

Metal-to-glass bonding properties of an acrylate adhesive (DELO GB368) and an ionoplast interlayer (SentryGlas) at 23, -20 and 60°C.

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Abstract

The metal-to-glass bonding properties of an acrylate adhesive (DELO PHOTOBOND GB368) and an ionoplast interlayer (SentryGlas) have been investigated by a series of 'pull-out' tests, performed at 23, -20 and 60°C, on small glass laminates with a metal insertion. The specimens consisted of three layers of glass with a small stainless steel box section adhesively bonded or laminated in between. The test results showed a temperature dependency of both bonding systems. The acrylate adhesive showed high bond strength at 23°C and a reduction in bond strength at -20°C and 60°C of 45% and 55% respectively. The SentryGlas interlayer showed high bond strength at 23°C and a reduction in bond strength of 60% at 60°C, whereas no significant change in bond strength between the 23°C and -20°C specimens has been observed. However, both at 23°C and -20°C the specimens might not have been tested to full bond strength, since their strength might have been limited by the limited tensile capacity of the metal insertion, which started to contract, thereby loading the bond in an unfavorable manner. The results showed high bond strength of both bonding systems. A direct comparison between both systems, however, could not be made since the SentryGlas specimens might have profited from a geometrical advantage.

Introduction

Adhesive bonds or lamination techniques offer the possibility of joining metal to glass in a fully transparent manner without using any mechanical connection. Due to the visco-elastic properties of most adhesives and interlayers, however, their bond strength might vary at different temperature levels.

To investigate the temperature dependency of different bonding systems, a series of 'pull-out' tests has been performed at 23°C (room temperature), -20°C, and 60°C. Small three-layered glass laminates with a metal insertion bonded between both

outer layers have been loaded in tension to determine the bond strength of the specific bond system. Two bond systems have been tested; a UV-curing acrylate adhesive (DELO PHOTOBOND GB368) and an ionoplast interlayer (SentryGlas or SG). From the properties listed in table 1 it can be seen that the acrylate adhesive has a relatively high glass transition temperature of 102°C compared to the glass transition temperature of the ionoplast interlayer which amounts ~55-60°C. This might result in a more constant bond strength of the acrylate adhesive at the tested temperature range of -20 to 60°C than the ionoplast interlayer.

The bonding systems have been selected in concordance with the ongoing research at Delft University of Technology (TU Delft) on metal reinforced glass beams [1]. Both the DELO acrylate adhesive and the SentryGlas interlayer are promising techniques for bonding the metal reinforcement to the glass beam. Parallel to the 'pull-out' tests, also bending tests have been performed at different temperature levels on metal reinforced glass beams, in which the metal reinforcement was bonded to the glass using either the DELO acrylate adhesive or the SentryGlas interlayer. The bending tests showed promising results for both bonding systems and are presented by Louter et al. in [4] for the DELO acrylate bonded beams and at this conference in [5] for the SentryGlas laminated beams. Besides the bond systems also the shape and size of the metal insertion has been selected in concordance with the metal reinforcement commonly applied in the metal reinforced beams. This way the

pull-out tests generate valuable data for the interpretation of the structural response of the metal reinforced beams.

Pull-out specimens

The pull-out specimens consist of small glass plates (2 outer glass layers of 10*100*100 mm and 2 inner glass spacers of 10*40*100 mm or 10*45*100mm) bonded together with a small stainless steel box section (10*10*1 mm), see figures 1 and 2. Within the box section a small circular steel section is inserted to prevent compression of the box section when clamped in the test setup.

The metal insertion is only bonded to both outer glass layer, and not to the inner glass spacers, so only a two-face bond is generated. Both the glass-to-glass and the metal-to-glass bonding have been executed using either the DELO acrylate adhesive or the SentryGlas interlayer. For each bonding system a series of 9 specimens has been manufactured.

The acrylate bonded specimens have been manufactured using a semi-automated production process in which the specimens are guided through two pressure rollers before the adhesive is cured by UV-light. This process guarantees an equal adhesive thickness for all specimens of < 0.1 mm. For the adhesively bonded specimens a gap has been made between the inner glass spacers and the metal insertion to prevent accidental bonding of the glass spacers to the metal insertion, see figure 3.

The laminated specimens have been manufactured using the vacuum bag

| Property | Unit | DELO GB368 | SentryGlas |
|-------------------------------|-------------------|------------|------------|
| Tensile strength | N/mm ² | 20 | 34.5 |
| Elastic modulus | N/mm ² | 900 | 300 |
| Glass-aluminum shear strength | N/mm ² | 23 | unknown |
| Glass transition temperature | °C | 102 | ~55-60 |
| Elongation at tear | % | 17 | 400 |
| Density | kg/m ³ | 1000 | 950 |

Table 1:
Material properties of DELO PHOTOBOND GB368 [2] and SentryGlas [3].

lamination technique. For all specimens a SentryGlas interlayer with a thickness of 1.52 mm has been applied. To facilitate the manufacturing process, no gap has been made between the inner glass spacers and the metal insertion, see figure 3.

The specimens bonded with the DELO acrylate adhesive will be referred to as acrylate-specimens or 'ACR-specimens' and the laminated specimens will be referred to as SentryGlas-specimens or 'SG-specimens'.

Test setup at 23, -20 and 60°C

The pull-out tests have been performed at a Zwick Universal 100 kN test machine, which has been provided with a specially devised steel bracket to host the pull-out specimens, see figure 4. The metal insertion has been clamped in the lower bracket and the upper steel bracket containing the glass laminate has been moved upwards by the crosshead at a rate of 2 mm/minute. This way the metal insertion is pulled out of the glass laminate.

For the pull-out tests at -20 and 60°C an insulated climatic chamber has been put around the test setup, see figure 5. This climatic chamber has been either cooled with vaporized liquid nitrogen or heated with an electric heating element. A fan at the back side of the climatic chamber generated an air flow throughout the climatic chamber which ensured an even temperature level throughout the climatic chamber.

Prior to the pull-out tests the specimens have been conditioned for several days. The specimens tested at room temperature have been conditioned for 1 week at 23°C (±1°C) in the same room as the test setup. The specimens tested at -20°C have been conditioned for 1 week at -23°C (±1°C) in an ordinary fridge. The conditioning temperature has been selected 3 degrees lower than the testing temperature to compensate for any heat gain during the mounting of the specimens in the test setup, which took about 2 minutes per specimen. The specimens tested at 60°C have been conditioned for 5 days in an oven at 63°C (±1°C); again with an additional 3 degrees to compensate for any heat loss during the mounting of the specimens in the test setup.

During the tests the inflicted force and the displacement of the crosshead has been measured.

Test results

Figure 6 shows both the force-displacement diagrams and the numerical results of the pull-out tests performed at 23, -20 and 60°C. The indicated displacement in the force-displacement diagrams corresponds with the displacement of the crosshead (see figure 4) and includes both the strain of the specimens and the strain

Figure 1:
Exploded and assembled view of the pull-out specimens.



Figure 2:
The pull-out specimens consist of several small glass pieces and a metal insertion bonded together.

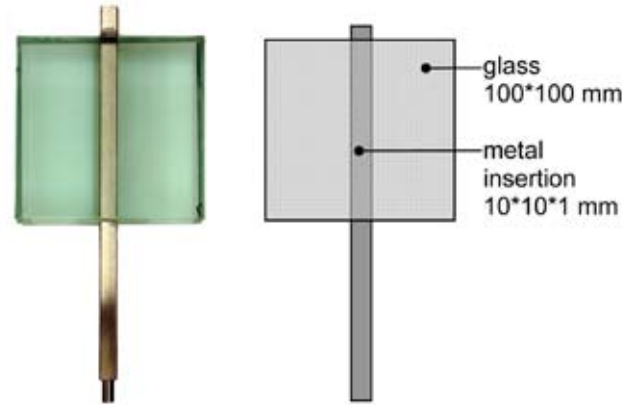


Figure 3:
Cross section of the pull-out specimens.

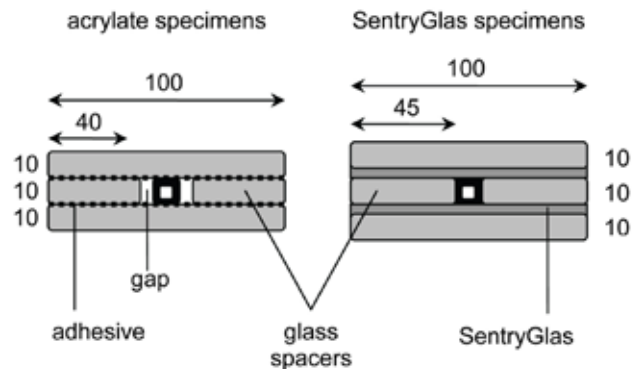


Figure 4:
Pull-out test setup at 23°C (room temperature).

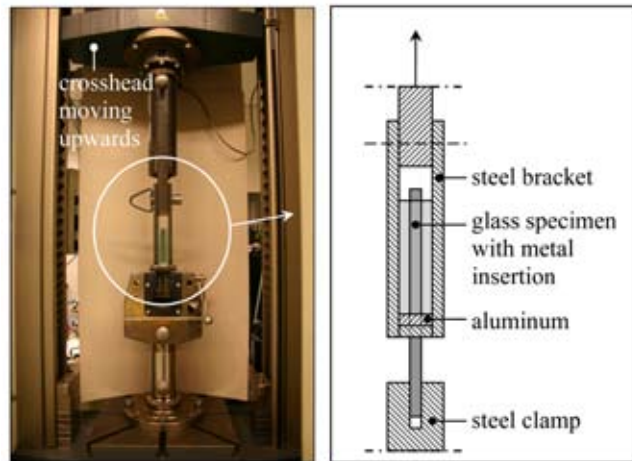
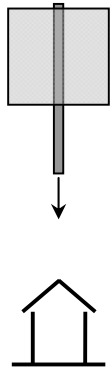


Figure 5:
A climatic chamber has been put around the test setup to either cool to -20°C (left) or heat to 60°C (right).

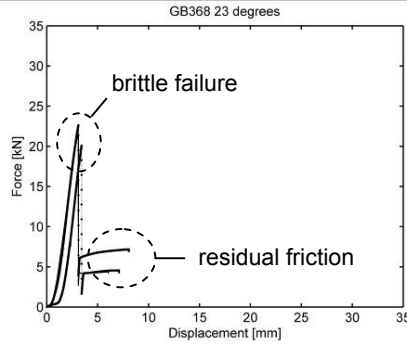


DELO GB368 acrylate specimens

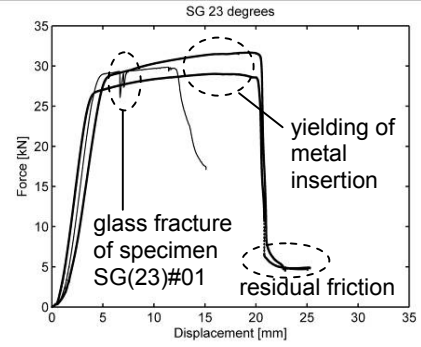
SentryGlas specimens



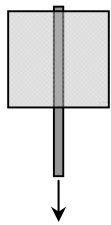
23°C



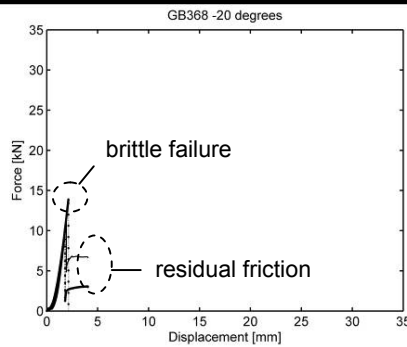
| specimen | max.force | |
|--------------|-----------|----|
| ACR (23) #01 | 21.3 | kN |
| ACR (23) #02 | 22.6 | kN |
| ACR (23) #03 | 20.1 | kN |
| average | 21.3 | kN |
| S.D. | 1.3 | kN |
| rel. S.D. | 6.0 | % |



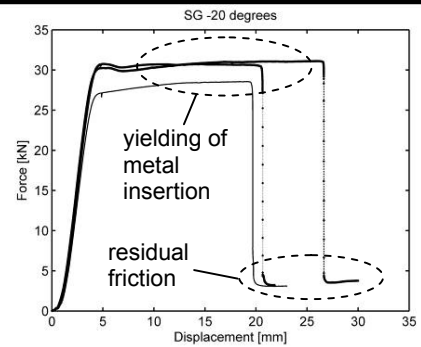
| specimen | max.force | |
|-------------|-----------|----|
| SG (23) #01 | 29.9 | kN |
| SG (23) #02 | 29.0 | kN |
| SG (23) #03 | 31.7 | kN |
| average | 30.2 | kN |
| S.D. | 1.3 | kN |
| rel. S.D. | 4.5 | % |



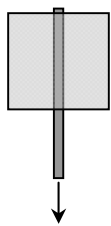
-20°C



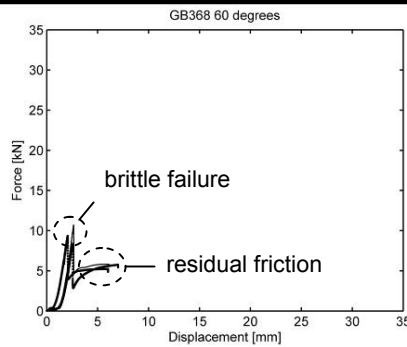
| specimen | max.force | |
|---------------|-----------|----|
| ACR (-20) #01 | 11.3 | kN |
| ACR (-20) #02 | 10.3 | kN |
| ACR (-20) #03 | 13.9 | kN |
| average | 11.8 | kN |
| S.D. | 1.8 | kN |
| rel. S.D. | 15.6 | % |



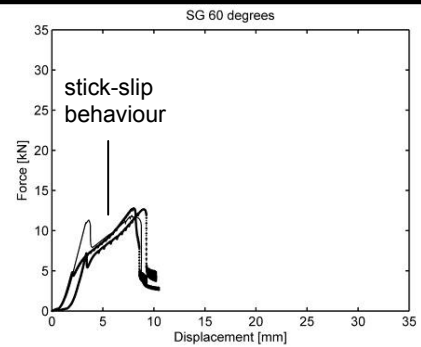
| specimen | max.force | |
|--------------|-----------|----|
| SG (-20) #01 | 28.6 | kN |
| SG (-20) #02 | 31.1 | kN |
| SG (-20) #03 | 30.8 | kN |
| average | 30.1 | kN |
| S.D. | 1.4 | kN |
| rel. S.D. | 4.6 | % |



60°C



| specimen | max.force | |
|--------------|-----------|----|
| ACR (60) #01 | 10.7 | kN |
| ACR (60) #02 | 9.3 | kN |
| ACR (60) #03 | 8.4 | kN |
| average | 9.5 | kN |
| S.D. | 1.2 | kN |
| rel. S.D. | 12.6 | % |



| specimen | max.force | |
|-------------|-----------|----|
| SG (60) #01 | 11.9 | kN |
| SG (60) #02 | 12.7 | kN |
| SG (60) #03 | 12.8 | kN |
| average | 12.4 | kN |
| S.D. | 0.5 | kN |
| rel. S.D. | 3.8 | % |

Figure 6: Force-displacement diagrams and numerical results of the pull-out tests.

of the test setup itself. The results will be discussed by temperature level in the next paragraphs.

Results at 23°C

At the beginning of the loading procedure the acrylate specimens showed no adhesive failure and the part of the metal insertion sticking out of the specimen started to elongate in a linear elastic manner. At an average force of 21.3 kN, however, brittle failure of the adhesive bond occurred, which caused slip of the metal insertion within the glass specimen. The load dropped to a residual friction load of about 5 kN, see figure 6, which was needed to pull the metal insertion further out of the glass. Since the strength of the specimens did not significantly increase anymore the loading procedure was stopped at a displacement of 5-8 mm. The acrylate specimens were removed from the test setup and inspected. Except for specimen ACR(23)#02, which showed a linear crack along one bond line (see figure 7), no cracking of the specimens was observed. All acrylate specimens showed white discoloration of the adhesive bond line which indicates deformation of the adhesive.

Also the SG-specimens showed linear elastic response at the beginning of the loading procedure without showing any slip of the metal insertion, until at a load of about 26 to 28 kN the response became more ductile. At this loading level the applied force approached the tensile strength of the metal insertion and the metal insertion started to yield and contract. For specimens SG(23)#02 and #03 the applied force showed only limited increase until at a displacement of 22 mm the metal insertion started to slip, see figure 6. At this displacement level the load suddenly dropped to a residual friction load of 5 kN, which was needed to pull the metal insertion further out of the glass laminate. Visual inspections of the specimens showed debonding of the metal insertion, stretching of the SG-interlayer and some minor cracks in the inner glass spacers, see figure 8 (left). The response of specimen SG#01 was different since it showed severe cracking at a load 29 kN, see figures 6 and 8 (right). This cracking did not severely affect the load carrying capacity of the specimens, and after a small drop the load further increased, see figure 6. Subsequent cracking of both glass spacers, however, caused slip of the metal insertion and a drop in load.

Results at -20°C

Similar to the specimens tested at room temperature the acrylate specimens tested at -20°C showed brittle failure of the adhesive bond and a residual friction strength of about 2 to 7 kN. The strength of the acrylate specimens was, however, decreased with 45% compared to the specimens tested

Figure 7:
Specimen ACR(23)#02 tested at room temperature shows a linear crack along one bond line.

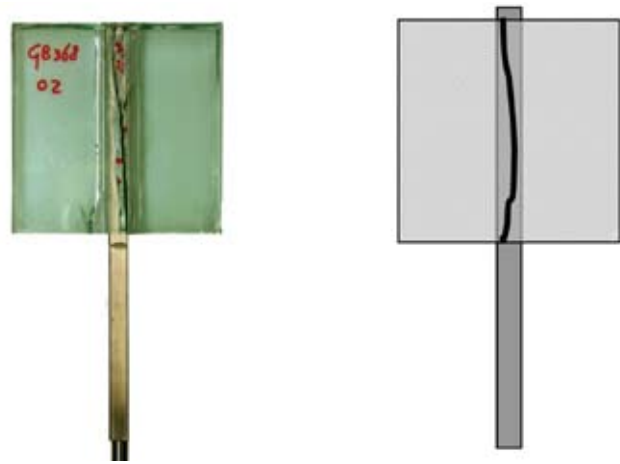


Figure 8:
Specimen SG(23)#02 shows only minor cracks in the inner layer, whereas specimen SG(23)#01 shows severe cracking. Both specimens have been tested at room temperature.



Figure 9:
Specimen ACR(-20)#02 shows a linear crack (left) and specimen SG(-20)#01 shows more severe cracking.



at room temperature (from average 21.3 to 11.8 kN). Specimen ACR(-20)#02 showed a similar linear crack at one bond line as was observed for an acrylate specimen tested at room temperature, see figure 9 (left), whereas the other specimens showed no cracking at all.

The SG-specimens showed similar response at -20°C as at room temperature. After a linear elastic response without any slip of the metal insertion, again a ductile behaviour was observed which was caused by yielding and contraction of the metal insertion.

The specimens showed some glass cracking along the bond line of the metal insertion. This cracking was more severe at specimen SG(-20)#01, which showed cracking of the outer glass layer, see figure 9 (left) than at the other two SG-specimens tested at -20°C, which showed some cracking of the inner glass spacers. On average the SG-specimens showed the same strength as the specimens tested at room temperature; namely 30.1 kN compared to 30.2 kN at room temperature. Visual inspection of the specimens showed debonding of the interlayer at the metal insertion and

stretching of the interlayer between the outer glass layers and the inner spacers.

Results at 60°C

Also at 60°C the acrylate specimens showed brittle failure and residual friction strength of about 5 kN. Compared to the tests at room temperature, however, the strength has decreased with 55% (from 21.3 kN to 9.5 kN). Only specimen GB368#02(60) showed some minor glass cracking.

The response of the SG-specimens tested at 60°C significantly changed compared to the room temperature tests. The average bond strength decreased with almost 60% compared to the tests at room temperature (from 30.2 to 12.4 kN). Furthermore, after an initial linear elastic response the stiffness of the specimens reduced and the specimens showed stick-slip behaviour. This stick-slip behaviour is shown in figure 10, which shows an enlarged force-displacement diagram of the tests at 60°C. The hick-ups in the curve indicate slip of the metal insertion followed by an instant regenerated bond/friction. However, at an average load of 12.4 kN and a displacement of about 10 mm the bond fails and the load drops to a residual friction load of about 5 kN. No glass cracking has been observed for the SG-specimens tested at 60°C.

Discussion

The pull-out tests performed at 23, -20 and 60°C showed a temperature dependency of the metal-to-glass bond strength of both the DELO acrylate adhesive and the SentryGlas (SG) interlayer. The specific response of the bonding systems, however, differed.

The bond strength of the acrylate adhesive is highest at room temperature, but decreases with 45% at -20°C and with 55% at 60°C, despite its relatively high glass transition temperature of 102°C. Compared to the acrylate adhesive the SG-interlayer has a lower glass transition temperature of ~55-60°C. Around this temperature level the properties of the SG-interlayer change and its stiffness reduces. As can be seen from the test results also the bond strength of the SG-interlayer decreases at 60°C; namely with almost 60% compared to the bond strength at room temperature. However, no significant change in bond strength between the specimens tested at -20°C and 23°C has been observed. This might have been the result of the limited tensile capacity of the metal insertion, which probably caused premature failure of the metal-to-glass bond. Both at 23 and -20°C the applied force approached the tensile strength of the metal insertion (about 32 kN, see figure 11), which started to yield and contract. Contraction of the metal insertion probably caused additional forces perpendicular to the SG-interlayer,

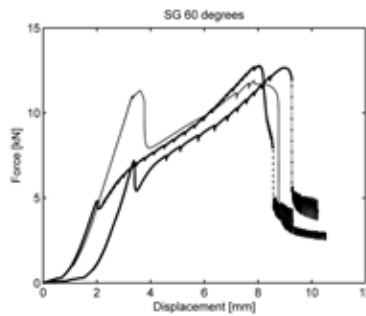


Figure 10:
Enlarged force-displacement diagram of the SG-specimens tested at 60°C.

which are highly unfavourable for the SG-bond. Due to these forces the bond failed prematurely and the shear bond strength of the SG-interlayer was not tested to the maximum. Additional tests at 23 and -20°C on specimens with a stronger metal insertion will be necessary to determine whether the bond strength of SG changes between 23°C and -20°C.

For some pull-out specimens glass fracture occurred during the tests. If glass fracture occurred for the acrylate specimens it occurred only in the outer glass layers to which the metal insertion was bonded. In those cases the glass fractured due to high stresses caused by the local transfer of (shear) forces through the adhesive bond. For the SG-specimens, however, glass fracture also occurred at the inner glass spacers. This seems a bit curious since the metal insertion should only transfer forces to the outer glass layers, as it has not been laminated to the inner glass spacers. However, it seems likely that during the lamination process the SG-interlayer melted and seeped between the metal insertion and the inner glass spacers, thereby creating a local bond between the metal and the glass. During the tests this bond was stressed and the inner glass spacers cracked. This additional bond area might have increased the strength of the SG-specimens. Since the acrylate bonded specimens did not profit from this geometrical advantage, a direct comparison in strength between the acrylate bonded and SG-laminated specimens could not be made.

Remarkable aspect is that all specimens, regardless of bonding type or temperature level, show residual friction strength after bond failure. This friction is probably generated by an interlocking effect of the failed adhesive or interlayer remainders.

The results of the pull-out tests will be implemented in the ongoing research at TU Delft on metal reinforced glass beams. Furthermore, the tests results will be implemented in finite element studies to investigate the effect of the bond layer on the structural response of a reinforced glass beam.

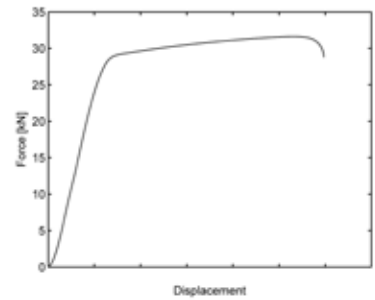


Figure 11:
Force-displacement diagram of a tensile test on the metal insertion.

Conclusions

From the pull-out tests, which have been performed at 23, -20 and 60°C on small glass laminates with a metal insertion, it is concluded that the bond strength of both the acrylate adhesive (DELO PHOTOBOND GB368) and the SentryGlas (SG) interlayer is dependent on temperature level. Compared to room temperature (23°C) the strength of the acrylate adhesive decreases with 45% at -20°C and with 55% at 60°C. For the SentryGlas interlayer a reduction in bond strength of 60% at 60°C compared to its bond strength at room temperature has been observed. However, no significant change in bond strength between the 23 and -20°C specimens has been observed. This might have been caused by the limited tensile capacity of the metal insertion, which started to contract, causing premature failure of the SG-bond both at 23°C and -20°C.

Both bonding systems showed significant bond strength. A direct comparison, however, could not be made since the SentryGlas-specimens might have profited from an unintentional geometrical advantage.

Acknowledgements

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