Multiphase Composites Obtained by Sintering Reaction of Boehmite and Zircon Part I: Development and Microstructural Characterization

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Abstract:
In this work, different composites (alumina-mullite, zirconia-mullite-zirconia, mullite-zirconia and alumina-zirconia-mullite) were developed by reactive sintering of a powder mixture of boehmite (AlOOH) and zircon (ZrSiO4). These powder mixtures were mixed and ground by ball milling and then pressed in cylindrical form. Finally, the green specimens were sintered in air during 2 hours between 1400°C and 1600°C, with a heating and cooling rate of 5°C/min.

The dilatometric curves show that there are several microstructural transformations in these mixtures. X-ray diffraction spectra showed formation of several composites depending on the initial conditions (% of boehmite and zircon and sintering temperature). The micrographic observations of the samples confirmed the presence of various phases.

Keywords: Multiphase composites, Reactive sintering, Development, Alumina, Zircon, Mullite, Zirconia.

1. Introduction

Nowadays, the multiphase composites ceramic-ceramic have an important industrial and technological role [1]. The Al2O3-SiO2-ZrO2 equilibrium diagram developed by Budnikov and Litvakovskii [2], suggests the existence of ternary compounds starting from these oxides. But we note the existence of two binary compounds which are: mullite (3Al2O3.2SiO2) and Zircon (ZrSiO4).

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The mullite ceramic has had and will continue to have a significant role in the development of traditional and advanced ceramics. Mullite is the only stable crystalline phase in the aluminosilicate system, under normal atmospheric pressure from ambient temperature up to elevated temperatures. Its chemical composition ranges from 3Al₂O₃-2SiO₂ to approximately 2Al₂O₃-SiO₂. Mullite exhibits a good mechanical behavior at high temperatures [3].

Zircon is the only zirconium silicate in the ZrO₂-SiO₂ diagram. It is an abundant and economical ceramic that has a good chemical stability at ambient and at high temperatures up to 1667°C where zircon dissociates into zirconia and silica [4]. This stability gives a good corrosion resistance to the material. Its low expansion coefficient makes it very resistant to thermal shock [5]. However, its low creep resistance limits its applications.

Multicomposites ceramics, based on zircon-zirconia-mullite-alumina, can be obtained by different processes [6]. They exhibit excellent corrosion and thermal shock resistances and enhanced mechanical properties [7]. The content and structure of phases present in these materials may be related to the phase content of the raw materials, to the eventual zircon dissociation and to monoclinic/tetragonal martensitic transformation [8].

According to the ternary alumina-silica-zircon diagram, adding small amount of alumina to zircon leads to composition located in the compatibility triangle zircon-zirconia-mullite ZSMZ [9]. Zircon is the matrix, while mullite and zirconia are the dispersant phases. Anjali et al. [10] has observed that addition of alumina enhances partial low temperature decomposition of zircon under the influence of plasma generated during Spark Plasma Sintering. Mullite formed as a result of this in situ reaction between alumina and silica acts as a bonding phase.

There are many main processing routes for producing mullite-zirconia composites, each leading to a special microstructure and then to specific properties. Mullite-zirconia materials can be produced by conventional sintering of mechanical mixtures of fused mullite or pre-mullite and zirconia powders. The second technique is based on sintering the constituent oxides of mullite (SiO₂ and Al₂O₃) or their precursors (Alkoxides, Kaolin,...) and zirconia content [11]. It can also be produced by an in situ reaction process between zircon and alumina to form mullite with dispersed zirconia [12].

Reaction sintering of alumina and zircon is an economic and relatively easy processing route to obtain homogeneous mullite-zirconia composite ceramics [13]. This reaction is expressed as follows:

\[ 3 \text{Al}_2\text{O}_3 + 2 \text{ZrSiO}_4 \rightarrow 3\text{Al}_2\text{O}_3-2\text{SiO}_2 + 2 \text{ZrO}_2 \]

This reaction-sintering is difficult to control because of occurring chemical reactions, such as decomposition of ZrSiO₄ and formation of mullite, as well as densification during sintering at various temperatures. S. H. Badiee [14] has observed that the additives like nano TiO₂, which do not form a solid solution with the matrix, encourage the sintering rate by a transitory liquid formation and increase the mechanical strength.

The zirconia dispersion in the ceramic matrix improves the thermomechanical properties by transformation and microcracking [15]. Besides, microcracks due to the martensitic transformation may cause the dissipation or adsorption of strain energy by crack spreading. For this reason these composites are likely to have a good thermal shock resistance. Claussen [16] was shown that a special sintering technique, which essentially consists of separation of the sintering and reaction steps, produced a fully dense matrix with zirconia dispersion.

The composite alumina-zirconia-mullite (AMZ) shows good mechanical strength at ambient and at high temperature. This behavior is significantly reduced in the presence of glassy phase [17].

In this work, we developed by reaction sintering composites with dispersed phases from boehmite and zircon powders. Several mixtures were used by varying the proportion of boehmite from 10 to 90% (wt%). Physical characterizations were conducted (shrinkage
behavior, X-ray diffraction and density measurements). Observations of microstructures, using scanning electron microscopy, were carried out for different obtained composites.

2. Experimental procedure

In this study, we mainly used like starting powders a monohydrate alumina (boehmite (AlOOH)) and a micronized zircon. 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 size of this powder is about 70 µm (Fig. 1). The zircon chosen for this study is provided by Moulin des Près (French Company). The morphology of this powder is irregular. The average diameter of the granules is, in this case, close to 1.5 µm (Fig. 2). Tab. I gives the starting powders chemical compositions.

<table>
<thead>
<tr>
<th>Tab. I Chemical compositions of the starting powders (% wt)</th>
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<tbody>
<tr>
<td>Elements</td>
</tr>
<tr>
<td>Al₂O₃</td>
</tr>
<tr>
<td>ZrO₂</td>
</tr>
<tr>
<td>SiO₂</td>
</tr>
<tr>
<td>F</td>
</tr>
<tr>
<td>Na₂O</td>
</tr>
<tr>
<td>CaO₂</td>
</tr>
<tr>
<td>Fe₂O₃</td>
</tr>
<tr>
<td>Ti₂O</td>
</tr>
<tr>
<td>K₂O</td>
</tr>
<tr>
<td>HfO₂</td>
</tr>
<tr>
<td>Structural water</td>
</tr>
</tbody>
</table>

The boehmite powder was attrition milled with alumina balls in aqueous media for 3 h to reduce d₅₀ to 1.5 µm. Milling was carried out under the following conditions: the powder to ball ratio was kept 1:10 by weight, the addition of approximately 0.25 wt. % of ammonium polymethacrylate dispersant (Darvan C) and ammoniac was used to adjust the pH of the suspensions to 10.4. The mixtures (X% boehmite + Y% zircon) are non-stoichiometric compositions for reaction sintering to form various composites in accordance with the mass dosages ranging from 10 to 90% of each component. Each mixture was mixed and homogenized by ball milling during 20 hours under the same preceding conditions. After milling, the mixtures were dried at 110°C and 1 wt % polyvinyl alcohol (PVA) + 0.5 wt. % polyethylene glycol (PEG) were added as binder by mortar mixing. Then the mixtures were granulated through a 45 µm sieve.
The samples were uniaxially pressed at 7 MPa and isostatically cold pressed at 250 MPa in the shape of disks (diameter: 15 mm) and a thickness varying from 8 to 10 mm. Isopressed samples were heated up to 600°C at a rate of 1°C/min to burn out the binder. To determine an optimum preliminary sintering temperature, the compacts were then sintered in air in the range 1400-1600°C with a step of 50°C for 2 h soaking using a heating rate of 5°C/min.

Linear thermal expansion was performed on the cylindrical green rods using SETARAM TMA 92 dilatometer up to 1600°C with 5°C/min heating rate. Phases present and their transformation in the heated powders were identified by X-ray diffraction using a RIGAKU diffractometer [using Ni-filtered CuKα X radiation (40kV-25mA) with a scanning speed of 2° (2θ) per minute and 0.05° of step. The morphology of powders was characterized by means of a JEOL 840 A. scanning electron microscope (SEM).

3. Results and discussion
3.1. Shrinkage study

During heating of boehmite and zircon mixture, two phenomena proceed simultaneously: the reaction between silica coming from the dissociation of zircon and alumina resulting from the boehmite transformation. During sintering of samples mixture between the ambient temperature and 1600°C, the shrinkage curves (Fig. 3) show many evolutions:
- For temperatures lower than 1000°C, no shrinkage is observed. These temperatures are
insufficient to induce any shrinkage.
- Towards 1050°C, we observe a beginning of shrinkage; it is allotted to the beginning of the connection zone between the particles [18].
- In the interval 1100°C-1200°C, there is a transitory alumina transformation to form sTab. alumina α [18, 19].

An important shrinkage is observed beyond 1200°C, it is related to the zircon decomposition which give tetragonal zirconia and silica (vitreous phase). The inflection curve observed at 1350°C is caused by the phase transformation of a residual quantity of the transitory alumina phase \( \text{Al}_2\text{O}_3-\theta \) into \( \text{Al}_2\text{O}_3-\alpha \) [20].

The shrinkage curves of the intermediate mixtures are displaced towards the high temperatures when the zircon concentration increases. However, it appears that for weaker zircon contents, the shrinkage is dominated by alumina-\( \alpha \) whereas for larger zircon rates, we note a clear influence of the zircon’s behavior on the total shrinkage.

Fig. 3. Shrinkage curves of bohemite-zircon mixtures during sintering.
The temperature to which the phenomenon of curve swelling occurs differs slightly according to the boehmite initial percentage: it is lower than 1345°C for boehmite high percentage; it is equal to 1345°C for 50% of boehmite and occurs higher than 1345°C for the mixtures whose boehmite content is very weak. We quantified this phenomenon by expressing the percentage of curve swelling with the starting and final temperatures of this expansion. The results obtained are gathered in Tab. II. We note a clear growth of swelling with the boehmite percentage increasing; it reaches a maximum value (1.54%) in the case of the samples containing 70% of boehmite. In the case of percentages of boehmite higher than this value, the curve swelling decreases by 0.97% to 0.28% when one passes from 50 to 30% mass of boehmite. This swelling is directly related to the boehmite starting percentage i.e. to the theta phase. This expansion is caused by the zircon dissociation to tetragonal zirconia t-ZrO₂ and silica (vitreous phase) [20]. In the case of samples containing 70% of boehmite, a large quantity of the θ phase is present, consequently the number of contact between the granules of zircon and the granules of the phase θ increases.

Tab. II Expansion characteristics observed between 1300°C and 1400°C, deduced from the expansion behaviors.

<table>
<thead>
<tr>
<th>Samples</th>
<th>% of Expansion</th>
<th>Start temperature of expansion (°C)</th>
<th>Finish temperature of expansion (°C)</th>
<th>Temperature difference (°C)</th>
</tr>
</thead>
<tbody>
<tr>
<td>90% B + 10% ZS</td>
<td>0.14</td>
<td>1315</td>
<td>1340</td>
<td>25</td>
</tr>
<tr>
<td>70% B + 30% ZS</td>
<td>1.54</td>
<td>1330</td>
<td>1370</td>
<td>40</td>
</tr>
<tr>
<td>60% B + 40% ZS</td>
<td>1.35</td>
<td>1336</td>
<td>1376</td>
<td>40</td>
</tr>
<tr>
<td>50% B + 50% ZS</td>
<td>0.97</td>
<td>1345</td>
<td>1380</td>
<td>35</td>
</tr>
<tr>
<td>40% B + 60% ZS</td>
<td>0.77</td>
<td>1358</td>
<td>1390</td>
<td>32</td>
</tr>
<tr>
<td>30% B + 70% ZS</td>
<td>0.28</td>
<td>1360</td>
<td>1390</td>
<td>30</td>
</tr>
<tr>
<td>20% B + 80% ZS</td>
<td>0.02</td>
<td>1361</td>
<td>1386</td>
<td>25</td>
</tr>
<tr>
<td>10% B + 90% ZS</td>
<td>-</td>
<td>1380</td>
<td>1405</td>
<td>25</td>
</tr>
</tbody>
</table>

The shrinkage of the samples begins again notably at high temperature in consequence of the sintering mechanism (elimination of open porosity). Silica formed by the dissociation of zircon, reacts with α-alumina towards 1500°C to form mullite (3.17 g/cm³) in the samples containing between 30 and 70% of boehmite. That explains the rapid shrinkage deceleration (the second swelling) starting from 1500°C, also observed by CLAUSSEN et al. [16]. The shrinkage takes once again considerably with the increase temperature which one allots to elimination open porosity (sintering mechanism). This shrinkage is more notable as the percentage of boehmite decreases up to 30%.

For the samples containing 10% of Boehmite, we do not notice this swelling. That is due to the weak Boehmite content which is insufficient to form a large quantity of mullite.
3.2. Phases identification by the XRD
3.2.1. Composites Zircon-Mullite-zircone

X-rays diffraction patterns obtained from the samples heated at different temperatures are presented in Fig.s (4 to 11). XRD Spectra for the samples containing 10% bohemite + 90% zircon are given in Fig. 4. This Fig. shows the crystalline phases of zircon and mullite. We note 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 from 1400°C. The presence of the impurities in the zircon powder (1.5% HfO₂; 0.10% TiO₂ and 0.08 Fe₂O₃) is probably the cause of the reduction of the dissociation temperature of zircon; this behavior was also mentioned by other authors [21, 16]. 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. During heating, zircon is in equilibrium with the glassy phase. This glassy phase is formed at a corresponding invariant point (around 1450°C). Alumina present in the mixture is dissolved in this glassy phase. Mullite is formed from this glassy phase by precipitation during cooling [9]. For small additions of boehmite (10%), all spectra did not reveal the presence of zirconia (t, m). This absence of zirconia is justified by the presence of 3.86% SiO₂ impurities in the starting boehmite.

![Fig. 4. XRD patterns for 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).](image)

![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, Zm: tetragonal zirconia).](image)
X-rays diffraction patterns recorded from the samples containing 20% boehmite + 80% zircon are represented in Fig. 5. We note the formation of the composites zircon-mullite-zirconia (m) at 1450°C. At 1600°C we notice the appearance of the tetragonal zirconia.

Fig. 6 corresponding to the samples prepared with 30% boehmite + 70% zircon show a clear modification of the formed phases. We note the presence of four phases at 1400°C: \( \alpha \)-alumina, zircon, mullite and monoclinic zirconia (m-ZrO\(_2\)). From 1450°C, the intensity of the mullite and zirconia (m) peaks increases with temperature. Simultaneously with the disappearance of the peaks corresponding to \( \alpha \)-alumina, a new crystalline phase (t-ZrO\(_2\)) appears. This observation confirms the complete reaction between \( \alpha \)-alumina and silica. At 1500°C, the formation of the composites zircon-mullite-zirconia (ZSMZ) is completed.

X-rays diffraction patterns for the samples prepared with 40% boehmite + 60% zircon (Fig. 7), show the formation of the composites zircon-mullite-zirconia at about 1450°C. At 1500°C, the formation of these composites (ZSMZ) is completed. In this case, we note that the quantity of the mullite and zirconia phases is larger than that in the preceding temperature.
3.2.2. Composites Mullite-zircone

XRD analysis realized from the samples containing 50% boehmite + 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 until 1500°C. At 1550°C, one notices the total dissociation of zircon to form the composite mullite-zirconia (MZ).

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 (ZS: Zircon, M: Mullite, A: α-Alumina, Zm: m-zirconia, Zm: t-zirconia).

In the case of specimens obtained with 60% of bohemite + 40% of zircon, the x-rays diffraction spectra are shown in Fig. 9. We note the presence of α-alumina and zircon with the appearance of a new phase (tetragonal zirconia) in the sample heated at 1400°C. Contrary, the diffraction of the sample treated at 1450°C show the formation of the mullite and zirconia (t) phases. At 1500°C, we note the total decomposition of zircon and the formation of mullite-zirconia composites with both tetragonal and monoclinic phases.

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 (ZS: Zircon, M: Mullite, A: α-Alumina, Zm: m-zirconia, Zm: t-zirconia).
3.2.3. Composite alumina-mullite-zirconia

The diffraction patterns obtained from the samples prepared with 70% boehmite + 30% zircon are represented in Fig. 10. These diagrams reveal the presence of α-alumina and zircon with the appearance of tetragonal zirconia at about 1400°C, which confirms the decomposition of zircon before this temperature. After 1500°C, we note the total decomposition of zircon and the formation of the composite alumina-mullite-zirconia (AMZ) with both zirconia phases (m+t).

![Fig. 10. XRD patterns for the 70% boehmite + 30% 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: m-zirconia, Zt: t-zirconia).](image)

Finally, the analysis of the XRD patterns showed that starting from two initial powders (boehmite and zircon) we obtain after sintering at different temperatures, various

XRD patterns of the samples prepared by 90% boehmite + 10% zircon and sintered at various temperatures are given in Fig. 11. We note the presence of α-alumina, zircon and tetragonal zirconia at about 1400°C. With increasing the temperature 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.

Finally, the analysis of the XRD patterns showed that starting from two initial powders (boehmite and zircon) we obtain after sintering at different temperatures, various
composites with several phases (alumina, mullite, zircon, zirconia (m + t)). If initial powder proportioning is equal to 50%, we let us obtain a composite mullite-zirconia (MZ). On the other hand, we obtain alumina-mullite-zirconia (AMZ) composites in the case of boehmite is in excess or a zircon-mullite-zirconia (ZSMZ) composites in the case of zircon is in excess.

3.3. Microscopy observations

Micrographs in Fig. 12 reveal that the structure of the samples, sintered at 1600°C, evolves according to percentages of the mixture (boehmite + zircon) used. We estimate that a treatment during 2 hours at 1600°C is sufficient to that the sintering-reaction takes place.

Fig. 12. Structures of the various sintered mixtures at 1600°C during 2h.
We note:
- For the weak boehmite rates < 30%, the first three micrographs, show primarily a zircon matrix (clear rounded particles) with dispersoids of mullite grains (sinks) of irregular form. The distribution of the latter seems more homogeneous as the starting boehmite rate increases. We note also the presence on weak scale of zirconia grains, of low size (white grains). An important porosity is also observed. It varies from a sample to another according to the mix composition of the starting powders. It increases according to the increase in the percentage of boehmite.
- For the boehmite rates of 40 and 50%, we note primarily the formation of the composite mullite-zirconia. The mullite matrix presents grains of random form. The zirconia grains, of round form, are distributed in a homogeneous way. They are of two types: smallest are will intra granular whereas largest are intergranular. In this type of composite, porosity is lower.
- For a high percentage of boehmite (> 50%), the microstructure shows elongated grains of alumina with a few grains of irregular shaped mullite and a small amount of zirconia. Porosity is very important in this structure. Through these microstructures, we confirm the results found by XRD.
- For a high percentage of boehmite (> 50%), the microstructure shows lengthened alumina grains with some mullite grains of irregular form as well as a small quantity of zirconia. Porosity is very important in this structure. Through these microstructures, we confirm the results found by the x-ray diffraction analysis.

3.4. Evolution of the density

The density is a very interesting parameter used to characterize a sintered product. In addition to the apparent density, this method makes it possible to consider the total porosity of the specimen. The results are reported in Fig. 13.

![Fig. 13. Density given by Arthur’s method.](image)

The curves have the same shape in despite of some disparities of the ones compared to the others. We can analyze these results from two following points of view:

From content point of view: we note a reduction in the apparent density in the vicinity of the composites obtained from 30% to 40% of boehmite, followed by a light increase towards 50% of boehmite then after a small decrease towards the 70% of boehmite.

The fall of the density can be explained by two factors: initially, in consequence of the increased porosity which is allotted to the decomposition of the zircon out of zirconia and
silica, and then in the second factor, during the appearance of the mullite which is formed by reconstitution (confirmed by the observations with the microscopy), and which is less dense than alumina. This approach agrees with the results found by certain authors [13] and confirms the X-rays diffraction analyzes. Indeed, the composites having mullite matrix are formed in the vicinity of the boehmite composition about 40-50%. The little increase of density is made with the densification of little remaining residual α-alumina. The increasing of density beyond 50% of boehmite, is caused by the sintering of the composite mullite-zirconia.

According to the sintering temperature, the minimal density is observed on the mixtures curves treated at 1450°C. That is related on one hand to the zircon’s progressive dissociation with the zirconia appearance and on the other hand at the beginning of low density mullite formation. It is obvious that at this temperature, sintering does not take place yet, thus giving a low density. Thereafter, we observe an increased density, allotted to the beginning of formation of the mullite-zirconia composite. This effect is less for the mixtures superior of 60% of boehmite because of the slow densification of α-alumina.

Thermal treatment at 1500°C is characterized by the complete dissociation of zircon from where diminution in the density curves. The latter goes up following the complete formation of the composite mullite-zircon. Beyond of 60% of boehmite, alumina takes again the top and controls the densification. It is starting from 1550°C that there is densification consequently to the sintering mechanisms.

The characterization of x-ray diffraction of the composites confirmed an increase in the quantity of mullite and zirconia phases with the increase in the quantities of zircon in the reaction. No other phase was found, like impurities or zircon; only alumina, mullite and zirconia (tetragonal and monoclinic phases) were identified. At the end of sintering, the densities reached for each grade rather acceptable (3.5 to 3.75 g/cm³), obtained for the composites presenting an initial zircon excess.

3.5. Evolution of open porosity

The general aspect of open porosity curve according to the various percentages of boehmite + zircon presents a great variation (Fig. 14). Indeed with 1400°C, the curve presents an instability in its behavior because the samples are not sintered yet, therefore with full interconnected pores. With 1450°C and 1500°C, the two curves of porosity present the same behavior; i.e.: Growth up to boehmite 40%, due to the zircon’s dissociation giving silica (vitreous phase) and zirconia (tetragonal+ monoclinic) which come to compete with the mechanisms of sintering.

![Fig. 14. Open porosity according to the percentages of starting powders.](RETRACTED)
A decrease which can be explained as follows: Resultant Silica of the dissociation of zircon (vitreous phase) is located at the grain boundaries, in addition to monoclinic zirconia (confirmed by microscopic observation) facts which support the diminution of open porosity. For samples treated at 1500°C, one notices a strong progression of porosity up to 40% of boehmite related to the total zircon’s decomposition. This phenomenon comes to disturb the sintering mechanisms. Beyond, porosity falls because of the complete formation of the composite which sintered. At the end of sintering open porosity falls with very small values, about 0.3% to 2%.

4. Conclusion

The characterization of the grades obtained makes us with various percentages of the starting powders (boehmite and zircon) it possible to conclude by the following points:
- The use of boehmite (to replace α-alumina) lowers the reaction sintering temperature to 1550°C.
- The shrinkage curves showed that they exist several microstructural transformations during its thermal treatment. The obtained results show the presence of two harmful swellings about 1350°C and 150°C.
- The x-ray diffraction patterns shows that the zircon’s dissociation starts from 1350°C whereas the formation of the composites begins towards 1550°C with an initial proportioning from 40% of boehmite and 60% of zircon. When the treatment is realized at 1500°C, mullite-zirconia composite was formed for 40, 50 and 60% of boehmite. In lower part of these percentages of boehmite (zircon is in excess) to obtain the composite zircon-mullite-zirconia. When alumina is in excess, a composite alumina-zirconia-mullite is obtained.
- The microstructures obtained from composites sintered at 1600°C shows a homogeneous distribution of all the phases.
- The density measurement reveals a good densification of composites sintered between 1550°C and 1600°C.

5. References

композита у зависимости от начальных условий (процент боямита и циркона и температуры синтеза). Микрографии указывают на наличие различных фаз. 

Ключевые слова: многофазные композиты, реакционная сinterация, развитие фаз, алюминий, циркон, мулит.