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# Sinterability, microstructure and compressive strength of porous glass-ceramics from metallurgical silicon slag and waste glass

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ABSTRACT

Porous glass-ceramics have been prepared by the direct sintering of powder mixtures of metallurgical silicon slag and waste glass. The thermal behavior of silicon slag was examined by differential thermal analysis and thermogravimetry to clarify the foaming mechanism of porous glass-ceramics. The mass loss of silicon slag below 700 °C was attributed to the oxidation of amorphous carbon from residual metallurgical coke in the silicon slag, and the mass gain above 800 °C to the passive oxidation of silicon carbide. The porosity of sintered glass-ceramics was characterized in terms of the apparent density and pore size. By simply adjusting the content of waste glass and sintering parameters (i.e. temperature, time and heating rate), the apparent density changed from 0.4 g/cm$^3$ to 0.5 g/cm$^3$, and the pore size from 0.7 mm to 1.4 mm. In addition to the existing crystalline phases in the silicon slag, the gehlenite phase appeared in the sintered glass-ceramics. The compressive strength of porous glass-ceramics firstly increased and then decreased with the sintering temperature, reaching a maximal value of 1.8 MPa at 750 °C. The mechanical strength was primarily influenced by the crystallinity of glass-ceramics and the interfaces between the crystalline phases and the glassy matrix. These sintered porous glass-ceramics exhibit superior properties such as light-weight, heat-insulation and sound-absorption, and could found their potential applications in the construction decoration.

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## 1. Introduction

Metallurgical silicon slag is the by-product in the pyrometallurgical silicon industry. The annual production of metallurgical silicon nearly amounts to two million tons in China, thus the generation of silicon slag is about 200 kt annually. The main applications of metallurgical silicon slag involve the recycling of metallic silicon component, the deoxidizing agent in steelmaking, the production of non-ferrous alloys, the economical feed stocks of colloidal silica and organosilyl derivatives [1–4].

Compared with huge amounts of metallurgical slag, e.g. steel and copper slags [5,6], metallurgical silicon slag received little attention due to its relatively limited production, thus the related researches were scarcely reported. Besides the glassy phase and metallic silicon, metallurgical silicon slag generally contains carbon and silicon carbide, which are the common foaming agents at elevated temperatures. This enables the facile production of porous materials from silicon slag by the conventional sintering process without the addition of extraneous foaming agents [7,8]. Similar to the application of steel slag [9], metallurgical silicon slag could be an ideal feedstock to produce light-weight porous glass-ceramics, which exhibit superior properties such as heat insulation, sound absorption and humidity control [10–12].

The objective of this study was to prepare the porous glass-ceramics from metallurgical silicon slag and waste glass without the addition of foaming agent. The thermal behavior of silicon slag was initially investigated to clarify the foaming mechanism in the sintering process. Then, the silicon slag was mixed with different amounts of waste glass, and directly sintered in the mold without the usual powder compaction. The sintering conditions were systematically studied to examine the sinterability and microstructure of porous glass-ceramics. The variation of mechanical strength of porous glass-ceramics was qualitatively interpreted in terms of the crystalline composition and microstructure.

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## 2. Experimental

### 2.1. Raw materials

The metallurgical silicon slag was obtained from Anyang Huaqiang Metallurgical Materials Co., Ltd. Henan, China. The slag lump was crushed into small pieces and ground with alumina balls in a planetary mill to obtain slag powder. The as-prepared powder was passed through a 200 mesh sieve. In the same way, waste glass powder was prepared from the exhausted fluorescent lamps, and passed through a 120 mesh sieve. The rectangular/cylindrical refractory molds were manually manufactured with ceramic fiber papers (Zhibo Huashuo Refractory and Thermal Insulation Materials Co., Ltd.), and thermally treated at 850 °C for 1 h to remove the organic binder.

### 2.2. Preparation of porous glass-ceramics

Silicon slag powder was ground with different amounts of waste glass powder (10–50 wt%) in a mortar with a pestle for 10 min, and passed through a 120 mesh sieve. This procedure was repeated three times to ensure the homogenization of powder mixtures. Afterwards, the powder mixtures were uniformly lay in the rectangular molds, dried in an oven at 200 °C for 2 h, and sintered in a traditional muffle furnace. The sintering process was conducted in the temperature range of 700–900 °C, with the soaking time of 10–120 min. The heating rate was varied in the range of 10–100 K/min. After removing the molds, the sintered samples were cut with a diamond grinding wheel, and polished by using SiC abrasive papers (2000#).

### 2.3. Characterization

The chemical composition of silicon slag was semi-quantitatively analyzed by using a scanning X-ray fluorescence spectrometer (XRF, Bruker S4 Pioneer). The crystalline structure of slag powder and sintered glass-ceramics were identified by X-ray powder diffraction (XRD, Bruker-axs D8 Advance) using Cu $K_\alpha$ radiation, and the degree of crystallinity was estimated from the XRD pattern simulation using the MDI Jade 5.0 software. The thermal behavior of silicon slag was analyzed using a simultaneous thermal analyzer (STA, Netzsch STA 449C). This measurement was conducted in a flow of synthetic air (30 m*l*/min) with a heating rate of 10 K/min.

The apparent density of porous glass-ceramics was determined via dividing the mass of rectangular samples by their apparent volumes. The optical image of porous glass-ceramics was recorded with a digital camera (Canon 750D), and the mean pore size was calculated using the software of Image pro plus 6.0 (Media Cybernetics, USA) after the binarization processing of digital images. The microstructure observation and elemental analysis of silicon slag and sintered glass-ceramics were performed using an environmental scanning electron microscope equipped with an energy dispersive spectrometer (SEM/EDS, FEI Quanta 200). The compressive strength of porous glass-ceramics was measured using cylindrical samples ($\varnothing$ 20 × 20 mm$^2$) in an Instron-type apparatus (WDW-50) with a crosshead speed of 0.05 mm min$^{-1}$. Each data point represents the average value of at least 5 individual tests.

## 3. Results and discussion

### 3.1. Characterization of silicon slag

The chemical composition of silicon slag was shown in Table 1.

**Table 1**
Chemical composition (wt%) of silicon slag and waste glass.

| Raw materials | $SiO_2$ | $Na_2O$ | CaO | $Al_2O_3$ | MgO | BaO | $Fe_2O_3$ | $K_2O$ | Others |
|---|---|---|---|---|---|---|---|---|---|
| Silicon slag | 72.6 | 19.3 | 2.3 | 2.3 | 2.0 | – | 0.7 | 0.1 | 0.7 |
| Waste glass | 71.5 | 14.2 | 5.7 | 2.4 | 3.0 | 1.7 | – | 1.5 | – |

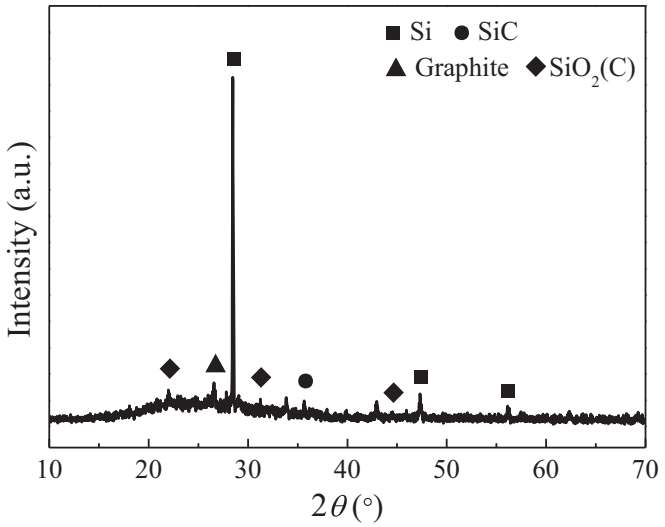


**Fig. 1.** XRD pattern of silicon slag.

It contains not only the major constituents of silica and sodium oxide, but also minor constituents of calcia, alumina, magnesia and ferric oxide *etc*. As a comparison, waste glass contains comparable amount of network formers ($SiO_2$ and $Al_2O_3$) and higher contents of network modifying oxides (CaO and MgO). Unfortunately, the XRF analysis merely presented the elemental composition in the oxide formulation, and could not determine the exact compounds and their crystallographic states of silicon slag.

The XRD pattern of silicon slag is shown in Fig. 1. The crystalline phases include Si (PDF# 77-2111), graphite (PDF# 75-2078), cristobalite (PDF# 82-0512) and moissanite (SiC, PDF# 75-0254). The graphite phase derived from the graphitization of metallurgical coke in the pyrometallurgical silicon production, and the Si phase from the residual silicon melt. The cristobalite and moissanite phases resulted from the reactions between the silicon melt with the oxygen and coke, respectively. Since the XRF results in Table 1 were presented in the oxide formulation, the crystalline Si and SiC phases were absent therein. By simulating the XRD pattern with the MDI Jade 5.0 software, the degree of crystallinity was estimated to be 18 vol%, suggesting the dominance of glassy phase in the silicon slag.

Fig. 2 shows the SEM micrograph of silicon slag. The microsized particles were embedded in the continuous matrix. From the EDS analysis the atomic ratio of C/Si is close to 1 for the spherical particles (Spectrum 1), evidencing the presence of SiC crystallites. However, multiple elements such as carbon, sodium, oxygen and silicon were detected for those particles with irregular shape, implying the glassy phase therein. The matrix is characteristic of Si element with trace of oxygen (Spectrum 2), attributable to the silicon and silica phases. Considering the rapid quenching process of silicon slag, it is reasonable to infer that the glassy phase is partially composed of the amorphous phases, including coke, amorphous silicon and silica.

Fig. 3 shows the thermal behavior of silicon slag. For the DTA curve two broad exothermic processes at 435 °C and 652 °C related with the successive mass loss in the TG curve, which should be attributed to the oxidation reactions of amorphous carbon from

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