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A Comparison of Different Methodologies for PVB Interlayer Modulus Characterization

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The proper measurement and interpretation of modulus data for glass laminate interlayers can be quite complex. The development of master curves using different deformation modes and the preparation of the samples for measurement can significantly affect the results. International standard ISO 6721, determination of dynamic mechanical properties, uses modulus as a primary criterion for method selection. The shear modulus of polyvinylbutyral (PVB) materials varies to a great extent, e.g. 1 – 400 MPa, over the temperatures and durations encountered for glass laminates in a building. We have evaluated the use of tensile and plate-plate geometries for a regular and a high rigidity ("structural") PVB interlayer material, as well as torsion geometry for a structural PVB interlayer material. In some cases, datasets from different sources have been compared. This paper will discuss the results we obtained using different methodologies, and explore the effect with regards to positioning of the interlayers in the "stiffness families" and the associated shear transfer coefficients as in draft European norms prEN 16612 and prEN 16613.

Keywords: Polyvinylbutyral, Structural PVB, Interlayer, Storage modulus, Laminated glass, prEN 16613

1. Introduction

Standard PVB interlayers have been used as an interlayer technology providing safety characteristics in laminated glass applications for decades. They can also provide some transfer between glass panes under specific conditions such as short load durations and/or modest temperatures. This is reflected in proposed or accepted glass standards such as prEN 16612 Determination of the load resistance of glass panes by calculation and testing and ASTM 1300 Standard Practice for determining the load resistance of glass in buildings. For cases where a higher level of structural performance is required, or load durations are longer, or occur at higher temperatures, PVB interlayers with high rigidity ("structural PVB") have recently become available on the market. The rigidity of these products contributes to reduced stresses and deflections in glass laminates for use in applications such as balustrades (see e.g. Stevels 2015), facades, and canopies. The trend towards ever larger glass surfaces is another driver for this development, as deflections need be limited to levels that allow full functionality of the glazing, meet standard requirements if absolute limits are in place, and are compatibility with constraints in the framing design. Structural PVB's are available in roll widths to up to 3.2 m and allow the use of conventional PVB lamination processing equipment and settings, facilitating standard production processes.

In order for an interlayer to be used in a structural glazing, the modulus of the material as a function of time and temperature must be known in detail. Draft European standard prEN 16613 Determination of interlayer mechanical properties (2013) is proposing a specific method for determination of interlayer modulus properties based on tensile vibration measurements. Whereas this specific method is certainly useful, no comparison to other potentially useful methods was made at the time. Generic standards for plastic materials exist under the ISO 6721 Plastics-Determination of Dynamic Mechanical Properties series. In ISO 6721-1-Part 1: General principles, it is stipulated that different deformation modes may produce results that are not directly comparable.

Measurements of this type provide detailed modulus data, which are used in engineering calculations to predict laminate strength under various load conditions. For conventional PVB interlayers, a number of datasets are available (Bennison et al. 1999; D'Haene 2001; D'Haene and Savineau 2007; Sackmann 2007; Hooper et al. 2012). Published tables may show the shear stress relaxation modulus as a function of time and temperature. However, an interpretation in terms of prEN 16613 is lacking, because publications are either preceding the publication of this draft norm, or were not of academic interest.

For structural PVB interlayers, detailed information on the methodology and measurement is available to an even smaller extent. A recent example of dataset that was both transparent and interpreted in terms of prEN 16613 was recently published (Zhang et al. 2015).

Several authors have expressed some concern over specific measurement methodology details in prEN 16613 (Andreozzi et al. 2014; Kuntsche et al. 2015; Zhang et al. 2015). These include the tensile deformation, especially above the glass transition temperature of the materials used, clamping forces affecting the uniaxial stress state, the high frequency range prescribed, and elements of the sample preparation used. These are potential topics of interest for further study in this area and over time some understanding of "best practices" may emerge.

From a practical perspective, in terms of the interlayer stiffness family classification of prEN 16612, it is more relevant to review if various methods of interlayer characterization would result in a change of stiffness family as determined by specific Young's modulus values under this norm.

Therefore, properties of standard and structural PVB interlayer were determined using dynamic mechanical analysis using various deformation modes, and the results have been interpreted in terms of the load scenarios proposed in prEN 16612. In some cases, test reports were obtained from independent laboratories to benchmark results, or have more equipment types available.

2. Experimental

2.1. Materials

Eastman Saflex® RB41 interlayer was selected as the representive regular interlayer, and Saflex® DG41 structural interlayer was selected as the representative structural PVB interlayer. Interlayers were obtained from commercial production with a nominal thickness of 0.76 mm. Test specimens were conditioned inside a desiccator at room temperature for a minimum of 48 hours before being tested, thus bringing the moisture content of the specimen to zero.

PVB interlayers are completely amorphous, thermoplastic materials, which undergo no chemical cross-linking or crystalline melting during the normal lamination process (Schneider et al. 2016). More importantly, this process will not change any physical properties of the material. Therefore, measurements on the non-processed interlayer is possible, and was used in this testing.

2.2. Measurements plate/plate geometry

A TA Discovery HR-2 hybrid rheometer was used for all shear mode characterization measurements, using a 8 mm plate/plate geometry for a single layer of, dried, non-processed PVB interlayer (interlayer not put through a lamination and autoclave process) punched by an 8 mm circular die. In order to ensure good bonding between the PVB sample and the metal plates, each test specimen was loaded at 65ºC and heated to 100ºC first under program controlled pressure and then cooled to the test temperature for each frequency scan. Strain was varied from 0.01% at 15 ºC to 0.4% at 70ºC based on the rigidity of the material, being certain to maintain all measurements within the linear viscoelastic regime. The controlled normal force was also varied by using a high setting below the specimen's glass transition temperature to avoid slippage, and a low setting at high temperatures to avoid the specimen flowing out from between the plates. Measurements were performed in 5ºC increments from 15ºC to 70ºC, using a frequency range of 0.01 -100 Hz with 8 datapoints per decade (33 datapoints per scan per temperature). Time-temperature superposition principles were applied using the Williams-Landel-Ferry to determine shift factors a_τ. Master curves at different reference temperatures and the corresponding shift factors were created and calculated by the TRIOS Software provided by TA Instruments.

Alternatively, independent measurements were executed on an Anton Paar Rheometer MCR 702 using Saflex® DG41 structural interlayer, again using 8 mm plates at a constaint strain of 0.05 % from 0.02 to 20 Hz from 70 ºC to – 20 ºC, with the normal force between 4 and 10 N to clamp the sample.

2.3. Measurements tensile geometry

Experiments were performed (independent laboratory) on a TA Instruments Q800 Dynamic Mechanical Analyzer (DMA) using a film tension setup (samples were cut to 15 mm x 5.6 mm). The instrument was equipped with a liquid nitrogen cooling accessory and is purged with N_2. Frequency sweeps (from 0.1 to 200 Hz, 5 points per decade) are recorded at different temperatures (stabilization at 20 °C, followed by stepwise heating from -20 °C to 80 °C, steps of 10 K), maintaining the dynamic strain at 0.1 % and the static stress at 115% of the dynamic stress.

2.4. Measurements torsion geometry

Measurements on Saflex® DG41 structural interlayer were executed indepedently on an Anton Paar Rheometer MCR 702, using a strip of 21.5mm (length) * 10 mm (width) *0.76 mm at a constaint strain of 0.05 % from 0.01 to 10 Hz from -60 ºC to 55 ºC, with the normal force of -0.1 N to strain the sample. No reliable measurements could be obtained at higher frequencies or temperatures.

3. Results and Discussions

3.1. Method selection and sample preparation

The ISO 6721 series and prEN 16613 were taken as leading documents for method selection. An overview of potentially applicable methods is given in Table 1. In this table E, represents the (complex) Young's modulus and G the (complex) shear modulus.

Table 1: Dynamic mechanical methods in ISO 6721 series

Reference	Methodology	Modulus type, Modulus range
ISO6721-2	Torsion-pendulum	G, None given
ISO6721-3	Flexural vibration resonance (acoustic materials)	NA
ISO6721-4	Tensile vibration	E, 10 MPa to 5 GPa
ISO6721-5	Flexural vibration	E, 10 MPa to 200 GPa
ISO6721-6	Shear vibration	G, 0.1 MPa to 50 MPa
ISO6721-7	Torsional vibration	G, 10 MPa to 10 GPa
ISO6721-8	Longitudinal and shear vibration (wave pulse)	G, 10 MPa to 200 GPa
ISO6721-9	Tensile vibration (sonic pulse)	E, 10 MPa to 200 GPa
ISO6721-10	Oscillatory plate/plate	G, up to 10 MPa
ISO6721-12	Compressive vibration	E, 10 MPa to 200 GPa

Considering the modulus range of interest (1-400 MPa), and making use of the methods that seem to most wide-spread for the characterization of plastic materials, we used tensile vibration (ISO 6721-4), torsional vibration (ISO 6721-7), and oscillatory plate-plate deformation (ISO 6721-10) for this study. A schematic representation of the deformations modes is given in Figure 1, and in more detail for a plate/plate geometry in Figure 2. The relative ease of sample preparation is an advantage for these methods. Kuntsche et al. (2015) have reported on the use of double shear vibration (ISO6721-6), but reported relatively complex sample preparation and installation.

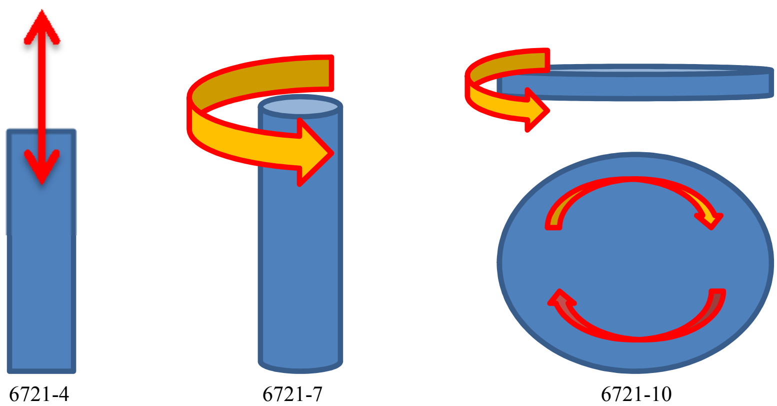

Fig. 1 Graphic representation of deformation modes used in this study, with reference to the relevant ISO method

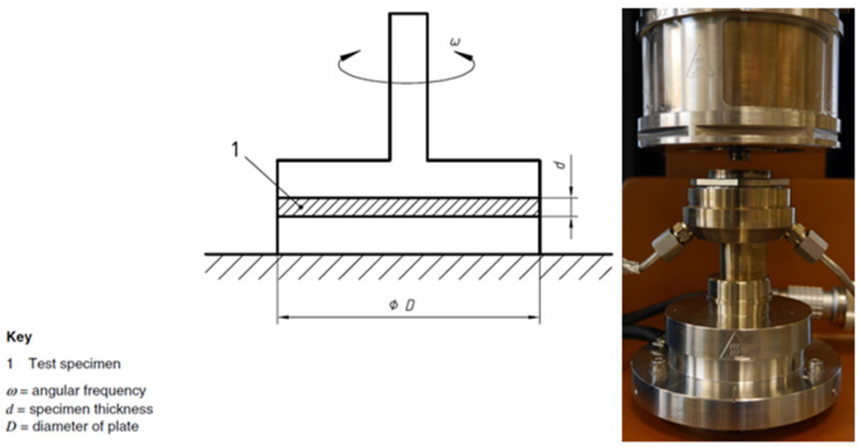

Fig. 2 Measurement in plate/plate geometry: schematically (left) and actual equipment (picture right)

In general, care should be taken to verify that the measurements are generated in the linear viscoelastic domain. This is not always easy over the range of measurement conditions required to establish the properties of the interlayers over the relevant range. Arguably speaking, creep compliance data might be a more accurate parameter to characterize and predict interlayers' long term performance under load. As both aspects are not addressed by prEN 16613, we have not made a detailed analysis in this work for these elements

Optimal resolution in the 1-10 MPa range is desirable, as this is the critical regime for shear transfer in laminated glass structure. However, for this study this was not taken as a stringent criterion as the intent was to review the effect on stiffness family classification

Controlling moisture levels in PVB specimens is important, as gradual water uptake by the hygroscopic PVB material is likely to result in an effect on the glass transition temperature (T_g). In commercial lamination practice, PVB moisture levels are always controlled within a narrow and low range. Therefore, samples were dried directly before measurement. Controlling moisture levels in samples for measurements in tensile mode or torsion mode is more difficult because of the relatively high surface exposure of these samples, required sample manipulation, and measuring time. The extent of the moisture level change will further depend on the surrounding environmental conditions during the test, unless the testing chamber is controlled to a constant humidity. This is expected to be especially true if the same sample is used through the entire characterization. It should be noted that neither using a new specimen for each individual scan nor employing a humidity control is a current requirement of prEN 16613.

Testing frequencies in line with the ISO recommendations (0.01 – 100 Hz), were used in this study regardless of the deformation mode used. prEN 16613 specifies testing frequencies from 1 to 400 Hz. Obtaining reliable results from measurements at the high frequency end can be challenging due to inertial effects in viscoelastic materials.

3.2. Characterization of materials used

A temperature sweep measurement in shear mode was executed, to get a basic understanding of the standard and structural PVB rheological characteristics (Figure 3).

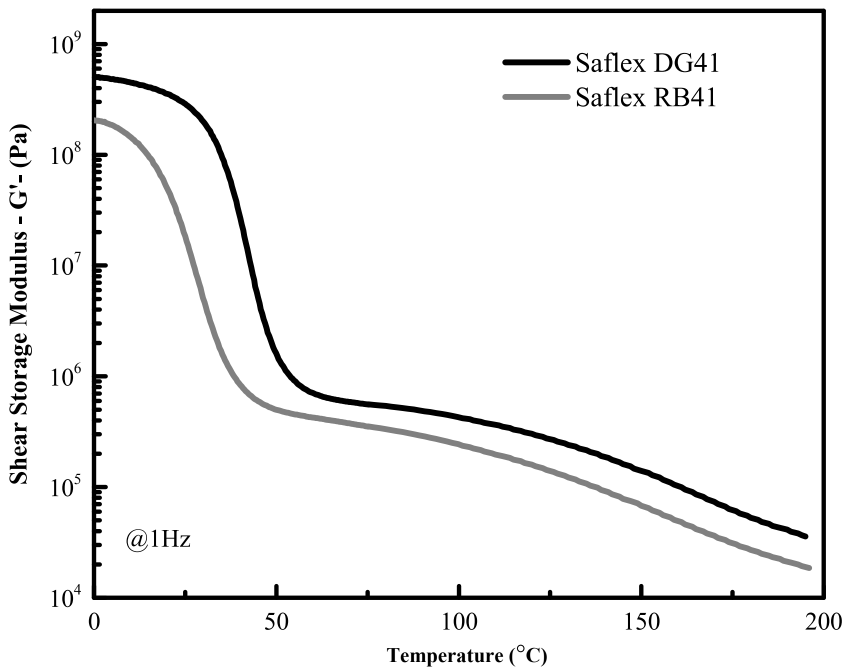

Fig. 3 Shear storage modulus of Standard and Structural PVB (Saflex® RB41 and Saflex® DG41 interlayers)

A distinctive reduction in modulus can be observed as the PVB interlayers are heated through their glass transition regions, between respectively 10 and 30 ºC for the regular PVB, and between 30 and 50ºC for the structural PVB. Although the general shape of the curve for structural PVB is consistent with that of regular PVB interlayers, it is shifted towards high temperatures. The associated glass transition temperature (T_g) of the structural PVB interlayer is approximately 43ºC, and around 25 ºC for the standard PVB, in line with the guidance given by Decourcelle et al. (2008) relative to T_g and stiffness family classification.

Multiple frequency sweep scans were performed as outlined in the experimental section and reference (Zhang et al. 2015) to generate a master curve, comprising around 500 data points over a wide range of frequencies. The result is shown in Figure 4 for a reference temperature of 20 ^{o}C.

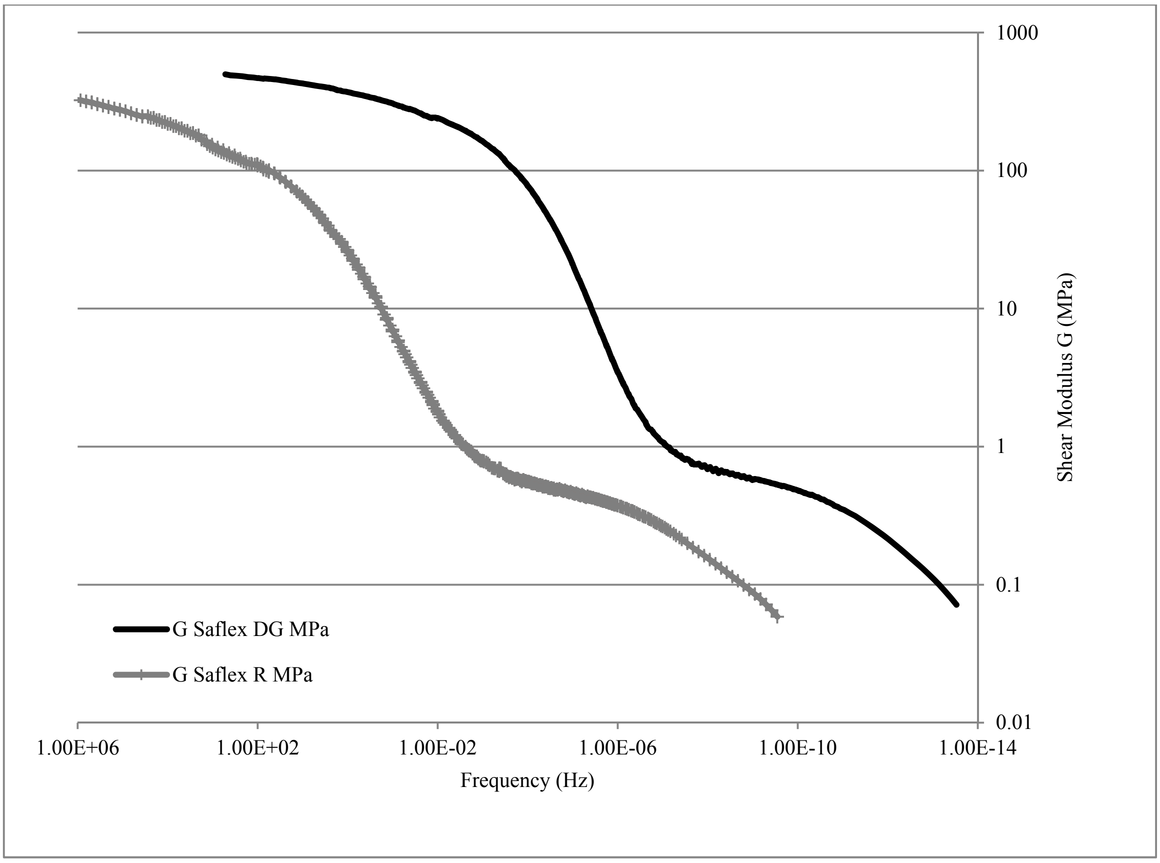

Fig. 4 Mastercurves at 20 ºC of Standard and Structural PVB (Saflex® RB41 and Saflex® DG41 interlayer)

As expected, the curves are of similar shape again, and the structural PVB curve is shifted towards lower frequencies, or longer durations. These master curves were then used for the determination of G_L (T,f) at different load scenarios as described in prEN 16613, as shown in Table 2 and 3 for a limited set of durations.

Table 2: Standard PVB (Saflex® RB41 interlayer) shear storage modulus in MPa

Time	Duration					
Temperature	3 seconds	30 seconds	5 minutes	30 minutes	5 days	3 weeks
20°C	14	3.5	1.1	0.7	0.4	0.3
25°C	3.4	1.1	0.6	0.5	0.3	0.2
30°C	1.2	0.7	0.5	0.4	0.2	0.1
35°C	0.8	0.5	0.4	0.4	0.1	0.1
40°C	0.6	0.5	0.4	0.3	0.1	0.1
45°C	0.5	0.4	0.3	0.2	--	--
50°C	0.5	0.4	0.3	0.2	--	--
55°C	0.4	0.3	0.2	0.1	--	--
60°C	0.4	0.3	0.2	0.1	--	--

Table 3: Structural PVB (Saflex® DG41 interlayer) shear storage modulus in MPa

Time	Duration					
Temperature	3 seconds	30 seconds	5 minutes	30 minutes	5 days	3 weeks
20°C	341	275	202	140	7.0	2.0
25°C	237	158	72	28	0.9	0.7
30°C	108	39	7.0	2.0	0.6	0.5
35°C	27	4.5	1.2	0.8	0.5	0.4
40°C	4.0	1.0	0.7	0.6	0.3	0.3
45°C	1.3	0.8	0.6	0.5	0.2	0.2
50°C	0.8	0.6	0.5	0.4	0.1	0.1
55°C	0.7	0.6	0.5	0.4	0.1	--
60°C	0.6	0.5	0.4	0.3	--	--

In order to convert the measured shear storage modulus, G_L to Young's modulus E_L, which is used in prEN 16613 as the basis for stiffness family determination, Equation 1 may be used:

$$E_L = 2G_L(1+\mu) \tag{1}$$

where μ is the Poisson's ratio value of the interlayer. Section 5.1 prEN 16613 specifies μ equals 0.49 for an isotropic interlayer, which leads to the approximation:

$$E_L \approx 3G_L \tag{2}$$

Equation 2 may be used to categorize the structural PVB interlayer under various load scenarios into different stiffness families according to the conditions specified in prEN 16613. Alternatively, if the Poisson's ratio is known for the material being tested, Equation 1 may be directly used. E_L values for the structural PVB interlayer were calculated from the shear modulus results in Table 2 and 3 using Equation 2. The relevant load scenarios and the associated Youngs' modulus criteria for stiffness family 2 and 3 are tabulated in Table 4. Finally, the stiffness family is determined for the materials used in this study using the methodology outlined. The results are tabulated to prEN 16613 load cases in Table 5. Permanent loads were not included, as these do not have an associated shear transfer coefficient for any of the stiffness families.

Table 4: Load scenario's and stiffness family criteria in prEN 16613

prEN 16613 Load scenarios and stiffness family criteria				
Load case	Duration	Max T (ºC)	Required for family 2 E (MPa)	Required for family 3 E (MPa)
Wind load (Mediterranean areas)	3 s	30	1-50	>50
Wind load (other areas)	3 s	20	10-100	>100
Personnel balustrade loads – normal duty	30 s	30	1-20	>20
Personnel balustrade loads - crowds	5 min	30	<5	>5
Glass for walking on for maintenance	30 min	40	<1	>1
Snow load -roofs of unheated buildings	3 weeks	0	1-10	>10
Snow load - roofs of heated buildings	5 days	20	<1	>1

Table 5: Stiffness family determination as per pr EN16613 for Saflex® RB41 interlayer and Saflex® DG41 interlayer

Load case	Saflex® RB41 (E Modulus, MPa)	Stiffness family	Default ω	Saflex® DG41 (E Modulus, MPa)	Stiffness family	Default ω
Wind load (Mediterranean areas)	3.6	2	0.1	324	3	0.6
Wind load (other areas)	42	2	0.3	1023	3	0.7
Personnel balustrade loads – normal duty	2.1	2	0.1	117	3	0.5
Personnel balustrade loads - crowds	1.5	0-2	0	21	3	0.3
Glass for walking on for maintenance	0.9	0-2	0	1.8	3	0.1
Snow load -roofs of unheated buildings	3.4*	2	0.1	69*	3	0.3
Snow load - roofs of heated buildings	1.2	3	0.1	21	3	0.1

*Measured data at 0ºC is not available in this data set, an assessment was made based on internal data

As expected from the glass transition temperature, Saflex® RB41 interlayer classifies as a stiffness family 2 material with modulus values well within the range given for this stiffness family, except for the last scenario. In this case, it is border line over the criterion for stiffness family 3 in this evaluation. Given the complexities and inevitable inaccuracies in interlayer modulus determination, we would not recommend it to be classified as such for this load scenario, and have investigated this further in the next section.

It can be seen that Saflex® DG41 structural PVB interlayer classifies as a stiffness family 3 material for all load scenarios with significant margin in all cases.

3.3. Standard PVB: comparison of methodology

The results in the previous paragraph were generated using a plate-plate geometry. A similar characterization of this material in tensile mode using an as-produced film, directly clamped, was executed at an independent laboratory. A comparison of directly measured frequency sweep results is shown in Figure 5, with the Young's modulus results transformed to shear storage modulus values using Equation 2. Only three frequency sweeps are represented for clarity, one well below the glass transition temperature (0 °C) , one around the glass transition temperature (20 °C), and one well above the glass transition temperature (40 °C) of this material.

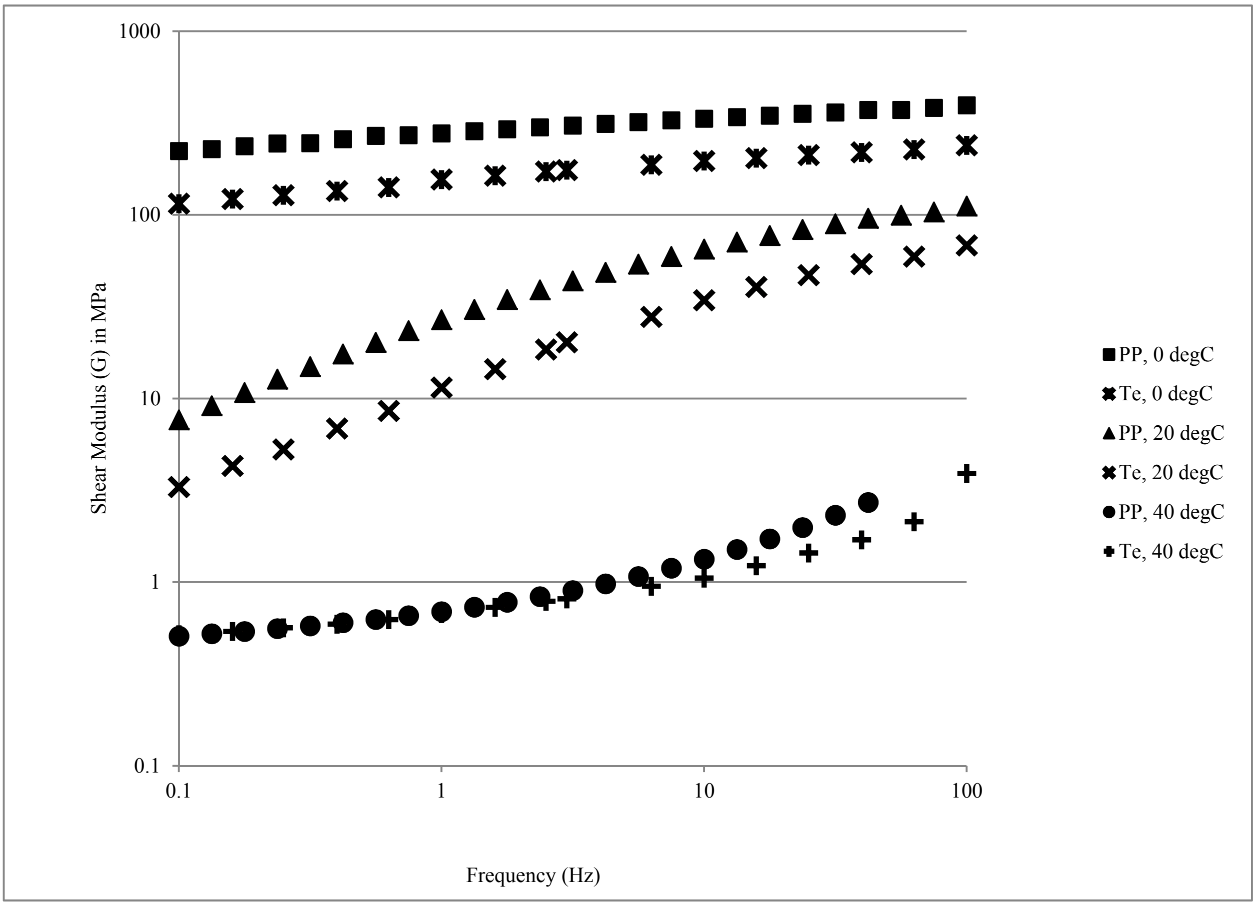

Fig. 5 Characterization of Saflex® RB41 interlayer using two different deformation modes
(PP refers to plate/plate geometry, Te to tensile geometry)

During the measurements in tensile mode, deformation of the samples was noted as of the onset of Tg, for the measurements taken above 10 °C. Initially, a shrinkage of the sample was observed followed by an elongation of the samples as the sample gradually softens during the measurement. The initial shrinkage might be attributed to "frozen in" tensions during the sample clamping procedure, the final elongation to the inability of the material to withstand the static stress required to avoid out-of plane deformation during application of the oscillatory forces. Both effects are undesirable for the accuracy of the measurement. Further studies reviewing the potential for optimization of the test method have been planned

The modulus values determined in tensile mode determined in this way were lower than those obtained during the plate-plate measurements. The differences were largest in the transition regime, and fairly well aligned at the low viscosity regime towards higher temperatures. Although there was a significant difference for the absolute values at low temperature, this is not very relevant in terms of glass laminate properties, as the material is stiff enough for significant shear transfer, regardless of the determination method.

These data were then treated to generate mastercurves, and values extracted pertaining to the load scenarios of prEN116613 that have a non-zero shear transfer coefficient in stiffness family 2, in order to assess the effect of the measurement methodology on the stiffness family classification. Snow loads of unheated buildings were not included, because mastercurves at 0 °C were not constructed for both datasets

The results are collected in Table 5.

Table 5: Stiffness family determination as per prEN 16613 for Saflex® RB41 interlayer using different deformation mode

Load case	Plate/plate geometry E (MPa)	Stiffness family	Tensile mode E (MPa)	Stiffness family
Wind load (Mediterranean areas)	3.6	2	3.2	2
Wind load (other areas)	42	2	20	2
Personnel balustrade loads – normal duty	2.1	2	2.3	2
Snow load - roofs of heated buildings	1.2	3	0.9	2

Despite the differences in values that were measured, the material is still classified as being a stiffness family 2 material for the relevant load cases. As the associated shear transfer coefficients are also modest, except for wind gust loads in other areas (ω = 0.3), little effect on glass dimensioning practice is expected. In tensile mode, modulus value was at or slightly below the threshold value for a stiffness family 3 material, a further indication that this material should be treated as a family 2 material for snow loads.

3.4. Structural PVB: comparison of methodology

For this material four different characterizations were carried out, using three different deformation modes. Property evaluation in plate-plate geometry was executed as described by Zhang et al. (2015), and further characterizations were executed in independent laboratories using again plate-plate, tensile and torsion geometries. A comparison of directly measured frequency sweep results is shown in Figure 6, with the Young's modulus results transformed to shear storage modulus values using Equation 2. Only three frequency sweeps are represented for clarity, one well below the glass transition temperature (20°C), one around the glass transition temperature (40 °C), and one well above the glass transition temperature (60 °C) of this material. Note that the temperatures are shifted by 20 °C as compared to Figure 5.

A number of observations can be made. The relative positioning of the plate-plate vs. the tensile measurements was as for the standard PVB in the previous section, but the torsion measurements providing higher modulus values than obtained using the plate-plate geometry. The differences between plate-plate and tensile characterization in the transition regimes seem relatively larger for this stiffer interlayer. The same issues with sample deformation in tensile mode were observed as in the previous section. The differences between the different methods were the largest in the transition regime. In torsion deformation, only a limited range of frequencies was available on the equipment used, and data could no longer be obtained at 60 °C, as the sample became too soft. These higher temperature measurements are still relevant, especially for longer duration load scenarios.

The large difference between torsion and tensile measurements in these experiments is somewhat surprising, given the finding of Kuntsche et al. (2015) that in their experimentation torsion and shear test delivered comparable results from the beginning of the glass transition region until high temperatures, where the tension test gets unreliable when the material starts to creep under the static offset force.

The differences between the two plate-plate determinations, one by the authors, one by an independent laboratory, were rather similar despite instrumentation differences.

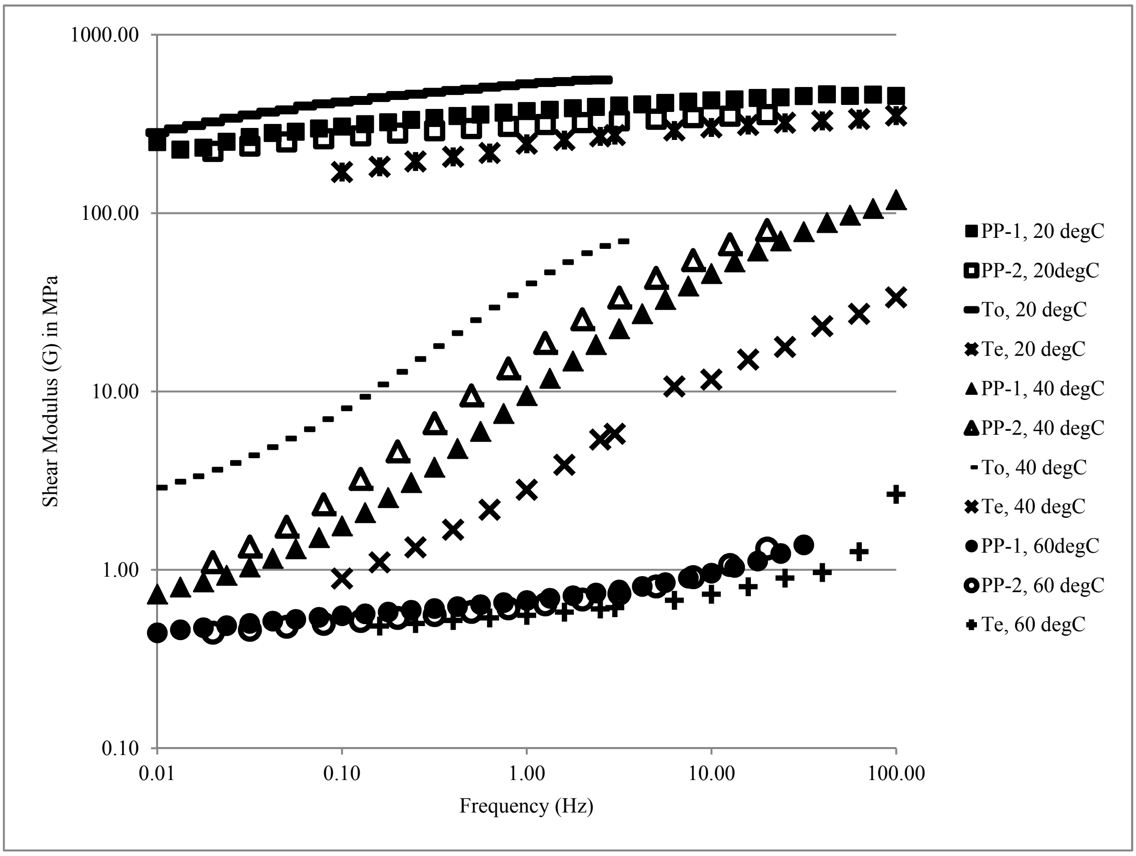

Fig. 6 Characterization of Saflex® DG41 interlayer using three different deformation modes
(PP refers to plate/plate geometry, Te to tensile geometry, To to torsion geometry)

These data were then treated to generate mastercurves, and values extracted pertaining to the load scenarios of prEN 16613 that have a non-zero shear transfer coefficient in stiffness family 3, in order to assess the effect of the measurement methodology on the stiffness family classification. Snow loads of unheated buildings were not included, because mastercurves at 0 °C could not be constructed for all datasets. The results are collected in Table 6.

Table 6: Comparison of Saflex® DG41 interlayer using different deformation modes

	Plate plate geometry (Det. I)	Plate plate geometry (Det. II)	Tensile mode	Torsion mode
Load case	E (MPa)	E (MPa)	E (MPa)	E (MPa)
Wind load (Mediterranean areas)	318	381	85	528
Wind load (other areas)	1023	819	679	1428
Personnel balustrade loads – normal duty	114	180	19	198
Personnel balustrade loads – crowds	20	63	4.3	96
Snow load - roofs of heated buildings	21	12	3.6	15

All these modulus results would put Saflex® DG41 interlayer in stiffness family 3, with the exception of the personnel loads as measured in this particular tensile determination with a very slight margin (< 1 MPa). Given the issues observed in the measurement as executed, and the seemingly different results by other authors, it would be recommended to repeat this work with more attention to sample dimension control by pre-stress and/or pre-strain setting, clamping practice and/or interlayer thickness. Optimization of this methodology is in progress. Even so, some of the concerns with regards to tensile mode deformation will remain.

4. Conclusions

In this paper, the rheological properties of PVB interlayer materials were determined using different deformation modes. The effect with regards to positioning of the interlayers in the "stiffness families" and the associated shear transfer coefficients as in draft European norms pEN 16612 and prEN 16613 was explored.

It was found that for the methods studied, there was significant variation in the individual values determined for the Young's modulus values E for different load scenarios. This variation tends to be largest around the glass transition temperature of the material. However, the positioning of the interlayer in the stiffness family as proposed in pEN 16612 was generally not affected. In some cases, a deviation was found in conjunction with technical issues in those measurements. More detailed studies comparing tensile and plate-plate geometries are ongoing. The classification of two well-known interlayer materials was in line with the expectations of prEN 16613, with a regular PVB classified in stiffness family 2, and a structural PVB classified in stiffness family 3.

It was found that a plate-plate geometry generated results within a relatively narrow range between different laboratories. It is recommended that further studies are executed to establish best practices in interlayer rheological property determination to confirm these findings.

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