

# Research on synthesis and optical testing of silicon carbide nanostructures

*As a wide band gap semiconductor material silicon carbide, with a stable chemical properties and good thermal conductivity of physical properties, in addition to the mechanical properties of the material also has a strong advantage. In order to explore more scientific and practical nanomaterials, this paper studies the preparation process of silicon carbide nanomaterials and the optical characterization of the materials, and analyzes the properties of the materials, 3C-SiC nanowires and 3C-SiC micron polyhedra, XRD patterns, infrared spectra, Raman spectra, thermogravimetric curves and room temperature photoluminescence spectra. By studying the experimental results of optical properties, it is concluded that silicon carbide products have great application prospect in optoelectronic devices.*

**Keywords:** Optical testing, silicon carbide nanostructures, XRD diffraction pattern, infrared spectrogram

## Introduction

(Lee, et al., 2014) points out *SiC* has excellent electronic, physical and chemical performance, *SiC* has extremely high mechanical strength and perfectly biocompatibility. has a wide range of applications in electronic field, machinery industry (Sreedhar, et al., 2017). Many scholars pay efforts on the research of *SiC* nanomaterial. (Olego, et al., 2012) points out that there are many problems existing in fabrication of one-dimensional *SiC* nanomaterials, such as the high temperature, large pressure and so on. The study of high-purity 1-D *SiC* nanomaterials' production and intrinsic properties are restricted by these problems. Based on the first principles method of density functional theory, (Shim, et al., 2017) studied the electronic structure properties of the core-shell structure d the  $\beta$ -*SiC* (15.0) carbon nanotube and  $\beta$ -*SiC* (16.0) carbon nanotube's core-shell construction. It is concluded that two kinds of structures shows metallic properties, and the metallicity is mainly contributed by the carbon nanotubes and the atoms on the surface of silicon carbon nanomaterial.

Silicon carbide is composed of IVA-IVA elements of the binary compound, with a diamond-like hexahedral crystal structure, with a high hardness and melting point (Li, et al., 2014). Pure silicon carbide is a colourless and transparent crystal, industrial silicon carbide because of its different types and content of impurities, and was light yellow, green, blue and even black, transparency varies with its purity (Keller, et al., 2015). The definition of nanomaterials is derived from the limitation of the material space scale, and the rule is defined as the dimension of at least one dimension in the range of 10<sup>-7</sup>m-10<sup>-9</sup>m (Motahari, et al., 2013). Therefore, the number of different dimensions of nano to meet the scale of the different, can be divided into one-dimensional, two-dimensional and zero-dimensional nanomaterials three categories.

Silicon tetrachloride is a liquid state in the normal temperature environment, and has a strong pungent odor. Because of its easy hydrolysis, resulting in the difficulty of preservation, in addition, silicon tetrachloride is a harmful substance (Mishra, et al., 2012). Based on this, the main direction of this paper is to reduce the reaction temperature to reduce the production cost. This paper mainly discusses  $CHI_3$  as carbon source to control the synthesis of different morphology and size, to lay the foundation for the studies on synthesis and properties of silicon carbide nanowires. In addition, the synthesis and properties of *SiC* polyhedron are also studied.

## Experimental

### 2.1 SYNTHESIS EXPERIMENTS OF 3C-SiC NANOWIRES

The chemical reagents used in the experiment are purely analytical. The precautions for taking metal sodium are to dry the surface kerosene with paper and cut off the surface oxide to increase the purity of the sodium metal. Other drugs other than sodium need not to be further purified before use. Typical experimental procedures are shown below.

Weigh the exact weight of the sodium metal (1.527g), silicon powder (0.500g) and (7.017g). After mixing drugs evenly, put it into stainless steel autoclave with a volume of 22ml. The autoclave is sealed and screwed and placed in a constant temperature oven (230°C) to react with 20h. When the reaction is over, take out the autoclave to cool to room

Messrs. Ai-Min Yang, Xiao-Lei Yang, Cui-Huan Ren, Xiang-yu Xu and Cheng-Ran Ren, Hebei Key Laboratory of Data Science and Applications, North China University of Science and Technology, Tangshan, 063000, China. Email: aiminyangtujian@163.com

temperature and collect the product. Remove the by-products and reactions with residual sodium, and silicon impurities. The products are washed by absolute alcohol and muriatic acid, then treated with 70% perchloric acid (reflux 3h under 150-180°C condition) and hydrofluoric acid/nitric acid mixed solutions. Washed products with distilled water 2-3 times, and then placed in 60°C vacuum drying oven to dried for 3h, and finally obtained the products of gray powder.

The experimental analysis tool of pure products are respectively Bruker D8 model X-ray powder diffractometer, VERTEX-70 model infrared spectrometer, NEXUS 670 FT-IR model Raman spectrometer, TGA/DTA851 model thermogravimetric tester and FLS920 model fluorescence spectrophotometer. The radioactive X-ray source in a diffractometer is Cu-K alpha radiation ( $\lambda = 1.5418\text{\AA}$ ), scanning angle  $2\theta$  range for  $[20^\circ, 80^\circ]$ . The xenon (Xe) lamp is used to test the luminescence of the sample at room temperature.

## 2.2 3C-SiC MICRO SCALE POLYHEDRON AND SYNTHESIS EXPERIMENT

The chemical reagents used in the experiment are all analytical pure. Precautions in when taking sodium metal, the precaution is that uses paper to absorb the surface kerosene and cut the oxide layer on the surface to increase the purity of the sodium metal. Other drugs other than sodium need not to be further purified before use. Typical experimental procedures are shown below.

Weigh the exact weight of the sodium metal (2.997g), silicon powder (1.000g) and  $\text{CHI}_3$  (14.030g). After mixing drugs evenly, put it into stainless steel autoclave with a volume of 22ml. The autoclave is sealed and screwed and placed in a constant temperature oven (230°C) to react with 20h. When the reaction is over, take out the autoclave to cool to room temperature and collect the product. Remove the by-products and reactions with residual sodium, and silicon impurities. The products are washed by absolute alcohol and muriatic acid, then treated with 70% perchloric acid (reflux 3h under 150-180°C condition) and hydrofluoric acid/nitric acid mixed solutions. Then oscillate solution containing the SiC sample

strongly for 2-3 minutes, and then remains stationary for 5 minutes. This process was repeated 3-5 times to collect the underlying products and dried 1h under 100°C conditions, and finally obtained the product of green micrometer polyhedron samples.

The experimental analysis tool of pure SiC products are respectively Bruker D8 model x-ray powder diffractometer, VERTEX-70 model infrared spectrometer, NEXUS 670 FT-IR model Raman spectrograph, TGA/DTA851 model gravimetric analyzer and FLS920 model fluorescence spectrophotometer. The radioactive X-ray source in a diffractometer is Cu-K alpha radiation ( $\lambda = 1.5418\text{\AA}$ ), scanning angle  $2\theta$  range for  $[20^\circ, 80^\circ]$ . The xenon (Xe) lamp is used to test the luminescence of the sample at room temperature.

## The discussion about experimental results

### 3.1 THE DISCUSSION ABOUT EXPERIMENTAL RESULTS ON 3C-SiC NANOWIRE SYNTHESIS

As shown in Fig.1(a), the silicon carbide product obtained after reaction at a temperature of 230°C for 20h was subjected a series of purified XRD diffraction patterns. By analyzing the XRD diffraction pattern shown in Fig.1(a), the following conclusions can be drawn. Each peak in the diffraction pattern can be measured into a cubic SiC cell, its lattice constant is consistent with standard powder diffraction data cards, and its lattice constant  $a = 4.549\text{\AA}$ . The diffraction peaks are strong and sharp, and other peaks are not detected in the XRD diffraction pattern, which proves that the sample has good crystallinity and high purity. The weak peaks marked with SF are caused by stacking faults.

The infrared spectrum of the final SiC nanowire is shown in Fig.1(b). By analyzing the infrared spectra shown in Fig. 1(b), the following conclusions can be drawn. There is a strong absorption peak at  $808\text{cm}^{-1}$ , which is the TO transverse photon vibration mode of the SiC bond, demonstrating the presence of silicon carbide in the product. No significant impurity peaks were detected in the infrared spectrum, and

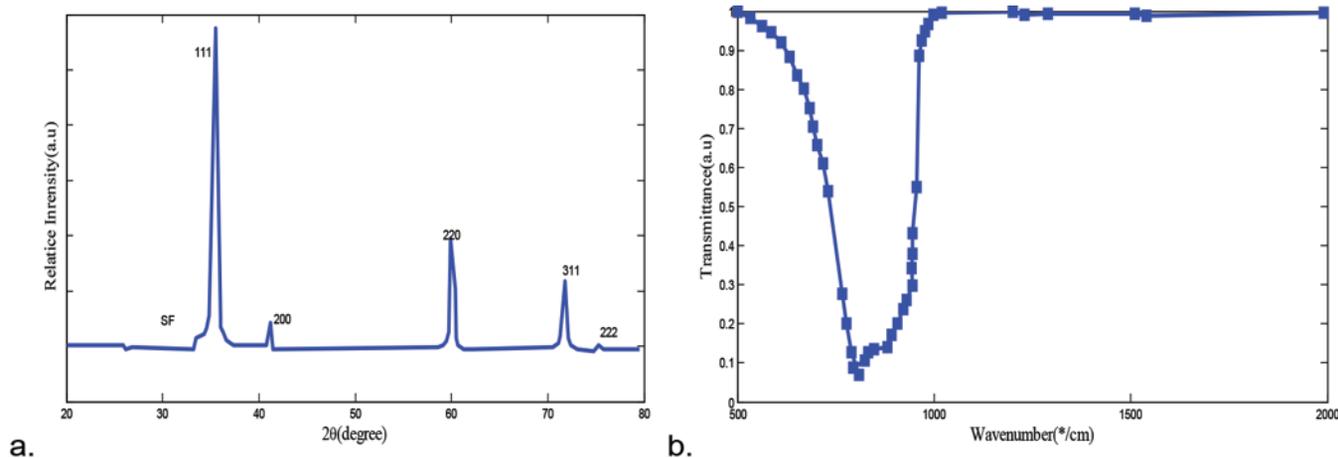


Fig.1 (a) XRD diffraction pattern and (b) the infrared spectrogram of 3C-SiC nanowire

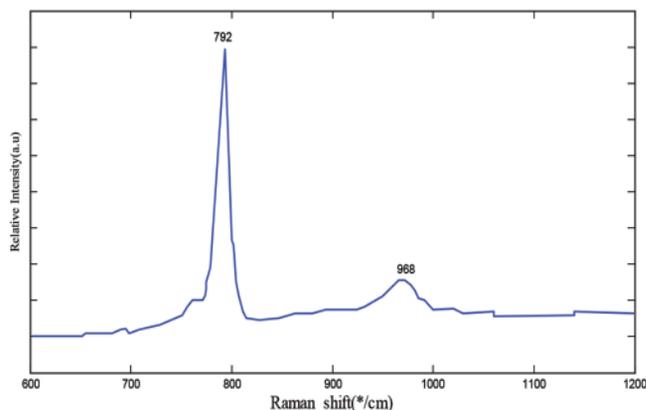


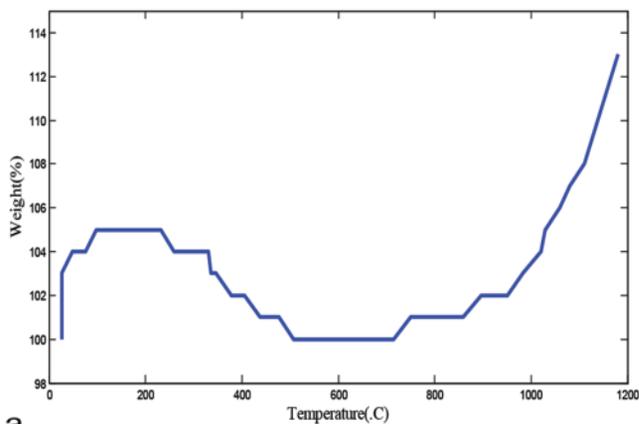
Fig.2 The spectrogram of 3C-SiC nanowire

the strong and sharp peaks proved that the silicon carbide product had high crystallinity.

Fig.2 shows the analysis of Raman spectrogram of silicon carbide nanowire product.

By analyzing the Raman spectrogram shown in Fig.2, the following conclusions can be drawn. The Raman spectra of the nanowire samples have two peaks at 792 cm<sup>-1</sup> and 968 cm<sup>-1</sup> corresponding to the transverse optical phonon mode (TO) and the longitudinal optical phonon mode (LO) of 3C-SiC. And has a redshift of 3-8 cm<sup>-1</sup> compared to the TO and LO phonon scattering peaks of bulk silicon carbide. The red shift of the above 3-8cm<sup>-1</sup> may be due to the quantum confinement effect or the stacking faults existing in the nanowires.

The Fig.3 shows the thermogravimetric curve of 3C-SiC sample. By analyzing thermogravimetric curve shown in Fig.3, the following inclusion could be drawn. The thermogravimetric curve has a weak peak at 40-400°C, which is thought to be due to drift of the TGA baseline, based on the effects of other factors such as gas adsorption. The further blank test thermogravimetric curve confirms that the weak peak at 40-400°C is indeed caused by instrument error. To remove the impact of instrument error, silicon carbide nano samples in the



a.

800°C below no significant weight gain, more than 800°C due to the sample cup oxidation significant weight gain.

Fig.4 shows the final room temperature photoluminescence of silicon carbide spectrogram, which excited at 220nm wavelength. From the figure we can clearly see that the sample has a strong emission peak at 360 nm, compared to blue and green emission peaks of 3C-SiC film that literature mentioned in [9]. The emission peak has a pronounced blue shift, this phenomenon may be due to differences in morphology of nanomaterials, or defects in materials or the quantum size confinement effect.

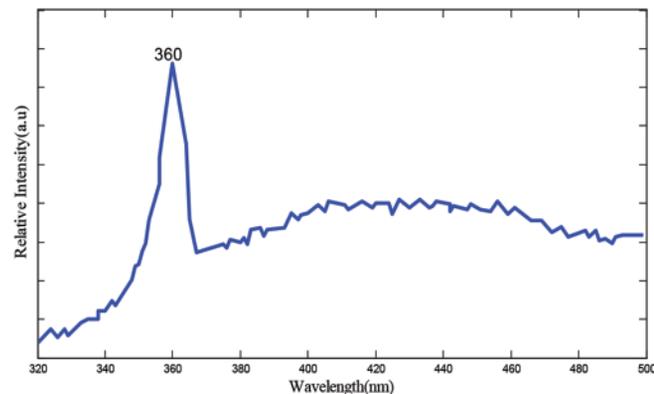
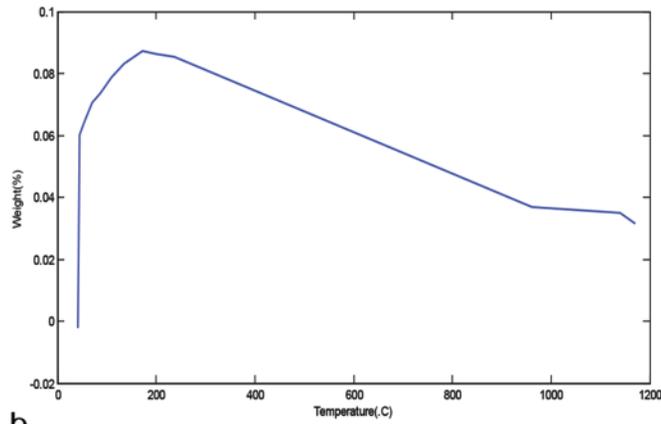


Fig.4 The room temperature photoluminescence of 3C-SiC sample spectrogram

### 3.2 THE DISCUSSION ABOUT EXPERIMENTAL RESULTS OF SYNTHESIS OF 3C-SiC MICRO SCALE POLYHEDRON

As shown in Fig.5, the silicon carbide product obtained after reaction of 20h at a temperature of 500°C has undergone a series of purified. Here are the XRD diffraction patterns.

By analyzing the XRD diffraction pattern shown in Fig.5, the following conclusions could be drawn. The peaks in the diffraction pattern can be indexed to the cubic SiC cell, and the lattice constant  $a = 4.357\text{\AA}$ . is consistent with the standard powder diffraction data card. The peak sample of the other phase does not detect the good crystallinity and



b.

Fig.3 The thermogravimetric curve of 3C-SiC sample (a) in the air (b) not in the air.

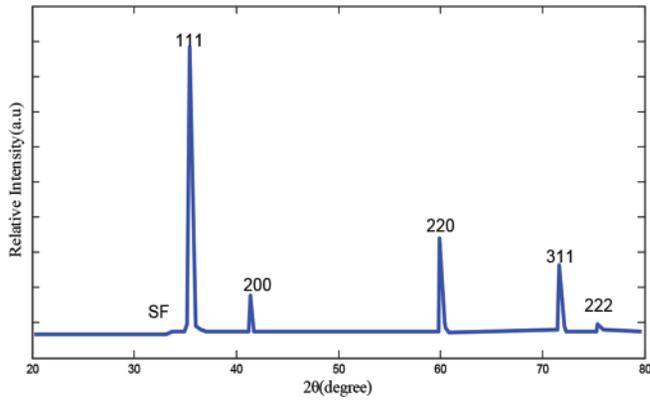


Fig.5 The XRD diffraction pattern

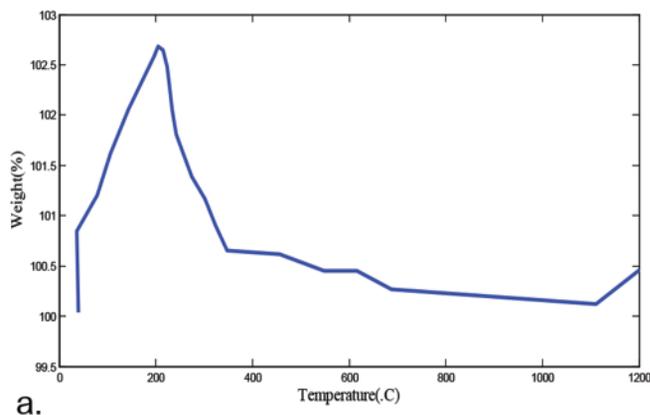
high purity. The weak peaks marked SF may be due to internal structural defects or twinning.

As shown in Fig.6, the product's infrared spectrum. By analyzing the infrared spectrum shown in Fig.6, the following conclusion could be drawn. There is a strong absorption peak at around  $875\text{cm}^{-1}$ , the peak is to transverse vibration mode photonic  $\text{SiC}$  bond, the vibration mode of the absorption peak at about  $1275\text{cm}^{-1}$  is weak  $\text{SiC}$  bond. Strong and sharp peaks indicate that the product has a high degree of crystallinity.

As shown in Fig.7, the Raman spectra of the samples were analyzed.

By analyzing the Raman spectrum shown in Fig.7, the following conclusions can be drawn. The product is cubic  $\text{SiC}$ , and the Raman spectra of the micronized silicon carbide polyhedral sample have two peaks at  $793\text{cm}^{-1}$  and  $969\text{cm}^{-1}$ , which correspond to the transverse optical phonon mode (TO) of  $3\text{C-SiC}$  and the longitudinal optical phonon mode (LO), which has a redshift of  $3\text{-}8\text{cm}^{-1}$  compared to the TO and LO phonon scattering peaks of bulk silicon carbide. The redshift of  $3\text{-}8\text{cm}^{-1}$  may be due to the quantum confinement effect or the stacking fault.

As shown in Fig.8(a), the thermogravimetric curves of the  $3\text{C-SiC}$  polyhedral sample.



a.

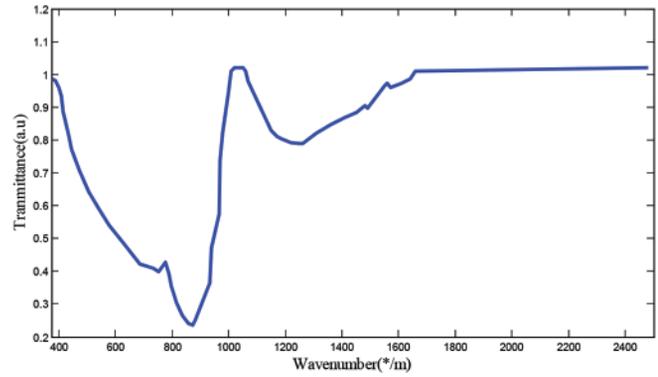


Fig.6 The infrared spectrum

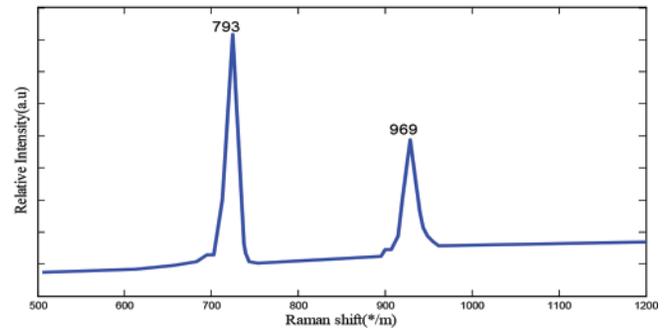
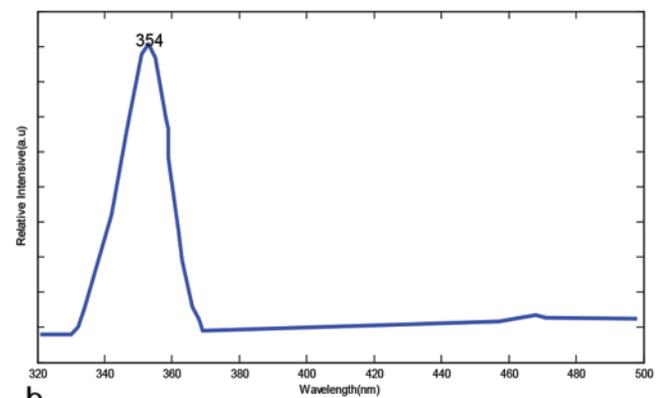


Fig.7 The Raman spectrum of  $3\text{C-SiC}$  micro scale polyhedron

By analyzing the TGA curve shown in Fig.8(a), the following conclusions can be drawn. The thermogravimetric curve has a weak peak at  $40\text{-}400^\circ\text{C}$ , which is thought to be due to drift of the TGA baseline, based on the effects of other factors such as gas adsorption. The further blank test of the thermogravimetric curve confirms that the weak peak at  $40\text{-}400^\circ\text{C}$  is indeed caused by the instrument error, removing the influence of the instrument error. Silicon carbide nano samples in  $1100^\circ\text{C}$  below no significant weight gain, more than  $1100^\circ\text{C}$  due to the sample cup oxidation obvious weight gain phenomenon, proved that the sample  $1100^\circ\text{C}$  above the air began to be oxidized. The thermal stability of this sample in the air is significantly enhanced, possibly due to the fact that the larger size of the sample has a smaller surface area that is more difficult to oxidize.



b.

Fig.8 (a) TGA diagram (b) photoluminescence spectra of  $3\text{C-SiC}$  micron polyhedron sample

As the Fig.8(b) shown, room temperature photoluminescence spectra of a silicon carbide polyhedral sample. As shown in the sample has a strong emission peak at 354nm, compared to the blue and green peaks method 3C-SiC film than the literature mentioned, the emission peak was observed the blue shift. This blue shift phenomenon may be due to differences in morphology of nanomaterials, caused by defects in materials or the quantum size effect.

### Conclusions

Through the discussion of the experimental results, the following conclusions are drawn. Powders produced by the reaction of 230°C with 20h in a autoclave in the presence of silicon powder as silicon source, CH<sub>4</sub> as carbon sources, sodium metal as reducing agent. When the reaction temperature is 500°C, the powders produced by the reaction with 20h in a autoclave in the presence of silicon powder as silicon source, CH<sub>4</sub> as carbon sources, sodium metal as reducing agent, the product is mainly 3C-SiC micron polyhedron. Thermogravimetric analysis shows that 3C-SiC micron polyhedron has superior stability, and micron scale polyhedron is oxidized until the temperature reach up to 1100°C, indicating that 3C-SiC polyhedron can be used to prepare high temperature resistant equipment. In photoluminescence test, silicon carbide nanowires and micron polyhedron respectively have strong emission peaks at 360nm and 354nm, therefore we can see that SiC products have great potential applications in optoelectronic devices.

### Acknowledgments

This work is supported by the National Natural Science Foundation of China (no. 51674121), by the National Natural Science Foundation of Hebei Province (no. E2017209178), Science and technology project of Hebei province (no. 15214104D), the National Natural Science Foundation of

Hebei Education Department (no. QN2016088), and Graduate Student Innovation Fund of North China University of Science and Technology, Graduate Student Innovation Fund of Hebei Province (2017S03, CXZZSS2017071).

### Reference

1. Lee S.K, Kim YC., Kim C.H., (2014): Microstructural development and mechanical properties of pressureless-sintered SiC with plate-like grains using Al<sub>2</sub>O<sub>3</sub>: Y<sub>2</sub>O<sub>3</sub> Additives. *J. Mater. Sci.* 29(20), 5321-5326.
2. Olego D., Cardona M., (2012): Temperature dependence of the optical phonons and transverse effective charge in 3C-SiC. *Phys. Rev. B.* 86(25), 3889-3890.
3. Shim H.W., Kim K.C., Seo Y.H., (2017): Anomalous photoluminescence from 3C-SiC grown on Si (111) by thermal chemical vapor deposition. *Appl. Phys. Lett.* 121(70),1756-1757.
4. Li P., Xu L.Q., Qian Y.T., (2014): Selective synthesis of 3C-SiC hollow nanospheres and nanowires. *Cryst. Growth Des.* 12(8), 2430-2431.
5. Keller N., Pham-Huu C., Roy R., Ledoux M.J., (2015): Influence of the preparation conditions on the synthesis of high surface area SiC for use as a heterogeneous catalyst support. *J. Mater. Sci.* 65(34), 3189-3190.
6. Mishra A, Buerlp P., (2012): Small molecule organic semiconductors on the move: Promises for future solar energy technology, *Angew ChemInt Ed.* 51(15), 2020-2067.
7. Motahari, A. Omrani, A.A. Rostami, M. Ehsani, (2013): Preparation and characterization of a novel epoxy based nanocomposite using tryptophan as an eco-friendly curing agent, *Thermochim Acta*, 574(574), 38-46.
8. Sreedhar B., Reddy P.S., (2017): Sonochemical synthesis of 1, 4-disubstituted 1, 2, 3- triazoles in aqueous medium, *Synthetic Communications*, 37(12), 805-812.

## Indian Journal of Power & River Valley Development

*Forthcoming International Conference on*

### **ADVANCES AND CHALLENGES IN SUPERCRITICAL POWER GENERATION TECHNOLOGY**

The Journal is planning to host an international conference sometime towards the end of this year at Kolkata. For details, please contact

*The Editor & The Organising Secretary  
International Conference*

*Indian Journal of Power & River Valley Development  
(Conference Secretariat)*

Mob: +91 9239384829 / +91 8479919829

E-mail: [bnjournals@gmail.com](mailto:bnjournals@gmail.com) / [pradipchanda@yahoo.co.uk](mailto:pradipchanda@yahoo.co.uk) • Web: [www.ijprvd.info](http://www.ijprvd.info)