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STUDY ON PULVERIZED SILICON CARBIDE POWDERS PRODUCED BY HIGH ENERGY BALL MILL

***Krishnamohan S[1] and Ramanathan S[2]**

[1]Department of Mechanical Engineering, E.G.S.Pillay Engineering College, Nagapattinam, Tamilnadu- 611002, India
[2]Department of Manufacturing Engineering, Annamalai University, Chidambaram,, Tamilnadu- 608002, India

ABSTRACT

The present study was undertaken in order to elucidate the effect of the ball-to-powder weight ratio of Silicon Carbide powder in ball milling. The Silicon Carbide powder(SiC) was pulverized in a high energy ball mill with constant milling time of 8 hrs, constant milling speed of 300 rpm and varying the ball to powder ratio (BP ratio) of 20:1 and 40:1. The wet milling media was WC balls in Toluene ($C_6H_6CH_3$). The powders of both samples were then characterized by Atomic Force Microscope (AFM), Particle Analyzer (Zeta sizer) and Scanning Electron Microscope (SEM). The average particle size was reduced to 432.7 nm with BP ratio 20:1 and 315.5nm by BP ratio 40:1 and there is no much change in roughness value.

Keywords: *Ball-To-Powder Weight Ratio, Silicon Carbide Powder, Atomic Force Microscope, Particle Analyzer and Scanning Electron Microscope*

1. Introduction

Particles, especially nanoparticles are of great scientific interest because of their properties change as the size approaches the nanoscale. The properties include optical, magnetic, mechanical, electronic and thermal property etc. due to this unique feature, nanopaticles not only attract much attention from the scientific community but also are becoming more and more important in practical applications such as coatings, data storage, catalyst and nano electronics. Recently, sub-micrometer-sized silicon carbide has received more attention because of a significant improvement in mechanical properties of ceramic nano-composites, compared to the micrometer-sized particulates

Silicon Carbide (SiC) is a very interesting ceramic due to its promising properties like high hardness, high strength, low bulk density, good wear resistance, high oxidation resistance which makes it useful for wide range of industrial applications(abrasives, cutting tools, heating elements, thermal barriers for aeronautic or aerospace applications)[1-3] A very efficient way to increase density is through the reduction of the particle size, tight distribution and increase of surface area, which strongly influence the overall processing route and the final properties of the product [4,5]. Silicon Carbide (SiC) nanoparticles exhibit characteristics like high thermal conductivity, high stability, high purity, good wear resistance and a small thermal expansion co-efficient. These particles are also resistant to oxidation at high temperatures.

By introducing nano SiC into aluminium alloys, higher mechanical strength with decreased ductility can be obtained[6-8].By introducing small amount of Nano SiCp into a soft aluminium matrix can yield mechanical properties comparable to those of precipitation strengthened aluminium alloys[9,10] The addition of smaller volumes (5-10%) of nano sized reinforcing agent may give a significant increase in creep resistance, strength and toughness of Al2O3 ceramics[11-20].The nano SiC particles addition can hinder obviously the coalescence of TiC grains and increase remarkably the fracture toughness of the composites from 3.1 to 5.76 MPa m1/2.[21] A large number of mechanical methods are available for preparing fine and ultra fine powders. Typical high-energy processes include planetary, attritor and jet milling [5]. Silicon Carbide powder (SiC) was comminuted in a planetary mill during time intervals of 0.5, 2, 4 and 6 hours. The wet milling media was ZrO2 spheres in isopropyl alcohol. The powders were then characterized with respect to chemical composition, particle size distribution, surface area and density for each milling time. The average particle size was reduced from 1.8 μm to 0.4 μm in 30 min [22].

In this study SiCp was milled in planetary mill by varying the parameters and powders were characterized.

**Corresponding Author - E- mail: mohan_rahul2005@yahoo.com*

2. Experimental Study

2.1. Milling of SiCp

In this study, fine 600 grit silicon carbide powder (Alfa Aesar A13561, A Johnson matthey company-German) was used. The chemical composition is specified in the table 1.

Table1: Chemical Composition of the received SiC powder

Composition	Wt %
SiC	97%,
free SiC	1.8%,
SiO_2	1.5%,
free Fe	0.4%
free Fe_2O_3	0.2%

The silicon carbide particle was pulverized by planetary milling. Two samples were prepared by changing the milling ball to powder ratio (BP ratio) with constant milling time of 8 hrs and constant rotational speed of 300 rpm. The powder was sealed in a cylindrical WC vial together with and 50WC balls of 10mm in diameter. The ball to powder weight ratio was maintained at the level 20:1. (Ball weight400gms and powder weight 20 gms) The ball milling experiment were conducted at room temperature by using palversitte 6 (Fritsch-Germany) high energy ball mill for first sample, hereafter mentioned as sample1. The wet milling method was used to mill the powder. The organic compound toluene (C6H6-CH3) was filled in the ball mill chamber in adequate quantity, and its level frequently checked. The milling experiment was interrupted at regular intervals of 5 minutes for cooling. After end of one complete cycle time, the milled powders with toluene were poured into a Pyrex container, dried in an oven at 70 0C for 24 hrs, de-agglomerated in a mortar and sieved. The same procedure was repeated for second sample with 100WC balls, BP ratio of 40:1, (Ball weight 800gms powder weight 20 gms) hereafter mentioned as sample2.

2.2. Characterization

2.2. (a) surface topography by AFM

The milled nano SiC powder of each batch mixed with acetone solution and treated in Ultrasonic sonicator (750 W, 20 kHz, Sonics-USA) for about 10 minutes to attain individual particles from agglomeration. Using drop cast approach, few drops of suspension prepared from sonicator was deposited on plain glass plate and allowed for 10 minutes for drying. Then the topography of the nano SiC powders were taken by using the Atomic Force Microscopy (AFM) (XE 70, Park Systems – Korea). For better understanding of the morphology of a surface, a quantitative description of the surface topography must be carried out. The topography matrix data should be treated in each profile line (2D) or over all profiles extending the analysis to surface (3D). The amplitude parameters are the principal parameters in characterizing the surface topography. The average roughness (Ra) is the most used amplitude parameter and was measured.

2.2. (b) particle analysis and distribution by DLS (zeta seizer)

The particle size analysis was performed from the sample prepared from sonicator using Zetasizer (Malvern, Nano ZS90 – UK) (FIG). The milled nano SiC powder were mixed with Double Distilled water and treated in Ultrasonic sonicator (750 W, 20 kHz, Sonics-USA)(FIG) for about 10 minutes to attain individual particles from agglomeration. The samples of each batch was taken in a polystyrene cuvette and kept in the Zetasizer for characterization.

2.2. (C) optical characterization by scanning Electron Microscope

A scanning electron microscope (SEM-VEGA3 TESCAN.SEM HV: 15kv (FIG)) was utilized to evaluate the milled SiCp and as received SiC particles for characterization

3. Result and discussion

3.1 AFM

Topography of milled powders Using AFM

AFM images of both samples in two dimensional formats are shown in Figs 1(a) & 2(a). Particle distribution found in the 10 × 10 μm area is explored by drawing a line profile drawn across the 2D image at 7.5 μm & 6 μm is represented as red line and Vertical line drawn at 6 μm & 5.75 μm is indicated by the green line respectively. The average surface roughness (Ra) was found as 1.277 nm and 1.485 nm for both samples respectively. An AFM image of SiCp displayed in a 3-D format is shown in Figs 1(b) & 2(b) gives a rendition of what the surface topography actually looks like. It is observed that there is no much change in roughness value which represents; roughness is irrespective of BP ratio

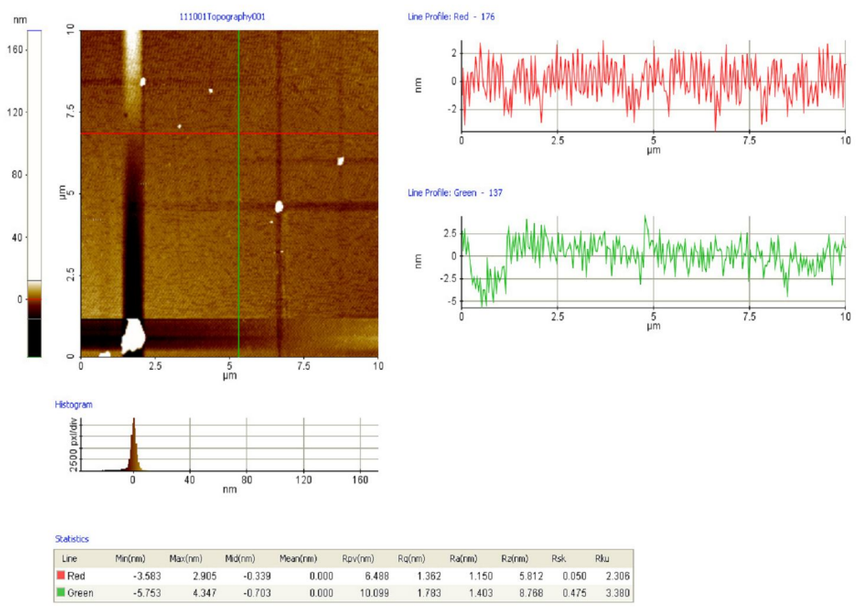

Fig. 1(a) Particle Distribution of Sample1 in 2D Format AFM Image

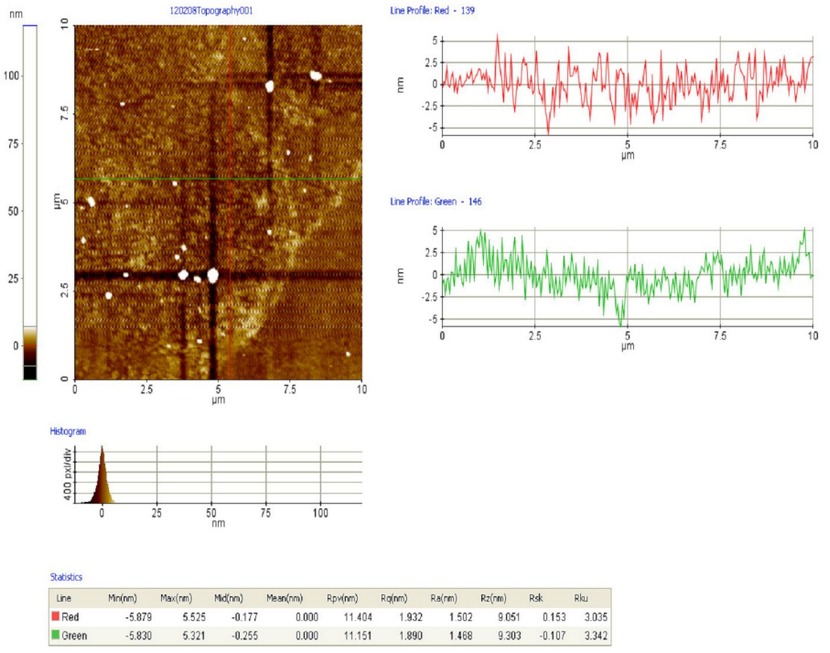

Fig. 2(a) Particle Distribution of Sample2 in 2D Format AFM Image

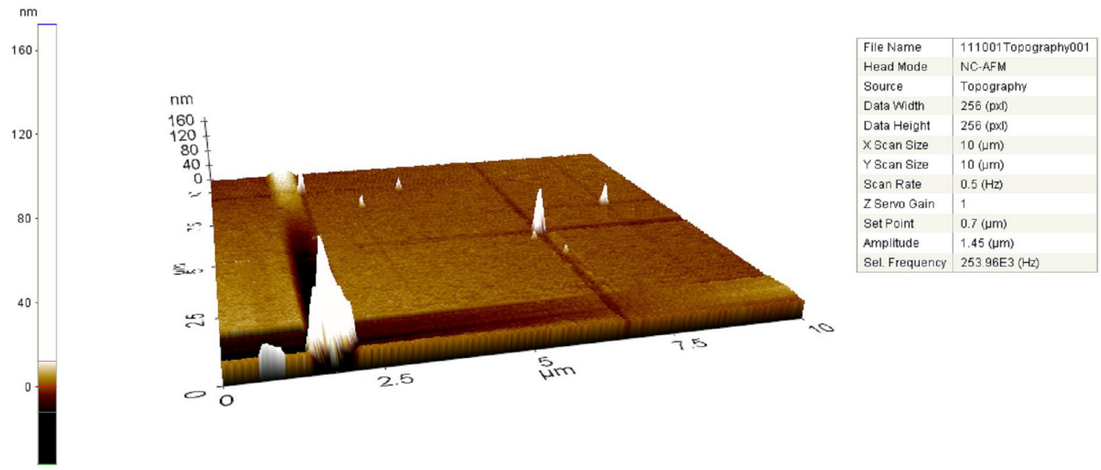

Fig. 1(b) Particle Distribution of Sample1 in 3D Format AFM Image

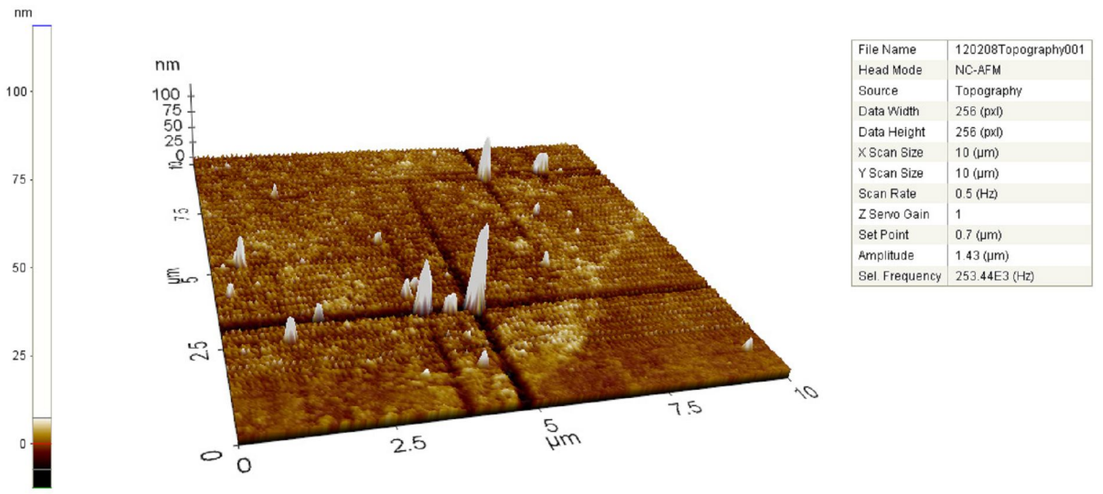

Fig. 2(b) Particle Distribution of Sample2 in 3D Format AFM Image

Results

		Diam. (nm)	% Intensity	Width (nm)
Z-Average (d.nm): 432.9	Peak 1:	438.0	84.6	202.1
PdI: 0.262	Peak 2:	3033	15.4	1181
Intercept: 0.778	Peak 3:	0.000	0.0	0.000
Result quality : Good				

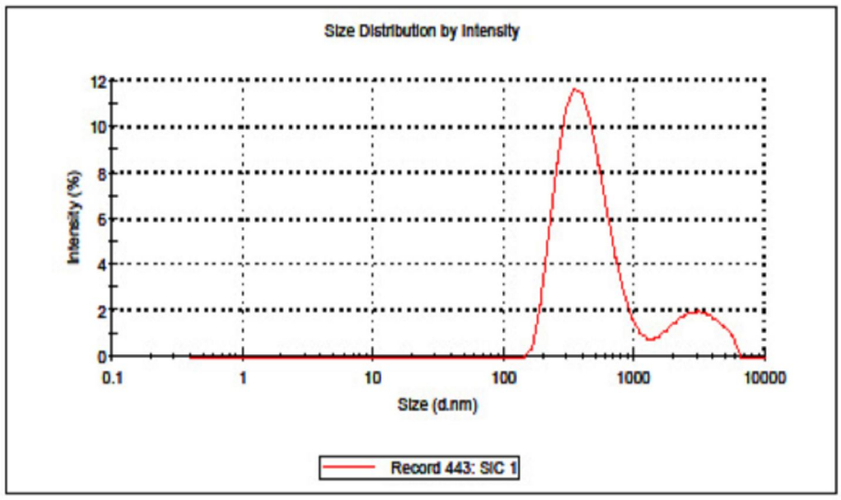

Fig. 3 Average Particle Size of Sample 1

Results

		Diam. (nm)	% Intensity	Width (nm)
Z-Average (d.nm): 315.5	Peak 1:	394.6	99.7	199.6
PdI: 0.202	Peak 2:	68.91	0.3	7.705
Intercept: 0.850	Peak 3:	0.000	0.0	0.000
Result quality : Good				

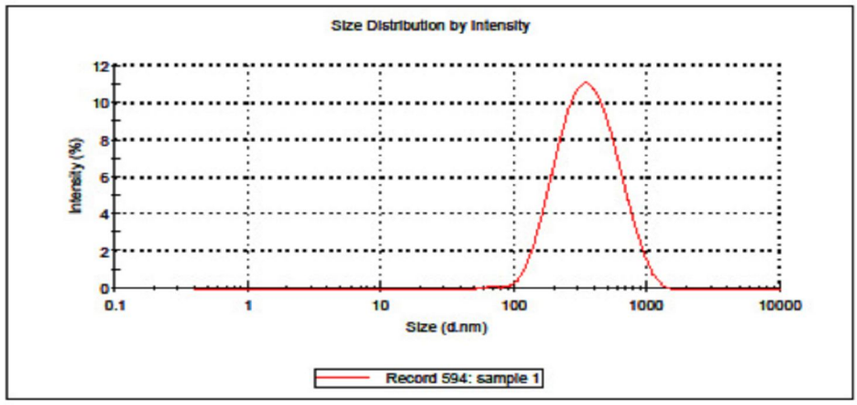

Fig. 4 Average Particle Size of Sample 2

Z-Average (nm):	432.9373	Derived Count Rate (kcps):	1079.46769079...
Standard Deviation:	0	Standard Deviation:	0
%Std Deviation:	0	%Std Deviation:	0
Variance:	0	Variance:	0

Size d.nm	Mean Intensity %	Std Dev Intensity %
0.4000	0.0	
0.4632	0.0	
0.5365	0.0	
0.6213	0.0	
0.7195	0.0	
0.8332	0.0	
0.9649	0.0	
1.117	0.0	
1.294	0.0	
1.499	0.0	
1.736	0.0	
2.010	0.0	
2.328	0.0	
2.696	0.0	
3.122	0.0	
3.615	0.0	
4.187	0.0	
4.849	0.0	

Size d.nm	Mean Intensity %	Std Dev Intensity %
5.615	0.0	
6.503	0.0	
7.531	0.0	
8.721	0.0	
10.10	0.0	
11.70	0.0	
13.54	0.0	
15.69	0.0	
18.17	0.0	
21.04	0.0	
24.36	0.0	
28.21	0.0	
32.67	0.0	
37.84	0.0	
43.82	0.0	
50.75	0.0	
58.77	0.0	
68.06	0.0	

Size d.nm	Mean Intensity %	Std Dev Intensity %
78.82	0.0	
91.28	0.0	
105.7	0.0	
122.4	0.0	
141.8	0.0	
164.2	0.3	
190.1	2.3	
220.2	5.4	
255.0	8.4	
295.3	10.6	
342.0	11.6	
396.1	11.4	
458.7	10.1	
531.2	8.3	
615.1	6.2	
712.4	4.3	
825.0	2.7	
955.4	1.6	

Size d.nm	Mean Intensity %	Std Dev Intensity %
1106	1.0	
1281	0.8	
1484	0.8	
1718	1.1	
1990	1.4	
2305	1.7	
2669	1.9	
3091	2.0	
3580	1.9	
4145	1.6	
4801	1.3	
5560	0.9	
6439	0.0	
7456	0.0	
8635	0.0	
1.000e4	0.0	

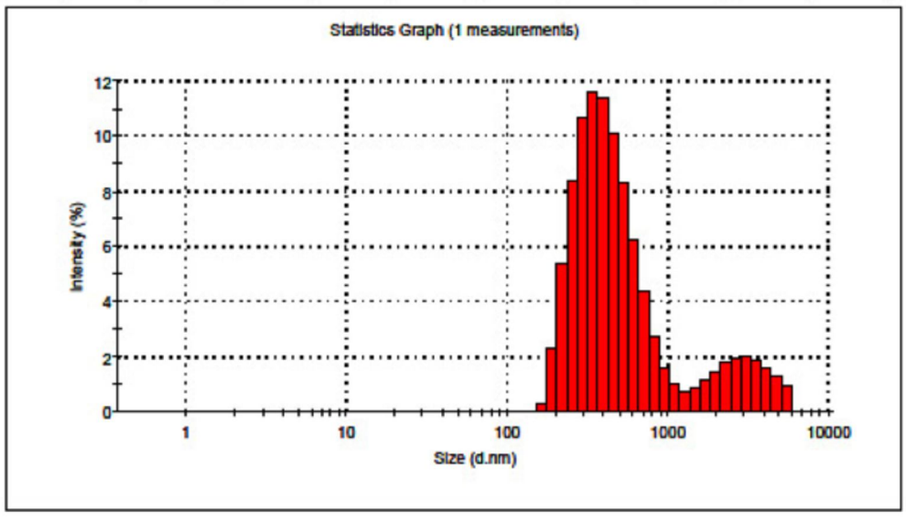

Fig. 5 Particle Size Statistics by Intensity of Sample1

3.2 Particle Analyzer Result

The results of particle analyzer are shown in the Figs. 3 & 4. It is observed that, the average particle size 432.9 nm & 315.5 nm obtained in 8 hrs and 20:1 Bp ratio milling and 8hrs and 40:1Bp ratio milling respectively. The results reveal that by increasing the BP ratio, it considerably reduces the particle sizes. The particle Size Statistics Report by Intensities of both sample are shown in the Figs. 5 & 6

Z-Average (nm):	315.5273	Derived Count Rate (kcps):	6480.10257417...
Standard Deviation:	0	Standard Deviation:	0
%Std Deviation:	0	%Std Deviation:	0
Variance:	0	Variance:	0

Size d.nm	Mean Intensity %	Std Dev Intensity %
0.4000	0.0	
0.4632	0.0	
0.5365	0.0	
0.6213	0.0	
0.7195	0.0	
0.8332	0.0	
0.9649	0.0	
1.117	0.0	
1.294	0.0	
1.499	0.0	
1.736	0.0	
2.010	0.0	
2.328	0.0	
2.696	0.0	
3.122	0.0	
3.615	0.0	
4.187	0.0	
4.849	0.0	

Size d.nm	Mean Intensity %	Std Dev Intensity %
5.615	0.0	
6.503	0.0	
7.531	0.0	
8.721	0.0	
10.10	0.0	
11.70	0.0	
13.54	0.0	
15.69	0.0	
18.17	0.0	
21.04	0.0	
24.36	0.0	
28.21	0.0	
32.67	0.0	
37.84	0.0	
43.82	0.0	
50.75	0.0	
58.77	0.1	
68.06	0.1	

Size d.nm	Mean Intensity %	Std Dev Intensity %
78.82	0.1	
91.28	0.1	
105.7	0.4	
122.4	1.2	
141.8	2.5	
164.2	4.2	
190.1	6.1	
220.2	8.0	
255.0	9.6	
295.3	10.7	
342.0	11.1	
396.1	10.7	
458.7	9.8	
531.2	8.3	
615.1	6.6	
712.4	4.8	
825.0	3.1	
955.4	1.7	

Size d.nm	Mean Intensity %	Std Dev Intensity %
1106	0.7	
1281	0.2	
1484	0.0	
1718	0.0	
1990	0.0	
2305	0.0	
2669	0.0	
3091	0.0	
3580	0.0	
4145	0.0	
4801	0.0	
5560	0.0	
6439	0.0	
7456	0.0	
8635	0.0	
1.000e4	0.0	

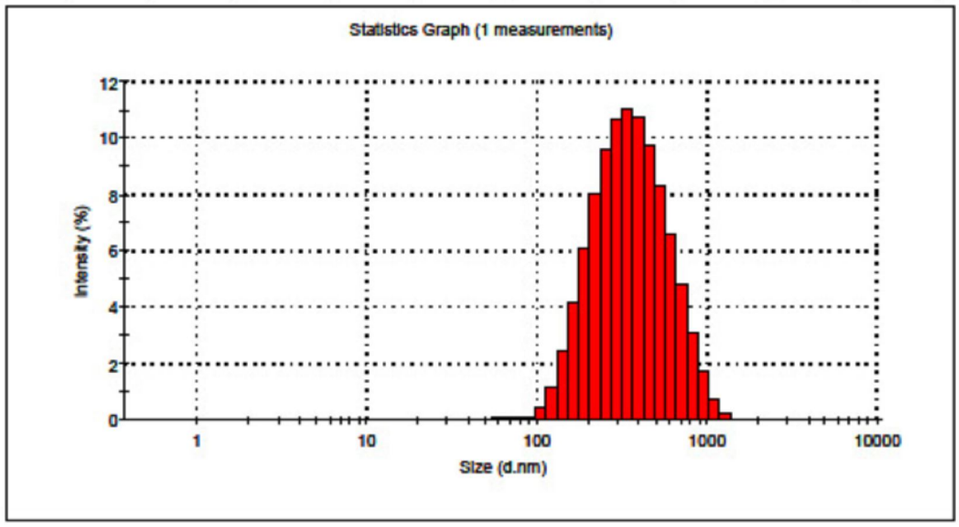

Fig. 6 Particle Size Statistics by Intensity of Sample2

3.3 SEM Result

The fast reduction in particle size and the agglormation effect was confirmed by SEM photographs of, as received powder image Fig.7, and the milled powder images of 8hrs with BP ratio 20:1shown in Fig.8 & 8hrs with BP ratio 40:1 shown in Fig.9. This confirms the generation of agglormation as particles becomes smaller. Another possibility that had to be considered was contamination that could occur in the processes, namely, the increase in surface area could be the result of breaking of SiC particles plus the addition of other small particles from the milling media.

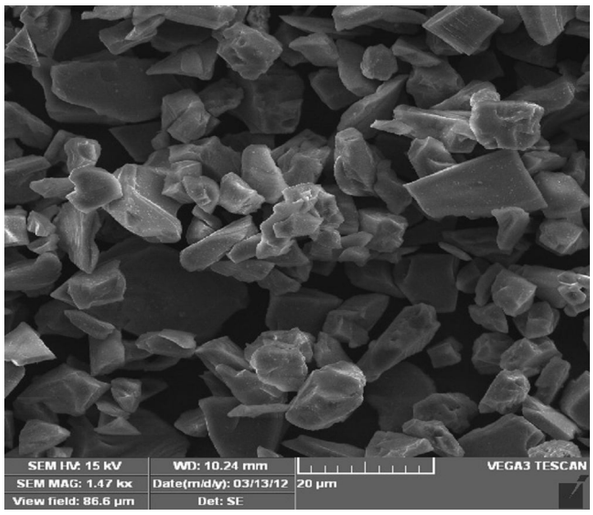

Fig. 7 SEM image of as Received SiC powder

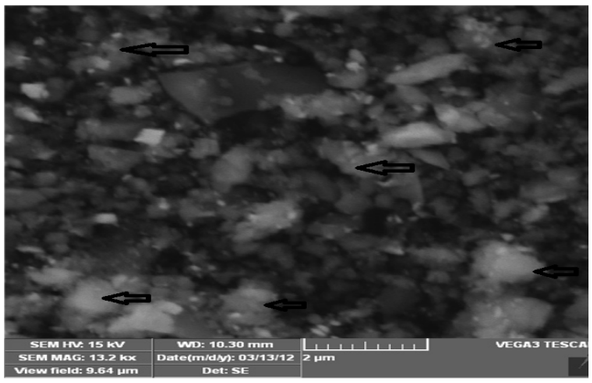

Fig. 8 SEM Image of Sample1

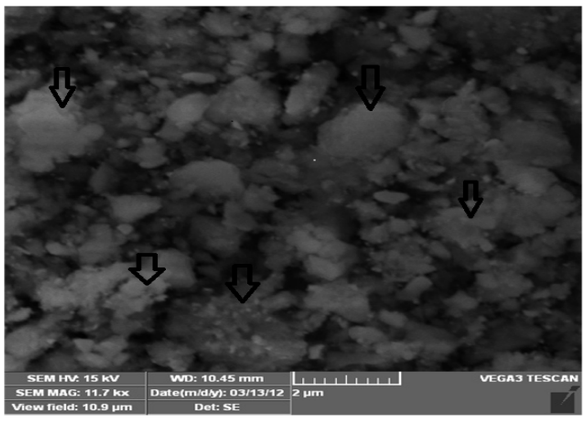

Fig. 9 SEM Image of Sample2

4. Conclusions

In this work, experiments were conducted to compare the silicon carbide powder, roughness value, particle size reduction and agglomeration by varying the BP ratio of ball milling parameters.AFM revealed that there is no much change in roughness value of samples, that is roughness value, is irrespective of BP ratio. Ball to powder ratio of 20:1 parameter reduce the particle size as average of 432.9nm Ball to powder ratio of 40:1 parameter reduce the particle size as average of 315.5nm It is observed that by increasing the BP ratio, the particle size can be considerably reduced, that is particle size is directly proportional to the BP ratio of ball milling parameter.SEM results revealed that the generation of strong agglomeration of particles during the particle size reduction by ball milling process.

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