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Elaboration and characterization of multiphase composites obtained by reaction sintering of boehmite and zircon

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Abstract

In this work, different composites (zircon–mullite–zirconia, mullite–zircon–zirconia, mullite–zirconia, mullite–zirconia–alumina and alumina–mullite–zirconia) were developed by reaction sintering of boehmite (AlOOH) and zircon (ZrSiO_4). Several mixtures were used by varying the boehmite content from 10 to 90 wt. %. All powders were mixed and grounded by ball milling and then pressed in cylindrical form. Finally, the green specimens were sintered under normal conditions for 2 hours at temperatures between 1400 and 1600°C, with a heating rate of 5°C/min. The dilatometric curves reveal several microstructural transformations in these mixtures. The X-rays diffraction spectra revealed factors such as percentage of boehmite and zircon and the sintering temperature lead to the formation of several composites. The presence of the various phases was confirmed by micrographic observations.

Key words: Multiphase composites, Reaction sintering, Development, Zircon, Alumina, Mullite, Zirconia.

INTRODUCTION

Nowadays, multiphase composites ceramic-ceramic have an important industrial and technological role [1]. The Al_2O_3 - SiO_2 - ZrO_2 equilibrium diagram developed by Budnikov and Litvakovskii [2], suggests the existence of ternary and binary (based on mullite and zircon) compounds.

Mullite continues to have a significant role in the development of traditional and advanced ceramics [3-4]. As it exhibits good mechanical behavior at high temperatures [5]. Zircon is an abundant and economical ceramic that has a good chemical stability at both ambient and at high temperatures up to 1667°C before dissociating into zirconia and silica [6]. This stability gives the material a good corrosion resistance. Its low expansion coefficient makes it extremely resistant to thermal shock [8]. However, it has a low creep resistance that limits its applications [6].

Alumina, on the other hand, is one of the most widely used structural ceramics [7]. It has many outstanding performances, including high strength, high hardness, good corrosion and high temperature resistance, thermal insulation, etc.

Multicomposites ceramics, based on zircon, alumina, mullite and zirconia, can be obtained by different processes [8]. They exhibit excellent corrosion and thermal shock resistances and enhanced mechanical properties [9].

Reaction sintering of alumina and zircon is an economic and relatively easy processing route for obtaining homogeneous composites ceramics (zircon–mullite, zircon–mullite–zirconia, mullite–zircon–zirconia, mullite–zirconia, mullite–zirconia–alumina and alumina–mullite–zirconia) to toughen the zircon, mullite and alumina matrix [10-14]. However, the reaction sintering is difficult to control because of the occurring chemical reactions (dissociation of zircon, formation of mullite and densification at various temperatures) [6].

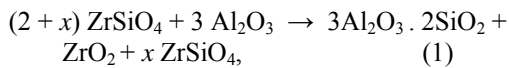
In previous work [15], the crystallization kinetics of mullite formation in boehmite and zircon were investigated using a nonisothermal method. In the present work, composites with dispersed phases from boehmite and zircon powders were developed by reaction sintering. Several mixtures were used by varying the proportion of boehmite from 10 to 90% (wt. %) with a step of 10. Several physical characterizations (shrinkage behavior, X-ray

diffraction and density measurements) were undertaken. Microstructures observations using scanning electron microscopy were carried out for the different obtained composites.

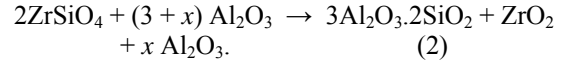
METHODS AND PROCEDURES

The starting powders used in this study are boehmite (AlOOH) and zircon microparticles. Boehmite is marketed by the Algerian company (Diprochim). Its morphology presents a regular distribution where the largest agglomerates are formed by coarse grains. The average particle size of this powder is about 70 μm (Fig. 1). The zircon chosen for this study is provided by Moulin des Près (France Company). The morphology of this powder is irregular with an average particle size of 1.5 μm (Fig. 2).

Table 1 gives the starting powders chemical compositions. The boehmite powder was milled by attrition with alumina balls in aqueous media for 3 h to reduce its average particle size (d_{50}) to 1.5 μm . Milling was carried out under the following conditions: the powder to ball ratio was kept to 1:10 by weight and we added approximately 0.25 wt. % of ammonium polymethacrylate (Darvan C) as a dispersant and ammoniac was introduced to adjust the pH of the suspensions to 10.4. Eight compositions were developed, while varying the percentage of the boehmite from 10 to 90 wt. % at 10% increments. The reaction between α -alumina and zircon at high temperature (around 1500°C) can be described according to both equations and the following molar proportions [15]:



If zircon is in excess and



If alumina is in excess.

Each mixture was homogenized by ball milling for a duration of 20 hours under the same preceding conditions. After milling, the mixtures were dried at 110°C and 1 wt % polyvinyl alcohol (PVA) with 0.5 wt. % polyethylene glycol (PEG) were added as binder by mortar mixing. Then the mixtures were granulated using various sieves (200, 150, 100 and 45 μm) to eliminate any coarse contaminations and agglomerated particles.

The samples were uniaxially pressed at 7 MPa and isostatically cold pressed at 250 MPa. This allows the formation of disks with diameter of 15 mm and different thicknesses ranged from 8 to 10 mm. The isopressed samples were heated up to 600°C at a rate of 1°C/min to burn out the binder. In order to determine an optimum preliminary sintering temperature, the compacts were sintered under atmospheric conditions at different temperatures within the range 1400-1600°C with a step of 50°C for 2 h of soaking. The heating rate was kept constant and equal to 5°C/min.

The linear thermal expansion characterization was performed on cylindrical green rods using SETARAM TMA 92 dilatometer up to 1600°C with 5°C/min heating rate. The phases present in the heated powders and their transformations were identified by X-ray diffraction using a RIGAKU diffractometer. The tests conditions were Ni-filtered $\text{Cu}\alpha$ X radiation (40kV-25mA) with a scanning speed of 2° (2 θ) per minute and at an increment of 0.05°. The morphology of powders and sintered samples were characterized by means of a JEOL 840 A. scanning electron microscope (SEM). The bulk density and open porosity of the sintered samples were determined by the Archimedes method using distilled water.

Table 1: Chemical compositions of the starting powders (wt.%)

Elements	Boehmite	Zircon
Al ₂ O ₃	88.34	-
ZrO ₂	-	63.50
SiO ₂	3.86	35.50
F	0.897	-
Na ₂ O	0.34	-
CaO ₂	0.18	-
Fe ₂ O ₃	0.096	0.08
Ti ₂ O	0.08	0.10
K ₂ O	0.07	-
HfO ₂	-	1.50
Structural water	0.6	-

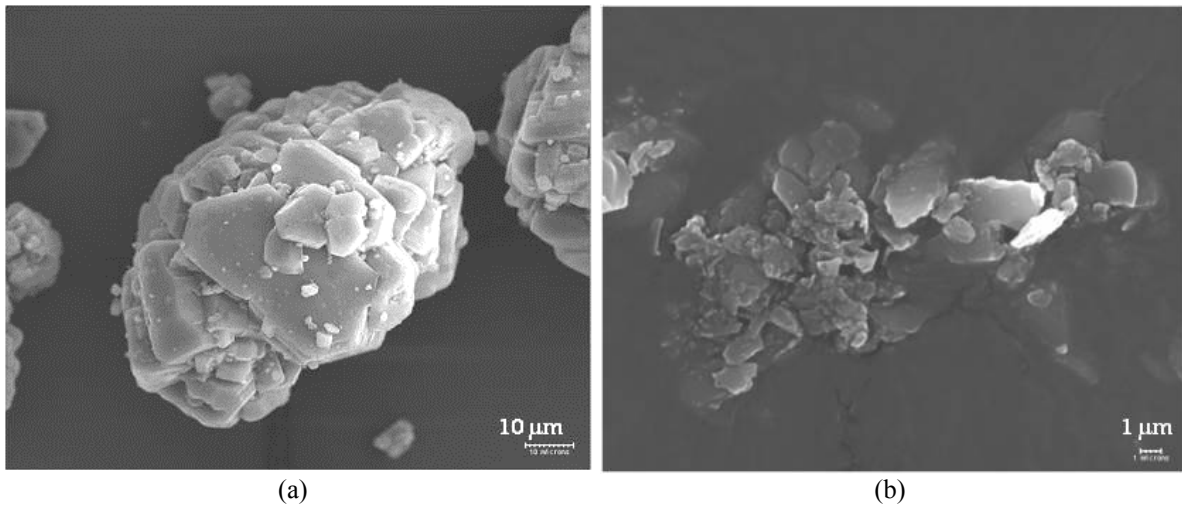


Fig. 1: Morphology of the raw (a) and milled boehmite powder (b)

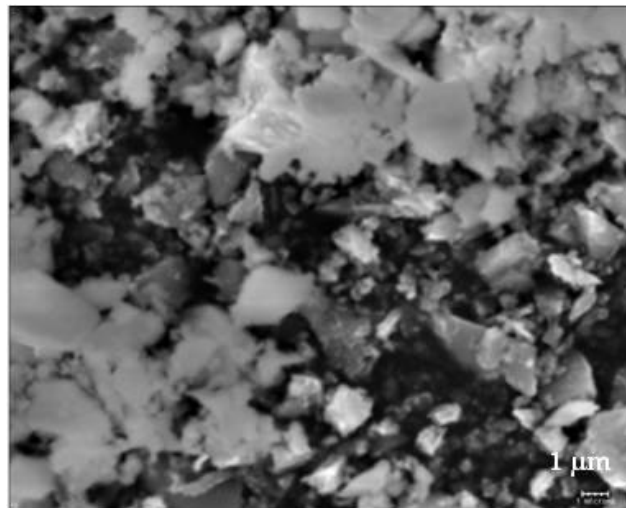


Fig. 2: Morphology of the zircon powder.

RESULTS AND DISCUSSION

3.1 Shrinkage study

During the heating of boehmite and zircon mixtures, two phenomena occur simultaneously: the reaction between silica coming from the dissociation of zircon and alumina resulting from the boehmite transformation. At high temperatures (up to 1600°C) the shrinkage curves (Fig. 3) show different evolutions:

At 1050°C, a beginning of linear shrinkage is observed; it is attributed to a necking formation between the particles [16-17]. An important shrinkage is observed from 1200°C which is related to the sintering and densification processes. An expansion was observed in all curves between 1300 and 1350°C that are caused by the phase transformation of a residual quantity of the transitory alumina phase θ -Al₂O₃ into α -Al₂O₃ [17]. The starting and final temperatures of this expansion are displaced towards higher temperatures when the zircon content increases (Table 2). However, for weaker zircon contents, the shrinkage is dominated by α -Al₂O₃.

The temperature at which the phenomenon of expansion occurs differs slightly according to the initial boehmite percentage: it is lower than 1345°C for high content of boehmite; it is equal to 1345°C for 50wt. % of boehmite and higher than 1345°C for the mixtures whose boehmite content is very weak. We quantified this phenomenon by expressing the percentage of expansion with the starting and final temperatures. The results obtained are gathered in Table 2. We observed a clear growth of expansion as the boehmite percentage increases; it reaches a maximum value (1.54 %) for the samples containing 70wt. % of boehmite. For lower percentages of boehmite, the expansion decreases from 1.35 % to 0.28 % with a decrease of boehmite from 60 to 30wt. %. This expansion is related probably to the zircon decomposition in t-ZrO₂ and silica (vitreous phase) caused by the transformation of a residual quantity of θ -Al₂O₃ toward α -Al₂O₃ [17]. In the case of the samples containing 70wt. % of boehmite, a large quantity of the θ -Al₂O₃ is present. Therefore, the number of contacts between zircon and θ -Al₂O₃ increases.

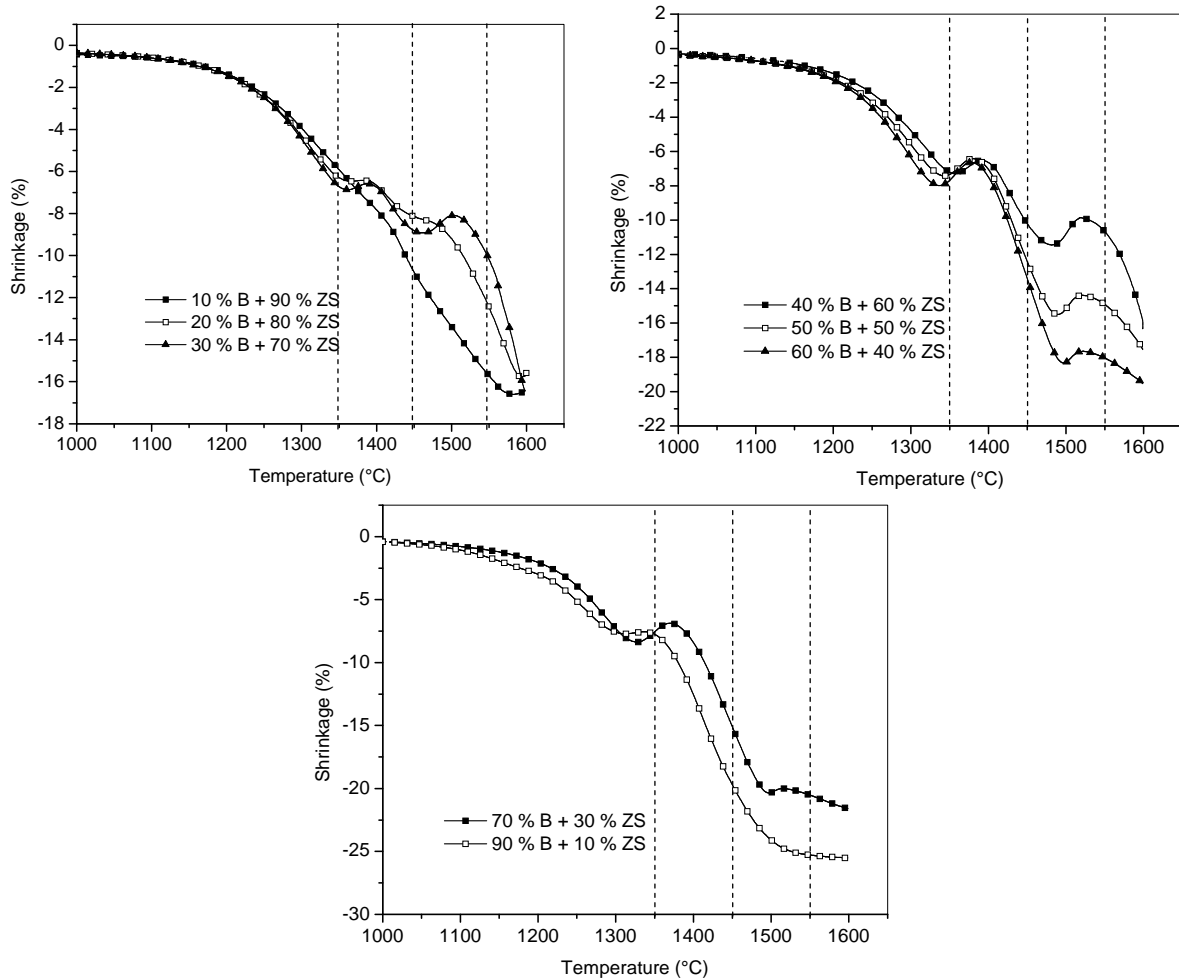


Fig. 3: Shrinkage curves of bohemite-zircon mixtures during sintering.

The shrinkage of the samples again commences notably at high temperature as a consequence of the sintering mechanism (elimination of open porosity). Silica formed by the dissociation of zircon, reacts with $\alpha\text{-Al}_2\text{O}_3$ at 1500°C to form mullite (3.17 g/cm^3) in the samples containing between 30 and 70wt. % of bohemite. That explains the rapid shrinkage deceleration starting from 1500°C , also observed by Claussen et al. [18]. The shrinkage starts over again increasing considerably with higher temperature. This is related to the elimination of open porosity (sintering mechanism). This shrinkage is more notable as the percentage of bohemite decreases up to 30wt. %. For the samples containing 10 and 90wt. % of bohemite, we did not notice any expansion.

Another perturbing expansion is observed on the dilatometric curves in all samples between 1450 and 1500°C as a consequence of simultaneous decomposition of zircon in zirconia and silica, and mullite formation. The shrinkage starts afterwards at 1440°C for the sample containing 50wt. % of bohemite. It starts at higher temperature for high bohemite content and lower temperature for very weak content. The shrinkage is also apparent in the samples containing between 30 and 70wt. % of bohemite.

Phase's identification by XRD: Zircon-mullite composites

X-rays diffraction patterns obtained from the samples heated at different temperatures are presented in Figures 4 to 11. The XRD Spectra for the samples containing 10wt. % bohemite + 90wt. % zircon (Fig. 4) shows the crystalline phases of zircon and mullite. We can notice that the intensity of the peaks corresponding to mullite is constant, according to the temperature treatment. Consequently, the formation of the composites zircon-mullite (ZSM) is starting at 1400°C . The presence of the impurities in the zircon powder (1.5% HfO_2 ; 0.10% TiO_2 and 0.08 Fe_2O_3) is probably the cause of the reduction of the dissociation temperature of zircon. This behavior was also mentioned by other authors [11, 18]. The formation of this mullite, resulting from the reaction between alumina and silica, comes from zircon dissociation. It confirms the presence of a vitreous phase which is in equilibrium with zircon. This glassy phase is formed at a corresponding invariant point (around 1450°C). The alumina present in the mixture is dissolved in this glassy phase. Mullite is formed from this glassy phase by precipitation during cooling [10]. For small additions of bohemite (10wt. %), all spectra do not reveal the presence of zirconia (t, m).

Table 2: Expansion characteristics observed between 1300 and 1400°C, deduced from the shrinkage curves

Samples	% of Expansion	Start temperature of expansion (°C)	Finish temperature of expansion (°C)	Difference of temperature (°C)
90% B + 10% ZS	0,14	1315	1340	25
70% B + 30% ZS	1,54	1330	1370	40
60% B + 40% ZS	1.35	1336	1376	40
50% B + 50% ZS	0,97	1345	1380	35
40% B + 60% ZS	0.77	1358	1390	32
30% B + 70% ZS	0,28	1360	1390	30
20% B + 80% ZS	0.02	1361	1386	25
10% B + 90% ZS	-	1380	1405	25

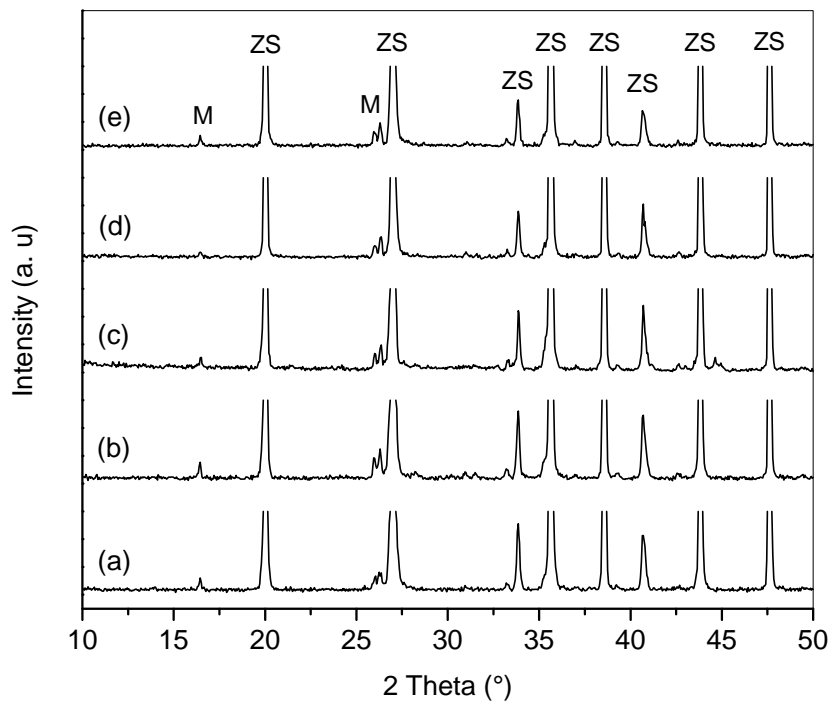


Fig. 4: XRD patterns for the 10% bohemite + 90% zircon mixture sintered at (a) 1400, (b) 1450, (c) 1500, (d) 1550 and (e) 1600°C for 2 h (ZS: Zircon, M: Mullite).

The absence of zirconia is justified by the presence of 3.86wt. % SiO_2 impurities in the starting bohemite.

Zircon-mullite-zirconia composites

The X-rays diffraction patterns recorded from the samples containing 20% bohemite + 80% zircon are represented in Fig. 5. We note the formation of the composites zircon-mullite-zirconia at 1450°C. In samples prepared with 30% bohemite + 70% zircon (Fig. 6), there is a clear modification of the formed phases. We note the presence of four phases at 1400°C: α -alumina, zircon, mullite and monoclinic zirconia ($m\text{-ZrO}_2$). Above 1450°C, the intensity of the mullite and zirconia (m) peaks increases with

temperature. Simultaneously with the disappearance of the peaks corresponding to α -alumina, a new crystalline phase ($t\text{-ZrO}_2$) appears. This observation confirms the complete reaction between α -alumina and silica. At 1500°C, the formation of the composites zircon-mullite-zirconia (ZSMZ) is completed.

The X-rays diffraction patterns for the samples prepared with 40% bohemite + 60% zircon (Fig. 7) show the formation of the composites zircon-mullite-zirconia at about 1500°C. In this case, the ratio of mullite+zirconia / zircon is larger than that in the preceding mixtures (30% bohemite +70% zircon).

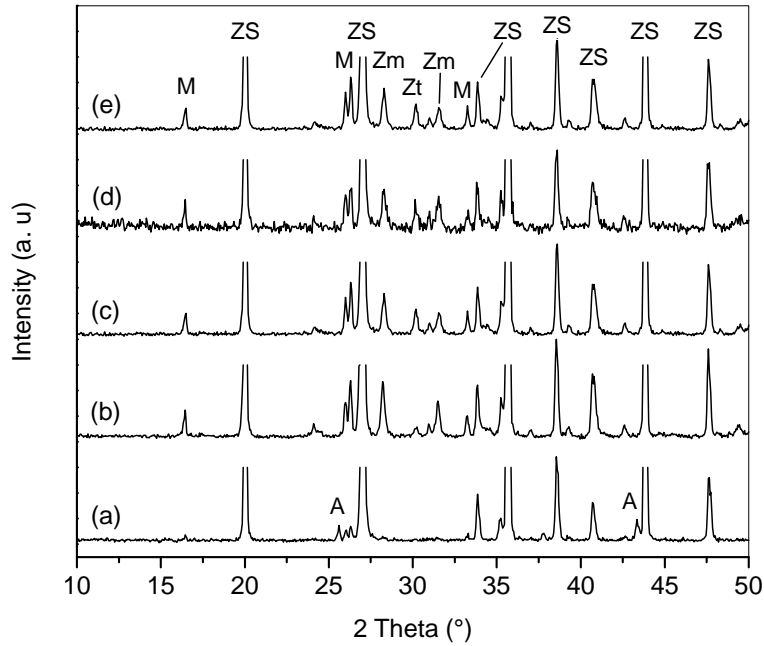


Fig. 5: XRD patterns for the 20% bohemite + 80% zircon mixture sintered at (a) 1400, (b) 1450, (c) 1500, (d) 1550 and (e) 1600°C for 2 h (ZS: Zircon, M: Mullite, A: α -Alumina, Zm: monoclinic zirconia, Zt: tetragonal zirconia).

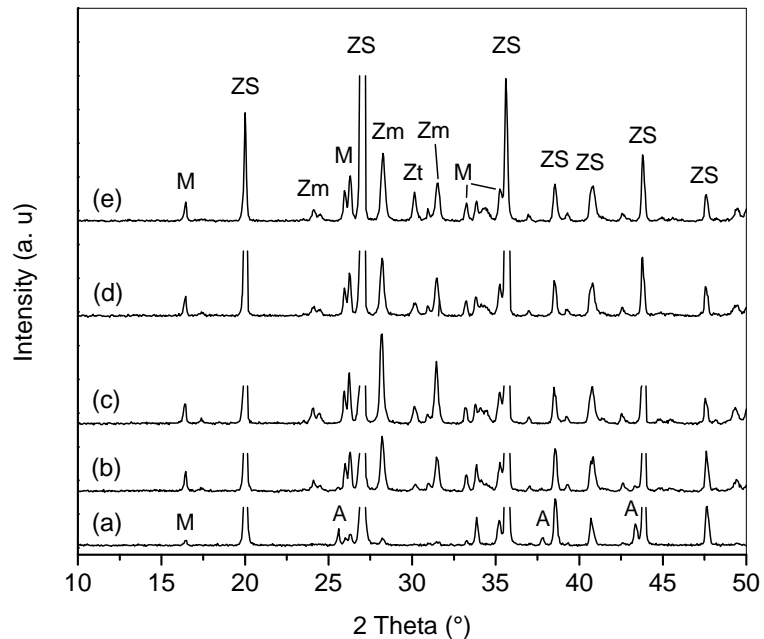


Fig. 6: XRD patterns for the 30% bohemite + 70% zircon mixture sintered at (a) 1400, (b) 1450, (c) 1500, (d) 1550 and (e) 1600°C for 2 h.

Mullite-zirconia composites

The XRD analysis realized from the samples containing 50% bohemite + 50% zircon (Fig. 8) shows the formation of mullite-zirconia composites at 1450°C with both tetragonal and monoclinic phases. In addition to the peaks corresponding to zircon, this spectra reveals the presence of the α -alumina of which the peaks characteristic disappear in the case of samples sintered up to 1500°C. At 1550°C, a total dissociation of zircon is obtained and the formation of the composites mullite-zirconia (MZ) is completed.

The x-rays diffraction spectra of the specimens obtained with 60% of bohemite + 40% of zircon (Fig. 9) reveal the presence of α -alumina and zircon with the appearance of a new phase (tetragonal zirconia) in the sample heated at 1400°C. On the other hand, the diffraction of the sample treated at 1450°C shows the formation of the mullite and zirconia (t) phases. At 1500°C, a total decomposition of zircon and formation of mullite-zirconia composites with both tetragonal and monoclinic phases are observed.

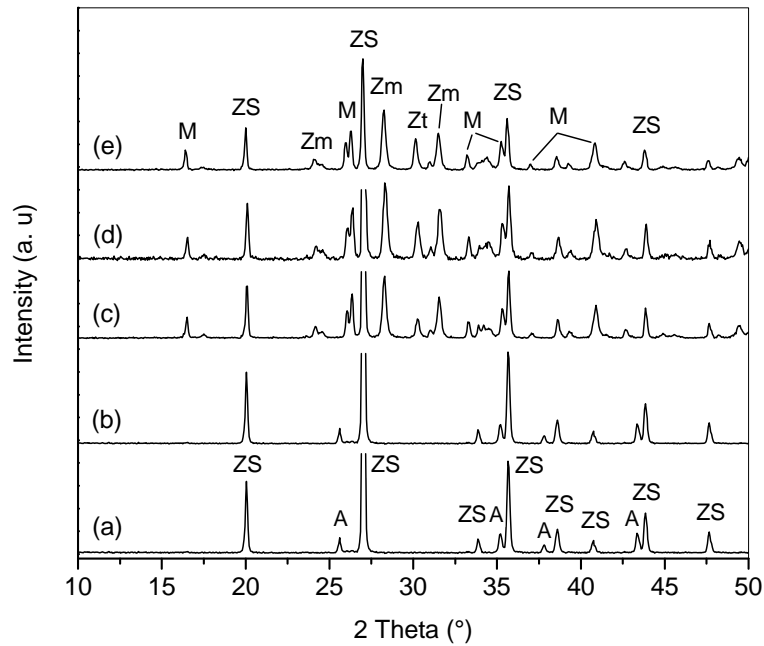


Fig. 7: XRD patterns for the 40% bohemite + 60% zircon mixture sintered at (a) 1400, (b) 1450, (c) 1500, (d) 1550 and (e) 1600°C for 2 h.

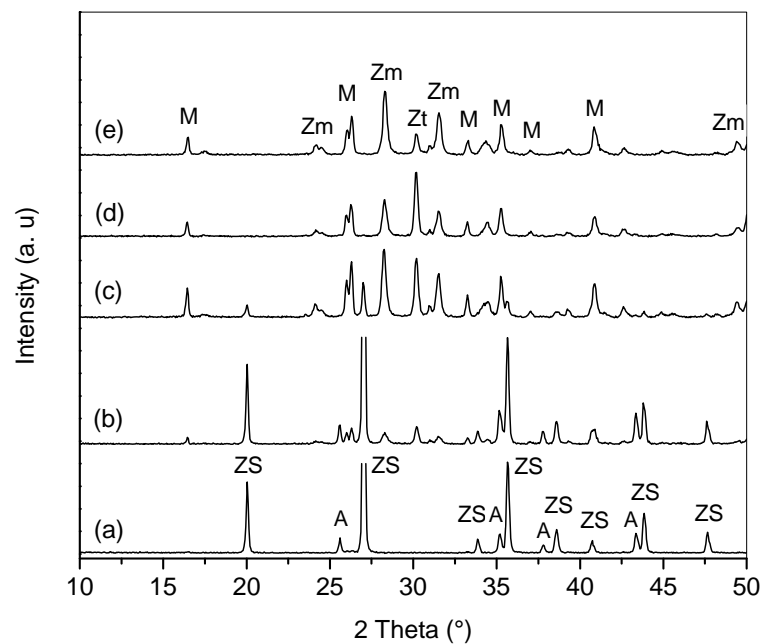


Fig. 8: XRD patterns for the 50% bohemite + 50% zircon mixture sintered at (a) 1400, (b) 1450, (c) 1500, (d) 1550 and (e) 1600°C for 2 h.

Alumina-mullite-zirconia composites

The diffraction patterns obtained from the samples prepared with 70% bohemite + 30% zircon (Fig. 10) reveal the presence of α -alumina and zircon with the appearance of tetragonal zirconia at about 1400°C confirming the decomposition of zircon below this temperature. At 1500°C, the total decomposition of zircon and the formation of the composite alumina-mullite-zirconia (AMZ) with both zirconia phases (m+t) is noted.

The presence of α -Al₂O₃, zircon and tetragonal zirconia at about 1400°C is revealed from the XRD patterns of the samples prepared by 90% bohemite + 10% zircon and sintered at various temperatures

(Fig. 11). With increasing temperature up to 1500°C, mullite peaks are observed. Complete dissociation of zircon is achieved at 1500°C. When alumina is in excess, the composite alumina-mullite-zirconia (AMZ) is obtained.

The XRD patterns global analysis showed that starting from a mixture of two initial powders (bohemite and zircon), various composites with different phases (alumina, mullite, zircon, zirconia (m + t)) can be obtained at different sintering temperatures and different ratios. Mullite-zirconia (MZ) composites were obtained for a 50% initial powder proportioning ratio.

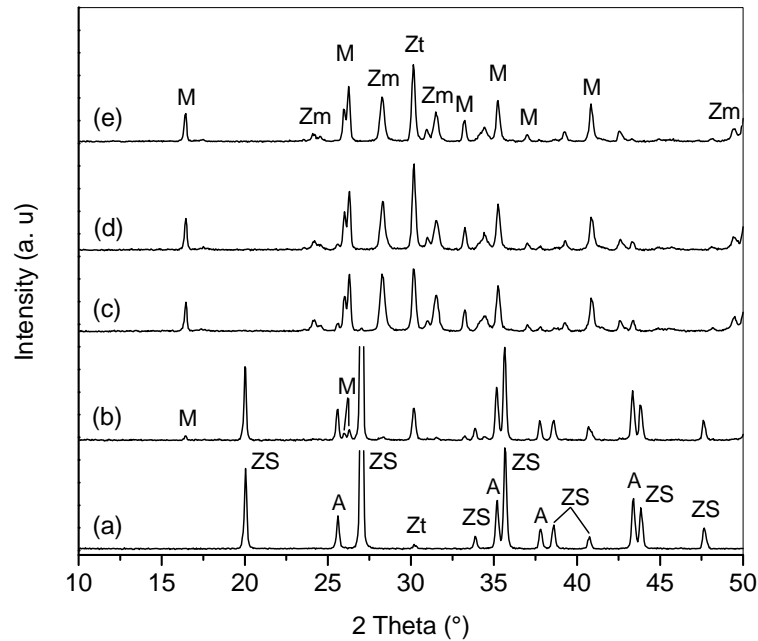


Fig. 9: XRD patterns for the 60% bohemite + 40% zircon mixture sintered at (a) 1400, (b) 1450, (c) 1500, (d) 1550 and (e) 1600°C for 2 h.

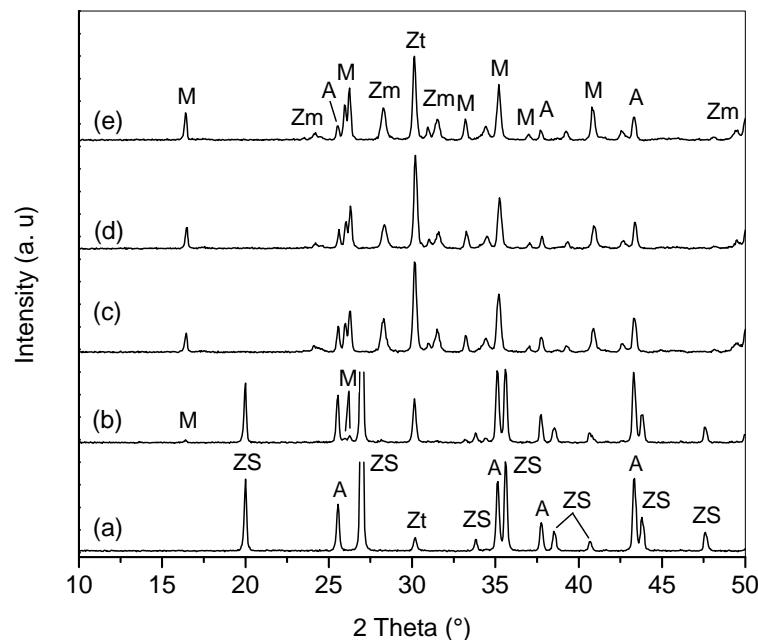


Fig. 10: XRD patterns for the 70% bohemite + 30% zircon mixture sintered at (a) 1400, (b) 1450, (c) 1500, (d) 1550 and (e) 1600°C for 2 h.

In cases where bohemite is in excess, alumina-mullite-zirconia (AMZ) composites were revealed. Zircon-mullite-zirconia (ZSMZ) and zircon-mullite (ZSM) composites are instead obtained when zircon is in excess.

Microscopy observations

Fig. 12 shows the scanning electron microstructure of all samples sintered at 1600°C. It appears that a treatment during 2 hours at 1600°C is sufficient for the sintering reaction between α -Al₂O₃ and zircon. For samples containing less than 30wt. % of bohemite (Fig. 12-a and 12-b) a zircon matrix (clear rounded particles) with dispersoids of mullite grains

(sinks) of irregular shape are primarily observed. An important porosity is also present. It varies from sample to sample according to the mixture composition of the starting powders. The distribution of the mullite seems more homogeneous in the zircon matrix.

In samples containing 30 and 40wt. % of bohemite (Fig. 12-c and 12-d), there is the formation of zircon-mullite-zirconia composites. All samples are composed of irregularly zircon and mullite shaped and distributed in a homogeneous way. The presence of a lower porosity is also apparent.

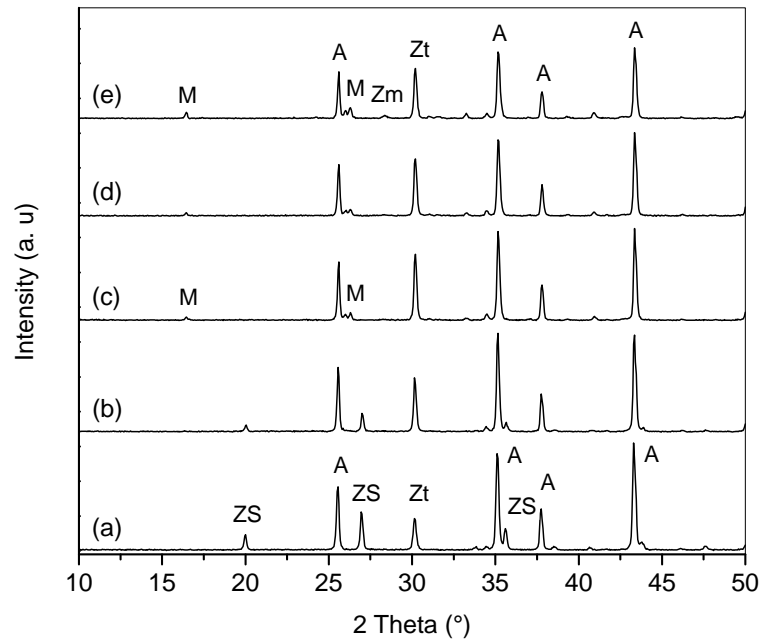


Fig. 11: XRD patterns for the 90% bohemite + 10% zircon mixture sintered at (a) 1400, (b) 1450, (c) 1500, (d) 1550 and (e) 1600°C for 2 h.

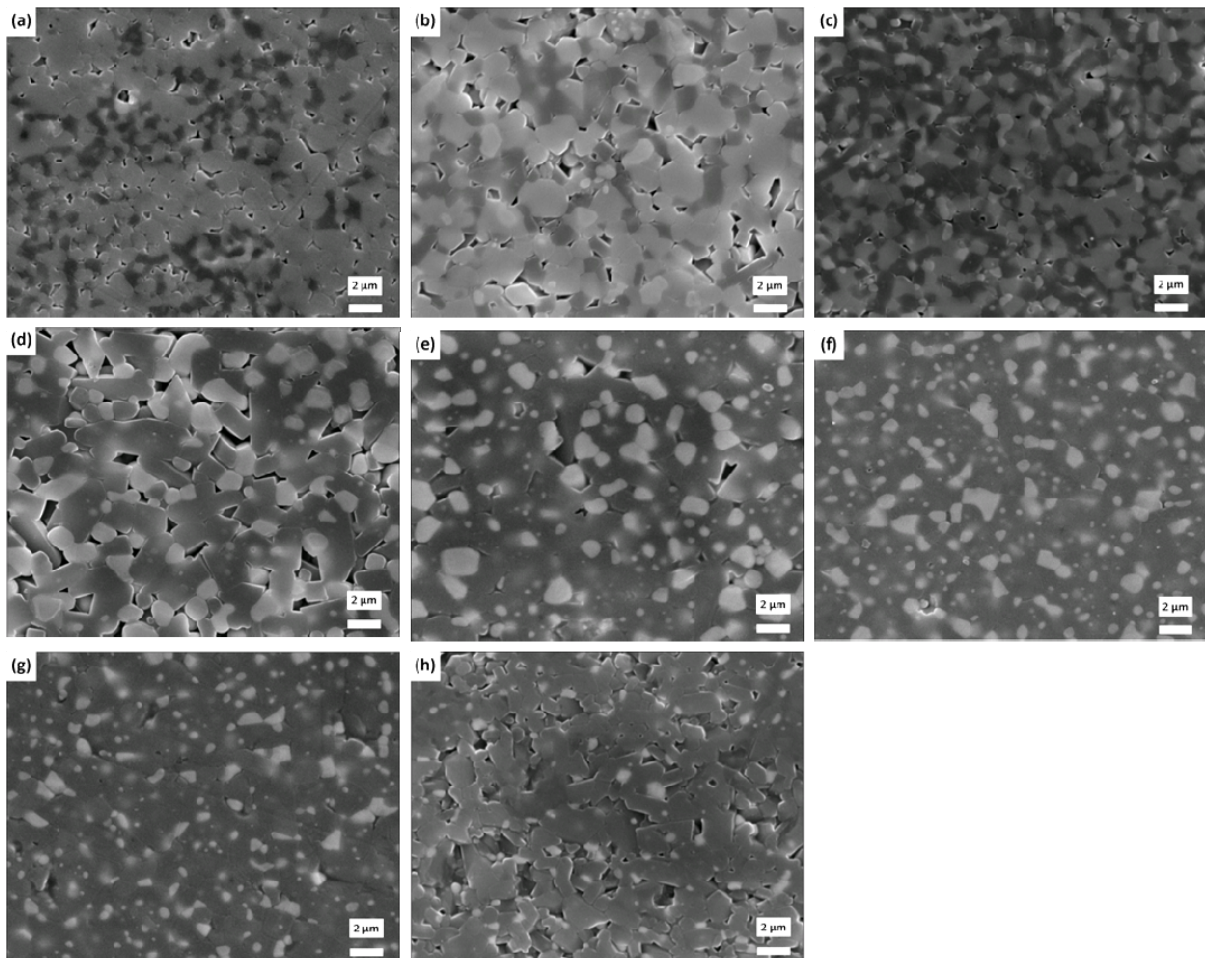


Fig. 12: SEM micrographs of composites sintered at 1600°C for 2h, a) 10wt. % of bohemite, b) 20wt. % of bohemite, c) 30wt. % of bohemite, d) 40wt. % of bohemite, e) 50wt. % of bohemite, f) 60wt. % of bohemite, g) 70wt. % of bohemite and h) 90wt. % of bohemite.

Fig. 12-e shows the formation of mullite-zirconia composites characterized by a more homogeneous structure and a uniformly distributed porosity. All samples are composed of irregularly shaped large mullite grains and round shaped zirconia grains distributed both intergranularly and intragranularly. For a high percentage of boehmite (> 50wt. %), the microstructure (Fig. 12-f, 12-g and 12-h) shows elongated and irregular grains of alumina with a few irregular grains of mullite and a small amount of zirconia. Porosity is very important in this structure. The XRD analyses confirm the results on these microstructures observations.

Density evolution

The density is a very interesting parameter used to characterize a sintered product. The relative density of all eight composites sintered at 1600°C for 2 h is reported in Table 3. All samples exhibits a high densification and obtains a relative density greater than 90%. The highest and lowest values (97.83 and 90.13 %) is obtained for 90 and 10wt. % of boehmite, respectively. It is evident from the table that the relative density decreases with boehmite addition from 10 to 20wt. %, then increases in the case of 50wt. % of boehmite. The presence of zircon and zirconia (m+t) is responsible for the high relative density in the sample containing 20wt. % of boehmite.

The increases in relative density may be due to the increase of the zirconia amount [high density $\approx 6.09 \text{ g/cm}^3$ for (Zt) and 5.87 g/cm^3 for (Zm)] in the samples containing 40 and 50wt. % of boehmite, as confirmed by the enhancement of the zirconia peaks intensity in the X-ray diffraction patterns. However, the presence of alumina (High density $\approx 3.98 \text{ g/cm}^3$) is responsible for the density increase in the sample containing 90wt. % of boehmite.

Fig. 13 shows the densification behaviour (bulk density) of the samples with boehmite content. In all samples, the densification rate change with the reaction develops (Fig. 4-11). For samples heated at 1400°C, it decreases linearly with boehmite

contents varying from 10 to 90wt. %. The density of all composition is very low at this temperature. The densification is similar for all samples heated at 1450 and 1500°C. A sharp decrease in bulk density is observed, reaching respectively 2.87 and 2.80 for 30 and 40wt. % of boehmite. This effect is due to the formation of mullite (low density $\approx 3.17 \text{ g/cm}^3$). Similar results were found in other works [8] in conformation with the x-rays diffraction analyzes. Composites with mullite matrix are indeed formed in samples containing 40 and 50wt. % of boehmite. The small increase in density is related to the densification of the remaining residual $\alpha\text{-Al}_2\text{O}_3$. For the same reasons, the bulk density increases in samples containing 50 and 90wt. % of boehmite.

At 1600°C, the bulk density decreases with boehmite addition from 10 to 50wt. % and then increases for higher levels (up to 90wt. %). The presence of zircon (high density $\approx 4.6 \text{ g/cm}^3$) is responsible for the high bulk density in the sample containing 10wt. % of boehmite. The decreases in density may be due to the increase of the mullite content in samples containing 40, 50 and 60wt. % of boehmite as confirmed by the enhancement of the mullite peaks intensity in the X-ray diffraction patterns. However, the presence of alumina with higher density ($\approx 3.98 \text{ g/cm}^3$) is responsible of the density increase in samples containing 70 and 90wt. % of boehmite.

Open porosity evolution

The curves of the open porosity variation with boehmite and zircon content have different aspect according to the sintering temperature (Fig. 14). At 1400°C, the curve presents instability in its behavior because the samples are not yet sintered. We still have interconnected pores. At 1450 and 1500°C, the porosity curves have similar shape characterized by a growth up to 40wt. % boehmite content followed by a decrease. The porosity increase is related to the zircon's dissociation into silica (vitreous phase) and zirconia (tetragonal+ monoclinic) competing with the sintering mechanisms. The following diminution of the open

Table 3: Relative density of composites heated at 1600°C for 2 h

Samples	Relative density (%)
90% B + 10% ZS	97.83
70% B + 30% ZS	94.30
60% B + 40% ZS	93.97
50% B + 50% ZS	92.68
40% B + 60% ZS	93.44
30% B + 70% ZS	93.32
20% B + 80% ZS	94.18
10% B + 90% ZS	90.13

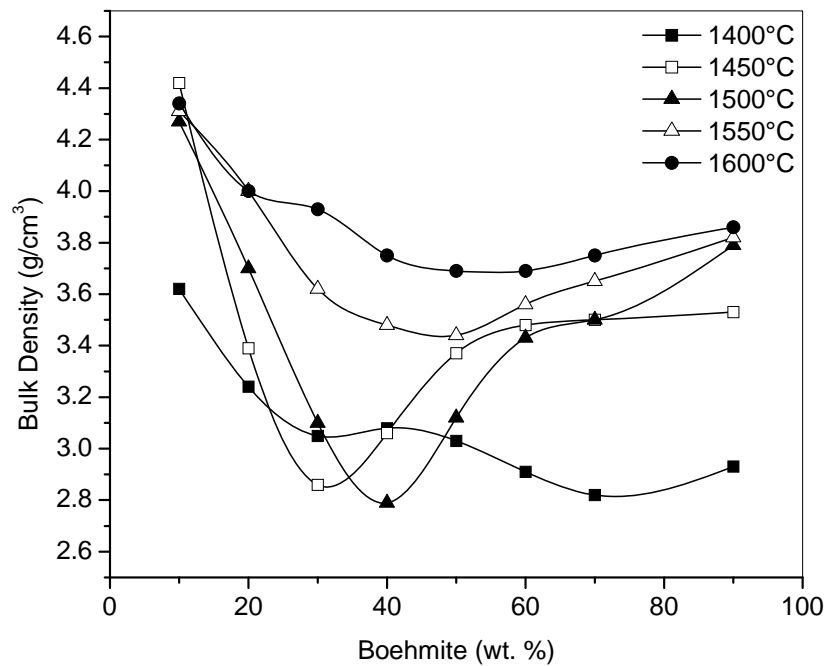


Fig. 13: Bulk density of composites as a function of boehmite addition

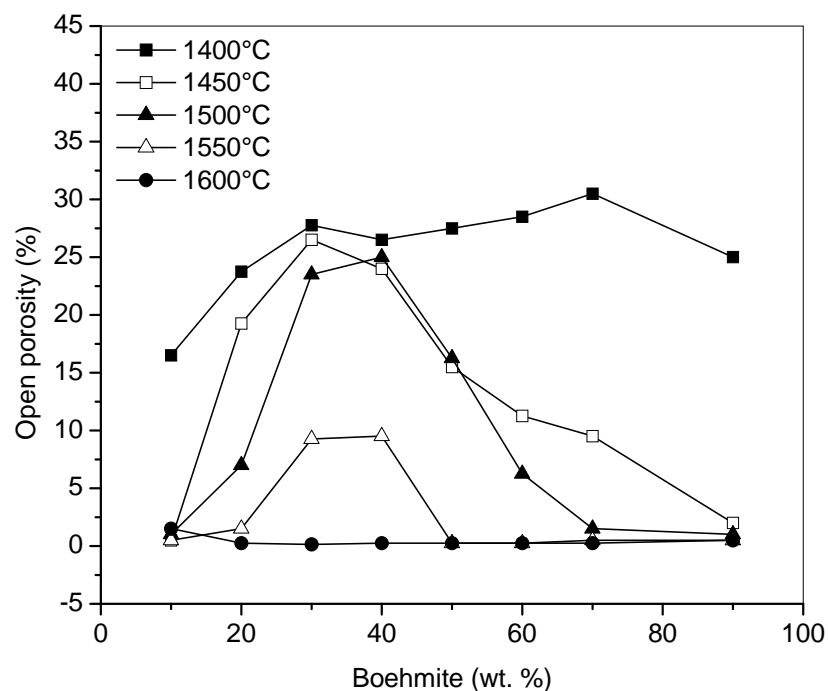


Fig. 14: Open porosity changes of the composites as a function of boehmite addition

porosity can be explained by the resulting silica from the dissociation of zircon (vitreous phase) which is located at the grain boundaries (confirmed by microscopic observation). For the samples treated at 1550°C, a strong progression of porosity up to 40wt. % of boehmite related to the total zircon's decomposition is noticed. This phenomenon disturbs the sintering mechanisms. Beyond, the porosity drops because of the complete formation of sintered composites. At 1600°C the open porosity is lowest than 2% in all composites.

CONCLUSION

From the characterization of the different composites and their phases obtained by varying percentages of boehmite and zircon starting powders, we can conclude the following points:

- The use of boehmite instead of α -Al₂O₃ lowers the sintering reaction temperature to 1550°C.
- The shrinkage is perturbed by the presence of two expansions at 1350 and 1500°C.
- The zircon's dissociation starts from 1350°C for the lowest boehmite ratios. A mullite-

zirconia composite is formed using 50 and 60wt. % of boehmite. For lower contents, zircon-mullite-zirconia composites are obtained. Alumina-mullite-zirconia composites are formed for higher boehmite content levels.

- The density measurement reveals a good densification of composites sintered between 1550°C and 1600°C.

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