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Effects of different carbon sources and reaction temperatures on the synthesis of SiC-TiC composite powders by carbothermal reduction

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SiC-TiC composite powders were synthesised in argon atmosphere by a carbothermal reduction method using silica sol, TiO₂, and different carbon sources (i.e., graphite and starch) as the raw materials. The effects of carbon source types and reaction temperatures on the phase composition, particle size distribution, and microstructure of SiC-TiC composite powders were investigated. The reaction mechanism of SiC-TiC composite powders was also discussed. Results showed that SiC-TiC composite powders can be synthesized under optimum conditions (i.e., 1600 °C for 2 hrs using graphite as carbon source). The SiC-TiC composite powders prepared at 1600 °C comprised a small amount of large-sized tabular grains, a large number of fine irregular rod-like and approximately spherical particles 200-400 nm in size using graphite as carbon source. The SiC-TiC composite powders prepared at 1550 °C were composed of flaky particles, spherical particles (~ 100-200 nm), and whiskers (50-200 nm in diameter) using starch as carbon source. The growth mechanism of SiC whiskers synthesised in powder samples followed the vapor-solid mechanism.

Key words: Graphite, Starch, SiC-TiC, Composite powders, Synthesis, Mechanism.

Introduction

Silicon carbide (SiC) ceramics possesses several advantages, such as high temperature strength, high thermal conductivity, good wear and corrosion resistance, and excellent oxidation resistance, making it a promising engineering material [1-3]. SiC is used in numerous fields, e.g., machinery, chemical industry, textile, petroleum, environmental protection, energy, military industry and aviation. However, the application of SiC ceramics is limited by its low room-temperature strength, poor fracture toughness, and high flaw sensitivity [4].

Introducing a transition metal boride or carbide as a dispersed phase into the SiC matrix is an effective solution [5]. Titanium carbide (TiC) is one of the most essential transition metal carbide discovered, that attracted great interest because of its excellent properties, including high Vickers hardness (28-33 GPa), high melting point (> 3000 °C), high Young's modulus (410-450 GPa), outstanding chemical stability, and high fracture toughness. These characteristics makes TiC widely used for cutting tools, wear-resistant parts, bullet-proof armor, aviation components, etc [6-7]. Reports concerning the effects of TiC particles dispersed in a SiC matrix have been published [8-12]. To improve the mechanical properties of SiC-TiC

composites, Robavie et al. [9] prepared SiC-TiC composites by hot-pressing sintering using finely grinded SiC and TiC particles as the raw materials, and investigated the influence of various processing parameters on microstructure resulting to flexural strength and fracture toughness. Balasubramanian et al. [10] investigated the effects of initial a-phase content of SiC on microstructure and mechanical properties of the hot-pressed sintering and subsequently annealed SiC-30 wt.% TiC composites. Chen et al. [11] fabricated SiC-TiC composites using TiC and SiC powders as raw materials at the range of 1650-2000 °C in Ar atmosphere by a two-step method and studied the mechanical, thermal, and electrical properties of SiC-TiC composites. Chen et al. [12] prepared TiC/SiC composites from Ti-enriched slag through an electro-deoxidization process in molten salts in argon and suggested a possible reaction pathway.

Most of the research work described above involves the direct mechanical mixing of SiC and TiC powders, and then sintering the powder mixture to prepare SiC-TiC composites. Nevertheless, mechanical mixing of the powders introduces inhomogeneity to the SiC-TiC composites. Therefore, high-quality SiC-TiC composite powders with uniform and non-agglomerating properties are crucial in the preparation of SiC-TiC composites with excellent properties. Various methods for synthesis of single SiC and TiC ultrafine powders, such as carbothermal reduction method [13-14], combustion method [15], mechanochemical method [16], and sol-gel method [17]. Among the synthetic methods available, the

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carbothermal reduction method is the most commonly chosen route to produce SiC and TiC powders because of simple processing and low-cost preparation. Unfortunately, reports on the synthesis of SiC-TiC composite powders by the carbothermal reduction method are limited.

In our previous studies, the SiC-TiC composite powders were successfully synthesised through carbothermal reduction using silica sol, carbon black, and titanium oxide as raw materials. The effects of reaction temperatures on the phase compositions and morphologies of SiC-TiC composite powders were investigated [18]. In the present work, SiC-TiC composite powders are synthesised in Ar atmosphere in a tube furnace by carbothermal reduction using varieties of carbon sources (such as graphite and starch) as raw materials. Effects of these variations, such as the phase composition, loss rate of reaction process, the particle size distribution, and the microstructure of SiC-TiC composite powders were investigated. Furthermore, the mechanism of synthesis reaction was also analysed.

Experimental

Silica sol (SiO₂, 28 wt%), TiO₂ (purity: \geq 99.0%, $D_{50} = 1.70 \ \mu m$), graphite (purity: $\geq 99.0\%$), and starch (purity: ≥99.0%) were used as raw materials for the preparation of SiC-TiC composite powders. The amounts of TiO₂, silica sol and varieties of carbon sources were adjusted to yield SiC-30 mol% TiC. The procedure is shown in Fig. 1. The raw materials were accurately weighed according to the proportion of each. These ingredients were uniformly mixed in a planetary ball mill in ethanol for 2 hrs using SiC balls as the grinding body. The well-mixed raw materials were dried at 80 °C for 24 hrs, placed into a graphite crucible and then placed in the high-temperature tubular furnace. Heating was performed at 10 K min⁻¹ from room temperature to 1300-1600 °C in an argon atmosphere. The holding time at the desired temperature was 2 hrs. Subsequently, the furnace was cooled naturally and the composite powders were obtained. The phase composition of powder samples synthesised at different reaction temperatures was analysed by X-ray diffraction (XRD, TD-3500). The quality of the powder samples before and after the reaction was measured by an electronic analytical balance to determine the extent of the high temperature reaction process. The particle size and distribution of powder samples were measured by laser particle size analyser (LPSA, Easysizer



Fig. 1. Procedure of preparation of SiC-TiC composite powders.

20). The morphology of the composite powders was observed by scanning electron microscopy (SEM, Quanta FEG 250).

Results and Discussion

Effect of reaction temperature on the phase composition of products

Fig. 2 shows the results of XRD analysis of powder samples synthesised at different reaction temperatures using graphite as the carbon source. The reaction temperature has a great influence on the phase composition of the synthesized powder samples. When the reaction temperature is at 1300 °C, the diffraction peaks of TiO₂ and C are observed on the XRD patterns of the obtained powder samples, and no SiO₂ diffraction peaks are found. This indicates that the amount of the carbothermal reduction reaction between the TiO₂ and the graphite mixture is still small at 1300 °C; moreover, the SiO₂ component in the silica sol material is present in the amorphous form in the mixture. When the reaction temperature is at 1400 °C, the XRD patterns of the synthesized powder samples changed significantly. In addition to the diffraction peak of C, the diffraction peak of Ti₃O₅ and SiO₂ appeared, whereas, the diffraction peak of TiO₂ disappeared. The results showed that the carbothermal reduction of TiO2 and graphite produces an amount of intermediate Ti₃O₅ at the reaction temperature. When the reaction temperature is increased to 1500 °C, the XRD pattern of the synthesised powder samples remained constant. Strong diffraction peaks of C, SiC and TiC in the powder samples resulted when the reaction temperature is increased to 1550 °C, whereas, the diffraction peak intensity of Ti₃O₅ and SiO₂ is weak., Some diffraction peaks of C in the XRD pattern aside from the main crystalline SiC and TiC, and the other impurity peaks are less when the reaction temperature reaches 1600 °C. The diffraction peak of SiC is more evident, and the shape of its characteristic peak is sharper.



Fig. 2. XRD patterns of the synthesised samples at different reaction temperatures using graphite as carbon source.



Fig. 3. XRD patterns of the synthesised samples at different reaction temperatures using starch as carbon source.

This specifies that more SiC and TiC powders are formed at 1600 °C, and C-SiC-TiC composite powders with a certain amount of C is obtained. Various studies have shown [19-23] that a small amount of graphite particulates in the metal matrix composites containing SiC have a positive effect on the mechanical and thermal properties of the resulting products because graphite particulates possess an excellent combination of good high-temperature resistance, good thermal conductivity, good thermal shock resistance, high strength, and low density. Therefore, SiC-TiC composites containing graphite particulates are excellent materials for future applications, having a wide application prospect in the field of high thermal conductivity and mechanical property.

Fig. 3 shows the XRD patterns of the powder samples synthesised at different reaction temperatures using starch as the carbon source. The diffraction peaks of TiO₂ and Ti₃O₅ are observed in the XRD pattern of the synthesised powders when the reaction temperature is at 1300 °C indicating that the carbothermal reduction of the elemental carbon produced by decomposition of starch raw material and TiO₂ begins to form intermediate Ti₃O₅ at the reaction temperature. When the reaction temperature is increased to 1500 °C, the XRD patterns of the synthesised powder samples change significantly. The diffraction peaks of Ti₃O₅ and TiO₂ disappeared in the XRD patterns of SiC and TiC. This indicates that the carbothermal reduction in the mixture system has occurred at high temperature, generating a large amount of SiC and TiC powders. When the reaction temperature is further increased to 1550 °C, the diffraction peak intensity indexed on SiC and the XRD patterns increased further, indicating that the system generates more SiC powders. When the reaction temperature reaches 1600 °C, compared with 1550 °C, XRD patterns do not change, the main crystalline phase is still SiC and TiC, but the shape of the characteristic peak of the powder samples is sharper, indicating that the generated SiC and TiC

powders have larger grains and better crystallinity at 1600 °C.

Degree of the synthesis reaction

The formation of CO gas during the high-temperature reaction of the SiC-TiC composite powders synthesised by the carbothermal reduction method leads to the loss of the quality of the powder samples. Therefore, the ignition loss is calculated from the mass loss of the powder samples caused by the escape of CO gas used to evaluate the extent of the reaction synthesis process of the SiC-TiC composite powders. A greater ignition loss implies a higher degree of completion of the reaction. In this study, the ratio of the measured weight loss to the theoretical calculated mass loss (the relative ignition loss) is used to determine the extent of the reaction. The relative ignition loss of SiC-TiC composite powders synthesised at different reaction temperatures using different carbon sources as raw materials is shown in Table 1. The relative ignition loss gradually increases with increased reaction temperature.

When graphite is used as the carbon source, the relative ignition loss of the samples is only 10.6% at 1300 °C. As the temperature is increased further to 1400 and 1500 °C, the relative ignition loss of the samples, likewise, increased to 28.8% and 35.8%, respectively. The relative ignition loss of the sample reached 105.5% when the reaction temperature is increased to 1600 °C, exceeding the calculation of the loss of 5.5%. These results suggest that the synthesis reaction of SiC-TiC composite powders is completed when the reaction temperature is at 1600 °C, in accordance with the previous XRD results. The ignition loss is higher than the calculated value at 1600 °C because [24] gaseous SiO is generated in the reaction process at high temperatures, it does not completely change into SiC and a little quantity with the CO gas is taken away by the flowing argon. This caused the actual ignition loss to be higher than the calculated value.

When starch is selected as the carbon source, the ignition loss of the powder samples reaches 91.2% at 1300 °C. The mass loss at this temperature is caused by the H₂O gaseous matter produced by the decomposition

Table 1. Relationship between the ignition loss and reaction temperature in the synthesis process of SiC-TiC composite powders with different carbon source at different reaction temperatures.

Reaction temperature /	Mass fraction /%		
°C	Graphite	Starch	
1 300	10.6	91.2	
1 400	28.8	96.4	
1 500	35.8	105.7	
1 550	94.3	118.6	
1 600	105.5	122.0	



Fig. 4. TG-DTA curve of single starch raw material.

of starch and the CO gas produced during the reaction of TiO₂ with carbon. At 1500 °C, the relative ignition loss reaches 105.7%, which is higher than the calculated value. A large amount of SiC and TiC ultrafine powders are formed during the intense synthesis reaction at 1500 °C. The relative ignition loss increases to 118.6% when the temperature is increased to 1550 °C. When the reaction temperature continues increase up to 1600 °C, the ignition loss is 122.0%, close to the value of the product obtained at 1550 °C. This research implies that the carbothermal reduction can reach completion at 1550 °C. In this study, ignition loss during the synthesis of SiC-TiC composite powders at 1300 °C is high, the higher relative ignition loss compared with the calculated value at above 1500 °C may be explained as follows: because the carbon source selected in this experiment is an organic starch, the starch in the raw material decomposes to produce carbon and H2O gaseous matter, but also decomposes to produce other gaseous products, such as CO, CO₂, C_xH_y, etc. at higher reaction temperature, thereby, leading to mass losses of the samples [25]. If a single starch is completely decomposed to form only carbon and H₂O gaseous matter, the ignition loss can be calculated as 55.6%. However, the TG-DTA curve of a single starch material (Fig. 4) at high temperature produces a mass loss of up to 78.2%, thereby verifying the results of the above-mentioned document [25]. Furthermore, during the carbothermal reduction of SiO₂ and carbon to form SiC powders, the resulting SiO gaseous material, along with CO gas, partially escapes from the system, resulting in more mass loss in the system [24].

Particle size distribution and morphology analysis of powder samples

The particle size distribution of SiC-TiC composite powders synthesised at the optimum reaction temperature was analysed, and the results are shown in Table 2. When the carbon source is graphite, the particle size of the sample synthesis at optimum reaction temperature (1600 °C) is larger, the median diameter is $D_{50} = 14.36$ µm; whereas, when the carbon source is starch, the median particle size of the powder sample synthesis at optimum reaction temperature (1550 °C) is $D_{50} = 1.02$ µm. When starch is selected as the raw material of the carbon source, the SiC-TiC composite powders synthesised under the reaction conditions are relatively fine and have a small particle size as a whole.

Fig. 5 shows the SEM photographs of SiC-TiC composite powder samples synthesised using graphite as the carbon source at 1550 and 1600 °C for 2 hrs, respectively. The composite powder samples prepared at 1550 °C consisted of larger particles, larger amounts of irregular rod-shaped particles, and nearly spherical particles. According to previous XRD patterns, SiC, TiC and a certain amount of C diffraction peaks in the powder sample. In combination with their crystal structure, larger flake particles should be the graphite particles in the raw material system [26], whereas, the irregular rod-shaped particles and the nearly spherical particles should be the mixed particles of SiC and TiC



Fig. 5. SEM morphologies of SiC-TiC composite powders synthesised at 1550 and 1600 °C using graphite as carbon source. (a) 1550 °C, low magnification; (b) 1550 °C, high magnification; (c) 1600 °C, low magnification; (d) 1600 °C, high magnification.

Table 2. Particle size distribution of the synthesised SiC-TiC powders at suitable reaction temperatures.

NO.	Carbon source	Reaction temperatures /°C	$D_{10}/\mu\mathrm{m}$	$D_{25}/\mu m$	$D_{50}/\mu m$	$D_{75}/\mu m$	$D_{90}/\mu m$
1	Graphite	1600	1.80	3.68	14.36	22.24	40.57
2	Starch	1550	0.56	0.73	1.02	1.35	1.65



Fig. 6. SEM morphologies and EDS pattern of SiC-TiC composite powders synthesised at 1550 and 1600 °C using starch as carbon source. (a) 1550 °C, low magnification; (b) 1550 °C, high magnification; (c) 1600 °C, low magnification; (d) 1600 °C, high magnification.



Fig. 7. EDS pattern of the whisker in Fig. 6.

[18]. As the reaction temperature increased to 1600 °C, the larger particles in the synthesised product (mainly graphite particles) were significantly reduced, indicating that at the reaction temperature, the graphite particles in the feedstock system react with SiO₂ to form more SiC particles, which is consistent with the results of XRD analysis in Fig. 2. Figs. 5(c) and 5(d) show that the composite powders prepared at 1600 °C are composed of a small amount of large-sized tabular grains, a large number of fine irregular rod-like, and approximately spherical particles 200-400 nm in size, moreover a serious agglomeration or cohesion phenomenon between the powder sample particles, results in a larger particle size than the actual particle size of the SiC-TiC composite powder samples (the sample particles are mainly composed of secondary aggregates).

Fig. 6 shows the SEM photographs of SiC-TiC composite powder samples synthesised with starch as

Table 3. Relationship between ΔG^{θ}_{T} and T for possible reactions during high-temperature synthesis of SiC-TiC composite powders.

No.	Reaction equation	$\Delta G^{\theta}_{\mathrm{T}}/\mathrm{KJ}\cdot\mathrm{mol}^{-1}$	Critical reaction temperature / K
(1)	$SiO_2(s) + C(s)$ = SiO(g) + CO(g)	$697.39-359.87 \times 10^{-3}$ T	1938
(2)	SiO(g) + 2C(s) = SiC(s) + CO(g)	$-83.34+8.78 \times 10^{-3} T$	949
(3)	$SiO(g) + 3CO(g)$ $= SiC(s) + 2CO_2(g)$	$-428.20 {+} 360.18 \times 10^{-3} T$	1189
(4)	$CO_2(g) + C(s)$ = 2CO(g)	$172.43-175.70 \times 10^{-3} T$	981
(5)	$3TiO_2(s) + C(s)$ = Ti ₃ O ₅ (s) + CO(g)	$229.40-171.48 \times 10^{-3} T$	1338
(6)	$Ti_{3}O_{5}(s) + 8C(s)$ = 3TiC(s) + 5CO(g)	$1354.15-885.09 \times 10^{-3} T$	1530

carbon source at 1550 and 1600 °C for 2 hrs, respectively. The SiC-TiC composite powders prepared at 1550 °C are composed of a small amount of coarse and flaky particles, a large number of fine spherical particles (~ 100-200 nm), and a certain amount of whiskers. The dispersion between the powder particles is relatively good. Additionally, the whiskers in the powder samples are relatively long and consist of fine whiskers 50-200 nm in diameter, and a certain overlap phenomenon is present among the whiskers. Compared with the powders synthesised at 1550 °C, the samples synthesised at 1600 °C show insignificant changes in their overall morphology, but the size of the large amounts of nearly spherical particles and a certain amount of whiskers increase. Fig. 7 shows the EDS pattern of the whiskers in Fig. 6(d). The whiskers exist in the synthesised products contain Si and C elements, indicating that the whiskers synthesised in the powder samples are SiC whiskers.

Reaction mechanism

The possible chemical reactions during the hightemperature reaction synthesis of SiC-TiC composite powders by carbothermal reduction method are shown in Table 3 [27-29]. Based on the thermodynamic data provided in Ref. [30], the relationship between the Gibbs free energy (ΔG^{θ}_{T}) and the temperature (T) of these reactions are calculated (see Table 3). As the starch easily decomposed to produce elemental carbon at low temperatures, the steps of synthesising SiC-TiC composite powders are Formulas (1) to (3) for forming SiC and Formulas (5) to (6) for producing TiC. SiC is produced by gas-solid reaction of SiO (g) and C (s) or gas-gas reaction of SiO (g) and CO (g). The step of the synthesis of SiC by carbothermal reduction is Formula (1) for forming SiO (g). ΔG_{T}^{θ} in Formula (1) starts to turn negative at 1665 °C (1938 K). However, in this experiment, the XRD patterns of the products synthesised

at 1500 °C in the presence of starch as the carbon source begin to show the diffraction peaks of SiC, indicating that SiC forms in the mixed raw materials at the reaction temperature, which is lower than the reaction temperature calculated by thermodynamics theory. The reason is that the thermodynamic theoretical analysis is based on the assumption that the gas phase pressure is standard pressure $(1.01 \times 10^5 \text{ Pa})$, and in this experimental conditions, a flowing argon gas atmosphere is used, furthermore, the gas partial pressure is maintained at a low level, so the formation of SiO (g) in the Formula (1) is facilitated. Additionally, elementary carbon particles, resulted from the decomposition of starch in the precursor gel powders, are small and have a high reactivity as the SiO_2 particles, facilitating Formula (1) to transpire at relatively low temperatures.

In this experiment, the SiO gas generated from Formula (1) and the C particle form SiC particles by Formula (2) when graphite and starch are selected as the carbon sources. The reaction is a gas-solid reaction with C particles as the nucleus; therefore, the shape and size of the reactant C particles determine the shape and size of the synthesised SiC particles. Thus, formula (2) is the main reason for the formation of SiC particles. The results are confirmed by the formation of SiC diversified structure particles in Figs. 5 and 6. Furthermore, the formed SiC hinders the solid-phase diffusion of C and the gas-phase diffusion of SiO, and SiC are formed by the reaction between SiO and CO according to Formula (3). Formula (3) is a gas-gas reaction between SiO and CO gases, and the reaction product SiC is independently nucleated. Therefore, Formula (3) may be the main reaction of SiC whisker growth [27, 31]. Two main growth mechanisms for the synthesis of SiC whiskers are present: vapor-liquid-solid mechanism and vapor-solid mechanism [32-34]. During the carbothermal reduction process without additives, the presence of eutectic balls and impurity phases at the tips of whiskers cannot be observed in the micrographs because of the absence of catalyst in the system, the formation of SiC whiskers follows vapor-solid mechanism. In this experiment, the SiO₂ in the feedstock reacts with elemental carbon decomposed from the starch to form SiO and CO, and a part of SiO and C undergoes gas-solid reaction to form SiC particles. The other part of SiO and CO reacts with gas-gas reaction to produce SiC nuclei. SiC nuclei seek to minimize its own surface energy and grow in a particular direction in a preferred manner, and ultimately lead to the formation of whiskers under appropriate condition [31].

TiC is mainly produced by Formulas (5) and (6). TiO₂ reacts with C in the reaction process to initially generate intermediate products Ti_3O_5 , and then Ti_3O_5 reacts with C to further generate TiC. Thermodynamic calculation shows that the initial reaction temperature of Formulas (5) and (6) is 1065 °C (1338 K) and 1257 °C (1530 K), respectively. A large amount of TiC should be generated in the reaction system according to Formula (6) when the reaction temperature is higher than 1257 °C. However, in this experiment, a large amount of TiC forms when the reaction temperature is higher than 1500 °C. The main reasons for this phenomenon are: TiO₂ in raw materials has a larger particle size resulting in low reactivity; TiO₂ and C in raw materials are solid-solid mechanical mixing and the mixing is insufficient, resulting in a reduction of the contact area between the reactant particles. The results of thermodynamic analysis and XRD show that TiC forms prior to SiC, and the formation of TiC inhibits the growth of SiC particles and changes its growth mode, which is consistent with the SEM results.

Conclusions

SiC-TiC composite powders are successfully synthesised by carbothermal reduction using graphite or starch as carbon source. The reaction temperatures greatly influence the synthesis process, phase composition, and microstructure of SiC-TiC composite powders. With graphite or starch as the carbon source, the optimum reaction temperature is 1600 and 1550 °C, respectively.

With starch as the carbon source, the starch in the raw material decomposes to produce carbon and H_2O gaseous matter, but also may decompose to produce other gaseous products, such as CO, CO₂, C_xH_y, etc. at higher reaction temperature, thereby, leading to mass losses of the samples. Additionally, during the carbothermal reduction of SiO₂ and carbon to form SiC powders, the resulting SiO gaseous material, along with CO gas, partially escapes from the system, resulting in more mass losses in the system.

The SiC-TiC composite powders prepared at 1600 °C comprised of a small amount of large-sized tabular grains, a large number of fine irregular rod-like and approximately spherical particles of 200-400 nm in size using graphite as carbon source, moreover there exists a serious agglomeration or cohesion phenomenon between the powder sample particles.

The SiC-TiC composite powders prepared at 1550 °C are composed of a small amount of flaky particles, a large number of fine spherical particles (~100-200 nm), and a certain amount of whiskers 50-200 nm in diameter using starch as carbon source. The dispersion between the powder particles is relatively good. The growth mechanism of SiC whiskers synthesised in powder samples followed the vapor-solid mechanism.

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