American Journal of Engineering Research (AJER)
e-ISSN: 2320-0847 p-ISSN: 2320-0936
Volume-03, Issue-03, pp-317-321
www.ajer.org

Research Paper Open Access

Effect of primary materials ratio and their stirring time on SiC Nanoparticle production efficiency through sol gel process

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Abstract: In this article, SiC (silicon carbide) Nanopowder was synthesized by sol-gel method. TEOS and sucrose were used as pricier source. Final gel were prepared by mixing in different stirring times and different ratio in pH=4 and drying. This mixture was heated at 650°C and carbothermal reduction was carried out at 1500 °C. XRD analyses showed that produced powder in this condition are only β-Sic. Investigating SEM and TEM images and PS diagrams for different samples one can realize that in TEOS/sucrose=4 and stirring time, 5 hours, maximum efficiency and minimum grain size and minimum agglomeration occurs. Experiment result indicated that nano silicium carbide along with tetraethoxysilane, ethanol, sucrose and water can be used as carbon supply. Average grain size which has been seen by SEM was less than 100 nm.

Keywords: *sol-gel, nanopowder, synthesize, SiC*

I. INTRODUCTION

SiC is one of the most important none oxide ceramics which is produced in the powder form in a wide range, because of having excellent mechanical property, high electron conductivity, high thermal conductivity, high chemical resistance against oxidation possess wide range of application in industry such as high thermal application, semiconductors and so on. The main process of SiC production is carbothermal reduction which is known as Acheson process. Produced powder through this process is coarse. This is obvious that micro structured materials have great properties. These properties include improvement in strength, wear resistance, corrosion resistance and toughness. To produce SiC a lot of methods have been reported like sol-gel, plasma, laser and microvio. Nanopowder production through sol-gel process is a quite new process which will possess high potential in industry in future. This paper represents effect of important parameter on SiC production by sol-gel process.

SiC as a ceramic material has been noted a lot in the last decades due to unique properties such as stability in high temperatures. But low toughness is one the most important downside of SiC and other ceramics [1].

In the last years a lot efforts have been made to solve this problem Due to planting fibers and viscures to produce multiphase structure. Although toughness of SiC and other ceramics improved to some extent, toughness of these materials is less than applied metals. By producing nano particles and controlling particle size bonding energy in structure, surface and grain interfaces, the hope of producing strong ceramic ceramics with high toughness has been achieved [2].

SiC nanopowder, because of having special property such as high hardness and strength, high corrosion and oxidation resistance ,low thermal expansion quotient and high thermal conductivity is a suitable substance for improving complex materials and high temperature application such as thermal elements and

fireproof [3,4,5].On the other hand, SiC as a semiconductor in designing electronic pieces with the ability of working in high temperatures, electric power and frequencies and high stressed media has attracted attention [6]. Conventional method of producing SiC which is famous in industrial scale is carbothermal reduction between Silica and petroleum coke in high temperature (about 2500 °c). Resultant SiC by this method is so 888grain and has a lot of impurities [1]. To produce piece with high quality, powder with high purity, spherical shape, small particle size, limited size distribution and low agglomeration intensity is needed. Nanopowder with these property can be produced through process like: carbonization of metals having Si [7], CVD [8], sol gel [9], heat plasma [10], grinding [6] and SiO2 carbothermal reduction [4]. Among these methods sol gel and carbothermal reduction are alike. But regarding different mixing condition of two reactors (SiO2 and carbon) , in sol gel method much more homogenous mixture is produced by having homogenous mixture, reaction rate increases and reaction temperature decreases so that producing fine grained powder is possible [11].

II. MATERIALS AND EXPERIMENT

In this research TEOS (Tetron Etil Ortho Silicate) with chemical formula Si (C2H5O4), ethanol, Sacarose (c12H22O11) and distilled water used as primary materials. Primary materials are mixed according to ratios in table 1 and were stirred with the speed of 250 rpm in 30˚Caccording to times in table2 in pH=4.5. Prepared solution was kept in an isolated place for 18 hours in order to finish hydrolysis reaction. Resultant gels were kept in the extraction fan for 4 hours in 60° C to dry. In order to perform paralysis, samples were kept in furnace with 888 atmospheres at 650˚ C at 15˚ C min-1 for 1.5 hours. For carbothermal reduction samples were kept in argon atmosphere. To remove remained Si and SiO2 pickling by HF was performed because of samples agglomeration a grinding process in neutral environment was done. After sorting the samples phase analyze through XRD was carried out. Then samples were studied by SEM and TEM images has been prepared in order to get information about morphology and different phase distribution and also approve or reject of presumption. After that PS test has been done to investigate grain size distribution in samples.

III. RESULTS AND DISCUSSIONS

3.1. Effect of TEOS/Eth ratio on produced SiC quantity

Figure 1 shows XRD spectrum related to TE1 sample. Presence of TEOS and carbon source leads to SiC production any way which spectrum above proves it. Since picks intensity are too close phase quantity cannot be obtained. But we have analyses like this:

According to the studies, equilibrium diagram for TEOS and water mixing can be plotted as figure2.

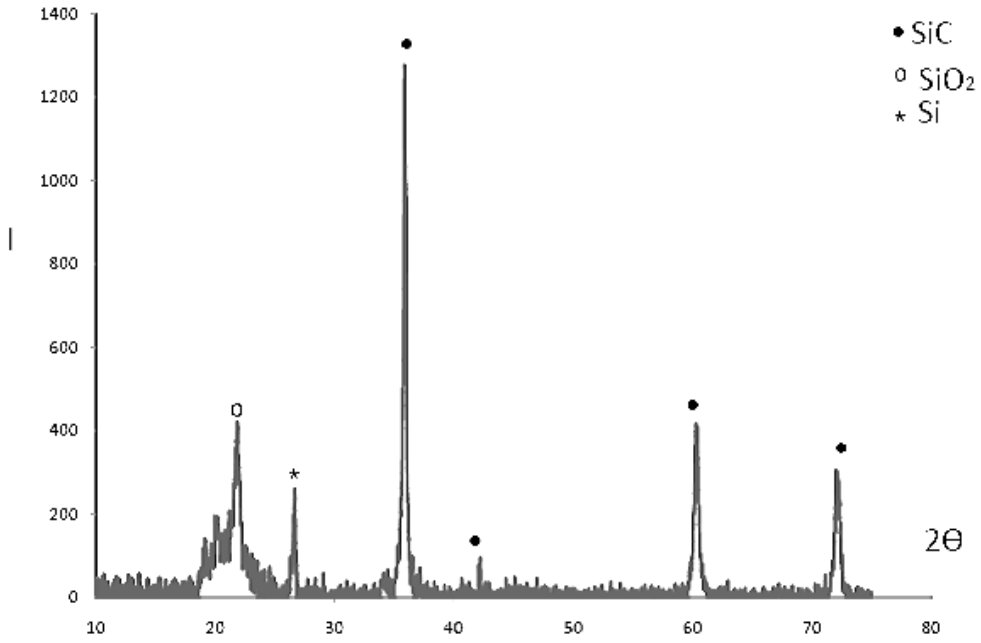


Fig.1 XRD spectrum for TE1 sample with T/E=6.

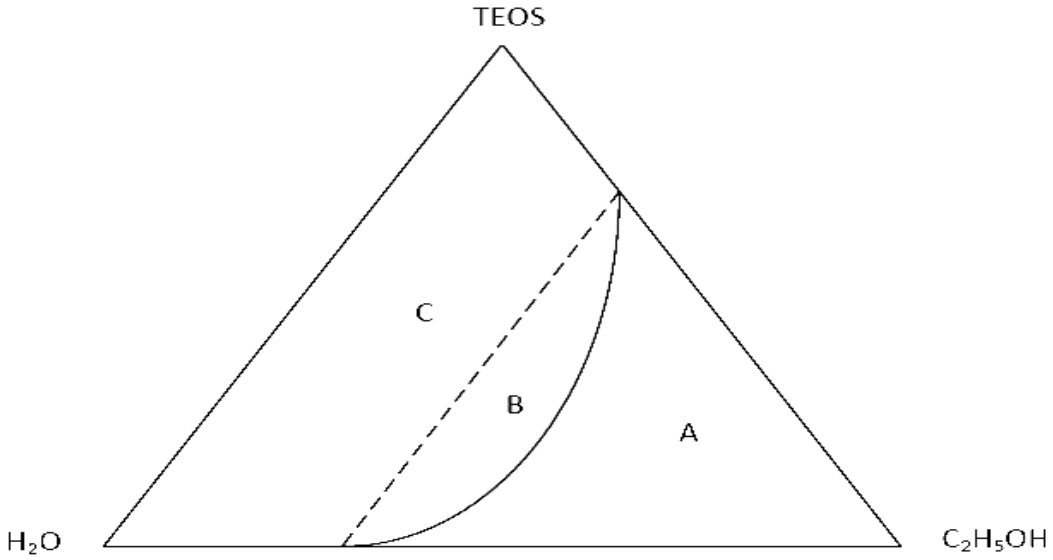


Fig.2. water and alcohol and TEOS mixing

In above mixing diagram region A is the main mixing region which can be developed to A_B region by increasing acid or decreasing alcohol amount. In fact by increasing alcohol so much it is deviated from ideal line hydrolysis reactions rate decrease and the amount of final phases will decline. Also decreasing solvent so much leads to increasing cross links and not occurring equilibrium reaction, so decreasing expected phases [6].

3.2. Effect of TEOS/Eth ratio on produced SiC particle shape

Figure 3 and 4 show SEM images for samples TE2 and TE4. SEM images indicate that in bigger grain size which growth nucleation powder shape changes from spherical shape to flat shape. Growth here is increasing cross links and chain length. Increasing particle size and getting out of Spherical shape with changing in alcohol ratio should be searched in mixing diagram. Figure 5 and 6 show TEM images for the same samples. Bigger flat shape grains are clear.

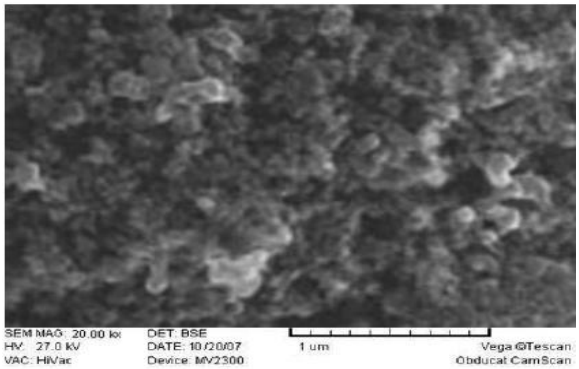


Fig.3. SEM image for TE2 with T/E=5**.**

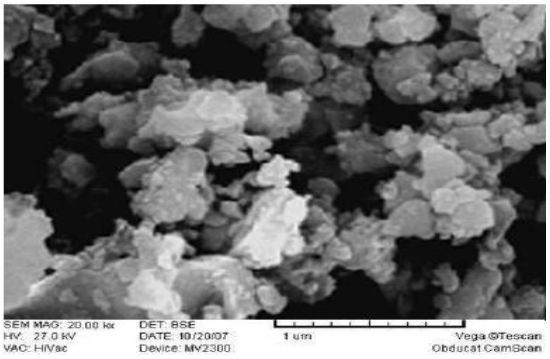


Fig.4. SEM image for TE4 with T/E=3**.**

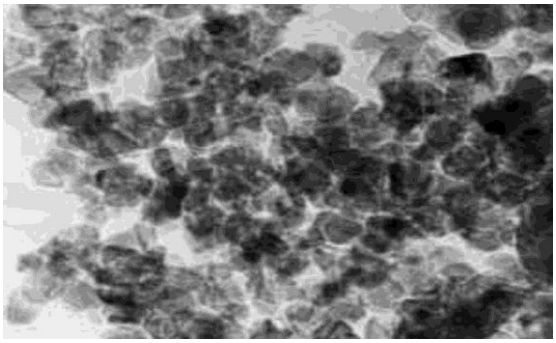


Fig.5. TEM image for TE2 with T/E=5**.**

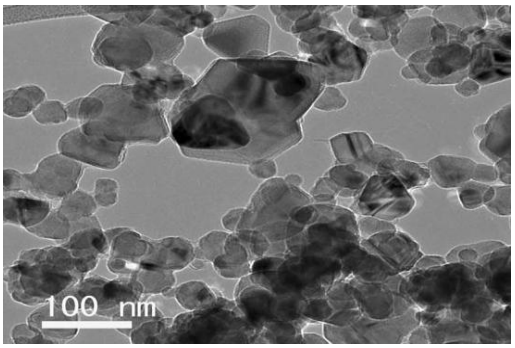
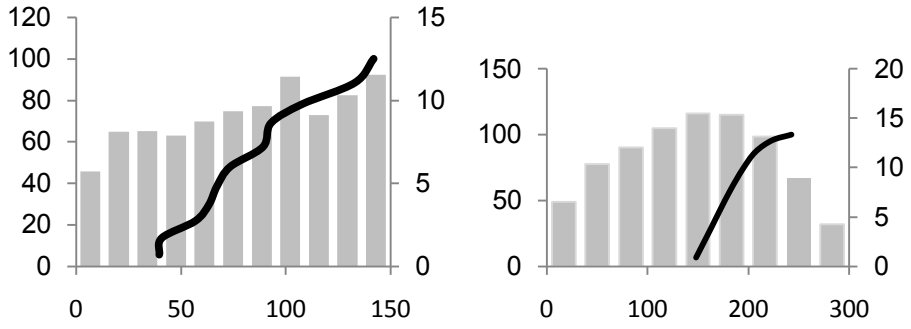
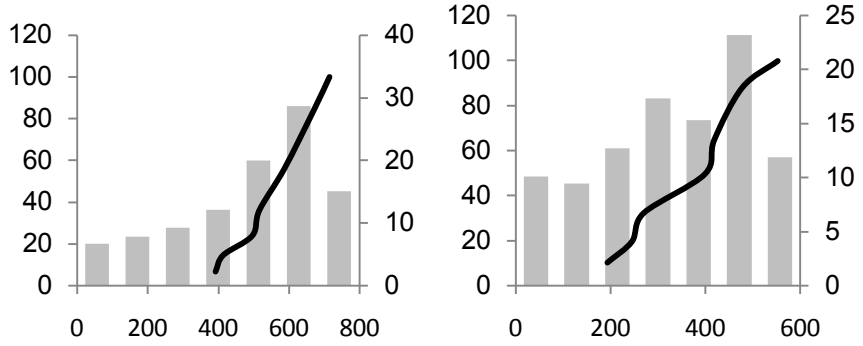


Fig.6. TEM image for TE2 with T/E=3**.**



TE1 sample with T/E=6 TE2 sample with **T**/E=5



TE2 sample with **T**/E=3 TE2 sample with **T**/E=4

Fig.7. PS diagram

According to PS diagrams, average size for each sample is obtained. And effect of alcohol ratio on particle size can be figured. As one can see in diagram, best applied ratio in this ratio research has been 5. By decreasing alcohol, since it has the roll of solvent, particles are formed faster and by increasing micro cross links make bigger molecules which have led to bigger grain size. By increasing alcohol reactions rate decline and there is more gel than precipitate.

3.3. Effect of stirring time

Hydrolysis reactions are performed in 4 stages in following order:

$$Si(OC_2H_5)_4 + H_2 \longrightarrow Si(OH)(OC_2H_5)_3 + C_2H_5OH$$
$$Si(OC_2H_5)_4 + 2H_2O \longrightarrow Si(OH)_2(OC_2H_5)_2 + 2C_2H_5OH$$
$$Si(OC_2H_5)_4 + 3H_2O \longrightarrow Si(OH)_3(OC_2H_5) + 3C_2H_5OH$$
$$Si(OC_2H_5)_4 + 4H_2O \longrightarrow Si(OH)_4 + 4C_2H_5OH$$

Completing these reactions requires enough time. In short mixing times, these reactions are not completed so final phases mount decreases. By increasing mixing time so much reactions were completed and increasing time has caused increasing cross links and bigger micro molecules, these micro molecules cause SiO_2production in bigger sizes and trapping carbon source in them. In higher temperatures carbonization reaction occurs with lower diffusion and total amount of phases has declined.

By performing experiment according to table 2 with the same route, the best mixing time obtained as 4 hours which has been already mentioned in references and there is no need to repeat.

IV. CONCLUSIONS

Maximum efficiency SiC production is obtained in T/E ratio 5. By changing this ratio production efficiency decreases. By deviation from this ratio primary sol solvent, resultant powder shape has changed from spherical shape to flat shape. By obtaining average particle size from bar diagram, decrease or increase in this ratio effects on primary molecules size. In all XRD spectrums other phases exist which in some points are amorphous and by pickling, these phases will remove to some extent. It should be mentioned that resultant data in this experiment have some deviation, due to some limitation and difficulties.

Table1. Materials ratio with different TEOS/Eth

Sample	TEOS/Eth	TEOS/Sac	Sac/H_2O	TEOS/H_2O	Time
TE1	6	4	0/5	2	**4**
TE2	5	4	0/5	2	**4**
TE3	4	4	0/5	2	**4**
TE4	3	4	0/5	2	**4**

Table2. Materials ratio with different stirring time

Sample	TEOS/Eth	TEOS/Sac	Sac/H_2O	TEOS/H_2O	Time
T1	5	4	0/5	2	**3**
T2	5	4	0/5	2	**4**
T3	5	4	0/5	2	**5**
T4	5	4	0/5	2	**6**

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