Temperature effects on laminated glass at high rate

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Abstract

The load bearing capacity of a laminated glass pane changes with temperature. In blast protection, laminated glass panes with a Polyvinyl Butyral (PVB) interlayer are usually employed. The post-crack response of the laminated pane is determined by the interlayer material response and its bond to the glass plies. An experimental study has been performed to determine the effects of temperature on the post cracked response of laminated glass at a test rate of 1 m/s for PVB thicknesses of 0.76 mm, 1.52 mm and 2.28 mm. Tensile tests were carried out on single cracked and randomly cracked samples in a temperature range of 0°C to 60°C. Photoelasticity observation and high speed video recording were used to capture the delamination in the single cracked tests. Competing mechanisms of PVB compliance and the adhesion between the glass and PVB, were revealed. The adhesion showed an increase at lower temperatures, but the compliance of the PVB interlayer was reduced. Based on the interlayer thickness range tested, the post-crack response of laminated glass is shown to be thickness

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1. Introduction

When an explosion occurs, a blast pulse propagates in the form of a pressure wave from the origin of the explosion. When this blast wave strikes a glass window, the glass can fail and the pressure wave then enters the building, causing damage. Furthermore, projection of glass fragments at high speeds can cause injuries. Laminated glass is often employed to avoid such effects. In protective design, three factors must be considered. Firstly, the laminated glass pane must be able to absorb the pressure pulse by its deformation. Secondly, the pane must stay in its frame and not detach from it. Finally the frame and its fixing must survive. All requirements are important. This paper addresses the first requirement.

As with most polymers, PVB’s mechanical response is temperature dependant [1, 2]. In some climates across the globe, air temperatures can reach up to 50°C and surface temperatures can exceed this value. This has various effects on the material properties of the interlayer, hence affecting its blast performance. This study aims to shed light on this issue.

The through-cracked tensile test is a testing method which has been used by various authors to characterise the post-crack behaviour of laminated glass. In this test, originally called the 'tension adhesion test', the laminated glass is scored normal to the loading direction, creating a coincident single crack on both sides and tested in tension. This test had originally been carried out in 1996 by Sha et al. [3] to
characterise the adhesion between the glass and the PVB interlayer. They developed this method as they believed it had advantages over the peel and pummel tests which are commonly used to assess adhesion levels.

Similarly, single cracked tensile tests were later used in 2000 by Seshadri et al. [4] to assess the mechanical behaviour of cracked laminated glass. They stated that if the material constitutive model is known, the interfacial fracture toughness and energy release rate can be calculated from the tensile tests on cracked laminates. The outcome of their work was the calculation of the energy dissipated in delamination of the interlayer.

Seshadri et al. [5] later extended their work by developing a finite element model which would allow analysis of more complex geometries other than the tensile specimens they tested; assuming a hyperelastic material model. This model predicts the behaviour of laminated glass as a function of geometry, interlayer properties and number of glass fragments. Delince et al. [6] continued the work of Seshadri, but they could not reproduce the results of Seshadri. They proposed that this was because their test specimens spent a very short time in the steady state.

In extension to the single crack tests, Hooper et al. [7] conducted tests with multiple parallel cracks normal to the loading direction at different spacings. They then used a rate dependent Johnson-Cook plasticity model to characterise the stress-strain post-crack response of laminated glass. Hooper [8] also conducted random cracked tests, in which the laminated samples were randomly cracked and tested in tension. Del Linz et al. [9] also looked at delamination of the PVB interlayer in single cracked tensile tests and compared experimental data with a finite element model.
In the literature, the work on temperature dependency of laminated glass is very limited. Bermbach et al. [10] have conducted shock tube experiments at 13°C and 30°C. They concluded that at 30°C, greater strain and strain rate are observed than 13°C. Hooper et al. [2] conducted DMA (dynamic mechanical analysis) tests on PVB alone and they reported that below 5-10°C the material behaves glassy. Above this temperature, it goes into transition and finally it behaves rubbery above 40°C.

From the literature, there is a clear gap in understanding the post-crack behaviour of laminated glass in the higher temperature range. The present study investigates this at a range of temperatures from 0°C to 60°C. Single cracked and random cracked tests were carried out for three different PVB interlayer thicknesses. These tests were carried out at a high rate of 1 m/s. As an example, in the delaminated PVB ligament at room temperature, this corresponds to an approximate strain rate of 21 s⁻¹ for the 0.76 mm interlayer in the steady state region (it should be noted that the initial strain rate in the test is greater than this and it also varies during the test). This is a representative order of magnitude of the strain rate which the laminated glass experiences, under blast loading. Hooper conducted blast tests and reported that on the edges of the pane, where strain is highest, strain rates can reach up to 20-25 s⁻¹ [8]. Bermbach et al. also conducted shock tube tests at which strain rates were reported to reach up to 19 s⁻¹ on the laminated glass panel [10].

The outline of this paper is as follows. The specimen geometry and test matrix are given, and the experimental setup is described. This is followed by the presentation of the results for the single and random cracked tensile tests on laminated glass. Finally a discussion is made on the behaviour of the laminate at different temperatures and interlayer thicknesses depending of stiffness and adhesion.
2. Materials and Methods

2.1. Test specimens

The experiments were carried out on laminated glass samples comprising of three different PVB interlayer thicknesses, namely, 0.76 mm, 1.52 mm and 2.28 mm. These interlayers were laminated with 3 mm annealed glass on each side. The sample length was 150 mm and the width was 60 mm. The specimens were prepared using a standard lamination procedure by a commercial company (Kite Glass). The PVB used in the laminate was sourced from Everlam.

To enable the gripping of the sample by the machine, aluminium tabs were bonded to the laminated glass using an adhesive (Araldite 2021). Prior to bonding, the aluminium tabs and the gripping ends of the glass were grit blasted; followed by cleaning of the surface with acetone.

The single cracks were prepared using a tool designed at Imperial College. The laminated glass samples were scored on each side, followed by a gentle tap over the crack with a light ball hammer. This propagated the crack on the opposite side of the hammer tap. This procedure was repeated on the opposite side of the glass, thus, enabling a coincident crack to form on either side of the glass. Figure 1 and Figure 2 display a schematic of the single crack and random crack samples.

The random cracked specimens are closer to what the laminated pane experiences under blast loading. They were prepared using a ball hammer by forming random cracks on the laminated glass sample. The location of the hammer impact and number of hits were kept almost the same between subsequent samples, such that
the size of the fragments produced were approximately 5-10 mm.

For the single crack tests, 30 specimens were tested, and for the randomly cracked test, 63 specimens. Experiments were carried out over a temperature range of 0°C to 60°C, in increments of 10°C. Tests were conducted at a constant test rate of 1 ms\(^{-1}\). In some cases up to four repeats per test condition were carried out. Tables 1 and 2 show the number of specimens tested for the single and randomly cracked tests respectively, at each interlayer thickness.
Table 1: Number of tests for single cracked samples.

<table>
<thead>
<tr>
<th>Thickness of PVB (mm)</th>
<th>Temperature (°C)</th>
<th>0</th>
<th>10</th>
<th>20</th>
<th>30</th>
<th>40</th>
<th>50</th>
<th>60</th>
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<td>1</td>
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Table 2: Number of tests for randomly cracked samples.

<table>
<thead>
<tr>
<th>Thickness of PVB (mm)</th>
<th>Temperature (°C)</th>
<th>0</th>
<th>10</th>
<th>20</th>
<th>30</th>
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2.2. Experimental setup

The tests were carried out on an Instron servo-hydraulic high rate testing machine. Figure 3 shows the experimental setup. The sample was connected to the grips through dowel pins. The load was applied through a lost-motion rod; this allowed for the system to accelerate to the desired test velocity, prior to pulling the sample. The load was measured using a piezoelectric load cell (PCB model 222B). This was placed between the sample and the machine base.
Since the glass was cracked, what was essentially being tested was the PVB. Since the compliance of the test rig is negligible compared to the PVB, the actuator extension was deemed acceptable as the displacement during the test.
In the single cracked tests, it was important to get an accurate measurement of
This was done through the use of a photoelasticity setup (Figure 4).

Linear polarising filters were placed front and behind the sample at a 90° difference. The camera was placed behind the front polarising sheet. Halogen light with a diffuser on front of it was placed behind the rear polarising sheet. Finally, the sample was placed between the polarising filters. This allowed different regions of the PVB delamination to be identified in the high speed recording. A clear delamination front was visible and using image processing techniques, delamination was quantified.

Figure 4: Photoelasticity setup.
To achieve the required temperatures, the samples were kept in an Instron environmental chamber for 24 hours prior to testing. This was to ensure that the PVB was at the same temperature as the surface of the glass. In this case it was impossible to measure the temperature of the PVB interlayer as thermocouples would have had to be placed during the lamination process. This could have significantly affected the lamination quality. Instead, the temperature of the environmental chamber was measured to $\pm 0.5^\circ$ using air temperature sensors built into the environmental chamber and also independent thermocouples.

In order to enable photoelasticity to be performed a camera was required at the front of the sample and light behind the sample. Linear polarising film was also needed on front and behind the sample. This would not have been possible if the samples were tested in an environmental chamber. The samples were conditioned in the chamber 24 hours prior to testing. They were then removed from the chamber and then tested within 30 seconds in all cases. The glass surface would have cooled slightly in that time; however, the more important temperature is that of the PVB. Since the PVB was sandwiched between two glass layers, the change in temperature during that time was assumed to be negligible.

2.3. Data Analysis

The load data was extracted and the engineering stress in the PVB ligament was defined by equation 2.1.

$$\sigma = \frac{P}{b \times t_{\text{Interlayer}}} \quad (2.1)$$
Where $P$ is the load, $b$ is the initial sample width and $\delta_{\text{interlayer}}$ is the initial interlayer thickness.

In randomly cracked specimens, a maximum load and an average load, followed by a corresponding stress was calculated. However in single cracked tests, a more relevant parameter is the adhesive load. This can be defined as the lowest load plateau at which delamination propagates [11]. As an example Figure 5 and 6 show how the adhesive load is calculated for different load-displacement profiles. The dashed red line represents the adhesive load.

![Figure 5: Single crack 0.76 mm interlayer at 20°C.](image1)

![Figure 6: Single crack 0.76 mm interlayer at 40°C.](image2)

For the randomly cracked specimens, the nominal strain, $\varepsilon$, in the cracked laminate was calculated from the gauge length, $l$, at the start of the test which was 105 mm, and the displacement, $\Delta l$, using equation 2.2.

$$\varepsilon = \frac{\Delta l}{l}$$  (2.2)
It should be noted that this is an average strain in the gauge length of the cracked laminate and not the strain in the PVB.

In the random cracked tests, the strain in the PVB was not considered, because it would be almost impossible to account for the strain in every ligament of the PVB. During the experiment, the PVB locally delaminates in multiple locations surrounding cracked glass fragments. This occurs in many directions and angles compared to the loading direction. Even though the local strain surrounding each glass fragment can be vary greatly, this measure of nominal strain in the cracked laminate as whole is still useful. This value can be used to input into computational models when considering the failure of the laminate as a whole on a global scale. Local strain becomes important when assessing the fracture mechanism of the PVB on a very local scale.

For the single cracked tests, it is more relevant if we look at the strain the PVB. This makes the calculations slightly more complicated. This is because the gauge length of the sample changes during the experiment due to delamination. Then the photoelasticity setup is important in allowing the delamination front to become visible.

The pictures taken by the high speed camera during the experiment were processed in MATLAB [12]. An edge was identified and tracked, which allowed for the displacement in pixels to be calculated. The pixel to mm ratio was calculated using a known distance in the picture. This was used to convert the displacement in pixels to displacement in mm. A similar technique was used to calculate the delaminated length, \( l_{\text{del}} \), by tracking the delamination front.

The original length is the total PVB ligament length minus any deformation. This
is equivalent to the total delamination length. The total delaminated length was calculated by summing the upper delaminated length \( (L_U) \) and the lower delaminated length \( (L_L) \). The nominal strain, \( \varepsilon \), in the PVB ligament for the single cracked specimens was calculated using equation 2.3. Figure 7 illustrates this. Note that the strain in the PVB, particularly around the edges can vary and the strain defined here is only an average strain across the PVB length.

\[
\varepsilon = \frac{\Delta \text{Length}}{\text{Original Length}} = \frac{\Delta l}{l_{\text{del}}} \tag{2.3}
\]

![Figure 7: Delamination of PVB from laminated glass.](image)

The total energy absorbed during the test was calculated as the area under the
load-displacement graph. The trapezoidal numerical integration method was used to calculate this. It should be noted that this includes the energy of delamination, viscoelastic losses, and the strain energy [13].

In all calculations, the standard deviation was calculated. In the plots in the following sections, the error bars represent one standard deviation.

3. Results

3.1. Single cracked

Figure 8 illustrates an example of the load-displacement trace obtained from the single cracked tests. This is for the specific case of an interlayer thickness of 0.76 mm. At lower temperatures high load with low displacement occurs. As the temperature increases, the load drops and displacement to failure increases. Finally at the greater temperatures, both the load and displacement decrease. Similar trends were also observed for the other interlayer thicknesses and repeat tests.

At lower temperatures, the load increases up to a maximum then drops to an almost constant plateau. Whereas at high temperatures, initially there is a small plateau, followed by a gradual increase in load and finally reaches a maximum towards the end of the test.
As an example, by having a closer look at the 20°C case (Figure 9). It can be seen that the load initially rises to a peak, followed by a small drop and settles at almost a constant load for most of the duration of the test.

Selected frames from the high speed recording are shown (Figure 9). The test begins by a mixture of delamination and stretching of the PVB. The PVB is also seen to bridge the gap between the two edges of the cracked glass. As the loading progresses, further delamination takes place, new material becomes available for deformation and the PVB also stretches. This leads to an extension of the PVB bridge between the two glass pieces. The delamination continues until either all the PVB has separated from the glass or until the point of PVB tearing. The latter occurs in this specific case and in most cases. However, for some of the samples with the thicker interlayer, full delamination did occur. In the discussion, it is further explained.
why the thicker interlayers have a greater tendency to delaminate.

Figure 9: Load vs displacement at 20°C for 0.76 mm interlayer thickness in single crack test.

Figure 10 displays the samples at half of failure time and Figure 11 displays the final states prior to failure, for the 0.76 mm interlayer. At the lower temperatures (0°C and 10°C) and at 60°C, the amount of delamination and interlayer extension is low compared with other temperatures. Similar patterns were also seen for other interlayer thicknesses. At some instances where the delamination front was uneven, the value at the mid-span of the sample was considered.
At greater temperatures the load required for delamination to propagate (adhesive load) is reduced (Figure 12). The thicker interlayers also hold a greater load before delamination propagates. A greater absolute difference in load is observed between the interlayer thicknesses at lower temperatures in comparison to higher temperatures.
Using the optical methods described in section 2.3, the strain in the PVB bridge was calculated. As an example, Figure 13 displays strain against displacement for the 1.52 mm interlayer. Greater strain was observed for the low temperatures (0°C and 10°C) and also for the high temperatures (50°C and 60°C). Similar trends were observed for the other interlayers. It should be noted that the strain displayed in the figure was calculated using optical methods. As a result of this, for some of the experiments, the plots shown are not for the full duration of the test due to the sample not being in the boundaries of the camera for the full duration of the test.
The variation of strain with interlayer thickness was also examined. All temperatures were considered in this study, however only two are displayed in Figures 14 and 15 as a demonstration. The general trend is that the thinner interlayers undergo a greater strain and less delamination for a given displacement.
3.2. Random cracked

For the random cracked tests, the stress and strain throughout the test was calculated. Figure 16 shows a stress against strain plot for the specific case of the 2.28 mm interlayer. As the temperature increases, the stress reduces and failure strain begins to increase. As the highest temperature is approached, the failure strain is reduced. Similar trends were also observed for other interlayers and repeats.
Similar to the single crack case, the maximum load drops with temperature increase (Figure 17). The rate of load reduction with temperature is the greatest for the 2.28 mm interlayer, followed by the 1.52 mm and the 0.76 mm interlayer. However, the trend for drop in maximum stress with increasing temperature is fairly similar for all interlayer thicknesses (Figure 18).
The average load and average stress show trends similar to that of maximum load and maximum stress (Figures 19 and 20).

The maximum and average stress are fairly independent of interlayer thickness,
however the failure strain, $\varepsilon_r$, is not (Figure 21). At higher temperatures, greater failure strain is achieved. The 0.76 mm interlayer had the lowest failure strain and the 2.28 mm achieved the greatest.

For all interlayers, the total energy dissipated rises as temperature increases up to a maximum, then begins to drop down (Figure 22). For the 0.76 mm interlayer this maximum peak occurs at 40°C, and for the 1.52 mm and 2.28 mm this occurs at 20°C and 10°C respectively. The 0.76 mm interlayer dissipated the lowest energy and the 2.28 mm achieved the greatest. This was consistent at all temperatures.
4. Discussion

4.1. Single cracked tests

The glass transition temperature, $T_g$, of the PVB has been reported to be in the range of 15°C to 20°C [4, 2]. At lower temperatures, below $T_g$, the single cracked tests showed a sharp rise in load at initial times during the test, prior to delamination. This is because the material behaves glassy and stiff. This is followed by a load plateau where the dominated failure mode is through delamination (adhesive failure).

At the higher end of temperatures tested (above 40°C), where the material is rubbery and compliant, the load gradually increases as the test progresses, and reaches a maximum towards the end of the test. Taking the 40°C case as an example (Fig-
ure 6). A plateau is reached initially; this is where the main mode of failure is adhesive (PVB - Glass bond). As the test progresses, since the material is compliant, the delaminated front rounds off (no longer as sharp as before), therefore requiring a much greater load to propagate (similar to blunting of the crack tip). The mode of failure at this stage is a mixture of adhesive (delamination) and cohesive (PVB - PVB bond). Finally, the delaminated ligament can no longer support this load, it fails mainly cohesively and eventually leads to tearing.

In the peel test, a load, P, is applied at a peeling angle, \( \theta \), this creates a slope of the arm at the peeling front, \( \theta_0 \) (Figure 23). By reducing the peeling angle, the slope of the arm is also reduced. This leads to a greater adhesive load [14]. The single cracked test is similar to the peel test, except that the peeling angle is 0°, however the slope at the delamination front can vary depending on the compliance of the delaminated PVB ligament. The increase in adhesive load at lower temperatures can be explained by the fact that at lower temperatures, the PVB is less compliant (more stiff), therefore it is stretched less. This creates a thicker delaminated PVB ligament, resulting in the angle at the peeling front being smaller than that of higher temperatures. This leads to a greater adhesive load. The opposite is also true for high temperatures, where the material is more compliant therefore being stretched more, leading to a greater slope at the delamination front, and a lower adhesive load. This is illustrated in Figure 24. A similar argument also holds for thicker interlayers having a greater adhesive load due to an increased effective thickness.
At the lower and higher temperatures mentioned, greater values of strain in the PVB bridge are obtained (Figure 13). In this instance, the strain is defined as the ratio between displacement and total delamination length. Greater values of strain means less delamination has taken place in comparison with the stretching of the PVB bridging ligament between the two parts of the specimen.

Lower strain (meaning more delamination) was expected when PVB is more stiff. PVB becomes less compliant at lower temperatures (below its transition temperature), thus a greater load can be transferred through it, therefore leading to a greater delamination. However this was not the case in the results obtained. This is because adhesive strength is increased at lower temperatures, making the propagation of the delamination front more difficult. Tian et al. conducted tests on adhesion between glass and PVB interlayer [15]. They concluded that from room temperature to -30°C, the adhesive tensile strength reduces by 65%, however the adhesive shear strength increases by 172%. In the single cracked tests, the main mode of adhesive failure is
through shear. This increase in adhesive shear strength causes less delamination to occur.

At temperatures in excess of $T_g$, it was expected that when the material is more compliant, greater displacements would be observed - however this was not the case due to reduced delamination. This is because as the PVB becomes more compliant, its load bearing capabilities are reduced; leading to lower delamination and thus a smaller delaminated PVB ligament. An optimum is reached between 20-40°C, where there is a balance between the adhesive strength and stiffness of the delaminated ligament.

The importance of choosing the correct interlayer thickness is apparent in the single cracked tests (figures 14 and 15). The thicker interlayer showed the lowest strain. Using the definition of strain for single cracked specimens, this means a greater ratio of delamination to stretching of the interlayer is obtained for thicker interlayers. The thicker interlayer has a greater effective stiffness, therefore resulting in a smaller mismatch with glass (even though the difference is still large). This results in a lower strain in the delaminated PVB ligament and therefore greater delamination.

The thicker interlayer generally showed the greatest variability in results. The lamination quality of the interlayer can severely effect the results. This is a possible source of uncertainty in the data.

A balance between the PVB stiffness and adhesive strength is required to reduced the chances of PVB tearing. By controlling the adhesion and transition temperature of the PVB, the desired optimum performance can be reached. As an example at
lower temperatures, below $T_g$, if the adhesion is reduced in the manufacturing process, greater displacements can be reached, hence the blast performance in colder environments is improved.

4.2. Random cracked tests

The failure mechanism of the single and random cracks are the same in the sense that when a load is applied, delamination occurs; the delamination ligament is stretched until failure occurs usually via tearing of the interlayer. However, the single cracked case cannot directly be related to the random cracks. This is because in the single cracked case, loading is perpendicular to the crack direction, whereas in the random case, the angle at which the load is applied varies in the sample (Figure 25).
The random cracked samples are therefore more comparable to failure of laminated panels under blast loading. As a result of this the main focus of the experimental program was put on random cracked tests. More repeats were performed on these samples; hence they show a lower error compared to the single cracked tests. From the low error bars on the random cracks, it can also be deduced that the variability in crack pattern between samples was not significant enough to affect the results.

The failure strain showed a general rise with increasing temperature (Figure 21). In this case, the strain is defined as the ratio of displacement to original gauge length.
The laminate is effectively being treated as a polymer with glass attached as extra mass. Therefore since at higher temperature, the polymer is more ductile, greater strain at failure is achieved. The failure strain for the 1.52 mm interlayer stays almost constant at temperatures above 20°C. It is unknown why this occurs.

Greater loads are observed at lower temperatures, due to increased adhesion and the PVB being more stiff. Greater displacements are observed at higher temperatures, due to PVB being more compliant. This results in the optimum temperature at which maximum energy was dissipated being between the high and low temperature extremes. The fracture behaviour of ductile thermoplastics are typically thickness dependant [16]. This resulted in the optimum temperature at which the maximum energy is dissipated being different for the three interlayers (Figure 22). It should be noted that there is a lot of overlapping of error bars (e.g. 0.76 mm), so the optimum temperature is not a single definite temperature.

The tolerable stress is severely effected by temperature. As the temperature is increased from 0°C to 60°C, for the 0.76 mm, 1.52 mm and 2.28 mm, the maximum stress decreases by 81%, 87% and 88% respectively. A graph which can be used as a conservative design guide based on the average stress data for random cracks is Figure 20. This should be used with a suitable safety factor. PVB is rate dependant, especially at high rates ([7, 17, 18, 19, 20]), therefore this data is only valid at the experimented strain rate (approximately 20 s⁻¹).
5. Conclusion

In this study the effects of temperature on the post-cracked response of laminated glass under blast loading were explored. Experiments were conducted at a high strain rate in the laboratory on samples consisting of a PVB interlayer laminated between 3 mm annealed glass. Three different interlayer thicknesses consisting of 0.76 mm, 1.52 mm and 2.28 mm were considered. Single cracked and randomly cracked tensile tests were conducted at temperatures in the range of 0°C to 60°C in increments of 10°C.

The post-cracked response of the laminated glass is influenced by the compliance of the PVB interlayer and the adhesion between the glass and PVB layer. From the single cracked tests, it was found that the adhesion between the glass and PVB layer is increased at lower temperatures. The glass transition temperature of the PVB is just below room temperature. As a consequence, the compliance of the PVB was very temperature dependant, in the temperature ranges tested.

The maximum energy absorbed by the cracked laminate was found to occur at a temperature where there is a balance between the adhesion and compliance of the interlayer. At greater adhesion the interlayer can tolerate greater loads, but lower displacements. Whereas, at lower adhesion, provided the interlayer is stiff enough to transmit the loads required to propagate the delaminated front, a greater delaminated PVB length is formed. This allowed for the cracked laminate to undergo greater displacements. Depending on thickness, this optimum temperature was also different. This is as expected, because fracture of ductile thermoplastics is thickness dependant [16].
The results from this study indicate that temperature dependence of laminated glass is important. By designing for a certain load at room temperature, it cannot be assumed the laminated pane will still survive blast loading at different temperatures. If extreme temperatures (above 50°C or below 10°C) are required, it is recommended that the adhesion should be modified to shift the temperature range of the optimum performance. Alternatively, the transition temperature of the PVB should be altered in the polymer forming process, thus allowing it to perform better at the extreme temperatures.

Acknowledgements

The authors acknowledge EPSRC (Engineering and Physical Sciences Research Council) and Arup Resilience, Security and Risk for financially supporting Mohammad Amin Samiei since the beginning of his PhD. The authors also thank Leroy Reed at Kite Glass for preparing, manufacturing and providing the test specimens.

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