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Material Properties of Polymeric Interlayers under Static and Dynamic Loading with Respect to the Temperature

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Looking at a current architecture, there are many examples of glass load bearing structures such as beams, panes, balustrades, columns or even stairs. These elements are mostly made of laminated glass panels. Panels are bonded together with polymer interlayer significantly influencing a shear forces transfer between them. There is still overall lack of knowledge in the task of shear forces transfer between these panels. It principally depends on the polymer stiffness, which is affected by an ambient temperature, humidity and load duration. Civil engineers currently tend to design laminated glass members on the safe side, generally not taking laminated panels interaction provided by the interlayer into account. This approach leads to uneconomical and robust glass bearing members significantly preventing the use of laminated glass more extensively. There are many polymer interlayers made for structural laminated glass applications available on a market. Most of them differ in stiffness and other important properties therefore these must be experimentally examined to design safer and more economical laminated glass members. This paper is focused on the shear modulus of PVB (polyvinyl-buthyral) and SentryGlas® (ionoplast) experimental investigations as a function of temperature and loading ratio. It is possible to find out these functions by static creep or relaxation tests as well as by dynamic mechanical thermal analysis-DMTA. A lot of DMTA experiments in shear with the aforementioned interlayers in various loading conditions have been performed in order to determine their shear stiffness. It also enables to identify their Prony parameters as a part of the next survey. Experimentally verified common polymer interlayer stiffness helps engineers to design safer and cheaper glass constructions. This is the way how to extend the use of laminated glass in a current architecture.

Keywords: Glass, Loading ratio, Temperature, Stiffness, Interlayer, DMTA, Strain

1. Introduction

Laminated glass, which is composed of two or more glass plies bonded together by transparent polymeric interlayer, is the subject of a current research (Serafinavičius and Lebet et al. 2013; Serafinavičius and Kvedaras et al. 2013). Bonding process is made usually in autoclave under the pressure of 0.8 MPa. The use of laminated glass for load bearing structural elements becomes necessary because of its load bearing capacity. Wheter the glass strength is exceeded under a certain load, glass fragments stay adhered to the interlayer and do not fall down potentionally causing an injury. Therefore, laminated safety glass is a suitable solution for load bearing glass applications installed above the heads of users and for other structural elements such as staircases and balustrades. Civil engineers currently tend to design laminated safety glass on the safe side, regardless positive shear coupling of the plies. This approach leads to robust and too expensive laminated glass constructions principally because of the lack of interlayer´s properties. There are many different polymeric interlayers for laminated glass applications available on a market. These generally differ in a chemical composition and therefore in a stiffness too. Interlayer´s shear stiffness plays a significant role in a task of a stress distribution in laminated glass panels loaded perpendicularly to their plane. Wheter economic and reliable design is requested, interlayer´s shear stiffness becomes an important quantity. This paper introduces the experimental data gained from the static and dynamic loading tests performed at Klokner Institute CTU in Prague for PVB and SentryGlas® polymeric interlayers, describes the experimental programme and proves a strong dependence of their stiffness on a temperature and loading rate.

2. Experimental programme

2.1. Materials and equipment

Two types of the interlayers such as PVB Trosifol BG-R-20 and SentryGlas were experimentally examined. Trosifol BG-R-20 will be denoted as Trosifol and SentryGlas will be denoted as SG hereafter in the text. Test specimens were made of 2 annealed float glass panes bonded by transparent interlayer at shear area 50 x 50 mm in autoclave. Specimen´s dimensions are shown in Fig. 1. Thickness of the interlayers was measured at six representative specimens along their shear area under the microscope. The average value of measured thickness for Trosifol was 1.49 mm and for SG it was 1.01 mm. Potenciometric linear transducers MMR 1011 were stuck on the sides of the specimen to be able to measure the relative slippage of the glass plies u in [mm] in the acting force direction, see Fig. 2a. There were totally 83 specimens statically and 3 specimens dynamically loaded with Trosifol interlayer. Then, 75 specimens were statically and 7 specimens were dynamically loaded with SG interlayer.

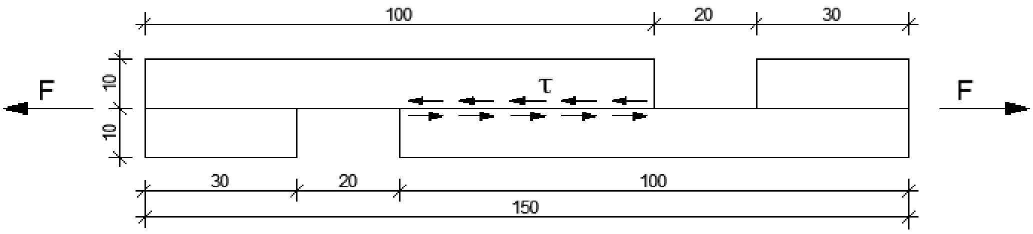


Fig. 1 Nominal dimensions of the testing specimen.

Fig. 2a) Specimen with potenciometric linear transducers, b) Specimen fixing in metal jaws (TIRA, MTS).

2.2. Test set-up

In the static loading tests, the specimens were put into the metal jaws of the testing device TIRA TEST, see Fig. 2b, and continuously loaded until their collapse. The experiments were performed as short duration tests to exclude the influence of time and were controlled by the plies displacement in three different loading rates such as 2 mm/min – short term, 0.5 mm/min – medium load and 0.125 mm/min – long term load. Moreover, the specimens were loaded in four different temperatures 0, 20, 40 and 60 °C such as room temperature multiplies in TiraTest TS 201 chamber. The temperature in the chamber was measured by Pt 1000 sensor. Therefore, the initial shear modulus was actually measured as a function of temperature and loading rate.

In dynamic mechanical thermal analysis tests (DMTA), the specimens were put into the metal jaws of the testing device MTS 500B with TiraTest TS 201 chamber, see Fig. 4. The tests were controlled by the displacement of the glass panes. The amplitude of MTS loading cylinder displacement was set as 0.2 mm for each loading cycle but real maximum transducers measured displacement was much lower-up to 0.1 mm due to other metal components axial deformation in the loading system. Temperature and loading frequency were changed during the tests. The range of testing frequencies was 0.05 – 4.95 Hz with the beginning at 0.05 Hz and finish at 4.95 Hz. Frequency step between the cycles was set as 0.05 Hz and the temperature was held constant in the whole range of testing frequencies (99 cycles). Prestressing force between the cycles was set as 1.2 – 1.5 kN depending on the testing specimen and its value assured reliable fastening of the specimen in metal jaws during the cycles not clearly influencing obtained results. Prestressing force duration between each cycle was set as 10 s and every cycle began from this set force without any further force steps. Cylinder´s displacement evoked interlayer´s shear strain according to the equation 2. The example of this strain as a function of time is displayed in Fig. 3. Tested temperature range for Trosifol was 25 – 40 °C and for SG it was 30 – 70 °C, both with a step of 5 °C. The temperature in the chamber was measured by Pt 1000 sensor and the temperature on the glass surface was mesured by two Pt 100 sensors stuck on the sides of the specimen.

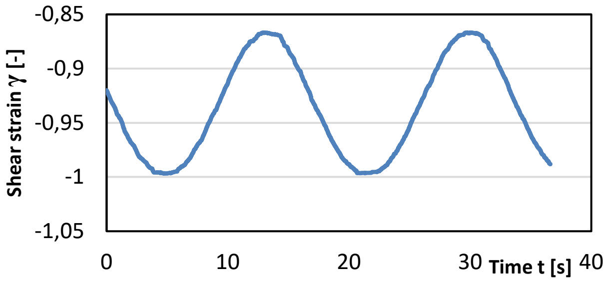
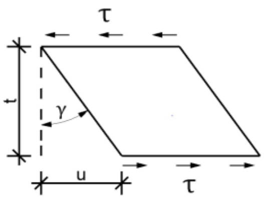


Fig. 3 Shear strain example of the interlayer during DMTA.

Fig. 4 MTS 500B with TiraTest 201 chamber for DMTA.

2.3. Evaluating method

Force transferred between glass plies caused shear strain of the interlayer, see Fig. 5. This strain γ can be calculated from the relative slippage *u* of the glass plies and the interlayer thickness *t*, see equation 1. This equation formulation refers to shear strain γ as the technical strain in both static and dynamic loading tests mentioned in the text. Large deflections are not considered. Additional bending moment resulting from the geometry of the testing device was neglected due to its low effect.



$$\gamma \approx tan\gamma = \frac{u}{t} \quad (1)$$

Fig. 5 Technical shear strain of the interlayer and its calculation formula

In the static loading tests, the appropriate value of shear stress obtained from the experiment of each specimen needed to be defined for the initial shear modulus assessment. Its acceptable value was determined as the intersection of the circle of 0.4 MPa radius with the corresponding strain-stress curve for all specimens besides Trosifol loaded at 60 °C where the value of 0.2 MPa was considered because of no higher values achieved. This procedure is indicated in Fig. 6 where it is possible to find out that stress-strain curves are not linear even in relatively low strain values. Therefore, all the initial shear modules presented hereafter should be considered as the secant ones. Circle radius values 0.4 (0.2) MPa were chosen in that way in order to obtain appropriate interlayer´s shear strains higher than those in real structures which assures that obtained initial shear modules are on the side of safety regarding concave stress-strain relations in the examined interval for most experimental cases.

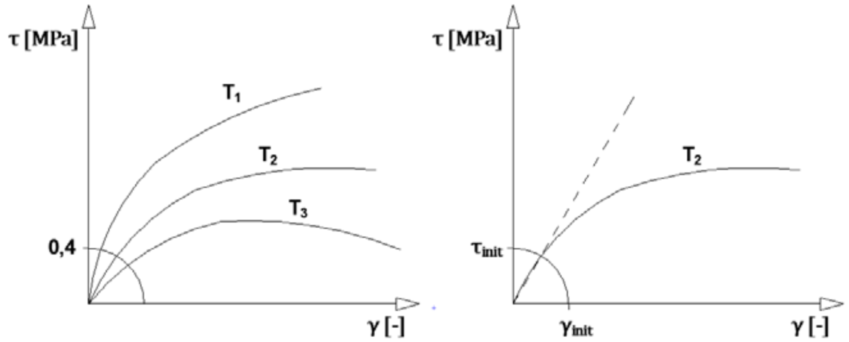


Fig. 6 Initial shear modulus evaluation where the temperatures $T_1 > T_2 > T_3$.

In the dynamic mechanical thermal analysis tests (DMTA), the shear strain of the interlayer was in each loading cycle controlled according to the equation 2

$$\gamma = \gamma_{max} sin(\omega t), \tag{2}$$

where γ_{max} is the maximum strain of the interlayer evoked by 0.2 mm loading cylinder´s displacement. This shear strain was in most cases in linear viscoelastic region-up to 1% (Kuntsche and Schuster et al. 2015) due to other loading device components axial deformation. ω refers to the loading angular frequency and t is the instantaneous time in each cycle. The appropriate shear stress (Lakes 2009) can then be calculated from the equation 3

$$\tau = \tau_{max}\, sin(\omega t + \delta), \tag{3}$$

where τ_{max} is the maximum shear stress of the interlayer evoked by 0.2 mm loading cylinder displacement and δ is the phase-lag between the oscillating strain and stress response. Stress-strain dependence in each loading cycle may be displayed graphically through the hysteresis loop (Lakes 2009) which is drawn in Fig. 7.

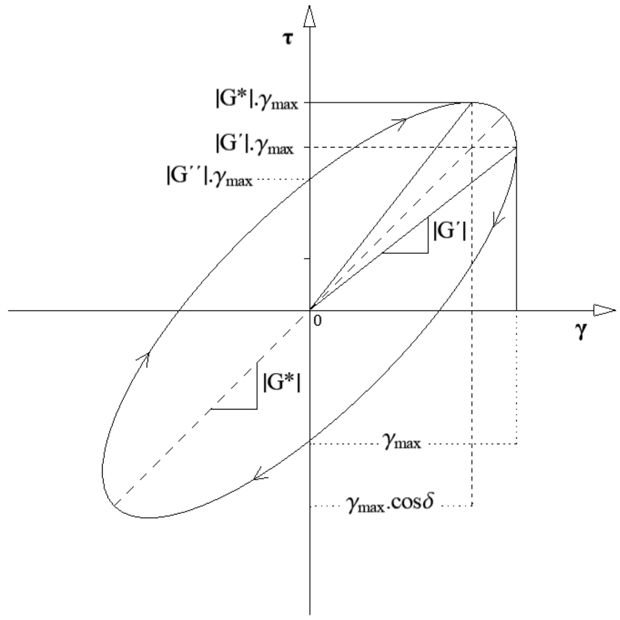


Fig. 7 Hysteresis loop showing stress-strain dependence in each loading cycle with its important points, (Lakes 2009).

On the hysteresis loop, there are some important points which can serve for required time and temperature dependent shear modulus *G(t,T)* evaluation. Loop symmetry axis slope indicates the value of dynamic complex shear modulus *G**. The slope of the line passing through the origin of the coordinate system and the point at which the maximum shear deformation is achieved indicates the value of the shear storage modulus *G´* being directly proportional to the average energy storage in each loading cycle (Ferry 1980). The intersection of hysteresis loop with shear stress axe allows shear loss modulus *G´´* determination. This value is directly proportional to the average dissipation or loss of energy as a heat in each loading cycle (Ferry 1980). Mathematical description of loading procedure in each sinusoidal cycle can be described by equation 4 (Lakes 2009)

$$\tau(t) = G^{*}.\gamma(t) = (G' + iG'').\gamma(t) = (G' + iG'').\gamma_{max}.\sin\omega t = G'.\gamma_{max}.\sin\omega t + \\ +G''.\gamma_{max}.\cos\omega t, \tag{4}$$

where $\gamma(t)$ is the shear strain input set according to the equation 2, γ_{max} is the maximum shear strain, ω is the loading frequency, i is the complex unit and G^*, G', G'' are the desired modules. These modules can be expressed as angular frequency ω or simple frequency f dependent keeping the relation $\omega = 2\pi f$ in mind. When $\omega t = \pi/2$, γ_{max} is achieved and then $\tau = G'\gamma_{max}$. On the other hand, for time $t = 0$ is $\gamma = 0$, therefore $\tau(0) = \gamma_{max}G''$ which is displayed on the hysteresis loop. All three complex modules were evaluated from the experimental hysteresis loops in MATLAB® for each loading cycle according the aforementioned procedure. Since DMTA delivers wide range of complex modules results in a frequency domain, a conversion to real numbers and time domain t is necessary. This can be performed by Fourier transform together with Boltzmann superposition principle for linear viscoelastic polymers (Kuntsche and Schuster et al. 2015). If this transform is not available, the following approximations are given here with the increasing accuracy to enable quick and simple way to compare the results gained from DMTA with those obtained by static relaxation tests (Kuntsche and Schuster et al. 2015):

$$G(t = 1/\mathrm{f}) = G'(\mathrm{f}) \tag{5}$$

$$G(t = 1/\mathrm{f}) = G'(f) - 0.4G'(\mathrm{f}/2) \tag{6}$$

$$G(t = 1/\mathrm{f}) = G'(f) - 0.4G''(0.4\mathrm{f}) + 0.014G''(10\mathrm{f}) \tag{7}$$

3. Results and discussion

3.1. Static loading tests

A sufficient number of specimens with both interlayers were tested in different loading rates at every temperature, see Table 1. The following charts summarise the most important stress-strain curves obtained from the experiments. Chart in Fig. 8 shows representative stress-strain curves for Trosifol BG-R-20. It is noticeable that when the temperature increases, material softens considerably. When Trosifol was loaded at 0 °C, it exhibited almost one hundred times stiffer response than when loaded at 60 °C as it is possible to find out in Fig. 8. Qualitatively speaking, molecular movement is thermally activated process and temperature increase causes polymer´s relaxation time reduction (Kuntsche and Schuster et al. 2015). 0 °C curves are almost linear apart from nonlinear curves in higher temperatures.

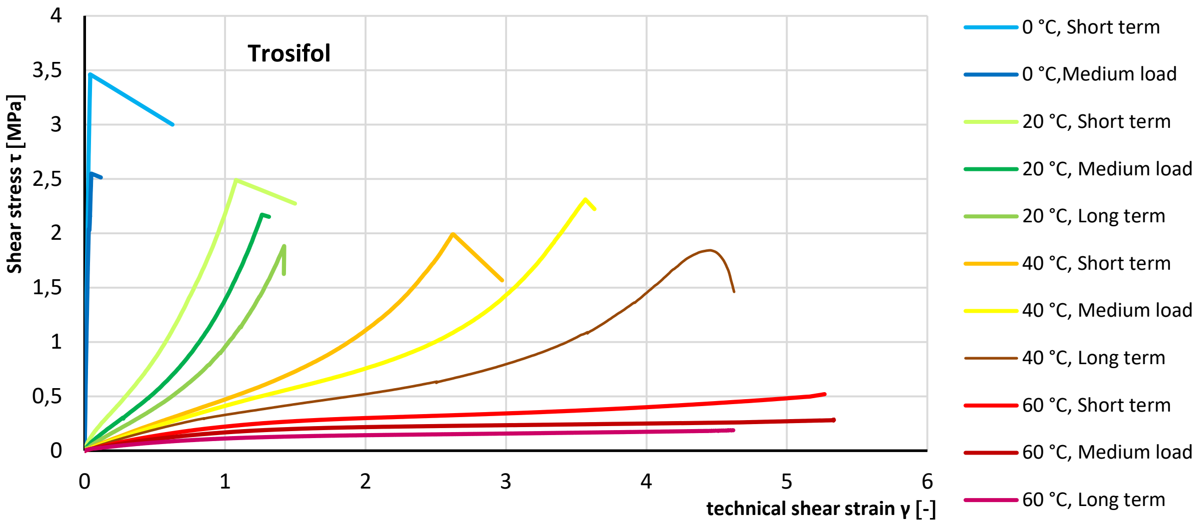


Fig. 8 Representative stress-strain curves for Trosifol interlayer.

Fig. 8 also proves the influence of the loading rate on the representative stress-strain curves of Trosifol interlayer. For example, the initial Trosifol stiffness increases about 40 % in every loading rate step when loaded at 20 °C which is in correlation with visco-elastic response of polymer. Lower the rate of the load, the effect of viscosity prevails

(Ferry 1980). Chart in Fig. 9 shows representative stress-strain curves for SentryGlas interlayer. In comparison with structural Trosifol, SG exhibits much stiffer response in the whole range of measured temperatures. And, apart from Trosifol, SG response for temperatures 0, 20 and 40 °C is almost solid-plastic so that stress-strain curves for these temperatures can be in Fig. 9 hardly recognised. The most significant stiffness reduction is attained between 40 and 60 °C. When SG was loaded at 40 °C, its initial response was almost twenty times stiffer than when loaded at 60 °C. The influence of the loading rate for SG is noticeable in case of 60 °C stress-strain curves having different shape. For the remaining curves, this influence can be hardly detected.

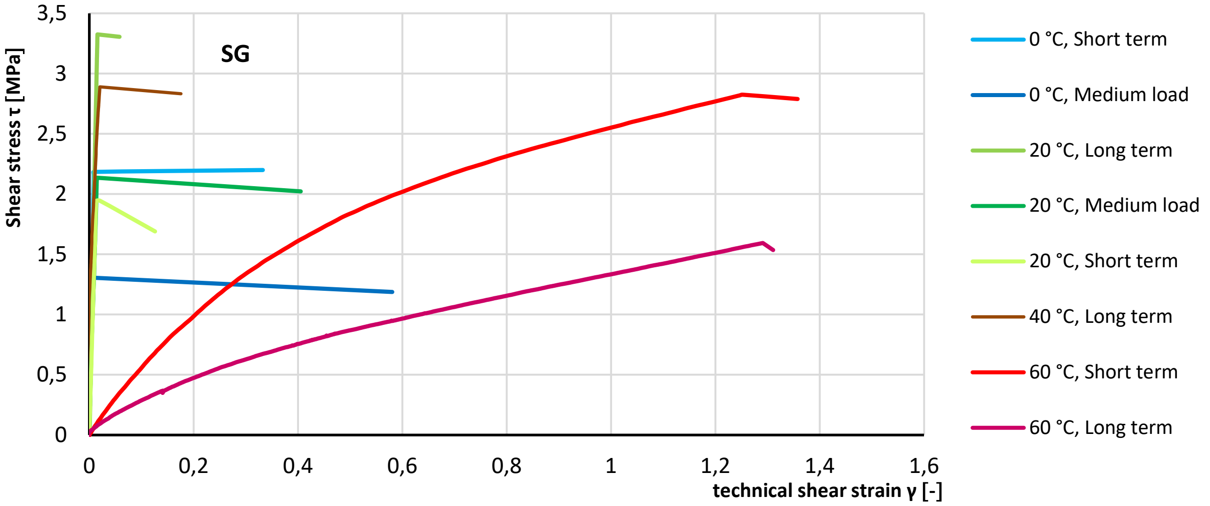


Fig. 9 Representative stress-strain curves for SG interlayer.

The initial secant shear modulus was determined for every stress-strain curve obtained from the experiments according to the following equation

$$G = \tau/\gamma, \quad (8)$$

where τ is the limit value of shear stress according to Fig. 6 and γ is the corresponding shear deformation determined by equation 1. In case of 20, 40 and 60 °C PVB curves, the corresponding shear strains were higher than 20 %. For 60 °C SG curves, strains were higher than 3%. Solid-plastic relations attained much lower strains. Table 1 summarises the average values of all initial secant shear modules calculated according to the equation 8 from the experimental stress-strain curves. It proves that the initial shear modulus is, as expected, temperature and loading rate dependent. When the temperature is increased, stiffness reduces and when the load is applied faster, stiffness increases. This statement doesn´t hold only for SG stiffness loaded at 20 °C. SG achieves, apart from structural Trosifol, satisfactory values of the initial shear modulus in the whole range of temperatures. It could be assumed that laminated glass panels with SG interlayer perpendicularly loaded would be much stiffer apart from those ones laminated with structural Trosifol.

Table 1: Initial secant shear modules obtained from the experimental data for Trosifol and SG.

Temperature [°C]	Number of Trosifol specimens	Trosifol, G_{mean} [MPa]	Number of SG specimens	SG, G_{mean} [MPa]
0 °C, Short term	4	144.13	3	684.65
0 °C, Medium load	7	103.32	7	290.83
20 °C, Short term	8	1.71	10	245.60
20 °C, Medium load	5	1.09	5	206.21
20 °C, Long term	10	0.80	10	214.23
40 °C, Short term	9	0.47	-	not specified
40 °C, Medium load	4	0.45	-	not specified
40 °C, Long term	10	0.31	10	144.49
60 °C, Short term	9	0.26	10	9.45
60 °C, Medium load	5	0.15	-	not specified
60 °C, Long term	7	0.12	9	5.44

3.2. Dynamic mechanical thermal analysis (DMTA)

Totally 3 specimens with Trosifol and 7 samples with SG were tested by DMTA. As it has been stated, the range of testing frequencies was 0.05 – 4.95 Hz with the frequency step 0.05 Hz between the individual cycles. The cycles were controlled by the loading cylinder´s displacement causing interlayer´s shear strain according to the equation 2. The following paragraph summarises stiffness-frequency curves obtained by DMTA for representative specimens and compares shear storage modules of both tested interlayers as a function of frequency and temperature. Curves in Fig. 10 of the representative Trosifol interlayer show, that the value of storage modulus depending on a frequency is affected by the ambient temperature. When the temperature is increased, the value of storage modulus *G´* for the fixed frequency decreases.

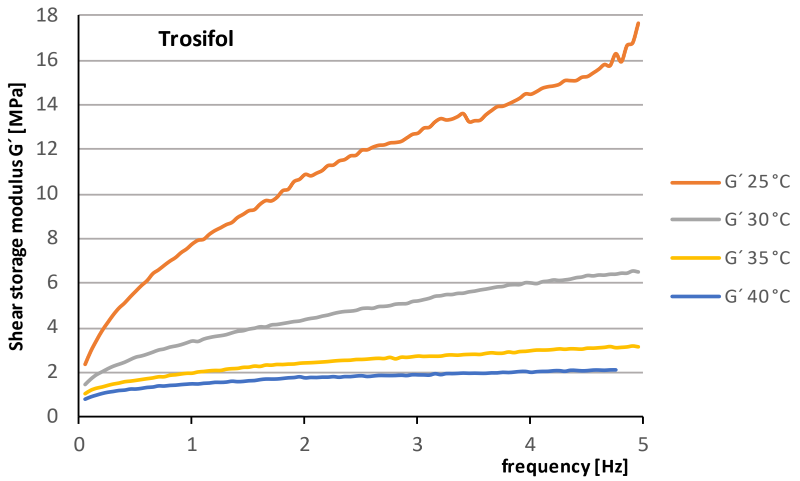


Fig. 10 Storage modulus-frequency curves of representative Trosifol interlayer obtained by DMTA.

Moreover, for the temperatures 25 and 30 °C the storage modulus increases with the increasing frequency (rate of strain). For higher temperatures, this feature is less noticeable. The interlayer´s delamination preliminarily interrupted the loading cycle at 40 °C and, as Fig. 10 indicates, Trosifol´s stiffness was at this temperature nearly negligible. Stiffness-frequency curves for the representative SentryGlas sample in Fig. 11 prove that the storage modulus *G´* is also temperature and whole range frequency dependent. For temperatures 30 – 45 °C, the curves are not sufficiently smooth but still have an increasing tendency. Their shape together with the values of storage modulus for these temperatures indicate, that the measurement was on the edge of the testing device accuracy. But apart from this, the drop of the stiffness values between the individual temperatures becomes noteworthy.

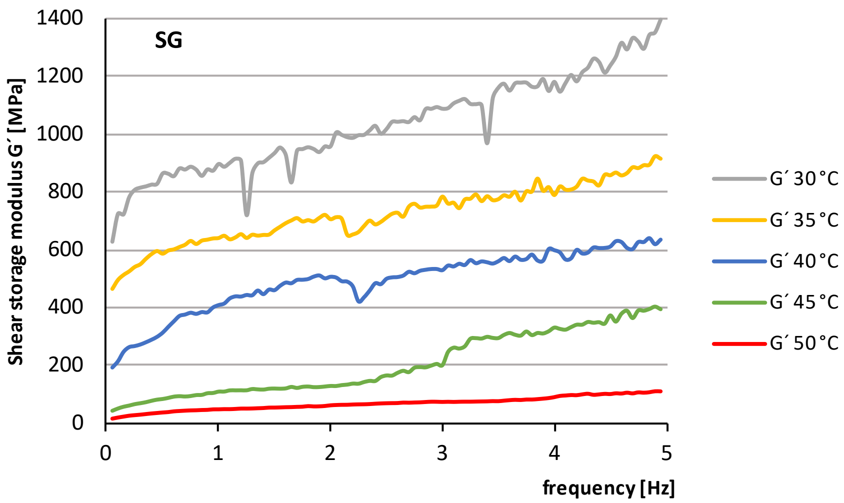


Fig. 11 Storage modulus-frequency curves of representative SG interlayer obtained by DMTA.

As the values of storage modulus for SG are still satisfactory at 50 °C and no specimen´s delamination was found, loading cycles at higher temperatures were performed as shown in Fig. 12. The frequency-stiffness curves of the representative SG were measured up to the temperature of 70 °C. Higher temperatures could not be tested because of interlayer´s delamination. These curves declare that SG is able to provide sufficient values of shear modulus *G* even in relatively high temperatures. This allegation becomes justified when checking the approximations given by the equations 5-7 and is going to be approved by the further research. Furthermore, stiffness-frequency curves are also temperature dependent. When the temperature is increased, the storage modulus values become lower. Another spectacular fact is that the storage modulus tends to increase with the increasing input frequency even at 70 °C as shown in Fig. 12. The phase-lag between the oscillating strain and stress response δ became negligible for both interlayers in the whole range of tested temperatures and frequencies meaning the fact that the stress response was almost in phase with the input strain and stifled the viscosity effect.

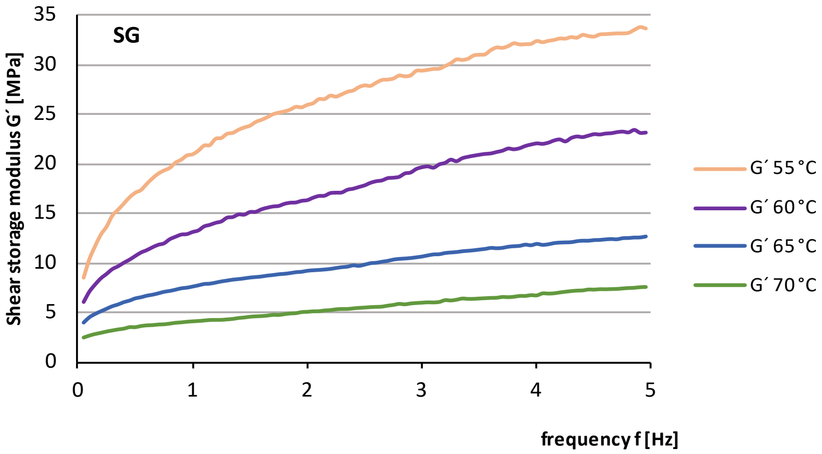


Fig. 12 Storage modulus-frequency curves of representative SG interlayer in high temperatures obtained by DMTA.

To compare the experimental data of the representative samples shear storage modules, their certain values are shown in Table 2 as a function of temperature and loading period of each cycle. Loading period T is reciprocal to the loading frequency f and is calculated according to the following equation

$$T = 1/f. \tag{9}$$

Table 2 indicates that the values of shear storage modulus are distinctly temperature and loading time cycle period dependent. When the temperature is increased, the storage modulus decreases and when the loading cycle takes more time, the storage modulus also decreases in case of both tested interlayers. Keeping the direct proportionality between the storage modulus *G´* and the shear modulus *G* in mind, this response is in correlation with viscoelastic properties of polymers. In addition, these dependences approve the indirect proportionality between the initial shear modulus and ambient temperature and the direct proportionality between the initial shear modulus and loading ratio input experimentally verified by static loading tests. When comparing the values of Trosifol and SG storage modules, it becomes obvious that SG loaded at 70 °C is almost as stiff as Trosifol loaded at 25 °C. For other temperatures, SG interlayer achieves several times higher shear storage modulus values.

Table 2: Representative Trosifol and SentryGlas Shear Storage Modules experimental values.

	Trosifol BG-R-20			SentryGlas		
Time T^*	1 s	5 s	20 s	1 s	5 s	20 s
Temperature	G´ [MPa]	G´ [MPa]	G´ [MPa]	G´ [MPa]	G´ [MPa]	G´ [MPa]
25 °C	7.8	3.9	2.4	-	-	-
30 °C	3.4	2.0	1.4	896.8	780.9	626.5
35 °C	2.0	1.3	1.1	640.2	524.7	463.9
40 °C	1.5	1.1	0.8	410.8	266.4	196.4
45 °C	-	-	-	110.0	61.2	43.0
50 °C	-	-	-	48.9	26.4	16.1
55 °C	-	-	-	21.0	12.9	8.6
60 °C	-	-	-	13.2	8.5	6.1
65 °C	-	-	-	7.6	5.1	4.0
70 °C	-	-	-	4.2	3.0	2.5

T^* refers to loading cycle period time [s] not being generally equal to the static loading time in a real structure

3.3. Failure modes

When the samples with Trosifol were loaded statically at 0 °C, they collapsed because of glass failure in tension due to the moment evoked by the load eccentricity. On the contrary, when the samples were loaded at 60 °C, the interlayer delamination caused their failure as shown in Fig. 13. In DMTA, as it has been said, Trosifol´s delamination preliminarily interrupted the loading cycle at 40 °C for all testing specimens. This delamination is shown in Fig. 14.

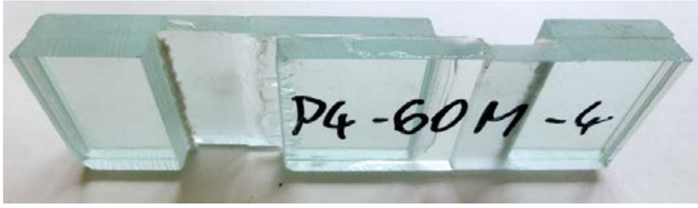


Fig. 13 Trosifol delamination, sample statically loaded at 60 °C.

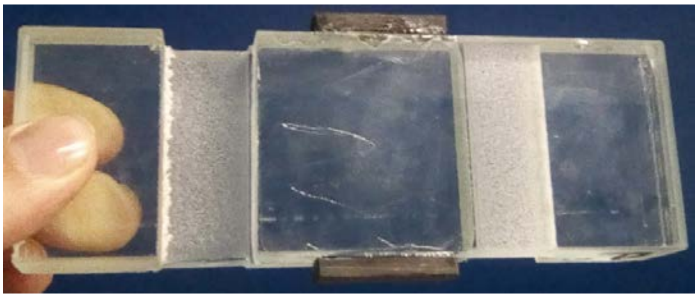


Fig. 14 Trosifol delamination, sample dynamically loaded at 40 °C.

In case of SG, no debonding at 50 °C was found and it enabled to perform DMTA up to relatively high temperatures. The delamination effect occurred at 75 °C, therefore this temperature could not be tested. But when checking the stiffness-frequency curves in fig. 12, some stiffness reserve might be still available. SG delamination dynamically loaded at 75 °C is shown in fig. 15.

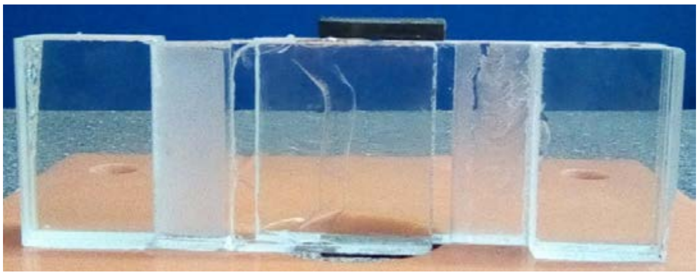


Fig. 15 SG delamination, sample dynamically loaded at 75 °C.

4. Conclusion

In this paper, details regarding the initial shear modules of Trosifol and SentryGlas® depending on a temperature and loading rate obtained from static shear loading tests were elaborated as well as the experimental data regarding shear storage modules as a functions of temperature and testing frequency obtained from DMTA. The effect of humidity was not considered. Stress-strain curves gained from the static shear load tests proved a strong dependence of these interlayers on temperature and loading rate and showed significant initial shear modules differences of both tested interlayers. This is the sign of visco-elastic behaviour of polymers which has been introduced into variational laminated glass panels calculations (Gallupi 2012). Static shear loading tests were performed as a short duration. To illustrate time dependent behaviour of both investigated polymers under permanent static loading, long time creep or relaxation tests should be executed. Dynamic mechanical thermal analysis (DMTA) revealed significant dependence of shear storage modulus on the ambient temperature and frequency, showed negligible phase angle between strain input and stress response in each loading cycle, compared experimental shear storage modules and confirmed the experimental data and tendencies gained from the static shear loading tests for both tested interalyers. In practice, it is necessary to check out the type of the interlayer used in laminated glass panels perpendicularly loaded. Different chemical compositions of the interlayers result in a different shear stiffness which plays a significant role in normal stress distribution in the whole laminate. This fact should be kept in mind when designing laminated glass panels as load bearing members. Stiffer interlayers should be used mainly in case of load bearing structural elements such as floors, roof panels, staircases and balustrades. Softer interlayers can be used in short term loaded laminated glass elements such as facades and windows.

Acknowledgement

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