Synthesis Cordierite Materials Starting from Algerian Kaolin

M. Kheloui(✉), K. Boumchedda, S. Debbakh, and B. Rabehi

Department Engineering of Materials, URMPE, FSI, University of Boumerdes, Boumerdès, Algeria
khelouimeriem@yahoo.com

Abstract. The objective of this work is to study the synthesis of the cordierite phase from Algerian kaolin known as DD kaolin. To do this, the kaolin is washed and sieved to prepare a particle size fraction <20 μ, rich in clay particles, and precipitated magnesium hydroxide is prepared to give magnesium and finally silica fume is added to the mixtures experimental, to fill the silica deficit. The weighing of the materials is carried out in such a way as to produce mixtures close to the ideal phase of the cordierite (2MgO.2Al2O3.5SiO2). The mixing is carried out in a large quantity of water with a metal stirrer at high speed. The mixture is heated during stirring until the sludge is obtained. The final drying is continued in an oven, the coarse powder obtained is finely ground in a porcelain mortar.

The results of investigations carried out by simultaneous thermal analysis (ATD-TG) show a first exothermic peak at 945 °C. which is attributed to μ-cordierite, a second exothermic peak at 1200 °C. which indicates the crystallization of μ-cordierite in Indialite, and finally an endothermic peak above 1400 °C which indicates the fusion of the indialite.

The results of investigations of the X-ray diffraction analysis on the calcined powders at high temperature, shows that only the Indialite phase is present from 1300 °C.

Keywords: Cordierite · Kaolin · Synthesis · Ceramic · Sintering

1 Introduction

Cordierite ceramic is composed mainly of a phase corresponding to the formula ($2MgO.2Al_2O_3.5SiO_2$), it is one of the main ternary compounds in the MgO-Al_2O_3-SiO_2 system.

Cordierite materials still interests research development for two main reasons; (I) for its remarkable properties; Thermal shock resistance, chemical stability, and stability of dielectric properties. Cordierite is well known in catalytic converters of vehicles, and refractory applications, but also as a substrate material for integrated circuit boards. Recently cordierite showed of real performances in the filtration applications.

(II) Low cost manufacturing from natural raw materials available throughout the world. Natural raw materials often used are Kaolin [2], talc [3], stevensite [3], sepiolite

B. Abdelbaki et al. (Eds.): SMSD 2017, *Proceedings of the Third International Symposium on Materials and Sustainable Development*,, pp. 141–146, 2018.
https://doi.org/10.1007/978-3-319-89707-3_17

[4], gibbsite [5], and magnesite. To respect the stoichiometric composition formula of cordierite ($2MgO.2Al_2O_3.5SiO_2$), it's added magnesium hydroxyde, magnesium compounds, alumina [4], silica powder [5].

This study presents our research on the development of cordierite materials from abundant Algerian kaolin. This kaolin known as DD is located in the mountain "Djebel Debbagh" in eastern Algeria; it has a blackish coloring that limits its use in paper, luxury porcelain and sanitary articles where brightness and whiteness are required. The precipitated magnesium hydroxide is added to the starting mixtures to compensate for the magnesium deficiency, thus approaching the stoïchiometric formula of the cordierite.

2 Experimental Procedure

2.1 Preparation and Characterization of Materials

The kaolin DD is dissolved in water for several days to separate the clay particles from the other particles (sand, limestone, etc.), then the barbotine is sieved under 20 microns under tap water to give fraction of kaolin after decanting and drying. The characterization show DD kaolin is composed mainly by kaolinite and halloysite (> 80 wt.%), the other minerals are represented by gibbsite which is bearer of alumina, and by manganese compounds which gives blackish color to that raw material (Fig. 1).

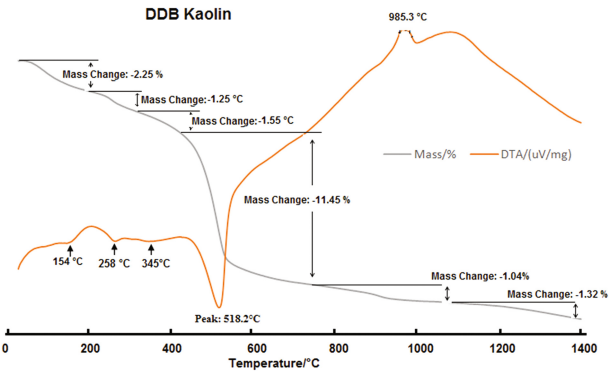

Fig. 1. ATD-TG of DDN kaolin

The chemical composition (Table 1) show that kaolin is very rich in alumina (Al_2O_3 > 46 wt.% at calcined state). The ignition loss exceed 17 wt.%, it's constituted mainly by H_2O because DD kaolin is poor in carbonates (CaO + MgO < 0.50%).

The simultaneous thermal analysis (TG/DTA) shows several transformations of DD kaolin. (1) First weight loss (−2.25 wt.%) corresponds to elimination of interlayer molecules H_20 of halloysite and todorokite. (2) Dehydratation of gibbsite is observed at 311.1 °C (−0.71 wt.%). (3) this weight loss may be attributed to the second dehydratation of todorokite. (4) Dehydroxylation of metahalloysite happens at 518 °C

Table 1. The chemical composition

Compounds	SiO_2	Al_2O_3	MnO	MgO	Other oxides	I. loss
DD black kaolin	53.60	46.25	1.68		<0.50	16.80
Magnesium hydroxyde	–	–	–	66	7	27
Amorphous silica	98	–	–	–	–	1

(−11.45 wt.%). The last weight losses (5, and 6) can be attributed to the reduction of manganese oxides. Exothermic peak observed at 985.3 °C can be attributed to the formation of spinel γ-Al_2O_3 and or mullite starting from metakaolinite.

The magnesium hydroxide ($Mg(OH)_2$) is precipitated from magnesium chloride solution by adding ammonia. Ammonia is added in excess (+5%) to guarantee the totality of the chemical reaction, and then the suspension is washed with distilled water several times until close to pH 7, for eliminate excess from ammonia. After decantation the magnesium hydroxide is dried at 100 °C and characterized.

STA analysis shows clearly shows the decomposition of $Mg(OH)_2$ at 390 °C, the associated loss of weight is 27.50% which gives a content of 88% $Mg(OH)_2$. The exothermic peak (680 °C) may be attributed to the crystallization of MgO (periclase). All peaks in the XRD analysis are attributed to periclase (Figs. 2 and 3).

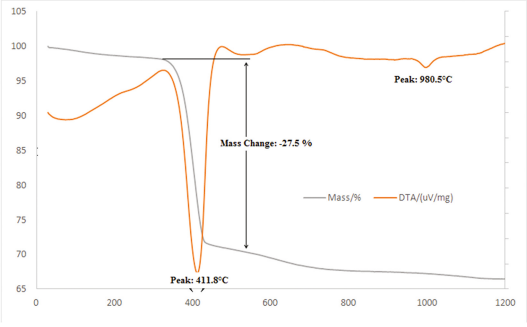

Fig. 2. DTA-TG curve of precipitated magnesium hydroxide

2.2 Preparation of Experimental Mixtures

The weighing of the starting materials is carried out in such a way as to produce mixtures close to the ideal phase of the cordierite (13.78%MgO.34.86%Al2O3.51.36% SiO2). The final calculation gives 72 g of kaolin, 20 g of magnesium hydroxide, and 12 g of silica fume (Table 2).

The mixing is carried out in a large quantity of water with a metal stirrer at high speed. The mixture is heated during stirring until the sludge is obtained. The final drying is continued in an oven, the coarse powder obtained is finely ground in a porcelain mortar.

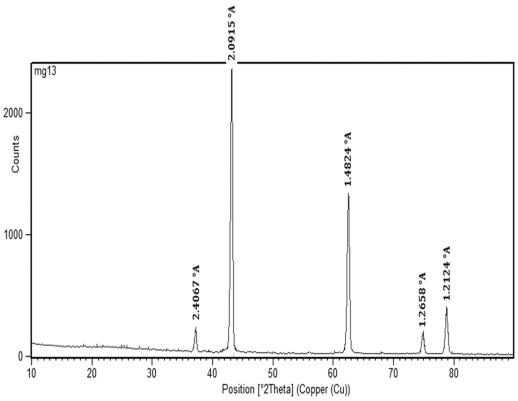

Fig. 3. XRD pattern of precipitated magnesium hydroxide calcined at 1000 °C

Table 2. Weight mixture of starting materials

Starting materials	SiO_2	Al_2O_3	MnO	MgO	OH
72% DD kaolin	38.60	33.12	1.20	0.08	8.24
20% Magnesium hydroxide	—	—	—	13.40	5.60
12% Amorphous silica	12	—	—	—	——
Total	50.60	33.12	1.20	13.48	

3 Results

3.1 Thermal Transformations of Starting Mixtures

According to the simultaneous thermal analysis, we observe that the mixtures with a MgO deficiency result in the crystallization of μ-cordierite at 940 °C followed by another crystallization around 980 °C which can be attributed to the mullite ($3Al_2O_3.2SiO_2$) (Fig. 4). Then, in the case where the mixture is close to the stoichiometric mixture of the cordierite, we observe the 2 exothermic peaks that of μ-cordierite at 940 °C, and that of

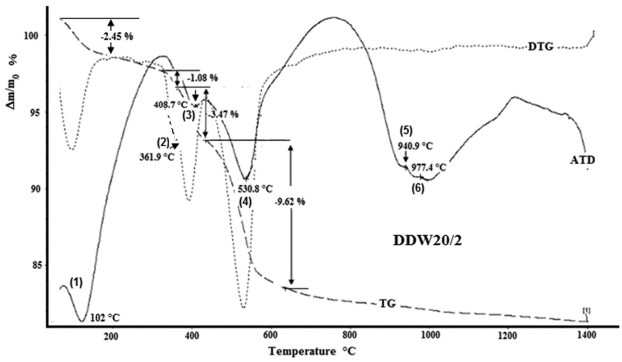

Fig. 4. ATD-TG of firstly mixture

the indialite from μ-cordierite at 1200 °C. The peak of the mullite has completely disappeared (Fig. 5).

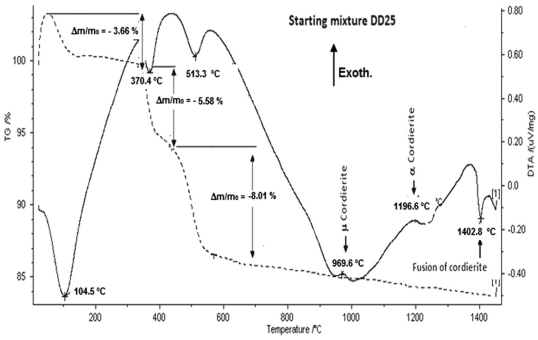

Fig. 5. ATD-TG of stoichiometric mixture

It appears that stoichiometric mixtures give only cordierite, while the other mixing compositions make it possible to synthesize cordierite-mullite composites.

The investigations of the thermal transformations carried out on the calcined stoichiometric mixture at 1000 °C, 1200 °C and 1300 °C for 2 h, show that the appearance of the indialite phase begins at 1200 °C with the presence of cristobalite. From 1300 °C, cristobalite decreases in intensity whereas the crystallization of indialite is perfectly observable (Fig. 6).

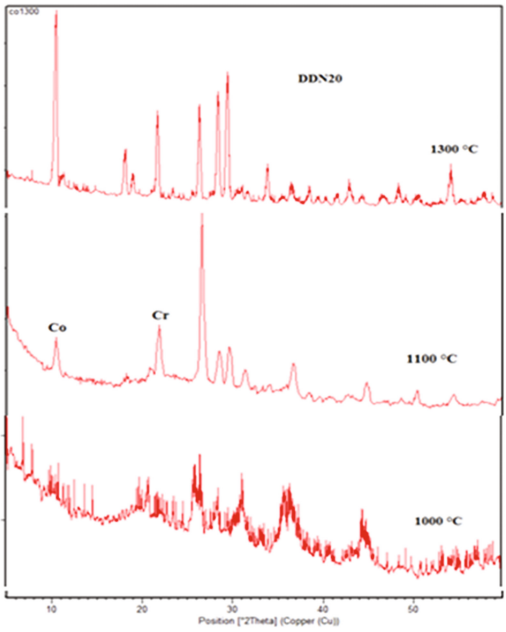

Fig. 6. XRD analysis of stoichiometric mixture calcined at 1000 °C, 1100 °C, and 1300 °C

4 Conclusion

In this study the cordierite ceramic was synthesized from a mixture of magnesium kaolin and hydroxide ($Mg(OH)_2$) precipitated from a magnesium chloride solution by adding ammonia to ensure its stoichiometric composition. The crystallization of the μ cordierite and α Indialite is well defined at 940 °C and 1200 °C respectively.

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