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Preparation of nano α-silicon carbide crystalline particles by attrition grinding

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Abstract Nano crystalline α-silicon carbide crystalline particles have been prepared from sub-micron sized particles by attrition grinding in the medium of water with surfactants in 12 hours with a ratio of weight of iron balls to the weight of α-silicon carbide of 12 : 1

Keywords : Nano particle, silicon carbide attrition grinding.

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1. Introduction

The synthesis of ultrafine powders has drawn a considerable attention because these powders (when sintered) have physical and chemical properties, which are superior to those of the bulk specimens. In particular, the emphasis is on the preparation of ultrafine ceramic powders for their applications in the development of sintered products having higher density and lower sintering temperature [1–4]. One such industrially important ceramic material is silicon carbide. 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 particulate [5–7].

Singh *et al* [8] have prepared nano crystalline silicon carbide particles from thermally pre-treated rice husk by a thermal plasma process. Chen *et al* [9] also prepared nano crystalline silicon carbide by chemical vapour deposition route. Martin *et al* [10] reported to have successfully prepared nano crystalline silicon carbide by carbo-thermal reduction of silica sol and sugar. The preparation of silicon carbide particles with nanometer-sized grains prepared from chlorine containing polysilane/polycarbosilanes (PS/PCS) has been reported [11-13]. In those investigations, the polysilane-to-polycarbosilane conversion leads to a conversion of organo-silicon compounds to a random silicon carbide network, and the cross-linking of the molecules has been investigated. Silicon carbide derived from polymeric precursors shows inhomogeneities in the nanometer range. No literature has been found for the preparation of nano crystalline silicon carbide particles by attrition milling of Acheson type (α-silicon carbide powder) and/or β-silicon carbide powders.

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2. Experimental procedure

For the preparation of nano particles, a commercial attritor (Model PRIS) of M/s Metisch Feinmath Technik GmbH, Germany, consisting of a water cooled 500 ml steel vessel and a motor driven steel agitator was used. In contrast to the conventional ball mills, in the attritor mill, the balls are agitated by a series of stirring arms mounted to a motor shaft. The attritor is very efficient in reducing the particle size. A commercially available Acheson type alpha silicon carbide of grade 1000 from M/s Grindwell Norton Ltd, India, with a silicon carbide content of 98.7%, was used. The initial particle size was measured in a Zetasizer (Model 1000-HS) of M/s Malvern Instruments Limited, UK.

The particles were ground in the above attritor mill in five different fluid media. These five fluid media were ethylene glycol, *n*-hexane, xylene, butanol and water with surface active agent. The ratio of the iron balls to silicon carbide to get nano particles was also studied in these fluid media for different grinding time in hours. Experimentally, for alpha silicon carbide, the findings of the ratio of the iron balls to silicon carbide and the required grinding time in hours were found to be the best in order to obtain nano crystalline particles from sub-micron sized silicon carbide in particular fluid medium.

3. Results and discussion

Figure 1 shows the distribution of the initial particle size of the as-received α-silicon carbide material. It is seen that the particle

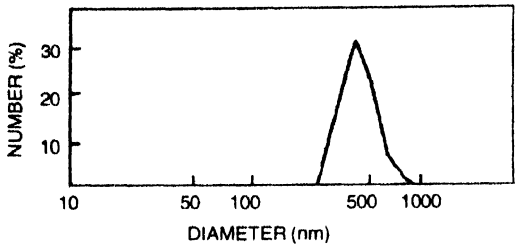

Figure 1. Size distribution of initial particles of α-SiC

size distribution is quite narrow. Therefore, the present work was started with α-silicon carbide particles with quite a narrow range of initial particle size. Figure 2 shows the transmission electron micrograph of the initial sub-micron size particles of α-silicon carbide.

Figure 2. TEM micrograph of initial particles of α-SiC

Figure 3 shows the ratio of the weight of the iron balls to that of α-silicon carbide, which is required to obtain the nano crystalline particles. It is seen that as the ratio increases, the particle size of α-silicon carbide sharply decreases. Several curves are shown in Figure 3, wherein each curve represents a different fluid medium of grinding, like water with surfactants, ethylene glycol, *n*-hexane, butanol and xylene, for a constant grinding time of 12 hours. The decrease of particle size is more pronounced for water with surfactants, compared to that of the latter four fluid media, wherein the difference is negligible in terms grinding efficiency in reducing the particle size. This is an important result in that it helps to optimize even the fluid medium for grinding to obtain nano size materials. It has also been found that at the ratio of 12 : 1 of the weight of the iron balls to that of α-silicon carbide, it is possible to obtain nano crystalline particles. The average particle size in the fluid medium of water with surfactants at the ratio of 12 : 1 is 37 nm.

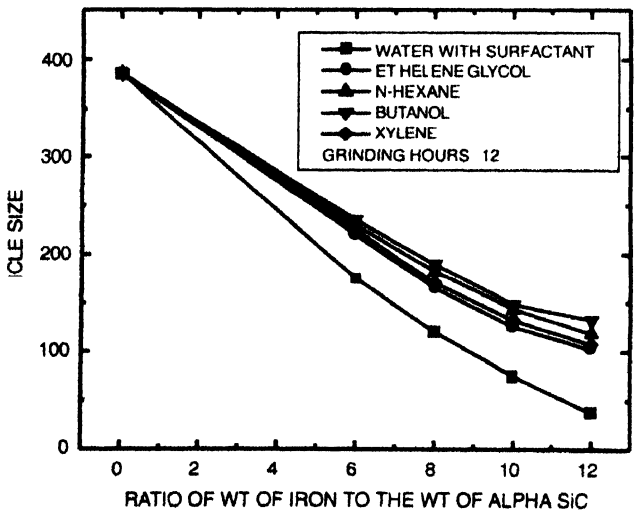

Figure 3. Effect of wt. ratio of iron balls to α-SiC on the particle size of α-SiC.

Figure 4 shows the particle size against the grinding time in hours, which are required to obtain nano size α-silicon carbide particles. It is seen that for a fluid medium of water with surfactants, the particle size continuously decreases with increasing time of grinding, as expected. The effect of grinding time is more pronounced for a fluid medium of water with surfactants, compared to that of the other fluid media, as also observed in Figure 3. It is also seen that nano size particles of α-silicon carbide of an average size of 37 nm are obtained for a minimum of 12 hours of grinding time.

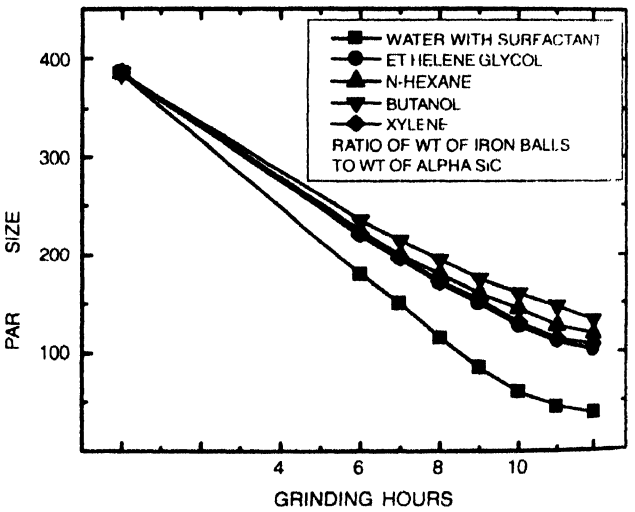

Figure 4. Effect of time of grinding in hours on the particle size of α-SiC

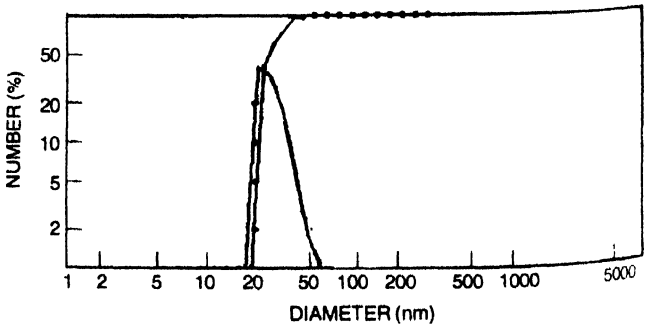

Figure 5. Size distribution of nanocrystalline α-SiC prepared in medium of water with surfactant.

Figures 5, 6, 7, 8 and 9, show the distribution of nano size α-silicon carbide particles for different fluid media of grinding. These five figures reflect the results on the effect of variation of the fluid medium on grinding to obtain nano size particles. From these figures, it can be concluded that the minimum nano size particles of α-silicon carbide of 37 nm can be obtained by attrition grinding of initial α-silicon carbide particles with an average size of 0.39 μm, in the medium of water with surfactants by iron balls with the ratio of the weight of the iron balls to that of α-silicon carbide of 12 :1 for a grinding time of 12 hours.

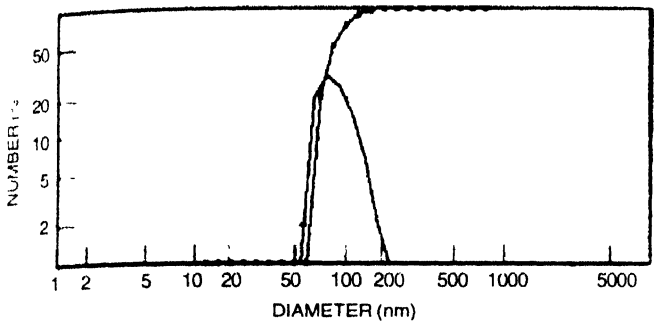

Figure 6. Size distribution of nanocrystalline α-SiC prepared in medium of ethylene glycol

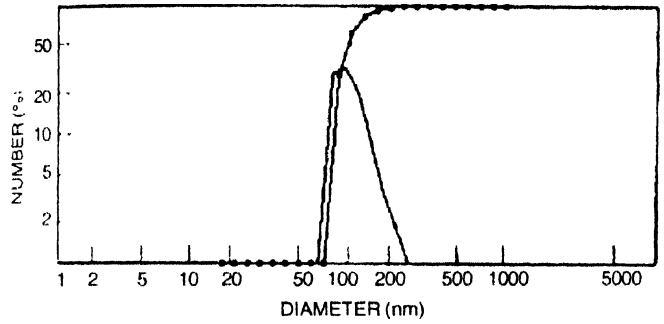

Figure 7. Size distribution of nanocrystalline α-SiC prepared in medium of *n* hexane

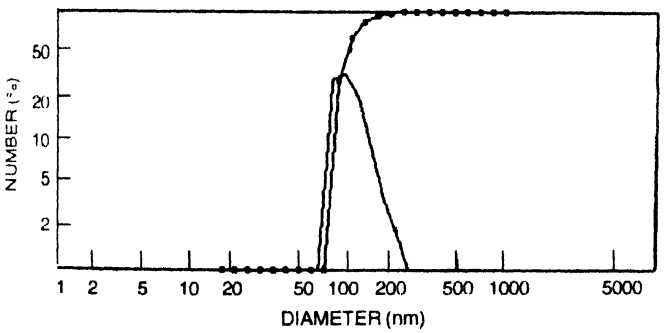

Figure 8. Size distribution of nanocrystalline α-SiC prepared in medium of *n*-butanol.

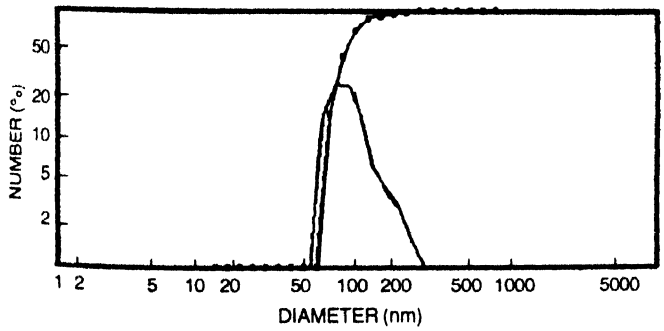

Figure 9. Size distribution of nanocrystalline α-SiC prepared in medium of xylene.

Figure 10 shows the transmission electron micrograph of the final particle size of the nano crystalline particles of α- silicon carbide (*i.e.* 37 nm), which was obtained by attrition grinding of α-silicon carbide of average size of 0.39 μm, in the fluid medium of water with surfactants by iron balls with the ratio of the weight of the iron balls to that of α-silicon carbide of 12 : 1 for a grinding time of 12 hours.

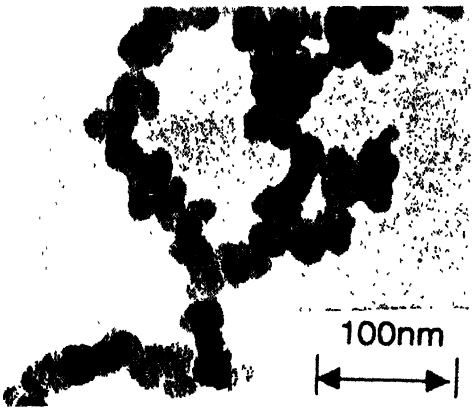

Figure 10. TEM micrograph of α-SiC processed in water with surfactant

4. Conclusion

Nano crystalline α-silicon carbide particle can be prepared from sub-micron size α-silicon carbide particles by attrition grinding with a ratio of weight of iron balls to α-silicon carbide particles of 12 : 1 for the minimum time of 12 hours.

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