Journal of the European Ceramics Society 7 (1991) 277–281

Fabrication and Properties of Al-infiltrated RBAO-based Composites

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(Received 23 October 1990; accepted 30 November 1990)

Abstract

The reaction-bonded Al_2O_3 (RBAO) process is applied to fabricate open porosity Al_2O_3-based composites with SiC and Al_2O_3 particulate inclusions. These are then gas-pressure infiltrated with liquid aluminum. The Al-infiltrated composites exhibit strongly improved mechanical properties, e.g. fracture toughness and bond strength of samples containing 30 vol.% 13 μm diameter Al_2O_3 platelets are enhanced 1·6 to 5·8 $MPa\sqrt{m}$ and from 85 to 760 MPa, respectively. In all cases, crack bridging by ductile Al ligaments is the main toughening mechanism. Filling of void defects, caused by particulate agglomeration, with Al is especially effective in reducing the strength-controlling flaw size.

Reaktionsbinden von Al_2O_3 (RBAO-Probzeß) wird zur Herstellung offenporiger Al_2O_3-Basisverbundwerkstoffe mit dispergierten SiC- und Al_2O_3-Partikeln verwendet. Diese Körper werden anschließend mit flüssigem Aluminium gasdruckinfiltriert. Dadurch werden die mechanischen Eigenschaften gegenüber den nichtinfiltrierten Körpern ganz erheblich verbessert. Beispielsweise steigt die Bruchzähigkeit von Proben, die 30 vol.% 13 μm große Al_2O_3-Platelets enthalten, von 1·6 auf 5·8 $MPa\sqrt{m}$ und die Festigkeit von 85 auf 760 MPa. In allen Fällen erweist sich Rißüberbrückung durch duktile Al-Ligamente als Hauptverstärkungsmechanismus. Das Ausfüllen von Hohlräumen, die durch Partikelagglomerate entstehen, mit Aluminium führt zu einer entscheidenden Reduzierung der kritischen Fillergröße.

On a élaboré des composites à porosité ouverte à base de Al_2O_3 et contenant des inclusions particulaires de SiC et Al_2O_3 par la méthode RBAO. Les matériaux étaient ensuite infiltrés par pression de gaz par de l'aluminium liquide. Les composites infiltrés par Al présentent une forte amélioration des propriétés mécaniques; dans le cas de composites contenant 30% volumiques de plaquettes de Al_2O_3 de 13 μm de diamètre, la ténacité passe de 1·6 à 5·8 $MPa\sqrt{m}$ et la résistance mécanique de 85 à 760 MPa. Dans tous les cas, le principal mécanisme responsable de l'accroissement de la ténacité est le pontage des fissures par des ligaments ductiles d'aluminium. Le remplissage par Al des pores induits par l'agglomération des particules permet de réduire de manière considerable la taille du défaut responsable de la rupture.

1 Introduction

It has been demonstrated recently that low-shrinkage reaction-bonded alumina (RBAO) can be fabricated from Al/Al_2O_3 powder compacts.[1,2] The heating cycle, Al/Al_2O_3 ratio and compaction pressure are used to control the microstructure development. Due to the low shrinkage and low temperatures associated with the processing, RBAO represents an ideal matrix for the incorporation of coarse and reactive particles, which has been demonstrated in preliminary experiments.[2] In order to test the suitability of RBAO as matrix phase, very coarse Al_2O_3 ($\sim 120\,\mu m$) and small SiC ($\sim 6\,\mu m$) particles as well as Al_2O_3 platelets ($\sim 13\,\mu m$) were incorporated. As previously shown, open porosity suitable for metal infiltration can be adjusted by a proper heating schedule.[2] In the present work, gas-pressure infiltration was used to prepare the RBAO/Al composites.[3] Their mechanical properties were investigated.

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Table 1. Mechanical properties of RBAO composites

Sample number	*Second phases in RBAO matrix*	*Bending strength (MPa)*	*Fracture toughness ($MPa\sqrt{m}$)*	*Hardness (HV 100) (GPa)*
1	SiC, Al_2O_3	$90 \pm 4{\cdot}7$	$2{\cdot}0 \pm 0{\cdot}1$	2·55
2	SiC, Al_2O_3, infiltrated	336 ± 22	$4{\cdot}7 \pm 0{\cdot}7$	8·22
3	Al_2O_3 platelets	85 ± 10	$1{\cdot}6 \pm 0{\cdot}1$	2·30
4	Al_2O_3 platelets, infiltrated	760 ± 30	$5{\cdot}8 \pm 0{\cdot}3$	5·70

2 Experimental Procedure

2.1 Preparation of porous composites

Aluminum powder (globular particles with diameter between 20 and 200 μm, >99% Al, Aldrich Chemie GmbH, FRG) and 13 μm Al_2O_3 abrasive powder (F500/13, H.C. Starck, Berlin, FRG) were used to mechanically alloy the RBAO precursor powder. A powder mixture of 40 vol.% Al and 60 vol.% Al_2O_3 with addition of 2 wt% Si, 1 wt% Mg and 0·4 wt% Zn powder (E. Merck AG, Darmstadt, FRG)† was attrition milled in an UHMVPE-lined vessel in isopropanol using Y-TZP balls (diameter 2–5 mm). After 8 h of milling, 30 vol.% of fine SiC (~5 μm, Norton Co., USA) and 20 vol.% of coarse Al_2O_3 (~120 μm, Norton Co., USA), were added in one case (see No. 1 in Table 1) and, in a second case, 30 vol.% Al_2O_3 platelets (~13 μm, Showa Aluminum, Japan) were added to the mixture (see No. 3 in Table 1). In order to homogenize these particles, milling was continued for another 30 min at reduced milling intensity. Further processing included rotary evaporator drying, sieving through 400 μm mesh, and isopressing at 400 MPa into square-sectioned plates of 36 mm × 36 mm × 6 mm. In the preparation of RBAO–SiC–Al_2O_3 composites, the samples were heat-treated in air in a box furnace with a heating cycle consisting of a heating rate of 10°C/min, a first hold at 1200°C for 10 h, a second hold at 1450°C for 4 h, and a cooling rate of 15°C/min. The density of the thus-fabricated samples was ~80% TD. The preparation of RBAO–Al_2O_3 platelet composites (No. 3) followed a similar procedure except that the heating cycle consisted of only one hold at 1200°C for 15 h. The density of these samples was approximately 76% TD.

2.2 Aluminum metal infiltration

Gas-pressure infiltration, schematically shown in Fig. 1 of Ref. 3, was used to prepare the RBAO–Al composites. The porous reaction-bonded bodies were mechanically fixed at the bottom of an alumina crucible and covered with an aluminum plate (>99·999% Al, Kryal S., VAW, FRG). The system was heated in a gas-pressure furnace in vacuum to 900°C. Then an Ar gas pressure of 8 MPa was applied for 10 min, forcing the molten aluminum to infiltrate the porous plates. On cooling, the pressure was kept up until the aluminum had solidified.

† Recently it has been shown that improved RBAO properties are obtained when no metal alloy additions are used.

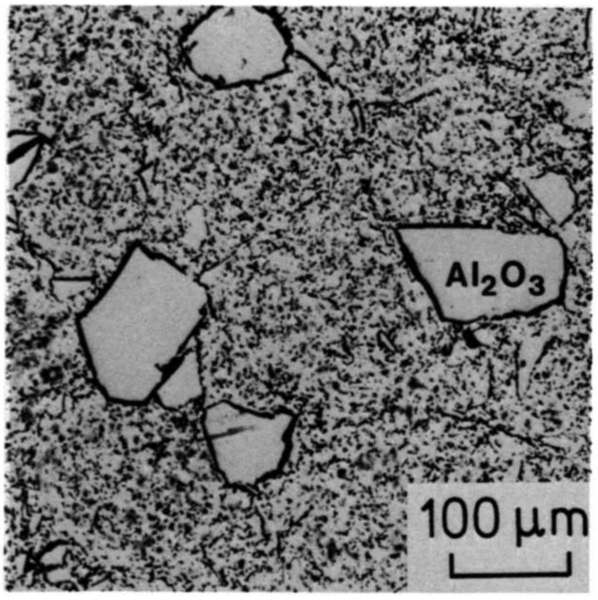
2.3 Mechanical properties

Uninfiltrated and infiltrated plates were cut into 5 mm × 5 mm × 34 mm bars. The fracture strength was measured in four-point bending with spans of 30 and 10 mm. Fracture toughness was measured by the indentation strength in bending technique. The indented samples (load 100 N) were broken in three-point bending with a 16 mm span. Tensile surfaces of the samples were polished to a 1 μm finish. The crosshead speed was 10 μm/min in all cases. Vickers microhardness was measured for all samples using a load of 100 N. Microstructure and phase content were studied by optical microscopy, SEM and XRD analyses.

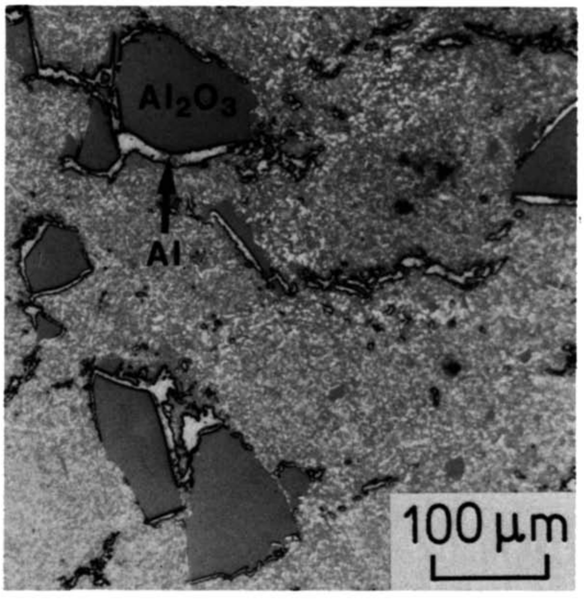
3 Results and Discussion

3.1 SiC–Al_2O_3 composites

After reaction bonding, the composites exhibit a slight expansion (~1%). XRD analyses of uninfiltrated RBAO–SiC–Al_2O_3 samples (No. 1 in Table 1) show traces of mullite ($3Al_2O_3 . 2SiO_2$) and t-ZrO_2 next to the dominant α-Al_2O_3 and SiC. The presence of t-ZrO_2 is due to wear of the ZrO_2 milling balls. No residual Al is detectable by XRD analysis. The presence of SiO_2, which reacts with Al_2O_3 to give mullite during the process, results from oxidation of Si and from surface oxidation of SiC particles. It is hypothesized that a protective mullite layer forms around the SiC particles, preventing the reaction with molten Al. The optical micrograph in Fig. 1(a) shows the microstructure of an uninfiltrated RBAO–SiC–Al_2O_3 composite. Fine SiC and coarse Al_2O_3 particles are easily distinguishable in the RBAO matrix. Figure 1(b) shows the microstruc-



(a)



(b)

Fig. 1. Optical micrographs of RBAO–SiC–Al_2O_3 and RBAO–SiC–Al_2O_3/Al composites: Gaps have formed around large Al_2O_3 particles (a) which are then pressure-infiltrated with Al (b).

ture of the same sample after Al infiltration (No. 2 in Table 1). The only additional phase in the infiltrated samples that could be detected by XRD analysis was aluminum. Aluminum has filled the open pores and especially the gap around the coarse Al_2O_3 particles. The morphology of the SiC particles was unchanged when compared to the as-received particles. This indicates that no or very little reaction has occurred with Al.

The mechanical properties of the uninfiltrated

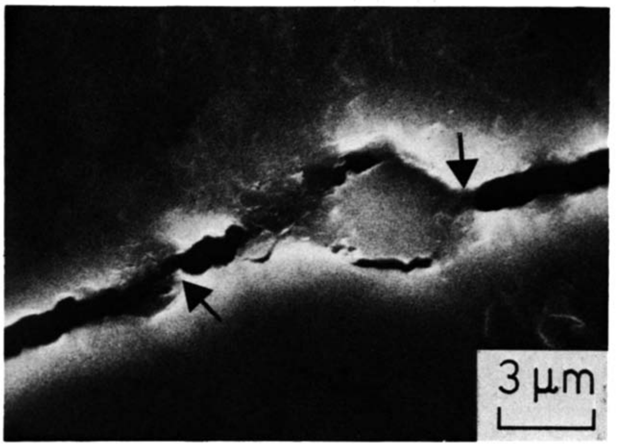


Fig. 2. Al phase bridging a crack.

and infiltrated composites are listed in Table 1. The increase in fracture toughness can be related to crack bridging of the ductile Al phase[5] as shown in Fig. 2. Al has necked down into chisel edges (see arrow in Fig. 3). The microstructural features suggest that the coarse Al_2O_3 particles have formed a skeleton and thus interfered with the sintering part of the RBAO process. Therefore, the RBAO matrix has shrunk away from the Al_2O_3 particles, leaving a gap which, on infiltration, was filled with Al (Fig. 1). For this reason, the large Al_2O_3 particles do not fully act as strength-controlling flaws.

3.2 RBAO–Al_2O_3 platelet composites

Before and after heat-treatment the RBAO–Al_2O_3 platelet composites exhibit no dimensional changes. XRD analyses of uninfiltrated composites show essentially α-Al_2O_3 and traces of t-ZrO_2. Residual Al was not detectable. The Al_2O_3 platelets are comfortably accommodated in the RBAO matrix. Due to inhomogeneities, the powder compacts contain localized Al_2O_3-platelet agglomerates.

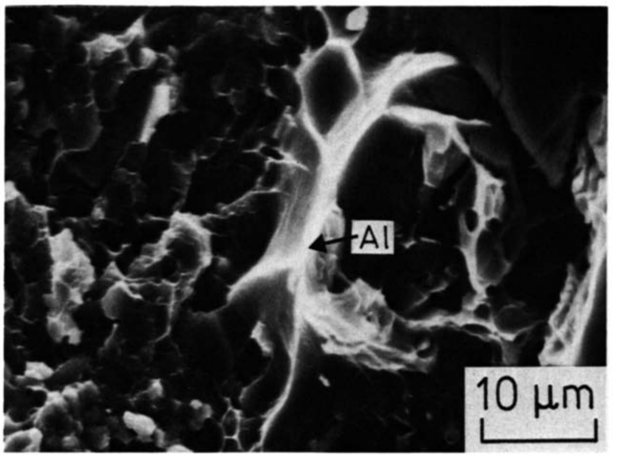


Fig. 3. Fracture surface of Al-infiltrated RBAO–SiC–Al_2O_3 composite. Al has plastically deformed to sharp edges (see arrow).

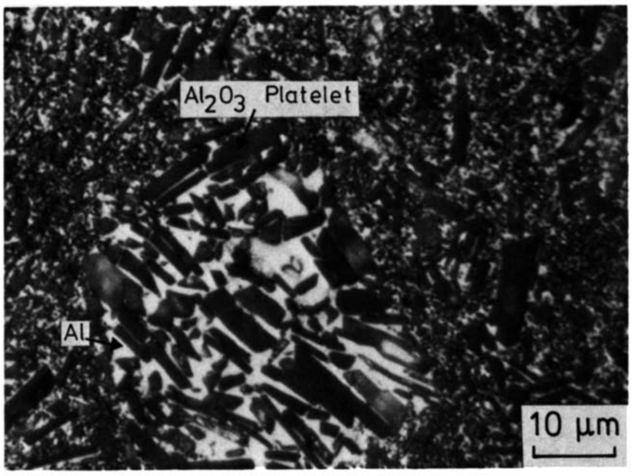


Fig. 4. Optical micrograph of an RBAO–Al_2O_3 platelet/Al composite (arrows indicate a localized Al_2O_3 platelet agglomerate filled with Al).

After infiltration, Al has filled the void spaces both in the RBAO matrix and the platelet agglomerates (see arrows in Fig. 4).

The mechanical properties of the uninfiltrated (No. 3) and infiltrated composites (No. 4) are also listed in Table 1. They are considerably improved after infiltration. The increase in fracture toughness can be explained by crack bridging of both the ductile Al phase and the Al_2O_3 platelets as well as by crack deflection along Al_2O_3 platelets. Figure 5 shows the crack propagation in an RBAO–Al_2O_3 platelet/Al composite. As in RBAO–SiC–Al_2O_3/Al composites, necking-down of Al into sharp edges is observed (Fig. 6).

4 Conclusions

4.1 RBAO–SiC–Al_2O_3/Al composites

(a) RBAO–SiC–Al_2O_3 composites exhibit near zero shrinkage.

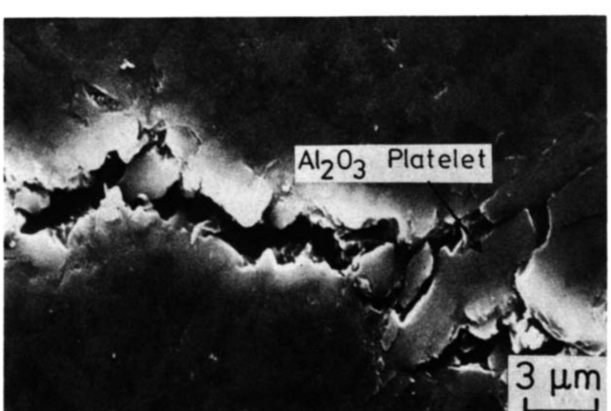


Fig. 5. Crack propagation in an RBAO–Al_2O_3 platelet/Al composite (arrow indicates an Al_2O_3 platelet bridging the crack).

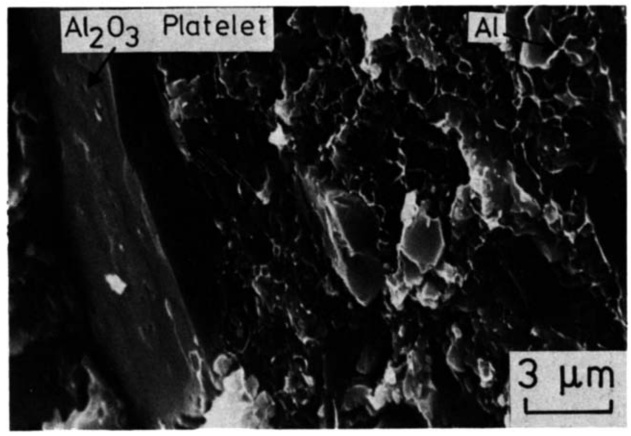


Fig. 6. Fractured surface of an RBAO–Al_2O_3 platelet/Al composite (arrows indicate chisel edges of plastically deformed Al and a platelet in the matrix).

(b) Fine SiC particles ($\sim 5\,\mu m$) are incorporated into the RBAO matrix nearly without reaction with Al.
(c) Coarse Al_2O_3 particles ($\sim 120\,\mu m$) are included without matrix cracking.
(d) Due to skeleton formation of the Al_2O_3 particles, the RBAO matrix shrinks away, leaving a gap around these particles.
(e) Pressure infiltration with molten Al fills both the void spaces and the gaps around the coarse Al_2O_3 particles which no longer act as flaws. The mechanical properties increase by about a factor of four when compared to the uninfiltrated composites, e.g. the strength increases from 90 to 336 MPa.

4.2 RBAO–Al_2O_3 platelet/Al composites

(a) RBAO–Al_2O_3 platelet composites exhibit zero shrinkage.
(b) Al_2O_3 platelets are incorporated into the RBAO matrix without strong bonding. Due to the small size of the platelets ($\sim 13\,\mu m$) shrinking away of the matrix from the platelets was not observed.
(c) Al infiltration fills the open pores in the matrix and the voids within the localized platelet agglomerates. Consequently, the platelet agglomerates do not fully act as strength-controlling flaws. Considerable improvement in mechanical properties has been achieved, e.g. strength increases from 85 to 760 MPa.

Acknowledgements

The authors wish to thank the Deutsche Forschungsgemeinschaft (DFG) for financial support under contract No. Cl 52/10 and one author (S.W.) thanks the Alexander von Humboldt Foundation for a scholarship grant.

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