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Rapid carbothermic synthesis of silicon carbide nano powders by using microwave heating

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Abstract

This paper reports an improved procedure for synthesis of silicon carbide nanopowders from silica by carbothermic reduction under fast microwave-induced heating. The powders have been prepared by direct solid-state reaction in a 2.45 GHz microwave field in nitrogen atmosphere after 40 h milling. For the first time, the formation of silicon carbide (β-SiC) as a major phase can be achieved at 1200 °C in 5 min of microwave exposure, resulting in nano sized particles ranging from 10 to 40 nm under optimized synthesis condition. The Rietveld quantitative phase-composition analysis confirmed that the major SiC polytype is cubic SiC (β-SiC) with 98.5(4) weight fraction and the remained is minor hexagonal SiC polytypic (α-SiC) phases. Therefore this method is the most efficient one for SiC powder synthesis in terms of energy and time saving as well as preparation of SiC nano powders.

Keywords: A. Microwave processing; A. Milling; B. X-ray methods; D. SiC; Nano powders synthesis

1. Introduction

Silicon carbide (SiC) is an important structural ceramic and a candidate for high temperature applications due to its strength, hardness, corrosion resistance, low thermal expansion coefficient and high thermal conductivity. Because of this set of attractive properties, it possesses great potential for industrial and engineering applications, such as abrasives, semiconductors, and composite reinforcements.[1–3] SiC powders can be produced through many methods. For instance, some authors have reported to synthesize SiC from silicon.[4,1,5] However, most of SiC powders produced nowadays are manufactured using the Acheson process.[6] This process is a several hours-long carbothermic reaction of SiO_2 at temperatures around 2200–2400 °C. Carbothermic reduction of silica is known to be a simple and economical process for the synthesis of SiC powders.[7] Due to the high reaction temperatures and long reaction times of the process, the produced powders have large particle size and consist of mostly alpha-phase SiC. Therefore, extensive milling is needed to convert the as-synthesized products to sinterable powders with particle sizes ranging from a few micrometers to submicrometer. However, the post milling process is inevitably accompanied by the contamination from the milling media. Thus, the drawbacks of this industrial production process lie in high-energy consumption and low product purity. So, this conventional method for the synthesis of pure SiC powders involves many steps and is an energy-intensive process.[8]

The overall reaction of formation for SiC through carbothermic reduction is normally written as:

$$SiO_2(s) + 3C(s) = SiC(s) + 2CO(g) \tag{1}$$

This reaction is strongly endothermic with $\Delta H^\circ_{298} = 618.5\ \mathrm{kJ\ mol^{-1}}$ and the SiC formation begins at a temperature of about 1500 °C. Also, above 1600 °C, the stable phases are SiC and CO(g).[9,10] As the reactants exist in separate

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particles, the extent of this reaction is limited by the contact area between the reactants and the distribution of the carbon within the silica. These limitations result in a silicon carbide product that contains unacceptable quantities of unreacted silica and carbon. Although the reaction (1) involves both reactants in solid form, the carbothermic reduction of silica consists of gas–solid reactions in two steps. The most accepted reaction mechanism for the gas–solid carbothermic synthesis of SiC at low temperatures is given in Eqs. (2) and (3)[11,12]:

$$SiO_2(s) + C = SiO(g) + CO(g) \tag{2}$$

$$SiO(g) + 2C(g) = SiC(s) + CO(g) \tag{3}$$

At 1600 °C or higher, the reaction is believed to be a SiO(g)–CO(g) gas–gas reaction, favoring whisker formation. Transition metals (especially Fe, Co, Ni) have been shown to act as catalysts in the gas–gas reaction to form whisker via VLS (vapor–liquid–solid) mechanism. At 1500 °C or lower, the reaction is attributed to the SiO(g)–C(s) gas–solid reaction, favoring powder formation.[13,14]

There is significant interest in the synthesis of nanostructured SiC including nanospheres, nanowires, nanorods and so on as novel functional materials for nanoscale engineering.[15,16] A great variety of alternate methods like sol–gel,[17] plasma,[18,19] laser[15] and microwave[20–23] have been reported in the literature for the synthesis of fine SiC powders.

Microwave heating is a promising preparation method that may resolve the problems arising from conventional synthesis methods. In microwave heating, energy is delivered to materials via molecular-level interactions with the electromagnetic field.[24]

In this case, materials couple with microwaves and absorb the electromagnetic energy volumetrically and transform it into heat. In other words, heat is generated at each point of the material by the dissipation of MW energy as a consequence of the interaction of the MW field with the molecular structure (phonons) of the material.[25] This is different from conventional methods in which heat is transferred between objects by the mechanisms of conduction, radiation and convection. In conventional heating, the material surface is first heated followed by the heat flow moving inward. This means that there is a temperature gradient from the surface to the inside. However, microwave heating generates first heat within the material and then heats the entire volume. This heating mechanism is advantageous due to uniform, rapid, and volumetric heating, high reaction rates and selectivity, dramatically reduced reaction times, and high product-yields. This method can save energy and time, thereby reducing the costs of final products.[26]

In this paper, we report the solid-state synthesis of β-SiC nano powders by carbothermic reduction of SiO_2 with graphite in a microwave furnace. The effect of temperature and time of microwave exposure on the synthesized SiC powder characteristics have been discussed.

2. Experimental procedure

Mixtures of amorphous nano silica (Degussa, purity >99.5%, particle size ≤ 20 nm) and crystalline graphite powders (Fluka, 99.9% purity, particle size ≤ 100 μm) in the stoichiometric ratio according to the reaction (1) were used as the starting materials. The mixture of powders was milled at room temperature using a planetary high-energy ball mill for 40 h. The ball milling media was a hardened chromium steel vial (40 mm diameter and 40 mm height) with three hardened carbon steel balls (10 mm). The vial is fixed onto a rotating disc and rotates in the opposite direction to that of the larger platform. The ball-to-powders weight ratio and the rotational speed of vial were 20:1 and 500 rpm, respectively. The powders and milling balls were loaded into the vial, which was filled with Ar gas to avoid air contamination. After milling, in order to increase contact between nano silica and graphite, pellets were made by using uniaxial press at 127 MPa in a 13 mm diameter steel die.

Table 1
Synthesis conditions for different samples.

Sample number	Microwave condition	
	Temperature (°C)	Time (min)
1	1450	60
2	1350	45
3	1350	30
4	1350	15
5	1250	15
6	1250	5
7	1200	5
8	1150	15
9	1150	5

Microwave heating was carried out in a multimode 2.45 GHz microwave furnace (Autowave™ VIS-300-01D).[27] This model is operated through a computer interface that is controlled using a LabVIEW™ executable program, which allows to generate an adjustable microwave power from 0 kW to 3 kW continuous wave, and to register temperature or supplied power schedules running in the synthesis processes.

The specimen and four SiC susceptors were placed inside an inner, microwave transparent chamber of alumina fiberboat for heat containment. The temperature of the specimen inside the microwave furnace was measured by using an infrared fiber optic pyrometer (Mikron-M770S/780) positioned directly above the sample at a distance of approximately 50 cm. Temperature measurement starts at 750 °C. The sample was viewed through a tube on top of the furnace cavity and a hole in the roof of the housing. Prior to a run in the microwave furnace, the cavity was pumped down and then backfilled with pure nitrogen for three times.

Different synthesis experiments were carried out in order to obtain the optimum process conditions (temperature and reaction time), which result in the lower particle size. Conditions used in the different synthesis experiments are summarized in Table 1. After the microwave treatment, the products were ground and calcined in air at 800 °C for 1 h to remove any excess carbon. In order to remove the probably unreacted SiO_2 or undesired phases, the as-prepared products were leached in a dilute mixture of nitric acid (65% HNO_3) and hydrofluoric acid (40% HF) (HNO_3/HF = 1:3, v/v) for 24 h to get the purified

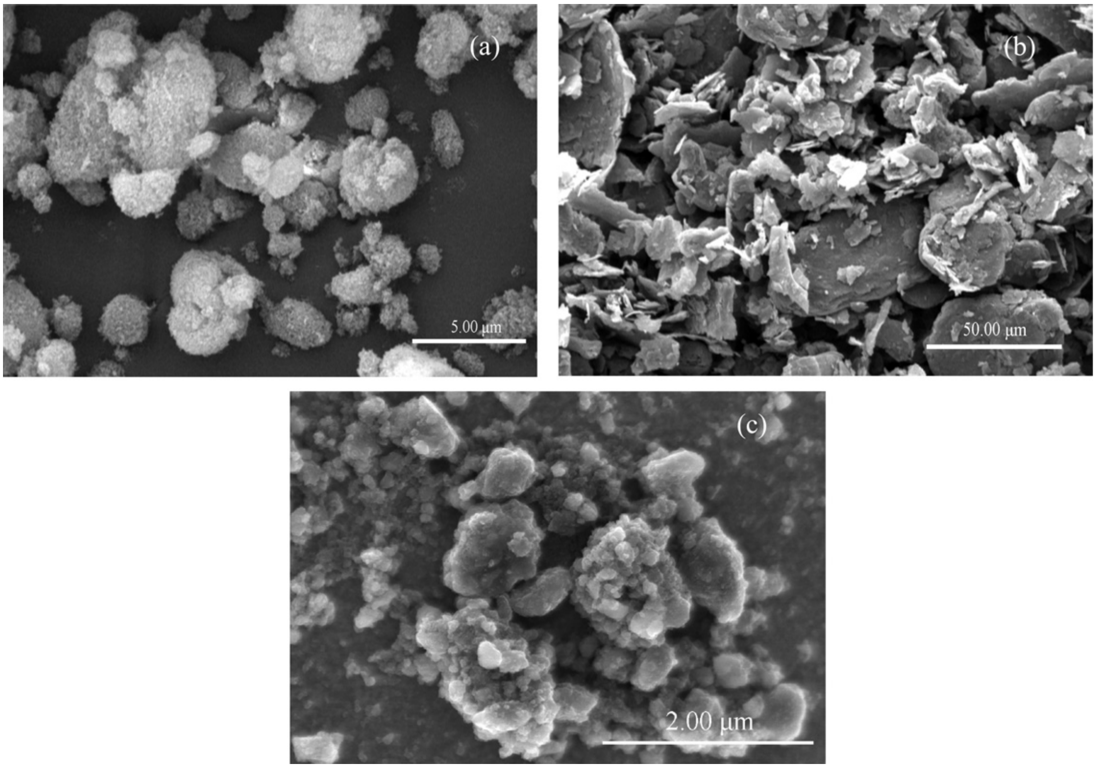

Fig. 1. SEM micrographs of (a) as-received nano silica powders, (b) as-received graphite powders and (c) stoichiometric mixtures of them after 40 h milling.

SiC products.[28] The solution was filtered after leaching and the purified products were washed by distilled water several times to eliminate extra acid until the pH value was about 7. Finally, the products were dried on a hot plate at 80 °C.

X-ray diffractometry (PANalytical X'Pert Pro diffractometer) was used to analyze the structural changes of powders before and after microwave treatment. Diffraction patterns were recorded using Cu Kα radiation over a 2θ range of 20–80° and a position-sensitive detector using a step size of 0.05° and a counting time of 1 s per step. In order to determine the crystallographic data and phase fractions of SiC polytypes, Rietveld refinement was performed for the best sample after leaching. For the Rietveld refinement, the XRD data were collected over a 2θ range of 20–120° with a step width of 0.033° and a counting time of 5 s per step. In this study, we used the version 3.5d of the Rietveld analysis program FULLPROF,[29] assuming the pseudo-Voigt function to describe the peak shapes. The refinement protocol included the background, the scale factors, and the global-instrumental, lattice, profile and texture parameters. The crystallite sizes of the powders were estimated from the (1 1 1) peaks of XRD patterns correspond to (2θ) of 35.61° using the Scherrer formula.

A scanning electron microscope (Philips XL30) and a high resolution scanning electron microscope (Hitachi S4800 SEM-FEG) and a transmission electron microscope (Philips CM200) were utilized to evaluate the morphology and particle size of as-received and the product powders. The synthesized powders were also tested for particle size analysis using a MALVERN Zetasizer 2000 instrument. For this investigation, the samples were sonicated for 1 h in a dilute NH_4OH solution in order to obtain stable suspensions. The size distributions were recorded in the main population number of particles. BET specific surface areas were measured by nitrogen adsorption at liquid nitrogen temperature (Micromeritics ASAP 2000). Before analysis, the samples were degassed 2 h at 150 °C under vacuum conditions.

3. Results and discussion

Fig. 1 shows the morphology of the initial powders and stoichiometric mixtures of them after 40 h milling. The morphology of the initial SiO_2 powders was spherical and strongly agglomerated and the graphite particles were angular and flaky in shape. It is obvious that after milling morphology of starting powders are changed and there are homogeneous mixed powders.

Fig. 2 shows the XRD patterns of as-received nano SiO_2 and graphite powders and stoichiometric mixtures of them after 40 h

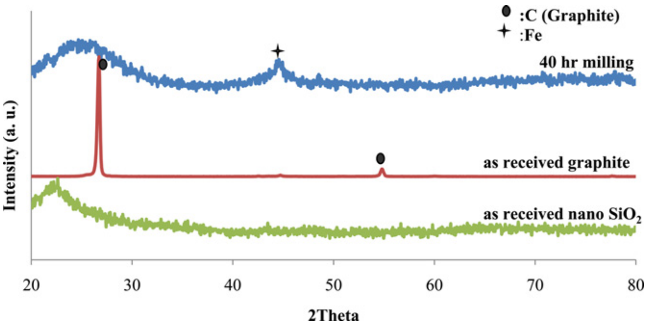

Fig. 2. XRD patterns of as-received nano SiO_2 and graphite powder and mixtures of them after 40 h milling.

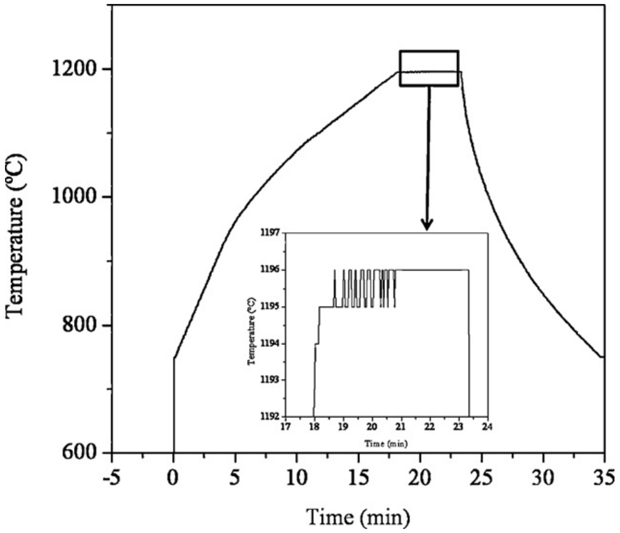

Fig. 3. Plot of temperature versus time for synthesized SiC in microwave field at 1200 °C for 5 min. The inset shows the temperature oscillation during the holding time at maximum temperature.

milling. The XRD pattern after 40 h milling shows the amorphous halo between 20 and 30° and no trace of SiC peaks. So ball milling up to 40 h had no effect on initiation of the reaction (1) due to positive $\Delta H°$. However, it has effects on destroying the crystalline structure of graphite and increasing the defect densities. Also, a trace of Fe contamination was observed. Therefore, the heat treatment of samples after milling was required to perform the carbothermic reaction of silica. Milling provides mechanical activation and the thermal activation energy required for the reaction between SiO_2 and C decreases to the temperature below 1600 °C.

Fig. 3 shows a plot of temperature versus time for one of the sample under microwave radiation. Microwave furnace is operated through a computer interface, which runs a temperature control mode through a feed-back microwave power. So, when the temperature is reached to maximum temperature, it kept stable by self-adjustable microwave power by feedback source. It is observed from inset that, any emissivity loss of the sample is compensates by increase of microwave power to keep temperature at the chosen fixed value. In fact, the controlling temperature software eliminates the possible heat loss by additional power supply. As a consequence of the feed-back operation mode, the hold temperature oscillates slightly around the mean value, being the oscillation not higher than 2°.

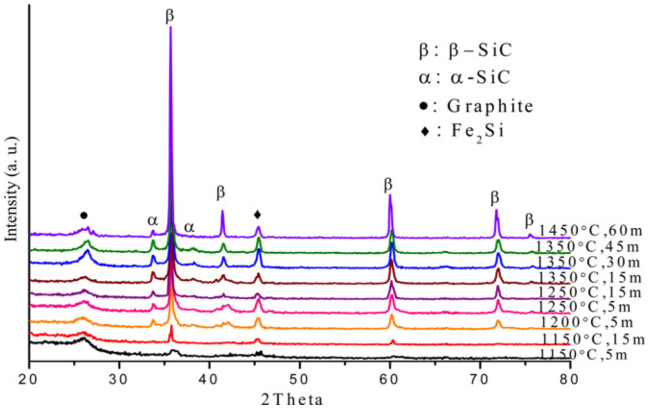

Fig. 4. XRD patterns of synthesized SiC samples at different temperatures and times under a microwave field.

XRD patterns of synthesized SiC powders at different temperatures and times under a microwave field are shown in Fig. 4. It can be noted for samples synthesized at temperatures ranging from 1450 °C to 1200 °C, the SiC is the major phase coexisting with small amount of Fe_2Si and graphite. No trace of SiO_2 peaks was found. However, for samples synthesized at lower temperature (1150 °C), the yield of synthesized SiC is low and the SiC synthesis is not completed. In this temperature, unreacted silica is remained as amorphous phase with a halo between 20 and 30° and does not crystallize to cristobalite or quartz. This is in contrast with the report by Carassiti et al.[23] By using silica in microwave synthesis of SiC, they observed the silicon dioxide phases (both quartz and cristobalite) as the major phases with silicon carbide and they illustrated that SiC synthesis was not completed using SiO_2 as starting material. The presence of Fe as the result of contamination during milling caused Fe_2Si phase formation after heat treatment.[30] Transition metals (especially Fe, Co, Ni) have been shown to act as catalysts and improved SiC synthesis. The positive effect of Fe on the SiC synthesis seems to be in the enhancement of the SiO(g) formation. When the concentration of SiO(g) increases, the second step of SiC formation (Eq. (3)) proceeds readily to the right. Therefore, it permits to decrease the (apparent) synthesizing temperature and increasing the yield of SiC.[14,31]

In order to eliminate Fe_2Si and probably residual SiO_2, and preparation of single-phase silicon carbide, the products were purified by using an appropriate leaching as mentioned above. Fig. 5 shows the XRD patterns of synthesized samples after leaching and filtering. It is evident that after leaching, pure β-SiC as a major phase with cubic structure and a small amount of α-SiC with a collection of hexagonal forms are presented. SiC predominantly consists of β-form, but a small volume fraction of α-form is always present under this processing condition. It shows that the β to α partial transformation took place during synthesis.[22] XRD results displayed in Figs. 4 and 5 showed

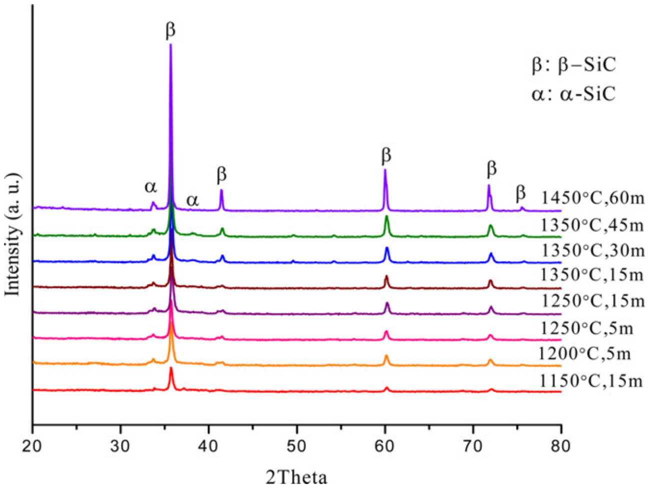

Fig. 5. XRD patterns of synthesized SiC samples after leaching and filtering.

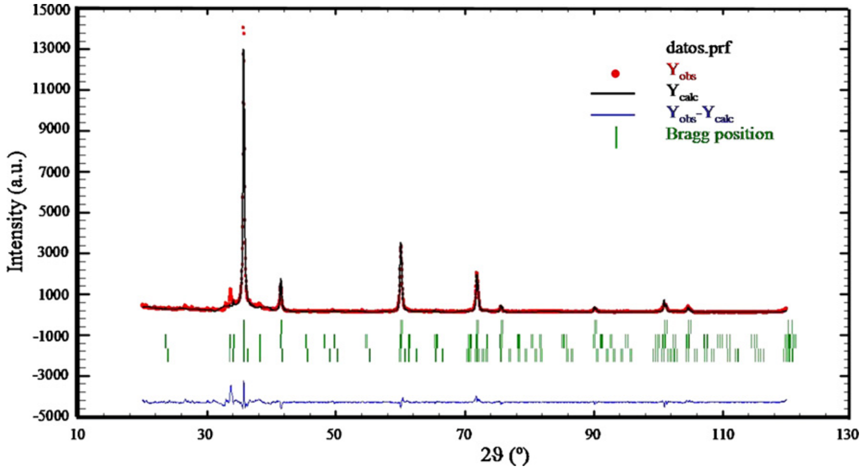

Fig. 6. Rietveld refined powder XRD data for SiC synthesized in microwave field at 1200 °C for 5 min.

no evidence of a peak due to some trace of any nitride. EDX analysis did not show any evidence of this either. This absence of nitrogen is in full agreement with reports by Dai et al.[20] who also performed microwave synthesis of SiC in N_2 atmosphere with no presence of silicon nitride in their samples.

The phase fractions of SiC polytypes for SiC powders synthesized at 1200 °C for 5 min is determined by using Rietveld refinement. Results corresponding to this sample are shown in Fig. 6 and also data for this sample are reported in Table 2. The graph shows excellent agreement between the observed and calculated XRD patterns. The results show that the major SiC polytype is cubic SiC (β-SiC:3C) with 98.5(4) weight fraction and the minor phases are two hexagonal SiC polytypic (α-SiC: 4H, 6H) with 0.50(1) and 1.0(2) weight fraction, respectively. Other polytypic of SiC (α-SiC: 15R) with rhombohedra structure does not fit in profile plot. It means that this phase is not present in this sample. Ortiz et al.[32] reported that in β-SiC phase there are always defects such as twins and stacking faults. They believed that the only difference between a highly twinned β-SiC structure and the structure of the different α-SiC polytype is the periodicity of the stacking faults. It is inferred that a central factor in the β to α transformation is the nucleation of discrete α-SiC lamellae at twins and stacking faults in the β-SiC because these defects already possess the configuration of α-SiC polytypes. But it should be noted that the β–α transformation is a kinetically controlled process, and often several days are needed to achieve complete conversion.[22] In SiC powders synthesized at 1200 °C for 5 min, the nucleation of α-SiC is just started, but the synthesis by microwave in this sample is very fast and the transformation of β to α is very small and negligible.

Table 2
Rietveld refinement of SiC synthesized in microwave field at 1200 °C for 5 min.

Phase	β-SiC	α-SiC	
	3C	4H	6H
Fraction (%)	98.5(4)	0.50(1)	1.0(2)
a (Å)	4.3523(2)	3.0813(2)	3.0887(2)
b (Å)	4.3523(2)	3.0813(2)	3.0887(2)
c (Å)	4.3523(2)	15.1198(2)	14.8695(2)
R_p	9.97		
R_{wp}	14.3		
R_{exp}	6.20		
Chi^2	5.34		

Particle size and morphology of the different synthesized samples were analyzed by scanning electron microscope (Fig. 7). High temperature and long time process (1450 °C, 60 min) yielded micron sized spherical particles (Fig. 7a). Size distribution is not uniform (agglomeration of particles from 100 nm to 1 μm). The high processing temperature and long time have resulted in partial fusion of particles to increase the overall particle size of powders. A decrease in temperature and time results in significant reduction in particle size to nano scale with narrow particle size distribution (Fig. 7b–d). It can be seen from all the images that particles aggregate to form agglomerates. It was noted that the agglomerate contained several individual and spherical particles. Agglomeration and fusion of synthesized SiC particles by microwave processing have been reported by other researchers.[5–23] Johnson et al.[16] studied the self-diffusion kinetics for silicon carbide synthesis by rapid carbothermic reduction and they showed that as soon as SiC forms on the surface of the carbon primary particles, these particles begin to grow by sintering. Particles have a tendency to grow significantly at higher temperatures, because of increased sintering.

The size and morphology details of synthesized SiC nano powders (for the sample nos. 7 and 8) were revealed using transmission electron microscopy (TEM) as shown in Fig. 8. The SiC powders synthesized at 1200 °C for 5 min have fine particle size of 10–40 nm, narrow particle size distribution, and are loosely agglomerated with nearly an equiaxed or spherical morphology (Fig. 8a). It seems that the particle size and morphology in the sample synthesized at 1150 °C for 15 min are similar to the sample treated at 1200 °C for 5 min (Fig. 8b). However, further decrease in temperature to 1150 °C caused incomplete SiC synthesis even with 15 min holding time, as shown by XRD analysis (Fig. 4).

The particle size and particle size distribution of synthesized SiC nano powders for sample nos. 5–8 were also measured using

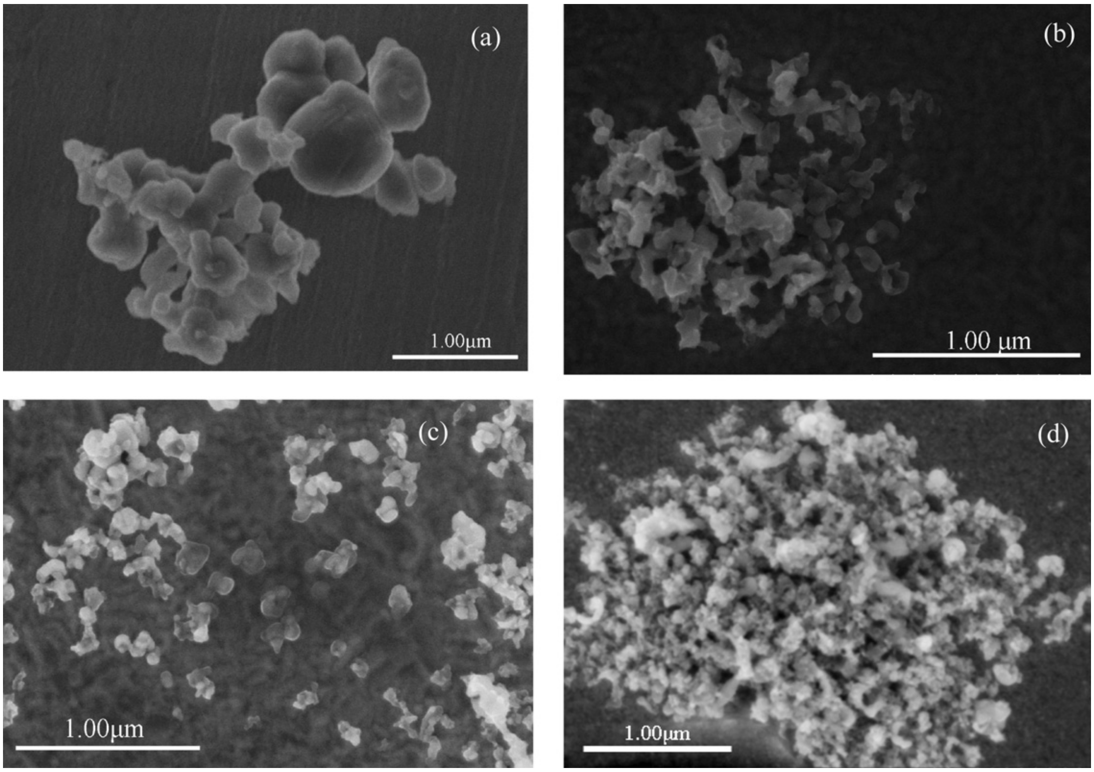

Fig. 7. SEM micrographs of synthesized SiC in microwave field at (a) 1450 °C for 60 min, (b) 1350 °C for 15 min, (c) 1200 °C for 5 min and (d) 1150 °C for 15 min.

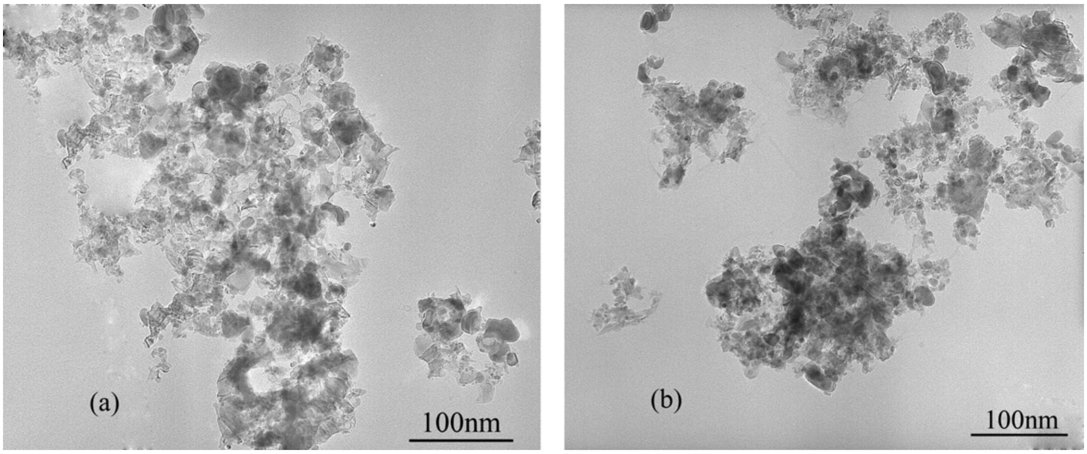

Fig. 8. TEM micrographs of synthesized SiC nano powders in microwave field at (a) 1200 °C for 5 min and (b) 1150 °C for 15 min.

Table 3
Properties of powders synthesized in a microwave field at different conditions.

Sample number	Synthesis conditions		Specific surface area (m^2/g)	Particle size (nm)/intensity		Mean crystallite size (nm)
	Temperature (°C)	Time (min)		Peak 1	Peak 2	
1	1450	60	5.36780	–	–	181
2	1350	45	19.2529	–	–	50
3	1350	30	25.9951	–	–	46
4	1350	15	29.7276	–	–	42
5	1250	15	33.5999	–	248/100%	40
6	1250	5	40.8060	60/9.2%	223/90.8%	37
7	1200	5	47.4716	40/12.1%	205/87.9%	35
8	1150	15	–	–	187.7/100%	–

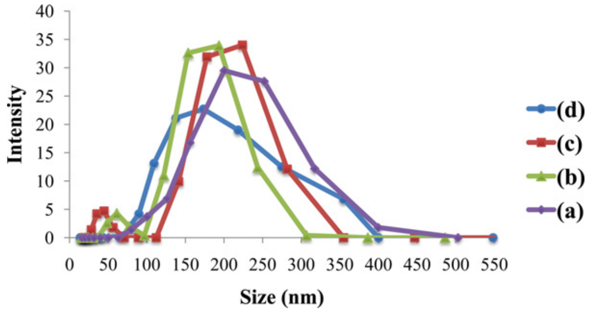

Fig. 9. Particle size distribution of synthesized SiC nano powders in microwave field at (a) 1250 °C for 15 min, (b) 1250 °C for 5 min, (c) 1200 °C for 5 min and (d) 1150 °C for 15 min.

a Malvern particle size analyzer as shown in Fig. 9. Table 3 shows the exact details of the peak position for each sample. A bimodal particle size distribution was obtained for the powders synthesized at 1200 °C and 1250 °C for 5 min, whereas the two samples synthesized with longer holding time showed unimodal particle size distribution. The bigger peak around 150–250 nm remains nearly fixed for all the samples and corresponds to agglomerates formed by 4–5 particles. The position of the first peak is altered slightly with increasing temperature and shows the coarsening of the particles from 40 nm in the sample synthesized at 1200 °C for 5 min to 60 nm in the sample synthesized at 1250 °C for 5 min. The first peak disappeared when increasing holding time, confirming the possibility of agglomeration and fusion between particles. Table 3 also lists the crystallite size of the synthesized powders calculated using the Scherrer formula from the (1 1 1) diffraction peak of β-SiC. As shown in Table 3, the mean crystallite size of samples produced at 1200 °C for 5 min is 35 nm and with increasing temperature and time to 1350 °C for 45 min, mean crystallite size increases gradually to 50 nm. But, for the sample synthesized at 1450 °C for 1 h, the crystallite size increased significantly more than three times magnitude, because a drastic sintering between particles begins to occur at this temperature. This is in agreement with the SEM micrographs shown in Fig. 7.

The reactivity of synthesized SiC powders was analyzed by measuring surface area using a BET surface area analyzer as it is shown in Fig. 10. The results are also mentioned in Table 3 in order to compare with the results from particle size analyzer. The BET surface area for the powders synthesized at 1450 °C for 60 min was 5.4 m^2/g. In this condition with high temperature and long duration time, the efficiency of fine particles forming agglomerates by sintering of the primary crystalline particles is very high. Thus, a drastic difference of the specific surface area was observed with other samples. It means that a decrease in temperature and soaking time has reduced the probable fusion between particles. Consequently, the surface area of the synthesized powders is increased from 5.4 m^2/g for powders synthesized at 1450 °C for 60 min to 47.5 m^2/g for powders synthesized at 1200 °C for 5 min. This is compliance with particle size values from Malvern analyzer. However, SEM micrographs showed large agglomerates while particle size analysis and BET did not. This phenomenon was also reported by Satapathy et al.[5] and they believed large agglomeration observed in SEM or TEM is due to physically bonded or just weakly bonded together because of surface charge on particles. They showed that very fine particles tend to coalesce due to surface charges. Continuous ultrasonic treatment that was done in both cases, tend to isolate the particles and keep them away from joining. The particle size analyzing experiments were carried out immediately after ultrasonic treatment, whereas the SEM or TEM experiments were carried out after a considerable time period until the liquid has dried out completely. Some micrographs in Figs. 5 and 6 clearly show isolated particles loosely sticking to the top surface indicative of bonding due to surficial charges.

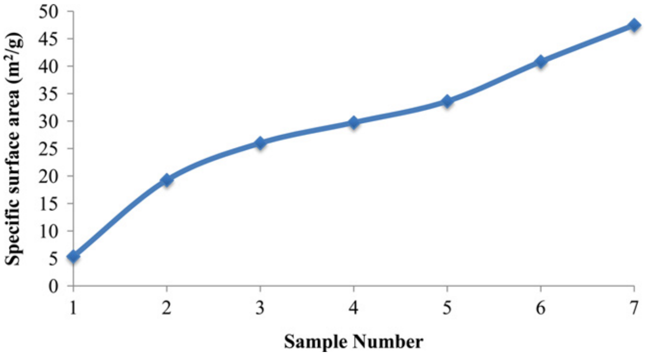

Fig. 10. BET surface area of synthesized SiC for sample nos. 1–7.

4. Conclusions

Silicon carbide nano powders have been synthesized from carbothermic reduction of nanosilica with graphite in a 2.45 GHz microwave field in nitrogen atmosphere after 40 h milling. At 1150 °C, the synthesis of SiC is started but it is not completed even with increasing duration time. Increasing in temperature to 1200 °C for 5 min could develop SiC particles of 10–40 nm with narrow particle size distribution. The Rietveld quantitative phase-composition analysis showed that the major SiC polytype is cubic SiC (β-SiC) with 98.5(4) weight fraction and the remained is hexagonal SiC polytypic (α-SiC). The synthesis by microwave in this condition is very fast and the transformation of β to α is negligible. Further increasing in synthesis temperature and time results in more possibility of agglomeration and fusion between synthesized SiC particles and sintering of the primary crystalline nano particles happens. Therefore, the particle size of synthesized SiC increases to micron scale in the sample treated at 1450 °C for 60 min with non-uniform size distribution.

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