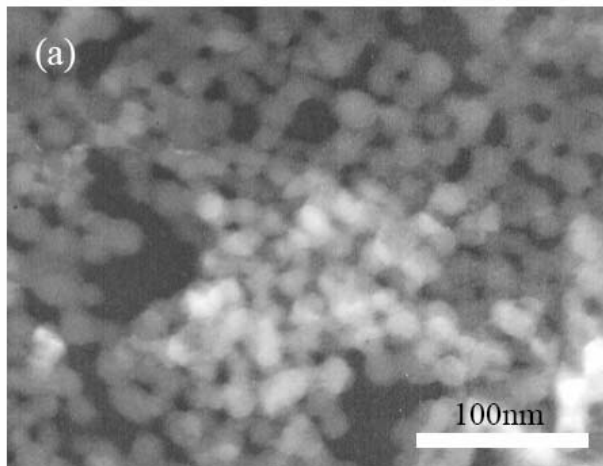
Fig.2. TEM images of SiC nanoparticles

Fig.3. The particle size and size distribution of SiC nanoparticles

The photoluminescence (PL) spectrum of the SiC nanoparticles is depicted in Fig. 4. The PL spectrum show a broad emission band with peaks centered at 450nm (2.76 eV) and 470 nm (2.63eV), which are far above the band gap energy of the bulk β-SiC (2.30 eV). The peak at 450 nm is similar to the result for the SiC hollow nanospheres [15]. This indicates that the SiC nanoparticles have a size-confinement effect similar to SiC hollow nanospheres. The peak at 470 nm is close to that of the porous SiC [16]. Various emission wavelengths from SiC nanostructures indicate that the luminescence characteristic depends strongly on the size and structure of the SiC nanoparticles. The PL emissions of the SiC nanoparticles are due to the influence of size effects.

The diameter of the SiC nanoparticles is further measured by using a Zeta Potential and Particle Size Analyzer. The research laser light scattering photometer is equipped with a He-Ne solid-state laser at an operating wavelength of 675 nm and power at 25mW. Measurements of the average size of nanoparticles are performed at 25 °C with an angle detection of 90° in optically homogeneous quartz cylinder cuvettes. The SiC sample is ultrasonicated in ethanol for 5 min before test. The sample is measured ten times, and average serial data are calculated [14]. Fig. 3 shows he sample has dimensions in the range of 10-40 nm. The results from XRD, TEM and DLS all suggest that both samples are composed of small particles with narrow size distributions.
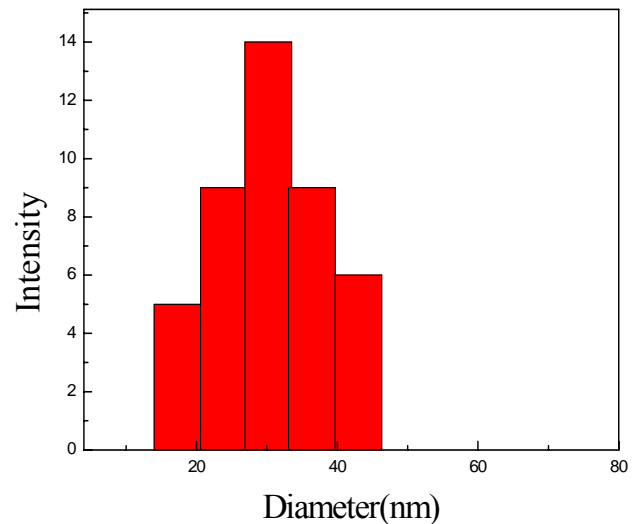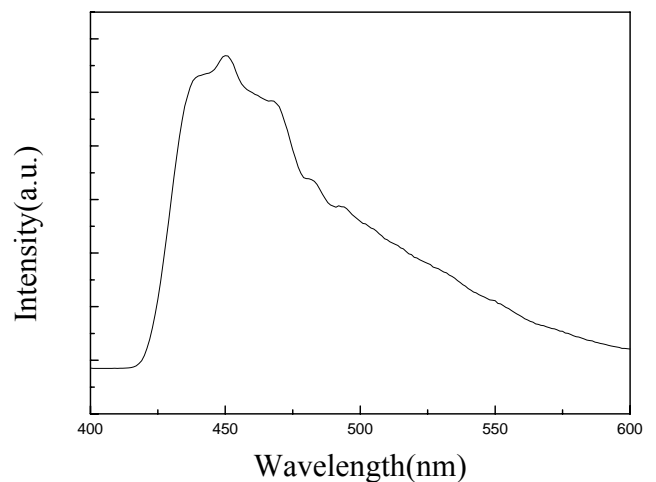

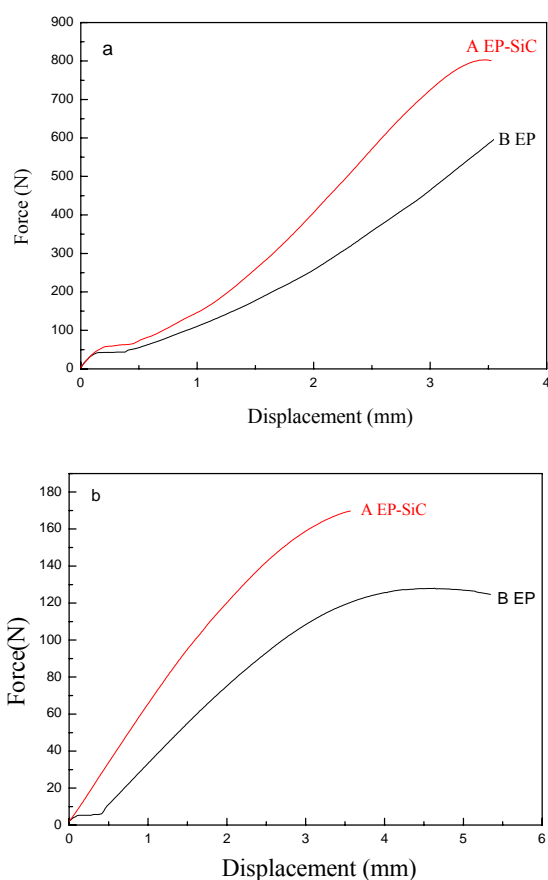*Fig.4. PL spectra of SiC nanoparticles*

Fig.5. Force-displacement curves of the epoxy resin composites for (a) tensile strength, and (b) bending strength.

3.2 Reinforcement effects

The reinforced epoxy resin is the most commonly used polymer matrix which has high mechanical strength, good thermal stability and low density, so they are developing fast as preferred materials in aerospace, automotive and marine industry [17]. To investigate the reinforcement effect of the SiC nanoparticles in epoxy resin, EP-SiC and EP are tested. Mechanical properties of all samples are tested by a SANS material testing machine. It can be seen from Fig. 5a and b, the composite rigid increase when epoxy resin is reinforced by the smaller sizes of SiC particles. The sample EP has the lowest average tensile strength and bending strength, 59.5 and 100.3 MPa. When epoxy resin is reinforced by SiC nanoparticles, its tensile and bending strength slight increase to 68.1 and 107.0 MPa, respectively. Because all other parameters in composites' preparation are the same, the differences in the strengths should come from the sizes

of SiC particles. In all composites, 0.5 wt% SiC is embedded in epoxy resin. There are more particles in the resin matrix when the sizes of SiC particles are small. The composites reinforced by the SiC nanopartcles with smaller size will have better mechanical properties because there are more particles in the composites acting as obstacles for destruction propagation.

4. Conclusion

The SiC nanoparticles were successfully prepared by a simple carbothermal method in which foam plastic wastes was employed as carbon precursors. The SiC nanoparticles had small sizes in the range of 10-40nm. These SiC nanoparticles had special photoluminescence spectra at room temperature due to their unique structures. The composites reinforced by the SiC nanopartcles with smaller size showed that the composites had better mechanical properties.

References

[1] Y. Li, P. S. Dorozhkin, Y. Bando, D. Golberg, Adv. Mater., **17**(5), 545 (2005).
[2] V. G. Pol, S. V. Pol, A. Gedanken, S. H. Lim, Z. Zhong, J. Lin, J. Phys. Chem. B, **110**(23), 11237 (2006).
[3] X. L. Luo, A. Morrin, A. J. Killard, M. R. Smyth, Electroanalysis, **18**(4), 319 (2006).
[4] Y. Katoh, A. Kohyama, T. Nozawa, M. Sato, J. Nucl. Mater., **329–333**, 587 (2004).
[5] X.L. Wu, S. J. Xiong, J. Zhu, J. Wang, J. C. Shen, P. K. Chu, Nano. Lett., **9**(12), 4053 (2009).

[6] J. Y. Fan, X. L. Wu, P. K. Chu, Prog. Mater. Sci., **51**(8), 983 (2006).

[7] I. K. Yu, J. H. Rhee, S. Cho, H. K. Yoon, J. Nucl. Mater., **386-388**, 631 (2009).

[8] V. Buschmann, S. Klein, H. Fuesz, H. Hahn, J. Cryst. Growth, **193**(3), 335 (1998).

[9] N. Herlin-Boime, J. Vicens, C. Dufour, F. T´en´egal, C. Reynaud, R. Rizk, J. Nanopar. Res., **6**(1), 63 (2004).

[10] N. Miskolczi, L. Bartha, F. Antal, Cs. Dudas, Talanta, **66**(5), 1264 (2005).

[11] S. Ucuar, S. Karagoz, T. Karayildirim, J. Yanik, Polym. Degrad. Stab., **75**(1), 161 (2002).

[12] Z. C. Ju, L. Q. Xu, Q. L. Pang, Z. Xing, X. J. Ma, Y.T. Qian, Nanotechnology, **20**(35), 355604 (2009).

[13] Y. J. Hao, J. B. Wagner, D. S. Su, G. Q. Jin, X. Y. Guo, Nanotechnology, **17**(12), 2870 (2006).

[14] G. Y. Wang, Z. P. Du, Q. X. Li, W. Zhang, J. Phys. Chem. B, **114**(20), 6872 (2010).

[15] G. Z. Shen, D. Chen, K. B. Tang, Y. T. Qian, S. Y. Zhang, Chem. Phys. Lett., **375**(1-2), 177 (2003).

[16] M. K. Lee, R. K. Peng, Appl. Phys. Lett., **62**(24), 3159 (1993).

[17] J. Cho, I. M. Daniel, D. A. Dikin, Compos. A, **39**(12), 1844(2008).

_____

*Corresponding author. wangdongh1978@163.com