



Fabrication and characterization of porous cordierite ceramics prepared from ferrochromium slag

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Abstract

Porous cordierite ceramics were successfully fabricated using ferrochromium slag, commercial alumina and silica powder as raw materials, without any additional pore forming agents, by sintering at temperatures ranging from 1100 to 1350 °C. The phase evolution, porosity, microstructure, mechanical properties, thermal expansion property and chromium leachability were systematically investigated. The results showed that the internal oxidation of forsterite in the slag contributed to the massive glassy phases formed in the prepared porous ceramics as well as the low-temperature sintering synthesis of cordierite, which started to form at 1100 °C and at 1350 °C, comprising up to ~87.1 wt% of the ceramic. Furthermore, the iron oxides in the ferrochromium slag acted as pore forming agents at high temperatures, thereby resulting in significant volume expansion of the sample. The SEM images showed that the prepared cordierite ceramics had a porous microstructure composed of massive glassy phases embedded in polyhedral spinel and prismatic cordierite. In addition, the ceramic had a flexural strength of 47.26 ± 1.01 MPa, coefficient of thermal expansion of $3.5 \times 10^{-6}/^{\circ}\text{C}$, and chromium leachability that was only half as large as that of the green sample.

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1. Introduction

Porous ceramics materials have many interesting properties including low density, high permeability and good heat insulation. These characteristics make them widely used in various industrial fields such as liquid or gas fillers, catalysis supports, selective membranes, thermal insulation and pollution control [1]. Among those materials, porous cordierite is a very good candidate for these applications due to its low thermal expansion coefficient and high resistance to thermal shock [2,3]. Porous cordierite is synthesized primarily via a solid-state sintering method, using talc, kaolin, magnesite and other natural minerals or the pure chemical oxide as the raw materials. Sintering temperatures of up to ~1400 °C are typically used

[4–6]. Attempts to lower the sintering temperature have included the use of sintering additives [7,8] and the development of novel synthesis methods [9–11]. However, these attempts incur high fabrication costs and require a complex preparation process. Finding a meaningful way to use inexpensive and rich-sourced raw materials, especially the industrial byproducts, to synthesize porous cordierite ceramics is therefore essential. Some studies have focused on the preparation of porous cordierite ceramics using sepiolite, rectorite and vermiculite as raw materials [12–14]; industrial byproducts such as rice husk, and fly ash have also been used [15,16]. There are, however, no published reports regarding porous cordierite ceramics prepared mainly from ferrochromium slag.

Ferrochromium slag is a waste material produced during the manufacturing process of ferrochrome that is an essential component in stainless steel. The massive ferrochromium slag produced every year occupies significant land space and contaminates the environment owing to the presence of leachable heavy metals, especially chromium [17,18]. Effective use

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of the slag is therefore essential. Many approaches for reusing the slag have been reported. In fact, most of the slag is used for traditional construction materials, such as concrete [19,20] and brick [21]. Unfortunately, with these approaches, the slag is simply moved from one place to another and the potential hazard of the slag remains.

Ferrochromium slag has a similar chemical composition to cordierite. Both contain mainly silicon (Si), manganese (Mg), and aluminum (Al), which together constitute ~83% of the slag [19]. The slag also contains metallic elements such as iron (Fe), titanium (Ti), and heavy metals such as chromium (Cr), cobalt (Co) and nickel (Ni). These components are expected to act as sintering additives and have a positive effect on the phase evolution as well as the crystallization of cordierite [22–24]. In addition, the high-temperature reduction of iron oxides in the slag generates a large amount of oxygen, which acts as a pore forming agent [25]. Therefore, in this study, in order to carry out an innocuous treatment of ferrochromium slag, porous cordierite ceramics were prepared from a mixture of the slag, commercial alumina (Al_2O_3), and silica powder (SiO_2) without additional pore forming agents. The phase evolution, volume expansion, porosity, microstructure, mechanical properties, thermal expansion behavior, and Cr leachability, of the prepared ceramics were evaluated. Furthermore, the phase evolution during sintering is discussed from the viewpoint of chemical reactions of the various MgO (FeO)– Al_2O_3 – SiO_2 compositions.

2. Experimental procedure

2.1. Sample preparation

The porous cordierite ceramics were prepared from raw materials of ferrochromium slag (Leshan, Sichuan Province, China), commercial alumina and silica powder (both from Zibo Jiezhong New Material Co., Ltd., China). Cordierite having the composition $2\text{MgO} \cdot 2\text{Al}_2\text{O}_3 \cdot 5\text{SiO}_2$, was prepared by weighing and mixing ferrochromium slag, alumina and silica powder for 6 min at room temperature in a vibration mill rotating at a constant speed of 710 rpm. The milled powder mixture was then mixed with organic binder PVA-1750 (3 wt% solution) and uniaxially pressed under 45 MPa into 25 mm(diameter) \times 5 mm (thickness) pellets (weighing ~5.5 g each). The green pellets were subsequently dried at $105 \pm 2^\circ\text{C}$ in a baking oven in order to remove the free water, and then sintered for 3 h in a muffle furnace at temperatures ranging from 1100 to 1400 °C. In addition, the green pellets were sintered at 1350 °C for 0.5, 1.0, 2.0 and 4.0 h, respectively, in order to investigate the effect of holding time on the properties of the cordierite. The heating rate was fixed at 5 °C/min, and the added organic binder was removed by holding the sample at 600 °C for 20 min. The sintered pellets were furnace-cooled to room temperature.

2.2. Characterization

The chemical compositions of the raw materials were determined via quantitative X-ray fluorescence spectrum analysis (XRF; Axios-Advanced, PANalytical Corporation, The Netherlands). The

corresponding particle size distributions were determined by a laser particle size analyzer (Mastersizer 2000, Malvern Instruments Ltd., UK) using water as a dispersing medium. Moreover, X-ray diffraction (XRD; X'Pert Pro, PANalytical Corporation, The Netherlands) was used to determine the phase compositions of the raw materials and the sintered samples. The actual phase fractions were obtained via quantitative Rietveld analysis using the HighScore Plus software [26,27]. Furthermore, the morphology of the slag and the fracture surfaces of the porous cordierite ceramics were examined by using a field emission scanning electron microscope (FESEM; Ultra 55, Carl Zeiss Ltd., Germany); electron energy disperse spectroscopy (EDS) was performed with equipment attached to the microscope.

The coefficient of thermal expansion (CTE) of the sintered samples was evaluated at temperatures between room temperature and 1000 °C by using a thermal dilatometer (DIL402PC, NETZSCH- Gerätebau GmbH, Germany). The CTE was calculated from:

$$\alpha = \Delta L / (L_0 \times \Delta T) \quad (1)$$

where α , $\Delta L/L_0$, and ΔT are the CTE, linear expansion ratio, and applied temperature difference, respectively. The change in the diameter (i.e., linear shrinkage or expansion) of the sintered pellets was measured by using a vernier caliper. In addition, the open porosity and bulk density were measured in accordance with Archimedes' principle using distilled water as an immersion liquid. The distribution of pore sizes of the prepared porous cordierite ceramics was determined by using an automatic mercury porosimeter (AutoPore IV9500, Micromeritics Instrument Corp., America). This porosimeter allows the analysis of pores with sizes ranging from 0.005 to 630 μm , and the pore radius, r_p , can then be determined from Washburn's equation that is given as:

$$r_p = \frac{-4\gamma \cos \theta}{P_{Hg}} \quad (2)$$

where γ is the surface tension (0.485 N m^{-1}) of Hg, θ is the contact angle between the Hg and the solid phase (equal to 140°) and P_{Hg} is the pressure of Hg. Three-point flexural strength tests of the sintered samples (length: 45 mm, width: 4 mm and height: 3 mm) were performed in accordance with ISO 14704:2000(E), using a universal material testing machine (ReGeR-3010, Reger Instrument Co., Ltd., China). A cross-head speed of 0.5 mm/min was used in the testing of at least five specimens.

The pH-dependent leaching behavior [28,29] of Cr was investigated in the green samples and the pellets sintered at 1350 °C for 3 h. The sintered pellets were initially ground and then sieved through a 200-mesh sieve for leach testing. A series of 5 g sub-samples was mixed with 100 mL of leaching solutions, of varying acidity, ranging from distilled water to 0.03 M HNO_3 solution (with pH value of ~1.5). The samples and leachates were mixed in sealed containers for 24 h on an oscillating shaking table, before being centrifuged. The leachates were then extracted by filtering through a 0.45 μm membrane filter and their pH was measured before being analyzed via inductively coupled plasma

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