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Influence of Temperature on Laminated Glass Performances Assembled with Various Interlayers

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Laminated glass is widely used for its safety properties (accident prevention, mechanical resistance), security properties (hard impact, explosion, burglar and bullet resistance), acoustic performances, for its decorative purposes or encapsulation of other materials. The increasing demand for laminated glass use in building facades is the driving force of new interlayers testing and implementation. In some instances it can be difficult to choose an interlayer that would perfectly fit to all architects' and engineers' requirements. The behaviour of the different interlayers is well known at ambient temperature, but can vary significantly at different temperatures or exposure. The aim of the research is to investigate temperature influence on laminated glass performances. The behaviour of several interlayers of different chemical composition (PVB based, EVA based, ionomer based) were evaluated at several temperatures ranging between -20°C and 80 °C. Resistance to hard impact was determined by use of ball drop test according to R43 standard. Regarding acoustic performances of laminated samples, mechanical impedance method (MIM) was used to characterize the interlayers sound transmission loss (STL) and weighted sound reduction index (Rw). Strong influence of temperature on final interlayers performances was observed and commented. Based on these observations, it is obvious that one unique type of interlayer cannot be used to meet all needs at any temperature. Several interlayers with complimentary performances at a wide range of temperatures are therefore required to meet the requirements.

Keywords: Glass, Laminated, PVB, EVA, Ionomer, Temperature, Resistance, Acoustics

1. Introduction

The increasing demand for use of laminated glass in building facades is the driving force for new interlayers testing and their implementation. Architects' and engineers' requests require searching for new solutions and processes of lamination. Intelligent facades, electrochromic windows, integrated photovoltaics, integrated lighting, enhanced solar control, acoustic pollution reduction, wires incorporation, use of special sealants and glues are some of the design drivers of new laminated structures.

Laminated safety glass is composed of two or more glass panes bonded by one or more interlayers. The interlayers are typically soft polymers like polyvinyl butyral (PVB), ethylene vinyl acetate (EVA), polyurethane (PU) or ionomer. Today, there are many interlayers on the market with a large variation in their properties. For some projects, it is a challenging task to choose the optimal interlayer.

In some cases, mainly due to exposure of façade to higher temperature, use of EVA instead of PVB is necessary since EVA shows lower sensitivity to delamination, yellowing or bubbles appearance at higher temperature. As it will be shown later, high temperature resistance doesn't necessarily mean high performance at higher temperature.

Thick patterned glass panes of special yellow colour laminated with EVA were used for instance on the Canopée project in Paris (parisleshalles.fr, Fig. 1, Fig. 2). Les Halles is a former food fresh market renovated to a modern shopping mall and directly connected to metro.

Fig. 1 Canopée Paris – Global view

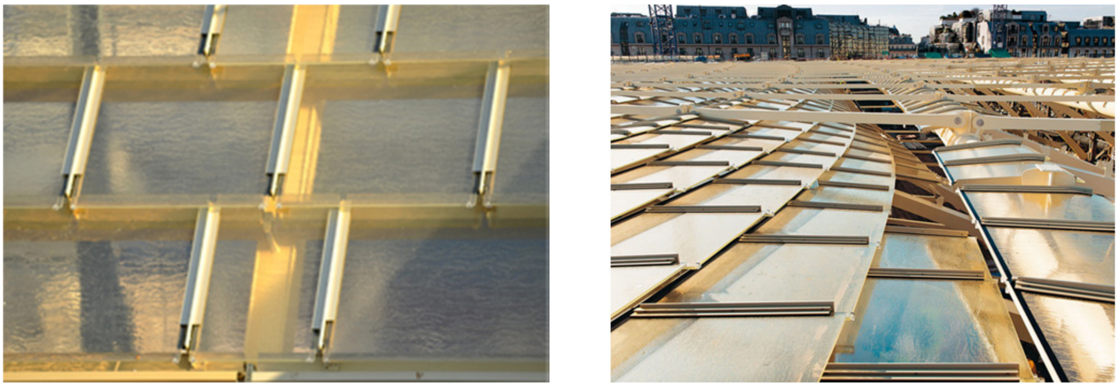

Fig. 2 Canopée Paris – Glazing detail

An example of encapsulation of photovoltaic cells in laminated glass is the Hikari building in Lyon, France (Fig. 3). Hikari is the first positive-energy mixed-use city block in Europe thanks to its photovoltaic glazing that generates slightly more energy than it consumes (yourglass.com 2016).

Fig. 3 Hikari Lyon – Global views

Laminated glass is widely used for its safety properties (accident prevention, mechanical resistance), security properties (hard impact, explosion, burglar and bullet resistance), acoustic performances, for its decorative purposes or for encapsulation of other materials. Glass assemblies with properly selected glass and interlayer combinations are capable of meeting various safety glazing standards such as EN 12600, EN 356, ISO 140-3, ISO 16940, ANSI Z97.1, JIS 3205 and R43. Most of the testing methods are defined at about 20°C, with exceptions like impact with a small ball (227g) included in the mentioned R43 regulation used for automotive glazing. The penetration resistance in this test is performed at -20, +20 and + 40 °C.

Nevertheless, the question rises what are the performances of laminated glass exposed to higher or lower temperature. Changes in interlayer performances could lead for instance to unexpected failure of the glazing elements.

2. Interlayers

2.1. PVB

Thanks to its optical and mechanical performances, ease of lamination and capacity to be adapted to meet a variety of requirements such as acoustic, solar, rigidity, this thermoplastic polymer (Fig. 4) is the most used and cheapest interlayer for laminated glass fabrication in the architectural and automotive business. PVB sheets are produced by an extrusion process where PVB resin is blended with a plasticizer and other additives such as antioxidants, UV blockers, adhesion agents or colorants to incorporate additional performances. There are many kinds of special PVB available on the market - acoustic, solar, coloured, structural and others (Schimmelpenningh 2012; Keller 1999; Speelman 2013).

The combination of residual polyvinyl alcohol (PVOH) groups and plasticizer forms the basis for the mechanical and rheological properties of the PVB. In particular, the glass transition temperature (Tg) depends on the percentage and type of plasticizer and the residual PVOH present in the PVB (Schimmelpenningh 2012; Keller 1999; Speelman 2013).

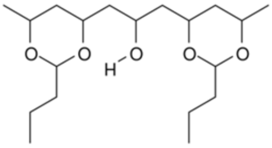

Fig. 4 Chemical composition of PVB

Acoustic PVB (tri-layer) combines a very thin and soft “core” layer with two layers of normal “skin” PVB (Fig. 5). The soft layer contains more plasticisers and due to its softness dampens better the transmitted sound. Skin layers are of the same composition as a standard PVB that guarantees the same processability (Yoshioka 2003, D’Haene 2003; Schimmelpenningh 2012).

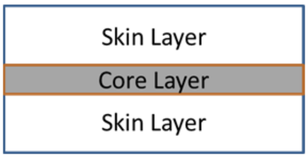

Fig. 5 Structure of an acoustic PVB

Structural PVB has the highest Tg of all PVBs for architectural use, resulting in a high stiffness at room temperature. Its stiffness is given by the lower plasticiser content compared to the standard PVB. The lower plasticizer content also changes the performances of the interlayer, making it more resistant to delamination (Schimmelpenningh 2012; Keller 1999; Speelman 2013, Zhang 2015).

2.2. EVA

The development of EVA copolymers (Ethylene Vinyl Acetate, Fig. 6) started in the middle of 20th century. EVA resin is based on copolymerization of ethylene (C_2H_4) with acetic acid (CH_3COOH) to form vinyl acetate and its subsequent reaction with ethylene to form Ethylene Vinyl Acetate (Bridgestone.com 2016, Hanson 2014).

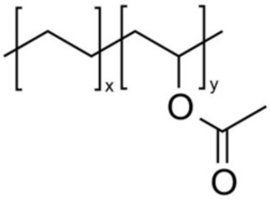

Fig. 6 Chemical composition of EVA.

EVA sheets are mainly used for the manufacturing of laminated glass for buildings, solar cell encapsulation and decorative laminates. EVA is well suited for small production units, it can be processed only by use of vacuum and temperature; no autoclave is required (Bridgestone.com 2016, Hanson 2014).

The final and irreversible three-dimensional cross-linking of the thermoset material occurs during lamination process by use of organic peroxide as catalyst (Fig. 7). Time and temperature are thus crucial parameters determining final film performances (haze, impact resistance, ageing resistance, wind and snow load resistance among others). If well processed, EVA has good durability, high adhesion and good resistance to heat, moisture and UV(Bridgestone.com 2016, Hanson 2014).

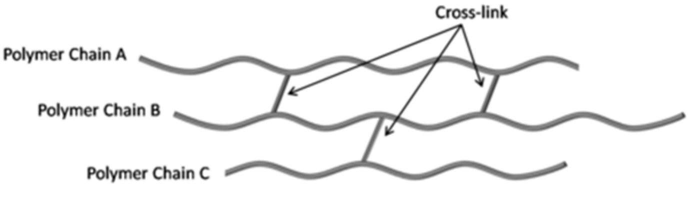

Fig. 7 Cross-linking mechanism

2.3. Ionomer

Ionomer based interlayers were introduced first for security and “anti-hurricane” markets in the second half of the 20th century. Due to its stiffness, other fields such as blast resistance and structural applications were identified afterwards. Ionomers are produced by a copolymerisation of ethylene and methacrylic acid in the presence of a small amount of metal salts which bind permanently to the polymer (Fig. 8). Thanks to the elastic-plastic polymer chain and rigid metal-oxygen ionic bond, ionomers show higher stiffness compared to other interlayers. They are usually extruded in rigid sheets, except for thickness of 0.89 mm (Stelzer 2010).

Fig. 8 Ionomer structure

3. Samples Preparation

For the purpose of this paper, only 4 mm glass panes were used for lamination and testing. The use of thicker glass would lead to mechanical resistance increase and could hide the interlayers performance contribution. Five common interlayers available on the market were selected for laminated glass fabrication and testing.

- Standard PVB (Stand PVB)
- Acoustic PVB (Ac PVB)
- Structural PVB (Rigid PVB)
- EVA
- Ionomer

These materials have diverse physical and chemical properties; therefore, different temperature dependent performance profiles were expected.

Regarding the interlayer thickness, the target was to be as close as possible to 0.8 mm, which means use of 0.76 mm foil of PVB, 0.8 mm foil of EVA and 0.89 mm of ionomer. Samples were prepared by use of vacuum bags and a middle-size industrial autoclave according to producers requirements; either by use of lamination under vacuum (EVA) or by use of vacuum + autoclave process (PVB, ionomer). Annealed glasses taken the same float production batch were washed according to internal standard procedure.

4. Testing Procedure

4.1. Ball Drop Test

Resistance to hard impact is described in this paper. Other types of impact (e.g. punching ball according to EN12600, hard impact according to EN356, static loads and others) are not part of this study; they will lead to different interlayers behaviour and thus to different results.

European Automotive R43 regulation with minor modifications was used for testing. The modification consists in impacting by a 2.26kg steel ball at different temperatures instead of at ambient one. Since the study required assembly of large amount of samples and their manipulation at very low and very high temperatures, R43 is easier to be applied (samples of 300x300mm) compared to EN356 (samples of 900x1100 mm) and EN12600 (1938 x 876 mm).

Laminated glasses were placed in a horizontal position within a metallic frame and impacted at different temperatures and heights. Penetration resistance of such glass laminates can be characterised by use of Mean Break Height (MBH). MBH is generally defined as the ball drop height at which 50% of samples would hold the ball and 50% would allow penetration. Iterative method was used for the MBH determination - starting from higher impact heights and approaching the final MBH value. About fifteen samples per interlayer were impacted to set the MBH at defined temperature, see the exact amount in the annex.

4.2. Acoustics

The effectiveness of glass as a material that acts as sound barrier is well known. Nevertheless, monolithic glass has specific critical or coincident frequency at which the wavelength of bending waves in the glass surface equals the wavelength of sound in air. This critical frequency changes with the glass thickness, moving towards lower frequencies as the glass gets thicker. Unfortunately the critical frequencies are located in the zone of highest human hearing ability (D'Haene 2003, Lilly 2004).

Efficient technique for improving the acoustic performance of glass is to use a laminated glass. The inner layer provides a significant amount of internal structural damping to the glass. This damping effect has a major impact on the sound transmission properties of glass at high frequencies, especially near its critical frequency and decreases the sound transmission loss dip (Schimmelpenningh 2012; Lilly 2004).

The MIM method described in the ISO 16940 was used for evaluation of acoustic performances at different temperatures. Three laminated samples composed by the interlayer and two 4mm glasses of 25x300 mm dimension were used for loss factor measurement and subsequent Sound Transmission Loss (STL) and Weighted Sound Reduction Index (Rw) calculation.

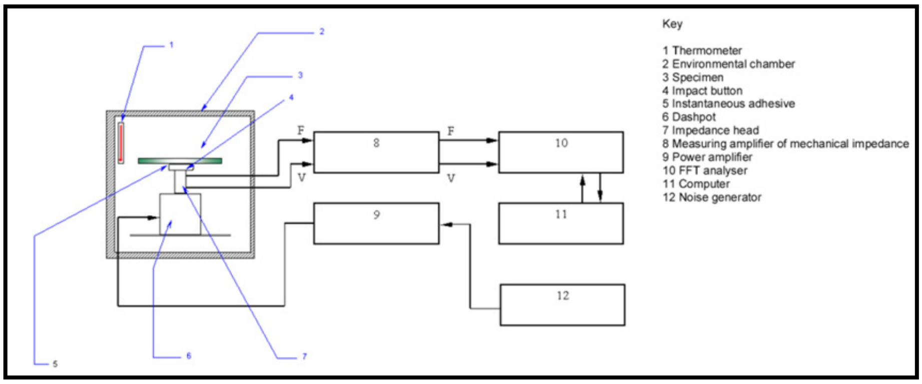

Fig. 9 Principle of the MIM method

Laminated samples of defined dimensions were glued in the centre with a steel made impact button by use of cyanoacrylate glue and fixed to an impedance head. White noise of 0-5000 Hz was generated to introduce the vibration into the specimen. First three resonance frequencies were recorded and used for following STL and Rw calculation.

Weighted sound reduction index, Rw, is defined as ten times the common logarithm of the ratio of the sound power W_1 which is incident on a partition under test to the sound power W_2 transmitted through the specimen. This quantity is denoted by Rw and is expressed in decibels.

$$Rw = 10 * \log \frac{W_1}{W_2} \tag{1}$$

This standard, as well as the ISO 140-3, which is used for acoustic performance measurement of large scale specimens, are designed for about 20°C. Nevertheless, the question rises same again what are the acoustic performances of a laminated glass at different temperatures.

5. Results

5.1. Hard Impact Resistance

Samples made of different interlayers were impacted with 2.26 kg steel ball according to R43 regulation. As seen in the graph below, MBH was identified for every interlayer at -20, 0, +20, 40 and 80 °C.

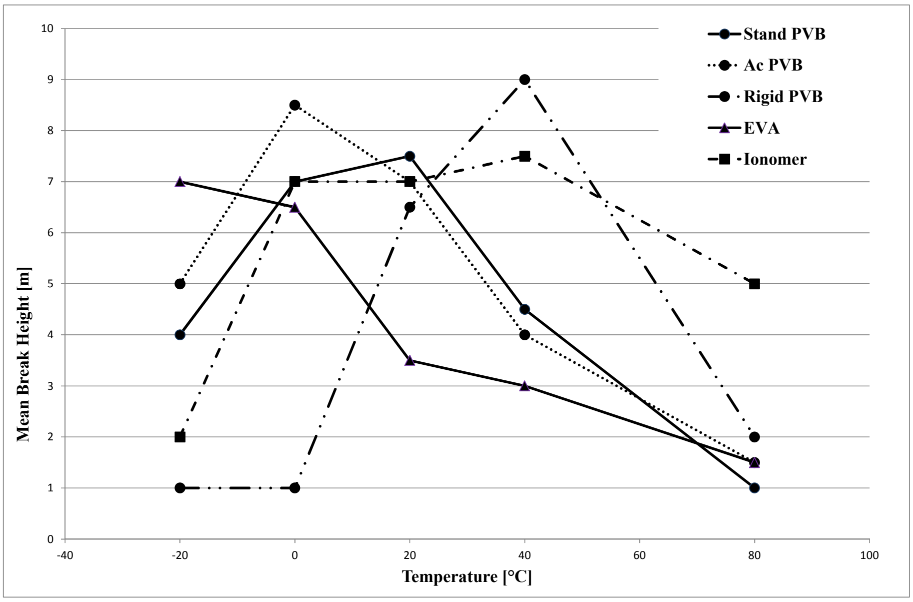

Fig. 10 Mean Break Height (MBH) of laminated interlayers at different temperatures

At ambient temperature, all laminated interlayers, except the EVA, showed similar MBH of about 7 meters. The specimens laminated with an EVA had a lower penetration resistance characterised by MBH of 3.5 meters. This EVA's drawback is well known and described.

Having a look at lower temperatures, the situation dramatically changes. At -20°C, the rigid interlayers (ionomer and rigid PVB) don't have enough elasticity to resist to the impact leading to very low MBH, while the EVA is more resistant than at ambient temperature. Its resistance at -20°C is the highest among all tested interlayers. Acoustic PVB resists more to impact than a standard one due to its slightly higher plasticizer content that increases its softness.

At 0°C, we still see very low impact resistance of the rigid PVB, while EVA, ionomer and standard PVB resist approximately to the same impact height (7m). At this temperature, the acoustic PVB is the most resistant interlayer.

Going up to 40°C, we see a decrease of performances for EVA, acoustic PVB and standard PVB compared to 20°C. Ionomer resits to about the same height while rigid PVB softens and shows high MBH of 9 m. At 80°C, all interlayers performances decreased. Except the ionomer, they were not able to resist to more than 2 m.

Correlation between maximum MBH and glass transition temperature (Tg) was tried to be put in evidence (Table 1). The Tg is the temperature region where the polymer transitions from a hard, glassy material to a soft, rubbery material. Tg is not a discrete thermodynamic value, but a temperature range over which the mobility of the polymer chains increases significantly. Its determination is very dependent on the used method, see the indicative values found in the literature (Dhaliwal 2002; DuPont 2016; Lilly 2004; Monserez 2013, Decourcelle 2009).

Table 1: Temperature of maximal Mean Break Height and Glass transition temperature of tested interlayers

Specimen	T of MBH max [°C]	Tg [°C]
Standard PVB	20	12-25 [1,2,3,4]
Acoustic PVB	0	10-15 [2]
Rigid PVB	40	45 [2]
EVA	-20	-28 [5]
Ionomer	40	55 [6]

[1] Dhaliwal 2002, [2] Decourcelle 2009, [3] Monserez 2013,[4] Weller 2005, [5] Hanson 2014, [5] DuPont 2016

As seen in Table 1, the Tg and temperature of maximal MBH lie very close one to the other. Unfortunately, there are some limitations in this study; the Tg of EVA was not reached since the minimal temperature of the impact resistance was -20°C while the Tg of the EVA is about -30°C. A second limitation is the big gap between tested temperatures, especially between 40 and 80°C, which is an important interval for rigid PVB as well as for ionomer. Complementary study with finer temperature intervals close to the interlayers' Tg would be required.

5.2. Acoustic Performance

As in case of impact resistance testing, only 4 mm glasses were used for lamination, as it is defined in the ISO 16940 standard. Weighted sound reduction index (Rw) was calculated to assess laminated glass acoustic performances. The Rw is a single-number quantity which characterises the airborne sound insulation of a material or building element over a range of frequencies; for results, see the graph below.

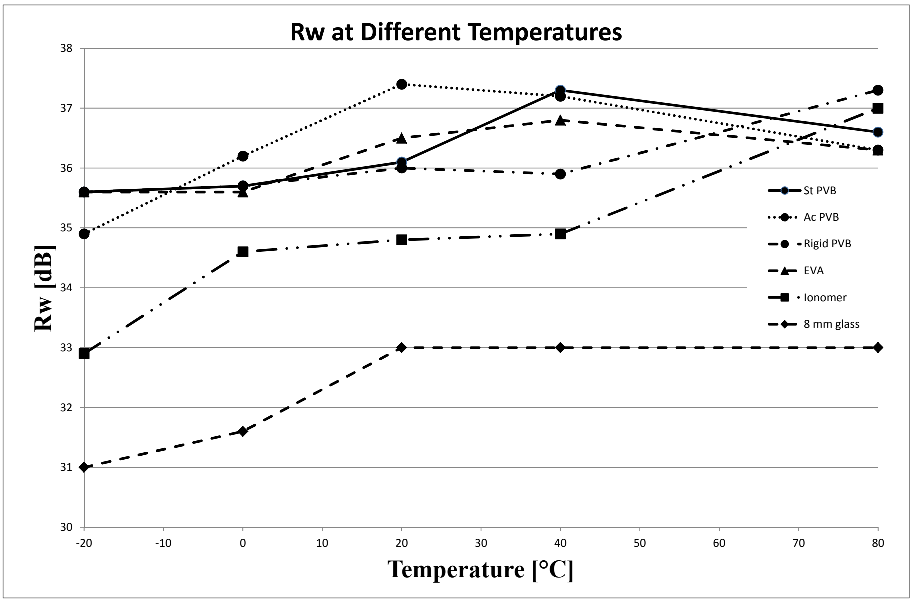

Fig. 11 Weighted Sound Reduction Index (Rw) at different temperatures

As a general trend, laminated composites show better acoustic performance compared to a single 8mm glass sheet. Acoustic PVB is designed to be most performant at ambient temperature. Standard PVB is most efficient at 40°C and stiff materials (rigid PVB and ionomer) even at higher temperatures. EVA shows similar performance all over the measured temperature range with a slightly better Rw value at 40°C. Since the ear sensitivity is 2dB, most of the Rw differences would not be perceived by a human ear.

Moreover, Rw value is just a single number that indicates "global" material performance. To understand the behaviour of the laminates within the whole frequency range, especially at critical frequency, Sound Transmission Loss curves need to be analysed. These curves were calculated for each material according to ISO 16940 standard, see Fig. 12 - Fig. 18.

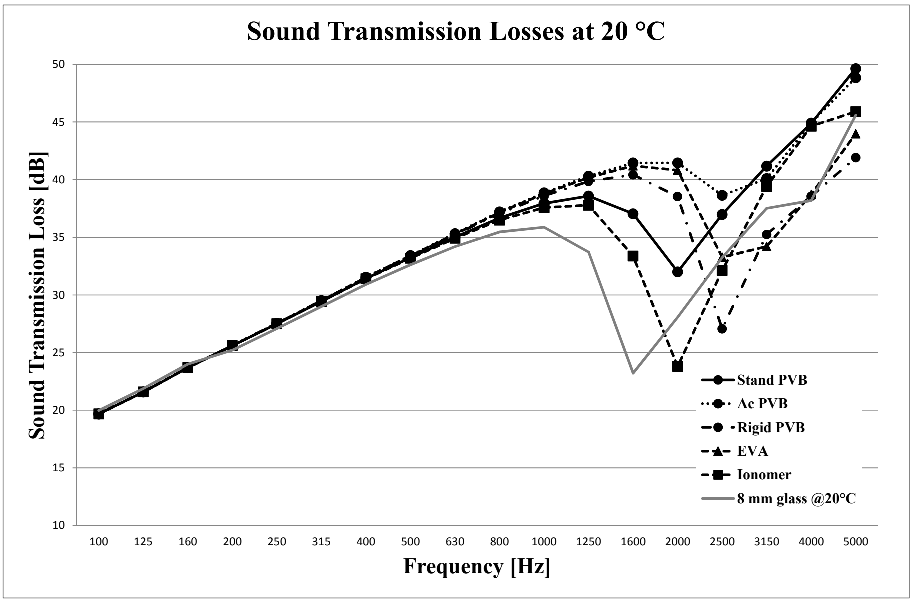

Fig. 12 Sound Transmission Loss Curves of laminated glass at 20°C

Several phenomena can be deduced from the STL curves. Since the specimens are not mounted or fixed to a frame but have free edges, there is no STL increase in the very low frequencies. In the frequency range of 100 to 800 Hz, acoustic performance is driven by the weight of the glass and since the glass stiffness is very similar at -20 and at 80°C, the curves are almost identical.

Above 800 Hz, the interlayers start to contribute to the STL profile. Acoustic PVB shows best performance with very shallow dip at 2500 Hz while ionomer and rigid PVB have a drop comparable with a clear 8 mm glass shifted to higher frequencies. If you compare STL value of the acoustic PVB and the ionomer at 200 Hz, there is almost 20 dB difference. If you would compare only the Rw values, you would find about 3.5 dB difference.

Regarding EVA, it shows better performances than other interlayers (except acoustic PVB) between 1000 and 2000 Hz following by a coincidence dip at about 2500 Hz.

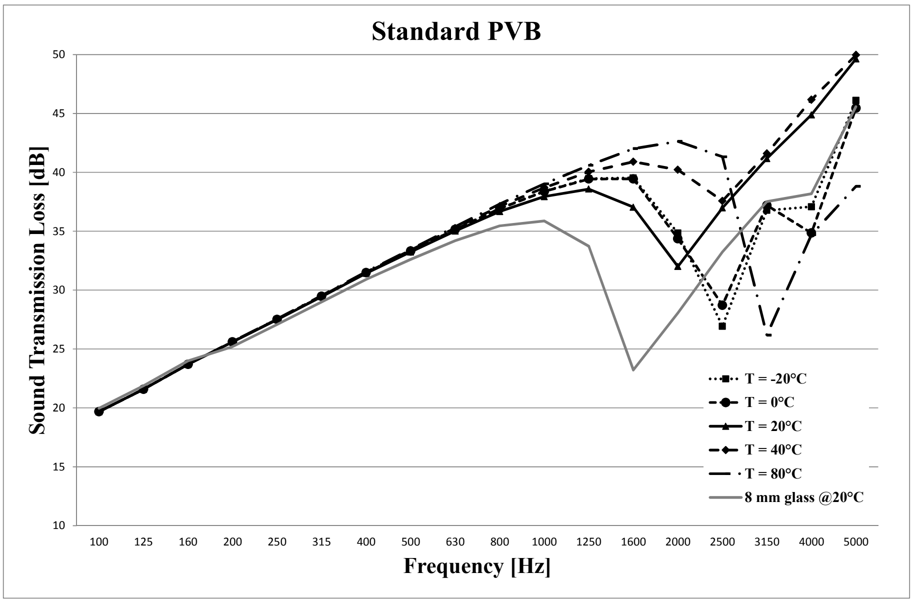

Fig. 13 Sound Transmission Loss Curves of glasses laminated with standard PVB at different temperatures

Standard PVB (Fig. 13) shows a performance dip at 2000 Hz at ambient temperature. Because the interlayer stiffness is temperature dependant (Délincé 2014), there are differences in the STL profile for each type of interlayer. As see above, sample laminated with the standard PVB has improved acoustic dampening at 40 °C resulting in a low STL decrease.

Acoustic PVB (Fig. 14) was designed for its maximal acoustic performances according to the standards that are defined at ambient temperature. Below and above this temperature, the interlayer loses its dampening properties resulting in an important coincidence dips.

Rigid PVB (Fig. 15) shows poor dampening at temperatures up to 40°C due to its stiffness. At 80°C, the interlayer is soft enough to be able to dampen the sound and behave similarly to the acoustic PVB.

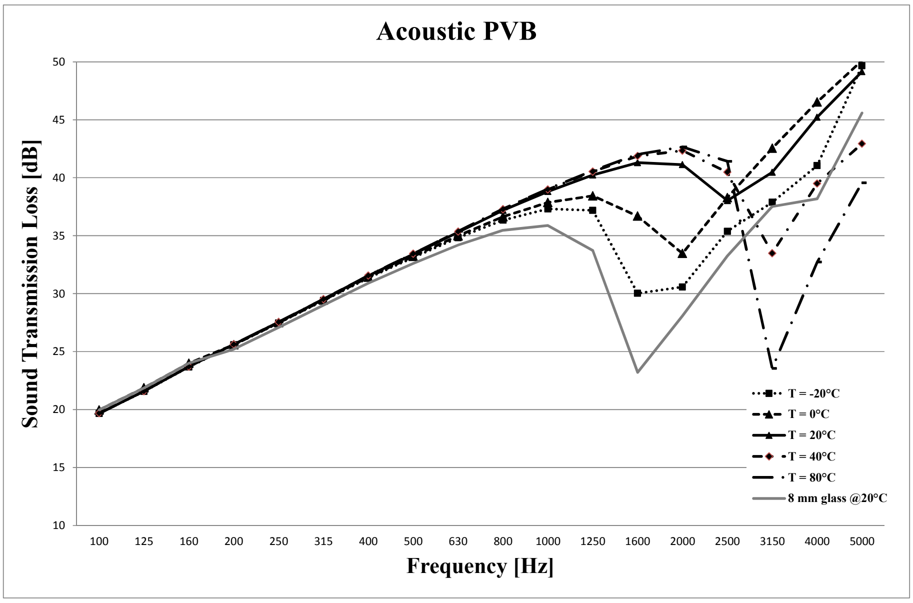

Fig. 14 Sound Transmission Loss Curves of glasses laminated with acoustic PVB at different temperatures

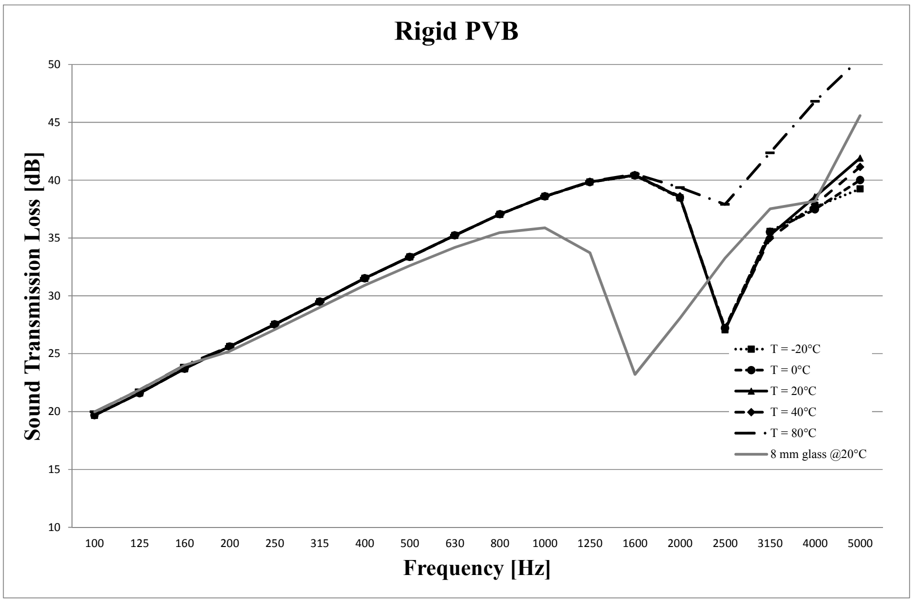

Fig. 15 Sound Transmission Loss Curves of glasses laminated with rigid PVB at different temperatures

Fig. 16 shows the comparison between the STL of standard, acoustic and rigid PVB at different temperatures. Rigid PVB at 80°C and standard PVB at 40°C behave similarly to the acoustic PVB at ambient temperature. Standard PVB as well as rigid PVB soften at higher temperatures that results in a better dampening. EVA at room temperature and ionomer at 80 °C were also included for comparison; their dip is more significant than in case of PVBs.

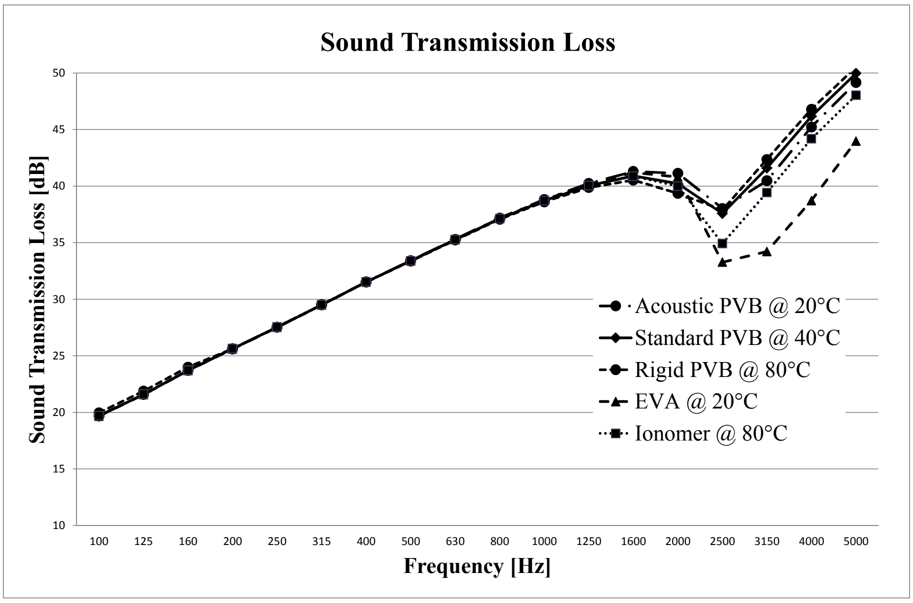

Fig. 16 Sound Transmission Loss comparison of different PVBs at different temperatures

Regarding EVA (Fig. 17), this interlayer is typically not being used for its acoustic performances. Even if it shows better transmission loss between 1000 and 2000 Hz, there are important dips in the coincidence frequencies at all measured temperatures.

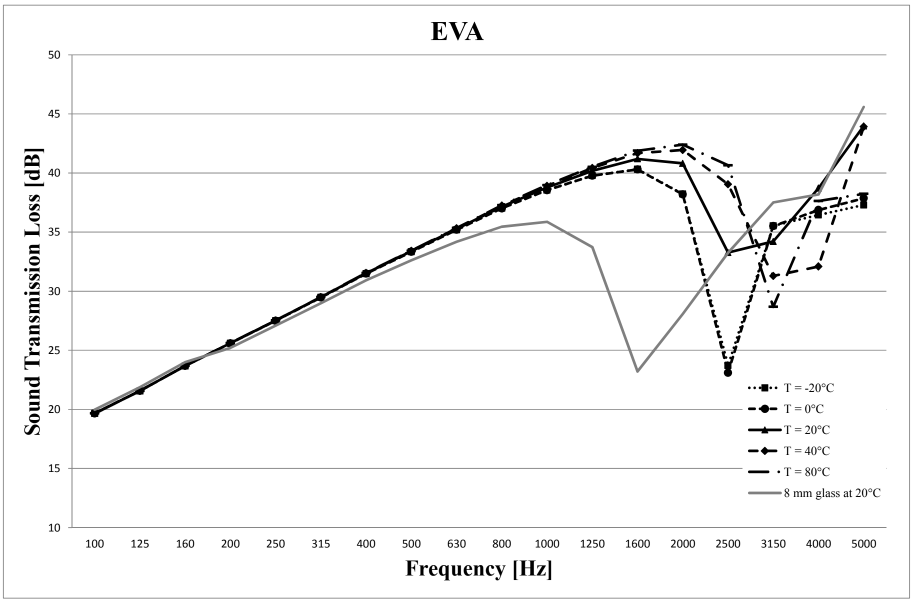

Fig. 17 Sound Transmission Loss Curves of glasses laminated with EVA at different temperatures

Same trends as for the rigid PVB can be stated for the ionomer interlayer (see Fig. 18). Nevertheless, the dampening effect is not as significant as for the rigid PVB. It’s possible that additional temperature increase would lead to STL improvement.

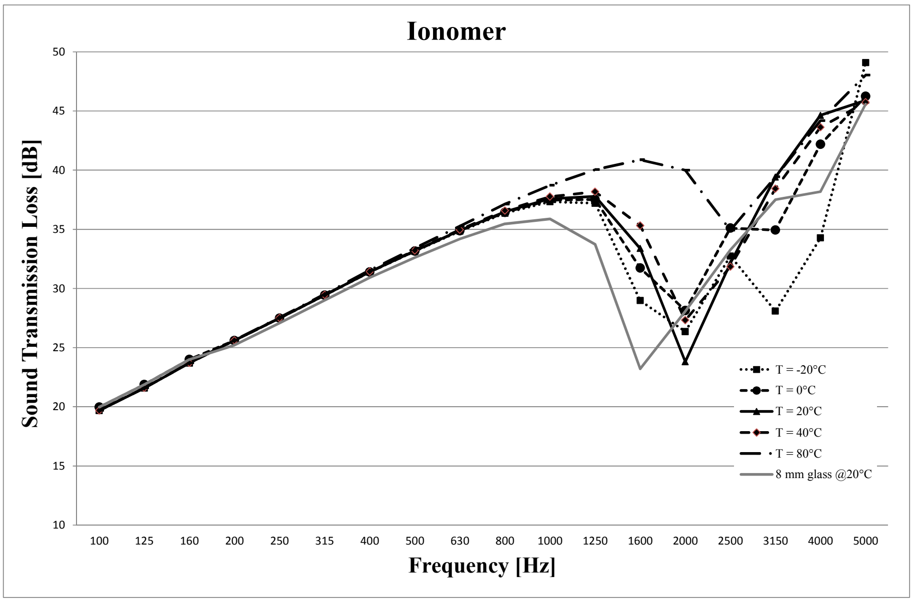

Fig. 18 Sound Transmission Loss Curves of glasses laminated with Ionomer at different temperatures

6. Conclusions

Impact resistance as well as acoustic performance of laminated glasses at standardized conditions are well known and described. Laminated glass behaviour and its temperature dependence is deeply studied and theoretically well described. However, limited information is available about laminated glass performances at low and high temperatures when tested according to industrial norms and standards.

Therefore, a study was launched to investigate the influence of temperature the impact resistance and acoustic performance of laminated glass. PVB, EVA and ionomer based interlayers were laminated with 4 mm glasses and tested. Resistance to hard impact at different temperatures (-20°C up to 80 °C) was determined by use of modified ball drop test according to the automotive R43 standard. Mechanical impedance method (MIM) was used for acoustic performance determination within the same temperature range. The general trend amongst all samples is that mechanical as well as acoustic performances of an interlayer are strongly temperature dependent. Unfortunately, current normative standards are defined at ambient temperature. The research showed that a different approach might be needed for some special applications. As it is the case e.g. for EVA, better temperature resistance (yellowing, delamination) doesn't mean better performance (mechanical, acoustic) at higher temperature. Based on this evidence, it is clear that one unique type of interlayer cannot fulfil all design requirements throughout the whole temperature range. It is worthy to remember that laminated glass is usually placed on the internal side of the façade glazing, therefore in standard conditions it will have a surface temperature close to the temperature of the internal air. However, in order to achieve specific aesthetics, facades become more complex and glazing can be in some cases exposed to relatively warm environment. In addition, the trend of glazed facades has also been spread to cold regions. For both cases, safe solutions are required by the client.

Since the hard impact resistance is only a piece of the complete laminated glass characterisation, other studies need to be undertaken (loading resistance, soft impact, use of thicker glass…) to gain a global understanding of the behaviour of these common commercial interlayers.

7. Annex

Table 2: Amount of impacted samples per temperature for MBH determination

Specimen	-20 °C	0 °C	20 °C	40 °C	80 °C
Stand PVB	15	15	15	12	7
Ac PVB	15	15	15	12	8
Rigid PVB	7	7	15	15	15
EVA	15	15	15	10	7
Ionomer	15	15	15	15	15

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