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Reference:
Lauriks Leen, Wouters Ine, Belis Jan.- Compressive and lap shear tests on traditional putty and polymer sealants
Full text (Publisher's DOI): http://dx.doi.org/doi:10.1016/J.IJADHADH.2015.10.015
To cite this reference: http://hdl.handle.net/10067/1284340151162165141
Compressive and lap shear tests on traditional putty and polymer sealants

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Abstract
In the 19th century, iron-and-glass roofs were built using linseed oil based putty as sealant to fix the glass plates in the iron glazing bars. Since then, two evolutions influenced the construction details of roofs drastically. First, new sealants with better mechanical properties and higher durability displaced the use of traditional putty. Second, as the insights in the properties and behaviour of glass improved, glass plates can today play a structural role in the global stability of an iron-and-glass structure.

This article examines the mechanical properties of the traditional linseed oil based putty to assess the structural integrity of existing iron-and-glass roofs. Single-lap shear and compressive tests are carried out on a traditional linseed oil putty sealant. Next, to determine the impact of a renovation campaign, a modern MS polymer sealant with comparable viscosity and texture but higher durability, is subjected to single-lap shear experiments.

The experiments demonstrate that the linseed oil putty can have significant compressive stiffness. However, the shear strength is negligible. The modern polymer sealant has higher shear stiffness and strength as well as a cohesive failure in shear.

Keywords
sealants, metals, lap-shear, mechanical properties of adhesives, heritage
1. Introduction

When renovating 19th-century iron-and-glass roofs, the question rises how we can preserve this built heritage while fulfilling the modern standards on safety and structural integrity. The goal of a structural recalculation is to assess the safety level of the structure. At the same time, the heritage value of the roof and its components define the boundary conditions in which restoration proposals are presented. The structural role glass plays in modern constructions [e.g. 1–3], poses the question how this could be applied in 19th-century iron-and-glass roofs. Incorporating the glass cladding into the structural model might limit new interventions necessary to fulfil the modern requirements for structural integrity.

If the glass plates and the iron frame must structurally work together, both parts have to be bonded in such a way that loads can be transferred. In 19th-century joints, the glass plates were mostly sealed to the iron T-shaped glazing bar (Figure 1) by using linseed oil putty [4,5]. To evaluate the force transfer between iron and glass, the mechanical properties of the joining material must be known.

Modern adhesive technology is able to connect glass and metal structurally [e.g. 6]. For application on 19th-century iron-and-glass roofs, we face the problem that the mechanical properties of the 19th-century linseed oil based putty are not known. Traditional materials however can have considerable value [e.g. 7,8]. Consequently, this paper will go into the experimental work that is carried out to determine the mechanical properties of linseed oil putty and to compare properties with a modern replacement sealant. These experiments will lead to a first insight in the mechanical possibilities of traditional putty materials to determine whether or not further experimental work would be useful.

Figure 1: Section of a typical 19th-century construction detail: the single glass plate is connected to the iron section with putty.
2. Materials and experiment design

Taking into account the common joints used in current adhesive bonding, the structural
behaviour of a typical 19th-century iron-glass connection (Figure 1) can be categorised as a butt
strap joint (Figure 2): a combination of a single-lap shear joint (working in shear) and a butt joint
(working in tension or compression). When the glass is loaded in-plane, the single-lap shear
part is located at the flange of the T-section and the butt joint at the web.

When a tensile force is applied parallel to the glass surface, the butt joint is loaded in tension.
For this load situation, Adams [9] indicates that the lap shear part will resist the majority of the
forces. Thus, to gather information about the structural behaviour of the connection, a single-lap
shear test can be performed on the sealant.

However, when a compressive force is applied parallel to the glass plate, the contribution of the
butt joint could be important if the ratio between the compressive strength and the shear
strength of the sealant is high. The latter is the case for traditional linseed oil putty. Determining
the compressive strength of the sealant via a compressive test is then appropriate.

Both lap shear and compressive tests are performed on two sealant materials and will be
discussed in the following paragraphs.

![Figure 2: Common engineering adhesive joints [10]](image)

2.1. Putty and polymer sealants

Originally, glass claddings were sealed to the iron glazing bars with putty [5]. Linseed oil mostly
served as the basis for the putty throughout the 19th century and continued to be used in
traditional putty manufacture by glazing craftsmen. The other ingredients vary per recipe. The
A general principle was to make a paste by mixing linseed oil with a drying agent. This drying agent could be chalk or ceruse (white lead). In addition, if desired, a pigment was added to it. Both the drying oil and the filler pigment were decisive for the quality of the putty: equilibrium had to be found between flexibility for handling during construction and hardening on the longer term [11]. Glaziers often made their own linseed oil putty on-site or in their workshop, adjusting the composition based on experience. Glaziers today still use these traditional recipes to make their own putty for restoring historic glazing (both plane and stained glass).

Adhesive manufacturers also compose products based on the traditional recipes. In this experimental research, Soudal putty (Stopverf or Mastic vitrier) is tested. This glazier's putty is made of synthetic resin modified linseed oil. It can be painted. The technical data sheet states a maximum allowable strain of 5% [12]. It will be referred to as "putty sealant" in the proceeding text.

According to literature, traditional putty has to be repainted regularly after which it has a life expectancy of three to five years [13]. Therefore, adhesive manufacturers search for replacement products with increased life span. Modern polymers are developed to obtain a product with similar workability, viscosity, etc. as linseed oil putty. The main goal is to create a product that stays elastic to ensure the water-tightness of a joint over a longer period of time. Soudaseal Tradition is a MS (modified silane) polymer that was developed as a replacement product for traditional linseed oil putty. The maximum allowable strain is around 20% [14], illustrating the higher elasticity compared to the Soudal putty. This product will be referred to as "polymer sealant" in the proceeding text.

2.2. Metal substrates

The material of the substrates for the putty compressive tests is not critical. Firstly, the expected stiffness of the putty sealant is much lower than the stiffness of a metal substrate, thus the geometry of the samples will be invariable. Secondly, when acting in compression, the influence of the adhesion to the substrate surface is negligible, therefore the compressive strength is independent from the substrate material. The substrates for the putty compressive samples were thus manufactured out of aluminium (AlMgSi0,5).
The substrate materials for the single-lap shear test samples are chosen based on practical considerations. The selected polymer sealant was tested in a broad research project on glass-metal adhesive bonds [15–17] where the adhesion to both aluminium and glass was tested. To complement these two substrate materials, steel substrates are chosen for the experiments reported in this paper. The putty shear samples are manufactured with the same steel substrate material to allow comparison of the results. Therefore, two lap shear sample sets are made of construction steel (S235 with a yield stress of 235 MPa and a Young’s modulus of 210 GPa).

### 2.3. Test sample matrix

The putty sealant is tested in compression. Traditional putty is known for drying out and thus hardening during its curing time. Therefore, two sample series out of aluminium were manufactured to test the putty sealant at different curing times. The tests were performed after 1 and after 3 months of curing (choice of these curing times will be explained in section 3.1). Additionally, the putty sealant as well as the polymer sealant are tested in shear after 1 month curing time. Two single-lap shear sample series are made out of construction steel to test both sealant materials.

An overview of all the test sample series is given in Table 1. In the proceeding text, the sample series are named with abbreviations relating to the test set-up (CO for compression, SH for single-lap shear).

<table>
<thead>
<tr>
<th>Table 1: Matrix of sample series</th>
</tr>
</thead>
<tbody>
<tr>
<td>number of manufactured samples</td>
</tr>
<tr>
<td></td>
</tr>
<tr>
<td>CO - putty 1 month</td>
</tr>
<tr>
<td>CO - putty 3 months</td>
</tr>
<tr>
<td>SH - polymer sealant</td>
</tr>
<tr>
<td>SH - putty sealant</td>
</tr>
</tbody>
</table>
3. Experiment execution

3.1. Compressive test samples

The compressive samples are made out of aluminium. The individual moulds are built up of a PVC holder and an aluminium spacer block (Figure 4). The samples are constructed as a representation of a traditional 19th-century iron and glass connection: a thin plate (representing the glass plate) is attached to an L-shaped section (Figure 3 and Table 2).

The geometry of the compressive samples is chosen differently from the standard dumbbell type specimens. Preliminary try-outs raised doubts about the possibility to manufacture dumbbell specimens with the flexible putty sealant. Therefore, a geometry is designed where the putty sealant is loaded in compression and where the substrates help to maintain the overall geometry. Due to the low shear stiffness of the putty sealant (paragraph 4.2), interference with other than compressive load paths is limited.

One sample series is tested after one month curing while the other series is given three months of curing time. The excess of putty of all samples is cut off after one month curing. These curing times were decided during follow-up of the hardening process of the sample series. A curing time of 1 month was chosen for the first sample series: after 1 month, a construction should be able to stand on its own, without external struts necessary. After testing, the heart of the putty sealant showed not to be fully cured after the first month. Therefore, a curing time of 3 months was decided for the second sample series.

<table>
<thead>
<tr>
<th>Table 2: Geometry of putty sealant compressive samples</th>
</tr>
</thead>
<tbody>
<tr>
<td>number of manufactured samples</td>
</tr>
<tr>
<td>-------------------------------</td>
</tr>
<tr>
<td>CO - putty 1 month</td>
</tr>
<tr>
<td>CO - putty 3 months</td>
</tr>
</tbody>
</table>
3.2. Lap shear test samples

Two construction steel sample series are manufactured for the lap shear tests of the putty sealant and the polymer sealant (Figure 5). The steel surface is sandblasted to remove all corrosion and impurities. The samples are cleaned with acetone both before and after the sand blasting. Subsequently, the samples are bonded with the putty sealant or the polymer sealant (Figure 6 left and middle and Table 3). The excess of sealant is cut off the sides of the test samples one day before testing.

Both series of lap shear samples cure one month at room temperature before being tested.
Table 3: Geometry of sealants lap shear samples

<table>
<thead>
<tr>
<th></th>
<th>number of manufactured samples</th>
<th>average width $b$ [mm]</th>
<th>average overlap $l_a$ [mm]</th>
<th>average thickness $d_a$ [mm]</th>
</tr>
</thead>
<tbody>
<tr>
<td>SH - putty sealant</td>
<td>1(^{(a)})</td>
<td>25.44</td>
<td>11(^{(b)})</td>
<td>2</td>
</tr>
<tr>
<td>SH - polymer sealant</td>
<td>9</td>
<td>24.87</td>
<td>11.15</td>
<td>2</td>
</tr>
</tbody>
</table>

(a) Only one sample is included in the results and therefore the dimensions are only given for this sample (more details in Section 4.2)
(b) Due to the fragility of the putty sealant samples, the overlap can only be measured with 1 mm accuracy

Figure 5: Sample geometry of the lap shear samples in construction steel

Figure 6: Sample series: (left) polymer sealant lap shear samples; (middle) putty sealant lap shear samples; (right) putty sealant compressive samples
3.3. Compressive test equipment

The compressive tests on the putty sealant samples are performed with an Instron 100 kN universal electromechanical test machine at the Vrije Universiteit Brussel. The tests are carried out at room temperature. The displacement rate is set at 1 mm/min. Due to the flexibility of the sealants (compared to the stiff substrate materials), the displacements of the grips can be supposed equal to the strain of the sealant under testing. Free play of the grips is avoided by mechanically tightening the tapered grips around the plate (which was executed long enough for secure tightening). The samples are placed on a spherical cap hinge to ensure the centric application of the compression load in the test samples (Figure 7).

![Figure 7: Ensure centric compression force by using a spherical cap hinge during compressive tests](image)

3.4. Lap shear test equipment

The single-lap shear tests on the sealants are performed with a Zwick 10 kN universal electromechanical test machine at the Soudal research lab. The grips are designed to be adjustable so that the loading is put centric on the adhesive bond. The tests are carried out at room temperature. A displacement rate of 5 mm/min is applied for the polymer sealant samples, however this is lowered to 1 mm/min for the putty sealant lap shear samples because of the expected low resistance. The displacements of the grips are recorded and supposed equal to the displacements of the adhesive bond (which is accurate for flexible sealants). The shear strain is calculated based on these displacements.
4. Results and discussion

All results from the experiments are reported in Table 4 and Figure 8 and will be discussed in the next paragraphs.

Table 4: Experimental results of sealant materials - overview

<table>
<thead>
<tr>
<th></th>
<th>number of tested samples</th>
<th>compressive strength [N/mm²]</th>
<th>standard deviation on strength [N/mm²]</th>
<th>elongation at max. stress [%]</th>
<th>standard deviation on elongation [%]</th>
<th>Young's modulus 100% [N/mm²]</th>
<th>standard deviation on Young's modulus [N/mm²]</th>
</tr>
</thead>
<tbody>
<tr>
<td>CO - putty 1 month</td>
<td>4</td>
<td>0.11</td>
<td>0.05</td>
<td>19.47</td>
<td>9.01</td>
<td>1.20</td>
<td>1.11</td>
</tr>
<tr>
<td>CO - putty 3 months</td>
<td>5</td>
<td>1.72</td>
<td>0.66</td>
<td>13.78</td>
<td>2.37</td>
<td>20.65</td>
<td>9.81</td>
</tr>
<tr>
<td>SH - polymer sealant</td>
<td>9</td>
<td>0.77</td>
<td>0.05</td>
<td>135.57</td>
<td>13.70</td>
<td>1.27</td>
<td>0.26</td>
</tr>
<tr>
<td>SH - putty sealant</td>
<td>1</td>
<td>0.0028</td>
<td></td>
<td></td>
<td></td>
<td></td>
<td></td>
</tr>
</tbody>
</table>

Figure 8: Experimental results of sealant materials – box plot

4.1. The putty sealant compressive tests

The results of the putty sealant compressive tests are summarized in Table 4. The mean compressive strength and Young’s modulus are an arithmetic mean of the results of all samples. One sample of the series that cured for one month (CO - putty 1 month sample 6.01), fails while positioning it in the hinge.
The compressive samples are opened up after the tests, showing a diversity of curing stages with often an alternation of soft material with harder grains (Figure 12 top).

For the putty compressive tests, the Young’s modulus is taken as the stress value at 5% strain (see curve 1 on Figure 9), the maximum allowable strain defined in the technical data sheet, divided by that same strain.

The experimental curve of compressive tests show strong nonlinear character. The curve up to 5% strain is considered for the determination of the Young’s modulus, although it does not coincide with strict linear behaviour. Above 30-50% strain (dependent per sample), the higher stress rates are caused by compressive force transfer between the aluminium substrates of the test specimen. In this context, the compressive stress cannot be determined by the maximum stress reached during the test but at the same time the division between elastic and plastic behaviour is not clear. Therefore, a methodology was applied based on the principle used for the analysis of stress-strain curves of metals. The compressive strength is determined by constructing the linear behaviour (the Young’s modulus) (see curve 2a on Figure 9) starting from 5% strain, so that the intersection with the experimental curve is the maximum allowable compressive strength (curve 2b on Figure 9). The resulting compressive strength is indicated in Figure 10 and Figure 11. This methodology was consequently applied for both the 1 month and 3 months curing sample series.

Figure 9: Derivation example of Young’s modulus (curve 1) and compressive strength (curves 2)
The Young’s modulus of the putty sealant samples reaches 1.20 N/mm² after one month curing under compressive forces. Nevertheless, the stiffness increases significantly after three months curing time. The elongation at the maximum stress also decreases, which confirms the hypotheses that the putty becomes more brittle after a longer curing time.

### 4.2. The putty sealant shear tests

The results of the putty sealant shear tests are summarized in Table 4. Nine samples of the putty sealant lap shear joints are manufactured. Seven samples fail while positioning them in the testing machine due to their fragility (the putty was still very soft). Finally, two samples are
tested. Only one sample resists force during deformation. This high loss of samples is to be taken into account in the statistical interpretation of the experimental results.

The sole tested putty lap shear sample undergoes such a major elongation that the test is interrupted. The shear strength (maximum stress reached during testing) as well as the G-modulus are very low. The bond at the surfaces is tested manually afterwards and is very weak (Figure 12 middle).

The shear strength of the putty sealant is negligible. All putty samples fail adhesively and are thus possibly subject to improvement when the adhesion to the substrates could be improved. The putty samples have an elongation of 31% at its maximum stress during shear. These results clearly confirm the non-structural function for which the putty sealant is intended.

4.3. The polymer sealant shear tests

The results of the polymer sealant tests are summarized in Table 4. The mean shear strength is an arithmetic mean of the maximum stress reached during testing of all samples. The polymer sealant lap shear samples all fail cohesively (Figure 12 bottom).

For silicone sealant connections, linear behaviour of single-lap shear tests is considered to last at least up to 12.5% of the strain at failure [1]. The same approach is used for the polymer sealant lap shear samples: the G-modulus was for every sample taken at the strain value of 12.5% of the strain at failure (Figure 13).

The stress-strain curve in Figure 13 shows to distinct groups of samples between 0 and 40% strain. The first group comprises of samples 1.01 and 1.02 and have a larger initial stiffness than the other samples. The explanation for this difference is the fillet of the adhesive at the end of the substrate plates. This fillet is cut off starting from sample 1.03 but causes a slightly higher force transfer in the first two samples.
Figure 12: Failure surfaces: (top) putty sealant compressive tests; (middle) adhesive failure of putty sealant lap shear test; (bottom) cohesive failure of polymer sealant lap shear tests

Figure 13: Stress-strain curve of polymer sealant single-lap shear tests
The polymer sealant reaches higher values for its shear strength (0.77 N/mm²) and its elongation at its maximum shear stress (136%). This confirms the expectations, since the polymer sealant was explicitly developed to improve the flexibility compared to the traditional linseed oil putties.

The Young’s modulus of the polymer sealant is taken from the technical data sheets of the adhesive manufacturer (at 100% strain: 1.35 N/mm²) [14] and is comparable to that of the putty sealant under compression after one month curing. The technical data sheet also mentions a tensile strength of 1.40 N/mm² however no compressive strength is known for comparison with the putty sealant sample series.

5. Conclusions

In 19th-century iron-and-glass roofs, glass plates were positioned on the iron glazing bars using traditional linseed oil putty. The loads in a representative 19th-century iron-glass connection are transferred in shear (single-lap shear joint) and in tension and/or compression (butt joint). This experimental research extends the knowledge about the mechanical properties of traditional linseed oil based putty and its modern replacement polymer sealant by conducting tests in shear and compression.

The linseed oil putty samples reveal that the shear strength of this material is very low, after one month curing, compared to the MS polymer sealant. However, the compressive tests on the linseed oil putty point out that the stiffness increases considerably over time.

The adhesive manufacturers developed modern sealants that have comparable viscosity and texture parameters as linseed oil putty, but stay flexible for a longer time, for example the tested MS polymer sealant. This sealant has a higher shear stiffness than linseed oil putty. These replacement sealant materials could be interesting when the structural behaviour of the joint has to be improved to fulfil modern requirements for structural integrity of a 19th-century iron-and-glass roof.

This research was born out of concern about the heritage value of 19th-century iron-and-glass roofs. Preserving the traditional connection method and traditional materials during a restoration campaign is preferred from the heritage point of view. However, the mechanical properties of
the putty that result from the experiments are not convincing for its preservation in historical constructions.

Nevertheless, structural calculations demonstrated that the traditional putty achieves sufficient stiffness to let the glass cladding contribute to the overall structural behaviour [5,18]. Therefore, further investigation in the mechanical properties of traditional putty materials is considered useful. Different aspects are today still unclear: the curing process, different putty compositions, the influence of humidity and temperature, the tensile strength of the putty sealant, the structural behaviour of the putty sealant under cyclic loading, the adhesion properties to substrates, as well as the compressive strength of the polymer sealant.

**Acknowledgements**

This research was funded by the Institute for the Promotion of Innovation by Science and Technology in Flanders (IWT-Vlaanderen).

For technical advice and help with the experimental part of this research, the authors would like to thank Baeck & Jansen NV, Steven Vandebril and Filip Van Mieghem (Soudal), Arno Van Hulle, Dieter Callewaert, Freek Bos (Ghent University), Frans Boupaep, Daniel Debondt, Gabriël Van den Nest (VUB MeMC), Iris De Graeve, Jean Vereecken, Marnix De Pauw, Marc Raes (VUB SURF), and Heidi Ottevaere (VUB TONA).
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