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The viscosity window of the silicate glass foam production

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ABSTRACT

The production of silicate glass foam allows diverse resources and waste materials to be used in the production. Testing of such large palette of materials complicates and prolongs the optimisation process. Therefore, it is crucial to find a universal criterion for foaming silicate glass melts which can offer a practical starting point for the optimisation procedure. The melt viscosity might be the most important parameter for controlling the foaming process and the glass foam density. In this work, we attempt to define a viscosity range in which foaming of different glasses results in a maximum of foam expansion. The expansion maximum is obtained for different glasses (labware, E-glass, CRT panel, soda-lime-silica) by foaming with CaCO₃ at isokom temperature and from literature data. In general, the viscosity window was found to be within 10⁴–10⁶ Pa s when foaming with MnO₂ or metal carbonates (CaCO₃, Na₂CO₃, MgCO₃, SrCO₃, dolomite) whereas SiC requires higher temperatures and correspondingly lower viscosities (10^{3.3}–10^{4.0} Pa s). These findings can help assessing the implementation of new resources in the glass foam production.

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1. Introduction

The production of silicate glass foams is becoming increasingly important as a valuable recycle option for post-consumer glass and industrial waste, such as fly ash, slags and ore tailings. The process proceeds at relatively low temperatures (650–1000 °C) by utilising virgin glass and/or inexpensive waste materials which would otherwise be landfilled.

The ability for a glass melt to foam through the sintering approach is controlled by many factors, e.g. glass composition, particle size, temperature, type and concentration of foaming agent. Optimising all the parameters is extensive and a good starting point is essential to reach optimal conditions within reasonable time. The foaming temperature is easy to control and is perhaps one of the most studied parameters. An accurate temperature control is important, since it affects properties such as melt viscosity, bubble pressure, rate of bubble coalescence, crystallisation and gas formation.

Several experimental studies show that foam density goes through a minimum when the treatment temperature is varied [1–5]. The origin of this minimum is related to several factors. At low temperatures, the melt viscosity is high and the foam expansion is slow. At high temperatures, the melt viscosity is low and gas formation is fast, leading to foam collapsing. Since viscosity decreases monotonically with temperature, viscosity must be the major cause of the observed minimum in the density. The temperature, at which the density reaches a minimum, depends also on many other parameters. For example, when a slow heating rate (0.8 °C/min) is used, the density minimum is found at a

100 °C lower in comparison to when faster heating rates (1.6 or 2.5 °C/min) are used [2]. Thus, the discussion of the density minimum is relevant only if other factors are kept within reasonable values.

The glass foam quality is determined by an optimal balance of properties. The most important ones are density, closed porosity, compression- and flexural strength and the thermal conductivity. The density is strongly correlated to thermal conductivity and the mechanical strength. It is challenging to obtain closed pores in a low density glass foam (<0.18 g/cm³), but very easy in high density glass foam. Hence, there is a strong connection between density and closed porosity. The density is therefore the most important property of glass foams and it is accordingly meaningful in a first attempt to relate density with melt viscosity.

Due to the important role of the viscosity factor, good foaming conditions are often defined in terms of a viscosity range. Mugoni et al. [6] suggest that the foaming agent should release gas when the melt viscosity is between 10⁶ and 10⁷ Pa s. Scarinci et al. [7] recommend that the viscosity should be between 10³ and 10⁵ Pa s for optimal foaming conditions. These statements do not agree well with each other at first sight. Therefore, in this work, we attempt to define the optimal viscosity range for different glass foam productions. We foam different glasses (labware glass, E-glass, soda-lime-silica glass, CRT panel glass) at different viscosities (i.e. isokom temperatures) by using the sintering approach and CaCO₃ as the foaming agent. The foaming of glass melts with CaCO₃ has been studied extensively for many decades [1,8–14]. In the present investigation, factors such as particle size, sample size, compaction pressure, and concentration of CaCO₃ were kept constant during preparation and only the viscosity was varied by changing temperature. The viscosity range at which we obtained a minimum density is compared with

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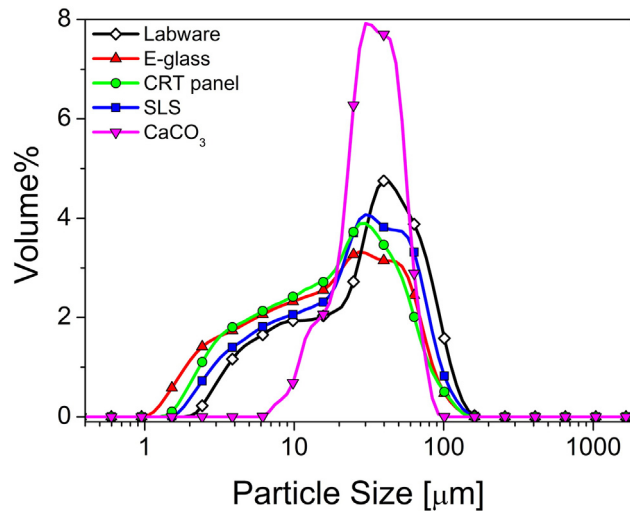


Fig. 1. Particle size distribution of CaCO_3 (foaming agent) and the glass powder. Only a fraction (1/5) of the data points is shown.

literature studies on glass foams which involve CaCO_3 and SiC as foaming agent. Based on these data, we suggest a universal viscosity window for the glass foam production. The viscosity window provides a helpful starting point for optimising the foaming process.

Several factors affect viscosity. First, inclusions in the glass melt can change the rheological behaviour of a glass melt. Suspended crystals [15,16] and gas bubbles [16,17] increase the viscosity of a melt suspension at low strain rates. There is no external shear stress applied to the suspension during the foaming process, however, the bubble growth induces minor shearing on the glass melt. Second, crystallisation changes the melt composition and the melt viscosity will change gradually as crystallisation proceeds.

The surface chemistry of many foam systems differs from the bulk part. Surface active species can immobilise the surface, increasing the kinetic stability of the cell wall and, hence, the lifetime of the foam. Though the surface chemistry of glass melt differs from the bulk, calculations indicate that the glass melt surface of a bubble is mobile [18]. Avramov's recent work on viscosity of silicate melts [19] show the surface viscosity could play an important role for bulk glass at very high viscosities ($>10^{12}$ Pa s). Extending the surface concept into the temperature regime of foaming (e.g. 800 °C for window glass), the surface viscosity can be several orders of magnitude lower [19]. Since a foam system has a large surface, the surface viscosity could play an important role on the overall glass foam viscosity. Measurements, however, show that the effective viscosity of glass foams is higher than the bulk glass [16,17]. Hence, the relation between surface rheology and foam viscosity is complex.

Table 1

Glass composition measured with XRF. Besides the reported oxides, labware and E-glass contain B_2O_3 (approximately: $\text{X}_{\text{B}_2\text{O}_3} = 100\% - \text{SUM}$).

Oxide	Chemical composition [wt%]			
	Labware	E-glass	CRT panel	Soda-lime-silica
SiO_2	80	53	61	74
Al_2O_3	2.6	14.0	1.9	
Fe_2O_3	0.9	1.0	0.5	0.5
Na_2O	3.9	0.9	7.3	15.6
K_2O	0.1	0.1	7.5	
CaO	0.4	22.9	0.1	9.2
SrO		0.2	8.8	
BaO		0.1	10.1	
TiO_2		0.6	0.5	
ZrO_2			1.4	
ZnO			0.5	
SUM	87.9	92.8	99.6	99.3

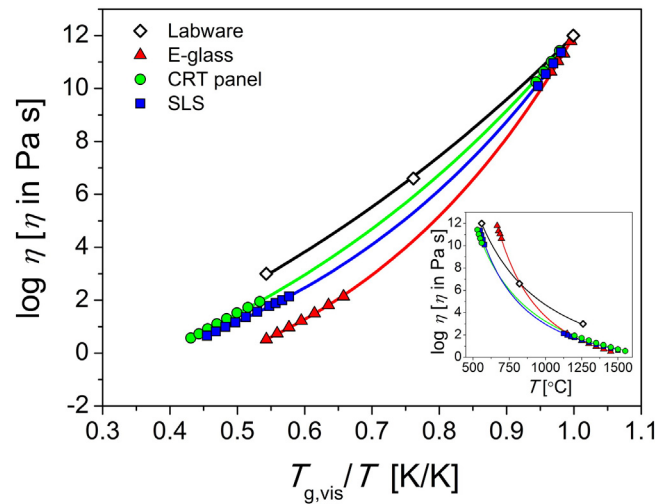


Fig. 2. Angell viscosity plot of the glasses (see legend) showing logarithmic viscosity ($\log \eta$) as a function of the inverse temperature (T) scaled to the glass transition temperature ($T_{g,\text{vis}}$) obtained from fitting the viscosity data to the MYEGA equation. The viscosity error at high temperature (measured with concentric cylinder) and at low temperature (measured with ball penetration) is $\eta = \pm 0.13$ Pa s and $\Delta \log_{10} \eta = 0.24$ (η in Pa s), respectively. The data for the labware glass are taken from the manufacturer. The solid lines represent fits made using the MYEGA equation.

It is challenging to account for the crystallisation effect and the inclusion effect on the effective viscosity during the foaming process. Our approach is simplified in sense that we compare the minimum density with the viscosity of the bubble- and crystal free glass melt. The decomposition and oxidation of foaming agents (e.g. CaCO_3 , SiC) produce solid oxide residues (CaO , SiO_2). These oxides will have an effect on the viscosity as solid residues and as components dissolved in the glass melts. The effect of foaming agents and solid residues on the viscosity is complicated. Since the concentrations of foaming agents are low, we simplify the approach by ignoring the effect of foaming agents as well.

2. Materials and methods

2.1. Powder and melt characterisation

The different glasses were first crushed with jaw crusher (BB51, Retsch) and then ball milled (PM 100, Retsch). The particle size distribution of the glass powder and CaCO_3 powder (99.9%, Sigma Aldrich) was determined with light scattering (LS 13320, Beckman Coulter) using Mie theory and refractive index of the respective glasses and CaCO_3 (Fig. 1).

The chemical composition of the glass powders was analysed with PW2400 X-ray fluorescence (PANalytical) using SuperQ software and oxide standards (Wroxi, PANalytical). The glass compositions are shown in Table 1. The iron content originates from the crushing procedure. The density (ρ) of the glasses was determined on glass

Table 2

The density (ρ) and fitting parameters ($T_{g,\text{visc}}$, m , $\log \eta_\infty$) obtained with the MYEGA viscosity model. $T_{g,\text{visc}}$ is the glass transition temperature, which corresponds to the viscosity of 10^{12} Pa s, m is the liquid fragility index and $\log \eta_\infty$ is the logarithmic viscosity at infinite high temperature. The uncertainty values are fitting errors.

Glass	ρ [g/cm ³]	$T_{g,\text{visc}}$ [°C]	m [—]	$\log \eta_\infty$ [Pa s]
Labware	2.2248 ± 0.0003	559.3 ± 3.2	25.5 ± 0.5	-2.93^*
E-glass	2.6317 ± 0.0005	663.4 ± 0.6	44.2 ± 0.8	-2.7 ± 0.2
CRT panel	2.7730 ± 0.0009	512.5 ± 1.1	30.7 ± 0.9	-2.9 ± 0.3
Soda-lime-silica	2.4803 ± 0.0017	533.5 ± 0.7	36.2 ± 0.6	-2.0 ± 0.1

* Fixed fitting parameter.

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