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Preparation of Al_2O_3-SiC composite powders in the presence of Fe-catalyst via molten-salt-assisted method

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Abstract

Alumina-silicon carbide (Al_2O_3-SiC) composite powders were synthesized by molten salt method utilizing silicon dioxide (SiO_2), carbon black (C) and aluminium (Al) powders as the starting materials, and sodium chloride (NaCl) and potassium chloride (KCl) as the molten salt medium. The reaction was carried out in the presence of iron (Fe) as a catalyst. The effects of the reaction temperature and Fe (catalyst) content on the formation of SiC were investigated. The optimum synthesis conditions for the Al_2O_3-SiC composite powder were: heating at 1250 °C for 4 h while using 3.0 wt.% Fe as a catalyst. It was also found that the catalyst favours the formation of β-SiC whiskers with diameter range of 30–100 nm, which were larger and longer than those prepared in the absence of the catalyst. It was suggested that the growth of β-SiC whiskers in the presence of Fe catalyst follows the solid-liquid-gas-solid growth mechanism.

Keywords: *Al_2O_3-SiC, β-SiC whisker, Fe catalyst, molten salt method, solid-liquid-gas-solid growth*

I. Introduction

Aluminium-silicon carbide composites combine the excellent properties of alumina (Al_2O_3) and silicon carbide (SiC) in a single material, such as excellent bending strength, fracture toughness, good corrosion resistance, wear resistance, high-temperature corrosion resistance and other properties [1–4]. When silicon carbide is present in a whisker form in ceramic-ceramic composites, it can improve their mechanical properties, especially by increasing fracture toughness and creep resistance at high temperatures, which is attractive for demanding applications [5,6]. The morphology and structure of SiC whiskers are important in this regard, which are influenced by different growth mechanisms in the presence of different catalysts such as metal elements (Fe, Co, Ni) and rare earth oxides (Y_2O_3, Sm_2O_3, La_2O_3) used for the synthesis [7–9]. These catalysts not only promote the growth of SiC whiskers, but also increase the reaction rate. For instance, Wang *et al.* [10] used tetraethoxysilane (TEOS) and sucrose as raw materials and iron(II) chloride ($FeCl_2$) as a catalyst to prepare SiC nanowires with a diameter of 100–300 nm and a length of tens to hundreds of micrometres by carbothermal reduction of the carbonaceous silicon dry gel. The addition of $FeCl_2$ was found to promote the carbothermal reduction at lower temperature. Li *et al.* [7] studied the effects of Fe and Ni on the yield and morphology of SiC nanostructures prepared in a chemical gas reactor at 1300 °C using silicon-silica (Si-SiO_2) powder and methane (CH_4) gas as the raw materials. All the prepared nanostructures were identified as β-SiC single crystals that grew in the [111] direction.

Becher *et al.* [11] used hot-pressed SiC whiskers to strengthen alumina and found that the fracture toughness value of the composite at 20 vol.% of SiC reached a value of 8.7 MPa·$m^{1/2}$. Gac *et al.* [12] prepared a composite material composed of 20 vol.% SiC whiskers dispersed in a hot-pressed MoSi matrix. The bending strength and fracture toughness of the composite material increased by 100% and 54% compared to the virgin matrix, respectively.

Due to the high melting point of alumina and silicon carbide, the synthesis of Al_2O_3-SiC composites requires a high synthesis temperature (sintering tem-

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perature >1450 °C) [13–15] resulting in high energy consumption and long processing time. Currently, the molten salt method is widely employed to prepare high-temperature-melting inorganic composite powders. For example, Dai *et al.* [16] synthesized Ti_2SC powder with Ti powder, carbon black and TiS_2 as starting materials, NaCl and KCl as molten salt medium under argon atmosphere, and heating at 1150 °C for 1.5 h. The experimental results showed that the molten salt method was a simple and economic method for synthesizing Ti_2SC powder. The average particle size of the synthesized Ti_2SC nanocrystals was 22.6 nm. Sun *et al.* [17] synthesized Al_2O_3-SiC composite powder by the molten salt method using silicon dioxide, carbon black and Al powder at 1573 K for 4 h.

In the present study, Al_2O_3-SiC composite powder is synthesized via a molten salt method utilizing silica fume (SiO_2), Al powder and carbon black as Si, Al and C sources, respectively. Sodium chloride and potassium chloride (NaCl-KCl) mixture is used as the molten salt medium, whereas Fe is added as a catalyst to the reaction mixture. The effect of the process temperature and the amount of catalyst on the phase composition and morphology of the product are studied, and the growth mechanism of SiC whisker grown *in situ* during the synthesis process is revealed.

II. Experimental

2.1. Sample preparation

Al_2O_3-SiO_2 composite powders were synthesized via the molten salt method based on the following equation:

$$4\,Al + 3\,SiO_2 + 3\,C \longrightarrow 2\,Al_2O_3 + 3\,SiC \qquad (1)$$

Silica fume (93% purity; particle size: ~10 μm; Hebei Kexu Building Materials Co. Ltd, China) was used as the raw source material for the Si. Ferrous powder (>99% purity; particle size: ~3.5 μm; Qinghe County Yuan Yao Alloy Products Co. Ltd, China) was used as the catalyst. Aluminium powder (>99% purity; particle size: 2.6 μm; Gongyi Borun Refractory Materials Co. Ltd, China) was used as a reducing agent and source of Al. Carbon black (>99.9% purity; particle size: ~15 μm; Zhengzhou Xinke Chemical Products Co. Ltd, China) was utilized as the carbon source. NaCl and KCl (99.5% purity; Sinopharm Chemical Reagent, Shanghai Co. Ltd, China) were employed together as the molten salt medium.

The mass of each raw material was calculated according to the proportion n(Al) : n(SiO_2) : n(C) = 8 : 3 : 12 from previous work [18]. The added amounts of Fe were 1, 2, 3 and 5 wt.% of the total mass of the reactant. The raw materials were weighed in the predetermined proportions and mixed with salts using a ball mill. The mixture samples were then placed in alumina crucibles provided with lids in a high-temperature tube furnace for high-temperature synthesis. Simultaneously, high-purity argon gas was pumped into the furnace at a flow rate of 20 ml/min, and the reaction temperature was raised to 1100, 1150, 1200, 1250 or 1300 °C at a heating rate of 5 °C/min and held constant for 4 h. After the tube furnace had cooled down to room temperature, the samples were taken out and rinsed several times with hot distilled water to wash away the residual salt in the product. Finally, the washed samples were dried in a drying oven at 100 °C for 12 h to obtain the finished product for characterization.

2.2. Characterization

The phase composition of the composite powder was analysed through X-ray diffraction (XRD; Cu $K\alpha$ radiation; PANalytical X'Pert Powder, Netherlands). The obtained X-ray diffractogram was analysed using Highscore Plus software and compared with the standard cards to determine the phase composition of the product. The microstructures of the powders were observed under field-emission scanning electron microscopy (FE-SEM; SUPRA™ 55, Germany) coupled with an energy-dispersive X-ray spectrometer (EDS).

III. Results and discussion

3.1. Effects of temperature on phase composition

Figure 1 shows the XRD diffractograms of the Al_2O_3-SiC composite powders prepared by the molten salt method utilizing 3 wt.% Fe catalyst at different temperatures for 4 h. In the powder synthesized at 1100 °C, different peaks of α-Al_2O_3 and 6H-SiC associated with Si and Fe_3Si characteristic peaks were detected, which indicated that a reaction occurred between the Fe catalyst and SiO_2. The minor proportion of the 6H-SiC phase that appeared in the composite powders is attributed to

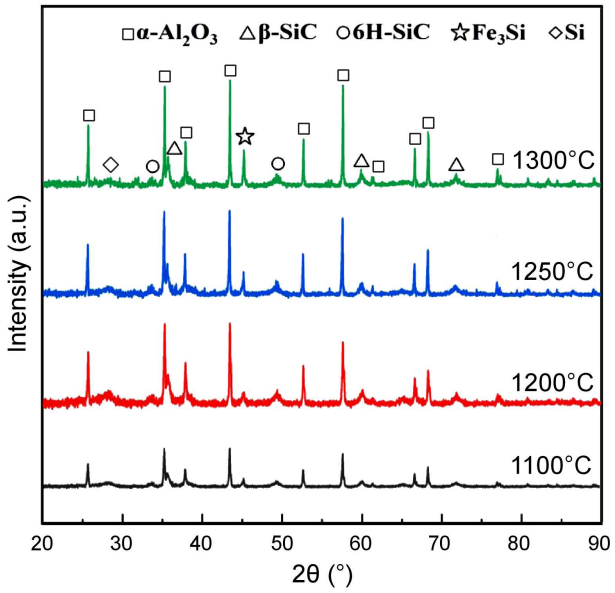

Figure 1. XRD diffractograms of Al_2O_3-SiC composite powders prepared by molten salt method with 3 wt.% Fe catalyst at different temperatures for 4 h

the presence of Al and C, promoting the transformation of β-SiC to the 6H-SiH phase. With the increase in the synthesis temperature to 1200 °C, the diffraction peaks of α-Al_2O_3, 6H-SiC and Fe_3Si became significantly stronger, while the intensity of Si decreased. At 1250 °C, the diffraction peak of Si almost disappeared while the intensity of other phases enhanced further. The strong diffraction peak observed at 45.6°, which is characteristic of Fe_3Si [19], confirmed that Fe_3Si acted as a catalyst during the formation of SiC whiskers. The further rise in temperature to 1300 °C had no obvious effects on the diffraction peak intensities of individual phases. Therefore, it can be suggested that the optimum synthesis temperature is 1250 °C to synthesize Al_2O_3-SiC composite powder while using Fe as a catalyst. Thus, compared with the optimal synthesis temperature of 1300 °C for the composite powder prepared without Fe catalyst [17], the addition of Fe effectively reduced the synthesis temperature by 50 °C.

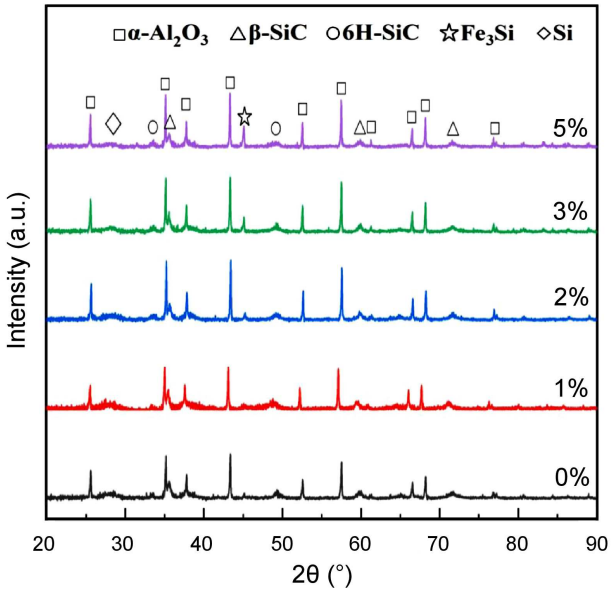

Figure 2. XRD diffractograms of the products synthesized by adding different amounts of Fe at 1250 °C for a 4 h reaction

3.2. *Effect of Fe amount on phase composition*

To explore the effect of added amount of catalyst on the product, Fig. 2 depicts the X-ray diffractograms of the products synthesized by adding different amounts of Fe at 1250 °C in a 4 h reaction. The addition of catalyst can significantly promote the transformation from Si to SiC. Therefore, the diffraction peak intensity of silicon in the sample decreased with the increase in iron catalyst content. When no Fe catalyst was added to the sample, the main crystalline phases were α-Al_2O_3 and β-SiC, with a small quantity of Si. By increasing the Fe content to 1 wt.%, the diffraction peaks of α-Al_2O_3 and β-SiC became stronger while the intensity of the Si peak became weaker. At this point, the main crystalline phases were α-Al_2O_3 and β-SiC, with a small quantity of Fe_3Si and unreacted Si. By increasing the Fe content to 2 wt.%, the diffraction peaks of α-Al_2O_3 and β-SiC became significantly stronger, while the intensity of the Si peak became weaker. When the added amount of Fe was increased to 3 wt.%, the Si phase almost disappeared. Under this condition, only α-Al_2O_3, β-SiC and Fe_3Si were detected in the product. This indicated that the reaction was completed for the amount of Fe used, and any further increase in the catalyst amount would have no effect on the phase composition of the product. When the added amount of Fe was 5 wt.%, the number of whiskers decreased and the whiskers became curved (Fig. 3f), which might influence the mechanical proper-

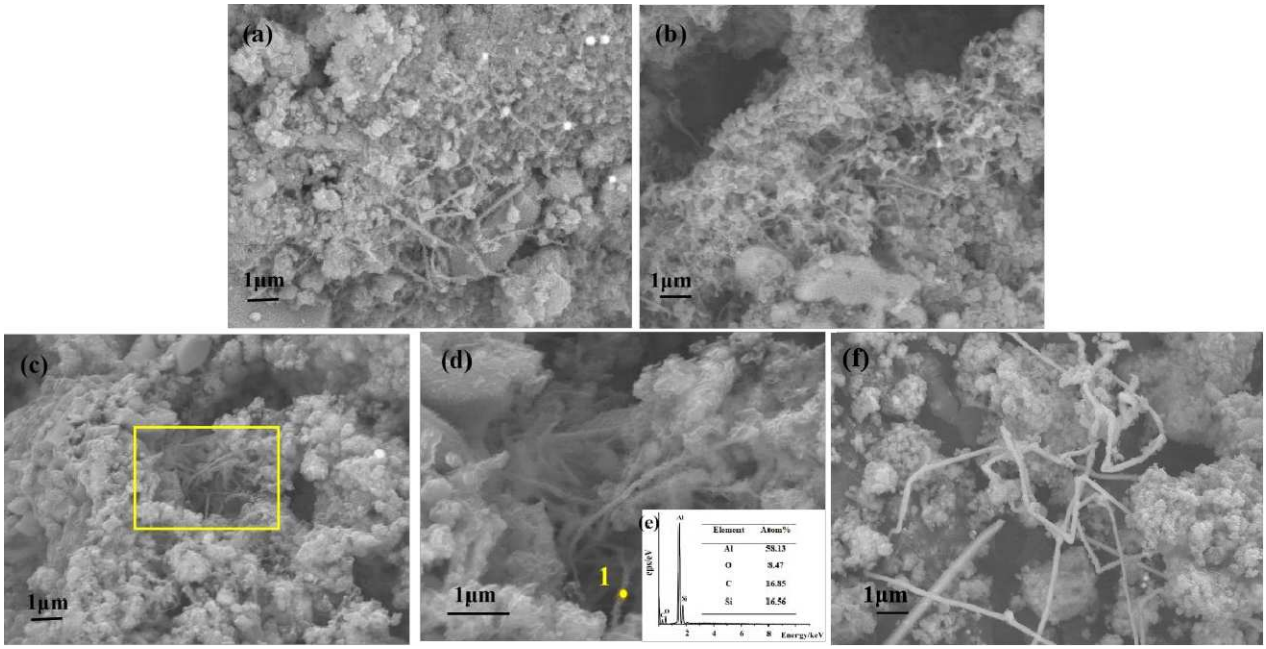

Figure 3. SEM micrographs of Al_2O_3-SiC composite powders synthesized at 1250 °C for 4 h and adding different amounts of Fe: a) 1 wt.%, b) 2 wt.%, c,d) 3 wt.% and f) 5 wt.%; (inset in (d) is EDS spectrum of point 1 in (c))

ties of the Al_2O_3-SiC composites. To sum up, the optimal amount of Fe catalyst is suggested to be 3 wt.% under the reaction conditions.

3.3. Microstructures of Al_2O_3-SiC composite powder

Figure 3 shows SEM micrographs of the Al_2O_3-SiC composite powders synthesized at 1250 °Cin a 4 h reaction with different Fe amounts. The prepared samples comprised of both α-Al_2O_3 particles and β-SiC whiskers. The SEM images revealed that with the increase in Fe content from 1 to 3 wt.%, the amount of SiC whiskers increased gradually. When the added amount of Fe was 5 wt.%, the number of whiskers decreased and the whiskers became curved (Fig. 3f), which was probably due to the increase in the amount of added catalyst. Although the active sites for growth of SiC whiskers increased, the relative concentration of SiO gas ($SiO_2(s) + C(s) \rightarrow SiO(g) + CO(g)$) decreased, rendering it insufficient to meet the growth demand of the whiskers. This can be combined with the XRD analysis (Fig. 2) and the fact that in the composite powder with 3 wt.% of Fe only α-Al_2O_3 and β-SiC exist in the product which contains the β-SiC whiskers with a diameter of 30–100 nm and a length of several microns (Fig. 3). The β-SiC whiskers can be seen stacked in the matrix in the form of very long, fine fibres intertwined with each other. It can also be observed that most of the whiskers are generated in empty spaces. Relevant studies have shown that the whisker morphology of SiC can only develop well under the condition that locally available CO gas is sufficient [20]. EDS analysis (Fig. 3e) at point 1 of Fig. 3d indicated that the whisker surface comprised of Al, O, Si and C elements, and that the atomic ratio of Si and C was close to 1 : 1, which corresponds to the results of the XRD (Fig. 2). This confirms that the product phase is composed of Al_2O_3 and SiC. The above analysis indicates that the addition of an appropriate amount of Fe as a catalyst contributes to the formation of the whisker morphology.

3.4. Growth mechanism and Fe catalyst mechanism

The vapour-liquid-solid (V-L-S) mechanism is often used to explain the growth mechanism of SiC whisker in the presence of a catalyst. Figure 3d shows the morphology of the SiC whisker synthesized with Fe as a catalyst. It can be seen that the tip of the SiC whisker does not have the morphology characteristic of the whisker growing in line with the V-L-S mechanism. In addition, the absence of small droplets on the whiskers tips observed in Fig. 3f suggests that there may be other mechanisms for the Fe-catalysed growth of SiC whiskers. Yang *et al.* [21,22] found that after the preparation of silicon nitride by the pyrolysis of polysilazane precursor in the presence of $FeCl_2$, many silicon nitride nanoribbons did not have small droplets on the tip of the product phase. Subsequently, he proposed a new growth mechanism: the solid-liquid-gas-solid (SLGS) mechanism. The mechanism suggests that at the beginning of the process, the

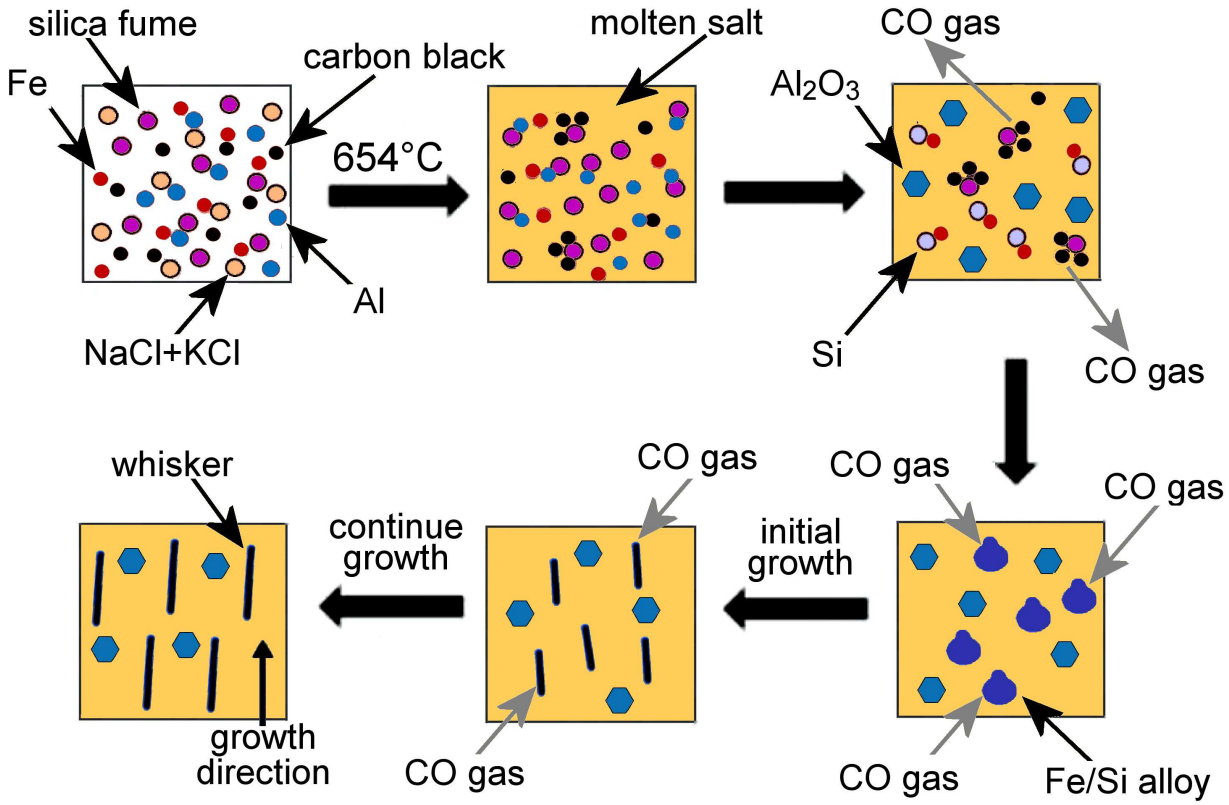

Figure 4. Schematic illustration of the synthesis mechanism of Al_2O_3-SiC composite powders by molten-salt method using Fe as a catalyst

amorphous SiCN reacts with Fe to form a liquid Si-Fe-C alloy at a temperature higher than the eutectic temperature of the Si-Fe-C ternary system while releases N_2 gas. Further reaction of the SiCN and the liquid alloy forms a liquid phase supersaturated with Si and C. This supersaturated liquid phase then reacts with N_2 gas (at least part of N_2 was provided by the protection gas) on the liquid/gas interface to precipitate the Si_3N_4 nanobelts. In addition, Yang *et al.* [23] also proposed the SLGS mechanism for the synthesis of Si_3N_4 to explain the whisker formation mechanism.

Based on the above analyses, the SiC whiskers generated in the presence of Fe catalyst without small droplets on the tip in the present study can also be explained by the SLGS mechanism. The occurrence process of SLGS in the SiO_2-Al-C system can be described by the following steps:

1. Fe and the reduced Si atom ($3\,SiO_2(s) + 4\,Al(s) \rightarrow 2\,Al_2O_3(s) + 3\,Si(s)$) formed Fe/Si alloy.
2. The Si in the low eutectic Fe/Si alloy reacted with CO gas (formed by the reaction of C with the residual oxygen in the high-temperature furnace or the reaction of silicon dioxide with C) to form SiC crystal nuclei at the gas-liquid interface. When the SiC crystal nucleus reached supersaturation in the droplet, it precipitated.
3. The precipitated SiC crystal continuously absorbed the Si element from the small droplet and the carbon element from the CO and eventually grew into whiskers.

The proposed mechanism is illustrated in Fig. 4. It is important to mention that the SLGS (solid-liquid-gas-solid) mechanism is very similar to the VLS mechanism; the only difference is that the SLGS mechanism proposes that the Si element is derived from the liquid phase, while the VLS mechanism suggests that the Si element is derived from the gas phase (such as SiO gas).

During the catalysis of Si powder to prepare SiC, by metal nanoparticles form alloys with Si, thus playing a catalytic role [24,25]. As it is well-known, the reaction of Si and C to form SiC includes the breaking of Si–Si bond and the formation of Si–C bond. The existence of Si–Si bond makes it difficult for silicon particles to decompose into silicon atoms.

The above experimental results and analysis show that Fe plays an important role in accelerating the transition from Si to SiC. Therefore, the role of Fe in the present study can be described as follows:

1. Fe was first adsorbed on Si and then formed an Fe/Si alloy with Si;
2. With the formation of Fe/Si alloy, the bond length of the Si–Si bond increased, which made the stability of Si atoms lower, resulting in easier activation of the Si atoms;
3. Subsequently, the activated Si atoms readily reacted with the carbon atoms so that SiC could be formed at relative low temperatures.

IV. Conclusions

Al_2O_3-SiC composite powders were prepared by molten salt method at 1250 °C for a 4 h reaction utilizing Fe as a catalyst, while silica fume, Al and carbon black were used as raw materials. The optimum amount of Fe was 3 wt.% of the total reactant mass. The addition of Fe was found to effectively reduce the synthesis temperature of the composite powder by about 50 °C. The phase composition and morphology of the product were affected by the synthesis temperature and the amount of catalyst added. Increasing the temperature (1100–1250 °C) and the content of the catalyst (1–5 wt.%) were conducive to the growth of SiC whiskers. The resulting whiskers ranged in diameter from 30 to 100 nm and had a length of several micrometres. During the synthesis process, Fe is suggested to promote the formation of SiC whiskers by forming Fe/Si alloy in the first reaction step, which follows the solid-liquid-gas-solid mechanism of the whiskers' growth.

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