High temperature flexural strength in monolithic boron carbide ceramic obtained from two different raw powders by spark plasma sintering

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High density monolithic boron carbide specimens with grain size of 3–5 μm were consolidated by spark plasma sintering using identical heating conditions and Ar or N₂ atmospheres. The effect of impurities from two different raw powders on the mechanical properties is not trivial and although much work was devoted1)–3) there are many open questions about the complex material-processing-properties correlations. The reasons are related to:

(i) high diversity of raw materials regarding particle size, shape, uniformity, boron to carbon ratio of the boron carbide considered a solid solution, and impurities;
(ii) bulks‘ features such as density, micro and nano structures;
(iii) specific aspects of processing technologies, e.g. pressure-assisted or pressure-less methods, liquid-phase-assisted or solid state processes;
(iv) different reaction and formation routes of secondary phases.

Enhancement of the mechanical properties is usually realized through introduction of different additives. Additives may also influence favorably sintering processes. The optimum amounts for the two situations can be different and solution is to take advantage of a pressure assisted processing technology. Popular is hot pressing, but in the last years spark plasma sintering is gaining more ground. This is because this technique is more flexible: high heating rates useful to avoid particle coarsening can be used. SPS is also debated10) as a method able to produce activated sintering effects. These effects are the consequence of the pulsed current utilization. The pulsed current is applied simultaneously with the axial pressure on the mold system loaded with the powder to be sintered.

It is also noteworthy that only a limited number of articles are investigating high temperature mechanical properties of boron carbide such as flexural strength. Results presented in literature often show contradictory trends.5)–15) In articles,5,8,10,13) samples were polycrystalline, while in Refs. 14, 15 samples were B₄C–TiB₂ eutectic crystals. Results point on different mechanical behavior. For example, when compared with room temperature bending strength, the bending strength at high temperature can be approximately constant,8,10) can decrease11) or it can be higher.12,14,15) Various explanations were given, but it is clear that when the samples and processing technologies, including starting powders, are very different and it is impossible to compare results.

Since boron carbide plays paramount role as material for high-temperature, cutting and armor applications, information on bending strength at different temperatures of boron carbide samples is required, especially for polycrystalline ceramic specimens. Hence in this study we consolidate bulk B₄C by SPS in Ar or N₂ gas from two different commercial powders supplied by different companies in order to investigate the effect of impurities on the mechanical properties of bulk ceramic specimens. Furthermore, this work we focused on the bending strength, σ, behavior of these ceramics. Measurements are performed using the three-point method at room temperature and up to 1600°C.

1. Introduction

Boron-carbide-based materials show useful mechanical properties for different practical applications. However, pure boron carbide is a difficult-to-sinter material and it is brittle due to its strong covalent character. Control and improvement of the mechanical properties is not trivial and although much work was devoted5)–7) there are many open questions about the complex material-processing-properties correlations. The reasons are related to:

(i) high diversity of raw materials regarding particle size, shape, uniformity, boron to carbon ratio of the boron carbide considered a solid solution, and impurities;
(ii) bulks‘ features such as density, micro and nano structures;
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2. Experimental

Boron carbide raw powders denoted A and B were purchased from SinoPharm Chemical Reagent Co Ltd. (Singapore) and International Labs (US), respectively. As-received powders were used to prepare bulk samples by SPS (SPS-1050, SPS-Syntex Inc., Japan). An amount of 3 g of the raw powder was wrapped into C-paper and loaded into a graphite mold system with the inner diameter of 2 cm. The loaded mold system was introduced
into a SPS furnace and a uniaxial pressure of 5 kN was applied on the punches. After vacuuming down to a pressure of 10 Pa, Ar or N₂ gas was introduced (0.8 atm). Heating was performed up to a maximum temperature of 1850°C and dwell time was 10 min. Heating from 700 to 1850°C was performed within 25 min, and during this processing step the uniaxial pressure was increased up to 80 MPa. Cooling from the maximum processing temperature down to room temperature was with a rate of 90°C/min and afterwards down to room temperature with a rate of 170°C/min. Bulk samples were labeled A-N₂, A-Ar and B-N₂ and B-Ar. Conditions of SPS processing were selected based on our previous studies16,17 as they produced samples from raw powder A with optimum mechanical properties (high σ₂5%, σ₁600, HV and intermediate KIC).

The Archimedes method was used to determine the apparent density of the samples. The relative density was 96.2, 98.6 and 97, 97.9% for samples A-N₂, A-Ar and B-N₂, B-Ar, respectively. Theoretical density of the samples was considered constant at 2.52 g/cm³11,12.

Bulk samples were extracted from the mold after SPS processing, cleaned, polished with diamond paper and paste and cut into rectangular bars of 3 × 2.5 × 20 mm using a spark erosion cutter. Bars were used as specimens in the three-points bending experiment.

X-ray diffraction (XRD) patterns on the raw powder and on spark plasma sintered samples were taken with a Bruker (CuKα radiation) diffractometer. Phase identification was made using EVA software and JCDPPowder diffraction database. Samples were observed by electronic microscopy. We used scanning electron microscopes (SEM) SU 8000 cold-emission FE-SEM Hitachi and JEOL 6500 F equipped with energy dispersive spectroscopy (EDS) detectors. Observations were made on fractured surfaces after SPS and after bending tests.

Three - points flexural tests were conducted using an “Instron 4505” setup (UK) in vacuum (0.1–0.2 Pa) at room temperature and at 1600°C, and in some cases at additional high temperatures. Results were averaged for 3 measurements.

3. Results

3.1 Structural and microstructural features of the raw powders and of the SPSed samples

Raw powders of boron carbide present some differences. XRD patterns (Fig. 1) show the presence of the boron carbide as the main phase. Composition of the boron carbide powder particles is different since XRD lines are better matched by those of B₄C (JCDP 35-0798) in powder A and of B₆.₅C in powder B (JCDPP 01-078-1574). Impurity phases are H₂BO₃ (B₂O₃ with water), graphite, and Si-based phases such as SiO₂, Si-B-O, SiC and FeSi. The powder A contains a higher amount of H₂BO₃ and a significantly lower amount of C-free and of Si-based phases comparative to powder B. The average particle size in the raw powders is not much different (3–5 μm). However, in the powder A, a higher amount of large agglomerates (≈10 μm) were observed.

X-ray diffraction patterns of sintered samples are presented in Fig. 2. Different 2θ positions of the XRDPeaks for the main boron carbide phase indicate that its composition is different among the samples and it is also different comparative to raw powders. Additional peaks that can be ascribed to boron carbide compositions B₂C or B₁₂(B, C, Si)₁₈ can be observed with difficulty. Results suggest that boron carbide composition changes during SPS.

The amount of the impurity phases is low. This aspect and the fact that peaks of different phases are overlapping do not allow precise phase identification. Si-based phases are SiC, Si₆B₅ and Si₅N₄ (sample A-N₂). Peak or peaks located around 2θ = 28.8° visible in the samples obtained from the powder B are also ascribed to Si-based phases [Si-B-C-O(N)] considering that Si (JCDP 078-2500) or SiO₂ (JCDP 01-070-2537) have intensive diffraction lines close to the indicated 2θ value. Sintered samples obtained from powder B have a higher amount of Si-based phases than in the samples obtained from powder A.

The presence of Si-based phases in samples A or B is confirmed by SEM/EDS investigations (Fig. 3). These phases are mainly located at triple points. Fe-containing phases such as Fe–B are present as traces according to XRD patterns and they are also found at triple points (not shown). Their amount is much lower than for Si-based ones. A higher amount of Fe-containing phases is in samples B. The triple point grains show distinctive morphology depending on their size. Namely, larger grains than 500 nm are of angular prism-like morphology and usually are identified as SiC. Smaller ones are of spheroidal-like or of star-like morphology (arms of the star are expanding along the grain boundaries). This morphology suggest that they precipitated from...
a melt. They show a large variation of compositions and sometimes exhibit a nonuniform spatial distribution of elements in the EDS measurements indicating the multiphase nature of these grains. Presented behavior can be considered as an evidence for the presence of a Si-containing melt during SPS processing. A conclusion cannot be formulated since the size of the grains and of the electron spot is comparable and the background can influence the measurement. The Si-containing liquid phase reacts with boron carbide, graphite, or B-O phases. One consequence is that the structure and composition of the boron carbide grains can change. Literature\(^1\) report occurrence of boron carbide phases containing Si in Si infiltrated boron carbide ceramic, and, for our samples, XRD data support this idea.

Si-containing thin films or nano precipitates denoted Si-B-C-O(N) are observed at boron carbide grain boundaries [Figs. 4(c), 4(g)]. Si-containing layers at grain boundaries as light edges of boron carbide grains are easily visualized for samples B that are rich in Si-based phases. In monolithic samples of Si\(_3\)N\(_4\), SiC or their composites [19 an therein refs.], the liquid phase at grain boundaries is identified as a silicon oxide.

In the samples processed in nitrogen, there are impurity phases of h-BN type (Fig. 2 and Ref. 17). In all samples, graphite (graphite) is available. These phases show a layered morphology and they are located at boron carbide grain boundaries [Figs. 4(a), 4(b)]. In the case of C-rich B- samples, graphite layers are frequently observed [Figs. 3(b), 4(a), 4(b)] and they can show large sizes comparable with the size of the boron carbide grains.

Presented information is summarized as follows: boron carbide changes their composition during SPS processing and this is expected to influence mechanical properties. In the boron carbide matrix there are grains located at triple points and along the boron carbide grain boundaries. The triple point grains of large size usually identified as SiC show an angular prism-like morphology, while the small ones are of sphere- or star-like shape and they have a complex Si-B-C-O(N) composition. Particular features of the small triple point grains provide arguments for the presence of a liquid phase during sintering. The liquid phase is the origin of the thin films at boron carbide grain boundaries from the Si-rich B-type samples and of the sphere-like nano precipitates located at grain boundaries. At grain boundaries are also found layered graphite and h-BN type phases. The last ones form when SPS is performed in nitrogen atmosphere. A higher amount of secondary phases are in samples B than in A.

### 3.2 Fractography observations after the bending test

Different fracture mechanisms are active for our samples. Mechanisms can be grouped as transgranular and intergranular. Breakage of transgranular type is observed for large boron carbide grains and for large triple point grains such as SiC (Fig. 5). Small boron carbide and triple point grains show an intergranular pull out mechanism: on the one hand, boron carbide grains are extracted from the matrix, and on the other hand triple point sphere-like grains are often found as self supporting grains standing up from the corners of the boron carbide grains or from the sintered boron carbide blocks. It can be also observed that intergranular phases such as layered ones, especially large ones (e.g. graphite for samples B in Fig. 5), contribute to boron carbide pull out through intergranular sliding. Sliding can be favored also by Si-based film-like phases located along the boron carbide grain boundaries, but situation is complex because if nano precipitates are available at grain boundaries they can influence sliding, e.g. suppressing it. If the pull out of the boron carbide grain is not realized, small triple point grains promote deflection of the propagating crack. For room temperature bending, the main mechanism is the transgranular fracturing of the boron carbide grains.
In samples subject to bending at high temperature, small triple point grains are found in the close vicinity of the crack origin. It is possible that crack opening [Figs. 5(b) and 5(b) detail] occurs as a consequence of the thermal expansion mismatch. Si-based phases are expected to change significantly their mechanical behavior at high temperatures (above 1200–1400°C) comparative to room temperature. On the one hand, the silicon oxide frozen melt is considered the reason for subcritical fracturing and bending strength decrease with temperature in monolithic Si₃N₄, SiC or Si₃N₄–SiC composites and, on the other hand, in SiC samples obtained by chemical vapor deposition that are free of silicon oxide frozen melt, the bending strength increases with temperature in the 900–1500°C range. The presence of such silicon oxide phase at grain boundaries and at triple points (likely as the arms of the star-like triple point grains) in our samples may influence fracturing of the Si-rich samples especially at high temperatures. Indeed for Si-rich B-samples surface roughness after bending test at 1600°C is the highest and this indicates on a higher plasticity for samples B than for A (Fig. 4). Actually, sintered samples B subject to bending test at high temperatures show some kind of macro bridging [Figs. 4(e), 4(f)]. Surface morphology strongly resembles the morphology from Si₃N₄ or SiC monolithic samples reported in literature. For all our samples there are also some similarities as well as differences with B₄C presented in Ref. 8. The B₄C sample mentioned in literature was fabricated from a powder with a purity of 99% containing 0.1% wt. Fe and 0.1% wt. Si. De With found that this sample has constant $K_{IC}$ and $\sigma$ with temperature (20–1500°C), and determined that no surface stress is present. He also reported that stress–strain curves were entirely linear up to 1500°C with no indication of a plastic component. Based on these results and on microscopy observations De With concluded that fracture mode was entirely transgranular at all testing temperatures and the flaw size [or the crack length in Eq. (1), see Section 4, Discussion] is temperature independent. Assuming the behavior proposed by De With, our data suggest that apart from the major trangranular mechanism imposed by the B₄C grains, for our samples, due to impurity phases also a certain share of intergranular-type mechanisms are active, and the ratio transgranular/intergranular changes with temperature. A higher level of impurity phases in samples B and their influence depending on their type can produce a more plastic fracture when compared to samples A and they will also increase the samples bending strength sensitivity with temperature. We shall note that for our samples stress–strain curves at all temperatures were linear (Fig. 6), i.e. a significant plastic contribution was not revealed. This result and that the transgranular mechanism is known to be specific for large grains, i.e. mainly to boron carbide from our samples, the observed boron carbide compositional changes during SPS with the involvement of Si, as already addressed in Section 3.1, would be the main factor to explain the extended plasticity for Si-rich samples. Nevertheless, the interplay of boron

Fig. 5. SEM images taken on the surface of the samples (a): A-N₂ and (b): B-N₂ samples after bending test at room temperature and at 1600°C, respectively. Square (yellow, red, blue): transgranular fracturing of triple-point large size phases (mainly SiC, >500 nm). Blue: step-like fracture, red: transgranular fracture of B₄C and SiC couple, yellow: microcrack opening (for a higher magnification see inset) with an impurity phase at the crack origin Rectangle (orange): typical transgranular fracturing of boron carbide grains; Circle (green): sliding and pull out (intergranular) mechanism of small boron carbide grains aided by the presence of layered or thin film melt-frozen phases (silicon oxide) located at grain boundaries and indicated with arrows; Triangle (blue): crack deflection at triple-point grains; Rectangle with rounded corners (red): very small precipitates at boron carbide grain boundaries Star: small impurity phases at triple points (often standing outwards surface).

Fig. 6. Typical loading diagrams of the B₄C ceramics tested at room and elevated temperatures by the three-point flexural strength test for samples processed by SPS in N₂ atmospheres from raw powders A and B.
carbide with the other phases should be also considered.

4. Discussion

Bending strength for SPSed samples are presented in Fig. 7. The bending strength \( \sigma \) according to Griffith-Irwin formula\(^\text{21}\) is

\[
\sigma = A \cdot K_{IC} \cdot c^{-0.5}
\]

where \( K_{IC} \) is the critical stress factor taken as fracture toughness \( K_{IC} \), \( c \) is the crack length and \( A \) is a constant. Therefore, bending strength \( \sigma \) enhances when fracture toughness enhances providing that the crack length \( c \) is constant, decreases, or the increase of \( c^{-0.5} \) is slower than the increase of \( K_{IC} \).

Samples processed in nitrogen have higher values of bending strength comparative to similar samples processed in Ar. This result suggests that nitrogen-containing phases such as those of h-BN type, Si\textsubscript{3}N\textsubscript{4} or boron carbide containing nitrogen enhance the bending strength at room or at high temperature.

Another observation is that samples B containing a higher amount of Si-based secondary phases and of layered graphite phase display a lower bending strength at room temperature than for samples A, but, at the same time, they have a significant increase of the bending strength from room to high temperature (48% in Ar and 35% in N\textsubscript{2}), while for the samples from powder A the bending strength increases by a smaller increase (16% in Ar and 4% in N\textsubscript{2}) from room to high temperature (Table 1, Ref. 22). The enhancement trend of bending strength with temperature is general regardless samples and processing. This indicates that the trend is given by boron carbide as the main phase as it was anticipated from the fractography details presented in Section 3.2. Secondary Si-based phases and layered graphite phase control bending strength values and the enhancement rate from room to high temperature. Literature\(^\text{23}\) indicate that large graphite grains in boron carbide samples decrease mechanical characteristics under compressive dynamic loads. In our case, C-layers favor an intergranular pull out fracture mechanism (Figs. 4(a)–4(d) and 5) as already noted in Section 3.2. The graphite is structurally and mechanically similar to h-BN and it is expected to provide a similar effect. We have seen that both phases promote sliding of boron carbide under load, but for h-BN the effect is to enhance the bending strength and for graphite is to decrease it. This contradiction leads to idea that for our samples the influence of layered phases, h-BN or graphite, is not significant and it is washed out by the influence of boron carbide and silicon-containing phases. If we admit this situation, and considering the boron carbide influence, already pointed out, the silicon-containing phases affect the values of the bending strength and the enhancement rate. Microcrack formation (crack opening), deflection of propagation cracks caused by thermal expansion mismatch and crack confinement realized for a certain amount of additive well dispersed in a boron carbide matrix with relatively small grains (<1 \( \mu \)m) improved the fracture toughness in \( B_{2}C-CrB_{2} \) or \( B_{2}C-TiB_{2} \) composites\(^\text{24,25}\). In turn, this led to a higher room temperature bending strength meaning that parameter \( c \) in formula (1) was not significantly influenced. If we apply this result to our samples (Table 1, Fig. 8), the scenario is the triple point Si-based phases involved in microcrack formation and deflection of the propagating cracks (see Section 3.2) can enhance the bending strength, while the Si-containing films at grain boundaries promoting sliding will enhance plasticity and the crack size so that the bending strength can decrease.

Therefore, the presence of Si-based films may explain why at room and at high temperature samples B containing a higher amount of Si-based phases have a lower bending strength than samples A. The enhancement rate from room to high temperatures, that is higher for Si-rich samples B, can be correlated with a more effective microcrack formation at high temperatures due to a higher amount of small Si-containing triple point phases. Samples B-N\textsubscript{2} and B-Ar show bending strength enhancement curves more parallel than for samples A-N\textsubscript{2} and A-Ar (Fig. 7). A stronger effect of Si-containing triple point grains specific for Si-rich samples B is reducing the effect of nitrogrenation on bending strength enhancement.

5. Conclusion

Our results provide arguments that boron carbide has the

Table 1. Physical and mechanical properties of boron carbide ceramics processed by SPS in Ar or N\textsubscript{2} atmospheres from raw powders A and B (Theoretical density of \( B_{2}C \) samples was taken constant at 2.52 g/cm\textsuperscript{3}).

| Sample ID | Relative Density, % | Indentation fracture toughness, \( K_{IC} \), MPa m\textsuperscript{1/2} | Three-point flexural strength, MPa |
|-----------|---------------------|--------------------------------|-------------------------------|
| A-N\textsubscript{2} | 96.2 | 4.1 ± 0.7 | 574 ± 16 612 ± 14 |
| A-Ar | 98.6 | 4.6 ± 0.4 | 508 ± 21 598 ± 17 |
| B-N\textsubscript{2} | 97.0 | 4.4 ± 0.3 | 394 ± 17 532 ± 11 |
| B-Ar | 97.9 | 4.7 ± 0.5 | 329 ± 21 373 ± 12 |

*Indentation fracture toughness \( (K_{IC}) \) was calculated considering the length of the cracks in indentation experiments and by using the formula for hard materials given in Ref. 22 using load of 1000 gf (9.8 N).
strongest influence on bending strength at room and at high temperature and this is in agreement with literature. In addition, compositional variation of the boron carbide grains has to be carefully considered: in our case Si inclusion in the crystal lattice of boron carbide is anticipated, but further research is necessary.

For our samples we observe a general enhancement trend of the bending strength from room temperature to high temperature. The values of the bending strength and the rate of enhancement are in agreement with literature. In addition, it is necessary to make a distinction between grain boundary phases favorable to intergranular sliding such as layered phases e.g. large grains of graphite, (to a less extent) h-BN for samples processed in nitrogen, and the silicon-based melt phases that decrease or does not strongly influence the bending strength from room temperature to high temperature. For our samples we observe a general enhancement trend of the bending strength through the decrease of the crack length.

Values of the bending strength at high temperatures (above 400 MPa) obtained for our samples are higher than the reported ones for monolithic polycrystalline B₄C.

The work brings arguments for a careful selection of the boron carbide precursor powders in order to control or optimize mechanical properties even in the absence of purposely introduced additives.

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