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Synthesis of Nano sized SiC Powder

Satinder Singh
Dept. Mechanical Engineering
Baba Banda Singh Bahadur Engineering College
Fatehgarh Sahib, Punjab, India

Prof. Amandeep Singh
Assistant Professor
Dept. Mechanical Engineering
Baba Banda Singh Bahadur Engineering College
Fatehgarh Sahib, Punjab, India

Abstract— Metal matrix composites (MMCs) are defined as the system that have metal matrix. Metal matrix composites are widely used in large range of engineering applications because of their enhanced properties [1]. Aluminium, titanium and magnesium are some examples of such composites. SiC is widely used because it is easily available. %). The mechanical properties of composites improves are mainly because of high hardness of SiC [2]. The mechanical properties of pure aluminium can be enhanced with reinforcement of Nano alumina particles. In the present work nano sized SiC powder is synthesized and then it is analyzed in scanning electron microscope and laser particle analyzer.

Keywords— SiC, SEM, Grain size

I. INTRODUCTION

Nano structured materials are characterized with grain size less than 100 nm in at least one dimension. When the grain size becomes smaller, more atoms are associated with grain boundaries. Thus, the properties of nano structured material are altered by the grain size effect due to the large volume fraction. Nano structured materials have many better properties over conventional coarse-grained material including the good corrosion resistance, improved ductility, hardness, toughness and strength etc. The Nano-ceramic coating has become an alternative for the hexavalent chrome on conversion as it improves the wear resistance of almost all types of the materials in an environment friendly way. The methods used for the synthesis of the Nano crystalline powder are physical vapour deposition (PVD), chemical vapour deposition (CVD), sol-gel, mechanical alloying/milling etc. The mechanical milling/alloying is used to convert the micron size particles into the Nano size particles because of its simplicity.

II. EXPERIMENTATION

SiC powder of size 37µm is converted into the near Nano material with the help of ball bearing machine by using the 20 tungsten carbide ball of diameter 5mm. The ball bearing machine used during the process is of Fritsch make. Toluene was used as an active media to avoid the cold welding and agglomeration during the process. The ball to powder ratio (BPR) was kept 10:1. The rotational speed of machine was 300 rpm and to decrease the temperature, the process was stopped after each 30 minute for 10 minutes. The particle was repeatedly welded; fractured and welded during the process as a result the particle size gets reduced.

Figure 1 Ball Bearing Machine

The process was done for 10, 15 and 20 hr to know the size of the powder. The powder was taken from the vials for the characterization. The size of the particle and their characterization was done with the help of X-ray diffraction, scanning electron microscopy and laser particle size analyzer.

III. CHARACTERIZATION OF POWDER

A). X-ray diffraction study

The milled powder was characterized with the X-ray diffractometer (Xpert-Pro) using a filtered CuKα radiation (λ= 0.1542nm).

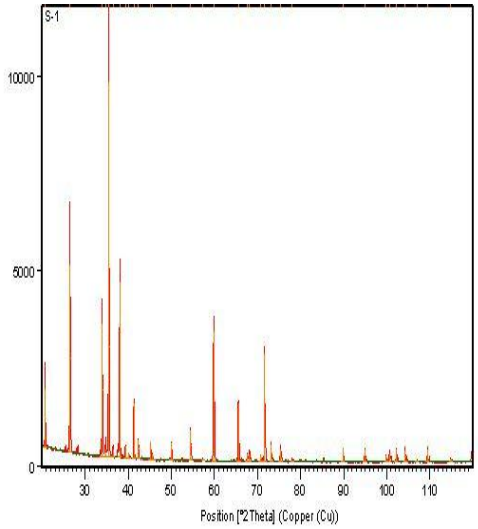

Figure 2(a) 0hr

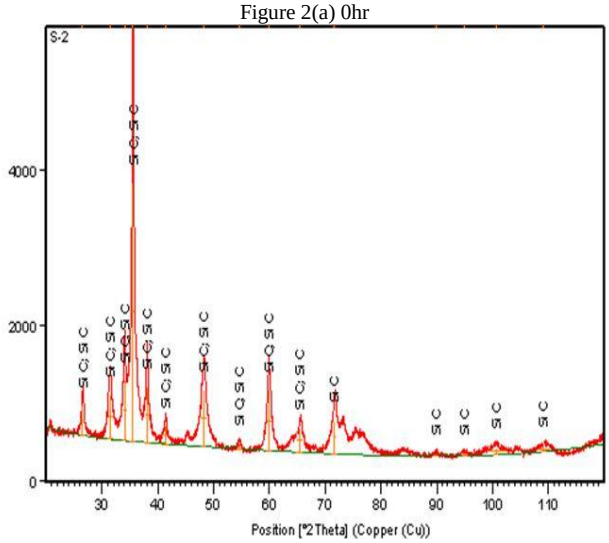

Figure 2(b) 10hr

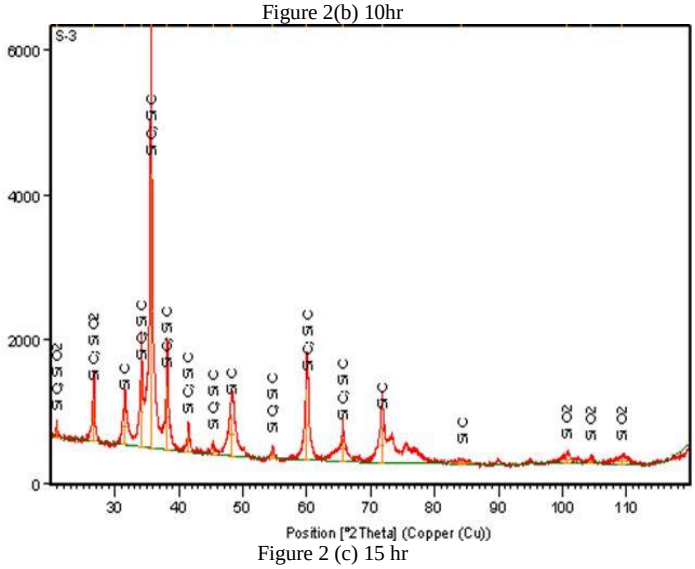

Figure 2 (c) 15 hr

Figure 2 Shows the XRD analysis of 2(a) 0 hr milled powder 2(b) 10 hr milled powder 2(c) 15 hr milled powder.

The goniometer (scan axis) was set over 2θ range of 20˚ to 120˚.The X-ray diffraction was carried out with the help of a Goniometer, using Cu Kα radiation at an accelerating voltage of 40 kV and a current of 40 mA. Figure 2 shows the X-ray diffraction pattern of the mechanically milled SiC powder of 0, 10 and 15 hr. Figure 2 (a) shows the X-ray diffraction of the initial SiC powder where peaks present in the figure are very narrow. As the milling time increases from 0 to 10 hr, the peak gets broaden. The broadening of peaks shows the reduction of the powder size. Scherer's equation is used to calculate the average crystallite size of the particles. Scherer's equation is described as:

$$d = \frac{k\lambda}{B\cos\theta}$$

Where d = Diameter of the particle
λ = X-ray wavelength
B = Full width at half maximum
θ = Diffraction angle
k = Scherer's constant

The x-ray diffraction pattern shows the reduction in the intensity of the peaks where as the peak gets broaden as the milling increases. Two major phases were identified for all the milling times; which were silicon carbide (SiC) was observed during the milling at 15 hr.
By substituting the values k, λ, B and θ, the diameter of the particle was calculated which was found less during the 10 hr milling of the SiC powder.

B). Scanning electron microscopy

Scanning electron microscopy was used to study the morphological changes occurred during the mechanical milling/alloying of the SiC powder. The size of initial as well as the nano structured SiC was also measured with the help of scanning electron microscopy. The figure 3 (a) shows the scanning electron microscopy image of the initial SiC powder. Initially, the shape of the SiC powder was angular. During the milling of the powder, the powder gets crushed and the figure (b) shows the scanning electron microscopy image of the 10 hr milled SiC powder. Image shows that there were particles which were less than micron in size as compared with scale given at the bottom of the image.

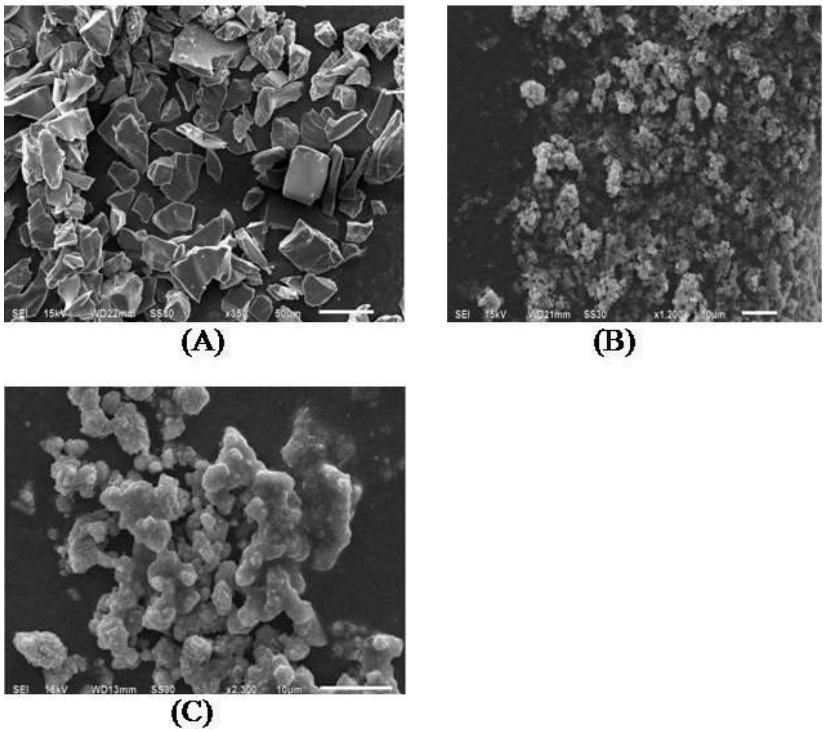

Figure (A) SEM 0 hr (B) SEM 10 hr (C) SEM 15 hr

From the scanning electron microscopy, it is evident that the SiC powder size reduces from micron size to the near Nano size at 10 hr. This decrease in size occurs due to the impact of the balls on the powder as a result of which the powder between the balls and vial get crushed and reduction of the powder size takes place.

C). Laser particle size analyzer

Particle size of the SiC powder was also characterized with the help of laser particle size analyzer. The powder milled for 10 hour is put into the beaker containing the acetone. Then the beaker was shacked for 10 min. so that particle of SiC gets suspended into the acetone. The beaker was shacked at the ultra sonification machine. The beaker containing the suspended particles of SiC powder was placed under the laser particle size analyzer. The laser particle size analyzer was of make Nano track at Indian Institute of Technology, Ropar used to measure the size of the milled SiC powder.

A laser was passed through the solution of SiC and acetone for 30 sec for the 3cycles on one sample.

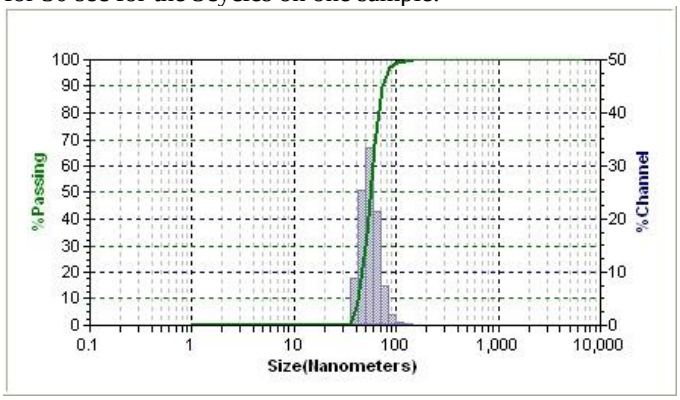

Figure 4 Size given by Laser particle size analyzer

The equipment gives the size of the SiC powder that was suspended in the acetone media. Figure 4 showed the size of the SiC powder determined by the laser particle size analyzer.

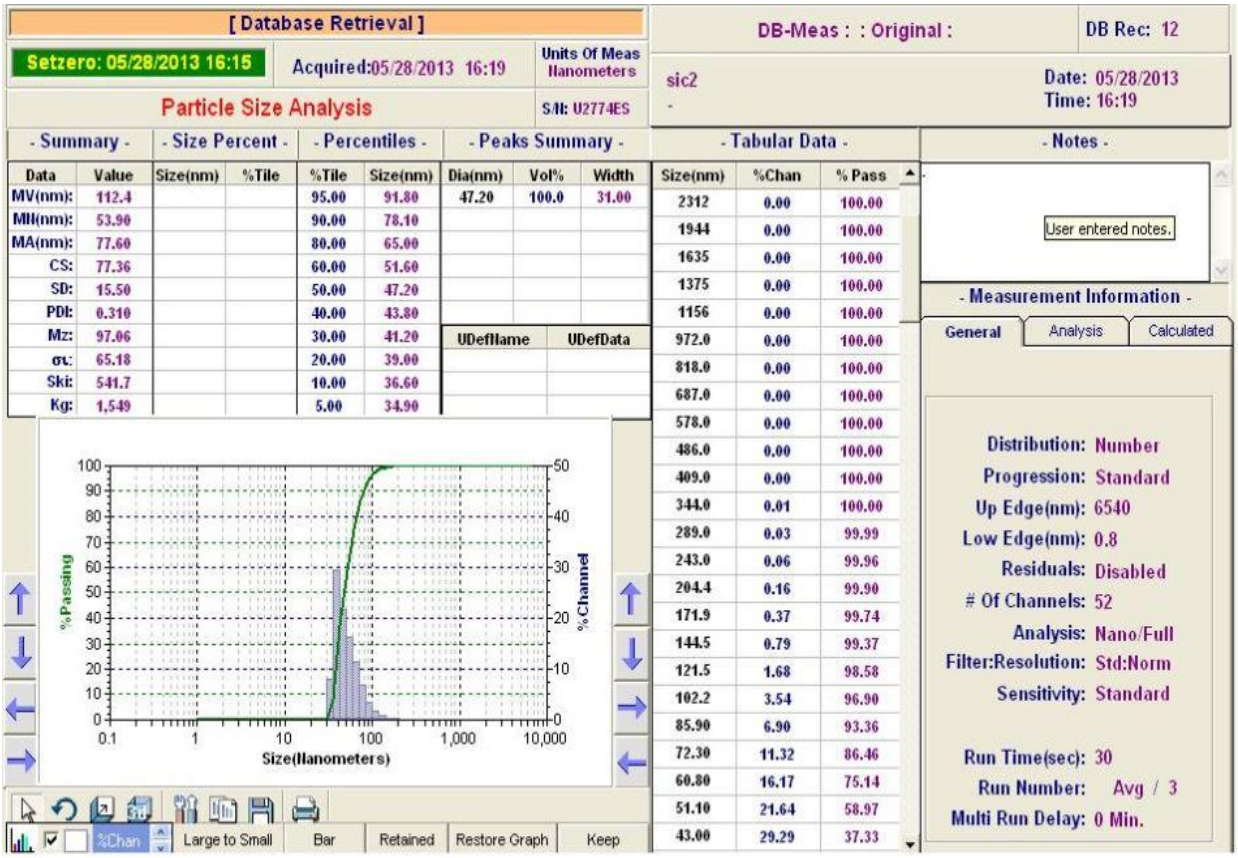

Fig 5 Report showing the size of SiC after 10 hr

CONCLUSION

From the above experimentation, it is evident that the SiC powder milled by the ball mill has become nano size powder. The SiC powder is converted from 37 µm size to 47.20 nm size of the powder. From the Scherer's equation, it can also be verified the average grain size of the particle falls near 16.50 nm and the SEM analysis of the 10 hr milled powder also showed that the SiC powder is converted into the near Nano meter size. Sic nano sized powder can be used as reinforcement in aluminium matrix composites so as to improve the hardness and wear resistance of the composites. Aluminium based matrix composites (AMCs) possess tremendous potential for number of applications in addition to their present uses in different engineering fields.

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