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EXPERIMENTAL DETERMINATION OF VISCO-ELASIC PROPERTIES OF LAMINATED GLASS INTERLAYER

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Abstract: *A specific experimental procedure employing the dynamic shear rheometer for the determination of dynamic properties of laminated glass interlayer is examined. An ethylene-vinyl-acetate (EVA) polymer interlayer experiencing strong rate and temperature depended behavior is considered as one particular example. This has been confirmed by performing cyclic measurements at various frequencies and temperatures to get corresponding variations of storage, loss, and complex dynamic shear moduli. One particular master curve for the reference temperature $T_R = 20\,°C$ was constructed for both the storage and loss modulus. Such master curves serve as a stepping stone for the calibration of the Maxwell-chain viscoelastic model needed in the actual analysis of the laminated class.*

Keywords: Laminated glass, Viscoelasticity, Complex shear moduli, Rheometer, Generalized Maxwell-chain model.

1. Introduction

Apart from classical application to windows the use of glass as structural elements with particular requirements for strength and safety has been on continuous rise over the last few decades. In this regard, attention has been accorded to the development of laminated glass. Multilayered glass units composed typically of two glass sheets bonded to a polymer interlayer have expanded into the building constructions, such as roof and floor systems, columns, staircases, hurricane-resistant windows, or pedestrian bridges. Such applications inevitably call for a proper characterization of the behavior of such structures often subjected to complex mechanical and atmospheric loading conditions adopting reliable and accurate computational methods.

While glass units can be considered as perfectly homogeneous, isotropic material experiencing more or less brittle response, the behavior of polymer interlayer is more complex manifesting itself by rate and temperature dependency. The most common interlayers are made of a polyvinyl butyral (PVB), ethylene-vinyl acetate (EVA) or ionoplast polymer. At minimum the viscoelastic constitutive models should therefore be adopted. This essentially precludes the use of analytical methods and suggest the application of various numerical methods such as the finite element method (FEM). The literature offers a number of methodologies allowing for the implementation of composite laminated glass into FEM computations. The most simple but the least accurate concept of effective thickness was introduced by (Galuppi et al., 2012) where the laminated glass element is converted to a solid one with an equivalent stiffness. Much more accurate approach, particularly when studying the geometrically nonlinear effects, has been presented in Zemanová (2014). Therein, the refined laminated plate theory by Mau (1973) was adopted to account for the viscoelastic behavior of the polymer interlayer. In particular, the viscoelastic properties of the polymer interlayer are described by the generalized Maxwell chain model, which describes the material response at a material point through a parallel connection of purely elastic spring and several

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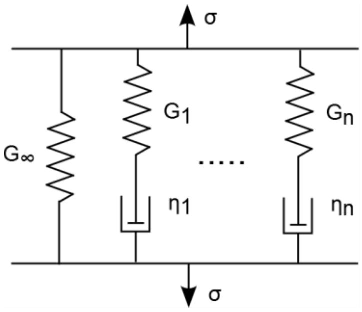
Maxwell cells, see Fig. 1. Since the behavior of the interlayer is driven by shear, see Fig. 2, we consider the formulation in terms of the viscoelastic shear modulus G and constant Poisson's ratio.

The dependent response of the shear modulus is then written in terms constants $G_\infty; G_1 \dots G_n$ and selected relaxation times τ_i as

$$G(t) = G_\infty + \sum G_i\, e^{-\frac{t}{a\tau_i}} = f(G_\infty, G_1, \dots, G_n). \quad (1)$$



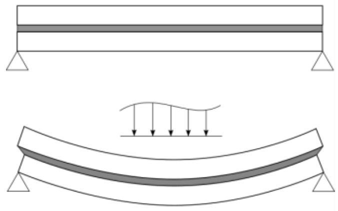


Fig. 1: Generalized Maxwell chain model. *Fig. 2: Shear strain in interlayer.*

The same material constants can be used for the description of the response to loading caused by harmonic excitation

$$G^*(\omega) = G'(\omega) + \mathrm{i}G''(\omega) = f(G_\infty, G_1, \dots, G_n). \quad (2)$$

The ability of the generalized Maxwell chain model to represent both the static shear modulus given by Eq. (1) and the dynamic shear modulus given by Eq. (2) thus allows us to investigate the viscoelasticity and temperature dependency of the polymer interlayer with the help of dynamic mechanical analysis. Typically, the tests are conducted in tension mode, see e.g. (Mohagheghian et al., 2017), resulting into the complex Young modus. To avoid a subsequent transformation into the shear modulus we proceed in the footsteps of (Andreozzi et al., 2014) and perform the dynamic measurements directly in the shear mode using rheometer testing device as described in detail in Section 2.

It has been already mentioned that the mechanical properties of the polymer layer depend strongly on temperature. Increasing temperature significantly reduces the interlayer stiffness. The temperature dependency is conveniently expressed in terms of the temperature shift factor $a(T)$, recall Eq. (1), fitted to the William, Landel and Ferry (WLF) equation

$$a(T) = \exp\left(\frac{-C_1(T-T_R)}{C_2+T-T_R}\right). \quad (3)$$

where C_1, C_2, are the model parameters, T_R is the reference temperature and T is the actual temperature. The model parameters are found by constructing a master curve from measurements carried out at various temperatures but at the same frequency sweep. The individual curves are then horizontally shifted to match the results corresponding to the reference temperature that would be obtained at either much smaller or much higher frequencies such that $G^*(\omega, T) = G^*(a(T)\omega, T_R)$.

2. Measurement setup

The dynamic shear rheometer HAAKE MARS, typically used for asphalts, was employed to derive the frequency characteristics of the polymer interlayer with attention limited to EVA material. This device operates on the plate-plate shear principle and allows for stress as well as strain loading conditions applied either in a static (simple rotation) or dynamic (oscillatory) mode under the prescribed temperature. Unlike asphalts, which do not require any special treatment, testing the glass composite sample requires more elaborate approach as described in detail in (Andreozzi et al., 2014 and Zulli et al., 2016).

The measurements were performed on layered cylindrical samples having diameter 20 mm and thickness of 5 + 0.76 + 5 mm. To ensure that the oscillatory loading is taken by the interlayer only a relatively stiff epoxy glue had to be applied when mounting the specimen on the two plates (one fixed and one rotating). Figs. 3 and 4 show a sample of the laminated glass fixed between the two plates.

Note that testing the actual composite is particularly advantageous as the polymer properties may change during the composite production and thus may differ from the properties of the original material. Clearly, this cannot be accounted for when testing the polymer interlayer only, e.g. in tension.

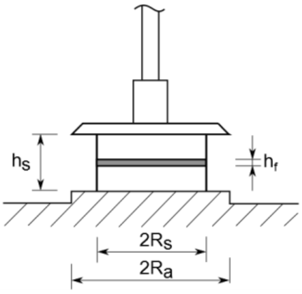


Fig. 3: Rheometer setup.

Fig. 4: Specimen glued to rheometer plates.

In the rheometer oscillatory tests the cyclic loading undergoing a certain frequency and temperature sweep is prescribed. The rheometer provides directly the frequency dependent variation of the storage and loss moduli for a given temperature. However, such results apply to the geometry of the entire composite. Since it is expected that the entire torsion is concentrated in the EVA sample, the results provided directly by the rheometer have to be adjusted to reflect properly the thickness h_f and the radius $R_s = 10$ mm of the interlayer with reference to the actual sample thickness h_s and the plate radius $R_a = 12.5$ mm in our particular case, see Fig. 3. As suggested in (Andreozzi et al., 2014) the complex modulus G_f^* of the interlayer relates to the rheometer modulus G_s^* as

$$G_f^* = \frac{R_a^4}{R_s^4} \cdot \frac{h_f}{h_s} G_s^*. \tag{4}$$

3. Measured results

The tested specimens were drilled from laminated glass with two-ply EVA (Ethylene-vinyl acetate) polymer interlayer. Each of six tested samples experienced a slightly different total height. On average the interlayer thickness h_f amounted to 0.81 mm with standard deviation of 0.028 mm. The measurements were performed for the following set of temperatures: 10 °C, 20 °C, 30 °C, 40 °C, 50 °C, 60 °C. The following range of frequencies was considered: 0.001 Hz, 0.01 Hz, 0.05 Hz, 0.1 Hz, 0.5 Hz, 1 Hz, 5 Hz, 10 Hz, 20 Hz, 30 Hz, 40 Hz, 50 Hz. The range of frequencies 50 Hz – 100 Hz was also examined. However, for the present testing device, as seen from the available results, the predicted response is highly unreliable suggesting a rapid decrease in the storage modulus for frequencies beyond 50 Hz, see Fig. 5. The results in Fig. 6, pertinent to the loss modus, show even larger discrepancies.

As already mentioned the proposed experimental program was applied of the six specimens one day after being fixed to the testing device to allow for sufficient curing of the epoxy glue. Performing the entire temperature and frequency sweep required about 9 hours. The resulting variations of the storage and loss moduli are plotted for one particular specimen in Figs. 5 and 6. For each specimen such measurements were repeated three times one day apart (runs 1 – 3) to examine the influence of both loading and heating/cooling sequence. The results for the reference temperature $T_R = 20$ °C are shown in Fig. 7. This trend, manifested itself by the stiffness reduction with repeating tests, has been observed for all samples. Such a dependency on the history of loading is, however, difficult to reflect through a simple Maxwell chain model.

The final step needed for the calibration of the Maxwell chain model is to construct a master for the selected reference temperature set equal to $T_R = 20\,°C$ in our present study. The master curve is created by horizontal shifting (in a logarithmic scale) original curves in Fig. 5. This automatically prolongs the frequency range. The result appears in Fig. 8. Such a master curve then allows us to determine the parameters C_1 and C_2 in Eq. (3), being equal to 300 and 350, respectively.

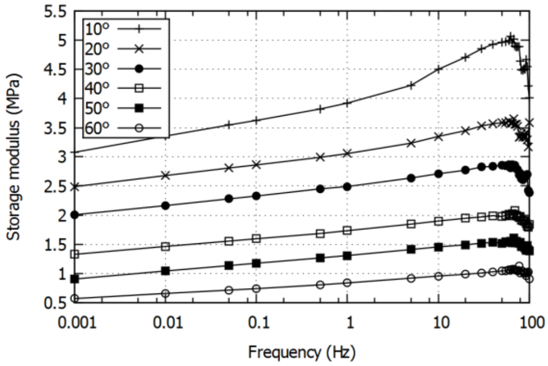


Fig. 5: Storage modulus.

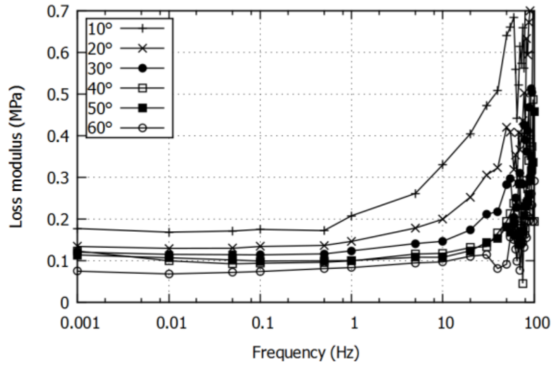


Fig. 6: Loss modulus.

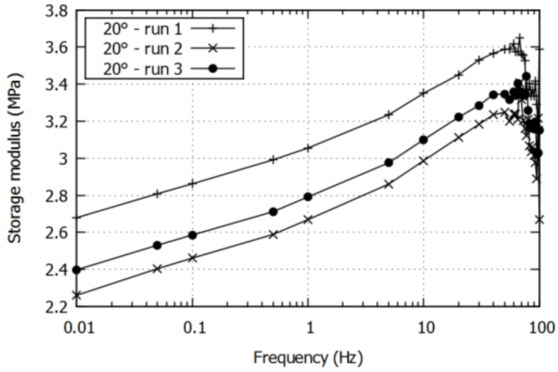


Fig. 7: Storage modulus for three consecutive runs.

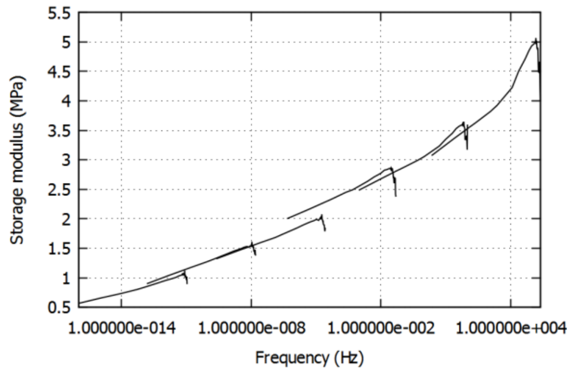


Fig. 8: Master curve for storage modulus.

4. Conclusions

This paper summarizes the results of the dynamic oscillatory measurements using the dynamic shear rheometer to address the viscoelastic behavior and temperature dependency of the EVA interlayer in a three-layer laminated glass composite. The applicability of the selected testing device was demonstrated through the construction of one particular master curve, which in turns is to be used in the calibration of the generalized Maxwell chain model needed in complex FEM simulations of a low velocity impact. Similar experimental program is currently under way to acquire the viscoelastic properties of PVB interlayer. In this regard, special attention will be devoted to the influence of loading history in order to reconcile the results plotted in Fig. 7.

Acknowledgement

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