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Broad screening of adhesives for glass-metal bonds

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Keywords
1=Adhesive  2=Screening  3=Glass-metal bond  4=Lap shear test  5=Humidity exposure

Abstract
Two archetypes (more specifically a glass sheet on a circumferential frame and a circular point fixing) of adhesively bonded glass to metal connections are subjected to extensive research in a two-year project run by Ghent University in cooperation with the Adhesion Institute of Delft University of Technology.

The main objective of the project is to facilitate innovations by providing technology transfer on adhesively bonded glass to metal connections to the building industry. Consequently, the project is coached by a varied group of industrial partners, which has specified five relevant applications and appropriate performance criteria to be further investigated.

Over ten adhesive manufacturers and suppliers have been approached to suggest adhesives for these applications. Of the 40 suggested adhesives, about 30 have been subjected to experimental testing in a broad screening to be able to select adhesives for further detailed investigations. This way, also adhesives which usually are outside the scope of glass research could be investigated, such as methacrylates. Furthermore, each major adhesive family is featured: silicones, polyurethanes, MS-polymers, acrylates, and epoxies.

The screening consists of lap-shear tests on three sets of specimens for each adhesive. The first set is tested without conditioning. The other sets were tested after 4 weeks storage at 90 °C, 50 % RH and 2 weeks of storage at 90 °C, 50 % RH.

The proposed paper summarises the experimental results of the screening research. Adhesives are proposed for further detailed research.

Introduction
This contribution reports on progress made in a joint research project at Ghent University (BE) and Delft University of Technology (NL) on the use of adhesives in structural bonds between glass and metal [1, 2]. In general, the project aims at transferring technical knowledge from academics to industry in order to facilitate innovation related to glass to metal bonds.

Consequently, the project is not only supported by the Agency for Innovation by Science and Technology in Flanders (IWT), but also by a board of industrial partners to whom is reported.

Four large phases can be distinguished in the project. The first phase consisted mainly of determining the exact needs and according performance requirements relevant to the industrial partners. Consequently, the main serviceability parameters and performance requirements of five typical applications (cases) have been determined. In short, the latter can be subdivided in two linearly bonded cases (to be constructed in workshop conditions and on site, respectively) and three adhesive point fixings (exposed to outdoor conditions, located at the inside of a façade, or exposed to ambient conditions only, respectively). Additionally, by means of an extensive literature research and by interviews with manufacturers and distributors of adhesives, a preliminary selection was made of 32 adhesives which are potentially suitable for the defined cases.

Subsequently, in the following phases the performance criteria determined in phase 1 are used as a basis to narrow down the number of adhesives to be investigated experimentally. The general idea during the experiments is to start with many adhesives (32) and a limited number of different tests (1) (this is phase 2), and to reduce the number of adhesive products (initially to 14, then 10, and finally 5) while increasing the number of additional tests (this is phase 3).

Finally, phase 4 of the project will be dedicated to dissemination of the results, towards industry and education.

The current contribution presents the results of phase 2, in which the number of different adhesives is reduced drastically based on the results of a well-chosen experimental program.

Adhesives and substrates
Consequent to a previous phase in the research project, 32 adhesive products have been selected for further investigation in the broad screening. A general overview of the adhesive types is listed in Table 1. As this phase of the research intended mainly to further decrease the number of adhesives by about 50 %, only two substrates were used: polished annealed soda-lime silicate glass and anodized aluminum type EN AW 6060 T6. Both substrates did undergo a precise cleaning of the bonding surfaces with isopropanol and clean cloths just before applying the adhesive, and for a few products an additional specific pretreatment was performed according to the product datasheets.

Test specimens
In a first step, 800 mm long samples were prepared in lab conditions by bonding 800*80*5 [mm] aluminium plates to 800*30*10 [mm] glass strips, with a lateral overlap of typically 5 or 10 mm. The thickness of the bond was adjusted to the requirements mentioned in the product datasheet of each adhesive individually, and ranged from about 0.3 mm (e.g., for some acrylics) to 4 mm (e.g., for some silicones).

Subsequently, after curing of the adhesives the 800 mm long samples were further cut down laterally to the actual test specimen size of about 25 mm wide using a water jet cutter. This way, about 30 specimens could be obtained from one sample, resulting in approximately 960 test specimens in total. Obviously, production tolerances caused the actual specimen size to deviate from the nominal size. Consequently, the actual bonding surface, characterized by the geometrical parameters indicated in Figure 1, was measured precisely for all specimens using an optical microscope.

<table>
<thead>
<tr>
<th>Type</th>
<th>Number of adhesives tested</th>
</tr>
</thead>
<tbody>
<tr>
<td>Silicones</td>
<td>8</td>
</tr>
<tr>
<td>MS-polymers</td>
<td>4</td>
</tr>
<tr>
<td>Polyurethanes</td>
<td>8</td>
</tr>
<tr>
<td>Acrylates</td>
<td>9</td>
</tr>
<tr>
<td>Epoxies</td>
<td>6</td>
</tr>
<tr>
<td>Isocyanates</td>
<td>1</td>
</tr>
</tbody>
</table>

Table 1: general overview of adhesive types

1=Adhesive  2=Screening  3=Glass-metal bond  4=Lap shear test  5=Humidity exposure
Exposure of the test specimens

As the objective of the test was to narrow down the number of remaining adhesives in the test program based on their performance with respect to a single test, the latter should impose rather critical conditions. Consequently, based on previous experiences, it was chosen to expose all specimens in a climate chamber to maintain 90% relative humidity at 50°C for zero, four and twelve weeks, respectively. Even if it is not possible to directly correlate these conditions to "real" conditions, it was reasonable to expect that for the test geometry chosen, all adhesives had reached full moisture saturation after twelve weeks (or even less) exposure time.

From every sample of about 30 specimens per adhesive, one series of seven specimens was mechanically tested immediately (corresponding to zero weeks exposure time), whereas two series of seven specimens were exposed simultaneously in a climate chamber. After four weeks, a first series was extracted from the climate chamber and mechanically tested, while the other series remained in the chamber until the end of the twelve weeks. The remaining unexposed specimens were stored for eventual back-up testing.

Mechanical testing

The specimens were designed for application in a lap shear test using a Zwick 250 kN universal electromechanical test machine. As can be seen in Figure 2, the aluminium was clamped directly in the machine, whereas the glass was positioned in a secondary device enabling a compressive force transfer to the glass. To reduce eccentricities to the minimum, the machine was adjusted in such a way that the line of forces was situated in the middle of the adhesive bond.

Table 2 Mean results on series of seven identical lap shear tests per adhesive after zero, four and twelve weeks exposure to 90% relative humidity at 50°C
All lap shear tests were performed at a constant test velocity, which was chosen in function of the stiffness and thickness of the adhesive and amounted to 1, 2 or 5 mm/sec. The data registered during testing included failure load, displacements of the machine crossheads, test temperature and relative humidity of the testing lab.

**Results**

The mean value of the maximum shear stress out of seven lap shear tests in every series and the corresponding variation coefficients are listed in Table 2 per adhesive and per exposure time, according to the conditions mentioned above. In addition, in the last two columns the shear strength difference after four or twelve weeks is given as the relative gain (positive values) or loss (negative values), compared to the initial (zero weeks) strength values.

**Discussion**

As is beyond the scope of this contribution to present all results in detail, the following discussion will focus on general observations and remarkable deviations to generally observed trends.

As expected, global differences can be detected when comparing the resulting strengths of different adhesive types. For instance, epoxies and acrylates are in general significantly stronger than silicones. However, at first sight a remarkable low value is found for VHB-B23F. Although the bonding is indeed realised by means of an acrylic, the latter product is a two-sided adhesive tape which typically failed at the foam of the tape, not at the bond itself.

In general, most of the tested adhesives tend to have a decreasing shear strength value after exposure (four and/or twelve weeks) compared to unexposed specimens (zero weeks). In spite of high strength values at zero weeks of exposure, some adhesives, such as Araldite 2048 and 2029, even turned out to maintain no residual resistance to the test conditions whatsoever, as they fell apart during water jet cutting or in the climate chamber without any additional loading. However, for several adhesives, such as Araldite 2015 or Sikasil SG500, a significant increase of the shear strength was observed compared to the initial strength (zero weeks). Probably this is due to the relatively high temperature during exposure in the climatic chamber, which inhibited hardening of the material.

**Selection of adhesives for further investigation**

Based on an analysis of the experimental results, 14 out of 32 adhesives are selected for successive in-depth applied research. Obviously, all selections are made in the context of the five application cases determined before.

One of the overall objectives is to select one promising adhesives for every case and to build according prototypes.

In the current phase, major criteria for selection are strength value, sensitivity to moisture (after four or twelve weeks of exposure), and failure mode. With respect to the latter, typical modes across the experiments include cohesive failure of the adhesive, adhesive failure at the glass or aluminium, substrate failure (glass breakage), or combinations. Usually, cohesive failure modes are preferred, because for a certain loading type they are fully determined by the material properties of the adhesive only. However, adhesive failure is not automatically rejected in this investigation, as it should always be evaluated taking into account the variation coefficient and the design stress values in the specific applications considered here. In other words, adhesive failure modes may be acceptable in case they have a low variation coefficient and their failure strengths are well above the expected stress levels. Examples are Scotch Weld 9323 and DP490, which failed (partially) adhesively after conditioning at the aluminium side, at strength levels significantly above 15 MPa and with variation coefficients of about 10%.

The adhesives which are selected and which are currently being exposed to a number of additional tests are tabled in Table 3.

**Conclusions and summary**

In general, the strength levels of the different unexposed adhesive types corresponded to the order of magnitude of values found in literature. However, the results of the lap shear tests after exposure to 90% relative humidity at 50° C revealed that the resistance of several adhesives was changed significantly in function of the duration of the exposure. Consequently, the suitability of this test as a basis for a critical selection of potentially suitable adhesives for the following test phases was in retrospect confirmed.

More precisely, the strength of many specimens was significantly reduced after exposure. However, in contrast to the previous conclusion, a few adhesive products seemed to benefit from the exposure conditions as they yielded better shear strength results. Most probably, the latter is caused by further curing, evaporation of weakeners and consequent hardening at the exposure temperature of 50° C.

Furthermore, the test results were not intended and should not be used to deduct quantitative shear strength values: the latter will be examined more in detail in the following phases of the experimental program.
Acknowledgements

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References


Table 3 selection of 14 out of 32 adhesives promising for the cases defined. All 14 cases are to be subjected to successive in-depth applied testing.

* A selection of the most appropriate adhesives for the point-fixing sub-cases will be made only after the results of future tests are known.

Linear applications
Linear applications
Point-fixings
(shop conditions)
(on site conditions)
(all conditions)*

<table>
<thead>
<tr>
<th>Linear applications (shop conditions)</th>
<th>Linear applications (on site conditions)</th>
<th>Point-fixings (all conditions)*</th>
</tr>
</thead>
<tbody>
<tr>
<td>Sikafast 5211</td>
<td>Dow Corning 993</td>
<td>3M Scotch Weld 9323 B/A</td>
</tr>
<tr>
<td>Sikalack-Drive</td>
<td>Dow Corning Instant Glaze 3</td>
<td>3M Scotch Weld DP 490</td>
</tr>
<tr>
<td>Sikasil S550</td>
<td>Delo Duopox AD 821</td>
<td>Dow Corning TSSA</td>
</tr>
<tr>
<td>Sikaflex S21 UV</td>
<td>Soudaseal 270 HS</td>
<td>DuPont Serrycil NS</td>
</tr>
<tr>
<td></td>
<td></td>
<td>Huntsman Aradite 2047</td>
</tr>
<tr>
<td></td>
<td></td>
<td>or MMA/Lord 406/17</td>
</tr>
</tbody>
</table>

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