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Influence of sintering temperature on the microstructural and mechanical properties of cordierite synthesized from andalusite and talc

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ABSTRACT

Cordierite-based ceramic has been synthesized by solid state process from andalusite and talc mixture sintered at 1300 and 1350 °C for 2 h. The phase, microstructural and thermal characterizations of the samples were investigated using X-ray diffraction (XRD), scanning electron microscope (SEM), dilatometric and thermal analysis (TG/DTA). Brazilian and three-point bending tests were performed to evaluate the mechanical properties. XRD results showed a significant improvement in the cordierite formation at 1350 °C, whereas the sintering at 1300 °C was insufficient. The SEM observation showed that the sample sintered at 1350 °C was relatively dense. As a result of this densification, the flexural and tensile strengths at 1300 °C (15.4 ± 1.2 and 12.2 ± 1.9 MPa, respectively) were significantly enhanced with increasing temperature to 1350 °C (32 ± 4.2 and 18.3 ± 3.6 MPa, respectively).

1. Introduction

The cordierite-ceramics ($2MgO.2Al_2O_3.5SiO_2$) due to their low thermal expansion coefficient, low dielectric constant, high resistance to thermal shock and high refractoriness are promising candidate in a variety of areas. The most important applications of cordierite are: electronic components [1], industrial foundry [2], diesel emissions control [3], catalyst support [4], automobile exhaust purification [5] and membrane support [6]. Several techniques have been developed to produce cordierite including: i) solid-state reaction of MgO, Al_2O_3 and SiO_2 or their precursors and ii) wet-chemical methods such as sol-gel, co-precipitation, hydrothermal synthesis and glass crystallization [7–9]. The cordierite synthesis via solid-state reaction has been considered as the most-widely used in the industrial field, due to its simplicity and low cost. The major advantage for cordierite manufacturing using this method is the exploitation of abundant and inexpensive raw materials. However, this method necessitates very high temperatures which result generally the appearance of cracks and deterioration of the mechanical properties. The sintering via solid-state reaction can be improved by the synthesis of dense cordierite using some additives such as zirconia (ZrO_2) or titania (TiO_2) [10,11]. The sol-gel process has as main advantage the reduction of the sintering temperature [12]. However, this method is relatively expensive because the price of starting compounds and solvents is high.

The raw materials frequently used for the cordierite synthesis via solid-state sintering are talc, kaolinite, sepiolite, fly ash, diatomite and feldspar [13–17]. Recently, we have reported results on the synthesis of porous cordierite from stevensite-rich clay, andalusite and oil shale [18,19]. The main objective of this study is to produce cordierite ceramic from talc (source of $MgO–SiO_2$) and andalusite (source of $Al_2O_3–SiO_2$). The effect of sintering temperature on the phase formation and microstructure evolution have been studied and described below.

2. Materials and methods

2.1. Materials

The phase analysis was carried out by X-ray diffraction (XRD) technique using a Siemens D500 diffractometer (Cu Kα radiation $\lambda = 1.5406$ Å). All samples were scanned in the 2θ range 3–80° with step size of 0.03°/min. The thermal analysis (TG/DTA) was performed

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in a Setaram SETSYS 24 from ambient temperature to 1200 °C with a heating rate of 10 °C/min. The dilatometric study was performed up to 1200 °C at a heating rate of 5 °C/min (NETZSCH DIL 402 PC). The microstructure of the sintered samples was observed by scanning electron microscope (SEM Stereoscan S260). The chemical compositions of the talc and andalusite determined by X-ray fluorescence are given in Table 1.

The three-point bending and Brazilian tests were performed on rectangular samples (length=50 mm, width=20 mm, and thickness=4.5 mm) using a universal testing machine (Ametek Lloyd Instruments), with a displacement rate of 0.1 mm/min. The Brazilian tests were performed on cylindrical samples (diameter=22 mm and height=6 mm).

The flexural strength (σ_f) and tensile strength (σ_t) were evaluated according to the equations:

$$\sigma_f = \frac{3}{2}\frac{F_f L}{w t^2} \quad (1)$$

$$\sigma_t = \frac{F_t}{S} = \frac{2F_t}{\pi D h} \quad (2)$$

where "F_f and F_t" are the load (N); "w" and "t" are the width and thickness of the specimen (mm); "L" is the span length (24 mm); "D" is the diameter and "h" is the height (mm).

Table 1
Chemical composition of the talc and andalusite (wt%).

Material	SiO_2	Al_2O_3	Fe_2O_3	CaO	MgO	K_2O	Na_2O	LOI
Talc	57.8	2.8	0.8	0.3	31.9	0.1	1.1	6.2
Andalusite	38.5	57.1	1.4	0.1	0.1	0.2	0.05	0.6

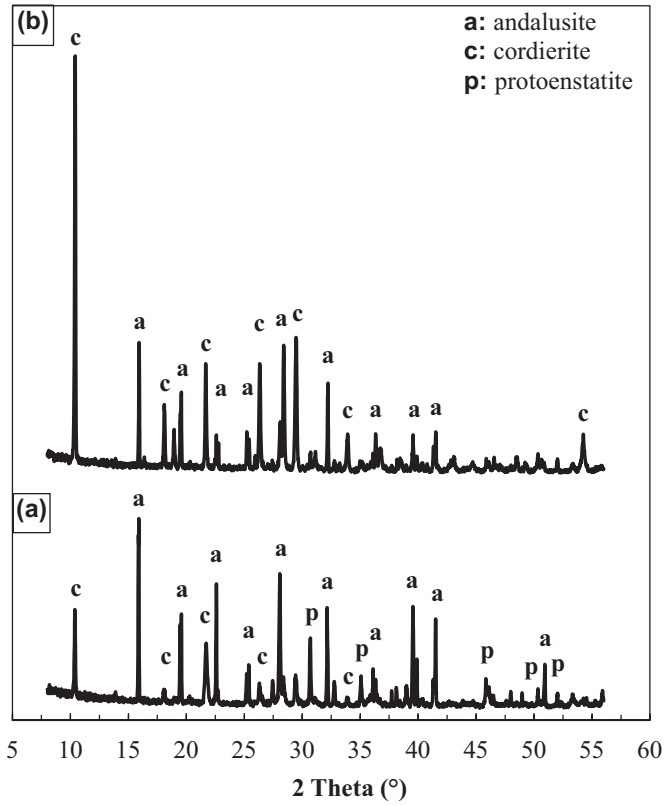

Fig. 1. XRD patterns of the mixtures sintered at: (a) 1300 °C and (b) 1350 °C.

3. Results and discussion

3.1. Phase, thermal, and microstructural characterization

The XRD results showed that the main phase formed at 1300 °C (Fig. 1a) was andalusite (pdf #04-009-4464) with a significant amount of cordierite (pdf #04-016-0633) and protoenstatite (pdf #04-007-8621). This is probably due to the insufficient sintering temperature to form cordierite as major phase. The appearance of protoenstatite can be attributed to the thermal decomposition of talc ($Mg_6Si_8O_{20}(OH)_4$) (Eq. (3)), as confirmed by Wesołowski et al. [20].

The formation of cordierite ($Mg_2Al_4Si_5O_{18}$) may be related to the reaction between andalusite (Al_2SiO_5), protoenstatite ($MgSiO_3$) and amorphous silica (Eq. (4)).

$$Mg_6Si_8O_{20}(OH)_4 \rightarrow 6(MgSiO_3) + 2SiO_2 + 2H_2O(g) \quad (3)$$

$$2(Al_2SiO_5) + 2(MgSiO_3) + SiO_2 \rightarrow (Mg_2Al_4Si_5O_{18}) \quad (4)$$

However, the main phase observed after increasing temperature to 1350 °C was cordierite with a significant decrease in the intensity of andalusite reflections (Fig. 1b). As well as, the protoenstatite reflections were totally disappeared at 1350 °C. This remarkable improvement in the cordierite formation is probably due to the thermal decomposition of residual andalusite to mullite ($Al_6Si_2O_{13}$) and silica (Eq. (5)). Then, these two intermediate phases can react with protoenstatite to improve the cordierite formation (Eq. (6)).

$$3(Al_2SiO_5) \rightarrow (Al_6Si_2O_{13}) + SiO_2 \quad (5)$$

$$2(Al_6Si_2O_{13}) + 6(MgSiO_3) + 5SiO_2 \rightarrow 3(Mg_2Al_4Si_5O_{18}) \quad (6)$$

The DTA and TGA curves for talc and andalusite mixture heated up to 1200 °C at a heating rate of 10 °C/min are shown in Fig. 2. From the TG results, only a small mass loss attributed to the dehydration of talc was observed in the temperature range of 30–500 °C. The dehydroxylation process of talc takes place in the temperature range of 500–820 °C and the equivalent mass loss was 3.3 wt%. The endothermic peak centered at 870 °C can be assigned to the thermal decomposition of talc, in agreement with XRD results (Fig. 1). However, the andalusite begins its thermal decomposition to mullite and silica at higher temperatures (exceeding 1250 °C) (Eq. (5)). Kakroudi et al. [21] showed that the mullitisation process of andalusite used in this work took place at about 1280 °C. The last endothermic peak at 1150 °C,

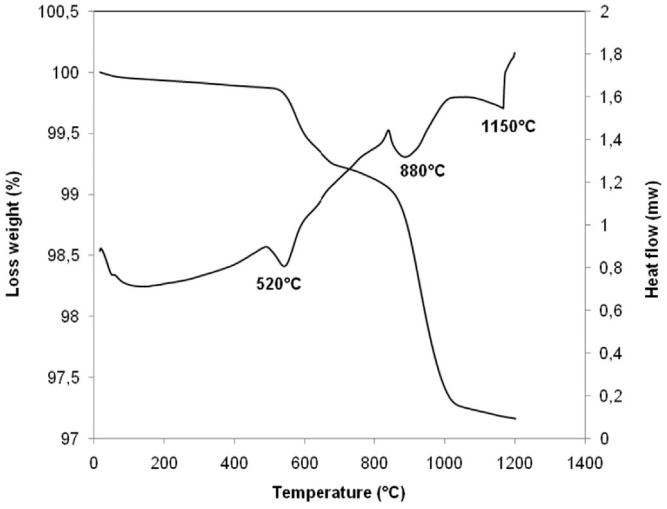

Fig. 2. DTA/DTG curves of the mixture.