RHEOLOGICAL RESPONSES OF GLASS MELTS

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In the paper viscous-elastic responses of silica glasses with different chemical compositions (Lead crystal, Crystal glass, Float glass and Container glass) are evaluated. For measurement of rheological properties of investigated glass melts the uniaxial compression method was used. Cylindrical glass samples were compressed with constant speed between two parallel flat forming tools under isothermal conditions in the furnace of a special design. Experiments were performed at temperatures corresponding to the Newtonian viscosity 10^{7,47} Pas. Constant speeds of moving plunger were chosen from the range 0.05 to 4 mm/s. On the basis of realised experiments, the elastic and viscous characteristics including shear-thinning effect at the mentioned viscosity were identified.

INTRODUCTION

The knowledge of rheological properties of silica glasses including their dependence on temperature, deformation and deformation rate is important for effective virtual simulation of glass forming processes. However, the viscous-elastic characteristics of only several types of silica glasses (moreover in very limited extent) can be found in the literature [1-3].

Elastic properties of glass melt can be investigated by several static and dynamic experimental methods. Static methods, e.g. the cylindrical compression method [2], are not used frequently, as they make possible the evaluation of only instantaneous module. On the other hand, the advantage of this method is a possibility of simultaneous evaluation of both elastic and viscous responses of glass melt on applied load. The aim of this work is the investigation of viscous-elastic characteristics of several types of silica glasses.

EXPERIMENTAL

The principle of isothermal compression method is simple. The cylindrical glass sample (Figure 1) is



Figure 1. Cylindrical samples processed by free pressing.

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compressed between two flat forming tools to a certain value of deformation and its viscous-elastic response depending on a deformation rate and temperature is measured [5]. The pressing device (Figure 2) is controlled by servo testing machine LLOYD 50 LR



Figure 2. Laboratory pressing device.

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Oxide (mol.%)	Crystal glass	Float glass	Container glass	Lead crystal glass
SiO_2	71.5	71.7	70.6	59.1
Al_2O_3	0.1	0.7	1.7	0.04
CaO	6.4	9.1	8.6	0.006
MgO	-	4.1	3.6	0.006
BaO	4.1	-	0.5	-
PbO	-	-	-	24.7
ZnO	1.3	-	-	1.4
Fe ₂ O ₃	0.02	0.08	0.4	0.007
Na ₂ O	10.0	13.6	13.8	2.1
K_2O	6.1	0.1	0.7	12.4
Property				
E_{20} (MPa)	74 000	73 900	74 690	60 700
G_{20} (MPa)	30 400	30 030	30 660	25 020
ρ_{20} (kg/m ³)	2520	2460	2480	2960

with a maximum piston load of 50 kN and maximum piston speed of 8 mm/s. To get reliable outputs, strictly isothermal conditions have to be ensured during the experiment. Therefore forming tools and glass samples are heated in a cylindrical laboratory furnace of a special design that is an integral part of a laboratory apparatus. To avoid sticking of the glass melt to the steel working plates, 0.27 mm thick mica foils are placed in between the glass and working surfaces.

The application of this method enables the comparison of the viscous-elastic response of different types of glass melts (Table 1) to applied load (with various but constant piston speed) at the same Newtonian viscosity.

RESULTS AND DISCUSSION

The force responses of glass melts were measured for piston speed range 0.05 to 8 mm/s at Newtonian viscosity changing in the interval $10^7 - 10^{10}$ Pas. Glass samples had equal dimension, 15.3 mm of the diameter and 9.95 mm of the height, and were pressed with the constant piston velocity (0.05; 2; 4 mm/s) at fixed viscosity ($10^{7.47}$ Pas). Therefore the obtained graphic outputs of measured viscous-elastic response of tested glass melts correspond to a unified initial dimension of used sample.

Figure 3 shows selected experimentally determined force responses of investigated glass melts. It is clear that from the course of viscous-elastic response two qualitatively different states can be identified. In the initial loading step (maximum up to 2 mm), the elastic deformation component is initiated in the glass melt to a great extent. A viscous flow of glass melt becomes more evident when the compression is higher than 2 mm.

Mutual comparison of individual force responses of tested glasses validates that all the realised experiments were made under same loading conditions. Experimentally determined viscous-elastic responses of glass melts (Figure 3) allow evaluation of instantaneous values of the elastic modulus E(t) in the first stage of experiment [2]. Corresponding courses of instantaneous Young's

modulus for tested glass melts are shown in Figure 4.

The analysis of measured results indicates that in the first stage of the deformation loading, instantaneous elastic modulus of investigated glass melts is almost identical for the whole range of applied deformation rates (Figure 4). With increasing deformation rates, there is an evident increase of relaxation modulus. In the last stage of deformation (the stage of viscous flow) a considerable difference was discovered between deformation characteristics of Lead crystal glass and other glass melts. This deviation may be caused by a different chemical composition of Lead crystal glass (Table 1).

For identification of the actual viscosity during experiments the viscous model (FEM) created in program MSC.Marc was used. The glass melt was assumed to be incompressible; therefore a relation between equivalent stress (σ_{ekv}) and equivalent strain rate ($d\epsilon/dt_{ekv}$) was expressed as

$$\sigma_{ekv} = 3\eta(T)\dot{\varepsilon}_{ekv} \tag{1}$$

The numerical model [4] was applied in order to specify actual viscosity in the later stage of compression during the shaping process. With respect to the viscous-elastic character of glass melt it is necessary the mechanical energy dissipation to be taken into account. The courses of real viscous response (experimental data correspond to the curve 1 in Figure 3. Virtual force responses of lead crystal at viscosity of 107.47 Pas and at various piston speeds are drawn up in Figure 5. They are in a good agreement mutually, especially at low piston speed confirming the achievement viscosity 10^{7.47} Pas. With increasing compression speed and compression level, virtual force response starts to deviate from experimental data gradually. These experiments allowed us also to define values of critical strain rate for onset of shearthinning [1] that can be for Lead crystal glass expressed as

$$\dot{\varepsilon}_k = A \, e^{-B \cdot \eta_0} \tag{2}$$

where *A*, *B* are constant (A = 48.9, B = 0.59) and η_0 is Newtonian viscosity.



c) v = 4 mm/s

Figure 3. Viscous-elastic response of analyzed glass melts under compression load at viscosity $10^{7.47}$ Pas. 1 – Lead crystal glass (612°C); 2 – Container glass (696.6°C); 3 – Float glass (685.1°C); 4 – Crystal glass (655°C).

Figure 4. Typical course of elastic modulus of chosen glass melts at viscosity $10^{7.47}$ Pas.

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Figure 5. Viscous response of lead crystal under compression load at $10^{7.47}$ Pas. 1 - 0.05 mm/s; 2 - 2 mm/s; 3 - 4 mm/s. — experimental; \circ FEM with total heat dissipation

CONCLUSION

In this paper the viscous-elastic responses of different types of glass melts at the constant viscosity $(10^{7.47} \text{ Pas})$ and at various piston speeds were evaluated. It was found that the viscous-elastic behaviour of investigated glass melts is very similar, except for Lead crystal glass at higher compression. Experimental results indicated that

the glass melt viscosity undergoes change expressed by converting the glass melt behaviour from a Newtonian to a non-Newtonian state when the critical value of the strain rate is exceeded.

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