Partially Stabilized Zirconia (3Y-ZrO$_2$) Aluminum Borate (Al$_{18}$B$_4$O$_{33}$) Needles Composite Materials by Direct Sintering

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Abstract:

Novel 1:1 Yttria partially stabilized zirconia (PSZ) - aluminum borate (AB: Al$_{11}$B$_4$O$_{33}$) ceramic composites were prepared and characterized by a simple direct sintering process of PSZ-AB mixtures. Sintering was performed below 1300 °C to minimize the decomposition of borate. Phases were corroborated by XRD. SEM permitted to characterize the microstructure as interlocking needles of AB - rounded by PSZ grains. Porous microstructure configuration was achieved by the proposed processing route. Porosity was within 30 % for samples thermally treated at 1200 and 1300 °C. Mechanical behavior was evaluated by diametral compression. A fragile behavior was observed. Both strength and apparent Young modulus were evaluated being ≈30 MPa and ≈3 GPa respectively; values four times higher than the corresponding alumina-AB composite. This is more important when firing at higher temperature. The density of the obtained composites coupled with the mechanical behavior are the main characteristics of this novel composite with potential structural, insulating and filtering applications.

Keywords: *Composites; Stabilized zirconia; Aluminum borate.*

1. Introduction

Composite materials have an important industrial and technological role. Usually, the designing capability of properties and behaviors is enhanced by combining the combination of two or more different materials. Namely, the properties of the composite will not always be the average between the pure material ones, rather, the properties can be considerably improved if appropriate design is performed [1].

Fine grained partially stabilized zirconia (PSZ) ceramics have been widely employed and studied for nearly four decades [2–9]. Generally, in ceramics field, both mono-phasic and multiphasic ceramics present distinctive features and have been employed in many different fields, from structural ceramics, nuclear functions, biomaterials, energy materials, etc. [1, 8, 10–12]. In zirconia, the most employed dopants are yttrium, cerium, calcium and magnesium, but other cations are also used (La, Gd, etc.). These ceramics are involved in toughening

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mechanisms [2, 6, 13], and doping proportion may be partial or full in order to achieve different ratios of monoclinic, tetragonal and/or cubic structures. 3 % Yttria PSZ has proved its merits for structural applications that involve tough ceramics, biomaterials, thermal barriers, etc. [5, 12].

On the other side, Aluminum borate (AB: $Al_{18}B_4O_{33}$) present high refractoriness up to 1300 °C, accompanied by chemical inertness in some specific environments [14–16], with adequate thermo-mechanical behavior for several applications that include alloy reinforcement phase [14].

This phase presents a theoretical density of 2.96 (g/cm^3); it is well known that thermochemical properties of ceramic materials are strongly related to the microstructure, both porosity and grain size and shape affect the material properties. The catalytic properties of AB for some technological reactions are also remarkable [17–19]. Its low density, in comparison with other similar structural ceramics such as alumina, zircon, zirconia and mullite, is one of the principal merits [16, 20].

In recent works we have reported a simple processing and sintering routes for obtaining dense and porous AB ceramics [14, 21] with alumina and boric acid as starting powders.

One way of further improving AB performance is to use it as a component in a ceramic composite. Composite materials have an important industrial and technological role. Usually, the designing capability of properties and behaviors is enhanced by the combination of two or more different materials. When appropriate design is performed, properties of the composite will not be the average between the pure components, rather, the properties can be considerably improved [1].

Fine grained PSZ ceramics have been widely employed and studied for nearly four decades [2–6]. Generally, in ceramics field, both mono-phasic and multiphasic ceramics present distinctive features and have been employed in many different fields, from structural ceramics, nuclear functions, biomaterials, energy materials, etc. [1, 10–12]. In zirconia, the most employed dopants are yttrium, cerium, calcium and magnesium, but other cations are also used (La, Gd, etc.).

Due to PSZ and AB distinctive properties, we believe that PSZ-AB composites could derive in ceramics with better or distinctive overall performance. It is important to compare the potentiality of these composites with previous studied ones. In another article we also presented the technological features of an alumina-aluminum borate composite [15]. In these, the structural application of this family of ceramics is encouraged, especially for their density, cost and needle morphology merits. These features can be designed by modulating the typical processing variables, like particle size and heating program.

The plate or needle shape morphology hints at a very high damage tolerance, this kind of materials could be suitable not only for structural application but maybe also through for 3D-printing of e.g biomedical scaffolds.

As mentioned, in a recent article a series of porous (≈ 45 %) refractory materials from the Al_2O_3-B_2O_3 system were developed [14]. The processing strategy resulted in materials with AB: $Al_{18}B_4O_{33}$ as the main phase accompanied by alumina or as unique crystalline phase. Needle grains with diameters between 0.2 and 1 μm and an aspect ratio over 20:1 were obtained. The developed monoliths obtained by direct reaction sintering of boric acid and calcined alumina presented almost 50 % of porosity.

The mechanical strength, stiffness and the overall mechanical behavior of materials ought to be described if the application of a selected material is proposed. This is the case of aluminate needle borate based monolithic porous materials, especially for structural applications [14, 15, 21].

AB forms at 800 °C and develops its distinctive needle morphology up to 1300 °C. Moreover, it is worth pointing out that the sintering process starts at 1000 °C but is restricted by the AB partial decomposition at higher temperatures (above 1300 °C).

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Several works had been carried out recently studying the incorporation of boron oxide containing compounds in refractory and structural materials, like alumina, magnesia and mullite based castables [22–24]. The boron oxide containing compounds were proposed as effective ceramic mineralizers during thermal treatments of the refractory castable achieving the in situ formation of new crystalline phase with mechanical behavior enhancements, thus showing the potentiality of boron containing materials in the refractory industry.

The principal objective of the present article is to obtain PSZ - AB composites 1:1 (volume basis) by a simple direct sintering from inexpensive starting powders, and to establish the effect of the key processing variables on the material properties. This will also enlighten design processing materials strategies.

2. Materials and Experimental Procedures

Calcined alumina (alumina A2G, ALCOA), boric acid (Borax Argentina SA) and 3 %-Yttria stabilized zirconia (TZ-3Y; Tosoh, Japan) were employed as starting powders. The properties of the these materials can be found elsewhere [4, 14].
 The aluminum borate (AB; $Al_{18}B_4O_{33}$) preparation method has been previously reported [14] therefore only a brief summary is described. A stoichiometric alumina (Al_2O_3) - boric acid (H_3BO_3) mixture was co-milled in ethanol in a ball mill for 12 hours, dried and fired at 1100 °C. This temperature was chosen in order to have completely reacted borate but small grain or needle development [11].

AB was grounded in a hand mill, and later mixed with the stabilized zirconia powder (1:1 volume ratio) using ethanol as liquid media. Intimate mixture and co-milling of this powders was performed in an alumina ball mill with alumina milling elements for 12 hours [4].

Disc shaped compacts (10.0 and 4.0 mm diameter and height respectively) were prepared by pressing the dried and de-agglomerated powder in a die. Two samples were prepared by thermal treatment of these compacts using an electric furnace, with a 5 °C/min heating rate in air atmosphere up to 1200 and 1300 °C for 120 min. Samples obtained this way were labeled Z-AB 1200 and Z-AB 1300 respectively.

2.1. Characterizations

Apparent density (D) and open porosity (P) of the sintered samples were evaluated by the Archimedes method. Crystalline phases of the sintered samples were determined by X-ray diffraction (XRD) using Cu:Kα radiation operating at 40 kV and 30 mA, and quantified by the Rietveld-method using the Fullprof commercial program [4,25]. The microstructure analysis was done by observing the gold sputtered (approximately 50 nm) free fracture surface of the sintered composited using a scanning electron microscopy (SEM, JEOL; JCM-6000).

The materials mechanical behavior was evaluated by diametral compression, also known as Brazilian test. It is a mechanical test where the compression force is applied diametrically on disc-shaped samples.

For each composite evaluated in this work, 8 discs of 10 mm diameter (D) and 4 mm thick (T) where diametrically compressed between steel plates, at a constant strain rate of 0.1 mm/min using an INSTRON 5985, EE. UU. Universal mechanical machine test equipped with a 5 KN cell.

From the experimental results obtained as a load (N) vs displacement (d in mm), a mechanical resistance (σ_d) vs displacement (ε) was built by using the following equations:

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$$\sigma_d = \frac{2L}{\pi D t} \qquad (1)$$

$$\varepsilon = \frac{\Delta d}{D} \qquad (2)$$

where *L* is the load, *D* is the initial diameter, *t* is the thickness of the disc-shaped sample and Δd is the universal testing machine displacement.

The maximum mechanical strength (σ_d^{max}) was calculated using the strength just before the sample collapsed (at peak load). Apparent Young modulus was obtained by calculating the slope of the final portion (last 25 %) of the curve before sudden breakage.

3. Results and Discussion
3.1. Textural parameters

Fine Zirconia powders started sintering above 1100 °C [4, 26], the AB powders sinters at a similar temperature, as studied elsewhere [21]. After this, two simple heating programs up to 1200 and 1300 °C were studied. Full densification was not achieved however sintering was advanced, sample diameters shrinkage was 5.1 and 6.6 % respectively. The achieved densities are low if compared with other structural materials [3]. As expected, higher thermal treatment (1300 °C) temperatures caused a decrease in open porosity and an increase in apparent density (Table I).

Tab. I Textural properties of the studied composites.

Sample	Sintering temperature	Shrinkage $\Delta D/D_0$	Apparent Density	standard deviation	Porosity	standard deviation
	(°C)	(%)	(g/cm³)		(%)	
Z-AB 1200	1200	5.1	3.017	0.042	30.9	1.8
Z-AB 1300	1300	6.6	3.238	0.042	26.3	0.5

3.2. XRD of the sintered composites

Diffraction patterns of the studied materials are shown in Fig. 1. Table II presents the Rietveld based quantification results and the PDF codes of the identified crystalline phases. The main crystalline phases are monoclinic (m-ZrO_2) and tetragonal zirconia (t-ZrO_2) and aluminum borate; these are accompanied by alfa-alumina. The presence of alumina inferred the possible presence of boron oxide, consequence of the partial AB dissociation. Although this was not observed, orthoboric acid was identified and refined.

Estimated standard deviations are also showed in Table I, these were calculated by Fullprof and derived from the estimated standard deviations in individual scale factors for the respective phases, excluding other error contributions. In all the cases the obtained Rietveld refinement profile residuals (Rwp) were satisfactory for the experimental conditions used (below 20). The quantified ZrO_2: AB ratio corresponds to the 1:1 starting volumetric proportion, within the possible uncertainties of the technique. Slight partial dissociation of AB was observed to be more important in Z-AB 1300 sample compared to Z-AB 1200. The m:t zirconia ratio was also affected by the heating program; the m amount is higher (1:3.7) for the samples fired at 1200 °C if compared with the sample fired at 1300 °C (1:5.4).
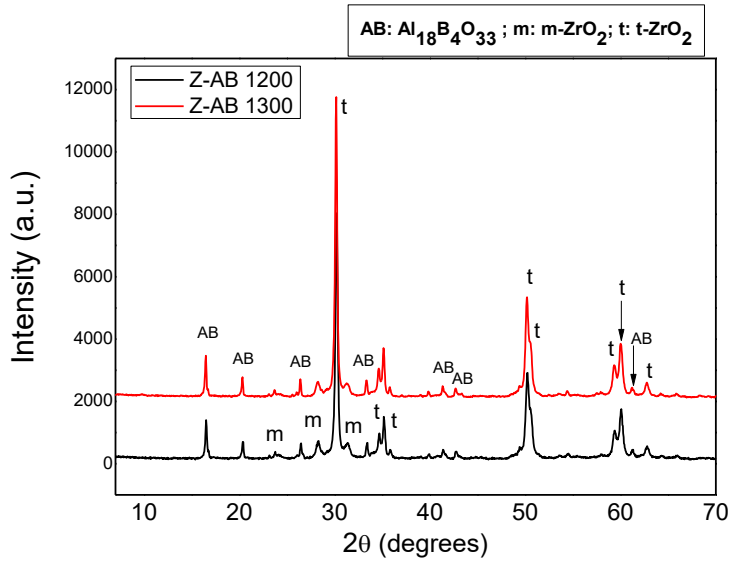
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Fig. 1. XRD patterns of the studied composites (only main phases are labeled).

Tab. II Results of the Rietveld based phase quantification.

| Phase	Formula	PDF codes	Content (%vol.)			
			Z-AB 1200	s.d	Z-AB 1300	s.d
monoclinic ZrO$_2$	m-ZrO$_2$	00-037-1484	9.03	0.03	6.46	0.09
tetragonal ZrO$_2$	t-ZrO$_2$	01-078-1808	32.96	0.05	35.17	0.10
aluminum borate	Al$_{11}$B$_4$O$_{33}$	01-080-2300	51.54	0.14	48.60	0.27
α-alumina	Al$_2$O$_3$	01-089-7716	0.95	0.05	3.39	0.10
orthoboric acid	H$_3$BO$_3$	01-078-0470	5.51	0.14	6.39	0.28

3.3. Microstructure characterization of the developed composites

Free fracture surface of the materials was analyzed by SEM, and the images obtained are depicted in Fig. 2 and 3. These images confirm a non-densified microstructure, as described before by the Archimedes method results. Further microstructural characterization of these samples was also done, assessing the presence of interlocked needles with rounded zirconia morphology in both developed materials. Aspect ratio of the needles was around 10:1 and ZrO$_2$ grain diameters are 1 micron in both cases.

Measured porosity is ascribed mainly to the space present between needles and the rounded zirconia grains. The needle thickness was relatively homogeneous for both materials, being this morphology consistent with what was previously reported for aluminum borates [16, 20, 27–30] and with the presence of borate observed by XRD. No important difference can be observed between the studied samples. Alumina particles could not be observed, probably due to the low amount present (Table II). Borate needles seem to be randomly oriented, presenting angled and rectangular sections with a tendency to overlap.

The observed fracture surface resulted from diametral compression test (described in the next section). No cracks were observed in the needles, showing the high mechanical properties of the individual grains. Presumably, the interlocked needle microstructure configuration leaded to good mechanical behavior, and, coupled with their high porosity (≈50 %), these results are encouraging for the potential filtering application of these materials. As

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both PSZ zirconia and AB have low thermal conductivity (4-6 and 2-4 $Wm^{-1}K^{-1}$ respectively) [12], these materials also show promising features for thermal insulating applications. Finally, the microstructures have a high specific area and might be employed for heterogeneous catalysis and adsorption.
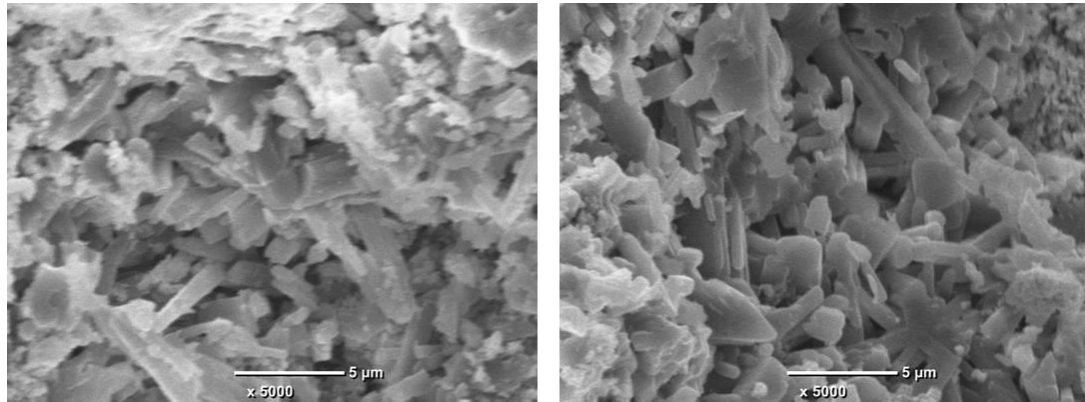
Fig. 2. SEM Images of the fracture surface of the Z-AB1200 sample.
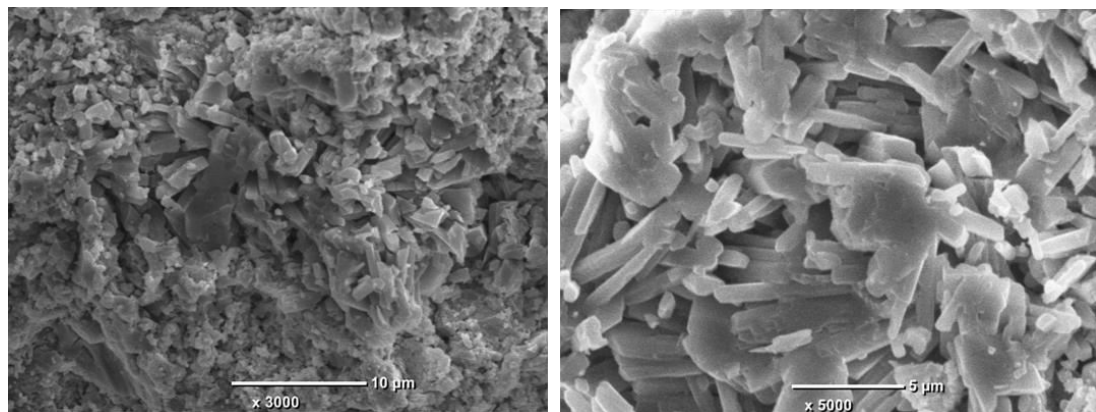
Fig. 3. SEM Images of the fracture surface of the Z-AB1300 sample.

3.4. Mechanical behavior of the developed composites

Presumably, properties of pure Y-TZP would be superior than the ones of the composite material developed, and probably to any other Z-AB Composite. This work seeks to address the compatibility of ZrO_2 and $Al_{18}B_4O_{33}$ for the future design of composite materials with different chemical environments. Perhaps, in certain applications the introduced properties of the $Al_{18}B_4O_{33}$ phase could be interest. For example, lower density, superior resistance to Boron environments, or even catalytic $Al_{18}B_4O_{33}$ properties.

Typical stress–strain curves for Z-AB 1200 and 1300 materials are shown in Fig. 4. Values of mechanical strength and apparent Young modulus are shown in Table III.
Both materials presented a similar behavior, which is expected taking into account that the microstructures and phase compositions were similar. Mechanical strength and apparent Young modulus obtained in these samples are higher than the corresponding alumina aluminum borate composite (A-AB) [11-12], with standard deviations within the typical values for ceramic materials. The achieved values corroborate the influence of the interlocked-needles microstructure observed by SEM, and encourages the structural application of the obtained composites. It's important to remark that the porosity of those A-

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AB materials sintered at 1200 and 1300 °C was 45.4 and 39.3 % [11-12], which is higher than the porosity of the composites sintered at the same temperatures in these work (30.9 and 26.3 % respectively) (see Table I).

However, the increase in both mechanical properties studied (for Z-AB materials) when compared to the A-AB materials is more than four times, which implies a better mechanical performance of the solid matrix and the influence of zirconia when compared to alumina in the preparation of aluminum borate composites.
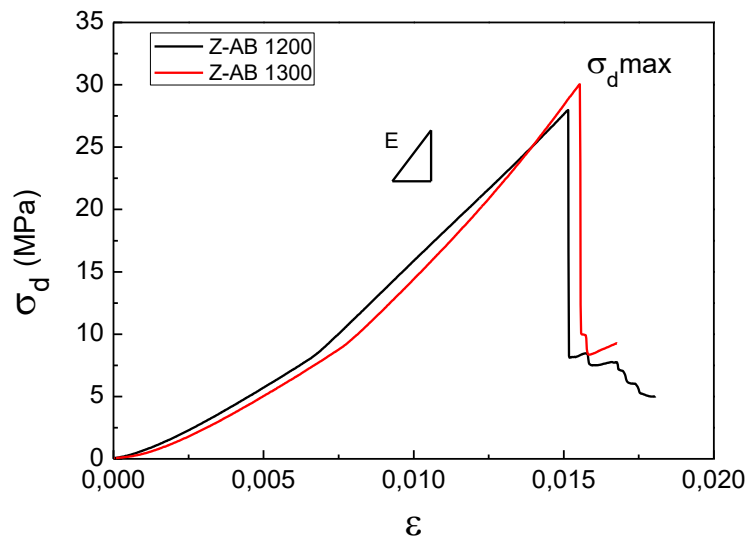

Fig. 4. Diametral compression curves of the studied materials (examples).

Tab. III Mechanical properties of the studied materials.

Sample	$\sigma_d^{\ max}$(MPa)	Standard deviation	E (MPa)	Standard deviation
Z-AB 1200	27.8	1.7	2230	350
Z-AB 1300	30.9	5.8	2160	220

The initial region of the stress–strain curves (see Fig. 4) showed a nonlinear segment that could be related to several factors: arrangement of the specimen in the load system, load distribution in the load contact region, and/or elastic strains of the load system [15, 31]. This initial ε region was below 0.01 for the obtained composites.

For both materials, after this first region, an almost linear response was observed up to the sudden fall of the load due to the brittle behavior in which these specimens failed. Therefore, porous interlocking AB needles-rounded zirconia grains composite microstructure presents a brittle behavior, similar to zirconia ceramics [12] and alumina-aluminum borate needles porous composite. This mechanical behavior is different from that of a pure interlocking AB needles material [15].

4. Conclusion

Novel ceramic composite materials were processed by a simple powder co milling route followed by conventional direct sintering; textural, micro-structural and mechanical behavior was analyzed. Commercial partially stabilized zirconia and previously prepared

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aluminum borate powders [14] were the only starting powders. Heating programs between 1200 and 1300 °C proved to be adequate for sintering.

The developed microstructure consists of aluminum borate (AB) interlocked needles with rounded PSZ grains. The observed porosity (30 %) corresponds to the space between needles and the rounded zirconia grains. A well-defined fragile behavior was observed, with mechanical properties that encourages the structural application of the developed composite materials. 30 MPa was achieved in a diametral compression test, being this result considerably higher than the corresponding alumina-borate composite. The results obtained allow the design of structural AB-PSZ ceramic materials with adequate mechanical behavior. Further microstructure design can be performed after this study; both heating programs and phases proportion can be explored for properties optimization; moreover, porogen additives could incorporate as well furthermore complex microstructure design.

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Сажетак: *Нови керамички композит итријум делимично стабилисан цирконијум (PSZ) – алуминијум борид (AB: Al$_{11}$B$_4$O$_{33}$) у односу 1:1 је припремљен директним синтеровањем смеше PSZ-AB и окарактерисан. Синтеровање је изведено испод*

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1300 °C да би се минимизирало разлагање борида. Фазни састав је одређен XRD. SEM - ом је одређена микроструктура као иглице AB - окружене PSZ зрнима. Порозна микроструктура је постигнута предложеним поступком синтезе. Порозност износу око 30 % за узорке синтеровање на 1200 и 1300 °C. Механичка својства су одређена дијаметралном компресијом. Примећена је крхкост узорака. Снага и Јунгов модул су износили ≈30 MPa и ≈3 GPa истим редом; вредности су 4 пута више од одговарајућег алумина-AB композита. Ово је јако важно када је температура синтеровања већа. Густина добијеног композита са механичким својствима су главна карактеристика овог новог композита са потенцијалном структурном, изоторском и филтерским применама.
Кључне речи*: композити, стабилисан цирконијум, алуминијум борид.*