

# Adhesion, creep and relaxation properties of PVB in laminated safety glass

Prof. Ing. Maurizio Froli, Dr. Ing. Leonardo Lani,  
Department of Civil Engineering, Structural Division, University of Pisa,  
Via Diotisalvi 2, Pisa -Italy

## Keywords

1=Laminated Glass    2= Adhesion    3 = Creep    4=Relaxation    5=Viscoelasticity

## Abstract

In Compression Shear Tests (CST) specimens of Laminated Safety Glass (LSG) are inserted in a special test device with an angle of 45° with respect of the loading force so that the compression and the shear components acting in the plane of the plastic interlayer have always the same magnitude.

The CST procedure has been adopted in the present research in order to assess the ultimate shear stresses of PVB interlayers of different kinds. Critical values of shear and normal stresses have been taken as measures of the adhesion properties of PVB to be implemented in calculations to model interfacial adhesion.

Different sets of specimens were prepared under different bond process conditions and tested at the Laboratory for Testing Materials and Structures of the University of Pisa.

The shear viscoelastic properties, as creep and relaxation, have been investigated on large specimens restrained by a eight point fixing. A numerical viscoelastic model of LSG was adopted to compare the FEM predictions with experimental results.

## Introduction

It is well known that adhesion between glass panes and plastic interlayers is a requisite of fundamental importance for the safety and the structural integrity of laminated glass.

The level of adhesion depends on many factors: type of materials, autoclave temperature, pressure and time of bonding process, cleaning process etc.

Surprisingly, any national or international standard requires minimum adhesion properties in spite of the importance to know and model bonding mechanisms between glass and polymers in order to get high quality LSG and to avoid delamination phenomena between glass and PVB.

Furthermore, the high adhesion of PVB to glass ensures, in the post-breakage phase, that fragments remain attached to the plastic film. On the other hand, a Laminated Safety Glass

with low PVB adhesion guarantees a higher impact resistance, since more energy is absorbed by elastic deformation of the plastic material. Therefore, the control of the adhesion properties should be such to satisfy at the same time the capacity to absorb impacts and the need for a sufficient bond strength [1].

Adhesion proprieties of PVB to glass are usually measured with the Compression Shear Test (CST) [2] that allows to reach the ultimate shear stress of PVB before glass collapses as it really often happens in single or double shear lap tests. As known, in a CST test a small specimen of LSG is inserted in the interface plane of two metallic units which is inclined of 45° with respect of the compression loading force so that the compression and the shear components acting in the plane of the plastic interlayer have at any instant the same intensity (see Fig. 2). The adhesion strength if given by the minimum shear force that causes the collapse of PVB before the collapse of glass.

### Test phase 1: Compression Shear Tests (CST)

Four rectangular LSG main panes each composed by two 500x150x6mm glass sheets have been prepared under different laminating conditions of autoclave temperature and pressure, as

indicated in Table 1. The 0.76 mm thick PVB foils have been previously stored under two different humidity conditions.

For each of the four panes, 30x50x50mm specimens have been cut and labelled as indicated in Figure 1 in order to exactly specify their original position in the pane. The firsts letter of the label is referred to humidity condition of PVB, the second is referred to the conditions of the lamination process.

Therefore the generated test population consists of 120 specimens subdivided in four homogeneous families: N-R, O-R, N-S, O-S. With reference to Fig.1, for example, label N-S/5B indicates a specimen cut from the central part of pane N-S.

CST tests were all performed at room conditions of 18°C temperature and 55% relative humidity with a test deflection velocity of 5 mm/min.

For each of the tested specimen the applied load and the relative displacement of the two steel units was recorded.

Figure 3 shows, for example, the results of test O-R/5A, where each curve is referred to one of the two inductive transducers. The lack of coincidence between the curves indicates a imperfect parallelism between the two loaded edges of the specimen. Table 2 collects the obtained test results.

Family name	Number of samples	Thickness (mm)	Dimensions (mm)	Pressure (bar)	Temperature (°)	Storage Humidity (%)
N-R	30	6+0.76+6	50x50	9.4	146	new
N-S	30	6+0.76+6	50x50	12.0	140	new
O-R	30	6+0.76+6	50x50	9.4	146	60
O-S	30	6+0.76+6	50x50	12.0	140	60

Table 1  
Labeling and lamination process parameters.

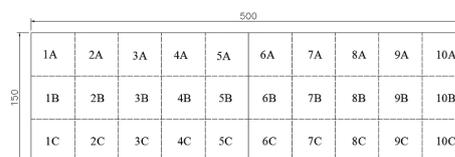


Figure 1  
Labeling and location of the specimens over the original LSG pane.



Figure 2  
CST test device

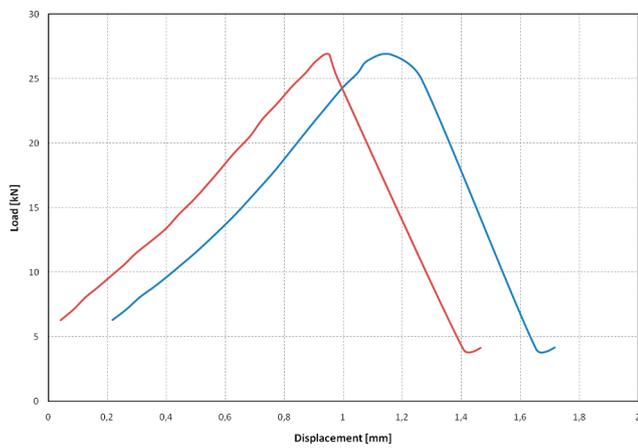


Figure 3  
Load vs. Displacement for O-R/5A specimen

Family name	$\tau_{av}$ [MPa]	Standard dev.
N-R	11,91	5,22
N-S	6,40	2,27
O-R	7,01	1,45
O-S	8,54	2,92

Table 3  
CST test results summary

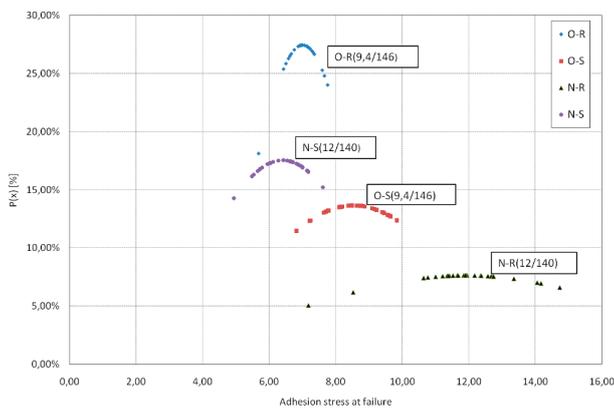


Figure 4  
Adhesion stress Vs. probability of failure

	O-R	O-S	N-R	N-S
Sample	$\tau$ [Mpa]	$\tau$ [Mpa]	$\tau$ [Mpa]	$\tau$ [Mpa]
1A	6,88	9,16	12,36	5,53
2A	6,93	9,45	11,94	7,14
3A	7,13	9,64	14,06	7,01
4A	6,66	8,82	12,38	6,87
5A	7,60	8,64	14,17	6,62
6A	7,29	9,10	11,22	6,28
7A	6,92	9,84	12,73	6,12
8A	6,95	9,23	12,58	7,18
9A	6,43	9,64	11,00	6,02
10A	6,98	8,20	12,72	5,95
1B	7,36	7,70	11,53	6,03
2B	7,16	8,87	11,95	5,48
3B	7,00	9,59	13,36	7,14
4B	6,58	8,19	11,42	6,71
5B	7,06	8,46	14,74	6,84
6B	7,23	7,76	11,85	6,96
7B	7,14	8,76	11,92	6,82
8B	7,66	9,41	12,67	5,72
9B	7,15	9,56	11,66	5,72
10B	5,68	7,21	12,18	6,92
1C	7,32	6,82	10,64	4,94
2C	7,76	8,11	11,68	5,78
3C	6,87	8,69	7,18	6,65
4C	6,94	7,24	12,73	5,95
5C	7,21	7,64	12,75	5,65
6C	6,76	8,39	11,35	6,54
7C	7,14	7,78	11,90	6,99
8C	6,62	8,48	11,39	7,61
9C	6,50	8,16	10,77	6,01
10C	7,17	7,64	8,52	6,43

Table 2  
CST test results

From test results of Table 2 and 3 it can be deduced that the most important parameter in the lamination process is the humidity storage of the PVB. Beside that these results show until now that the specimen's location on the main plate can be neglected.

The graph of Figure 3 indicates on the other hand that the stiffness in the ultimate limit state is constant with a linear response of PVB and a shear modulus  $G_{PVB} \cong 10$  MPa.

Table 3 shows the collection of test results with average values of adhesion and standard deviation. The high dispersion level of family N-R is due to the high number of glass failures occurred in this case, so the real value of adhesion is greater.

Figure 4 shows the adhesion value as function of the normal probability of failure. The limited number of samples does not allow to define completely each Gauss curve but is

possible to compare the main values of adhesion and the dispersion of results for different parameter of lamination process.

#### Test phase 2: Tests on large plates

Two large samples of glass panes were tested in the Laboratory of the University of Pisa in order to evaluate the creep and relaxation properties of PVB in laminated safety glass. In addition, a destructive test was performed to estimate the post-brakeage behaviour and the capacity of laminated safety glass to bear self weight after partially and totally rupture has occurred.

The specimen was a 2000x2600 mm rectangular glass plate made with two tempered glass sheets 10 mm and 8 mm thick with 1.52mm PVB interlayer.

The restrain devices were 8

countersunk point fixing positioned on small edge only in the lower glass of 10 mm, so the upper glass of 8 mm was free from holes.

Three tests have been performed at a temperature of 30°C by applying increasing loads in a small area around the centre of the pane.

The fourth test was performed at 35 °C. It consisted in artificially breaking the 10 mm glass sheet and observing the post breakage behaviour. Since after 3 days the pane was not yet collapsed, also the other sheet was intentionally broken and the whole pane resisted for other 5 minutes before completely falling down.

Table 4 shows specifies the number of load steps, the value of the applied force and the time [h] of load application:

Figure 6 illustrates vertical displacements vs. time for test number 3.

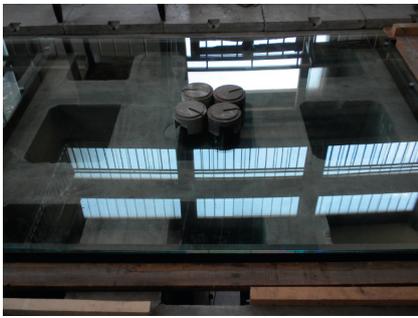


Figure 5  
Experimental phase on large specimen

Test n.	Number of step	F <sub>1</sub> [kN]/h.	F <sub>2</sub> [kN]/h
1	1	0,35 / 24	-
2	1	0,70 / 36	-
3	2	4,10 / 15	6,80 / 24
4	2	Intentional failure	Intentional failure

Table 4  
Test program on large sample

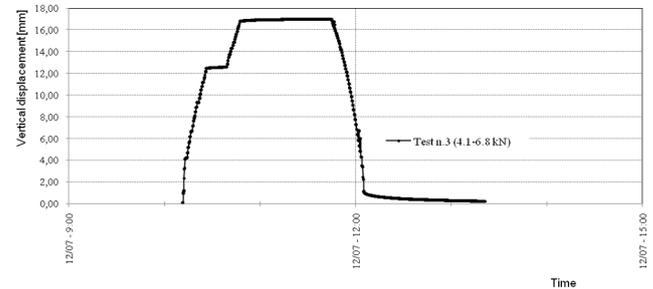


Figure 6  
Test n.3 - Vertical displacement Vs. time

The graph shows how PVB loses at 30°C every viscoelastic property so the displacements are practically constant during time and when the load is removed the recover is instantaneous.

In the fourth test the glass pane was positioned vertically and the 8 mm layer restrained with eight countersunk point fixings was subjected to intentional rupture. The residual capacity to care self weight was exerted by the adhesion of PVB to steel and to the confinement of fragments near the hole. The glass pane resisted for three days with a visible bending but without any catastrophic failure. After that the other 10 mm glass layer was also intentionally broken, it resisted over other 5 minutes (fig.8).

### Numerical analyses

Numerical analyses was performed and compared with the results of the available creep tests of virgin PVB.

The glass layer and PVB material were modeled with 8-node isoparametric elements to evaluate the behavior of PVB and the capacity to transfer shear action between glass layer.

Soda-lime-silica float glass is modeled as a linear-elastic material with a Young's modulus of 70.000 MPa and Poisson ratio of 0.22.

In FEM analysis the rheological behavior of PVB was taken into account by means of the shear relaxation modulus  $G(t)$  represented by a generalized Maxwell series [3]:

$$G(t) = G_{\infty} + \sum_{i=1}^n G_i e^{-t/\tau_i}$$

where  $G_{\infty}$  is the long-time plateau modulus,  $G_i$  the moduli of individual terms in the generalized Maxwell series, and  $\tau_i$  the associated relaxation times. The instantaneous or glassy modulus  $G_0$  is given by:

$$G(0) = G_0 = G_{\infty} + \sum_{i=1}^n G_i \cong \sum_{i=1}^n G_i$$



Figure 7  
Test n.4 - fragmentation near the hole



Figure 9  
Test n.4 - total collapse



Figure 8  
Test n.4 - macroscopic bending

In particular, we have adopted the shear relaxation modulus published by two different PVB producers [4], [5] to study the sensibility of the FEM model when different  $G(t)$  properties of PVB are introduced.

Figure 11 shows that the two trends of the shear modulus versus time are quite similar and it could be concluded that apparently the differences are negligible. Also the effect of temperature was taken into account with shift factor [8].



Figure 10  
Finite Element Model

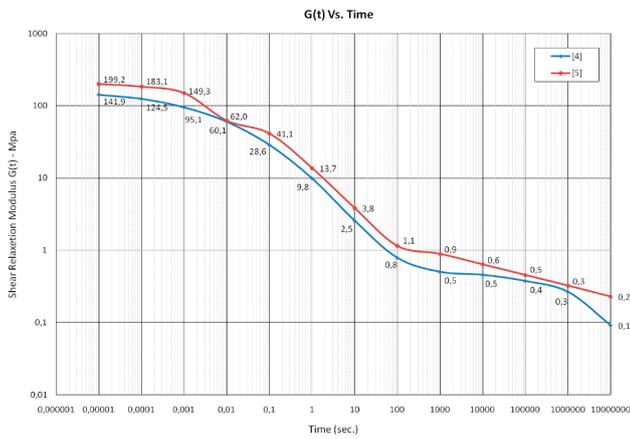


Figure 11  
Master curve of PVB (20°C)

The numerical analyses performed were:

- a linear static analysis to evaluate the secant modulus of PVB,
- a linear quasi-static analysis to evaluate the viscoelastic behavior of LSG,
- a non linear, quasi static-analysis to evaluate the viscoelastic behavior of LSG with large displacements.

Figure 12 shows the good correspondence between numerical and experimental results of test number 3. The graph shows also the importance to perform non-linear analysis with large displacements to simulate the real behaviour of glass plates. These results confirm the good quality of the constitutive model of PVB, so it is possible to apply just shift factors to evaluate the structural behaviour of glass plates at any temperature (fig. 13).

Figure 14 shows the principal stress along the thickness in the middle of the plate for different displacement values.

It is possible to see that for large displacements membrane stresses prevail over flexural stresses as the whole thickness is under traction.

### Conclusions

A Compression Shear Test (CST) programme is presently running at the University of Pisa over a population of 120 LSG specimens divided in 4 groups characterized by different lamination conditions.

The variable parameters are : autoclave temperature and pressure, time of process, storage humidity of the PVB.

The first tests confirmed the importance of the influence of storage humidity on the adhesion property of PVB.

The viscoelastic properties of PVB and post-breakage behaviour of LSG were investigated with tests on large specimen.

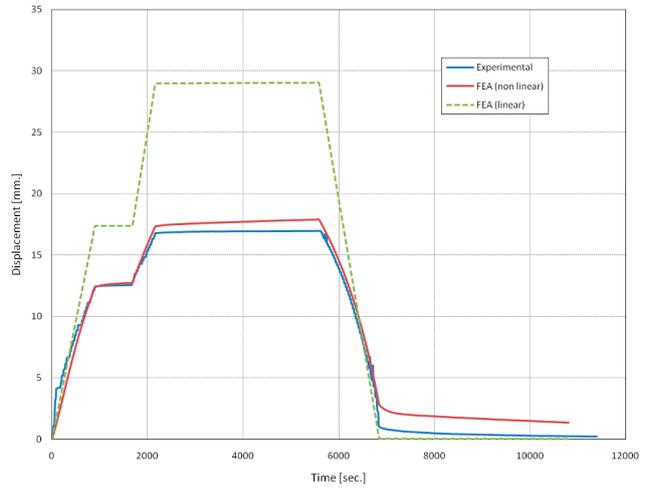


Figure 12  
Displacement vs. Time – Experimental and numerical analysis

Numerical analyses were also performed to evaluate how sensitive numerical simulations are with respect to the implemented relaxation shear modulus  $G(T,t)$  law of PVB.

### Acknowledgements

The research was conducted with the financial support of the Regione Toscana, Programma Operativo Regionale FSE "Competitività Regionale e Occupazione 2007-2013".

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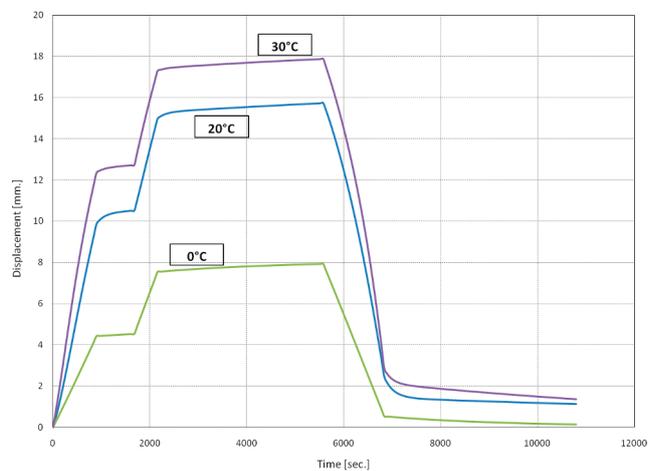


Figure 13  
Displacement vs. Time for different values of temperature.

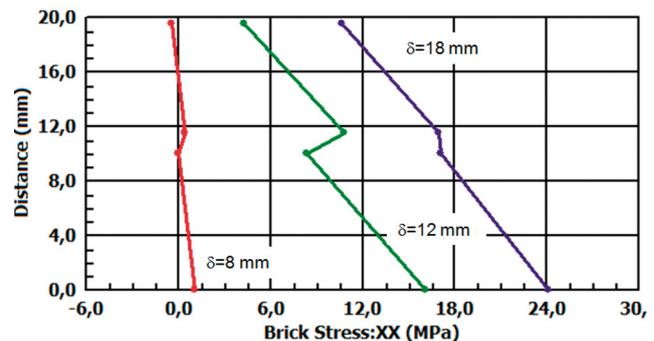


Figure 14  
Principal stress along the thickness.