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Ceramics International 40 (2014) 4359–4366

CERAMICS INTERNATIONAL

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Densification behavior and related phenomena of spark plasma sintered boron carbide

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Received 18 June 2013; received in revised form 18 August 2013; accepted 26 August 2013
Available online 3 September 2013

Abstract

In this work, boron carbide ceramics were sintered in the temperature range of 1400–1600 °C by spark plasma sintering (SPS). The influence of sintering temperature, heating rate, and holding time on the microstructure, densification process and physical property was studied. The heating rate was found to have greater influence than that of the holding time on the microstructure and the densification of boron carbide. The optimal sintering temperature was 1600 °C under the heating rate higher than 100 °C/min. The relative density, flexural strength, Vickers hardness and fracture toughness of the sample synthesized at 1600 °C were 98.33%, 828 MPa, 31 GPa and 2.66 ± 0.29 MPa $m^{1/2}$, respectively. The densification mechanism was also investigated.

Keywords: Boron carbide; Spark plasma sintering; Sintering behavior; XRD; TEM

1. Introduction

Boron carbide was a covalently bonded compound with high melting point (2427 °C), a relative low density (2.52 g/cm^3), extremely high hardness (only after c-BN and diamond) and high neutron absorption cross-section [1]. It had been used for light weight armor, wear-resistant components and control rods in nuclear reactors due to the attractive properties [1,2]. Pressureless sintering method to get high density pure boron carbide had proved to be difficult. Pressure-assistant sintering methods (hot pressing (HP) or hot isostatic pressing (HIP)) had usually been employed to produce almost fully densified boron carbide ceramics [1,2]. The pressure was recognized to promote the densification by facilitating particle rearrangement, plastic deformation and pore elimination [3].

Spark plasma sintering (SPS) was a special sintering process by combination of uniaxial pressure and powerful DC pulsed current to consolidate materials [4–6]. SPS can be used to produce specimens with outstanding properties and limited grain growth at lower temperature within shorter heating time [4–7]. Abnormal grain growth usually occurred in pressureless sintered boron carbide, which resulted in the performance degradation. In addition, boron carbide was electro-conductive material, the applied pulsed current could possibly contribute to the densification process and result in the material with attractive properties [1]. Thus, SPS was a promising approach for the preparation of boron carbide with high performance.

SPS had been used to consolidate boron carbide ceramics [8–22]. In SPS sintering, pore free ceramics were usually fabricated by adding sintering aids (e.g. SiC, TiB_2) or through reaction sintering method [9,11–15,21,22]. Dense boron carbide ceramics prepared directly using only boron carbide powder were rarely reported [10,16–20]. Hayun et al. [16] fabricated dense pure boron carbide ceramics using boron carbide powder and studied the effect of sintering temperature, holding time, applied pressure and heating rate on the sintering

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http://dx.doi.org/10.1016/j.ceramint.2013.08.106

behavior of boron carbide ceramics in the temperature range of 1800–2200 °C. It was found that the relative density increased with the increase of sintering temperature, holding time and applied pressure. Boron carbide with finely equiaxed grains and homogeneous microstructure can only be obtained at high sintering temperature of 2200 °C with very high heating rate (600 °C/min). It was also found that the impurities possibly improved the densification at the early stage of sintering by forming liquid phase. Moshtaghioun et al. [10] explored other factors influencing the sintering behavior of boron carbide. Results showed that the powder size is an important factor that influences the sintering temperature. The densification mechanism was not referred in the literature.

In the present work, boron carbide ceramics with high performance were fabricated without employing sintering additives. The effect of sintering temperature, heating rate and holding time at different sintering temperatures on the densification and microstructure was studied in the temperature range of 1400–1600 °C. Moreover, the densification mechanism of SPS sintered boron carbide was also deeply investigated.

2. Experimental procedure

Commercially available boron carbide powder (Mudanjiang Jingangzuan boron carbide Co. Ltd., Mudanjiang, China) with the mean particle size of 493.8 nm was used as the starting powder. To reduce the impurity content, the as-received powder was firstly dispersed in dilute HCl solutions and then washed with deionized water. After drying at 100 °C for 2 h, the powder was sieved using 200 mesh grids. The as-treated powder was then filled into a cylindrical graphite die with the inside/outside diameter and height of 20 mm/50 mm and 40 mm, respectively. Two graphite discs of 20 mm diameter were placed on the top and bottom of the sample. Graphite paper was also rolled and placed on the inner surface of die before the boron carbide powder was filled in. A SPS system (SPS 2040, Sumitomo Heavy Industries Ltd., Japan) was used to consolidate the boron carbide powder. The pulse cycle of the DC current was 12:2 (i.e., twelve 3 ms pulses on and two 3 ms pulses off). The DC current flowed from top punch down toward bottom punch, while the pressure was applied along the opposite direction. After sintering, the disc shaped sample was cut into bar-shaped sample of 2 mm × 4 mm × 18 mm in size.

The three-point flexural strength measurement was performed using a universal testing machine (Mold 5566, Instron, America) with a span of 12 mm. Vickers hardness was determined using a hardness tester (Wilson–Wolpert Tukon 2100B, America) with 1 kg load on polished surface. The fracture toughness was also measured in the hardness testing machine by the indentation method. The density of sample was measured using Archimedes' method. The phase composition of the sample was determined by X-ray diffraction (XRD, D/max 2550V, Rigaku, Japan). The microstructure of sample was observed using a scanning electron microscopy (SEM, TM-1000, Hitachi, Japan) and a transmission electron microscopy (TEM, JEM-2100F/200F, Joel, Japan) with an energy dispersive X-ray spectrometer attachment (INCA, Oxford instruments, Britain). To determine the grain size of boron carbide ceramics, the specimens were polished and etched in dilute KOH solutions under DC current of 0.02 A for 30–60 s using a DC current equipment (LW15J2, Shanghai Liyou, China). The mean grain size was determined from SEM micrograph, and at least 300 grains were measured to get the statistical mean size.

3. Results and discussion

3.1. Densification

The relative density of boron carbide ceramics sintered at different temperatures under the heating rate of 100 °C/min and the holding time of 3 min was shown in Fig. 1. The density noticeably increased with the increase of the temperature up to 1500 °C, and then kept almost constant thereafter in the temperature range of 1500–1600 °C. The densities of boron carbide ceramics were 65.39% and 75.17% after sintering at 1400 °C and 1450 °C (Fig. 1), respectively. High relative density over 95% was achieved when the temperature was above 1500 °C. The relative density of 98.32% can be reached at 1600 °C (Fig. 1). A increase in density from 92–93% to 99% had ever been observed when the SPS sintering of pure boron carbide was carried out from 1650 °C to 1750 °C [10]. However, the reason for density increase was not discussed in the literature. The abrupt increasing of relative density from about 60% at 1600 °C to 95% at 1675 °C was observed in SPS-reaction-sintered SiC [23,24], which was related to the disorder-to-order transformation and accompanied by mass transport in this temperature range. In our case, a different mechanism will be reasonably proposed for the increase in density, which would be discussed in following section.

The microstructure of the samples under different sintering temperature was shown in Fig. 2. No any obvious grain growth was observed at 1400 °C as shown in Fig. 2(a). At 1450 °C (Fig. 2(b)), sintering necks appeared while the compact was still in interconnected porous structure. Though the most part

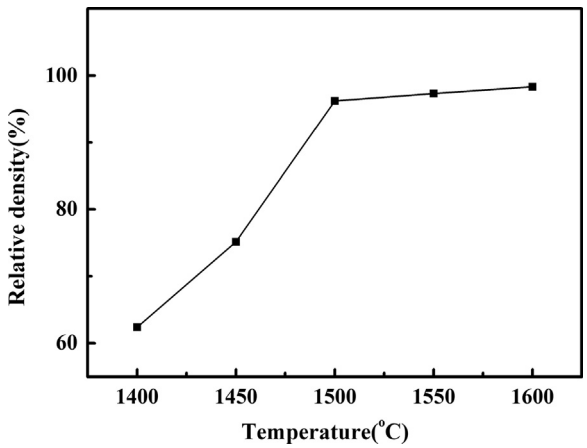

Fig. 1. Relative densities of boron carbide ceramics sintered at 1400 °C, 1450 °C, 1500 °C, 1550 °C and 1600 °C for 3 min under the heating rate of 100 °C/min.

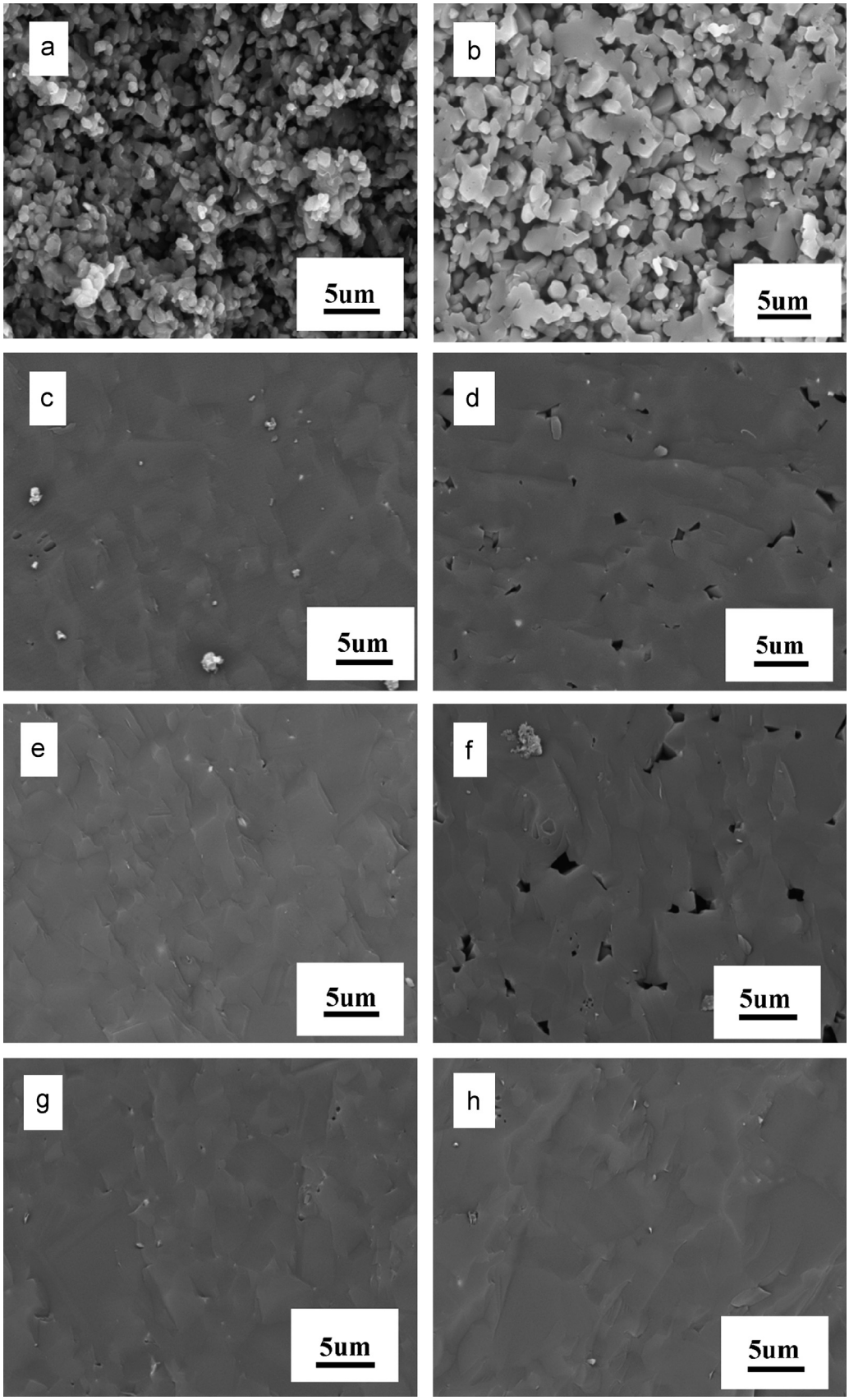

Fig. 2. SEM images for the fracture surface of the samples sintered at 1400 °C (a) and 1450 °C (b), the most part of the samples sintered at 1500 °C (c), 1550 °C (e) and 1600 °C (g) and the region at the upper central surface of the the samples sintered at 1500 °C (d), 1550 °C (f) and 1600 °C (h) under the heating rate of 100 °C/min and the holding time of 3 min.

of boron carbide was in dense state at 1500 °C and 1550 °C (Fig. 2(c) and (e)), the disc-shaped samples exhibited a small porous region at the upper central surface (Fig. 2(d) and (f)). It revealed that the temperatures of 1500 °C and 1550 °C were not high enough to remove all the pores in the samples. Nevertheless, a nearly dense sample from top to bottom was observed after sintering at 1600 °C (Fig. 2(g) and (h)), which suggested that the higher temperature of 1600 °C could further reduce the porosity of boron carbide. Thus, the suitable sintering temperature was 1600 °C.

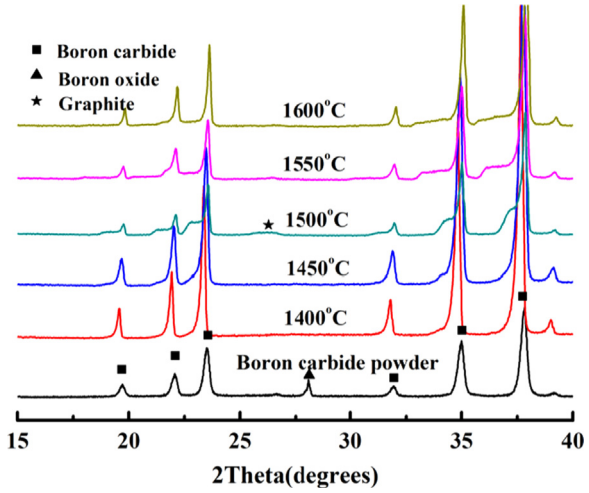

Fig. 3. XRD patterns of original boron carbide powder and boron carbide ceramics sintered at 1400 °C, 1450 °C, 1500 °C, 1550 °C and 1600 °C.

In the present work, it was also found that the heating rate have significant effect on the densification of boron carbide, compared with the soaking time. At the sintering temperature of 1500 °C, the relative density (94.87%) of boron carbide was higher at lower heating rate of 100 °C/min even with shorter holding time (3 min) compared with that (84.15%) at higher heating rate of 150 °C/min and longer holding time (10 min). Similar behavior was also found at 1550 °C, the density of 97.32% can be reached with the slow heating rate of 103 °C/min and the short soaking time of 3 min, which was higher than that of 96.36% with the heating rate of 130 °C/min and soaking times of 5 min. Since 1500 °C and 1550 °C were relatively low sintering temperature, the slow heating rate may be beneficial for the removal of surface oxides, the mass transport and reduction of porosity in boron carbide. At the higher

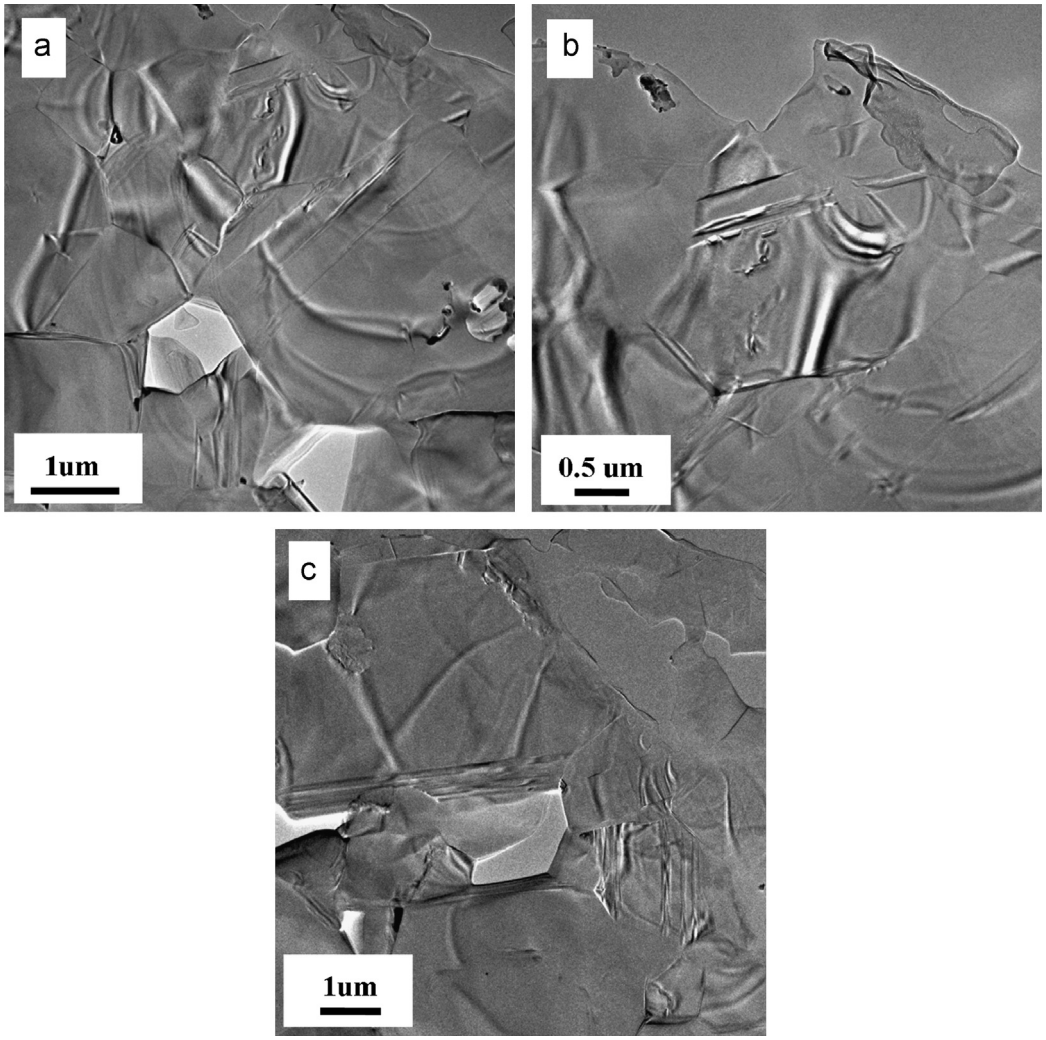

Fig. 4. TEM micrographs of boron carbide samples sintered at 1500 °C (a), 1550 °C (b) and 1600 °C (c).

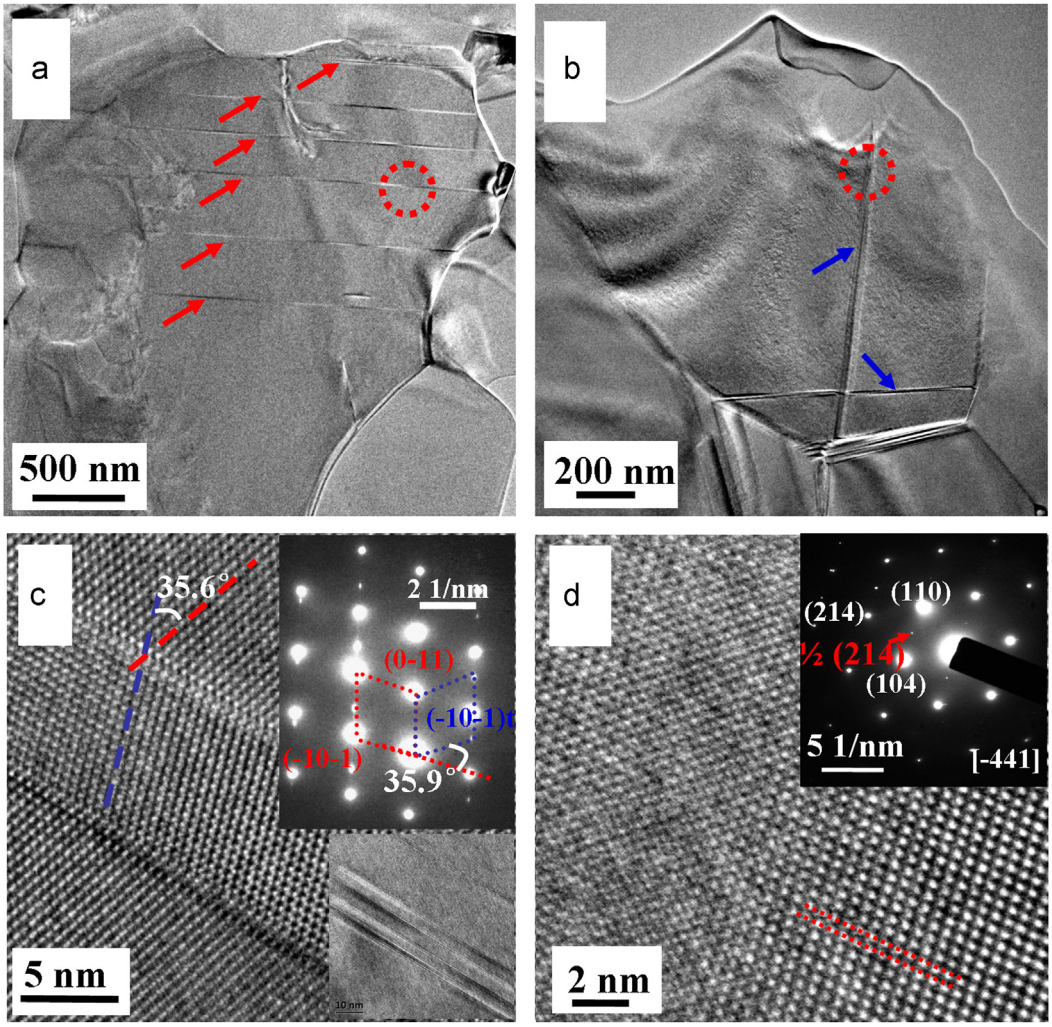

Fig. 5. TEM micrographs of the boron carbide sintered at 1550 °C (a) and (b), high resolution TEM (HRTEM) images and selected area electron diffraction (SAED) patterns of defects (c) and (d), which were corresponded to the circle regions in (a) and (b), respectively.

temperature of 1600 °C, the heating rate below 85 °C/min resulted in the presence of abnormal grains at bottom region of the samples, whereas no abnormal grains appeared at higher heating rate of 106 °C/min or 114 °C/min. The slow heating rate at higher temperature might lead to the grain coarsening, which was in good agreement with the literature [16]. This may suggest that the sintering temperature of 1600 °C was enough for the removal of surface oxides and effective mass transport. Thus, the proper sintering condition may be 1600 °C at the heating rate of 106 °C/min or 114 °C/min for attaining homogeneous and dense boron carbide ceramics.

3.2. Phase and microstructure analysis

After sintering, humps adjacent to the XRD main peaks at lower angle side were clearly observed in the XRD patterns of the samples sintered at 1500 °C, 1550 °C and 1600 °C (Fig. 3). The asymmetric XRD peaks were also detected for the samples sintered at 1400 °C and 1450 °C (Fig. 3). The humps and asymmetric peaks were not present in the starting boron carbide powder, which indicated that they were generated in the SPS sintering process. The intensity of the humps was highest at 1500 °C and decreased with the increasing of sintering temperature. A small bump in the XRD pattern of 1500 °C was observed at about $2\theta = 26.5°$, which was assigned to the graphite. The graphite was not detected in the XRD patterns of 1400 °C, 1450 °C, 1550 °C and 1600 °C. However, no any kind of carbon was added before sintering. Carbon diffusion from the graphite mold toward sample had been observed in HP sintering [1]. The samples were surrounded by graphite foil in the present work. The graphite probably originated from the graphite foil. Carbon dissolving into boron carbide can take place based on the B–C phase diagram [1]. The graphite was not observed at 1550 °C and 1600 °C, which indicated that the graphite may have been incorporated into the boron carbide structure at high temperature.

In literatures, the humps adjacent to diffraction peaks had been observed in SPS-sintered SiC and boron carbide using elemental Si or B with C as starting materials [21–24], in which the humps simply accompanied with specified peak (e.g. the peak corresponding to (111) lattice plane in SiC) at lower angle. In our case, almost each measured peak at 1500 °C, 1550 °C and 1600 °C was accompanied by a hump

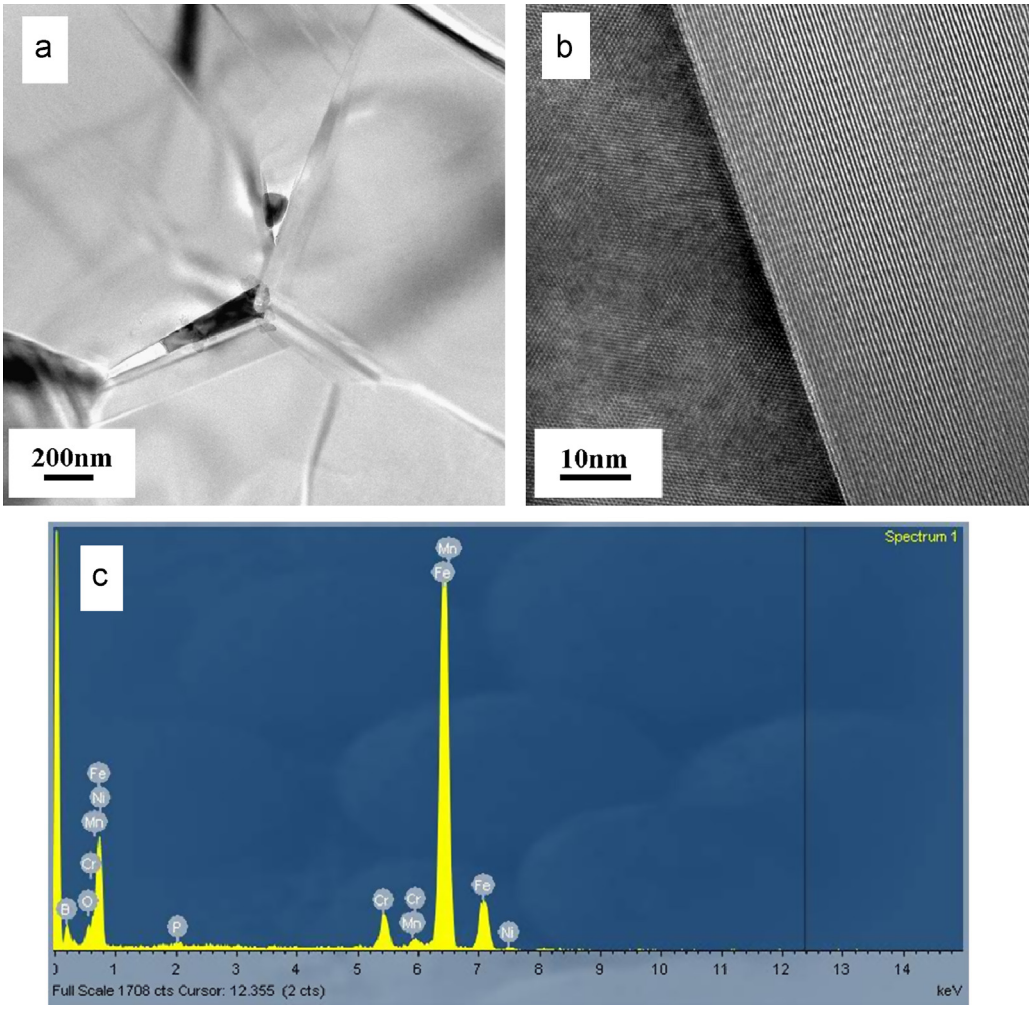

Fig. 6. Microstructure of boron carbide sintered at 1550 °C at the triple junction (a) and the interface of two boron carbide grains (b), and the EDS analysis of the triple-junction phase (c).

at lower angle region, and the measured peaks at 1400 °C and 1450 °C showed asymmetry as shown in Fig. 3. The asymmetric peaks suggested that the structure of boron carbide sintered at 1400 °C and 1450 °C evolved toward those of 1500 °C, 1550 °C and 1600 °C. In the SPS-reaction-sintered SiC [22,23], hump was reported as the indication of the defects such as stacking faults or twins in the sample. In the present work, the defects were easily observed in the samples of 1500 °C, 1550 °C and 1600 °C. The planar defects were clearly displayed in the TEM images, Fig. 4, which showed typical morphologies of the 1500 °C, 1550 °C and 1600 °C samples. HRTEM was utilized to characterize the microstructure of the samples. Fig. 5(a) and (b) showed the typical microstructure of boron carbide samples sintered at 1550 °C. The planar defects which the arrows in Fig. 5(a) pointed out were assigned to the twins. Fig. 5(c) showed the enlarged area of the circle shown in Fig. 5(a), from which the twins were clearly observed. The selected area electron diffraction (SAED) in Fig. 5(c) displayed the typical diffraction pattern of twins, in which the angles mismatch of the same lattice plane was about 35.9°. The angles mismatch was in good agreement with the result of the HRTEM, in which the measured angle was about 35.6° and the twinning plane was (0–11). The defects which the arrows in Fig. 5(b) pointed out were stacking faults. The circle area in Fig. 5(b) was corresponded to Fig. 5(d), in which the forbidden reflection 1/2 (214) in the SAED was present. The larger inter planar spacing in HRTEM was about two times of the (214) plane spacing, which indicated the appearance of the forbidden reflection 1/2 (214). The literature also reported that the stacking faults can resulted in the presence of forbidden reflection in SAED pattern [25]. The forbidden reflection was probably the evidence of stacking faults in this SPS sintered boron carbide. Huyun et al. observed the superposition at grain boundary in SAED, in which the forbidden reflection was not present [16]. The cause of superposition was not referred in the literature. The defects in the microstructures of the 1500 °C, 1550 °C and 1600 °C sintered samples were probably represented by the humps at the XRD patterns. The defects can increase the diffusion coefficient and thus improve the mass transfer [26], by which the densification can be enhanced. As shown in Fig. 3, the defects firstly appeared at 1500 °C, at which the significant increasing of relative density occurred, Fig. 1. The defects were also present in the high density samples sintered at 1550 °C and

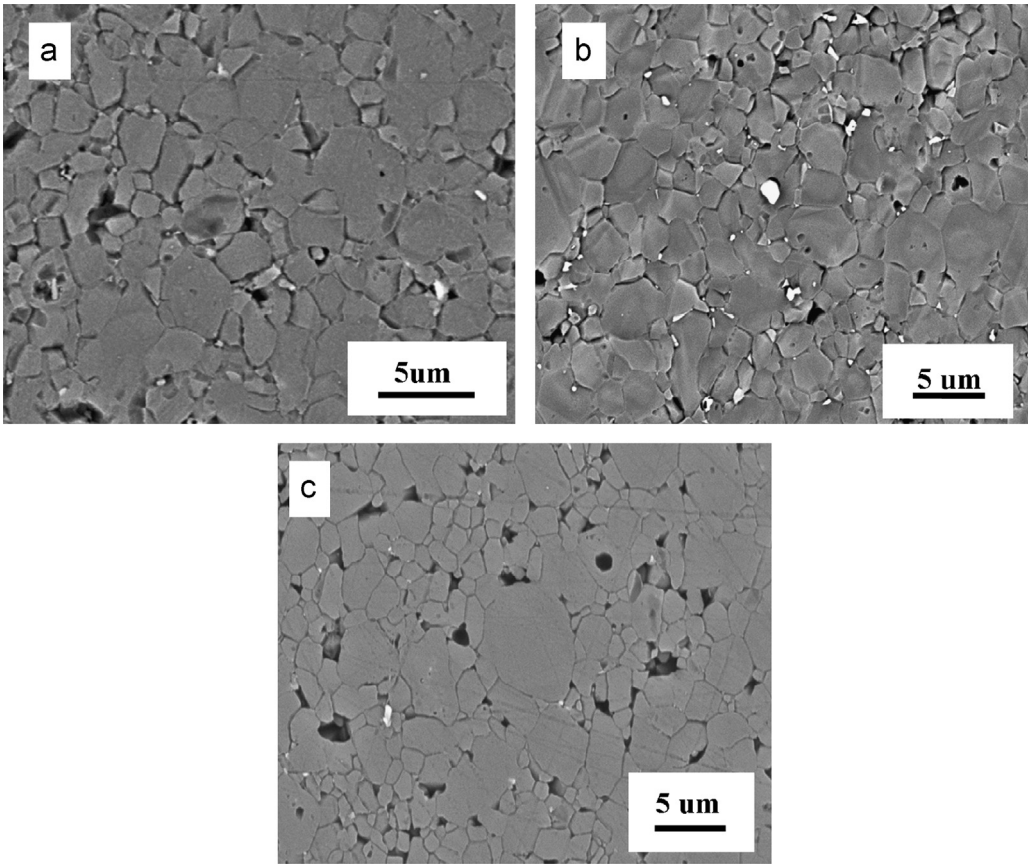

Fig. 7. Electrochemical etched polished surface of boron carbide samples sintered at 1500 °C (a), 1550 °C (b) and 1600 °C (c).

1600 °C. Therefore, the defects can effectively improve the densification of boron carbide in SPS sintering process.

The grain boundary structure was analyzed by TEM. Fig. 6 (a) and (b) showed the characterized straight and sharp grain boundaries, which was clean and impurities free. While the dark phase at the triple junction in Fig. 6(a) was determined to be impurities, which composed of Cr, Mn, Fe, Ni, P, B and O. The triple junction phase probably originated from impurities in the boron carbide powder. Since the impurity phase containing B and O, which was probably the borate phase. The impurities were considered to contribute to the densification of boron carbide by forming liquid phase during HP and SPS sintering [16,27]. Since the melting points of most borates were lower than the present sintering temperatures, the impurities may also enhance the densification by forming liquid phase. During sintering process, accompanied with the grain growth, the residual impurities were pushed toward the triple junction and the grain boundary became clean (Fig. 6 (b)).Certain kind of impurities may also escape from the sintered samples at high temperature as have reported [16].

3.3. Mechanical properties

The flexural strength values of the 1500 °C 1550 °C and 1600 °C sintered boron carbide were 316 MPa, 585 MPa and 828 MPa respectively. The flexural strength increased with the increasing temperature. The change of the flexural strength can be corresponded to the microstructure of the samples. Pores can be observed in the samples sintered at 1500 °C (Fig. 2(c) and (d)) and 1550 °C (Fig. 2(e) and (f)), while nearly fully densified microstructure was observed for the 1600 °C sintered boron carbide (Fig. 2(g) and (h)). Fig. 7 showed the electrochemical etched surface of the 1500 °C,1550 °C and 1600 °C sintered boron carbides. The mean grain sizes of samples sintered at 1500 °C, 1550 °C and 1600 °C were about 1.95 μm, 3.41 μm and 4.17 μm, respectively. The grain growth with the increasing of sintering temperature was not obvious. The fine grain size and pore free microstructure probably led to the high flexural strength of boron carbide sintered at 1600 °C. The flexural strength of the boron carbide sintered at 1600 °C was also much higher than those of HP [2] and SPS sintered [15,28] boron carbide ceramics.

The Vickers hardness and fracture toughness were measured for boron carbide samples sintered at1500 °C,1550 °C and 1600 °C. The Vickers hardness of samples sintered at 1500 °C, 1550 °C and 1600 °C were 27.63 GPa, 32 GPa and 31 GPa respectively. The Vickers hardness of samples sintered at 1600 °C was slightly lower than that of 1550 °C. The fracture toughness of samples sintered at 1500 °C, 1550 °C and 1600 °C were 2.95 ± 0.26 MPa $m^{1/2}$, 2.62 ± 0.08 MPa $m^{1/2}$ and 2.66 ± 0.29 MPa $m^{1/2}$, respectively. The fracture toughness of samples sintered at 1600 °C was lower than that of 1500 °C. The Vickers hardness and fracture toughness of the 1600 °C sample were comparable with that of pure boron carbide by HP [2]. The result revealed that 1600 °C was the

proper SPS sintering temperature and the as-prepared boron carbide ceramic was promising for practical use.

4. Conclusion

In this paper, boron carbide ceramics sintered by SPS was studied. It was found that the heating rate played a more important role on the densification than that of the holding time. Boron carbide samples can be densified in the temperature range of 1500–1600 °C without much grain growth. The suitable sintering temperature was 1600 °C. The relative density, flexural strength, Vickers hardness and fracture toughness of boron carbide sintered at 1600 °C were 98.33%, 828 MPa, 31 GPa and 2.66 ± 0.29 MPa $m^{1/2}$, respectively. The pore free microstructure and fine grain size may lead to the high flexural strength of boron carbide. Based on the microstructure, the defects (twins and stacking faults) and impurities both probably contributed to the densification of boron carbide ceramics.

Acknowledgments

This work was supported by the National Natural Science Foundation of China (No. 50990301, 51072210), Shanghai Science and Technology Committee and the State Key Laboratory of High Performance Ceramics and Superfine Microstructures.

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