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Effect of mechanical activation and microwave sintering on crystallization and mechanical strength of cordierite nanograins

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Abstract

In this study mechanical activated powders were synthesized by high-energy ball milling of starting powders containing 34.86 wt% Al_2O_3, 51.36 wt% SiO_2, and 13.78 wt% MgO. The activated powders were then sintered by either conventional or microwave sintering methods at temperatures between 900 and 1400 °C and the crystallization and sintering behavior of the samples were investigated. SEM observations showed that microwave sintering of the 30 h milled sample led to a microstructure consisting of equiaxed nanograins with average grain size of 38 nm. The specimens that were sintered by the microwave method exhibited lower porosity, higher density and higher bending strength compared to those sintered by the conventional method. The mechanical strength of the materials was evaluated by three-point bending test and the optimum sample, which was milled for 30 h and microwave sintered at 1390 °C, showed a maximum strength of 110 ± 5 MPa.
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Keywords: Microwave sintering; Mechanical activation; Microstructure; Bending strength; Cordierite

1. Introduction

Cordierite is one of the most important phases of the MgO-SiO_2-Al_2O_3 system, which has low thermal expansion coefficient, excellent thermal shock resistance, low dielectric constant, high volume resistivity, high chemical durability, and relatively high refractoriness and mechanical strength. These ceramics are, therefore, widely used as honeycomb-shaped catalyst carriers in automobile exhaust systems, substrate materials in integrated circuit boards, and also as refractory materials [1–3].

There are several methods for synthesizing cordierite including solid-state reaction, sol–gel, and crystallization of glass. Among these methods, sintering of oxide powders through solid-state reactions has gained the highest interest [4–7]. Microwave has been used as an efficient heating source for the synthesis and densification of ceramics at low temperatures and short times due to its uniform and rapid heating capability resulting from the direct absorption of energy into materials [8–11]. In addition, the densification in some ceramic materials has been observed [12–17] to get accelerated when sintered in a microwave furnace.

On the other hand, mechanical activation processes have been widely used to enhance the reactivity of materials by particle size reduction and amorphization process. This process overcomes the kinetic constraints and the demand for high processing temperatures, which are common in conventional solid-state synthesis methods. Moreover, the mechanical activation of powders promotes the rate of diffusion of reactants across the phase boundaries and is a way to increase the kinetics of the solid-state reactions [18]. Many researchers have also emphasized the efficiency of mechanical milling of precursors in the densification and final amount of relevant phases in sintered bodies [19–22].

The main objective of this work is to investigate the effect of mechanical activation as well as the sintering method of cordierite composition on the crystallization, densification, microstructure, and flexural strength of the final product.

2. Experimental procedures

MgO (РЕАХИМ, 1 21239, ГОСТ 4526-75, d_{50} =0.3–0.4 mm, purity ≈98%), Al_2O_3 (reactive grade alumina MR70, d_{50} =0.5

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–0.8 μm, purity ≈99.8%), and SiO_2 (Setabran, Iran, d_{50} =0.1–0.8 μm, purity ≈99.9%) powders were used as the starting materials. A stoichiometric cordierite composition containing 13.78 wt.% MgO, 34.86 wt.% Al_2O_3, and 51.36 wt.% SiO_2 was wet milled in a planetary mill using a plastic bottle and alumina balls with the average size of 15 mm and ethanol as the milling media for 1 h. The obtained slurry was first dried on a magnetic heater-stirrer and then in an oven for 24 h at 120 °C.

Mechanical milling experiment was performed on 44 g of the precursor powder using a high-energy ball mill, which had an alumina ball to powder mass ratio of 15:1. The planetary mill pot was rotated at about 270 rpm around the common axis. The powder mixtures were also ground for three different times of 10, 20, and 30 h. The weight loss of the alumina balls was measured before and after each milling run. After 10, 20, and 30 h of milling, weight loss of the alumina balls was 3.77, 5.65, and 7.11 g, respectively. Extra amounts of SiO_2 and MgO were added to the powder mixture to compensate for this increase in the alumina content and to maintain the stoichiometric ratio of the cordierite.

Rectangular samples were fabricated using a uniaxial hydraulic press with pressure of 220 MPa and a steel die with the dimensions of 5 mm × 5 mm × 25 mm. The samples were then sintered at a temperature between 900 and 1400 °C using either a microwave furnace (2.45 GHz and 900W) without soaking time or an electrical furnace with soaking time of 2 h. In the microwave method, an SiC crucible was used as the susceptor. All the runs were performed by fast heating up to the maximum temperatures measured by an optical pyrometer (Model: RAYR312MSCL2G) [23]. Mean heating rates in the microwave and electrical furnaces were approximately 37 and 10 °C/min, respectively.

Porosity and bulk density of the sintered specimens were measured by the Archimedes method (ASTM C373–88) using deionized water at ambient temperature. Flexural strength of the sintered samples was measured using Instron 1923. At least five specimens were tested to ensure the repeatability of the results. X-ray diffraction (XRD) (Siemens, D500 system, CuK_α radiation, 30 kV, 25 mA, and 0.5°/sec) was used to identify the phases present in the milled powders, before and after the heating. Surface morphology was investigated using VEGA\\ TESCAN scanning electron microscope on the powder and etched samples. The agglomerate size of the powders milled for 1, 10, 20, and 30 h was determined using scanning electron microscope (SEM). Moreover, at least 50 micrographs containing more than five particles or agglomerates were utilized for size measurement and the average values are reported.

3. Results and discussion

Fig. 1 shows the morphology of the adopted cordierite composition milled for different times. According to Fig. 1a, the powder that was milled for 1 h had an average particle size of about 1.1 μm. Fig. 1b shows the powder that was milled for 10 h. It can be seen in this figure that some particles retained their shapes, and some smaller particles attached to the surface of other bigger ones.

Thereafter, spherical particles associated with heterogeneous agglomerates appeared with increasing milling time up to 20 h (Fig. 1c). After 30 h of milling (Fig. 1d), the average size of both the particles and the agglomerates decreased. Average agglomerate sizes of the cordierite composition were 485, 220, and 130 nm after 10, 20, and 30 h of milling, respectively.

XRD patterns of the powders (Fig. 2) indicated the peaks of quartz (File 01-085-0795), alumina (File 00-005-0712), and magnesia (File 01-087-0652) as the only crystalline phases present in the powder after 1 h of milling, which was similar to that of the starting powder. As can be seen in Fig. 2, the magnesia peaks disappeared after 30 h of milling. The disappearance of the magnesia peaks and the decrease in the intensity of the alumina and quartz peaks indicated the possibility of formation of an amorphous phase. However, alumina and quartz were the only crystalline phases present in the powder after 30 h of milling.

The powders milled for 1 h were pressed and then heated at different temperatures in the microwave (MW) and conventional (CS) furnaces. Based on Fig. 3, cordierite (File 01-085-1722) did not form in the samples sintered at temperatures lower than 1200 °C in both MW and CS furnaces. The XRD patterns of the MW samples milled at different times and sintered at 1200 °C (Fig. 4) confirmed that the microwave process resulted in the formation of cordierite as the major phase. The intensity of the cordierite peaks increased with the increase in the milling time.

In the conventional sintering of the samples milled for 1, 10, and 20 h (Fig. 5), even after sintering at 1200 °C for 2 h, no cordierite peaks were observed. Cordierite was observed in these specimens only when milled for 30 h and sintered at 1200 °C for 2 h. Figs. 4 and 5 revealed that the intensity of the cordierite peaks in the MW samples was much higher than that of the cordierite peaks in the CS samples at the same sintering temperature of 1200 °C. The obtained peaks were identified using the JCPDS cards of 00-003-0523 and 01-082-2424 for Proto-enstatite and spinel, respectively.

The bulk density and porosity of the MW and CS specimens milled for 1, 10, 20, and 30 h and sintered above 1200 °C are shown in Figs. 6 and 7, respectively. According to these figures, it can be concluded that at the same sintering temperature, microwave processing led to higher density and lower porosity than the conventional one. After 30 h of milling, the samples sintered at 1390 °C in the microwave (without soaking) and the conventional (held for 2 h) furnaces showed a bulk density of 2.49 and 2.38 g/cm^3, respectively. With both heating methods, the density increased and the porosity of the sintered samples decreased with the increase in the milling time. MW samples exhibited higher densification compared to the CS samples, whereas, the density trends showed some fluctuations in the former and a steady increase in the latter. The main reason for the decrease and the increase in the density while increasing the sintering temperature in the microwave processing was attributed to the enhancement of the cordierite, having a low density (2.5 g/cm^3) instead of the higher density materials such as alumina, silica, and magnesia (3.99, 2.65 and 3.58 g/cm^3, respectively) and the formation of liquid phase at high temperatures, respectively. As further revealed in Fig. 6, the crystallization of the cordierite

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