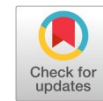


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Pressureless sinterability study of ZrB_2 –SiC composites containing hexagonal BN and phenolic resin additives

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ABSTRACT

This research is dedicated to investigating the role of different amounts of hexagonal BN (hBN: 0, 1.5, 3, and 4.5 wt%) on the pressureless sinterability of ZrB_2 –25 vol% SiC ceramics. Phenolic resin (5 wt%) with a carbon yield of ~40 % was incorporated as a binder to the powder mixtures and after initial cold pressing, the final sintering process was performed at 1900 °C for 100 min in a vacuum furnace. The as-sintered specimens were characterized by X-ray diffractometry, field emission scanning electron microscopy, and energy-dispersive X-ray spectroscopy. The results disclosed that the incorporation of 1.5 wt% hBN could increase the relative density to ~92%, while the sample with zero hBN content just reached ~81% of full densification. Appropriate hBN content not only facilitated the particle rearrangement during the cold pressing, but also removed the harmful oxide impurities during the final sintering. Nevertheless, the addition of higher amounts of hBN remarkably lessened the densification because of more delamination of the non-reacted hBN flakes and release and entrapment of more gaseous by-products induced by the reacted hBN phases.

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KEYWORDS

Ultrahigh temperature ceramics
Fractography
Pressureless sintering
Densification
BN additive

1. Introduction

Zirconium diboride (ZrB_2) is one of the compounds known as ultrahigh temperature ceramics, which has a chain of perfect properties such as high hardness, ultra-high melting point, and chemical inertness [1–4]. Although this ceramic exhibits such excellent properties, sintering of ZrB_2 is not easy because of the surface impurities on the starting particles in powder-based manufacturing processes, as well as low diffusivities [5–8]. By introducing SiC as a secondary phase to the ZrB_2 matrix, the resultant ZrB_2 –SiC composite will have boosted oxidation resistance and mechanical performance as compared to the unreinforced ZrB_2 [9–12].

Dense or near fully dense structural ceramics can be made by hot pressing or field-assisted sintering [13–18]. Anyway, utilizing the mentioned routes is only limited to simple shape geometries since

manufacturing complex products needs time-consuming and expensive post-sintering activities such as machining. Hence, as a good alternative, pressureless sintering can be employed for the production of near-net-shape components [19–21]. Several efforts have been done to fabricate ZrB_2 materials by pressureless sintering. The densification of ZrB_2 can be increased and sintering temperature can be decreased by either removal of oxide contaminations or liquid phase formation via adding appropriate additive materials [22–25]. For example, the densification is greatly improved by adding polymeric precursors due to the production of residual carbon after pyrolysis of such polymer additives, which can remove the oxide layers that existed on the ZrB_2 powders [26].

Zhu and Wang [27] prepared ZrB_2 –SiC ceramics by pressureless sintering of liquid polycarbosilane and ZrB_2 powders. They showed that adding the liquid polycarbosilane effectively reduces the sintering

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temperature. During the pressureless sintering, carbon and silicon were provided by this polymeric additive at high temperatures. The surface oxides of ZrB_2 were removed by the carbon, and the densification was enhanced by the silicon.

Li et al. [28] studied the influence of hBN grain size on microstructural and mechanical characteristics of the hot-pressed ZrB_2 -SiC ceramics comprising 10 vol% hBN. The fracture toughness of the ZrB_2 -SiC-BN composites was higher than those reported for the monolithic and SiC reinforced ZrB_2 -based materials. This outcome was related to the high aspect ratio of hBN additive with weak interfacial bonding, which enhanced stress relaxation adjacent to the crack tip and cracks deflections. However, the weaker interface bonding and the lower relative density resulted in the reduced flexural strength. Wu and Zhang [29] reported that the addition of BN tremendously decreased the hardness while noticeably improved the machinability of ZrB_2 -SiC ceramics fabricated by hot-pressing. They also indicated that the oxidation resistance of ZrB_2 -SiC material can be enhanced by the addition of BN, due to the formation of immense borosilicate and zirconium silicate films on the surface of BN-doped ceramics, which act as efficient barriers against further oxygen diffusion into the unaffected zones. Haghgooye Shafagh et al. [30] proved the beneficial role of incorporating 1 wt% hBN on fracture toughness, hardness, and consolidation enhancement of hot-pressed ZrB_2 -SiC ceramics. Anyway, they found that more hBN addition has a negative influence on the properties (densification, hardness, and fracture toughness) of the sintered compounds. Pourmohammadi Vafa et al. [31] reported that a near fully dense sample with enhanced bending strength was obtained by the addition of 2.5 vol% hBN in the hot-pressed ZrB_2 -SiC materials. Anyway, because of the poor sinterability of BN flakes, adding more additive (> 5 vol%) resulted in weakened hardness and strength.

Chen et al. [32] investigated the scratch behavior of ZrB_2 -SiC ceramics densified by spark plasma sintering and reinforced with hybrid BN nanotubes and BN nanoplatelets. The hybrid BN reinforced composite exhibited commonly ductile deformation. Easier grain sliding because of BN additives placed at grain boundaries and attached to the scratch surface could provide remarkable self-lubrication to lessen lateral force during scratch. Wu et al. [33] employed spark plasma sintering to synthesize the machinable ZrB_2 -SiC-BN ceramics through solid-state reaction of as-received ZrH_2 , B_4C , and Si_3N_4 materials. They found that the reactive approach is helpful for the production of dense, homogeneous, and fine-grained microstructures. Meanwhile, excellent machinability was achieved in the samples contained more than 20 vol% in-situ synthesized hBN. Zou et al. [34,35] also fabricated dense ZrB_2 -37 vol% hBN ceramics with attractive mechanical performance through reactive spark plasma sintering of ZrN and B precursors.

Liu et al. [36] studied the oxidation and mechanical behavior of laminated ZrB_2 -SiC/BN materials manufactured by tape casting and hot-pressing. The fracture toughness and flexural strength were improved by oxidation for 30 min at 1300 °C, due to the healing effect of the imperfections of the oxidation-induced glassy SiO_2 layer. Bai et al. [37] found that the laminated ZrB_2 -SiC_{whisker}/BN ceramics have better thermal shock resistance than the ZrB_2 -SiC_{whisker} composites due to the bifurcation, crack deflection, and delamination mechanisms. They proposed quenching-strengthening as a suitable route for the evaluation of the thermal shock resistance in laminated ceramic composites.

The goal of this research paper is to explain the role of different amounts of hBN additives on the densification performance and microstructure development of ZrB_2 -SiC composites. Pressureless sintering at 1900 °C for 100 min is used for the production of ceramic matrix composites. The relative density values and fractographical microstructures of the pressureless sintered samples as a function of hBN content are reported and discussed.

2. Experimental procedure

Pressureless sintering under vacuum conditions was used for the fabrication of ZrB_2 -SiC-based ultra-high temperature ceramic composites. Commercially available powders of ZrB_2 (particle size <5 μm and purity > 99.0%), SiC (particle size <8 μm and purity >98.0%), and hexagonal BN (particle size <2 μm, purity >99.0%) were used as initial materials. Different amounts of hBN sintering aid (0, 1.5, 3, and 4.5 wt%) and a fixed amount of phenolic resin binder (5 wt% with a carbon yield of ~40 % after pyrolysis) were added to the initial mixtures of ZrB_2 -25 vol% SiC powders.

The blending process was carried out in zirconia cups using ethanol medium by ball-mixing of the prepared slurries for 100 min soaking time. Afterward, a rotary evaporation facility was employed to dry the as-wet-mixed slurries at ~110 °C. Next, the dried mixtures were pulverized using a mortar and pestle and passed through a screen to provide uniform composite mixtures. The as-sieved powder mixtures were biaxially compacted by cold pressing to prepare appropriate pellets for the final pressureless sintering at 1900 °C for 100 min.

The theoretical density of each sample was calculated using the rule of mixtures, supposing that the components do not react with each other during the sintering process. The bulk density of the pressureless sintered UHTCs was measured using the Archimedes technique. Then, the bulk density was divided by the theoretical one to estimate the relative density.

Phase identification of the as-pressureless-sintered composite doped with 4.5 wt% hBN, the sample with the highest content of sinter additive in this research study, was performed by X-ray diffraction test. Some possible reactions between the initial materials and their surface oxide layers during the pressureless sintering were assessed thermodynamically through HSC Chemistry software. A field emission scanning electron microscope was used to investigate the microstructures of the as-fractured specimens. An energy-dispersive spectroscope was employed during fractographic studies to determine the elemental distributions in different phases/areas.

3. Results and discussion

Fig. 1 presents the relative density of the pressureless sintered composite specimens as a function of hBN content. It can be observed that the relative density significantly increased from 81.3% for the hBN-free sample to 91.6% for the sample doped with 1.5 wt% hBN. By further addition of the hBN, the composite relative density remarkably decreased as the values of 85.1% and 74.5% were measured for the samples doped with 3 and 4.5 wt% hBN, respectively. It should be noted that the relative density of the sample doped with 4.5 wt% hBN is even lower than that of the hBN-free one.

The presence of the low amount of hBN substantially boosted the densification of the ZrB_2 -SiC composite. The hBN is known as a great lubricant; hence, the presence of this phase among the primary powder

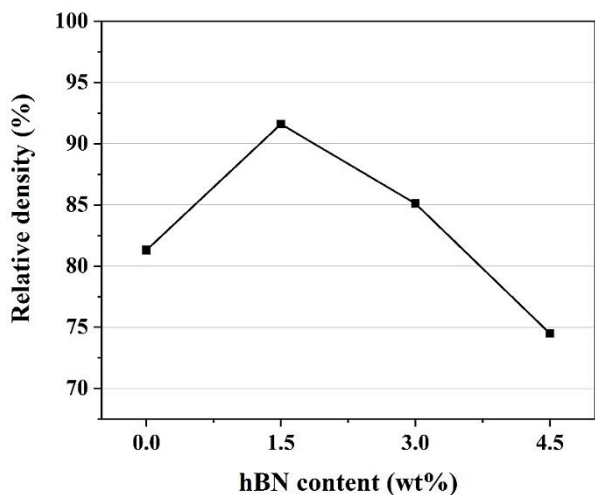
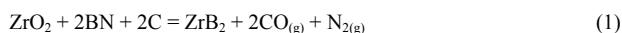


Fig. 1. Relative density of pressureless sintered ZrB_2 -SiC composites as a function of hBN content.

particles may accelerate their rearrangement via increasing the sliding of ZrB_2 and SiC powders during the cold pressing step. Such an additive may react with oxide impurities, which naturally form on the surface of every non-oxide ceramic material during the sintering step. In this research work, as the ZrB_2 is the dominant phase, the ZrO_2 can be supposed as the main impurity in this system. At elevated temperatures during the pressureless sintering (>1785 °C; extracted using the HSC Chemistry package), the ZrO_2 contamination reacts with BN and C additives according to Eq. 1:



Generally, the presence of such surface oxide layers disconnects initial ZrB_2 and SiC powders from reciprocal contact, which lessens the sinterability of composite precursors. The better densification obtained for ceramic containing 1.5 wt% hBN is attributed to the elimination of ZrO_2 impurity. Meanwhile, the in-situ synthesis of ultrafine ZrB_2 phases (according to Eq. 1) in the vicinity of the maternal ZrB_2 particles can promote the sinterability since the minimalization of surface free energy is also a driving force for better densification.

The XRD pattern of the pressureless sintered ZrB_2 -SiC specimen

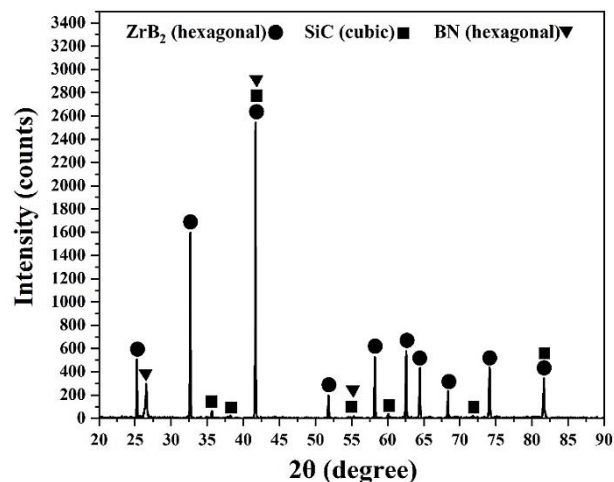


Fig. 2. Phase analysis of pressureless sintered ZrB_2 -SiC composite doped with 4.5 wt% hBN.

doped with 4.5 wt% hBN is shown in Fig. 2. As can be detected, there are three phases in the as-pressureless-sintered sample: ZrB_2 , the matrix phase; SiC, the reinforcement; hBN, the sintering additive. There are no detectable signals from the oxide impurities such as ZrO_2 , SiO_2 , or B_2O_3 related to the surface of ZrB_2 and SiC starting particles. There is also no visible diffraction peak from the phenolic resin because the as-pyrolyzed products are mostly amorphous.

The fracture surfaces of the pressureless sintered ZrB_2 -SiC composites doped with 1.5 wt% hBN are shown in Fig. 3. This sample has reached a relative density of 91.6%, which means that more than 8% of porosities have remained in the sintered product. Anyway, by comparing to the hBN-free sample with the relative density of 81.3%, it is disclosed that such a low amount of hBN additive (~10%) can significantly decrease the porosities content (~10%) in the sintered ceramic. The dark-gray areas in Fig. 3a are SiC phases dispersed in the light-colored ZrB_2 matrix. No obvious hBN phase is observed in this figure, but an amorphous phase can be seen at the higher magnification (Fig. 3b). Given the interfacial cleanliness of the ZrB_2 and SiC grains, seen in Fig. 3a, it can be supposed that the bonding between them is strong as a representative of the well-progressed sintering.

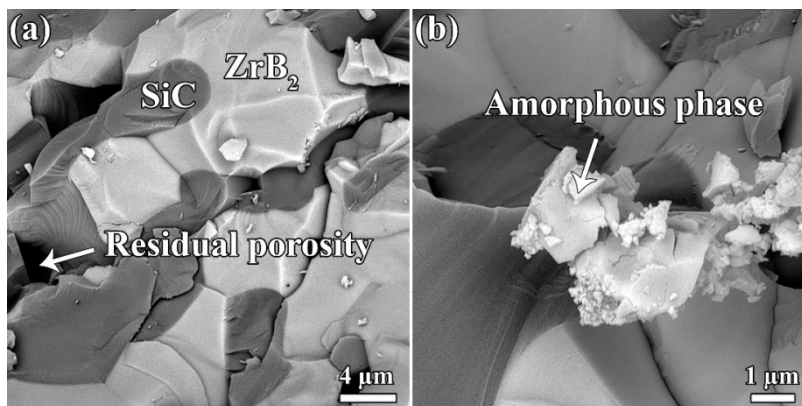


Fig. 3. SEM fractographs of pressureless sintered ZrB_2 -SiC composite doped with 1.5 wt% hBN.

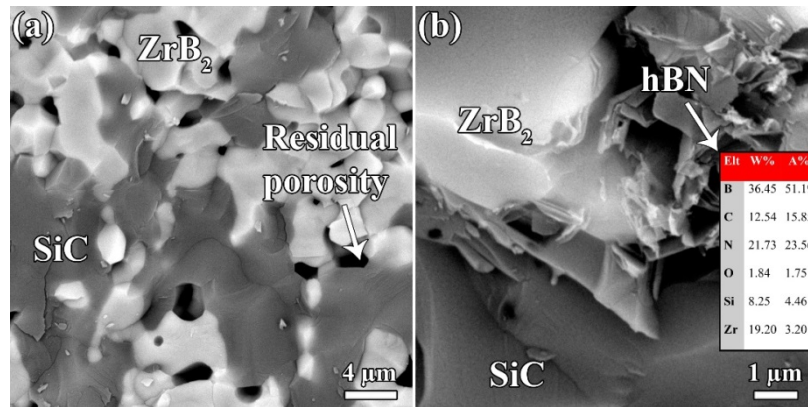


Fig. 4. SEM fractographs of pressureless sintered ZrB_2 -SiC composite doped with 3 wt% hBN.

Fig. 4 shows the fracture surfaces of the pressureless sintered ZrB_2 -SiC composites doped with 3 wt% hBN. Fig. 4a obviously discloses that the amounts of porosities in this sample are higher than those observed in the composite doped with only 1.5 wt% hBN (see Fig. 3a). In other words, with increasing the hBN content from 1.5 wt% to 3 wt%, the relative density drops from 91.6% to 85.1%. The elemental analysis (EDS) attached as an inset in Fig. 4b verifies the presence of the hBN phase with flaky morphology in the as-sintered specimen.

According to the chemical reaction of Eq. 1, although the hBN additive can increase the sinterability of the composites via the removal of the harmful ZrO_2 impurities and the in-situ formation of the pleasant ZrB_2 fine particles, the CO and N_2 gases are also formed as destructive by-products. Due to the applied vacuum during the pressureless sintering, the released gases could be removed from the sample doped with only 1.5 wt% hBN. However, the amounts of released gaseous by-products increase with increasing the amount of hBN additive. Therefore, it seems that the vacuum level is not high enough to remove the huge amounts of gas porosities in the samples doped with greater hBN contents.

The SEM fractographs of the pressureless sintered ZrB_2 -SiC composites doped with 4.5 wt% hBN are shown in Fig. 5. Fig. 5a clearly verifies that there are a lot of pores inside the sintered composite, which is in harmony with the measured low relative density

of 74.5%. The delaminated and bent hBN flakes are observed at the interfaces of the main ZrB_2 and SiC particles (Fig. 5b). It is well-known that the residual thermal stresses in the composite samples can play a great role in happening such a phenomenon. Wu et al. [33] reported that ZrB_2 and SiC have average thermal expansion coefficients of $5.2 \times 10^{-6} \text{ }^\circ\text{C}^{-1}$ and $3.3 \times 10^{-6} \text{ }^\circ\text{C}^{-1}$, respectively. Due to such a great thermal expansion mismatch between these phases, Watts et al. [38] approximated tensile stress of ~ 450 MPa in ZrB_2 grains and compressive stress of ~ 880 MPa in SiC grains, which were generated during the cooling process in the ZrB_2 -SiC composite materials [39]. In addition, because of the nature and morphology of hBN flakes, the thermal expansion coefficient of this material is extremely anisotropic. Therefore, the mismatch between ZrB_2 , hBN and SiC phases made the interfacial hBN flakes subject to different tensile or compressive stresses, when the composite was cooled down to the ambient temperature. It seems that the compressive stresses led to the bending of the hBN flakes, while the tensile stresses resulted in the delamination of the layers.

It was found from the fractographical observations that the porosities in the pressureless sintered composites are generally settled at the grain boundaries. This issue may be related to some phenomena which happened during the sintering process. For example, the surface oxide of B_2O_3 or some by-products, initiated from the possible reactions

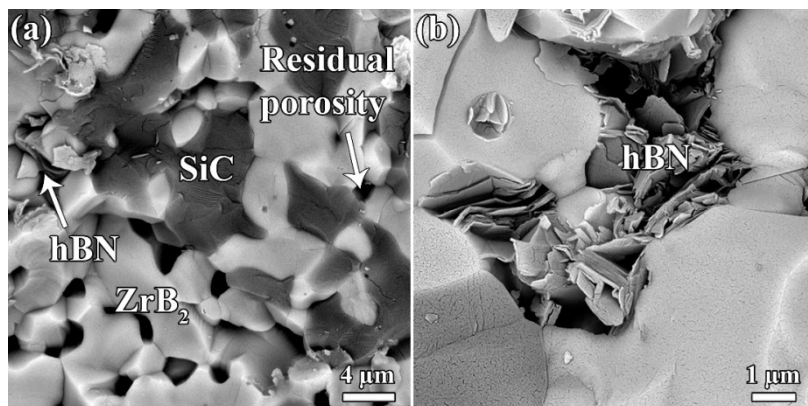


Fig. 5. SEM fractographs of pressureless sintered ZrB_2 -SiC composite doped with 4.5 wt% hBN.

between the precursors and the oxides, may evaporate and lead to the creation of porosities between the grains/phases. Meanwhile, when higher amounts of hBN are added to the starting powder mixture, more delaminated and bent hBN flakes with different orientations appear in the sintered specimens. Such phenomena, which are attributed to the thermal expansion coefficient mismatch between the components, may lead to the porosity entrapment at the interfacial areas and result in the decreased relative density.

4. Conclusions

The influence of various amounts of hBN additive (0, 1.5, 3, and 4.5 wt%) on the fractographical features and densification trend of ZrB₂-25 vol% SiC composite ceramics were reported and discussed. The samples were prepared using similar pressureless sintering conditions. The incorporation of 1.5 wt% hBN increased the relative density to ~92%, but the addition of higher amounts of hBN considerably ruined the densification progress. The effect of hBN on facilitating the particle rearrangement during the cold pressing and removing harmful oxide layers during the final sintering was discussed. If more nitride additive was introduced into the initial powder mixture, more delamination and bending were observed in the non-reacted hBN flakes, while more gaseous by-products were released around the reacted hBN phases.

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