STRESS RELAXATION CHARACTERISTICS OF SELECTED COMMERCIALLY PRODUCED GLASSES

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This paper describes a quantitative method of stress relaxation measurement in prismatic glass samples during two different time-temperature regimes using the Sénarmont compensator. Four types of glass (Barium crystal glass, Eutal, Simax, and Container glass) were subjected to observation in an assembled measuring device. Results will be used for parameterization of the Tool-Narayanaswamy-Mazurin model and consequently implemented in a finite element method code.

INTRODUCTION

Generation and relaxation of mechanical stress in glass play the key role in technological process of glass production. This was the reason why we focused our attention on commercially produced glasses for these measurements. The process of stress relaxation is described in detail by Gordon and Narayanaswamy in the work [1]. It is necessary for glass companies to optimize the process to reduce their costs, customize the shape of their products, and decrease the processing time if they want to succeed among the strong. One commonly used optimization tool is the finite element method (FEM). Available professional software based on this method deal mostly with empirical and simplified thermo-rheological models which are of little use in the field of the annealing and forming process of glass. Hence, the results obtained from measurements described in this work will be consequently used for more accurate parameterization of Tool-Narayanaswamy-Mazurin (TNMa) model and implemented in a FEM code. Due to the fact that obtaining the accurate quantitative values of stress is a relatively complicated process, which might be affected by an error, we choose to measure such characteristics of mechanical stress relaxation that are invariant to the potential proportional error.

EXPERIMENTAL

The measuring device (Figure 1), based on Sénarmont method [2, 3], consists of three basic parts. The first part consists of a light source and a polarizer. The second part is an electrically heated tubular horizontal oven where glass specimen is placed. A thermocouple, allowing the temperature control of the internal oven space by the programmable regulator CLARE 4.0 (CLASIC CZ s. r. o., Řevnice), is a component of the oven. The last part of this equipment consists of a quarter-wave plate, an adjustable analyzer, and an eyepiece with a scale.

Four commercial types of glass were studied. Their compositions are shown in Table 1. One prismatic sample from each type of glass of dimensions (5 × 5 × 20 mm) was cut and its bases were polished. Each sample was heated in the oven up to its annealing point temperature (Table 2) and kept at this temperature for 10 minutes. After removing from the oven, the samples were cooled in the air in order to generate reproducible stress. Stress measurements were performed on such prepared samples in the axial direction at a point in the middle of an edge of the square base.

Two types of measurement were performed. In the first one, we measured the temperature at which the tension disappears while heating the sample at constant heating rate. Six different constant heating rates

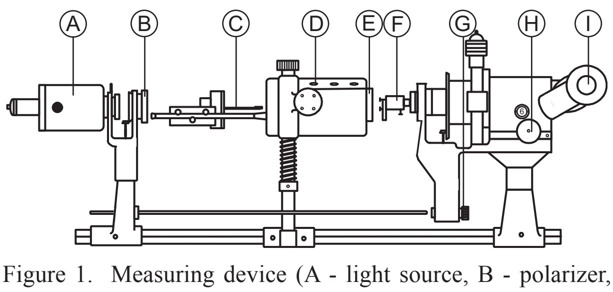

Figure 1. Measuring device (A - light source, B - polarizer, C - thermocouple, D - oven, E - monochromatic filter, F - quarter-wave plate, G - analyzer knob, H - horizontal shift, I - eyepiece).

were used, namely: 1°C/min, 2°C/min, 5°C/min, 10°C/min, 20°C/min, and 30°C/min. In the second type of experiment, the full relaxation time of stress was mea-sured at isothermal condition. Full relaxation time stands for a time period that starts by reaching the chosen temperature which is kept constant (temperature of full relaxation) and ends when the stress we measure is zero. The first particular temperature was derived from the annealing point. Each next measurement was performed at a temperature decreased by 10°C. Samples in this experiment were heated to the requested temperature from laboratory temperature at constant heating rate of 10°C/min.

Each single experiment was repeated five times in order to get more statistically significant result.

Table 1. Chemical composition of glasses in wt. % [4, 5].

Oxide	Ba - cryst.	Simax	Eutal	Container glass
SiO_2	72.68	83.22	56.37	71.15
Al_2O_3	0.70	1.24	8.94	0.89
CaO	10.23	–	21.37	7.79
BaO	1.91	–	–	–
Na_2O	10.99	3.67	0.19	12.79
K_2O	3.49	0.68	0.58	0.42
B_2O_3	–	11.19	7.67	–
MgO	–	–	4.88	6.96

RESULTS AND DISCUSSION

Measured results from the first type of experiment can be seen in Figure 2. The temperature of full relaxation increases in following order: Container glass < Barium crystal glass ≈ Simax << Eutal. From the practical speed independency of Eutal glass, the process of its relaxation was measured to be the fastest one, what can be caused by high networking degree of the glass structure.

The results of the experiments of the second type are shown in Figure 3. To the contrary of the preview experiment, results of the second experiment are well separated in the temperature scale. The highest full-relaxation times at the equivalent temperature shift were observed for Simax and Eutal glass what corresponds with the highly cross-linked glass structure. The order of the full relaxation time from the second experiment matches the order from the previous experiment.

CONCLUSION

Two types of experiment on four glass samples of different composition were performed. It has been proved that the hard glasses are characterized by higher full relaxation temperature and longer full relaxation time at the same temperatures. Both experiments give sufficiently exact data so that the results can be used for parameterization of the TNMa model and consequently implemented in a FEM code.

Table 2. Viscosity points (°C) [4, 5].

Name of point	log (η/dPa.s)	Ba - cryst.	Simax	Eutal	Container glass
Littelton Softening point	7.65	709	830	833	720
Deformation point	11.5	553	645	665	550
Annealing point	13.0	528	550	635	530
Strain point	14.5	490	494	594	498

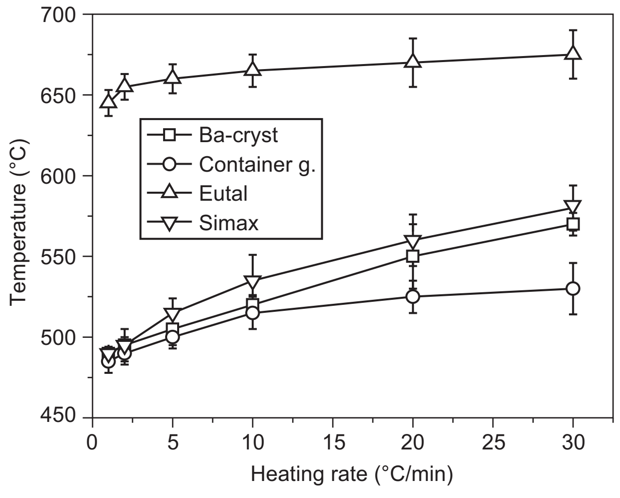

Figure 2. Temperature of full relaxation at different heating rate.

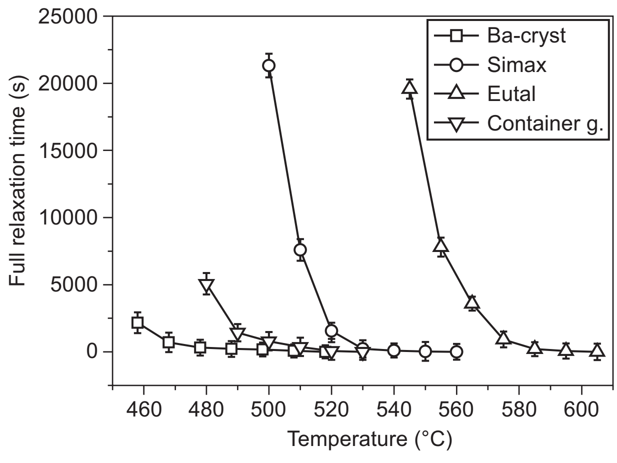

Figure 3. Full relaxation time of tension at various temperatures (heating rate 10°C/min).

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