PRODUCTION OF CEMENT BASED ON CALCIUM ALUMINATE BY MEANS OF SOLID STATE REACTIONS

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Abstract. Through powder techniques and *in situ* solid state reactions, a refractory cement $CaAl_2O_4$-based was fabricated, using $CaCO_3$ extracted from chicken eggshells and Al as precursor materials. To reduce the particle size and achieve a homogeneous mixture, the powders were subjected to high-energy milling in a planetary mill. The powders resulting from the grinding were compacted to form cylindrical tablets. These samples were pressureless sintered in air. A particle size distribution analysis indicates that they were obtained from the grinding particles ranging in size from nanometers to 2 microns. Differential thermal analysis indicates that the decomposition of $CaCO_3$ begins at 953 K and ends at 1073 K, a situation confirmed by X-ray diffraction analysis, the latter also indicating that the formation of the $CaAl_2O_4$ crystalline phase is completed at 1773 K. The microstructure observed by scanning electron microscope shows equiaxial grains in the form of flakes and sizes from 1 to 2 microns. The average density and hardness of the material was 3.08 g/cm^3 and 430 HV, respectively. With regard to thermal shock tests, the material showed cracks from cooling with temperature gradients of 873 K.

Keywords: refractory cement, calcium aluminate, solid state reaction, high alumina cement.

1. Introduction

Refractory materials are widely used in various industrial sectors by virtue of their excellent properties and performance in aggressive environments.[1] Refractories are materials that must be worked under the following conditions: to resist high temperatures, especially when they are in contact with molten metals or hot gases with strongly acidic or basic characteristics, to resist abrasion, to tolerate sudden changes in temperature, to retain heat, to have a low coefficient of thermal expansion, and not to interact chemically with the material with which they are in contact.[2] High alumina cements (HAC) have increased for refractory applications in recent years, being used mainly in low cementing molds. At present, the HAC are prepared from the mixture of clayey and calcareous materials, where, through a grinding, a fine powder is obtained, which is later submitted to a fusion or sintering above 1773 K.[3] However, over the years various methods have been developed for the synthesis of HAC. This is how Fernández-González *et al.*[4] synthesized this refractory material through a solar synthesis route. In another study developed by Xiao *et al.*,[5] where through a synthesis *in-situ* they have improved corrosion resistance, good crush resistance, and high cold modulus of rupture of HAC. The same researchers also developed the solution-burning method, in which calcium nitrate and aluminum solutions were added to glycine fuel, in the following ratios: total nitrate to fuel of 1:0, 4:1, and 1:2, the latter ratio being the one that forms small sizes.[6] Lee[7] has studied the manufacture of Al_2O_3 ceramics by the reaction bonding aluminum oxide (RBAO) process, using Al-Zn-Mg alloy powder to improve traditional RBAO ceramic processing where just pure Al is used. Another study, in which an RBAO matrix was implemented in processing total oxide ceramic matrix compounds, produced a reduced amount of shrinkage-related matrix cracks, a beneficial feature for refractory applications.[8] A publication by Rumyantsev and co-workers[9] indicates that peculiarities in the $Ca(OH)_2$-Al-H_2O system after heat treatment at 1273 K led to the formation of aluminates with the same molar ratio significantly improving the mechanical properties of the final product. A study conducted in 2019[10] showed that particle size significantly influences mechanical properties of HAC, with a better distribution of particles when they are tested at temperatures above 1723 K.

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On the other hand, the precursor method for obtaining alumina (the main element in HAC cements) is the Bayer process, which uses bauxite ore to obtain aluminum hydroxide, which is then calcined to obtain alumina.[11] However, a new technique for the reaction alumina formation called reaction bonded aluminum oxide (RBAO), has been developed.[12-14] Current studies of this technique have shown that the compositions, fruits of the RBAO process, have significantly helped the powders to adopt a nanometric grain size, thus improving their mechanical properties. Hence, the objective of this study is to produce a $CaAl_2O_4$ refractory material from a mixture of Al, Al_2O_3, and $CaCO_3$ powders through in-situ reactions involving the RBAO process and $CaCO_3$ decomposition.

2. Experimental

Materials. The raw materials used were aluminum powder <1 μm with 99.5 % purity, Al_2O_3 powder <10 μm with 99.5 % purity (both Sigma-Aldrich) and $CaCO_3$ obtained from chicken eggshells. Eggshell is considered a natural bio-ceramic material composed of a combination of organic and inorganic components. Within its chemical composition it contains 94 % of calcium carbonate ($CaCO_3$), 1 % of magnesium carbonate ($MgCO_3$), 1 % of calcium phosphate ($Ca_3(PO_4)_2$), and 4 % of other proteins.[15]

Grinding of eggshells to obtain $CaCO_3$. The first step was to wash the eggshell with water, crush it in a mortar to a homogeneous size and then grind it in the planetary mill (Retsch, PM100, Germany) at the speed of 300 rpm for 60 min.

Aluminum plus alumina grinding. A mixture composed of 30 % Al and 70 % Al_2O_3 was ground in a planetary mill for 2 h at the speed of 300 rpm, during the grinding isopropyl alcohol was used as a control agent.

Total grinding. Once $CaCO_3$ was crushed and Al and Al_2O_3 were mixed, quantities of each of these compounds were weighed according to the stoichiometry of reactions (1) and (2):

$$2Al + 3/2O_2 \rightarrow Al_2O_3 \qquad (1)$$
$$CaCO_3 + Al_2O_3 \rightarrow CaAl_2O_4 + CO_2 \qquad (2)$$
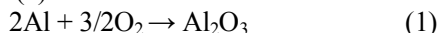
Subsequently, all the powders were ground in the planetary mill at the speed of 300 rpm for 2 h. After the grinding of the powders, their size and particle size distribution were determined with the help of a Mastersizer 2000, England equipment.

Interrupted heating test in powders after grinding. To follow the transformation sequence of the reagents (Al+Al_2O_3+$CaCO_3$) after grinding, interrupted tests were carried out in an electric furnace (Thermo Scientific Thermolyne FB1315M, USA) at different temperatures

(723, 873, 1023, 1173, 1273, and 1423 K) and atmospheric pressure. For this purpose, the powders were heated at the speed of 2 K/min to the desired temperature and cooled down inside the furnace. After heating, all powders were analyzed by X-ray diffraction (Panalytical x'pert pro, Japan) to determine the crystalline species present in them. To follow the progress of reactions (1) and (2), a sample of the total powder mixture is subjected to thermogravimetric analysis in a Shimadzu, DTG-60-H, Japan equipment.

Compaction. The ground powders were uniaxially compacted in a hydraulic press (Montequipo, LAB-30-T, Mexico) at the pressure of 30 MPa obtaining tablets of 2 cm radius and 0.3 cm thickness.

Sintering. The samples were sintered at the heating rate of 5 K/min at different temperatures (1573, 1673 and 1773 K) remaining at that temperature for 2 h, and at the end of the cycle they were allowed to cool down to room temperature inside the furnace. The microstructure of the sintered samples was analyzed by scanning electron microscopy (JEOL, 6300, Japan). The SEM was equipped with an X-ray dispersive energy analyzer (Hitachi, SU9000 UHR FE-SEM, Japan).

3. Results and Discussion

3.1. Granulometric Analysis in the Total Mixture ($CaCO_3$, Al, Al_2O_3)

Fig. 1 shows the distribution of particles sizes after milling stage. In this figure it is possible to observe that there is a good distribution of particle sizes in the powder mixture after the milling stage, since approximately 30 % of the powders have sizes smaller than 1 micron, there are 35 % of powders with sizes between 1 and 2 microns and the remaining 35 % of powders have sizes between 2 and 3 microns.
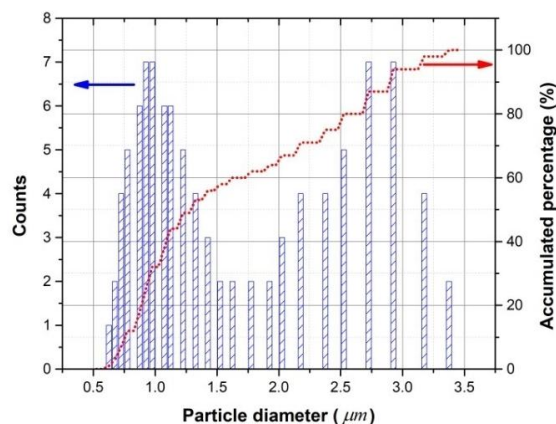

Fig. 1. Granulometry of the $CaCO_3$+Al+Al_2O_3 powder mixture

Very fine particle sizes and the homogeneous distribution of the particles help to consolidate well the desired cement and to carry out the reactions of oxidation of the aluminum, decomposition of the calcium carbonate, as well as formation of calcium aluminate – fundamental objectives in this work.

3.2. Thermogravimetric Analysis

Fig. 2a shows the thermogravimetric analysis carried out on the $Al+Al_2O_3$ powders mixture that were subjected to the heating cycle described in the experimental part, and which is used to form alumina through the oxidation of aluminum (RBAO process). In this figure it can be seen that from room temperature and up to approximately 773 K the weight of the sample remains constant, but from 773 K there begins to be a slight gain in weight which ends at 923 K, while at 1073 K again there is a gain in weight, in this case more significant, which ends at approximately 1173 K. Both weight gains are related to the oxidation of aluminum to form alumina. Observing the temperatures at which these weight gains occur, aluminum (melting temperature 933 K) oxidizes slightly when it is in the solid state (first gain 773 K) and later its oxidation is stronger when it is in the liquid state (gain at 1073 K). The total weight gain was approximately 12.5 %, which corresponds to the amount of oxygen that entered the mixture.
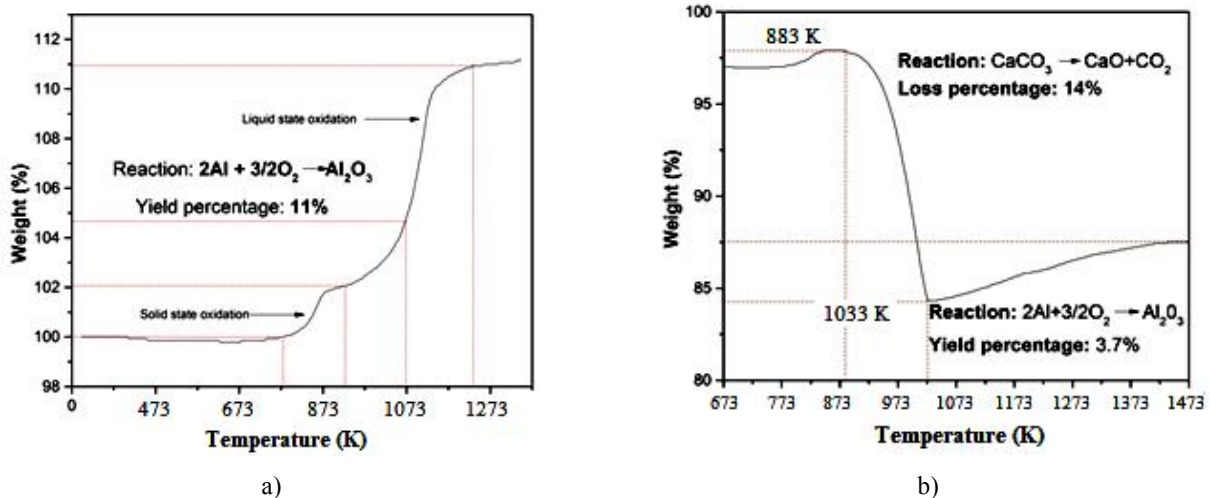

Fig. 2. Thermogravimetric analysis of $Al+Al_2O_3$ (a) and $Al+Al_2O_3+CaCO_3$ (b) mixtures

Fig. 2b shows the results of the thermogravimetric analysis performed on the total mixture ($Al+Al_2O_3+CaCO_3$) that was subjected to the heating cycle described in the experimental part. In this analysis, the weight of the sample must remain constant at room temperature until it reaches approximately 773 K, but from 773 K there is a slight gain in weight, which ends at 873 K. At temperatures above 953 K there is a considerable amount of weight loss, which ends at approximately 1033 K. The weight gain is caused by the oxidation of aluminum, while the weight loss is due to the decomposition of $CaCO_3$ which releases from the sample CO_2 and other organic elements. The formation of $CaAl_2O_4$ is not observed in the curve, because the formation of this compound is not accompanied by a change in weight. During the heating process there is a competition of two phenomena, the first is due to the oxidation reaction of the aluminum, that implies weight gain, and the second one is due to the decomposition of the carbonate, that implies weight loss. At the end of the stage the sample lost approximately 11 % of its weight, which indicates that the loss in weight due to the decomposition of the carbonate was much greater (release of CO_2) than the gain in weight achieved by the oxidation of the aluminum.

3.3. X-Ray Diffraction

Fig. 3 shows the diffraction patterns of the powder mixture ($Al+Al_2O_3+CaCO_3$) heated at temperatures ranging from 723 to 1773 K. The figure shows that as the treatment temperature increases, the intensity of the Al peaks decreases, while the intensity of the alumina peaks increases, which is mainly observed at temperatures of 873 and 1023 K. At 1173 and 1273 K some low intensity peaks of aluminum are still observed. They disappear at 1423 K indicating that reaction 1 is completed (RBAO process). In the same figure it is shown that from the temperature of 1173 K the $Ca_{12}Al_{14}O_{33}$ compound starts to form, at 1273 K new species corresponding to $Ca_5Al_6O_{14}$ and $CaAl_2O_4$ are formed, and at 1423 K the formation of

CaAl$_2$O$_4$ intensifies. This indicates the formation of different stoichiometric calcium aluminate phases at the temperatures from 1173 K. In the pattern of the samples heated to 1573 K and 1673 K the phases CaAl$_2$O$_4$, CaAl$_4$O$_7$ (grossite), Ca$_4$Al$_6$O$_{12}$SO$_4$, Ca$_{12}$Al$_{14}$O$_{33}$ (mayenite), and Al$_2$O$_3$ (corundo) are identified. At 1773 K the pattern is identified with the phases CaAl$_2$O$_4$ and CaAl$_4$O$_7$ (grossite), where the CaAl$_2$O$_4$ phase predominates, which indicates that at this temperature the desired compound is obtained in a stable way in this study.
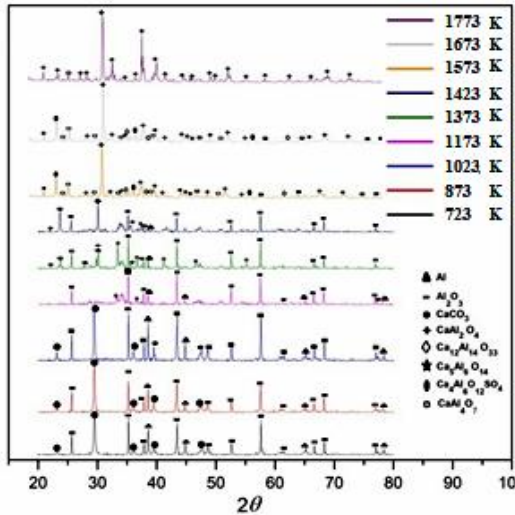

Fig. 3. X-ray diffraction of the powder mixture (Al+Al$_2$O$_3$+CaCO$_3$) heated to temperatures ranging from 723 K to 1773 K

3.4. Lattice Parameter

With the help of the crystallographic chart[16] 53-0191 of CaAl$_2$O$_4$ it was determined that the crystalline structure of this material is monoclinic. From the X-ray diffraction pattern of the sample treated at 1773 K and with the help of the above crystallographic chart, the lattice parameter of the compound was determined. This was done by using the peaks and planes shown in Table 1 and Eq. (3).[17]

$$\frac{4\sin^2\theta}{\lambda^2} = \frac{1}{\sin^2\beta}\left(\frac{h^2}{a^2} + \frac{k^2\sin^2\beta}{b^2} + \frac{l^2}{c^2} - \frac{2hl\cos\beta}{ac}\right) \quad (3)$$

Table 1. Planes and peaks to obtain the lattice parameter

Variable	Plane	2θ
a	(600)	64.31624
b	(040)	44.86483
c	(006)	35.50269
β	(313)	37.48548

Table 2 shows the comparison of the compound lattice parameter obtained by the calculations according

Eq. (3) for the sample treated at 1773 K and the values reported according to the crystallographic chart 53-0191. From these results it can be seen that the values are very close, therefore, it can be said that the lattice parameter of the synthesized material agrees as reported.

Table 2. Comparison of the CaAl$_2$O$_4$ lattice parameters

Variable	Lattice parameter according to the 53-0191 crystallographic chart, nm	Lattice parameter calculated according to Eq. (3), nm
a	8.687	8.680
b	8.083	8.071
c	15.193	15.153
β	90°	90°

3.5. Crystallite Size

To determine the size of crystallite, the diffraction patterns of the samples treated at 1573, 1673 and 1773 K were used, taking the most intense peak, which is located at approximately 30.1 (2θ). Debye-Scherrer equation[17] (Eq. 4) was used for the calculation:

$$D = \frac{0.9\lambda_{k\alpha}}{\beta_{2\theta}\cos\theta} \quad (4)$$

where D is the crystallite size, 0.9 is the shape factors for spherical particles, λ is the wavelength of radiation (1.5406 nm), $\beta_{2\theta}$ is the maximum width at half the maximum, and θ is the Bragg diffraction angle for each crystalline plane.

Table 3 shows the data collection for obtaining the final crystallite size. It is seen from the Table that the size of crystallite presented by the material at the temperature of 1573 K is 44.1256 nm, while at 1673 K this size increases by 1.2 % (44.6520 nm). The size presented by the sintered material at 1773 K (40.2865 nm) decreases by 8.7 % and 9.7 % compared to the size presented at 1573 and 1673 K, respectively.

Table 3. Data for calculating crystallite size at 1573, 1673 and 1773 K

Temperature, K	2θ	θ, rad	Crystallite size, nm
1573	30.0942	0.26262	44.1256
1673	30.1351	0.26297	44.6520
1773	30.1654	0.26324	40.2865

3.6. Microstructure

Fig. 4 shows the microstructure of CaAl$_2$O$_4$ taken with the scanning electron microscope. In this figure, it can be seen that the material presents grains in the form of flakes with a multimodal distribution of grain sizes,

ranging from approximately 30 microns to the sizes smaller than 1 micron. Porosity can also be observed in inter- and intra-granular positions. In general, a well consolidated and homogeneous sample is observed.

3.7. X-Ray Energy Dispersive Spectroscopy (EDS)

Fig. 5 shows the spectrum of the energy dispersive X-ray microanalysis (EDS) performed on the sintered sample at 1773 K. In this spectrum, the elements present in the sample (aluminum, calcium, oxygen, and carbon) are observed, the latter due to the carbon coating that was made to the sample to facilitate its study. It can be said that both aluminum and calcium are in the form of oxide according to the results of XRD analysis.

Scanning the sintered sample at 1773 K produced images of element distribution, also known as maps (Fig. 6), which help to reveal the surface topography of the sample and the distribution of the compound elements, which are aluminum, calcium, oxygen, and carbon. It can

be seen that there is a homogeneous distribution of all the elements that make up the compound.

3.8. Physical Properties

The density obtained in the refractory cement ($CaAl_2O_4$) is 3.08 g/cm^3, which is close to the data reported in the literature (2.99 g/cm^3). [18] On the other hand, the determined Vickers hardness was 430, which is also very similar to the value reported in the literature for this material.[18] To determine the resistance to thermal shock, different samples sintered at 1773 K were heated to the temperatures from 673 to 1373 K and suddenly cooled at room temperature in water. After cooling, the samples were under an optical microscope to observe the appearance or absence of cracks in the material. Fig. 7 shows the micrographs taken for different samples. It can be seen that all the samples have cracks on their surface, which were caused by a sudden change in temperature they suffered. It is obvious that at the temperature of 873 K and higher the material shows cracks.
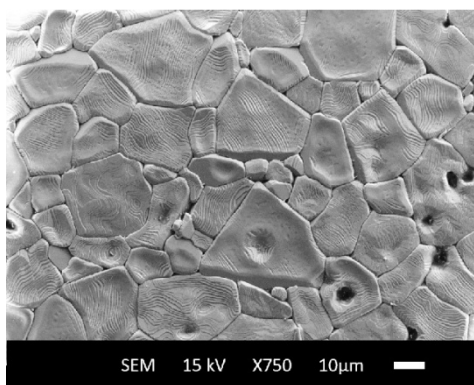

Fig. 4. Microstructure
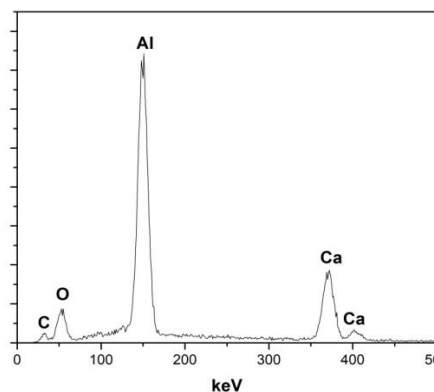of the sample sintered at 1773 K

Fig. 5. X-ray energy dispersive spectroscopy
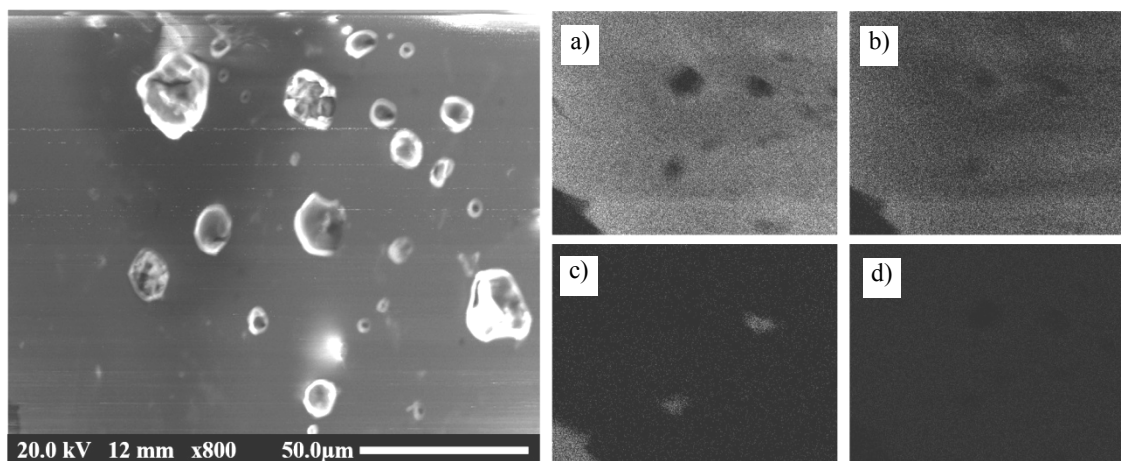of sintered sample at 1773 K

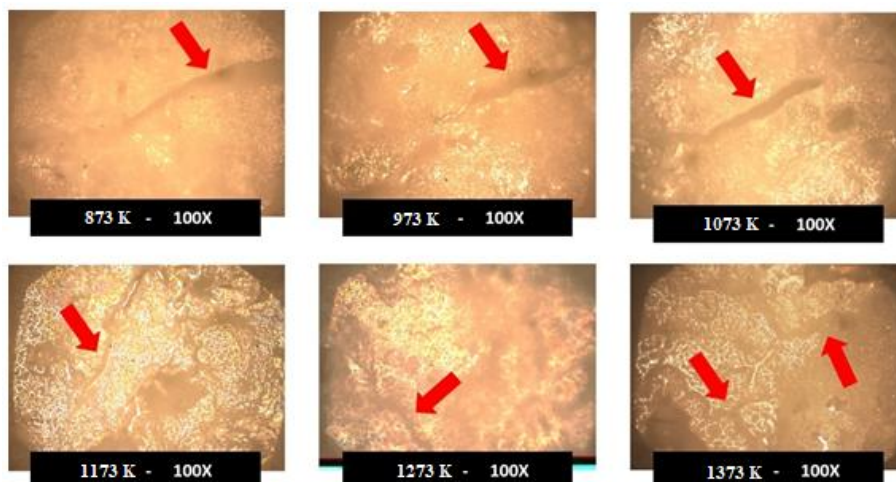**Fig. 6.** Mapping of the sintered sample at 1773 K: aluminum (a); calcium (b); carbon (c) and oxygen (d)

Fig. 7. Cracks presented in the cooled material under different temperatures

4. Conclusions

By means of TGA analysis it could be determined that the decomposition of $CaCO_3$ began at 883 K, and ended at approximately 1033 K. Through this analysis it was determined that the oxidation reaction of aluminum (RBAO process) occurred in two stages. The first one occurred at 773 K, when aluminum is in a solid state and another stronger one – at 1073 K, when it is already in the liquid phase. During the heating process there is a competition of two phenomena: weight gain due to the oxidation reactions of the aluminum and weight loss due to decomposition of the carbonate.

By means of X-ray diffraction analysis, it was determined that with the increase in temperature, Al begins to oxidize, and forms more Al_2O_3. Likewise, at 1023 K, the decomposition of $CaCO_3$ starts. $CaAl_2O_4$ began to form at 1423 K. During the heating stage, the reaction between CaO and Al_2O_3 occurs for the formation of $CaAl_2O_4$, and the formation of different species of calcium aluminate occurs according to the working temperature, achieving the desired compound ($CaAl_2O_4$) when the sample is treated at 1773 K.

The final product ($CaAl_2O_4$) presents the following lattice parameters: $a = 8.68$, $b = 8.0714$, $c = 15.1531$ and $\beta = 90°$, which are similar to the values reported in the literature. The calculated crystallite size was 40.28 nm.

As far as the resistance to thermal shock is concerned, the material shows cracks when cooling starting from temperature gradients of 873 K.

By means of high energy mechanical milling it was possible to obtain powders with sizes smaller than one micron, a fundamental characteristic that helped the material to obtain good physical properties such as density and hardness.

Acknowledgements

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ВИРОБНИЦТВО ЦЕМЕНТУ НА ОСНОВІ АЛЮМІНАТУ КАЛЬЦІЮ ЗА ДОПОМОГОЮ РЕАКЦІЙ В ТВЕРДОМУ СЕРЕДОВИЩІ

Анотація. *За допомогою порошкових методів та реакцій в твердому середовищі in situ виготовлено вогнетривкий цемент на основі CaAl$_2$O$_4$ з використанням CaCO$_3$, одержаного з шкаралупи курячих яєць, та Al як вихідних матеріалів. Для зменшення розміру частинок та досягнення однорідної суміші порошки піддавали високоенергетичному подрібненню на планетарному млині. Отримані порошки ущільнювали, утворюючи циліндричні таблетки і спікали у потоці повітря без тиску. За допомогою гранулометричного аналізу встановлено, що розмір зразків від декількох нанометрів до 2 мкм. За результатами диференціального термічного аналізу встановлено, що розкладання CaCO$_3$ починається за 953 К і закінчується за 1073 К. Цей факт підтверджений дифракційним аналізом який також вказує на те, що формування кристалічної фази CaAl$_2$O$_4$ завершується за 1773 К. Методом скануючої електронної мікроскопії встановлено мікроструктуру у вигляді рівноосних зерен у вигляді пластівців з розмірами від 1 до 2 мкм. Середня щільність та твердість матеріалу становила 3,08 г/см^3 та 430 HV відповідно. Проведено випробування на тепловий удар, і показано утворення тріщин матеріалу при охолодженні з градієнтами температури 873 К.*

Ключові слова: *вогнетривкий цемент, алюмінат кальцію, твердофазна реакція, високоглиноземний цемент.*