Production of high-quality glass foam from soda lime glass waste using SiC-AlN foaming agent

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Abstract
This research presents the possibility of producing durable foam glasses from glass cullet using SiC/AlN foaming agent. The foaming agent generated by the SiC and AlN couple results in a more homogeneous microstructure and thus the emergence of foam glasses with better properties compared to the nitride foaming agent used alone in our previous work. The fabricated foam had a crack-free, 3-D cellular structure with macropores whose geometries varied between elliptical-, pentagonal-, and hexagonal-shaped constructions. It also had a lightweight (≥ 0.18 g/cm^3), high cold crushing strength (≤ 4.5 MPa), low thermal conductivity (0.09–0.16 W/m K), and contained more than ~ 89 vol.% gas bubbles enclosed between 11 vol.% impervious glass walls. The properties accomplished by the foam prepared in this work conform with the requirements of international standard for commercial glass foams, demonstrating its strong capability to be utilized in potential applications in sustainable buildings and energy efficiency in industry.

Keywords Cellular glass foams · Recycled soda-lime glass waste · SiC/AlN foaming agent · 3-D cellular-like structure

1 Introduction

Due to the depletion of natural resources and the accumulation of huge amounts of solid waste, nowadays recycling of industrial waste and by-products has become a priority and has recently attracted a growing area of attention. The solid waste includes millions of tons of glass cullet which are produced annually. The glass waste is typically polluted by a high proportion of fine organic matter together with metallic and non-metallic particles. Removal of these organic and inorganic inclusions is relatively expensive. As a result, in many cases, the production of articles from glass cullet is a low-profit business. Consequently, the amount of accumulated glass waste is steadily rising in landfills [1–3]. The global quantity of glass wastes produced in 2018 was approximately 130 million metric tons (MMT); about 20% (~ 26 MMT) of these glass cullets are normally recycled and the rest of these municipal solid wastes (~104 MMT) is increasingly amassed in the landfills, causing severe environmental problems [4, 5]. Dealing with such large quantities of glass cullet is a serious issue that needs to be resolved.

Fortunately, incorporation of waste glass in the production of glass foam has recently shown a great deal of interest in the recycling concept and provided an important way to recycle these municipal solid wastes. Glass foams exhibit impressive combination of properties, such as lightweight (> 0.5 g/cm^3), nontoxicity, high porosity content (< 60 vol.% [3], chemical inertness, reasonable compressive strength values (0.4–6 MPa) [4], high thermal insulation (~ 0.1 W/m K), water- and fire-resistance along with the ease of operation, cutting, and drilling [5, 6]. That is why, in recent years, these foamed materials have been widely used in numerous applications, such as refractory linings, catalyst support, filters for hot gases and molten metals, as well as building materials [7–10].

In general, glass foam is typically prepared by foaming glass matrices with a pore-forming agent such as silicon carbide, carbon, and carbonates close to the glass softening temperature undergoing viscous flow sintering [11]. Soda-lime glass waste derived from the lapping machine has been utilized in glass foam production in a number of research...
works using various pore-forming agents; the characteristics of these foam glasses are summed up in Table 1. E. Erçenk has studied the effect of clay additives on the foaming and mechanical characteristics of glass foams prepared from soda-lime glass waste using dolomite as a pore-forming agent [12]; the resulting foams had 0.42–2.3 MPa compressive strength, 0.76–1.4 g/cm³ bulk density, and 33–62% apparent porosity in the temperature range 1000–1075 °C. Recently, our research team has paid more attention to the reuse of landfilled glass cullet in the production of glass foams, which contributes to preserving the environment and providing an added value to this waste. During the past years, Ewais et al. have synthesized foam glasses with 0.65–2.48 MPa CCS, ≤ 0.5 g cm⁻³ bulk density, and 0.09–0.106 W/m K thermal conductivity starting with soda-lime glass waste along with 2.5–7.5 wt.% AlN foaming agent [13]. Replacement of AlN by SiC in the previous work has led to emergence of glass foam with lower bulk density and higher porosity; however, the refractoriness of the foamed glass obtained by SiC was 50 °C lower than that of the glass foam obtained by AlN [14].

In this study, we were able to further improve the physico-mechanical characteristics of glass foams produced from soda-lime glass waste using SiC/AlN couple as a foaming agent rather than a single AlN or SiC. The use of SiC/AlN couple resulted in a more homogeneous microstructure and thus the emergence of a glass foam with better physico-mechanical and thermal characteristics (comparable CCS and BD to those obtained by SiC; as well as comparable refractoriness to those obtained by AlN) compared to the nitride and carbide foaming agents when used separately (please see Table 1). In addition, physical and mechanical characteristics of the international standard of the commercial glass foams are given in Table 1 to easily compare foam values obtained in this work with commercial foam products [15, 16]. The properties accomplished by the glass foam produced in this work are in line with the requirements of the international standard for commercial glass foams, demonstrating their strong capability to be used in potential applications in sustainable buildings and energy efficiency in industry.

### Table 1  Comparison between physico-mechanical properties of glass foams prepared in previous and current works via sintering of soda-lime glass waste with different foaming agents

| Glass waste       | Foaming agent, wt.% | Additives | Foaming temp., °C | Holding time, min | BD, g/cm³ | CCS, MPa | Por., Vol.% | References |
|-------------------|---------------------|-----------|-------------------|-------------------|-----------|----------|-------------|------------|
| Soda-lime glass   | Dol                 | Clay      | 1000–1075         |                   | 0.76–1.4  | 0.42–2.3 | 33–62%      | [12]       |
| Soda-lime glass   | AlN (2.5–7.5 wt.%)  | –         | 850–950           | 30                | ≤ 0.5     | 0.65–2.48 | ≤ 94        | Previous work [13] |
| Soda-lime glass   | SiC/AlN (2.5–7.5 wt.%) | –   | 850–950           | 30                | ≤ 0.4     | 0.3–4.0   | ≤ 94.5      | Current work |
| ISCGF             | –                   | –         | 800–1000          |                   | 0.1–0.3   | 0.4–6     | 85–95       | [15, 16]   |

* Dol. dolomite; Cal. calcite; BD bulk density; CCS cold crushing strength; Por. porosity; ISCGF international standard for commercial glass foam

### 2 Materials and experimental procedure

#### 2.1 Materials

The soda-lime glass waste derived from the lapping machine was provided by a municipal recycling company, Cairo, Egypt. A detailed chemical analysis of this glass waste was presented in our previous research work [13]; it composed mainly of SiO₂ (71.6 wt.%), Na₂O (13.5 wt.%), CaO (9 wt.%), MgO (3.87 wt.%) together with a small amount of Al₂O₃, Fe₂O₃, SO₃ and K₂O (about ~ 1.71 wt.%). High purity AlN and SiC raw materials were supplied by Hong Kong Tepu Refractory Co., Limited, China. Particle size distribution (PSD) results of raw material powders utilized in this study are listed in Table 2. According to this table, 90 wt.% of SiC and AlN powders comprised of particles with a diameter less than 1 and 1.3 µm, respectively.

#### 2.2 Experimental procedure

Typical experimental procedures were schematically outlined in Fig. 1. At first, the waste glass was crushed with a small crushing machine. The crushed glass was then ground in a planetary ball mill (weight ratio of glass:zirconia balls = 1:2) at 350 rpm for 90 min. The milled glass powder was subsequently sifted through a 50-mesh sieve. Three batch compositions (GF2.5–7.5) were manufactured from waste glass powders and SiC/AlN foaming agents according to the nominal compositions displayed in Table 3. The quantity of foaming agent was successively added in equal parts of AlN and SiC. For example, to add 2.5 wt.% AlN/SiC, 1.25 wt.% of each material was singly and successively added to the designed batch composition. These

### Table 2  Particle size distribution of raw material powders

| Sample name | D₁₀, µm | D₅₀, µm | D₉₀, µm |
|-------------|---------|---------|---------|
| SiC         | 0.42    | 0.6     | 1       |
| AlN         | 0.6     | 0.9     | 1.3     |

Dol. dolomite; Cal. calcite; BD bulk density; CCS cold crushing strength; Por. porosity; ISCGF international standard for commercial glass foam
batches were dry-blended in a planetary ball mill for 30 min, then dry-molded in cylindrical stainless steel moulds (interior surface coated with BN). The stainless-steel moulds were then placed in a muffle-furnace and heat-treated at 850–950 °C with 5 °C/min heating rate and 30 min retention time at the foaming temperature. After slow cooling to ambient temperature, demolding, cutting and finishing the sintered briquettes, 37 cm³ glass foam briquettes were obtained (Diameter = 3.07 cm; Height = 5 cm), as shown in schematic diagram in Fig. 1. The physical, mechanical and thermal characteristics of the resulting glass foams have been duly studied as a function of the foaming temperature and the pore-forming agent content. To avoid errors in the results, the measurement was carried out three times and the results were only accepted if the difference between the three values was less than 1.5%.

### 2.3 Characterization

The bulk density (BD) of the resulting cellular glass foams was measured by the simple equation, BD = W/V, where W: weight of glass foam and V: volume of glass foam. The relative density (RD) was estimated by RD = BD/GFD, where GFD is the true density of glass foam powders = 2.521–2.523 g/cm³. From the relative density value, the total porosity ($P_{total}$) was determined using the following equation, $P_{total} = 1 – RD$ [17, 18]. The mineralogical composition of the synthesized glass foams was investigated using SHIMADZU X-ray diffraction machine (Model: XRD-7000, Japan, Cu kα radiation ($\lambda = 1.5406$ Å), 2-theta range from 5 to 70°). The microstructure and morphology of the prepared glass foams were examined by means of a field-emission scanning electron microscope (FESEM, Quanta FEG 250, Holland). The exact cellulularity (number of pores per inch, PPI) was estimated by counting the number of pores intersected by a straight line (1") on the obtained photomicrographs [19]. The FESEM images were uploaded on the ImageJ software which automatically analyzed them giving the pore diameters of the obtained foams. This software is a free online application accessible to all researchers. The compression resistance (cold crushing strength) of the glass foam specimens was measured using Shimadzu testing machine at 1.3 mm/min strain rate (Model: UH-F 1000 KN, Japan). Thermal conductivity, thermal diffusivity and specific heat capacity were measured by the Hot Desk machine (Model: 2500 s, Sweden) at room temperature in normal atmosphere. Particle size distribution (PSD) of raw material powders was investigated by laser diffraction using a Fritsch laser diffractometer (model: Analysette 22 NanoTec). Before PSD measurement, the materials were precisely dispersed via ultrasonication for 1 min using 24 kHz Hielscher device (model: UP400S).

### 3 Results and discussion

The optimal foaming temperature of soda-lime glass waste with SiC/AlN foaming agent was found experimentally between 850 and 950 °C. In this temperature range, SiC and AlN oxidize according to Eqs. (1, 2) [20–22] releasing gas bubbles through the low-viscosity glass medium. These gases are entrapped through the viscous glass medium leading to expanding the glass matrix giving rise to high-quality cellular materials. The physical, mechanical, thermal, and microscopic properties of the resulting foams have been precisely studied, and the results are presented in subsequent sections.

\[
\text{SiC(s)} + 2\text{O}_2(g) \rightarrow \text{SiO}_2(s) + \text{CO}_2(g),
\]

\[
4\text{AlN(s)} + 3\text{O}_2(g) = 2\text{Al}_2\text{O}_3(s) + 2\text{N}_2(g).
\]

### 3.1 Microstructure

Figures 2, 3, and 4 display the FESEM photomicrographs of the resulting “GF2.5–7.5” glass foams sintered at 850,
900, and 950 °C, respectively. The obtained foams were crack-free and they presented predominantly closed porosity with different geometries that varied between hexagonal-, elliptical-, and pentagonal-shaped structures. FESEM photomicrographs were analyzed using ImageJ software and the variations of mean pore diameter and exact cellularity with the sintering temperature and foaming agent content are depicted in Figs. 5 and 6, respectively. The obtained results showed a good compatibility and a reciprocal relationship between the mean pore diameter and cellularity, where the former increased when the latter decreased and vice versa. This inverse relationship between average pore diameter and microcellularity is a logic, where the number of pores per inch (exact cellularity) diminishes as the pores grow larger. As shown in Fig. 6, the average cellularity of the three foams “GF2.5–7.5” at 850 °C was approximately...
43 ± 20 PPI, a value which declined to 21 ± 10 and 14 ± 6 PPI at 900 and 950 °C, respectively. In contrast, the average pore diameter of the foams varied from 0.46 to 1.2 mm at 850 °C; it went up to 1.355 ± 0.545 and 1.925 ± 0.625 mm at 900 and 950 °C, respectively. Such increase in the pore diameter with the sintering temperature could be explained in terms of a decrease in the glass viscosity at a higher sintering temperature and the increase in gas pressure in the pores which, in turn, force the pores to develop and the entire glass to expand further. Regardless of the sintering temperature, the mean pore diameter of the produced foams increased with the increase in foaming agent content from 2.5 to 5.0 wt.%, and subsequently decreased after increasing the SiC/AlN content above 5 wt.%. This can be explained as follows: (1) when the foaming agent content is low (2.5 wt.%), a small number of gases is evolved leading to small pore diameter and low porosity (2) The average aperture size increased when the foaming agent content increased to 5 wt.% due to the release of more gases, and coalescence between adjacent pores, a phenomenon that forced the pores to grow larger [7, 23–25] (3) Beyond 5 wt.% foaming agent,
The glass viscosity enhancement occurred by incorporation of the oxidation products of the foaming agent (Al₂O₃ and SiO₂) seems to be more responsible for the reduced bubble size. A similar trend for the variation of the mean pore diameter with the foaming agent content was observed in several previous works. In light of these findings, the sintering temperature and the amount of pore-forming agents are the primary factors controlling the porosity variation in the final cellular foam products.

3.2 Phase composition evolution

Powder XRD patterns of glass foam samples “GF2.5–7.5” fired at 850, 900, and 950 °C were displayed in Fig. 7a, b, c, respectively. The resulting foams had an amorphous nature with a wide halo in the 2θ range from 15 to 35° which is characteristic for the amorphous silica with silanol group (Si–OH) [26, 27]. Nonetheless, partially crystalline phases with very low-intensity diffraction peaks were observed in these glass foams, such as: (1) cristobalite phase (SiO₂, at 2θ = 21.72°, #JCPDS#01-082-0512) that was mainly detected in “GF2.5” specimen (2) diopside phase (CaMg(SiO₃)₂, at 2θ = 29.92 and 35.58°, #JCPDS#00–017-0318) which was detected in “GF5.0–7.5” specimens. Diopside and cristobalite crystallization was reported in a number of glass foams produced from glass cullet and various foaming agents [7, 12, 28–31].

3.3 Densification parameters

The densification parameters reflect what happened for the microstructure of the produced foams during sintering. Figures 8 and 9, respectively, show the dependence of bulk density and total porosity of the fabricated foams on the sintering temperature and foaming agent content. Apparently, the bulk density decreased throughout the studied temperature range, and contrarily the total porosity increased with increasing SiC/AlN content from 2.5 to 5 wt.%. This can be easily explained in terms of the formation of larger quantities of gas bubbles as the amount of foaming agent increases [28]. With the SiC/AlN content increase beyond 5 wt.%, a reverse trend occurred, where the density increased and the porosity decreased, featuring V and Λ-shaped trends, respectively. The decrease in porosity at the high SiC/AlN content (< 5 wt.%) was attributed to the generation of excessive gas bubbles, breakdown of the struts, and collapse of the structure. At 850 °C, foamed samples expanded slightly to 3.5 times compared to the green body, giving rise to foam with slightly high density (0.35 ± 0.5 g/cm³) and low porosity (89 ± 2 vol.%), suggesting that the viscosity at this foaming temperature was still relatively high. When the foaming temperature increased to 900–950 °C, foamed samples were expanded clearly to 5–5.5 times compared to the green body.

Fig. 7 X-ray diffractograms of glass foams fired at (a) 850 °C, (b) 900 °C and (c) 950 °C
resulting in foam with quite low density (0.25 ± 0.01 g/cm³ at 900 °C & 0.19 ± 0.01 g/cm³ at 950 °C) and high porosity (92 ± 0.5 vol.% at 900 °C & 93.5 ± 0.5 vol.% at 950 °C). The most efficient foaming effect was registered for samples containing 5 wt.% SiC/AlN, where GF5.0–900 has expanded to around 5 times compared to the green body, registering 92 vol.% porosity, 0.24 g/cm³ bulk density, 12 PPI cellularity, and 1.91 mm pore diameter, whereas GF5.0–950 specimen has expanded to about 5.5 times, recording 94 vol.% porosity, 0.18 g/cm³ bulk density, 10 PPI cellularity, and 2.54 mm pore diameter. Based on the densification results, the best sintering temperature was 900 to 950 °C, and the optimal foaming content was ranged from 2.5 to 5.0 wt.%.

3.4 Cold crushing strength (CCS)

The variation in the CCS of the resulting foams as a function of sintering temperature and SiC/AlN content is illustrated in Fig. 10. The CCS values of the resulting foamed specimens correlated well with their bulk density values. The foamed specimens at 850 °C demonstrated the highest CCS values at the expense of their elevated densities. The glass foams sintered at 850 °C had compressive strength values between 1.5 and 4.5 MPa, whereas the CCS values decreased to 0.7–1.7 MPa and 0.3–0.8 MPa at 900 and 950 °C, respectively. Such decrease in the CCS values corresponds to the porosity enhancement and density decline at higher sintering temperatures, since the compressive strength of porous ceramics is basically dependent on the ceramic particles around pores (effective load-bearing struts) which decrease with the increase in porosity at higher sintering temperatures [32, 33]. These findings are in agreement with the reported data in references [13, 34]. The obtained properties for the waste-derived foams synthesized in this work compare well with those displayed by commercially available glass foams in terms of bulk density (0.18–0.4 g/cm³), total porosity (89–94 vol.%), and compressive strength (0.3–4.5 MPa) [11].

3.5 Thermal conductivity

Figure 11 shows the variation of the thermal conductivity of the produced foams with the sintering temperature and SiC/AlN content. The thermal conductivity of glass foams had an inverse proportion with porosity as well as aperture size and homogeneity [35]. This means that the thermal insulation of the glass foams is improved with the increase in
2. The glass foam specimens expanded to around 5 times at 900 °C, resulting foams were characterized physically, mechanically, and thermally. The experimental results are set out below:

1. The foam glasses had a 3-D cellular structure with macropores of elliptical, pentagonal, and hexagonal geometries.
2. The glass foam specimens expanded to around 5 times at 900 °C compared to the green body, registering 92 ± 0.5 vol.% porosity, 0.25 ± 0.01 g/cm³ bulk density, 21 ± 10 PPI cellularity, 1.355 ± 0.545 mm mean pore diameter, 0.1 W/m·K thermal conductivity, and ~0.7–1.7 MPa CCS.
3. The physico-mechanical and thermal properties achieved by the specimens foamed at 900 °C are consistent with the requirements of the international standard for commercial glass foams, demonstrating its strong capability to be utilized for potential applications in sustainable buildings and energy efficiency in industry as lining or lightweight packing material.

4 Conclusions
High-quality foam glasses were successfully manufactured from soda-lime glass waste combined with SiC/AlN pore-forming agent via viscous flow sintering at 850–950 °C. The resulting foams were characterized physically, mechanically, and thermally. The experimental results are set out below:

1. The foam glasses had a 3-D cellular structure with macropores of elliptical, pentagonal, and hexagonal geometries,
2. The glass foam specimens expanded to around 5 times at 900 °C compared to the green body, registering 92 ± 0.5 vol.% porosity, 0.25 ± 0.01 g/cm³ bulk density, 21 ± 10 PPI cellularity, 1.355 ± 0.545 mm mean pore diameter, 0.1 W/m·K thermal conductivity, and ~0.7–1.7 MPa CCS.
3. The physico-mechanical and thermal properties achieved by the specimens foamed at 900 °C are consistent with the requirements of the international standard for commercial glass foams, demonstrating its strong capability to be utilized for potential applications in sustainable buildings and energy efficiency in industry as lining or lightweight packing material.

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