Adhesion in Laminated Safety Glass – What makes it work?

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Keywords:
1 = Laminated safety glass   2 = Adhesion

Session 8

Abstract

Level and durability of adhesion between PVB-interlayer and glass determine largely the performance of the laminate. Since the adhesion depends on both, the materials and parameters used in the bonding process, some of the most relevant processing steps are reconsidered and related to those mechanisms that are believed to govern adhesion.

Introduction

Laminated Safety Glass (LSG) is a composite material that combines the unique properties of glass with the benefits of a tough but highly elastic organic material. This results in a structure behaving superior in terms of risk of injury by shards, impact resistance and conservation of structural integrity as compared to a monolithic glass panel of identical thickness. In more than 90% of this type of application, the organic interlayer consists of plasticised polyvinylbutyral (PVB), a polymer that on the strength of high adhesive power and excellent mechanical properties is almost perfectly suited for this purpose.

Now, what do the requirements for the product LSG look like in more detail? First of all, they are different according to their use in architectural glazing as opposed to the automotive sector. Whereas for windshields, the aspect of passenger safety should be stressed, long term stability with regard to appearance of visual defects or delamination, obviously will be required from a product with service times of two decades or more in architectural applications. In this sector, the ability of LSG to retain glass fragments, provide residual resistance, limit the size of opening and reduce the risk of injury in case of breakage is usually pointed out.

Within the two domains mentioned above, a number of industry standards lay down the minimum requirements, that should be met. Architectural glazing for example is now covered by EN 12543 and therein, a clear understanding of the required properties of LSG is provided: Expect for visible defects and mismatch of the glass panes, long term stability and human impact test have to be rated. As an extension to this, standards of narrower scope cover more particular applications of LSG, such as overhead glazing for example.

Main frame

What about adhesion? Surprisingly, none of the relevant national or international standards even makes mention of the strength of the adhesive bond, let alone recommends specific values. This fact, however, should not mislead to the conclusion, that adhesion is of secondary importance in the production of LSG. On the contrary, adhesion between PVB interlayer and glass should be appreciated as an essential feature, not only playing a decisive role with regard to durability, but also providing one of the keys to the required safety performance of the LSG unit.

- In the event of breaking, **high adhesion** of PVB to glass ensures, that no dangerous fragments of glass release but instead adhere firmly to the PVB-interlayer. The whole laminate in this case, however, acts as a solid structure, allowing the penetration of objects or fragmentation into several larger parts.
- At **low adhesion** level, LSG exhibits much

higher impact resistance, since the PVB interlayer retains in part its mobility and can be pulled from between the glass panes. As a result, a considerable amount of the impact energy is annihilated by elastic deformation of the PVB. At too low adhesion level, however, retention of glass shards is unlikely to be effective.

Due to this conflicting interdependency *(Figure 1)*, one usually aims at a compromise that in view of impact resistance reliably fulfills all requirements, but does so at sufficiently high adhesion level.

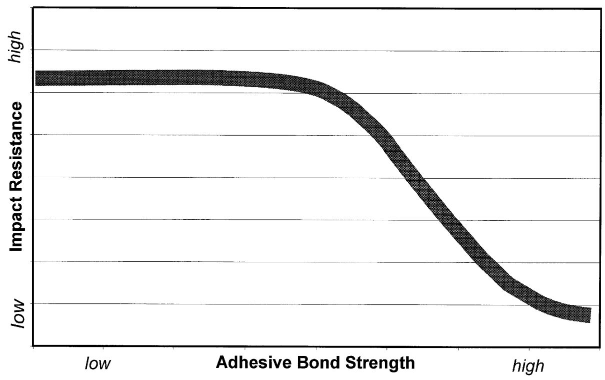

Figure 1. Impact resistance as a function of adhesive bond strength.

Clearly, adhesion should always be adjusted to its optimum level and should be as closely controlled as possible. To this end, the suppliers of PVB furnish their products with different grades of adhesion, that will preserve in the lamination process only, however, if a number of side effects, that potentially influence adhesion are taken into account at the lamination site. Nevertheless, since a number of small effects can severely interfere with adhesion, it is still advisable to monitor adhesion level by means of reliable tests of good predictive value.

How can adhesion be measured?

Although the underlying theoretical concept of adhesion is simple – it is the energy, necessary in order to separate two different materials at their interface – it turns out to be highly impractical, to obtain this energy by direct measurement. Instead, two widely used methods, namely Pummel- and Compressive Shear Test (CST) still prevail in the glass industry for routine adhesion control.

During the **Pummel Test**, a laminate of non-specific size is frozen to minus 18° C (zero degree Fahrenheit) and pummeled with a 1 pound hammer on a steel plate in such a way, that the glass on both sides pulverizes. Subsequently, the sample is visually rated according to a scale from 0 to 10, where 0 corresponds to complete exposure of the PVB interlayer and 10 to 100% coverage of the PVB by glass fragments. The test can be carried out by hand or by custom made apparatus. Still, treatment of the test specimen as well as rating can vary considerably from operator to operator. Therefore it should be stressed, that the Pummel test somewhat lacks from objectiveness.

The **Compressive Shear Test** involves cutting small samples from a laminate, usually 2,54 cm square, and loading them under 45° in a sliding head, that is mounted on a mechanical testing device *(Figure 2)*. The minimum force required to cause failure at the glass PVB interface is measured in units of PSI or N/mm^2. Although the CST gives more objective data as compared to the Pummel, the readings among other factors still depend on thickness and modulus of the interlayer.

The adhesion data obtained by Pummel respectively CST usually show almost linear correlation, provided that measurements are done on a set of laminates, that were fabricated employing the same materials under identical conditions. Since this rarely will be the case in practice, the relation of Pummel rating to CST data is presented only schematically *(Figure 3)*:

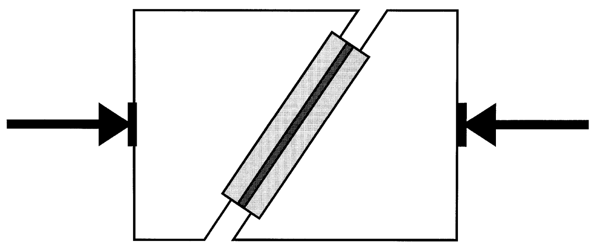

Figure 2. Position of the test specimen in the compressive shear test.

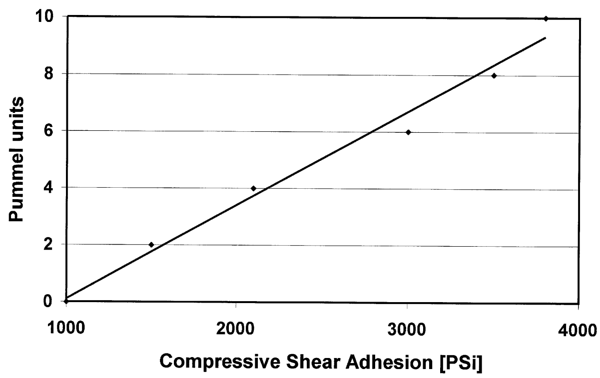

Figure 3. Correlation between Pummel and Compressive shear test.

Why does PVB adhere to the glass surface?

How the adhesive force is affected by different factors can best be explained on the basis of the

Glass Processing Days, 13–16 June '99
ISBN 952-91-0885-0
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mechanisms that govern adhesion on the molecular level. The PVB-interlayer lends its name from its main constituent, the organic resin polyvinylbutyral. This resin can be classified a copolymer, because it consists in two different units, that more or less alternate in the copolymer chain: The non-polar butyral group and the highly polar vinyl alcohol group, highly compatible with water *(Figure 4)*. The plasticizers employed, basically impart the desired flexibility to the sheet and allow to balance mechanical strength and elasticity whereas adhesion remains more or less unaffected.

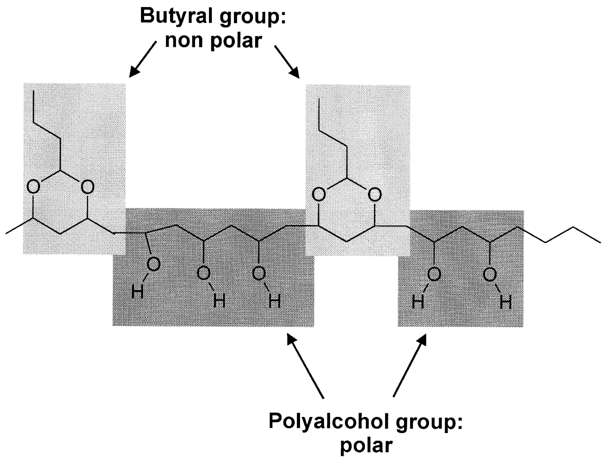

Figure 4. Chemical structure of PVB.

The glass surface on the other hand has to be considered an even more complex chemical system, dominated by polar silanol groups Si-OH that form on the surface through absorption of water with subsequent hydrolytic attack on the Si-O-Si bond. Moreover, the glass surface can be covered to a considerable degree with water or any other compound that has been in contact with it.

If brought into close proximity, the chemically similarly behaving alcohol groups of PVB and the silanol-groups on the surface of the glass combine to form a reversible but dense network of weak hydrogen bonds and beyond this point to some extent true chemical bonds. In order to do so, the PVB must be allowed to flow and adopt a shape that perfectly fits the microscopically rough surface encountered on the glass *(Figure 5)*.

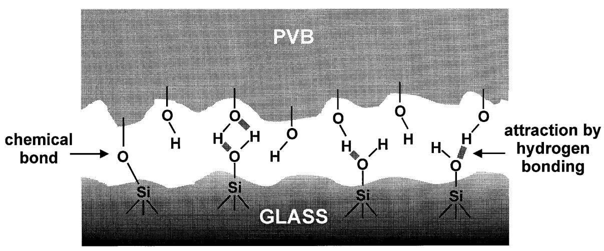

Figure 5. PVB-glass bonding mechanism.

In the autoclave at given pressure and hold time, this process calls for sufficient high temperatures in order to complete. The maximum adhesion level as determined from case to case by glass and PVB-type, will then safely be achieved. *Figure 6* illustrates this dependency of adhesive bond strength on the autoclave temperature:

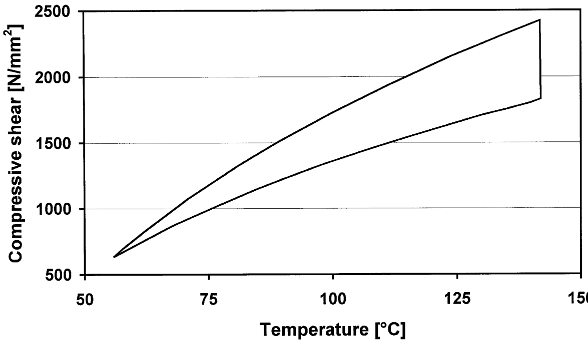

Figure 6. Compressive shear adhesion versus autoclave temperature.

What influences adhesion?

It is widespread knowledge that the properties and the treatment of the glass as well as bad conditioning of PVB can adversely affect adhesion in the laminate. Among the many factors that interfere with adhesion in PVB-glass laminates, two can be pointed out for being observed more frequently than the others:

- Glass composition
- Air versus tin side, the latter usually lower in adhesion
- Corrosion of the glass surface
- Contaminants on the surface
- Washing process and quality of water used therein
- Composition and type of PVB
- Moisture content of PVB

Why can residual salts reduce adhesion?

Since ionic contaminants present at the PVB-glass interface in most cases will interfere with the bonding mechanism as summarized above, salts can cause large deviations in adhesion, when involuntarily applied in the washing step. If the washing/rinsing machine operates with water heavily loaded with salts, adhesion may even drop by several pummel units. This relationship is shown in *Figure 7*, where the gross amount of salts dissolved in the rinsing water – commonly alkaline earth and alkaline metal – is expressed in units of ionic conductivity.

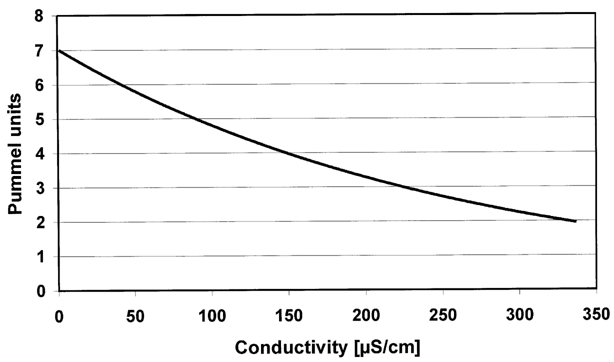

Figure 7. Pummel adhesion against conductivity of rinsing water.

Alkaline metals like potassium, when located at the PVB-glass interface exhibit a tendency to attract residual moisture from the sheet. This eventually leads to clustering of water between the alcohol groups of PVB and their silanol counterparts, resulting in weakening of the adhesive bond. It is this effect, that voluntarily can be used to adjust adhesion to lower levels and is widely exploited in formulating automotive grade PVB.

A much more pronounced reduction is observed however, if alkaline earth metals like the omnipresent calcium come into play *(Figure 8)*. They probably can interact directly with the alcohol groups of the PVB, rendering them unavailable for bonding. Therefore, at least the rinsing step should employ demineralized water instead of tab or industrial water, that usually carry minerals to some degree.

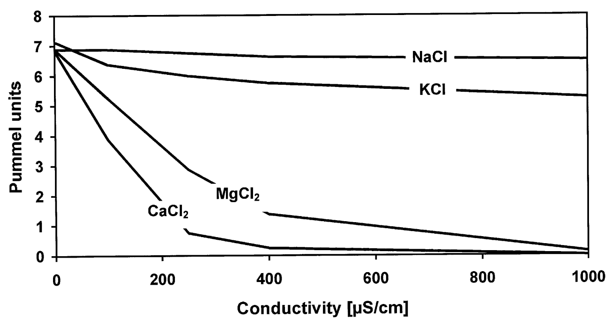

Figure 8. Compressive shear as a function of metal ion concentration.

How does moisture in the sheet reduce adhesion?

For its significant proportion of polar alcohol groups, PVB is a hygroscopic material, that eagerly tries to increase its water content when exposed to humidity. Whereas the recommended sheet moisture content in the laminate should vary around 0.4%, in the sheet itself, it can rise to 2% under unfavorable conditions. Since water, a highly polar compound itself, competes for bonding sites available on the glass surface, a progressive reduction of adhesive bond strength is observed with increasing moisture *(Figure 9)*.

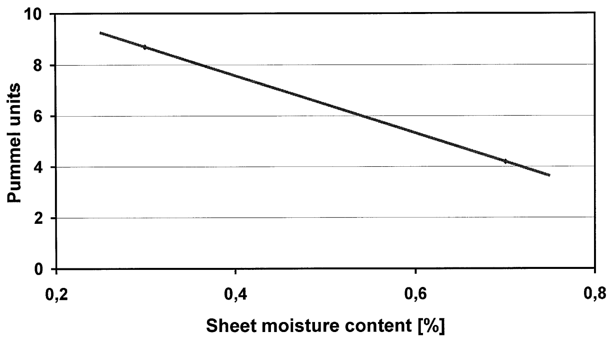

Figure 9. Pummel adhesion versus sheet moisture content.

In order to prevent adhesion problems arising from excessive moisture, it is adjusted to a low level and constantly monitored during manufacture of the PVB sheet. Subsequently, PVB stores and ships tightly sealed against humidity. At the laminators site however, the PVB should be stored and handled in a conditioned cabinet and should not be exposed to high humidity environments anymore, when not properly sealed. On the contrary, moisture level can change noticeable in the sheet within a couple of hours only, approaching a saturation level that depends on PVB grade and the relative humidity in the surrounding *(Figure 10)*.

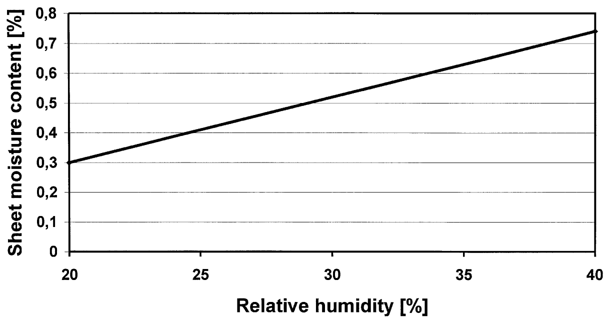

Figure 10. Sheet moisture content against relative humidity.

Conclusion

It becomes obvious that the bonding mechanism of PVB to the glass surface is highly complex. This is the reason, why minor variations in PVB conditioning, quality and treatment of glass potentially cause big changes in adhesion. For the production of high quality LSG, an understanding of the factors that affect the adhesive bond strength in the laminate should prove beneficial.

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