Study on Effect of Different Ball Milling Duration on SiC Particles

H.P. Raju, Professor and Head, PESCE, Mandya, India, rajuhp@hotmail.com

B.M. Madhusudan, Research scholar, PESCE, Mandya, India, bmmsudan@gmail.com

S. Ghanaraja, Professor, PESCE, Mandya, India, ghanaraja08@gmail.com

Abstract: Significant improvements in mechanical, chemical, and physical properties have been achieved through chemistry modifications and conventional thermal, mechanical, and thermo mechanical processing methods. However, the ever-increasing demands for ``hotter, stronger, stiffer, and lighter'' than traditional materials have led to the design and development of advanced materials. Mechanical milling is the one of such new approach used to synthesizing nano size materials, which exhibits extensive set of properties. The present study involves ball milling of Silicon carbide particles (SiC) for different duration and effect of ball milling on SiC particles are investigated by different techniques. Particle size characterization has been conducted by DLS zeta analyzer and Microstructural studies by scanning electron microscopy (SEM), X-Ray diffraction technique (XRD). The main objective of the work is to investigate the effect of ball milling on size, shape and phase of SiC particles.

Keywords- SiC, ball mill, particle characterization, SEM, XRD.

I. INTRODUCTION

Mechanical milling is one of the prominent solid state powder processing techniques. Initially ball milling was only limited to industrial applications; in 1966 one of the manufacturing companies INCO has made an effort to improve mechanical properties of nickel based super alloys, The first research article about mechanical milling was published by Benjamin [1]. Initially, ball milling was used in preparation of homogeneous materials by mechanical grinding of materials, in recent times heterogeneous mixture of newer kind of powders are developed by ball milling technique [2]. The milling was done in cylindrical jar called veils or container made up of agate materials, suitable quantity and numbers of balls are used. The volume of milling container was divided into 3 major parts. Lower part of the jar was filled with 20grams of the powder to be milled and middle part of the jar was filled by number of balls, milling parameters such as ball to powder weight ratio, number of balls, speed of milling has to be determined in the early stage of the milling. The top most layer of the jar was let free for circulation of balls with in the milling jar [3]. In order to minimize the contamination, milling operation was carried out in inert atmosphere and toluene was used in the study as process control agent. Powder particles in this method are subjected to greater mechanical deformation due to the impact of ball–powder-ball and ball-powder-container collisions that occurs during mechanical alloying. Strain hardening and fracture of particles decreases the size of the particles and creates new surfaces [4]. Powder particles in milling jar during milling are subjected to greater mechanical deformation due to the impact of ball–powder-ball and ball-powder-container collisions that occurs during mechanical milling. Strain hardening and fracture of particles decreases the size of the particles and creates new surfaces [5]. There are different milling parameters which can affect the milling process have to be optimizing before starting the milling process. Two opposite phenomena are induced by the milling process: on one hand, the particles split as a result of the important internal strain created by the high pressure applied to the grains and, on the other hand, the highly divide d particles tend to agglomerate due to the high reactivity of their surfaces, in order to minimize the surface energy [6]. When the initial particle size is relatively high (typically more than 10 µm), the milling starts by a rapid splitting and, then, there is successively splitting, crushing and coalescence of the particles [7], leading to an homogeneous powder, whose mean particle size depends on the nature of the powders and on the milling conditions, duration, rotation rate, charge ratio: ratio between balls and powders weights [8].

II. SELECTION OF MATERIALS

Silicon Carbide is the only chemical compound of carbon and silicon. It was originally produced by a high temperature electro-chemical reaction of sand and carbon. SiC nano particles have gained considerable attention because of their distinguished properties such as high thermal stability, hardness. Due to high resistance to oxidation and corrosion SiC nano particles are most preferable for advanced ceramic applications. Enhanced emission intensity blue shifted photoluminescence with decrease in particle size of silicon carbide has made SiC most suitable choice for optoelectronic applications. Hence

 DOI : 10.18231/2454-9150.2018.1148

synthesized nano SiC particles are suitable elements as blue and ultra violet light emitter in displays. In this investigation, Silicon carbide powder of purity 99.9% with average particle size of 15 µm was purchased from Sigma Aldrich chemicals are used for synthesis of nano particles by ball milling.

Table 1: Properties of Silicon carbide powder

Density (g/cm^3)	3.20
Melting point (^{o}C)	2,730
Modulus of Elasticity (GPa)	410
Thermal conductivity (W/m. K)	120
Coefficient of thermal expansion(m/m ^{o}C)	4×10^{-6}

III. EXPERIMENTAL METHODS

mechanical grinding of powder can be carried in Several types of high-energy milling equipment, they are differ in their capacity, efficiency of milling and additional arrangements for cooling, heating,

The planetary ball mills are most efficient and popularly used for milling of powders because of its simple operation. The experimental setup for ball milling of SiC powder used in this investigation is as shown in the Fig.1. The setup consists of planetary ball mill with varying speed facility up to 450 rpm. It consists of single jar holding station. Specially designed milling jar made of agate material with 100 gm maximum capacity and tungsten carbide balls of size 10 mm, [9].

Fig 1: The experimental setup for ball milling of SiC powder used in this investigation

Top end of the machine consists of rotor plate on which milling jar is to be fixed by offsetting little from the centre. Rotation of rotor plate advances the planetary motion of jar around their axis in the same speed and the centrifugal force on the plate causes internal strain between the balls which results in the fracture of powder particles stuck in between the balls [10]. The experimental procedures followed in present study concern with ball milling of SiC powder of size 15µm is as follows, 20 grams of SiC powder with purity of 99.9% and 14 number of agate balls weighing 10grams each was collected in a milling jar. Ball to powder weight ratio of 1:7 was maintained throughout the operation .The operation was carried out in wet grinding arrangement by using 30-40 ml of toluene as process control agent (PCA) [11]. It has been reported that wet grinding is a more suitable method than dry grinding to obtain finer-ground products because the solvent molecules are adsorbed on the newly formed surfaces of the particles and lower their surface energy [12]. The less-agglomerated condition of the powder particles in the wet condition is also a useful factor. It has been reported that the rate of amorphization is faster during wet grinding than during dry grinding. The milling operation was carried out up to 30 hours and Powder samples were taken out of the jar for every 10 hours and characterized in respect of microstructure, phase of particles and particle size. Particle size was analyzed by the use of DLS- Zeta potential; phase structure of powder was analyzed through X- Ray Diffraction technique. Micro structural study has also been carried out to understand the impact of milling parameters on mechanical properties by the use of Field Emission Scanning electron microscopy [13].

IV. RESULTS AND DISCUSSION

A. *SEM analysis*

SEM studies of ball milled SiC samples were conducted in objectives of studying the micro structure, morphology of SiC and changes in particle size and particle shape. SEM images were taken on different duration milled samples at different magnification. 30 hours duration milled SiC powder samples are observed in FESEM and its EDX analysis shown in the Fig 2. From the EDX results it's clear that SiC particles after milled 30 hours of duration sample consist of Silicon and carbon and it's free from contamination.

 DOI : 10.18231/2454-9150.2018.1148

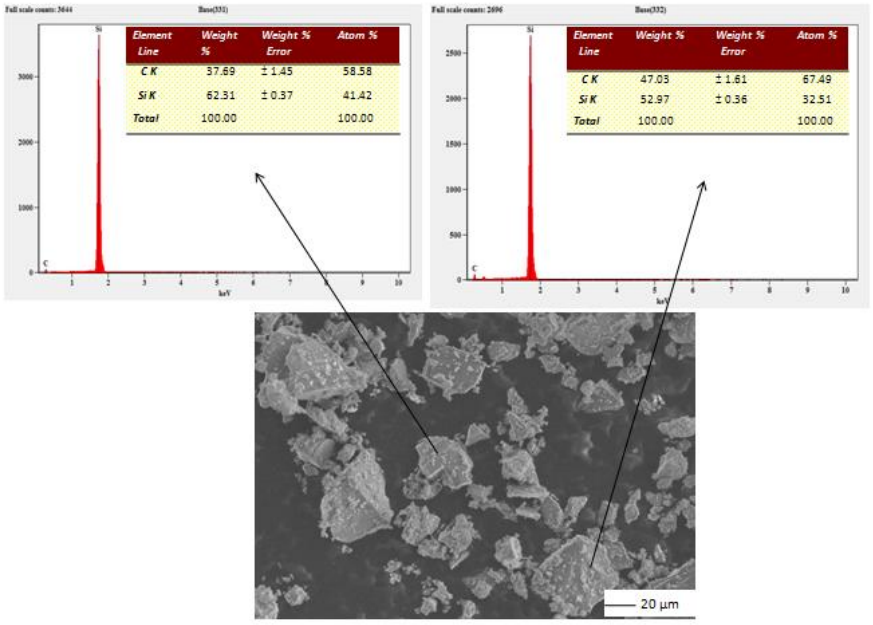


Fig 2: Shape and chemical composition of 30 hours milled SiC particles obtained by EDX analysis

Initially SiC particles before milling are spherical in shape and uniform in size and appear dull in color as shown in Fig.3 (a). Ball milling of SiC particles carried out in wet milling arrangement by using toluene as a process control agent, milled samples for FESEM analysis has been conducted in regular interval of time. During First 10 hours of milling, SiC particles are fragmented into smaller size and irregular shape particles are observed as shown in Fig.3 (b). The morphology of SiC powder after 20 and 30 hours of milling time observed in FESEM is as shown in Fig. 4. SiC particles are fractured and uneven size particles are noticed, small volume of nano sized particles are adhered to the surface of bigger particle, that may be due to higher surface energy of nano particles. From the Fig.4 (a) it's observed that sample contain small quantity of micron sized SiC particles. It is evident from the Fig (3) & (4). that SiC particles size of milled powder, as increase in the milling duration increases the reduction in particle size that could be due to greater impact force of balls on SiC particles. Comparatively more number of irregular shaped and uneven particles are observed after 30 hour milled powder is a shown in the Fig. 4 (b).

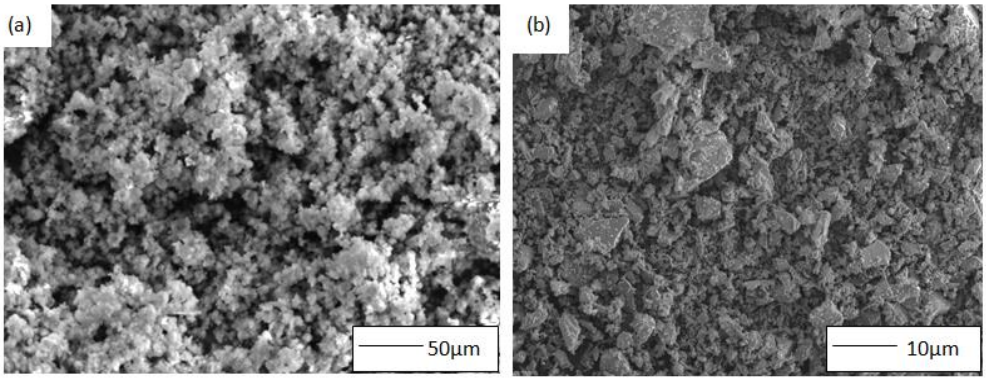


Fig 3: SEM micrographs showing change on morphology from (a) SiC powder before ball milling and (b) SiC powder after 10 hours of ball milling at 300 rpm

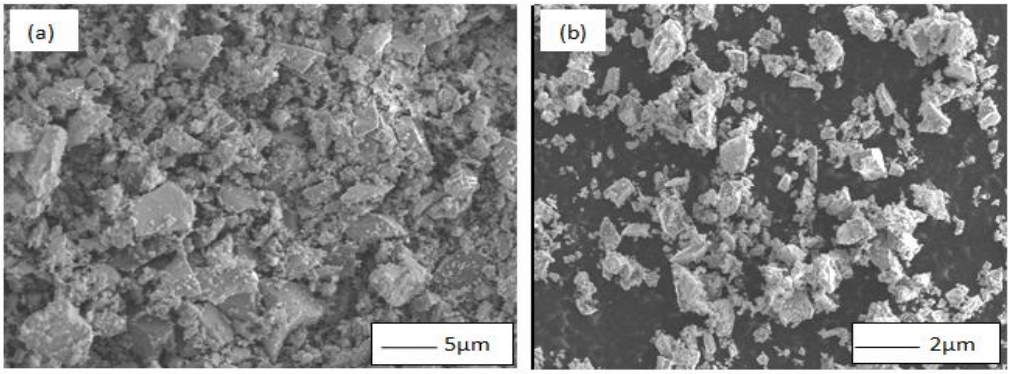
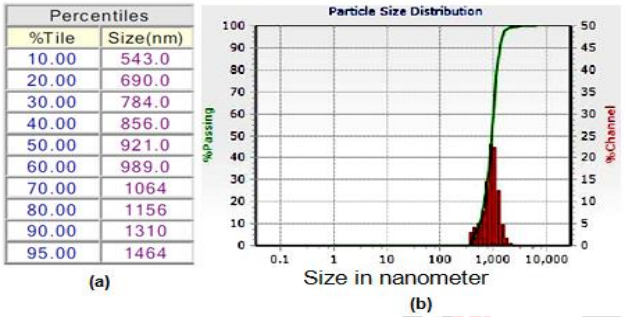


Fig 4: SEM micrographs showing change on morphology of SiC powder (a) ball milled after 20 hours and (b) ball milled after 30 hours of milling time at 300 rpm

 DOI : 10.18231/2454-9150.2018.1148

B. *DLS- ZETA analysis:*

Increase in milling time leads to decrease in mean particle size, which could be due to impact force acting on the powder particle struck in between the balls also between ball and surface of the jar. Powder particles in the container are subjected to greater mechanical deformation due to the impact of ball–powder-ball and ball-powder-container collisions. Strain hardening and fracture of particles decreases the size of the particles and creates new surfaces and irregular shape particles. Carbide particle of size fragmented from 15 µm to 200 nm average particle size after 30 hours of milling time. Powder size characterization was conducted by DLS-Zeta analysis on 10, 20 and 30 hours ball milled sample is as shown in the Fig.5, Fig.6, and Fig.7 respectively. It is observed from the Fig.5 (a) that higher order particles in the samples are in the range of 1464 nm, 10 percentiles of 543 nm sized particles are found.



From the Fig.5 (b) bar graph of SiC particles passing maximum percentage of the particles are in the range of 900 -1464 nm size.

Fig 5: DLS analysis after 10 hours milled sample (a) Percentile of particle passing chart and (b) Particle size distribution graph

In order to optimize the milling time silicon carbide particles are continued milling for another 20 hours, at the end of 20 hours milled samples are analyzed with the use of DLS analyzer. From the fig.6 (a) it is observed that particles are in the range of 565 nm to 1012 nm but average particle size was gradually decreases at the end of 20 hours of milling time. From the Fig.6 (b) it's observed that larger volume % of the sample is above 900 nm and small phase of higher size particle are also present. Generating Nano particles by planetary ball mill was the aim of the study but no phase Nano particles are recorded during the analysis.

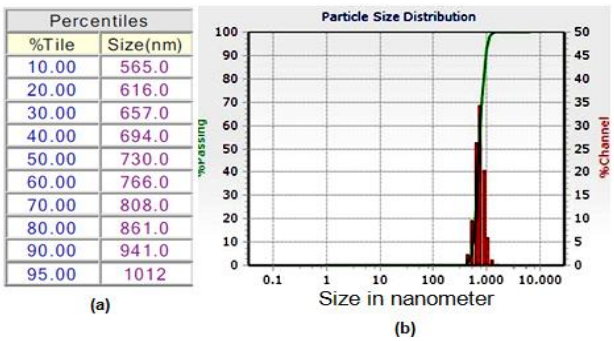


Fig 6: DLS analysis after 20 hours milled sample (a) Percentile of particle passing chart and (b) Particle size distribution graph

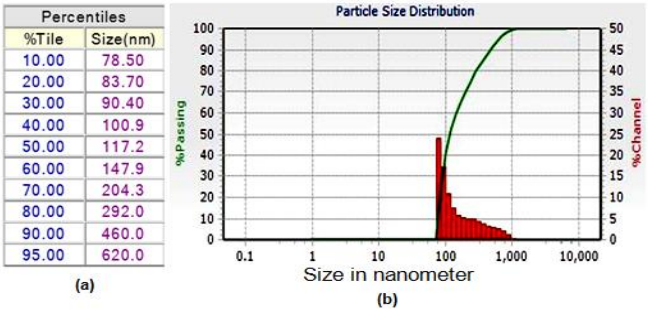


Fig 7: DLS analysis after 30 hours milled sample (a) Percentile of particle passing chart and (b) Particle size distribution graph

It is observed from the Fig.7 (a) that higher order particles in the samples are in the range of 620 nm, 30 percentiles of smaller size particles are noticed in 90.4 nm particles range and 60 percentile of 147.9 nm particles are noticed. From the Fig.7 (b) it's clear that 80 percentile of the particles are below the range of 292 nm size and particles gradually transforming from micron size to nano range and small phase of Nano size particles below 100 nm are noticed in the investigation that could be due to the effect of increase in milling time.

C. *XRD- analysis:*

The ball milled SiC particles has been examined for their X- Ray diffraction pattern before milling and also after milling for 10 hours, 20 hours and 30 hours the patterns are compared as shown in the Fig 8. Progressively with milling, the peaks of SiC are decreasing height but the extent of broadening increases. Some the minor peak in the early stage was disappeared in the 30 hours plot. The narrow peak positions are the indication of crystallite structure of the sample and peak broadening was the evident of decreasing the particle size in the sample. Hence it is evident from the XRD patterns that ball milling has the greater size effect of on the SiC powder sample.

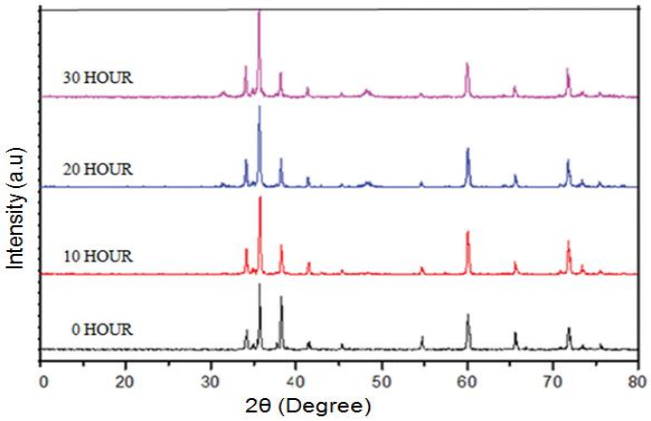


Fig 8: Comparison of XRD pattern of SiC powder milling for before milling, 10, 20 and 30 hours

V. CONCLUSION

The SiC powder of average size 15 µm was ball milled for different duration of milling time in wet milling arrangement and samples collected after every 10 hours was examined by SEM, DLS- ZETA, XRD and EDS

 DOI : 10.18231/2454-9150.2018.1148

analysis to study the effect of ball milling duration on the SiC powder.

1. FESEM Micrographs of SiC particles before milling shows that particles are fairly spherical in shape and even in size and EDS results confirms that sample was free from contamination.
2. FESEM Micrographs of 0, 10, 20 and 30 hours of milling confirms that as increase in milling time the particles got fractured due to the impact of ball–powder-ball and ball-powder-container collisions with ball to powder weight ratio 1:7 was maintained.
3. Size reduction of SiC particles on different duration milled sample were noticed in FESEM micrographs and the SiC powder also contains remaining fine particles of SiC along with nano particles.
4. DLS particle size analysis of different duration confirms that average particle size of SiC decreases from 15 µm to 620 nm and small percentile of SiC particles below 100 nm are observed in 30 hours milled sample.
5. The X- Ray diffraction pattern of different milling duration reveals that progressively with milling, the peaks of SiC are decreasing height but the extent of broadening increases. The narrow peak positions are the indication of crystallite structure of the sample and peak broadening was the evident of decreasing the particle size in the sample.

It was clear from the above results that, physical properties of the SiC particles such as size, shape and phase are dependent on ball milling hours. It was found that as increase in milling time (0-30 hours) decreases the particle size and more number of uneven size particles are observed also at the end of 30 hours of ball milling gradual transformation of SiC particles from micron to nano size was noticed. It was concluded from the results that synthesis of nano size SiC particles in inert atmosphere are possible from wet milling arrangement of ball milling method.

REFERENCES

[1] Nalwa H. S., Handbook of Nanostructured Materials and Nanotechnology, Academic Press, San Diego, 2000.

[2] Wilson N., Kannangara K., Smith G., Simmons M., and Raguse B., Nanotechnology. Basic Science and Emerging Technologies, Chapman and Hall/CRC, Boca Raton–London, 2002.

[3] Thakur Prasad Yadav, Ram Manohar Yadav, Dinesh Pratap Singh, “Mechanical Milling: a Top to Down Approach for the Synthesis of Nanomaterials and Nanocomposites”, Nanoscience and Nanotechnology, 2012, Vol 2(3), Pp. 22-48.

[4] Ghanaraja S., Ray S., Nath S K., “Synthesis and Characterization of γ-Al_2O_3, nano powder by disc milling of Al and MnO_2powder”, Procedia Materials Science, 2014, vol 5, pp. 416 – 425.

[5] Suryanarayana, Nasser Al-Aqeeli, “Mechanically alloyed Nano composites”, Progress in Materials science, 2013, vol 58, pp. 383-502.

[6] Majid Abdellahi, Hamed Bahmanpour, Maryam Bahmanpour, “The best conditions for minimizing the synthesis time of nano composites during High energy ball milling: Modeling and Optimizing”, Ceramics international, 2014, pp. 9675-9692.

[7] Suryanarayana C, Ivanov E. “Mechanical alloying and milling”, Prog. Mat science New York, 2001, vol 46, pp. 1-184.

[8] Jufu Jiang, Ying Wang, “Microstructure and mechanical properties of the rheo formed cylindrical part of 7075 Aluminium matrix composite reinforced with nano sized SiC particles”, Material and design, 2015, Vol79, pp. 32-41.

[9] Ray S, “Synthesis of cast metal matrix particulate composites”, Journal of Materials Science, 1993, Vol. 28, pp. 5397-5413.

[10] Madhusudan B.M, Raju H.P, Ghanaraja. S., “Microstructural characterization and analysis of ball milled Silicon carbide”, American institute of Physics, 2018, vol 1943, pp 020122-6.

[11] Metals Handbook, “Properties and selection: nonferrous alloys and special- purpose materials”, 10th Edition, Vol. 2, ASM International, Materials Park, OH, USA, 1990.

[12] Attard G. S., Goltner C., CorkerJ. M., HenkeS., and TempterR. H., “Liquid crystal templates for nanostructured metals,” Angew. Chem., Int. Ed. Engl., 1997, vol 36, pp. 1315–1317.

[13] Nadafi A., Golestani Fard F., Rezai H.R., Ehasani N., “A Study on Sol Gel Synthesis and Characterization of SiC nano powder”, Sol-gel sci Tech, 2011, vol 59, pp 205-214.

 DOI : 10.18231/2454-9150.2018.1148