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Influence of synthesis process on the dielectric properties of B-doped SiC powders

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Abstract

Fine powders (~0.7 μm) of SiC doped with 3 mol% and 10 mol% B were successfully produced by mechanical activation assisted self-propagating high-temperature synthesis (MASHS). The experimental results showed that the presence of B caused a reduction in the combustion temperature, shrinkage of the crystal lattice, an increase in the tendency of the grains to be crystallized, and a decrease in the dielectric properties in the frequency range between 8.2 and 12.4 GHz, specifically the real (ε') and the imaginary parts (ε'') of complex permittivity and the loss tangent ($\tan\delta$). Analysis of the results suggests that B ions should be preferably accommodated in the Si sites of the SiC lattice and cause a reduction in the number of defects (V_{Si}, V_C, and C_{Si}), which results in a decrease in the dielectric properties. Comparison of the experimental results of this study with results reported in similar earlier studies reveals that the influence of B on the dielectric properties of the B-SiC powders depends strongly on the synthesis process.

Keywords: B. Defects; B. Microstructure-final; C. Dielectric properties; D. SiC; Combustion synthesis

1. Introduction

Silicon carbide (SiC) ceramics are attractive materials in many high-temperature structural, electrical, and functional applications because of their outstanding properties, such as excellent strength, chemical resistance to high temperatures, semi-conductivity, high thermal stability, and thermal conductivity [1–3]. The increase in electromagnetic pollution due to the vast use of electronic and telecommunication systems has recently stimulated intensive investigation into the dielectric properties of SiC materials in the microwave range [4,5]. Doping of SiC with N (p-type) or Al (n-type) results in doped SiC powders with better microwave dielectric loss in the X-band than that obtained for pure SiC [6–9].

Boron (B), which is a group III element in the periodic table of elements, is expected to easily enter the SiC lattice (p-type doping). Earlier papers have reported studies on SiC materials with B impurities and focused on the preferred B-acceptor substitution sites in SiC [10,11], the diffusion of B in 4H–SiC [12] and the effect of native defects on diffusion of B in SiC [13,14].

With regard to the dielectric properties of SiC powders, only a few papers have studied the case of powders doped with B [15–17]. Therefore, the mechanism explaining the effect of B on the dielectric properties of SiC is still unclear. In particular, Li et al. [15] produced a B-doped SiC solid-solution *via* sol–gel. The products had higher dielectric constants compared to pure SiC. These authors attributed this behavior to the formation of bound holes in the SiC *via* acceptor doping. Su et al. [16] and Li et al. [17] produced B-doped SiC solid-solution by combustion synthesis in Ar and N_2 atmospheres, respectively. Su et al. [16] found that the dielectric constants of a B-doped SiC solid-solution decreased with an increase in B content. These investigators suggested that doping with B causes an effective decrease in the number of intrinsic defects in C_{Si} and transformation of the orbitals of C from sp^2 to sp^3. However, Li et al. [17] found that both the real and imaginary parts of complex permittivity of SiC reach a minimum at 5 mol% B content (in particular, the values of the complex permittivity followed the order 0 mol% B (pure SiC) >10 mol% B > 5 mol% B). They suggested that C_{Si} defects in SiC do not contribute to changes in the real and imaginary parts of

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permittivity in the frequency range of 8.2–12.4 GHz. They correlated this behavior to the formation of better crystallized SiC, which resulted in a decrease in the number of Si (V_{Si}) and C (V_C) vacancies and B_{Si} defects which developed due to B doping.

The above paragraph shows that there is no globally documented behavior of the dielectric properties of B-doped SiC powders. However, SiC is a material that is in high demand for modern dielectric applications. To contribute to this debate with new experimental results, in this study, B-doped SiC powders were produced *via* an alternative technique (compared to that used in Refs. [15–17]). Production of ceramic powders by the MASHS method, which was performed in this study, combines self-propagating high-temperature synthesis (SHS) and mechanical activation (MA). This method has gained interest for producing many inorganic refractory powders [18–21] because it features low cost and high purity of the final products along with the possibility of producing novel phases with unique properties [22,23] (compared to conventional techniques for synthesis of SiC powders, such as sol–gel, thermal diffusion, *etc.*).

Obviously, this technique is applicable in cases where strong exothermic reactions occur, because the combustion reaction must be self-sustained by the energy produced. Nevertheless, the reaction between Si and C is intrinsically a weak exothermic reaction. Therefore, additional energy is needed to fulfill the energy requirements for a complete combustion synthesis reaction, such as preheating and/or addition of chemical activators. Nersisyan et al. [24] used poly(tetrafluoroethylene) (PTFE, $[-C_2F_4-]_n$) as an exothermic promoter in an Ar atmosphere to produce fine SiC powders. Addition of NH_4Cl was also found to effectively enhance the reactivity of the raw powders; furthermore, it improves the pulverizing efficiency of milling and prevents agglomeration in some mixtures during the milling process [25].

In the present study, B-doped SiC powders were produced *via* MASHS, using PTFE and NH_4Cl as chemical activators. The influence of B on the microstructure and the crystalline structure as well as the electric permittivity and dielectric loss of the powders produced in the frequency range of 8.2–12.4 GHz was experimentally determined.

2. Materials and experimental procedure

Fine powders of silicon (99.995% pure, mean particle size 10 μm, Hebei Sinocera Advanced Materials Co. Ltd., Langfang, PR China), carbon black (99.9% pure, mean particle size 2 μm, Shandong Haihua Carbon Black Chemical Co. Ltd., Weifang, PR China), boron (99.9% pure, mean particle size 20 μm, General Research Institute for Nonferrous Metals, Beijing, PR China), PTFE (99.5% pure, mean particle size 0.5 μm, Sinopharm Chemical Reagent Beijing Co. Ltd., PR China), and NH_4Cl (99.5% pure, mean particle size 0.5 μm, Sinopharm Chemical Reagent Beijing Co., Ltd., PR China) were used.

Three different compositions were produced with B:Si:C molar ratios of 0:1:1, 0.03:0.97:1, and 0.10:0.90:1, which are simply denoted hereafter as 0% B, 3% B, and 10% B, respectively. The Si, C, and B powders were precisely weighed to satisfy the above molar ratios. Then, 1 wt% NH_4Cl and 6 wt% PTFE (with respect to the 100 wt% Si–C–B batch) were added to the powder batches. The powder mixture was dry milled by ball-milling for 3 h in a SiC jar using SiC balls as the milling media (the ball/powder weight ratio was 10/1). To protect the powders from oxidation, milling took place in an inert Ar atmosphere. The milled powders were sieved through a 60 mesh sieve and the finer fraction of the powder (<60 mesh) was collected.

Batches of *ca.* 100 g each were loosely loaded into a prismatic graphite crucible 200 mm in length, 50 mm in width, and 30 mm in height. The crucible was placed into a reaction chamber. A tungsten coil was placed on top of the powder for the purpose of triggering the combustion reaction by passing an electric current through it. To record the temperature, a W3/Re25 thermocouple protected by a BN tube with a diameter of 2.5 mm and a thickness of 0.3 mm was inserted into the center of the sample. The chamber was hermetically sealed and evacuated (primary vacuum, $\sim 10^{-2}$ mbar) with the aid of a rotary pump. Then, the chamber was filled with Ar. Three cycles of evacuation and refilling with Ar were repeated to reduce the oxygen partial pressure. Then, the chamber was filled with high purity Ar gas (99.999%) up to 0.5 MPa. After the reaction was complete, the chamber was allowed to cool, the exhaust gases were discharged, and the products were collected for characterization.

The crystalline phases in the produced powders were identified by X-ray diffraction analysis (XRD, Cu K_α, X'Pert PRO, PANalytical B.V., Almelo, The Netherlands). The microstructure of the powders was observed by scanning electron microscopy (SEM, JSM-6360LV, JEOL, Tokyo, Japan). The measurements of complex permittivity were carried out using an Anritsu 37269D (Kangawa, Japan) vector network analyzer. For the measurements of the dielectric parameters, the as-synthesized powders, after firing at 650 °C in air for 1 h to remove any possible excess of carbon, were dispersed in paraffin wax (the weight ratio of powder to paraffin wax was 3:7) by regular stirring for 1 h at 60 °C. Samples in the form of rings, with dimensions of 7.0 mm outer diameter, 3.0 mm inner diameter, and 2.0 mm thickness, were solidified at room temperature in air.

3. Results

The change in temperature during the combustion reaction followed a typical temperature profile. In particular, just before the temperature peak (due to the combustion reaction), the rate of increase in temperature was steep, being *ca.* 300°/s. After the temperature peak, the rate of decrease in temperature was also steep but soon (*ca.* 10–15 s) declined, and eventually a cooling rate of *ca.* 5°/s was reached. With regard to the influence of B content on the recorded combustion temperature, the temperature peaks were 1616 °C for the B-free powder (0% B), 1585 °C for the 3% B powder, and 1468 °C for the 10% B powder. Thus, it can be

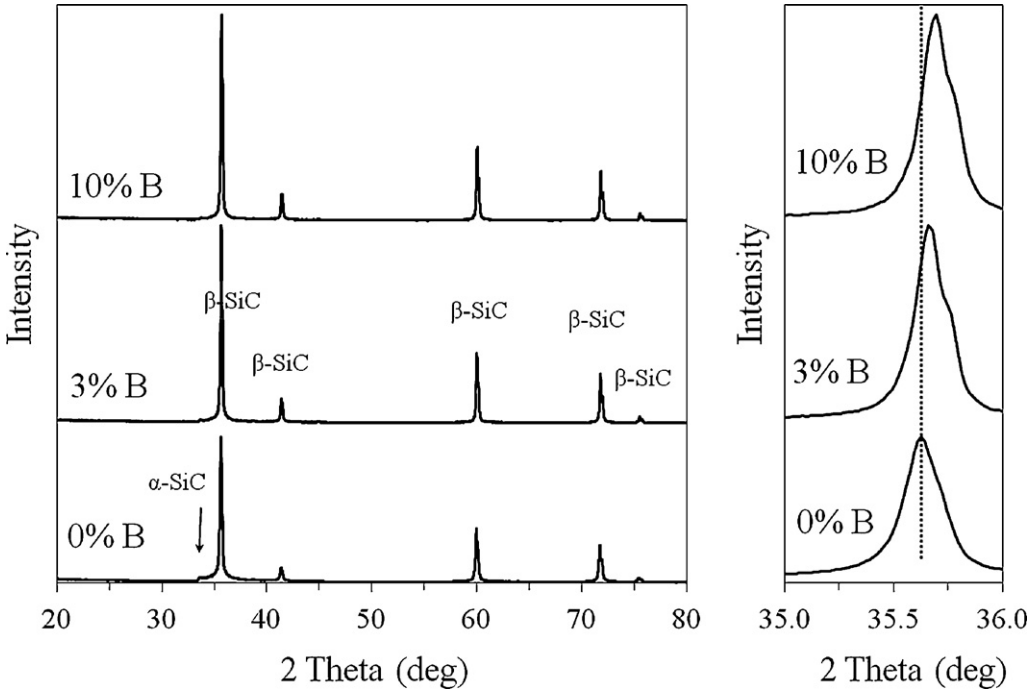

Fig. 1. X-ray diffractograms of the produced powders. The shift of the peak of the plane (1 1 1) of β-SiC is clearly shown in the plot at the right-hand side (the diffractograms have not been normalized; the peaks were identified using the ICDD cards 75-0254 for β-SiC, and 29-1126 for α-SiC).

stated that the increase in the presence of B caused a reduction in the combustion temperature.

The X-ray diffractograms of the powders produced are shown in Fig. 1 and suggest that well crystallized β-SiC powders were produced in all cases. There was no perceptible evidence of residual silicon or carbon. A weak peak of α-SiC was registered in the B-free powder. Formation of α-SiC has also been reported in an earlier study [26]. It has been postulated that this α-SiC derives from the transformation of β-SiC due to the presence of stacking faults in the crystal [27]. In this study (Fig. 1), the presence of B apparently suppressed the formation of α-SiC.

The X-ray diffractograms also reveal two important effects of B on the crystallographic regime of the SiC powders produced. The intensity of the peaks (which reflects the crystallinity of the powders) apparently increased from the B-free SiC to the B-doped powders. However, the most pronounced effect was that the peaks of the diffractograms shifted towards higher angles with increasing B content. The shift of the strongest peak, corresponding to the (1 1 1) plane of SiC, is clearly shown in the right hand plot of Fig. 1. Using the Bragg equation, the shrinkage of the lattice with increasing B content is shown in the values of the lattice constant a and the inter-planar distance d listed in Table 1.

Table 1
Lattice constant a and inter-planar distance d between the planes (1 1 1) of SiC, calculated from the diffractograms of Fig. 1 for the produced B-doped SiC powders.

Powder	a (Å)	d (Å)
0% B	4.3648	2.5201
3% B	4.3569	2.5155
10% B	4.3536	2.5136

Fig. 2 shows typical microstructures of the powders produced (Fig. 2a–c correspond to 0% B, 3% B, and 10% B, respectively). In general, B content did not perceptibly influence the grain size; a narrow particle size distribution of grains, which were sized at *ca.* 0.7 μm, was observed. However, there is evidence that B influenced the morphology of the powders. In particular, equiaxed round-shaped grains were observed in the B-free SiC powder with no evidence of preferential crystalline orientation (Fig. 2a). In 3% B (Fig. 2b) and 10% B (Fig. 2c) powders, the surfaces of the slightly elongated grains formed faceted shapes. This feature suggests better crystallization, which agrees fairly well with the aforementioned increase in the intensity of the peaks of the X-ray diffractograms in Fig. 1.

The experimental results of the dependence of the real (ε') and imaginary (ε'') parts of complex permittivity and the loss tangent (tan δ) on frequency are plotted in the diagrams in Fig. 3a–c, respectively. It can be clearly seen that the increase in B content in SiC powders lowers the values of ε', ε'', and tan δ within the investigated frequency range (*i.e.* 8.2–12.4 GHz).

4. Discussion

The experimental results suggest that B should be well accommodated in the β-SiC lattice (Fig. 1) and should affect the dielectric properties (Fig. 3) of the produced powders considerably. With the aid of the experimental results of this study and the results reported in earlier studies, this section aims to discuss the influence of the production method on the properties of the B-doped SiC powders.

4.1. Combustion reaction

The production method of MASHS successfully overcame the weak exothermic nature of the reaction between Si and C. The combustion reaction took place completely and β-SiC was formed rapidly (according to the temperature profile described in the results) and predominantly (Fig. 1).

Besides mechanical activation, the presence of PTFE has a seemingly multifold effect in providing energy to the system. In particular, at high temperatures, PTFE pyrolyzes exothermally to a variety of fluorocarbon compounds. These compounds are further pyrolyzed to F_2 and the produced F_2 reacts with the superficial passive oxide film that inevitably forms on the surface of Si particles to form volatile products. Obviously, the cleaning of the oxide film from the Si surface facilitates the occurrence of the reaction between the Si and C powders.

Meanwhile, the direct reaction of Si with PTFE to form SiF_4 is also highly exothermic [28–30]. On the other hand, the direct reaction of PTFE with B is expected to be less favored than the reaction of PTFE with Si since B is more electronegative (2.04) than Si (1.90). This can explain the strong effect of B content on the reduction of the combustion temperature. A similar behavior has been reported by Stobierski et al. [31].

4.2. Influence of the production method on the dielectric properties of B-doped SiC

The observed shrinkage of the SiC lattice with increases in the amount of B (Fig. 1 and Table 1) would plausibly suggest that B is preferably accommodated in the Si sites because the atomic radius of B (0.95 Å) is smaller than that of Si (1.34 Å). In the crystal lattice of SiC, every C atom is connected to four neighboring Si atoms and every Si atom is connected to four C atoms. Therefore, every B atom which is accommodated in a Si site should be connected with four neighboring C atoms. In this case, the difference in the valence between B^{3+} and Si^{4+} creates a hole, which is called a ‘‘bound hole’’. These bound holes can accept electrons and favor thermal motion around the B atoms.

Dielectric loss can occur due to either ion migration loss, including DC conductivity loss, or ion jump and dipole relaxation losses, or ion vibration and deformation losses, or electron polarization loss [32]. SiC is a non-magnetic loss material. Thus, the power dissipation at high frequencies is determined only by the dielectric loss.

In principle, a bigger number of defects results in a more pronounced dielectric loss and *vice versa*. In pure β-SiC, the defects, which intrinsically exist in the lattice, are vacancies of Si (V_{Si}) and C (V_C) and their anti-sites (Si_C, C_{Si}). These types of defects are energetically preferred since they are inherently bound to tetrahedral symmetry [10,33,34].

The experimental results of the influence of B content on the dielectric properties (Fig. 3) must be discussed in the light of the results of earlier studies, which were outlined in the introduction. Accordingly, it is clear that there are different (and opposing) experimental results with regard to the influence of B doping on the dielectric properties of SiC. In particular, the work of Li et al. [15], who produced a B-doped SiC solid-solution by the sol–gel method, suggests that bound holes exist in the material produced. These bound holes migrate to and fro under an alternating electromagnetic field, resulting in relaxation polarization and loss, which lead to higher values of ε' and ε'' with increasing B content. The work of Su et al. [16], who found that the dielectric constant decreases with an increase in B content, suggests that the presence of B causes a reduction in the number of C_{Si} defects. This decreases the number of dipole pairs and thus lowers the dielectric constant. The results of Li et al. [17] showed that the minimum real and imaginary parts of complex permittivity of SiC occur at 5 mol% B. These researchers postulated that the defects, which cause

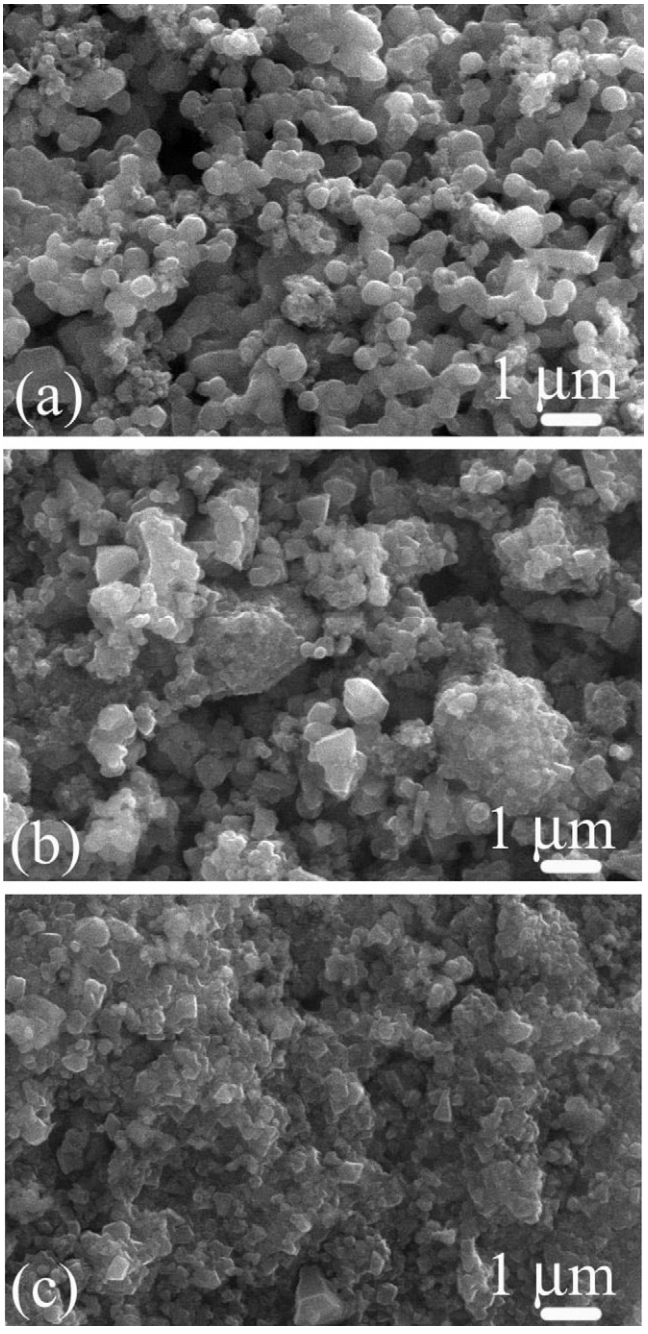

Fig. 2. Microstructure of the produced powders: (a) 0% B, (b) 3% B, and (c) 10% B.

the dielectric constant, such as V_{Si} and V_C, decrease with increasing B content. The experimental results of the present study (Fig. 3) apparently agree with the work of Su et al. [16] and Li et al. [17], since the experimental results have clearly shown that the presence of B caused a decrease in dielectric loss under an alternating electromagnetic field.

Accordingly, this study proposes that the aforementioned differences may be due to the preparation method. In the present discussion, the results of three different methods are considered and the following lines briefly discuss them. Li et al. [15] apparently followed the most benign (in terms of severity)

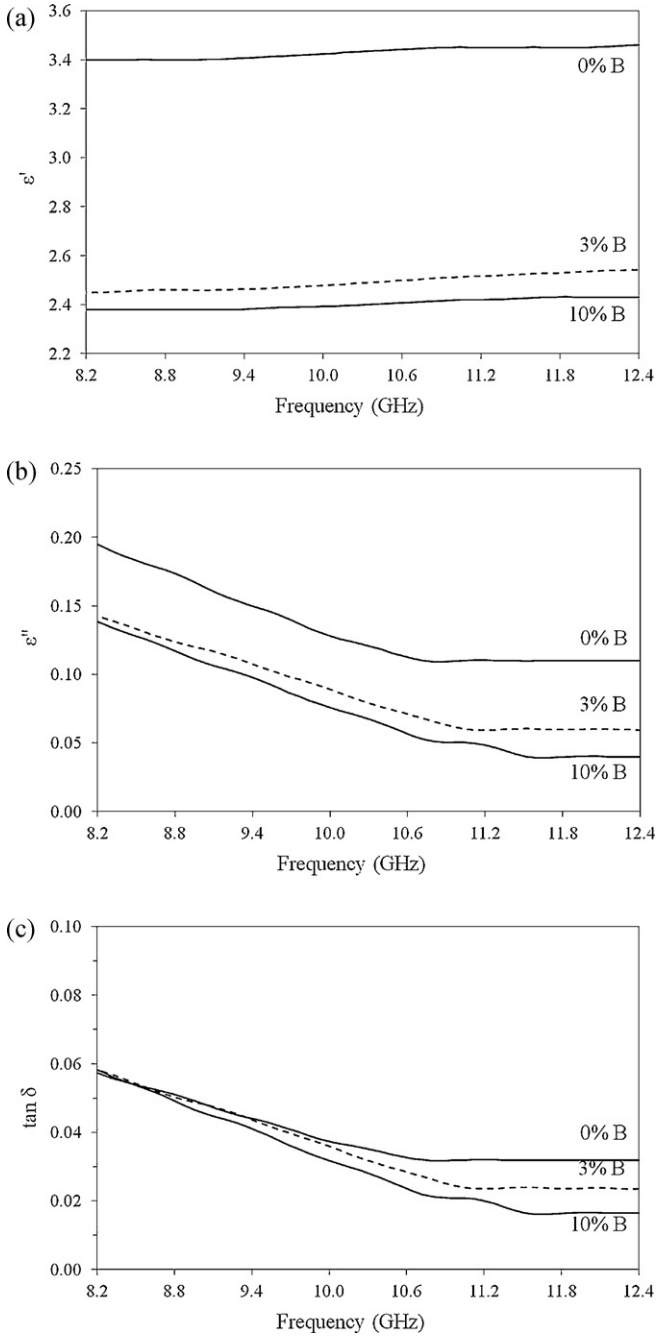

Fig. 3. Dielectric properties of the produced powders in the frequency band between 8.2 and 12.4 GHz: (a) real part (ε') and (b) imaginary part (ε'') of the complex permittivity, and (c) loss tangent ($\tan\delta$).

method among the selected references, that is, the sol–gel method. Su et al. [16] and Li et al. [17] employed an intermediate method (in terms of the total reaction time): the powders of Si, C, and B were preheated at ~1250 °C or 1350 °C. In the study of Su et al. [16], the X-ray diffractograms showed peaks of C for 0%, 5%, and 10% B. Therefore, those authors completely burnt the remaining C in the reaction products by firing at 650 °C for 1 h in air. In the present study, the SHS process was obviously not benign at all. There was almost no dwell at the peak temperature and the heating and cooling rates (immediately after the peak of the combustion temperature) were very fast; all these facts suggest that the reaction took place within a few seconds. However, the X-ray diffractograms (Fig. 1) showed that there was no evidence of either C or α-SiC in the B-doped SiC powders. Unfortunately, the papers of Su et al. [16] and Li et al. [17] do not provide information on the temperature profiles. Therefore, with the aid of the available data, it seems that the works of Su et al. [16] and Li et al. [17] resemble the present study. However, the use of the MASHS method in the present study resulted in better (*i.e.* more pure) final products (Fig. 1) (commending the methods used to produced B-doped SiC, it is also worthy of note that MASHS is superior because the instant ignition of SHS considerably reduces the production cost since it omits the expensive stage of preheating the furnace).

However, one should keep in mind that the MASHS method inevitably implies a pronounced discrepancy from thermodynamic equilibrium conditions. The influence of mass transportation phenomena under the extreme conditions of the MASHS method on the microstructure should be reflected in the SEM images of Fig. 2. These micrographs showed that the B-doped SiC grains apparently tended to develop shapes that indicated higher crystallinity. Earlier studies on the sintering behavior of SiC have reported that the presence of B in SiC matrix lowers the activation energy of diffusion and increases the mass transportation by several orders of magnitude [35–38].

Accordingly, the presence of B seems to favor the crystallization of SiC by enhancing the mass transport. Thus, the intrinsic defects of SiC (such as V_{Si}, V_C, and C_{Si}) should decrease, resulting in a reduction in the number of dipoles, as already suggested in earlier studies [16,17]. Considering the grid (*i.e.* because the covalent bond between Si and C atoms is strong) crystal lattice of SiC and the low solubility of B in SiC [39,40], the newly formed bound holes due to the addition of B are limited and do not compensate for the loss of defects.

Certainly, the aforementioned statement is seemingly not universal (*i.e.* for all the cases of B-doped SiC powders) because the discussion in this section has shown that the production method crucially influences the dielectric properties of the B-doped SiC powders.

Besides B, C may also contribute to the dielectric performance of the powders produced. It has been postulated [16] that the decrease in electrical conductivity of doped SiC is due to the transformation of the sp^2 orbital of C to sp^3 because the electrical conductivity of the p-state (resulting from sp^3) is lower than that of the s-state (resulting from sp^2) [41].

At this point, it is worthy to refer to a recent article [42] where boron was selected as a dopant of wide bandgap nanocrystalline silicon-carbide (nc-SiC:H) film in order to achieve a high conductivity. It was found that a relevant light doping is essential to improve the electrical conductivity without deteriorating significantly the crystallinity and optical bandgap.

5. Conclusions

The mechanically activated self-propagating high-temperature synthesis (MASHS) method successfully overcame the weak exothermic reaction between Si and C. Thus, B-doped SiC powders with high purity, high crystallinity, and fine microstructure ($\sim$0.7 μm) were successfully produced.

The presence of B considerably influenced the combustion temperature (*i.e.* it reduced it) and the features of the produced powders. In particular, the crystallinity of the powders increased with increasing B content. Analysis of the experimental results suggests that B should be preferably accommodated in the Si sites of the SiC lattice. The dielectric properties decreased, which was mainly attributed to the decrease in the number of defects due to the presence of B in SiC.

A comparison of the results of this study with the dielectric properties for similar powders reported in earlier studies suggests that the production method crucially determines the dielectric properties of the B-doped SiC powders.

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