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Microstructure of alumina reinforced with tungsten carbide

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Carbides and nitrides reinforced alumina based ceramic composites are generally accepted as a competitive technological alternative to cemented carbide (WC-Co). The aim of this work was to investigate the effect of dispersed tungsten carbide (WC) on the microstructure and mechanical properties of alumina (Al_2O_3). Micron size alumina and tungsten carbide powders were mixed in a ball mill and uniaxially pressed at 1600°C under 20 MPa in an inert atmosphere. The hardness of WC reinforced alumina was 19 GPa and fracture toughness attained up to 7 MPa $m^{1/2}$. It was demonstrated by TEM analysis that coarse, micrometersized tungsten carbide grains were located at grain boundaries of the alumina matrix grains. Additionally, sub-micrometer tungsten carbide spheres were found inside the alumina particles. Crack deflection triggered by the tungsten carbide at the grain boundaries of the alumina matrix is supposed to increase fracture toughness whereas the presence of intergranular and intragranular hard tungsten carbide particles are responsible for the increase of the hardness values of the investigated composite materials.

1. Introduction

Ceramics are increasingly used as cutting tool materials due to their high hardness combined with excellent chemical stability and wear resistance. Cemented carbide composites such as WC-Co are still the most used material for metalworking today [1]. The major advantage of Al_2O_3-based composite materials, compared to WC-Co, is their deformation stability at high temperatures. This behavior allows faster cutting speeds of the tools and increased cutting depths, which may result in improved surface quality of the metal part. The addition of hard particles such as SiC, TiN, TiC, NbC and (W,Ti)C on the mechanical properties of alumina has been investigated in order to improve hardness, fracture strength, and fracture toughness [2–10]. The presence of the dispersed particles generally increased the hardness and fracture toughness of the alumina matrix, however, a reduced density of the sintered material was often observed [2–10]. Some works have attributed this behavior to the pinning effect caused by the presence of hard particles in the alumina matrix [11, 12]. High density values of the composite materials can rely only on pressure-assisted sintering. Conversely to the extended investigations on Ti-based dispersions, experimental data on the addition of tungsten carbide in alumina is still scant. A recent work on the mechanical properties and microstructure of Al_2O_3-WC-Co [8] indicated that alumina-WC-Co composites produced by vacuum hot pressing exhibited a high bending strength, fracture toughness and hardness. Other studies on the same system proved increased flexural strength and fracture toughness and a higher temperature differential thermal shock resistance than alumina monolith [7, 9]. The present study reports results on the mechanical properties and microstructure of a high density hot-pressed alumina reinforced with tungsten carbide.

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2. Experimental procedure

Alumina APC-2011 SG (Alcoa, Brazil), $D_{50} = 2.3\ \mu$m, surface area 1.5 m^2/g and WC powder (Wolfram Bergbau, Austria), $D_{50} = 1.0\ \mu$m were used as the starting materials. Powders mixtures containing 5, 10, 20 and 30 wt.% WC were homogenized in a planetary ball mill for 4 h and subsequently uniaxially hot-pressed at 1600°C under 20 MPa in flowing argon. The density of the sintered samples was determined using the Archimedes method. The identification of crystalline phases was carried out by X-ray diffraction (XRD 6000- Shimadzu). The fracture toughness and vickers hardness were evaluated by indentation method, as described elsewhere [13]. The length of the cracks and the diagonal impression generated by a vickers indenter were measured at a load of 20 N.

Sections of the alumina/WC specimens with a thickness of 1 mm were prepared using a diamond coated circular saw for transmission electron microscopy (TEM) investigations. Discs with a diameter of 3 mm were obtained using an ultrasonic disc cutter (Gatan, model 601). The discs were polished on one side with various diamond laps and cloths. Subsequently, the sample was turned and mechanically ground to a thickness of approximately 60 μm. Further thinning and polishing was performed using a dimple grinder (Gatan, model 656) until WC became transparent at a thickness of approximately 6 μm or less. Electron transparency was ensured using ion-beam thinning on a precise ion polishing system (PIPS, Gatan, model 691, Ar-ions) at 5 kV at an angle of 5°. The samples were investigated in a Phillips CM30 TEM operated at 300 kV for bright field imaging. EDX-spectra in the TEM were performed at 200 kV using a SiLi-detector (type 7370, ISIS 30, Link, Oxford, UK).

3. Results and discussion

The X-ray diffraction analysis confirmed the presence of alumina (α-Al_2O_3) and tungsten carbide (WC) (Table I). Peaks derived from other crystalline phases or oxidation products were not detected, according to the results observed for hot-pressed alumina composites reinforced with NbC or TiC (Table I). Contrasting cemented carbides such as alumina-WC-Co composites fabricated by vacuum hot pressing revealed the presence of Al_2O_3, WC, Co and CoW_2C (Table I) [8]. It was demonstrated, that the presence of this intermetallic phase (CoW_2C) contributed to the properties of the composite [8]. Regardless of the tungsten carbide content the hot-pressed specimens attained almost full theoretical density (TD) of 97.6%-TD to 99.7%-TD. These values were comparable with other hot-pressed alumina composite materials (Table I).

TABLE I Properties of hot-pressed alumina reinforced with refractories carbides

Reinforced material		Density (g/cm^3)	Crystalline phases
TiC	[5, 6]	98.6–99.8	Alumina, TiC
NbC	[2–4]	97.7–99.5	Alumina, NbC
WC + Co	[8]	99–99.7	Alumina, WC, Co, CoW_2C
(W,Ti) C	[9]	99.0–99.7	Alumina,TiC, WC
WC	[7]	99.6	Alumina, WC
WC	This Work	97.6–99	Alumina, WC

TABLE II Mechanical Properties of hot-pressed Alumina-WC

Material	Hardness (GPa)	Fracture toughness (MPa·m$^{1/2}$)
Alumina	12	2.9
Alumina + 5 wt.% WC	16	3.0
Alumina + 10 wt.% WC	19	7.0
Alumina + 20 wt.% WC	18	7.1
Alumina + 30 wt.% WC	17	6.9
Alumina + 6 wt.% WC [7]	–	5.1

The experimentally determined values of hardness and fracture toughness observed in this work are summarized in Table II and compared with similar hot-pressed alumina reinforced with carbides (Fig. 1). The addition of tungsten carbide improves the hardness of alumina composite from approximately 12 to 19 GPa. Values between 16 and 19 GPa were obtained for all compositions tested. The diagonal hardness impression produced by the indentation method (25 μm) is much larger as the grain size of alumina (2.79 μm). Thus, the hardness values observed in this work can be attributed to both intergranular and intragranular WC particles. These values are slightly lower as compared to others carbide systems (Fig. 1), which might be attributed to the slightly lower density (Table I). The hardness of alumina reinforced with carbides was found to depend basically on their microstructure and density. Several studies have shown that pressure-less sintering resulted in considerably lower density values and consequently decreased hardness [2, 5, 6]. Contrary to what was observed for hardness, the fracture toughness of alumina/tungsten carbide exhibited a significant increase,

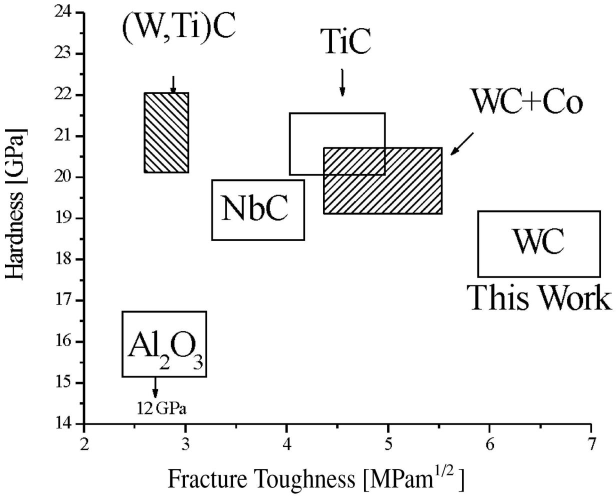


Figure 1 Mechanical properties of hot-pressed alumina composite ceramics.

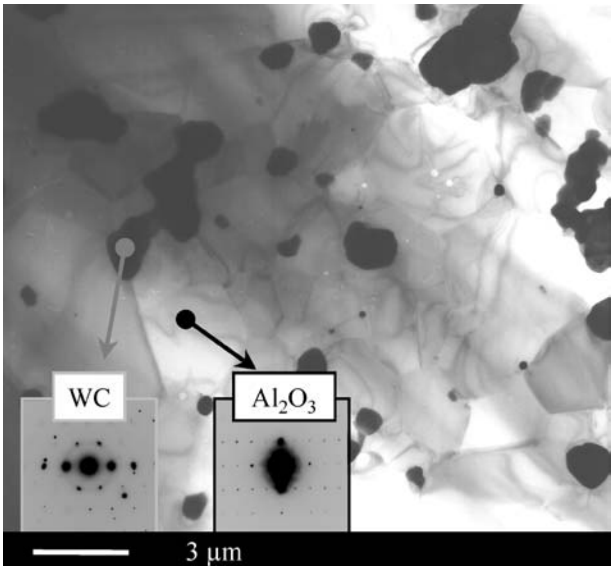
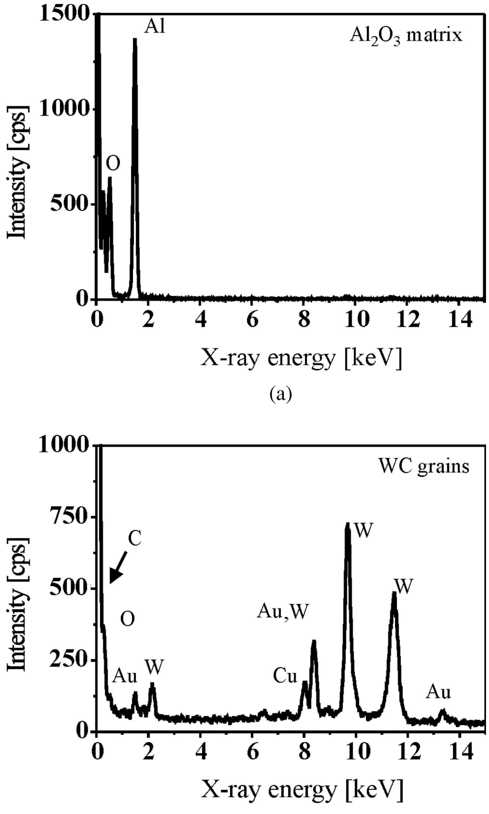


Figure 2 TEM bright field (BF) micrograph of the alumina-WC composite material; insets: selected area diffraction (SAD) of the alumina and WC.

as compared to the other composite materials (Fig. 1) and alumina matrix (Table I). The fracture toughness was nearly constant as the tungsten carbide content increased up to 10 wt.% WC (7 MPa·m$^{1/2}$). The addition of 5 wt.% WC did not cause a significant increase in the fracture toughness. This behavior is associated with the densities values of WC (15.8 g/cm^3) and Al_2O_3 (3.98 g/cm^3). The concentration of 5 wt.% WC causes only the incorporation of few WC particles that is not sufficient to improve the fracture toughness of the composite material. Similar behavior is also observed for Alumina+WC-Co [8] and also for others ceramic composite systems [2, 9].

TEM-investigation was performed to study the microstructure of alumina/tungsten carbide composite. The composite material exhibited a heterogeneous microstructure (Fig. 2). The phase composition was confirmed by selected area diffraction (SAD, insets in Fig. 2). The EDS-analysis confirmed the single phase characteristics of the components (Fig. 3). Coarse tungsten carbide grains with particle diameters between 0.7 and 2.0 μm were mainly located at the boundary between two alumina grains (intergranular WC-particles). Apart from the coarse intergranular tungsten carbide grains, submicron sized tungsten carbide grains of spherical shape were frequently observed inside the alumina (Fig. 4). The intragranular tungsten carbide grains seemed to be twinned, because sharp lines dividing areas with slightly different diffraction contrast from each other were observed within one grain (Fig. 4b). The particle size of the intragranular tungsten carbide spheres, embodied in the alumina grains (2.79 μm) did not exceed 500 nm. TEM-analysis did not show the presence of an intergranular phase between alumina and WC.

It was reported, that the presence of tungsten carbide in the composite can prevent to some extent crack propagation by deflection mechanisms, divergence, blunting,



(a)

(b)

Figure 3 EDS analysis of (a) alumina matrix and (b) WC grains; Cu- and Au-peaks derived from the specimen fixation used in the TEM.

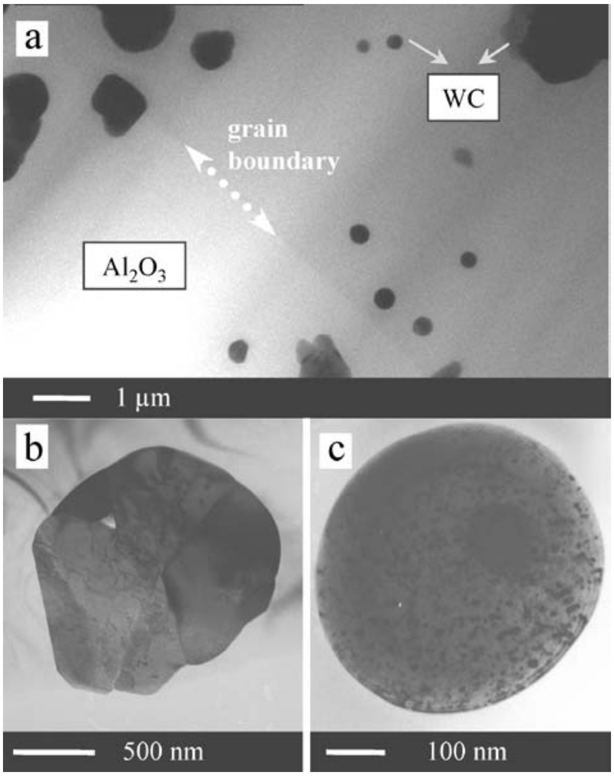


Figure 4 (a) Scanning transmission electron (STEM) image of WC particles in the alumina matrix, (b) TEM-BF micrograph of a WC particle, (c) TEM-BF micrograph of a WC sphere in alumina matrix; dotted doubled headed arrow indicates grain boundary.

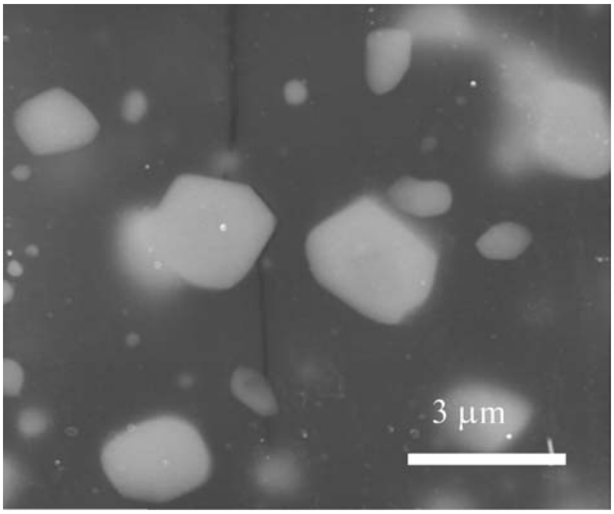


Figure 5 SEM micrograph of Al_2O_3+20 wt.% WC showing the crack deflection due the presence of WC particles.

arresting etc. [7, 9]. These mechanisms were partly related to the mismatch of the thermal expansion between tungsten carbide (CTE = 5.2–7.3 ppm/K) and alumina (CTE = 8–9 ppm/K). Crack deflection at alumina intergranular tungsten carbide interfaces is likely to appear and give rise to the observed increasing toughness. Fig. 5 illustrates the crack deflection produced by a WC particle. The same mechanism was considered to be responsible for a similar toughness improvement in alumina reinforced with WC-Co and (W, Ti)C [8, 14]. It is recognized, that the dispersion of TiC and (W, Ti)C particles in the alumina matrix can reduce the crack propagation essentially by the crack deflection mechanism [5, 6, 14]. The fracture toughness was improved by dispersion of tungsten carbide in alumina from 2.9 MPa $m^{1/2}$ to approximately 7 MPa $m^{1/2}$ by 75%. The presence of the tungsten carbide at the grain boundary indicates that large WC particles might act as pinning sites for the grain boundary movement of alumina during sintering. Similar results were also reported for alumina reinforced with (W,Ti)C [14].

Further investigations are under way to asses tribological properties and microstructure evolution upon different sintering conditions and its effect on the mechanical properties of the composite material.

4. Conclusions

Alumina/tungsten carbide composites combining good hardness with a high value of fracture toughness were fabricated by hot-pressing. The hardness values were increased by 58% for the addition of 10 wt.% of tungsten carbide, which was found to represent the optimum values for hardness and fracture toughness. Further increase of the added amount of tungsten carbide to the alumina matrix decreased the hardness as well as the fracture toughness. It was proved by TEM investigation, that the coarse micrometer sized tungsten carbide grains were located at the alumina gain boundaries, which might cause crack deflection responsible for the fracture toughness increase. The enhancement of the microhardness might be attributed to the inclusion of submicron sized tungsten carbide spheres in the alumina matrix and also to the intergranular WC particles.

Acknowledgment

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