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EFFECTS OF PARTICLE SIZE AND POWDER PACKING ON THE SiC FORMING REACTION OF Si AND C

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ABSTRACT

In this study, SiC was synthesized from the reaction between Si and C powders at 1350°C in a vacuum. The effects of particle size and particle packing of Si powder on the reaction were observed. The results showed that the complete reaction to form SiC was obtained from loose powder of all sizes of Si (5-12 μm) hence the particle size of Si in the range of 5-12 μm had no effect on the SiC formation. On the other hand in the closed packed powder system,the complete reaction was found in the large size of Si (12μm) while the small size Si showed an incomplete reaction.

Keywords : Si-C system; Particle size; Particle packing; SiC formation

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INTRODUCTION

Silicon carbide (SiC) is a ceramic material, which is good for high temperature applications. It possesses good mechanical, thermal, and electrical properties at high temperature, such as high strength, high corrosion resistance, high thermal conductivity, high dielectric strength and high electron saturation velocity. With these outstanding properties, SiC is widely applied in mechanical parts and in high temperature and high-power microelectronic devices. It is well known as a highly covalent and difficult-to-sinter substance. Using fine SiC powder is one of the methods to reduce the sintering temperature. Among the various methods to synthesize fine SiC powder, the carbothermal reaction is popular in the industrial process. (Krstic, 1992; Hatakeyama, *et al.* 1990; Kevorkijan, *et al.* 1992; Krishnam, *et al.* 2002; and Lin, *et al.* 2003). Silica and carbon powders are used as raw materials. In this process, the reaction to form SiC can be obtained above 1500°C. Thermodynamic data of SiC forming shows that the reaction of SiO_2-C systems becomes viable at temperatures higher than the reaction of Si-C systems. (Biernacki and Wotzak, 1989; and Ren, *et al. 2003*). According to some information, the method of SiC synthesis (Si-C system) without heating was also studied by using a high energy milling process (El-Eskandarany, *et al.* 1995; and Yang, *et al. 1996*). Although this method can produce SiC at ambient temperature, the purity of product was not described in the reports. In the present study, Si and C was used to produce β-SiC powder at low temperature. To control the Si and C reaction, various factors are required to investigate, such as particle size, particle packing and reaction atmosphere. The morphology and purity of the synthesized powder was examined in relation to particle size and particle packing of Si.

EXPERIMENTAL PROCEDURE

SiC powder was synthesized from the reaction between Si and C powders. The effects of Si particle size and particle packing on the purity of synthesized SiC powder were studied. Si powder of 97% purity was ground at three different grinding times to get three sets of median sizes(5, 8,and 12 μm) of Si powder then mixed with C powder, using a Si:C mole ratio of 1:1. Powder mixing was done in a dry process and then prepared in pellet form and loose powder. Using 0.5 tons pressure to the size of 12 mm diameter and 3.6 mm thickness formed the pellets. After forming the samples were put in a carbon crucible and fired at a temperature of 1350°C for 1 hour in a vacuum with a heating rate of 50°C /min.

The crystalline phases of samples were investigated by X-ray diffractometry (XRD: Model XD-610, Shimadzu).The microstructure of powders was observed by a field emission scanning electron microscope (FESEM: Model JSM-6304F, JEOL). Furthermore, the surface area and particle size of powder were measured by using BET (Model Autosorb-1, Quantachrome) and sedigraph (Model SA-CP3, Shimadzu) methods respectively.

RESULTS AND DISCUSSION

The free energies of Si-C and SiO_2-C systems were calculated at different temperatures and the results were plotted versus temperatures as shown in Figure 1. The SiC forming reactions of both systems show significant free energy difference. The reaction of the SiO_2-C system is possible at temperatures above 1500°C while Si-C and the related reactions occur at much lower temperatures. Therefore to synthesize SiC at low temperature, the Si-C system was chosen for the present study.

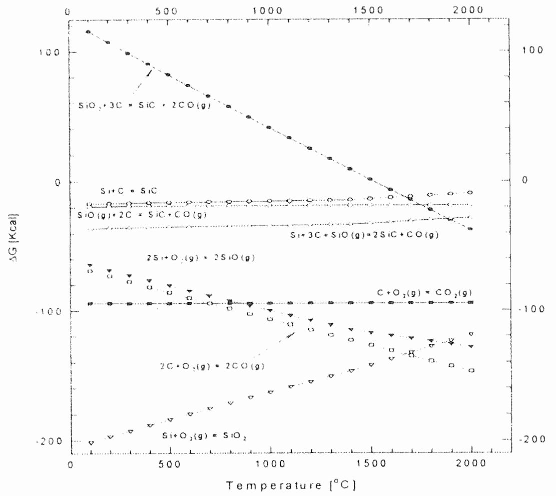

Figure 1 The plots of free energy versus temperature of SiC forming reactions.

The reactions between Si and C in a vacuum atmosphere were observed depending on the size of Si and powder packing. Three different sizes of Si, 12, 8, and 5 μm were used in the mixing with carbon powder. Two forms of specimens, pellet and loose powder, were prepared and fired at 1350°C for 1 h in a vacuum atmosphere. The surface areas of starting and synthesized powder were measured using the BET method as shown in Table 1. It was found that all the synthesized SiC has a larger surface area than that of the starting powder, and the SiC powder synthesized from pellets gives a larger surface area than that of loose powder. However, in Figure 2 the microstructures of both SiC powders do not show much difference. XRD patterns of Figure 3 and 4 show free Si peaks along with SiC peaks in pellet samples with 5 and 8 μm Si. On the contrary, free Si was not found in the sample synthesized from the 12 μm Si in pellet form. In the loose powder batch, the free Si peak was not observed in both samples and prepared from 5 and 12 μm sizes of Si.

Table 1 Surface area of Si, C and SiC powder synthesized at 1350°C for 1 h in vacuum.

Si Size (μm)	Si (m^2/g)	C-CN330 (m^2/g)	SiC, pellet (m^2/g)	SiC, loose (m^2/g)
12	1.62	7.61	20.37	14.36
5	3.63	7.61	27.87	19.14

It has been known that a better reaction can be obtained from the smaller particle size, due to its large surface area. However, the opposite result was obtained in the experiment with closed packing powder as previously mentioned (Figure 3 and 4). Therefore the surface area of Si particles may not be the only major factor of reaction. On the contrary, particle packing is investigated to be more effective than particle size in the SiC formation since the loose powder yields a better reaction than the closed packing powder and the reaction in a loose powder system was independent on the Si size in the study range.

a

b

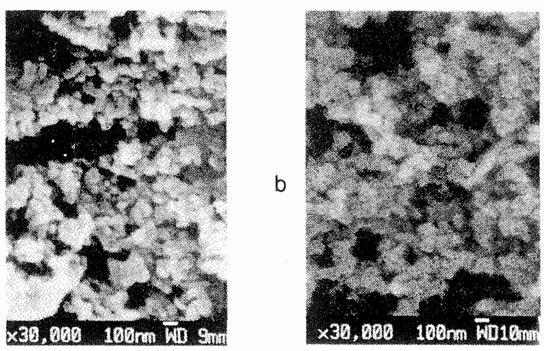

Figure 2 FESEM images of SiC powder synthesized as a) loose powder and b) pellet.

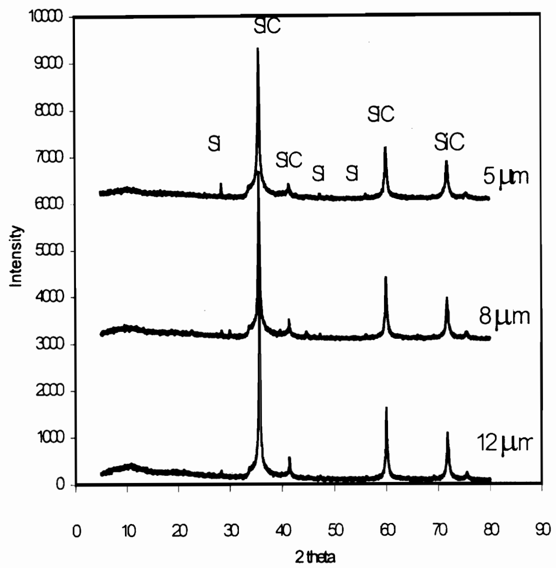

Figure 3 XRD patterns of SiC synthesized as a pellet from various sizes of Si .

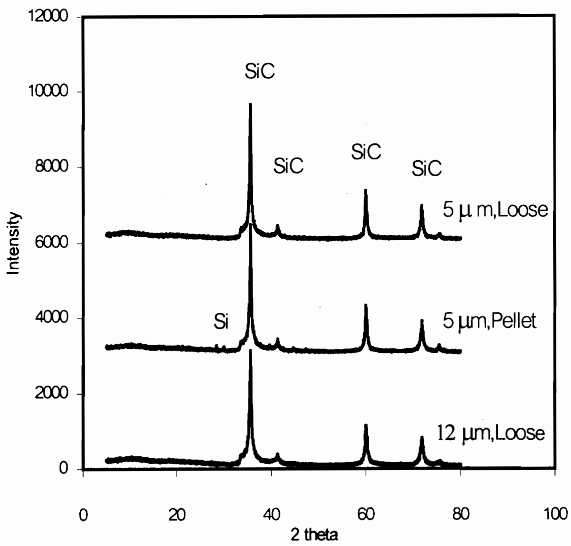

Figure 4 XRD patterns of SiC synthesized from a pellet and loose powder

CONCLUSIONS

According to the free energy values, the synthesis of SiC following the reaction of the Si-C system could be performed at a temperature lower than that of the SiO_2-C system. In the closed pack powder system, the reaction was affected by the size of the Si particles in the way that the larger size of Si resulted in a better reaction. On the contrary, the SiC forming reaction in a loose powder system was independent on the size of Si, and more effective than the closed pack system.

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