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Preparation and microwave absorption properties of Fe-doped SiC powder obtained by combustion synthesis

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Abstract

Fe-doped SiC powders were synthesized via combustion reaction of the Si and C system in a 0.1 MPa nitrogen atmosphere using iron as the dopant. The prepared powders have fine spherical particles and narrow particle size distribution. The electric permittivities of SiC samples were determined in the frequency range of 8.2–12.4 GHz. Results show that the permittivity of SiC increases with the increasing iron contents. The 5% Fe-doped SiC powder with 2 mm or 2.5 mm thickness exhibits the best microwave absorption over the frequencies ranging from 8.2 to 12.4 GHz.

Keywords: Silicon carbide; Combustion synthesis; Microwave absorption property

1. Introduction

Microwave absorbing materials are the materials to dissipate the electromagnetic energy of incident microwave into heat by magnetic or dielectric loss [1,2]. In recent years, they have been increasingly investigated due to their applications in civil and military fields [3,4]. But a large amount of microwave absorbing materials is not used at the higher temperature because of the low Curie temperature [5]. So the microwave absorbing absorbers with high structure strength, and chemical and temperature resistances in the high temperature environments have been focused in recent years.

Silicon carbide has many excellent properties, for example, high strength and hardness, good corrosion resistance, high thermal stability and high thermal conductivity, which has been considered to be one of the important microwave absorbing materials used in the higher temperature environments. However, the pure SiC material presents the poor absorbing ability in the gigahertz (GHz) band range. According to related studies, the dielectric property or microwave absorption property has been improved by the pure *n*-type or *p*-type doping. For example, Zhao et al. and Huan et al. have prepared the N-doped (*n*-type doping) SiC powder by laser synthesis and chemical vapor deposition, respectively, which presents a better dielectric property in the frequency range of 8.2–12.4 GHz [6,7]. Li et al., Luo et al. and Jin et al. have obtained the Al-doped (*p*-type doping) SiC powders using the combustion synthesis method, thermal diffusion and microwave method, respectively, which also showed a better dielectric property than the undoped SiC powder in the X-band range (8.2–12.4 GHz) [8–10]. Li et al. synthesized the B-doped SiC (*p*-type doping) nanopowder by the *sol–gel* and carbothermal reduction method and also gave the better dielectric property in the frequency range of 8.2–12.4 GHz [11]. Li et al. have prepared Ni-doped SiC powder by the mechanically activated self propagating high-temperature synthesis method and also presents the better dielectric property in the same frequency range [12]. Therefore, because the Fe element belongs to the VIII group, it presents the same effect on SiC powder as the Ni element possibly. However, little research has been focused on the effect of Fe-doping on the microstructure, dielectric property and microwave absorption property of SiC powder.

So in the paper, the Fe-doped SiC powders have been prepared by the CS method using silicon and carbon as raw materials, Fe as the dopant and PTFE as the chemical

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activator. The effects of Fe-doping on microstructure, dielectric property and microwave absorption property of SiC powder have been studied systematically. The sample with the best microwave absorption property has been presented.

2. Experimental procedure

Silicon powder (99% pure, mean particle size of 20 μm; Tianjin Kermel Chemical Reagents Development Centre, China) and carbon black (99% pure, particle size of 20–40 nm; Jiaozuo Chemical Co. Ltd., China) were used as reactant materials. Iron powder (Fe, 99% pure, mean particle size of 30μm; Shanpu Chemical Co. Ltd., Shanghai, China) and the PTFE powder (99% pure, mean particle size of 75μm; Sinopharm Chemical Reagent Co. Ltd., Shanghai, China) were used as doping source and chemical activator, respectively. The molar ratios of Fe, Si and C (n_{Fe}:n_{Si}:n_C) are 0:1:1, 0.05:0.95:1 and 0.1:0.9:1, respectively. Synchronously, the 15 wt% PTFE has been added to the powder. The powder batches were dry-mixed for 20 h using planetary milling with agate ball media, and then poured into a graphite crucible; the graphite crucibles were transferred into the cold chamber of CS reactor, which is shown in Fig. 1. When the temperature of the hot chamber of CS reactor reached the 1200 °C, the graphite crucibles were transferred into the hot chamber from the cold chamber quickly and kept for 15 min in 0.1 MPa N_2 atmosphere. Additionally, because the excess carbon and unreacted silicon in CS products will affect the accuracy of dielectric property, the products were calcined in air at 650 °C for 0.5 h to remove excess carbon, and then dipped into the hydrofluoric acid (HF) for 24 h to remove the unreacted silicon and then dried at 120 °C.

The products were identified by X-ray diffraction (XRD, X'Pert PRO MPD, Cu Kα) to detect the generated phases before the calcination, washing and drying of products. The 99.99% Si (a=5.43088 Å) was used as inner standard. The morphology of the CS powders was investigated by scanning electron microscopy (SEM, JSM-6360LV, JEOL, Tokyo, Japan), and the compositions were analyzed by energy-dispersive spectroscopy (EDS, NORAN System SIX Model 300, Thermo Electron Corporation) after the calcination, washing and drying of products. The dielectric property of prepared powders has been determined in the frequency range of 8.2–12.4 GHz by the waveguide technique [8].

According to the transmission line theory, the reflection loss curves (RL) can be calculated from the complex permittivity and permeability at a given frequency as well as the thickness of microwave absorbing materials by the method, which is shown in reference [5]. In this study, because silicon carbide is the dielectric loss material, the real part μ' and imaginary part μ'' of permeability are 1 and 0, respectively.

3. Results and discussion

Fig. 2 shows XRD patterns of the CS powders synthesized with different Fe contents. It can be seen that the β-SiC is generated in all products. The reason is that when the graphite crucibles were transferred into the hot chamber from the cold chamber quickly the reaction (1) of PTFE and Si powder, which released high heat and promoted combustion synthesis reaction (2) between Si and C, took place due to the preheating (the temperature of the hot chamber is 1200 °C) [13].

$$Si(s)+2(-CF_2-)(s)\rightarrow SiF_4(g)+2C(s)+Q \quad (1)$$

$$Si(s)+C(s)\rightarrow SiC(s) \quad (2)$$

In addition, the unreacted Si phase was also observed, the peak intensity of Si phase decreases and the peak intensity of β-SiC phase increases with increasing Fe content. It is possible that the Fe-doping improves the reaction of Si and carbon black and leads to the formation of SiC easily [14]. Additionally, the Fe–Si compound phase was also detected in samples (b) and (c). The Fe–Si

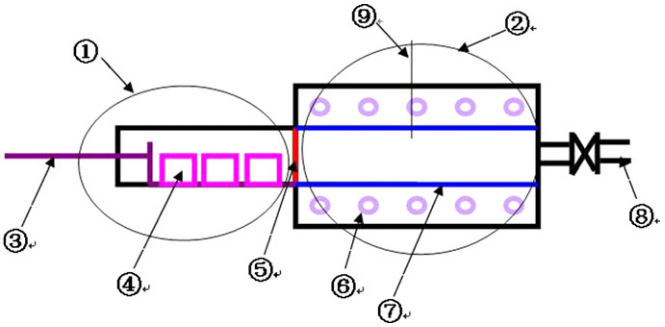

Fig. 1. Schematic diagram of CS reactor: 1—cold chamber; 2—hot chamber; 3—pushing rod; 4—graphite crucibles; 5—insulating plate; 6—heating resistance wire; 7—alumina tube; 8—pump; 9—thermocouple.

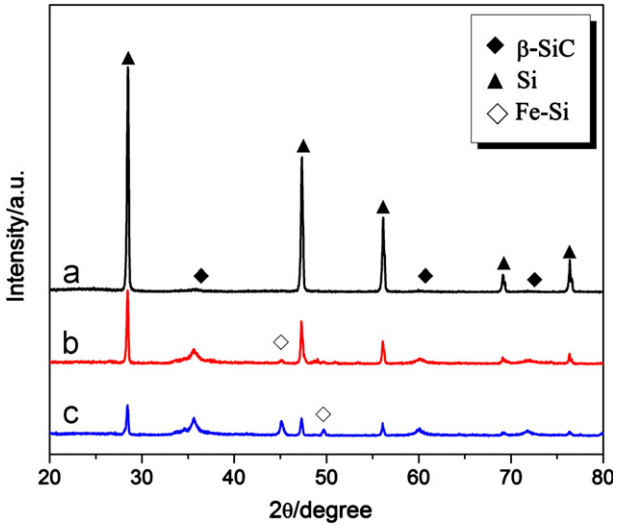

Fig. 2. XRD patterns of the CS powders synthesized with different Fe content: (a) n_{Fe}:n_{Si}:n_C=0:1:1; (b) n_{Fe}:n_{Si}:n_C=0.05:0.95:1 and (c) n_{Fe}:n_{Si}:n_C=0.1:0.9:1.