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Preparation and characterization of cordierite powders

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Abstract

The current paper presents different preparation methods for cordierite ceramic powders: solid state reactions and wet preparation. In the solid state method, γ-Al_2O_3, SiO_2 and $Mg(CO_3)_2$ were used as raw materials. For wet preparation, the following compounds were used as precursors: silica gel, $AlCl_3{\cdot}6H_2O$ and $Mg(NO_3)_2{\cdot}6H_2O$. The powders obtained by the different methods employed were characterized in what concerns mineralogical composition and granulometric distribution. It was determined that the results are influenced by the nature of raw materials used and the processing conditions.

Keywords: Powders-solid state reaction; Powders-chemical preparations; X-ray methods; Silicate

1. Introduction

Cordierite ceramics have been extensively studied in the last decades, due to their excellent properties, low thermal coefficient and dielectric constant, as well as high chemical and thermal stability.[1] These ceramics are used in various industrial fields, in applications such as refractories, electrical, thermal or phonic insulation, filters, membranes, heating elements, microwave absorbents, electromagnetic waves absorbents, etc.[2]

Cordierite exists in three polymorphs: α-cordierite (indialite), stable between 1450 and 1460 °C, β-cordierite, stable at temperatures lower than 1450 °C and μ-cordierite, metastable.[3]

Sugiura and Kuroda[4] and Evans et al.[5] have measured through X-ray diffractometry the thermal expansion coefficient of cordierite on different crystallographic axes.

These data are showing its important anisotropy and explains the low thermal expansion coefficient, through the negative thermal expansion along the "*c*" crystallographic axis. This property it is extremely important, conferring to cordierite high thermal shock resistance.

Solid state, sol–gel or co-precipitation reactions can be used for preparing cordierite, resulting powders with different purities.[6,7]

In the present work, wet and solid state methods were employed for the preparation of cordierite powders, starting from different types of raw materials. The characterization of the obtained ceramic powders showed that their properties depend on the parameters of the method used.

2. Experimental procedures

The methods used for preparing cordierite ceramic powder will be briefly described in the following. The raw materials were mixed in stoichiometric proportions for obtaining cordierite.

2.1. Preparation of cordierite powder through solid state reactions—cordierite I

The composition of the raw materials is presented in Table 1.

After dosing, the raw materials were homogenized in a planetary mill and then calcinated at 750 °C for 1 h, for eliminating the volatile compounds. In order to obtain the cordierite powder, thermal treatments at temperatures between 1050 and 1400 °C were realized. The ceramic powder prepared was then grounded in a planetary mill with alumina balls, for 4 h, at 180 rpm.

2.2. Preparation of cordierite powder from solutions—cordierite II

The raw materials were brought in solution or suspension, using distilled water.

First, the solution of $AlCl_3{\cdot}6H_2O$ and suspension of SiO_2 were mixed and homogenized in the planetary mill with alumina

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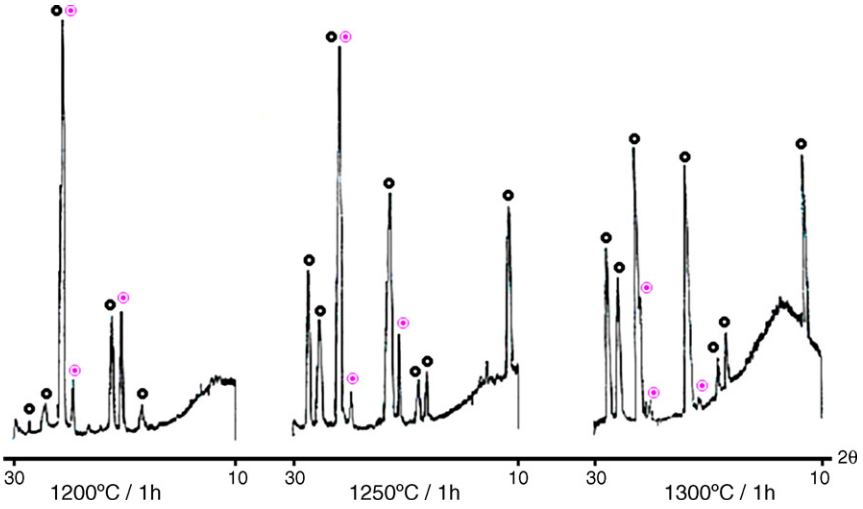

Fig. 1. X-ray diffraction for cordierite powder CI (◉) SiO_2; (○) $2MgO·2Al_2O_3·5SiO_2$.

Table 1
Raw materials used

Material	Raw materials (wt.%)		
	SiO_2	γ-Al_2O_3	$MgCO_3$
CI	50	35	28

balls for 1 h, at 180 rpm. The solution of $Mg(NO_3)_2·6H_2O$ was then added and once again the mixture was homogenized. In order to create the necessary conditions for precipitation, the pH was increased from 9.5 to 11 using ammonia.[3]

The precipitate was dried for 24 h, at 40 and 120 °C, respectively, and then milled in ethanol for 1 h, at 180 rpm. Two thermal treatments were performed, one at 950 °C, for 1.5 h in order to eliminate the volatile compounds and the other at temperatures between 1050 and 1400 °C, for the synthesis of cordierite.

3. Results and discussions

The cordierite powders were characterized through X-ray diffraction and laser granulometry.

3.1. *X-ray diffraction*

X-ray diffraction analyses performed on cordierite I ceramic powders treated at 1200, 1300 °C, respectively, 1350 °C have shown the presence of two components: cordierite and silica (Fig. 1). The diffraction peaks of cordierite increase with the increase in temperature, while those of silica decrease in intensity.

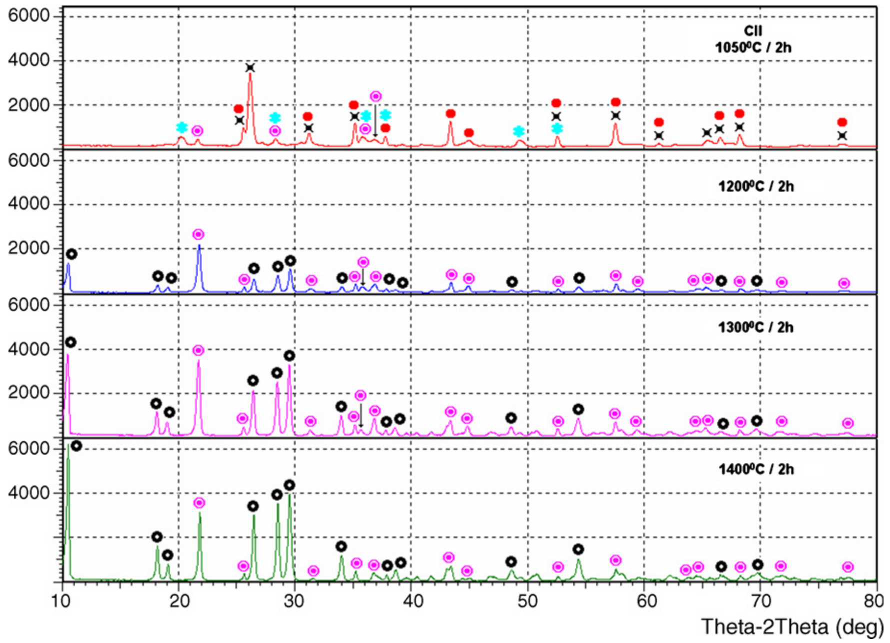

Fig. 2. Diffraction spectra for cordierite powder CII, thermally treated at 1050–1400 °C, for 2 h (●) α-Al_2O_3 [ASTM 46-1212]; (✖) A_3S_2 [ASTM 15-0776]; (✱) $2MgO·SiO_2$ [ASTM 74-1685]; (◉) SiO_2–cristobalite [ASTM 39-1425]; (○) $2MgO·2Al_2O_3·5SiO_2$ [ASTM 82-1541].

Table 2
Granulometric characteristics for the cordierite powder

Material	Milling time (h)	Milling medium	Average diameter (μm)	Specific surface (m^2/g)
CI	4	Dry	3.6	3.84
CII	1	Wet	71.8	0.23

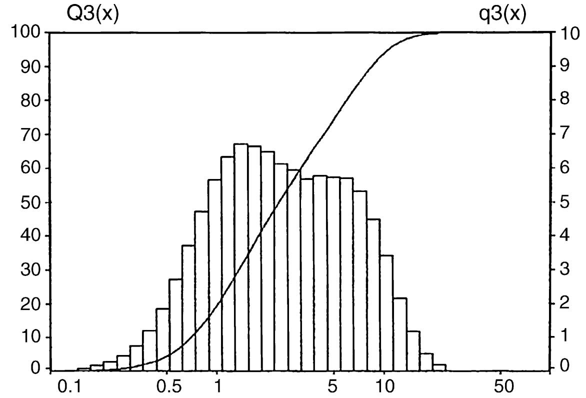

Fig. 3. Granulometric distribution of cordierite powders: (a) CI and (b) CII.

Fig. 2 presents the diffraction spectra for cordierite II powder, obtained from solutions, for different thermal treatment temperatures. The cordierite is not formed at 1050 °C, at this temperature mullite—as the main phase, and also forsterite, alumina and silica being identified, through their characteristic diffraction peaks. The magnesium silicate and alumina silicate were formed through chemical reactions from the inorganic precursors.

At 1200 °C the mineralogical composition of the ceramic powder changes, with formation of α-cordierite. Alumina, mullite and enstatite can no longer be detected, being probably consumed in the cordierite forming reactions. The diffraction interferences characteristic to silica are increasing.

However, at higher temperatures, the diffraction peaks characteristic to cristobalite are lowering, cordierite becoming the predominant phase.

3.2. Laser granulometry

The ceramic powders prepared were characterized in what it concerns the granulometric distribution, by laser granulometry analysis. The results are presented in Table 2 and Fig. 3.

The granulometric distribution for the cordierite I powder is relatively broad, showing particle dimensions between 0.1 and 50 μm. Another characteristic would be a maximum in particle dimension of about 2 μm. The cordierite II powder is constituted of particles with dimensions between 10 and 263 μm, with some particles between 1 and 10 μm. The granulometric distribution is characterized by a maximum of about 100 μm. The higher particle dimensions for the CII powder can be explained by its higher tendency to form agglomerates and also through the different milling parameters.

4. Conclusions

Cordierite ceramic powders were prepared both by solid state reactions (CI), from SiO_2, γ-Al_2O_3, $MgCO_3$ and from solutions (CII), using $Mg(NO_3)_2 \cdot 6H_2O$, $AlCl_3 \cdot 6H_2O$ and SiO_2 as raw materials. The powders were thermally treated at 750 and 950 °C, respectively, for the decomposition of the volatile compounds, and then at temperatures between 1050 and 1400 °C for the synthesis of cordierite. X-ray diffraction methods proved the apparition of cordierite at temperatures as low as 1200 °C in the presence of cristobalite silica. At higher temperatures, cordierite was identified as the main phase. The granulometric analysis shows that by a 4-h milling of the powder obtained in the traditional way, the granules obtained have dimensions as low as 3.6 μm, and a specific surface as high as 3.84 m^2/g.

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