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ISSN: 0976-3031

International Journal of Recent Scientific Research
Vol. 6, Issue, 2, pp.2630-2633, February, 2015

International Journal of Recent Scientific Research

RESEARCH ARTICLE

A NOVEL SONOCHEMICAL SYNTHESIS OF NANO-CRYSTALLINE SILICON CARBIDE CERAMIC POWDER AND ITS CHARACTERIZATION

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ARTICLE INFO

Article History:

Received 5th, January, 2015
Received in revised form 12th, January, 2015
Accepted 6th, February, 2015
Published online 28th, February, 2015

Key words:

Silicon Carbide, Ultrasound energy, Sonochemical method, Characterization

ABSTRACT

Nano-crystalline Silicon Carbide ceramic powder has been successfully synthesized by reacting the silicon dioxide amorphous powder with activated carbon in the molar ratio 1:3(SiO_2:C) using ultrasound energy, this method is generally referred as sonochemical method, which is a greener, facile, and safer method. The resultant material was characterized by using X-ray diffraction (XRD) to know the phase composition and average crystal size, Specific surface area of the synthesized SiC could be calculated by Brunauer–Emmett–Teller (BET) equation from the adsorption data.

INTRODUCTION

Silicon carbide is an important non-oxide ceramic which has diverse industrial applications.

In fact, it has exclusive properties such as high hardness and strength, chemical and thermal stability, high melting point, oxidation resistance, high erosion resistance, etc. All of these qualities make SiC a perfect candidate for high power, high temperature electronic devices as well as abrasion and cutting applications. Quite a lot of works were reported on SiC synthesis since the manufacturing process initiated by Acheson in 1892 (National, I. & Recherche, D., 2008) The outstanding oxidation behavior of SiC at high temperatures, even in harsh environments, makes it a candidate material for motor and gas turbine parts (Riedel & Gabriel 2000) Industrially used low cost silicon carbides are produced by reactions between silica or silicon and carbon (usually cokes) at 1400 – 1800 °C. This process involves gas-solid reactions and produces coarse particles so that they must be ground and classified in required sizes. Even after such processing, the resulting silicon carbide powder is not adequate to the relevant application [(Konno *et al*. 2004) (Ren *et al*. 2004)] some of the most important recent aspects of sonochemistry have been its applications in the synthesis and modification of both organic and inorganic materials (Suslick KS *et al*. 1993-97). High-intensity ultrasound can induce a wide range of chemical and physical consequences. The chemical effects of ultrasound fall into three areas: homogeneous sonochemistry of liquids, heterogeneous sonochemistry of liquid-liquid or liquid-solid systems, and sonocatalysis (which overlaps the first two). Applications of ultrasound to materials chemistry are found in all of these categories. Physical effects of high-intensity ultrasound, which often have chemical consequences, include enhanced mass transport, emulsification, bulk thermal heating, and a variety of effects on solids. The chemical consequences of high-intensity ultrasound do not arise from an interaction of acoustic waves and matter at a molecular or atomic level. Instead, in liquids irradiated with high-intensity ultrasound, acoustic cavitation (the formation, growth, and collapse of bubbles) (Leighton TG. 1994) provides the primary mechanism for sonochemical effects. During cavitation, bubble collapse produces intense local heating, high pressures, and very short lifetimes; these transient, localized hot spots drive high-energy chemical reactions. As described in detail elsewhere [(Suslick KS *et al*. 1999) (McNamara WB III *et al*. 1999)], these hot spots have temperatures of »5000±C, pressures of about 1000 atm, and heating and cooling rates above 1010 K/s. Thus, cavitation serves as a means of concentrating the diffuse energy of sound into a unique set of conditions to produce unusual materials from dissolved (and generally volatile) precursors. Chemical reactions are not generally seen in the ultrasonic irradiation of solids or solid-gas systems. Several methods have been reported to synthesize SiC namely sol-gel(Proc. NATO) , thermal plasma (Li, J., J. Tian and L. Dong, 2000), milling (Pierre, A.C., 1991) and carbothermal reduction of SiO_2 (Ronald William, R.W., 1989) However, to the best of our

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knowledge, there is no report on the synthesis of nano crystalline silicon carbide powder by sonochemical method. In this present work we report the ultrasound assisted synthesis of nano crystalline silicon carbide powder.

Experimental

A novel method is adopted to synthesize silicon carbide nano powder. All chemicals were analytical grade and used without further purification. 1g of SiO_2 amorphous powder used as a silicon source was dissolved in 30ml of ethanol then added 3g of activated carbon as a carbon source followed by 50ml of distilled water. Above solution was kept under magnetic stirrer for 15 min at 250 rpm. After that mixing, sonicate the solution for one hour by using direct probe sonicator. Process parameters were mentioned below. Sample was dried at 180^{o}C for 1 hour in the hot air oven. Table 1 shows the pH value of the solution, initially it was alkaline in nature after 1 hour Sonication pH of the solution partially turned to neutral value. Table 2 shows the temperature of solution before and after Sonication.

Sonication parameters

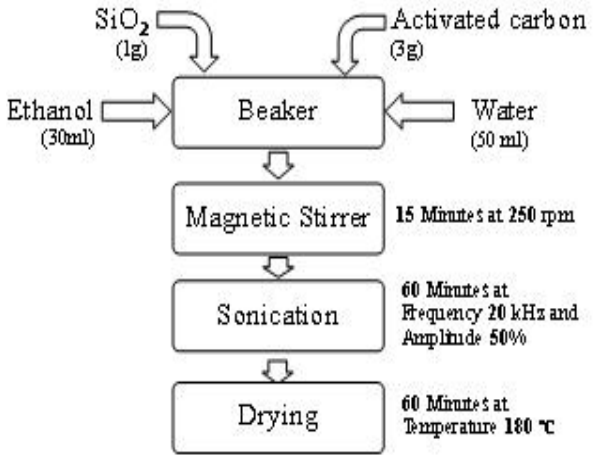
1. Frequency 20kHz
2. Pulse : 05 sec ON - 02 sec OFF
3. Amplitude : 50%
4. Time : 60min
5. Set point temperature 70^{o}C
6. Probe temperature 25^{o}C

Table 1 pH of solution

Initial	**8.95**
Final	7.39

Table 2 Temperature of solution

Initial	**30ºC**
Final	72^{o}C



The synthesized SiC nano-powders were characterized by using X-ray diffraction from which we found out the phase composition and crystallite size of the particles. We measured the specific surface area of the synthesized SiC nano powder by nitrogen adsorption using BET method. The mean particle size was calculated from measured specific surface area.

Reaction involved in this process

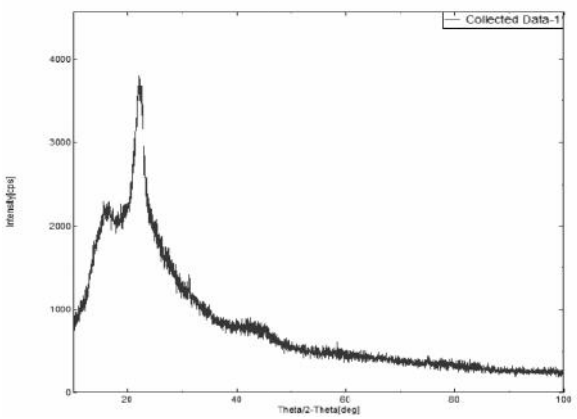
$SiO_2+3C \longrightarrow SiC+2CO$

Ultrasound

RESULTS AND DISCUSSION

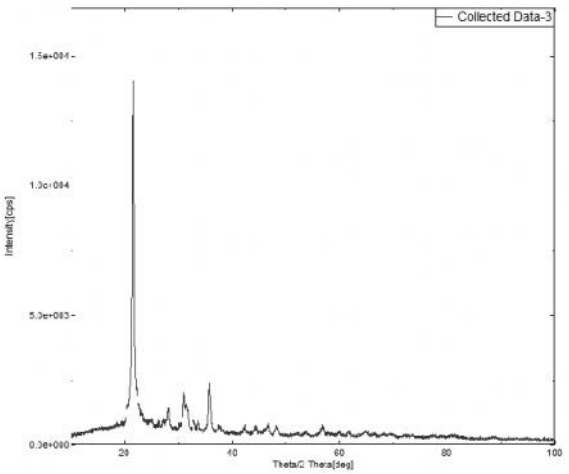
Phase composition and crystallite size characterization

XRD pattern of the synthesized powder confirms the presence of silicon carbide, sharp peak found at diffraction angle (2θ) 21.56^o, no other major peaks were found. It confirms the amorphous nature of synthesized SiC powder shown in Graph 1.



Graph 1 X-ray diffraction pattern of synthesized SiC nano-powders, dried at 180^o C for 1 hours.

After one hour heat treatment at 850^{o}C, XRD pattern clearly confirms the formation of β-SiC The sharp peaks of synthesized SiC nano powder found at diffraction angles (2θ) of 21.56^o, 28.07^o, 31.63^o, 35.68^o, 42.21^o, 48.22^o, 56.67^o (2θ are calculated with wavelength=1.54059) It confirms the crystalline nature of β-SiC shown in Graph 2.



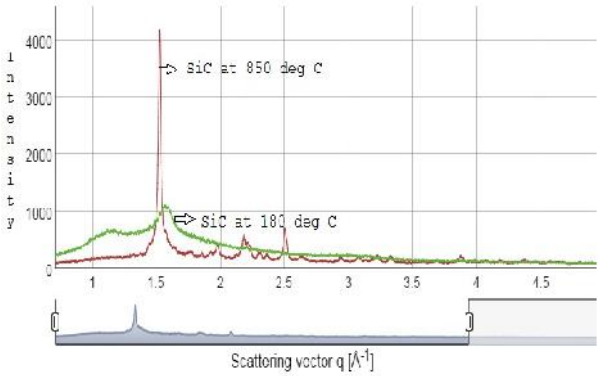
Graph 2 X-ray diffraction pattern of synthesized SiC nano-powders after heat treatment at 850^{o}C.

The average crystallite size of the synthesized nano crystalline SiC powder was calculated using Scherrer equation.

$$d = \frac{0.9\lambda}{\beta\cos\theta} \quad (1)$$

Where d is the average crystallite size, λ is the wavelength of the X-rays (0.154 nm), β is the full-width at half peak height of the diffraction peak (FWHM) and θ is the Bragg's angle. The smallest diffraction angle is 10.78^o (2θ=21.56^o). In our case β=0.3221(radians), θ=10.78^o, we calculated that average crystallite size of synthesized nano crystalline SiC powder was 25 nanometers from eq 1.

Comparison spectra report of the SiC at different temperature

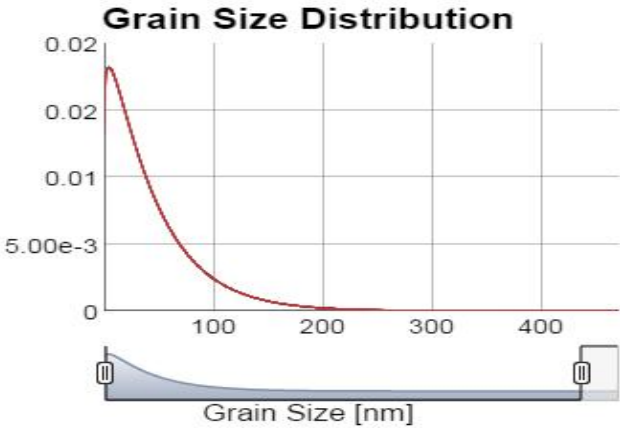


Graph 3 Comparison spectra of SiC at 180^{o}C and at 850^{o}C.

From the above comparison spectra we concluded that synthesized SiC nano powder was amorphous in nature initially at 180^{o}C. After the heat treatment of synthesized SiC nano powder at 850^{o}C clearly shows the crystalline nature.

Grain size distribution

We have done the grain size distribution analysis using Nano powder XRD Processor software from the data obtained during the XRD analysis which are diffraction angles (2theta) and intensity of the peaks.



Graph 4 Grain size distribution curve.

Average size distribution of synthesized SiC nano powder found to be 35-40nm.

Surface area and pore volume

Specific surface area and pore volume of the synthesized SiC nano powder was calculated by Brunauer–Emmett–Teller (BET) equation from the adsorption data. Assumption has been made that synthesized SiC nano particles were spherical in shape; the mean particle size of the synthesized particles can be obtained from the following equation.

$$d = \frac{0.9\lambda}{\beta cos\theta} \qquad (2)$$

Where d is the mean particle size of the synthesized SiC nanopowder, ρ is the density of SiC (3.2g/cm^3 is the theoretical density of SiC), S_{BET} is the specific surface area of the synthesized SiC nanopowder. The specific surface area was 878.92m^2/gm and the mean particle size (d) of the synthesized SiC nano particle was 2-3nm. Pore volume of the synthesized SiC was 0.5402cc/gm.

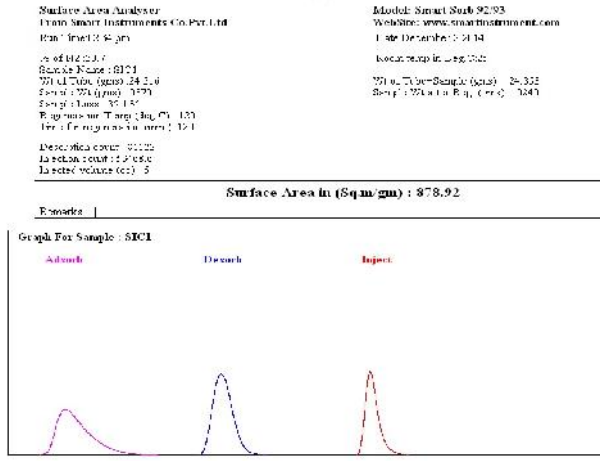


Fig 1 BET surface area analysis report.

CONCLUSIONS

The present work establishes that Nano-crystalline Silicon carbide powders are easily obtained by reacting silicon dioxide powder with activated carbon in the molar ratio 1:3 by using a novel sonochemical method. This process does not require any extra chemicals except that SiO_2 and activated carbon. XRD pattern of synthesized SiC nano powder and grain size distribution analysis showed the average particle size was 25-40nm. BET surface area analysis confirmed that synthesized SiC powder had a high specific surface area about 878.92m^2/gm. The synthesized powder consisted of SiC with free carbon, so free carbon was removed by the heat treatment at 850^{o}C for 1 hour.

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How to cite this article:

Kishor Kumar M J and S. Sivasankaran. A novel sonochemical synthesis of nano-crystalline silicon carbide ceramic powder and its characterization. *International Journal of Recent Scientific Research Vol. 6, Issue, 2, pp.2630-2633, February, 2015*

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