National Conference with International Participation

ENGINEERING MECHANICS 2008

Svratka, Czech Republic, May 12 – 15, 2008

RHEOLOGICAL RESPONSE OF SILICA GLASS MELT – MEASUREMENTS AND COMPUTER SIMULATION

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Summary: *The paper deals with a problem of a viscous-elastic response of glass melts under compression load. For an identification of rheological behavior isothermal cylinder compression method is used. Special attention is paid to influence of the strain rate on initialization of shear thinning process. Experimental material characteristics are used for virtual simulation of realized experiments. Evaluation of virtual experiments allowed defining critical values of strain rates for onset shear thinning.*

1. Introduction

Glass forming is a complex technological process the goal of which is to obtain solid product of required shape. From other substances glass differs by its amorphous structure in the solid state and a transformation (temperature T_g corresponding to viscosity of ca. 10^{12} Pas). Strong dependence of viscosity on temperature is a dominant property of glass. Glass melt is formed in the range of the glass forming interval (temperatures corresponding to the viscosities cca. $10^2 – 10^7$ Pa.s usually). In technological practice glass is usually considered to be viscous-elastic substance within the transition range, elastic one under this interval and viscous one above it. But from rheological point of view glass is a viscous-elastic matter in the whole range of temperatures applied.

Effective approach to meeting of industrial requirements (non-traditional shapes and dimensions, product quality) is an application of virtual modelling techniques in the pre-manufacture stage. To get acceptable results, the simulation model must be able to address all aspects influencing glass forming itself. Efficiency of numerical simulations is then dependent on the accuracy of description both of material properties and boundary conditions. Therefore it is necessary to pay increased attention to investigation of rheological properties of glass melt above T_g.

2. Experimental method

Rheological properties of glass melt can be investigated by means of a number of static and dynamic experimental methods. For the investigation of the viscous and elastic properties of glass melt we used the simplest (but the most powerful) method - uniaxial compression of

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cylindrical glass samples. The principle of this method is simple – the glass sample is compressed by defined manner with working piston and its force response depending on the constant piston velocity and temperature is measured. In our case cylindrical silica glass samples was compressed by laboratory machine LLOYD LR50K with constant speed of working piston under invariable temperature [Vítová, 2005]. Speed of working piston as well as temperature of glass sample has been changed subsequently.

To get reliable outputs strictly isothermal conditions was to be ensured during the experiment. Therefore forming tools and glass samples have been heated up in a cylindrical laboratory furnace of special design that is an integral part of a laboratory apparatus. The temperature of the pistons and the glass samples was held constant by an electronic temperature controller. Temperature was monitored by thermocouples (type K – chromel-alumel), two of them are soldered into the working surfaces of pistons and third one is located close to the free surface of the glass sample. To avoid sticking of the glass melt to the steel working plates, 0.27 mm thick mica foils were put in between the glass and working surfaces.

Application of this method allowed estimating the viscous-elastic response of glass to the constant piston velocity (range the 0.05 – 8 mm/s) and temperature interval corresponding to the viscosity of 10^7 to 10^{10} Pa.s.

3. Realized experiments

Viscous-elastic responses were measured for different kinds of silica glass (soda lime glass, float glass, bottle glass, lead crystal). In this paper the rheological response of cylindrical samples made of soda lime silica glass is estimated. Measurements were carried out in the temperature range 591 – 683 °C (corresponding to viscosity 10^7 – 10^{10} Pa.s) at speed varying in the range 0.05 – 4 mm/s. In Fig. 1 the progress of typical force response of the sample of mentioned chemical composition is drawn up. Forming glass samples, development of the pressing force runs in two qualitatively different stages. In the initial loading stage (compression interval $\Delta h = 0 - 1$ mm), the elastic strain component is initiated in glass melt to a great extent. Viscous flow of glass melt becomes dominant later on (above compression of 1 mm).

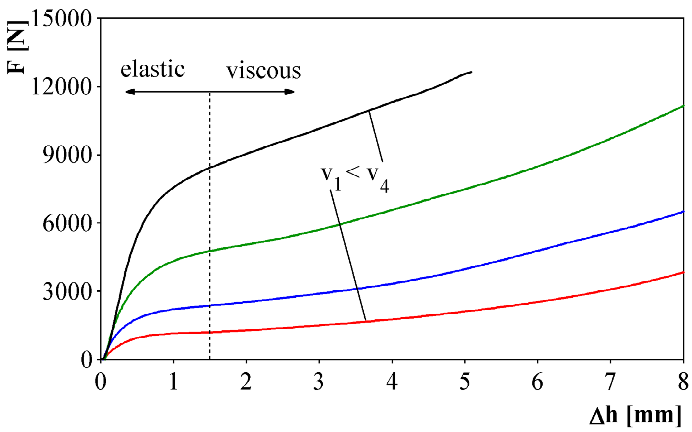

Fig. 1: Typical courses of force response of soda lime silica glass under compression load.

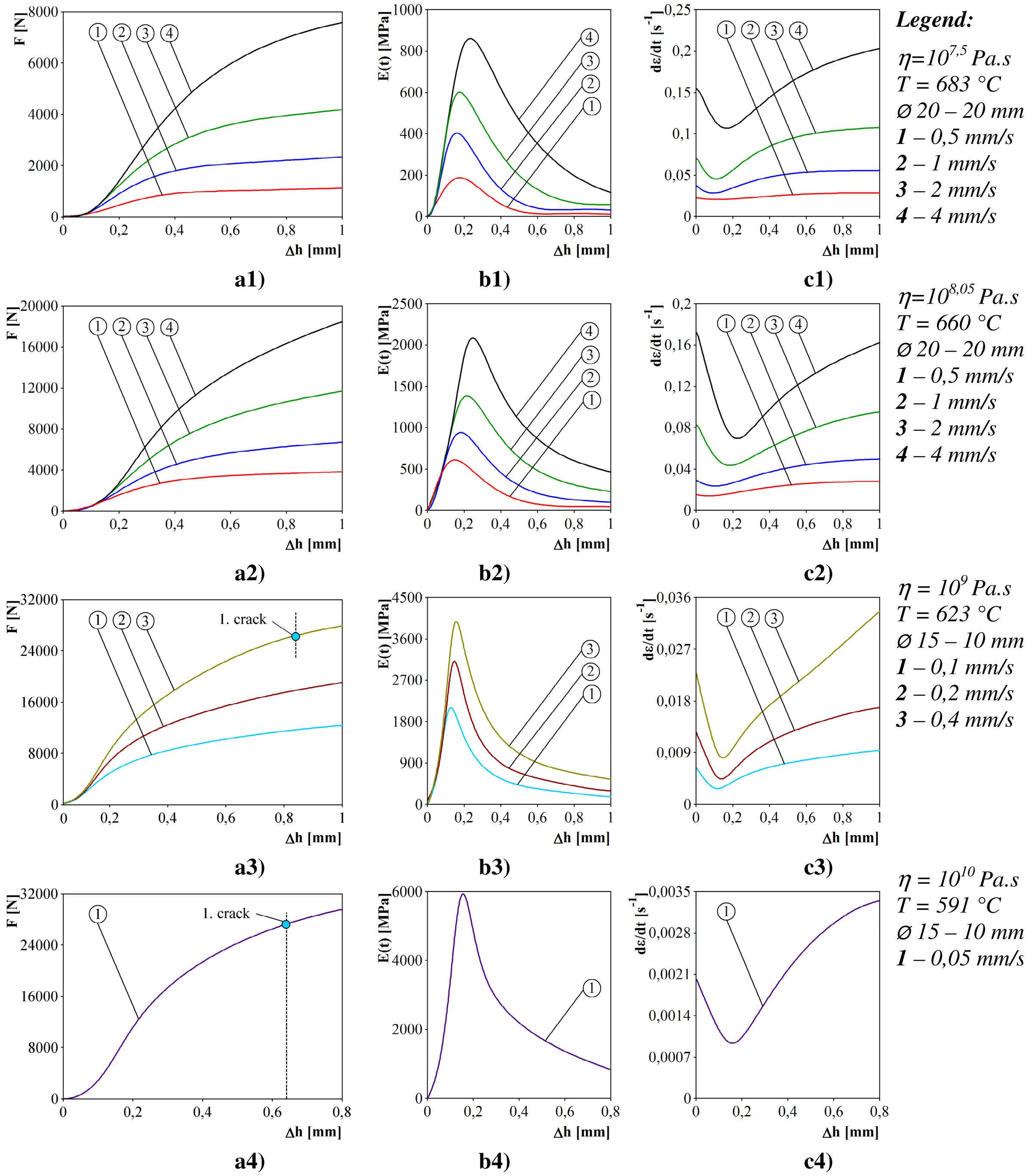

Fig. 2: The pressing force variation depending on the sample real compression (a1 – a4) and corresponding behavior of elastic moduli (b1 – b4) for temperatures chosen from the range (591 – 683 °C) at different speeds of the plunger movement (c1 – c4) document the strain rate variation during the pressing process depending on the sample real compression.

Experimentally determined viscous-elastic response of glass allows evaluation of instantaneous values of the elastic modulus $E(t)$ as

$$E(t) = \frac{d\sigma(t)}{d\varepsilon(t)} = d\left(\frac{F(t)}{A(t)}\right) / d\left(\frac{\Delta h(t)}{h(t)}\right). \tag{1}$$

Actual value of train rate was expressed as follows

$$\dot{\varepsilon} = \frac{d\varepsilon}{dt} = \frac{\dot{h}}{h(t)}, \tag{2}$$

where $h(t)$ is the sample height in time t; $A(t)$ is an instantaneous value of the sample cross section in time t; $\Delta h = h_0 - h(t)$ is an instantaneous value of the altitudinal deformation; h_0 is the sample initial height and $\dot{h}$ is a speed of the pressing plunger movement.

Due to increasing temperature both the instantaneous elastic modulus $E(t)$ and compression force decreases. Under temperatures corresponding to the viscosity of approximate 10^{10} Pa.s speed of working piston does not influence the course of deformation characteristic (sample behaves as an elastic substance).

4. Computer modeling

Final objective of realized experiments was to evaluate viscous response of glass samples under subjected load with emphasis on analysis of influence of the strain rate variation on the shear thinning generation process.

For the evaluation of the nature of viscous flow of soda lime silica glass under variable piston velocity the whole course of the experimental pressing was numerically simulated. The time running of individual experiments (isothermal compression process, under real boundary conditions) was modeled by means of customized FEM (Finite Element Method) code.

Special attention was paid to the specification of material properties – piston was considered to be rigid body. Friction conditions between pistons and working surfaces and sample were defined through the shear-based model. Samples were subjected to the real compression rate.

Contrary to the virtual simulation of the initial part of curve of the force response (up to compression of 2 mm – i.e. for description of pure viscous-elastic response) where the Maxwell viscous-elastic model has to be used [Matoušek, 2007], viscous flow in the later stages of compression experiment can be satisfactory described through Newtonian model. Glass melt was assumed to be incompressible, therefore relation between equivalent stress (σ_{ekv}) and equivalent strain rate ($\dot{\varepsilon}_{ekv}$) was expressed as

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

Used approach allowed analyzing individual stages of realized experiments (Fig. 3) and affording data for evaluation of critical deformation rates for onset of nonlinear behavior – shear thinning (Fig. 4-5). With respect to the visco-elasto-plastic character of glass melt behavior it is necessary the mechanical energy dissipation to be taken into account. Therefore two limited cases were analyzed, as follows:

- without mechanical heating,
- with total viscous heat dissipation.

Increase of temperature due to heat dissipation was described by equation:

$$\Delta T_{max} = \frac{1}{m \cdot c} \int_{0}^{\Delta h} F \, d\Delta h \, . \tag{4}$$

where m is weight of the sample, c is specific heat.

Analyzed problem was solved as coupled thermo-mechanical one in the environment of customized FEM code MSC MARC (Fig. 3). The courses of real viscous response (experimental data) and virtual force response of glass melt samples (for both cases mentioned above) at viscosity of $10^{7.5}$ Pas and $10^{8.05}$ respectively are drawn up in Fig. 4-a, b.

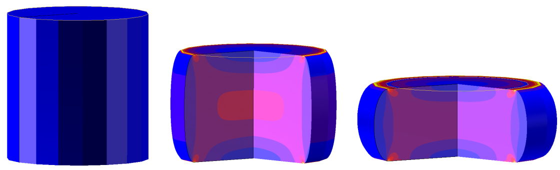

Fig. 3: FEM model.

It is evident that the influence of mechanical energy dissipation on the force response course rises up with the piston speed increase (4) steadily. Mutual comparison of experimental measurements and FEM model outputs results in a good agreement at small piston speeds (strain rates respectively). Viscous response of float glass can be approximated with classical Newtonian model up to piston speed 4 mm.s^{-1} at viscosity $10^{7.5}$ Pas (Fig. 4-a) and up to speed of 2 mm.s^{-1} at viscosity $10^{8.05}$ Pas (Fig. 4-b) relatively exactly. Due to increase both the piston speed and a compression level (strain rate respectively) virtual force response (the correct model considering total heat dissipation) starts to deviate from experimental data gradually.

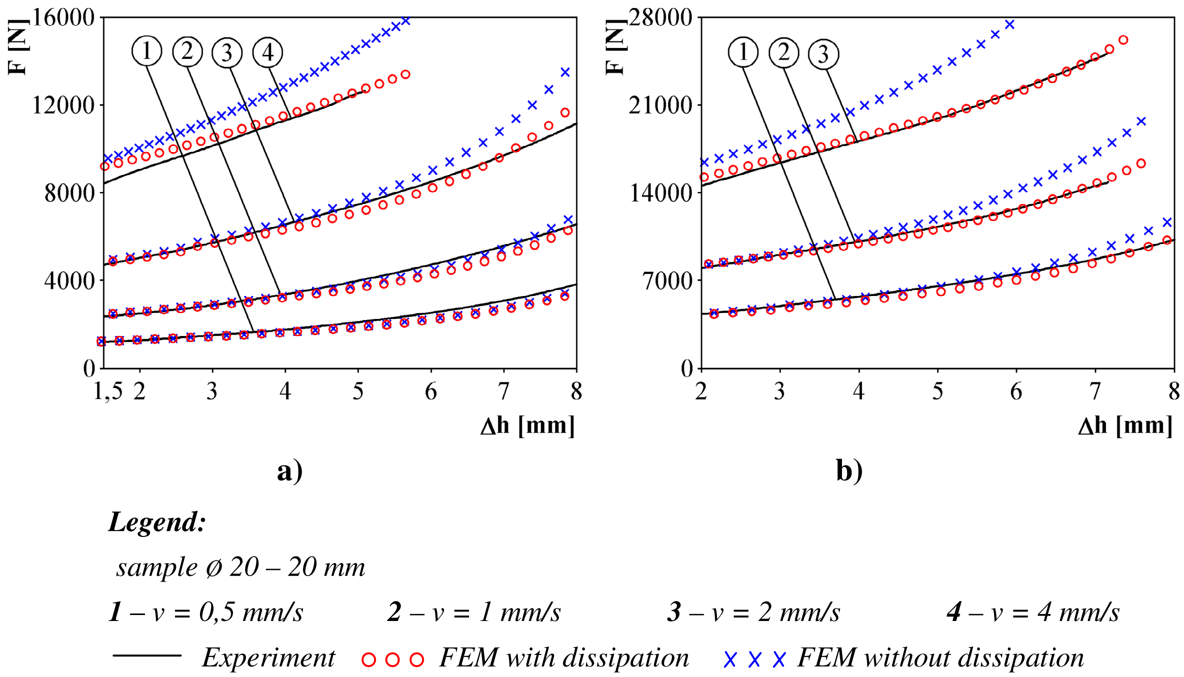

Fig. 4: Comparison of compression load-real deformation curves between experimental and simulation outputs at different viscosities; a) $\eta = 10^{7.5}$ Pa.s; b) $\eta = 10^{8.05}$ Pa.s.

Analysis of this tendency results in the evaluation of dependence of critical strain rate for onset of non Newtonian behavior (shear thinning) of soda lime silica glass melt on Newtonian viscosity (Fig. 5). For the soda lime silica glass characteristic equation defining critical strain rate for onset of shear thinning in the form (5) was found (its validity tested in the viscosity range $10^{7.5} - 10^{8.5}$ Pa.s).

$$\dot{\varepsilon} = A\, e^{-B \cdot \eta_0} \,, \qquad (5)$$

where *A, B* are constants (A = 3735; B = -1.27).

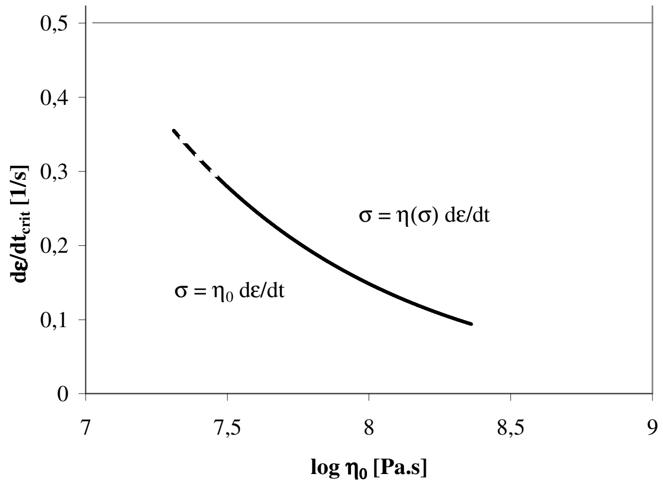

Fig. 5: Critical strain rate at which the initial compression load begins to deviate from theoretical linear plot as a function of the equilibrium viscosity of soda lime silica glass

5. Conclusions

In the paper analysis of viscous-elastic response of soda lime silica glass under variable piston speeds at different viscosities was done. Instantaneous moduli for different piston speeds (0.05 – 4 mm/s) at viscosity from $10^{7.5}$ to 10^{10} Pa.s were estimated. On the base of the comparison of the real experiments with the virtual models of the same the critical strain rates for onset of shear thinning were determined. For description of critical value of strain rates the exponential equation was suggested.

Acknowledgement

This work was done under research project no. MSM 4674788501 which is financed by the Ministry of Education, Youth and Sports of the Czech Republic.

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