A High Strength Alumina-Silicon Carbide-Boron Carbide Triplex Ceramic

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Abstract

A ceramic particulate composite composed of oxide, and carbide ceramics was found to have high strength, hardness, and fracture toughness values. A composition consisting of Al_2O_3 with 15 vol% SiC and 15 vol% B_4C additions was produced by hot-pressing at 1650°C for 30 min, with full density reached after ~ 5 min at temperature. Both WB and WB_2 were observed, with the W source presumably an impurity from WC milling media, and $\text{Al}_{18}\text{B}_4\text{O}_{33}$ was also detected following densification. Strength was ~ 880 MPa, which is greater than values reported for comparable composites of Al_2O_3 containing 30 vol% SiC or B_4C . Vickers hardness was ~ 21 GPa, and fracture toughness was $\sim 4.5 \text{ MPa}\cdot\text{m}^{1/2}$, comparable to values reported for the binary mixtures. The calculated critical flaw size of the material was similar to the size of the SiC/ B_4C clusters and microcracking at grain boundaries. The latter resulting from thermal expansion mismatch between the Al_2O_3 matrix and SiC/ B_4C reinforcing phases.

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1. Introduction

Aluminum oxide (Al_2O_3) based ceramics have been studied extensively due to their desirable properties such as high hardness, strength, toughness, low electrical conductivity, and good chemical stability.[1] Dispersed, second phase particles of ZrO_2 , SiC, TiB_2 , TiC, BN and B_4C have been incorporated into alumina ceramics resulting in improved mechanical properties.[2-9] Strength enhancements have typically been attributed to reduced grain growth during sintering due to grain pinning, while enhanced fracture toughness is due crack bridging and crack deflection as a result of the dispersed second phase.

Incorporation of SiC[5, 10-13] or B_4C [3, 4, 14] into Al_2O_3 ceramics improved mechanical properties. Shi et al. reported the properties of hot-pressed Al_2O_3 with submicron SiC additions. Strength was reported to increase from 280 MPa for pure Al_2O_3 to 615 MPa for 20 vol% SiC additions, with toughness values as high as $7.6 \text{ MPa}\cdot\text{m}^{1/2}$ reported for 5 vol% SiC additions.[5] Additions of SiC nanoparticles (SiC_n) to Al_2O_3 have led to even greater improvements in strength.[10-13] Chae et al. reported a strength of 810 MPa for Al_2O_3 -20 vol% SiC_n . [12] The high strength was attributed to increased propensity for transgranular fracture when SiC was present due to the presence of thermal residual stresses arising from the mismatch in coefficient of thermal expansion (CTE) between Al_2O_3 ($8 \times 10^{-6} / \text{K}$) and SiC ($4.2 \times 10^{-6} / \text{K}$), which led to the formation of residual stresses during cooling from processing temperatures. Similar improvements have been reported for Al_2O_3 - B_4C ceramics. Liu and Ownby reported an increase in strength for hot-pressed Al_2O_3 - B_4C with up to 30 vol% B_4C from ~ 420 MPa for pure Al_2O_3 to ~ 580 MPa for Al_2O_3 - B_4C , with fracture toughness increasing from $\sim 4.0 \text{ MPa}\cdot\text{m}^{1/2}$ to between 4.7 and $5.3 \text{ MPa}\cdot\text{m}^{1/2}$ for the two phase ceramics.[3] Jung and Kim investigated the Al_2O_3 - B_4C system, reporting improvement in strength (325 to 550 MPa), toughness (2.4 to $4.0 \text{ MPa}\cdot\text{m}^{1/2}$) and hardness (15.2 to 20.6 GPa) as a result of the finer grain size produced by grain pinning of the Al_2O_3 by the B_4C during sintering, and crack deflection around B_4C grains.[4]

Investigation of the processing and properties of ternary Al_2O_3 -SiC- B_4C ceramics is scarce. Swarnakar et al. investigated a Al_2O_3 – 60vol% B_4C composite with up to 5 wt% SiC carbide additions densified by pulsed electric current sintering (PECS).[15] Flexure strength, hardness, and toughness for the Al_2O_3 – 60 vol% B_4C ceramic were reported as 595 MPa, 24.3 GPa, and $7.2 \text{ MPa}\cdot\text{m}^{1/2}$, respectively, and remained unchanged with up to 5 wt% addition of SiC. Grains of an aluminum borate phase ($\text{Al}_{18}\text{B}_4\text{O}_{33}$) were found in the microstructure following PECS, which was attributed to the thermodynamically favorable reduction of Al_2O_3 by B_4C . Previous studies into alumina based ternary ceramics have investigated additions of SiC/TiC,[16, 17] WC/TiC,[17, 18] ZrO_2 /TiC,[17] and ZrB_2 / ZrO_2 [19] while several other studies have investigated the properties of ZrO_2 - Al_2O_3 -TiC,[20] TiB_2 -TiC-SiC,[21, 22] and ZrB_2 -SiC- B_4C [23] ceramics. In each of these systems, properties of the ternary ceramics were found to be improved compared to their constituent phases. The ternary ceramics also provide the promise of improved tailoring of properties such as strength, toughness, hardness, and electrical conductivity. For example, Al_2O_3 -WC-TiC ceramics were promising ceramic tooling materials that could be shaped by electrical discharge machining.[17, 18, 24]

The purpose of the present study was to examine the densification behavior, microstructure, and mechanical properties of Al_2O_3 -SiC- B_4C ceramics.

2. Experimental

Commercially available powders were used to prepare the ceramics in this study, which contained 70 vol% Al_2O_3 , 15 vol% SiC, and 15 vol% B_4C (designated ASB). The Al_2O_3 powder (Grade A16-SG, Almatis Inc., Leetsdale, PA) had a reported purity of 99.8% and a starting particle size of 0.5 μm . The SiC powder (Grade UF-25, H. C. Starck, Newton, MA) was predominantly α -SiC with a reported purity of >97.4% and a particle size of 0.45 μm . The B_4C powder (Grade HS, H. C. Starck) had a reported purity of >96.8%, a B/C ratio of 3.8, and starting particle sizes that ranged from 0.6 to 1.2 μm . The powders were ball milled in methyl–ethyl ketone with dispersant (DISPERBYK-110, BYK- USA Inc., Wallingford, CT) at 60

rpm for 24 h in a polyethylene jar, using cobalt-bonded tungsten carbide media. Solvent was removed from the milled slurry using rotary evaporation (Rotavapor R-124, Buchi, Flawil, Germany) at a temperature of 80°C, a vacuum of ~27 kPa, and a rotation speed of 90 rpm. Dried powders were lightly ground and passed through a 50 mesh screen before densification.

Blended powders were hot pressed (HP-3060, Thermal Technology, Santa Rosa, CA) in graphite die lined with graphite foil (GRAFOIL® Grade GTA, GrafTech International Holdings, Inc., Independence, OH) and coated with BN (SP-108, Materion Advanced Materials Group, Milwaukee, WA). Above ~600°C, the temperature of the graphite die was monitored using an infrared pyrometer (OS 3708, Omega Engineering, Stamford, CT). Powder compacts were heated under flowing argon to 1200°C with an average heating rate of ~60°C/min, when a uniaxial load was applied at ~6 MPa/min to 32 MPa while continuing to heat to 1650°. Compacts were held at 1650°C for 30 min for densification, the furnace was then cooled at ~10°C/min to room temperature. The load was removed when the die temperature dropped below 1250°C. Surfaces of hot-pressed billets were surface ground to a 120 grit finish to remove residual BN and graphite foil.

Bulk densities of the hot-pressed billets were determined using Archimedes' method with distilled water as the immersing medium. Microstructures were examined using scanning electron microscopy (SEM; Helios Nano Lab 600, Hillsboro, OR). Specimens were prepared for microscopy by cutting cross sections perpendicular to the hot-pressing direction and polishing to a 0.25 µm finish using progressively finer diamond abrasives.

Hardness was measured by Vickers indentation (Duramin V, Struers, Westlake, OH) using a load of 9.8 N and a dwell time of 15 s according to ASTM C1327. Ten indents were measured to determine the reported average values. Elastic modulus was calculated from load-deflection data collected during strength testing using a deflectometer. Flexure strengths were measured in four-point bending using a semi-articulated test fixture using type A bars (25 mm × 2.0 mm × 1.5 mm) according to ASTM C1161.

Bars were machined from the hot-pressed billets by diamond grinding on a fully automated surface grinder (FSG-3A818, Chevalier, Santa Fe Springs, CA), and fine ground to final dimensions using a 600-grit diamond grinding wheel. The flexure surface was polished to a 1 μm surface finish using diamond abrasives. Tests were performed using a screw-driven instrumented load frame (Model 5881, Instron, Norwood, MA). Fracture toughness was measured according to the direct crack method of Anstis et al.[25] A Vickers indent was made using a load of 10 kg and a dwell time of 15 s. Ten indents were measured to determine the average crack length.

3. Results and Discussion

The measured bulk density was 3.636 g/cm^3 for the hot pressed composite, which was close to the theoretical density of 3.625 g/cm^3 predicted for the nominal composition. The bulk density is higher than the predicted theoretical density for a combination of reasons: (1) incorporation of Co-bonded WC ($\rho = 14.6 \text{ g}/\text{cm}^3$) impurities during ball milling of the powder; (2) loss of SiC (3.21 g/cm^3) and B_4C (2.52 g/cm^3) due to reaction with oxygen; and (3) formation of a new phase from reaction of SiC and B_4C with Al_2O_3 . The additional phases present following hot pressing were identified by XRD (Figure 1). In addition to Al_2O_3 , SiC, and B_4C , WB, WB_2 , and $\text{Al}_{18}\text{B}_4\text{O}_{33}$ were detected by XRD. WB and WB_2 are likely formed from a multistep reaction of the WC, introduced by erosion of milling media, with Al_2O_3 and B_4C . $\text{Al}_{18}\text{B}_4\text{O}_{33}$ has previously been shown to form from the reaction of Al_2O_3 with B_4C . [15] Although the relative density could not be determined because the true density of the composite was not known, bulk density measurements indicated that the composite likely had high relative density.

The microstructure of the ceramics was examined by SEM (Figure 2) and by STEM (Figure 3). Al_2O_3 was the lighter gray phase and formed a continuous matrix with grain sizes of less than about 3 μm . SiC appeared as a slightly darker gray phase and was present as isolated particles. Some of the particles were equiaxed with diameters of up to $\sim 1 \mu\text{m}$, while others were elongated with thicknesses

on the order 0.5 μm with lengths up to $\sim 3 \mu\text{m}$. B_4C appeared dark gray and was present as isolated, equiaxed grains with diameters of less than about 3 μm ; however, some B_4C clusters composed of several particles were observed. The clusters were large as $\sim 6 \mu\text{m}$. A small volume fraction of BN and WB_x were observed, mainly at the grain boundaries. BN formed elongated lamellar inclusions of less than $\sim 0.2 \mu\text{m}$ in width with lengths up to $\sim 1.5 \mu\text{m}$. The WB_x phases were present as equiaxed particles of less than $\sim 0.2 \mu\text{m}$. A small volume fraction (less than 1 vol%) of porosity was observed as small features primarily located at triple grain junctions, and as internal porosity in larger B_4C grains/clusters. In addition, a fine ($< 0.1 \mu\text{m}$), intragranular, low-Z phase was present in some Al_2O_3 grains. This feature is likely SiC similar to what has previously been reported by Sternitzke et al.[13] The $\text{Al}_{18}\text{B}_4\text{O}_{33}$ phase identified by XRD was not distinguishable by SEM, presumably due to its similar Z-contrast to Al_2O_3 ; however, indications of a liquid phase were present at the grain boundaries in the STEM images. These regions could be $\text{Al}_{18}\text{B}_4\text{O}_{33}$, which is expected to be liquid above $\sim 1450^\circ\text{C}$. [26]

The densification behavior of the composite is shown in Figure 4. The onset of densification occurred almost simultaneously with the application of the load of $\sim 5 \text{ MPa}$ at $\sim 1225^\circ\text{C}$ during hot pressing. The ceramics reached full density after $\sim 4 \text{ min}$ at the maximum hot-pressing temperature of 1650°C . The initial observed densification was likely facilitated by particle rearrangement and compaction aided by formation of a liquid borate and aluminum borate phase. [26] Intermediate and final stage sintering likely occurred through a combination of liquid phase and solid state sintering, as the liquid phase was only observed at a limited amount of grain boundary interfaces. The formation of the aluminum borate phase has been predicted to occur at temperatures as low as 1000°C by thermodynamic calculations assuming amorphous B_2O_3 (a known surface impurity on fine B_4C powders) and Al_2O_3 by Swarnakar et al. [15] Additionally, the direct reaction of B_4C with Al_2O_3 was predicted to form $\text{Al}_{18}\text{B}_4\text{O}_{33}$ at 1650°C . [15]

Young's modulus four-point flexure strength, Vickers hardness, and fracture toughness were measured (Table I). Young's modulus was 408 GPa, which is similar to reported values for Al_2O_3 ceramics (~ 410 GPa),[27] even though the value was expected to increase due to the additions of SiC (~ 415 GPa)[28] and B_4C (~ 455 GPa),[29] and from the incorporation of WC (~ 640 GPa)[30] during milling. Presence of the aluminum borate may decrease the modulus of the final ceramic due to its lower Young's modulus (400 GPa).[31] In addition, microcracking could also reduce Young's modulus. Fracture toughness was $\sim 4.5 \text{ MPa}\cdot\text{m}^{1/2}$, similar to previously reported values for Al_2O_3 - B_4C ceramics (~ 3.5 - $5.5 \text{ MPa}\cdot\text{m}^{1/2}$)[3, 4, 14, 15] and Al_2O_3 -SiC ceramics (~ 2.5 - $7.5 \text{ MPa}\cdot\text{m}^{1/2}$).[5, 11-13] Figure 5 shows a radial/median crack originating from the Vickers indent used to measure toughness. The crack exhibited some deflection when encountering SiC and B_4C particles, though no bridging was observed. The fracture mode was found to be predominantly transgranular through the SiC and B_4C grains, while exhibiting mixed mode transgranular and intergranular fracture through the Al_2O_3 phase. Vickers microhardness was ~ 21 GPa, in agreement with previous studies for Al_2O_3 -SiC (~ 15 - 23 GPa)[5, 11-13] and Al_2O_3 - B_4C (~ 15 - 21 GPa).[4] Hardness of the present material was less than reported for the B_4C - Al_2O_3 -SiC ceramic (~ 26 GPa) due to B_4C being the continuous matrix phase in Swarnakar's material, compared to the continuous Al_2O_3 matrix in the present study.[15]

Flexure strength was ~ 880 MPa, which is greater than comparable Al_2O_3 -SiC (~ 300 - 750 MPa)[5, 11-13] and Al_2O_3 - B_4C (~ 200 - 600 MPa)[3, 4, 14] ceramics, and $\sim 50\%$ greater than previously reported B_4C - Al_2O_3 -SiC (~ 490 - 600 MPa).[15] This improvement is likely due favorable residual stress states in the ceramic that formed due to the CTE mismatch between Al_2O_3 ($7.2 - 8.6 \times 10^{-6}/\text{K}$)[32] and SiC ($4.3 - 5.6 \times 10^{-6}/\text{K}$),[32] B_4C ($5.7 \times 10^{-6}/\text{K}$),[33] and $\text{Al}_{18}\text{B}_4\text{O}_{33}$ ($3 - 5 \times 10^{-6}/\text{K}$).[34] The difference in CTE between the matrix and reinforcing phases places the Al_2O_3 matrix under a tensile stress state, while the reinforcing phases experience a compressive stress state. This behavior was investigated by Ferroni and Pezzotti for the Al_2O_3 -SiC system.[10] Ferroni and Pezzotti demonstrated that additions of SiC

nanoparticles to Al_2O_3 improved the fracture strength of the composite through an increase in the amount of transgranular fracture and a favorable bulk residual stress state that strengthened the Al_2O_3 grain boundaries, with the maximum effect occurring for SiC additions up to 10 vol%.

Residual stresses in the present material were large enough to cause spontaneous grain boundary microcracking between Al_2O_3 and SiC/ B_4C grains and clusters (Figure 6). The presence of microcracking is expected to decreased hardness and modulus compared to materials without microcracking. Griffith type analysis using a Y-parameter of 1.99 (long, elliptical surface crack where $c > 2a$) results in a calculated critical flaw size of $\sim 7 \mu\text{m}$. This is similar to the size ($\sim 6\text{--}8 \mu\text{m}$) and shape (aspect ratio > 2) of the largest SiC/ B_4C clusters present in the microstructure, in addition to the microcracking that accompanies these features. Thus, it is proposed that the strength limiting feature in the present material is the SiC/ B_4C clusters and accompanying spontaneous microcracking along the SiC/ B_4C - Al_2O_3 boundaries. Strength, hardness, and modulus improvements of the Al_2O_3 -SiC- B_4C system are likely to be realized through reducing the degree of percolation of the SiC/ B_4C phase in the Al_2O_3 matrix to a level below the critical microcracking threshold.

4. Summary

The microstructure, processing, and properties was reported for a three phase ceramic consisting of 70 vol% Al_2O_3 , 15 vol% SiC, and 15 vol% B_4C that was produced from commercially available powders. The composition reached near full density by hot-pressing at 1650°C for 30 min. In addition to Al_2O_3 , SiC, and B_4C , additional phases of WB, WB_2 , and $\text{Al}_{18}\text{B}_4\text{O}_{33}$ were present following densification. $\text{Al}_{18}\text{B}_4\text{O}_{33}$ may form from the reaction between Al_2O_3 and the B_2O_3 present on the surface of the B_4C powder beginning at 1000°C , or direct reaction between Al_2O_3 and B_4C at the processing temperature. The formation of aluminum borate during hot-pressing results in liquid phase sintering for process temperatures above $\sim 1450^\circ\text{C}$. Measured flexure strength was $\sim 880 \text{ MPa}$, Vickers hardness was $\sim 21 \text{ GPa}$, and fracture toughness was $\sim 4.5 \text{ MPa}\cdot\text{m}^{1/2}$. The strength limiting flaw in the material suggested to be

SiC/B₄C clusters which were directly associated with grain boundary microcracking, resulting from CTE mismatch between the Al₂O₃ matrix and SiC/B₄C reinforcing phases. Based on these results, the Al₂O₃-SiC-B₄C system offers a promising approach to improvement in the strength of alumina based ceramics using conventional processing methods and commercially available powders.

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Table I. Physical and mechanical properties of Al₂O₃-SiC-B₄C ceramic hot-pressed at 1650°C

Property	Value
Bulk density	3.636 g/cm ³
Young's modulus	408 ± 8 GPa
Flexure strength	876 ± 103 MPa
Fracture toughness	4.5 ± 0.2 MPa·m ^½
Vickers hardness	21.2 ± 0.8 GPa

List of Figures

Figure 1. XRD pattern of the hot-pressed Al_2O_3 – SiC – B_4C composite.

Figure 2. SEM micrograph of the Al_2O_3 - SiC - B_4C ceramic. Al_2O_3 and SiC are the medium grey phase, with similar Z-contrast, B_4C is the darker grey phase, WB_x is isolated white particles. Notice the intragranular porosity present in the B_4C phase.

Figure 3. DF STEM image of Al_2O_3 - SiC - B_4C highlighting the presence of W-containing high-Z phase along grain boundaries (a) and HAADF STEM image highlighting low-Z phase intragranular to Al_2O_3 (b).

Figure 4. Relative density, furnace temperature, and pressure as a function of hot-pressing time.

Figure 5. (a) Optical micrograph of the DCM Vickers indent, and (b) SEM micrograph of a radial median crack, in the Al_2O_3 - SiC - B_4C ceramic.

Figure 6. SEM micrograph of microcracking at the grain boundary interface of Al_2O_3 and $\text{SiC}/\text{B}_4\text{C}$ grains.

Al_2O_3

SiC

Pore

B_4C

WB_x

$5\text{ }\mu\text{m}$