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MOISTURE DEGRADATION OF OPEN-FACED SINGLE LAP JOINTS

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Abstract

To obtain experimental data in short time on the degradation of adhesives exposed to moisture, a valuable technique is represented using the open-face configuration. With this technique, a layer of adhesive is first applied on one adherend and exposed to the humid environment; then, the second adherend is bonded and the joint can now undergo mechanical testing. Apart from the acceleration of moisture uptake which is obtained due to the larger area exposed, a further advantage is the uniformity of degradation. A further acceleration can be obtained by adding a hygroscopic contaminant at the adhesive/adherend interface, which speeds up moisture uptake and accentuates the interfacial nature of the failure.

The main aim of this work was to evaluate the decay of the mechanical strength in absence or presence of a contaminating agent. The specimens studied were single lap joints, tested under static shear loading. Two sets of specimens were considered; in the first the adhesive was applied in standard way, in the second the adhesive/adherend interface was contaminated with droplets of CaCl₂ aqueous solution. Both sets were subjected to humid and warm environment (100% relative humidity, 50°C). After the desired exposure times, in the range 1-5 weeks, groups of specimens were dried and bonding of the second adherend was carried out. Then, mechanical testing was performed; the fractured surfaces were examined by scanning electron microscopy (SEM). The results show that before degradation the failure type is cohesive, but it changes to interfacial failure as the degradation proceeds. Uncontaminated specimens

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exhibit gradual degradation during the exposure time; contaminated specimens achieve almost half of the degradation in less than one week; after that, the process continues at lower speed and at the end of the observed period both methods show similar values of failure loads. Additional tests were carried out to assess the moisture absorption in the adhesive layer and relate it to the exposure time.

Keywords: Environmental degradation, Epoxy adhesive, Lap shear specimens.

1 INTRODUCTION

The durability of the joints exposed to a moist environment is –besides the need for reliable design methods to predict strength– the major concern in adhesive bonding. Although the subject has been investigated since long, it is still extensively studied at present [1-18].

The problem poses several issues, which research has tried to address. The first, obviously, is assessing the diffusion of water in the joint, which can affect both the adhesive bulk and the interfaces. Almost all studies regard Fick's law of diffusion as a starting point [1-3,5,11-13,15-18], but several researchers find that the process is non-Fickian and adopt more sophisticated laws, for instance dual-Fickian or Langmuir's [6,10,13,15,16,18]. A related aspect is the effect of water on the adhesive, which causes plasticization and swelling [2-4,6,8,11,15,16,17]. It is recognized that this is due to the free H₂O molecules diffused in the adhesive, therefore, the related weakening effects are reversible, i.e. the original strength is recovered when the material is dried [6,15]. Conversely, the bound H₂O molecules cause damages which cannot be recovered [18] by drying. These phenomena lead to different strength values (and also to different failure modes) if the specimens are tested in “wet” or “dry” conditions [6].

However, the most severe harm to joint strength is caused by the action of the moisture in the adhesive/adherend interface zone. Typical mechanisms are hydrolysis and breakage of the bonds at the interface, causing displacement of the adhesive [4]. In several cases it is observed that under loading, failure occurs close to the interface and a very thin layer of adhesive remains stuck to the adherend [4]. Another possible failure mechanism concerns the metallic adherends, as the surface oxide layer can separate from the bulk [9,14] under loading or even spontaneously. A significant role is played by the treatment applied to the adherend before bonding, ranging from simple degreasing plus grit blasting to chemical etching of different kinds [1,9]; in case of aluminium, anodizing is typically used [1,9,15]. Another key factor is the application of a primer on the adherends before bonding, which –apart from activating the

surface— prevents corrosion and also, in case of metallic adherends, inhibits penetration of the water in the surface oxide layer [9,10]. It is also possible that failure occurs in the primer [15]. Since the typical timescale of the environmental effects on joints is of months or years, testing is carried out under accelerated conditions, exposing the specimens to moist air or by immersing them in water. To this aim, the temperature is also increased with respect to ambient, but special care is required because at temperatures close to –or greater than– the adhesive glass transition temperature T_g , the absorption rate increases dramatically and this invalidates the results. Consequently, the amount of water absorbed may depend, apart from the adhesive type, on the condition of exposure.

All these aspects affect the transferability from laboratory conditions to real life applications. In general, the test results are more qualitative than quantitative, in the sense that they identify comparatively “the best”, in an assortment of materials, treatments, etc., candidate for a certain application. Conversely, it is difficult to assume as a design value the result obtained in terms of ultimate load, fracture energy etc.; this is possible, for instance, when the strength reduction is a function of the absorbed water only, as in [10], which is not always the case. Another related problem is that the actual exposure conditions for a joint in service are difficult to foresee. Considering the worst scenario, tests can give quantitative information when the joints exhibit a residual asymptotic strength (as, e.g., in [4]), that can be assumed as a “safe” limit for design.

Among the acceleration techniques which have been proposed, an interesting solution is given by the “open-faced” joints, described in more detail in the following section, which allow obtaining faster exposure and nearly uniform diffusion. This approach, reported first in [5], exploits the same principle as the wafers of adhesive used for diffusion measurements, and has become rather popular in the last years ([6,8,10,16,18]). In the present work the open-faced technique was applied to joints involving steel adherends (rarely treated in the literature,

[10]), including also the use of a contaminant [8,14] to accelerate moisture uptake at the adherend surface.

2 EXPERIMENTAL PROCEDURE

2.1 Open-faced joints

The traditional approach used to degrade the adhesive is to place the complete adhesive joint test specimen in a moist environment. In this way, moisture can diffuse just from the perimeter of the adhesive as shown in Fig. 1(a). This process is slow and completion of moisture absorption can take unacceptably long time. The other shortcoming of the traditional method is that the moisture distribution across the joint is not uniform, nor is the degradation [10]. Fig. 1(b) shows the open-face joint configuration with its water ingress path; with this method the entire bond area is exposed to the moisture. For example, by comparing conventional and open-faced methods with the dimensions of the Single Lap Joint (SLJ) illustrated in Fig. 2, it is found that the water path ingress in case of the open-face method is more than 30 times wider than in case of the conventional method.

2.2 Specimen preparation

The adherends used were carbon steel strips with dimensions 100 mm × 25 mm × 2 mm, cut from extruded plates. They were degreased with acetone both before and after roughening using sand paper P100, to provide a suitable surface for bonding. The adhesive used in this investigation was Hysol 3425, a two-component epoxy produced by Henkel [19]. This product reaches its full strength in one week if cured at room temperature. To accelerate the curing process, after applying the adhesive the specimens were cured at a temperature of 80°C for 2 hours; in this way the final strength which is reached is about 10% higher than in case of curing at room temperature, as reported by the product datasheet [19]. The stress-strain curves of the adhesive, as polymerised and after different periods of exposure to moisture, are shown in [20].

As shown in Fig. 3, the primary adhesive layer (0.2 mm thickness controlled using brass wires as spacers) was cured on the first adherend using a backing plate coated with Teflon films, to avoid adhesion, as a second adherend. To keep the exact dimension of the overlap length, the non-overlapping part of the substrate was covered with an adhesive tape. The single lap joint configuration with details of primary and secondary bonding is shown in Fig. 2.

After drying at room temperature for one day, the backing plate and the protective adhesive tape were removed. After curing the primary adhesive, the specimens were protected with an anti-corrosion paint. The perimeter edge of the bondline was sealed with silicone to avoid moisture ingress. Then the specimens were exposed, as shown in Fig. 4, to warm and wet environment in a climatic cabinet at 50°C and 100% relative humidity (same conditions as in [1]); such a temperature was deemed high enough (compared to ambient temperature) to accelerate degradation and, at the same time, was sufficiently below T_g (72°C, [19]) to avoid an unrealistically high absorption rate. The test duration was varied in six levels from 0 to 5 weeks of exposure, thus longer than in similar works ([10,14]), to compensate for the lower temperature. At the end of the corresponding exposure period, the degraded specimens were dried in an oven of sufficient internal volume (to avoid saturation of water vapour inside the oven) at 70°C for 72 h. Then, to allow mechanical testing, the degraded open-faced specimens were completed, forming closed joints, by bonding the second adherend using an additional adhesive layer, termed “secondary adhesive”. As proposed in [16], to ensure mechanical interlocking between the primary and secondary adhesive layers, the primary adhesive layer surface was roughened using 100 grit sandpaper, then degreased with acetone before secondary bonding. Then the joint was cured in identical fashion to that used for the first adhesive. Tabs were bonded to the two ends of each specimen (Fig. 2) to avoid offset in the grips when loading.

2.3 Contaminated interface

The application of a contaminant on the substrate surface is described in [8], and later in [14], with the aim of increasing the concentration of the moisture at the substrate surface and subsequent local degradation. In those works, the adherends were aluminium alloys. The contaminant is, typically, a hygroscopic substance which attracts the water from the adhesive bulk towards the interface with the adherend; to obtain significant results it must not reduce the initial (i.e. before exposure) strength of the joint [14]. Within this work, calcium chloride (CaCl_2) was chosen as a trade-off between effectiveness, availability and non-hazardous use; the adherends were again (as for the uncontaminated case) carbon steel.

After the surface preparation process, prior to application of the primary adhesive, half of the specimens were contaminated using an aqueous CaCl_2 solution 4 % by weight. Three droplets of 2 μl were deposited on the adherend surface using a micro-pipette; the specimens were dried in an oven at 90°C for one hour and kept, when cooling down, in a drying unit with silica gel to avoid absorption of environmental moisture. After that, the adhesive was applied as for the uncontaminated specimens.

3 RESULTS

3.1 Lap shear test

All mechanical tests were carried out under ambient conditions on an Instron 100 kN hydraulic machine at constant displacement rate of 1 mm/min.

A preliminary group of tests was carried out to investigate the potential effects of the fabrication technique, open-faced, and contamination on the strength of the specimens, to ascertain if the joints fabricated with these procedures could be intrinsically weaker than those fabricated in standard way. Three sets of five specimens each, manufactured as standard (traditional), open-faced, open-faced contaminated, with a total thickness of 0.4 mm, were tested as fabricated (i.e. not exposed). The results are shown graphically in Fig. 5; it can be

seen that the mean failure load of the joints fabricated in standard way (7.32 kN) is about 10% higher than the remaining two (6.64 kN and 6.74 kN) which, considering also the scatter in the values, can be regarded as equal. In most of the open-faced specimens, failure occurred at the interface between primary and secondary adhesive.

Thus, as stated previously, the two sets of uncontaminated and contaminated open-faced specimens were exposed to humid and warm environment for 1-5 weeks, then the specimens were dried, completed with the secondary bonding and tested. Testing under these “dry” conditions was chosen purposely, to account only for the irreversible effects [6].

Fig. 6 shows the reduction in failure load as a function of exposure time, for the two sets of specimens. In general, it can be noticed that the reduction is significant and proceeds over the entire time period, without any asymptotic tendency. The curve corresponding to the uncontaminated specimens exhibits a steady, more regular decrease; on the contrary, for the contaminated specimens, the strength drops dramatically after the first week –during which almost half of the total reduction occurs– then continues at lower rate. Analogous findings were obtained in [14] about the fracture energy G_c . After three weeks, the strength values of uncontaminated and contaminated specimens are similar and, subsequently, the two curves seem to decrease in a parallel fashion.

The reduction in the strength corresponds to the change in the failure mode from cohesive (as previously stated, after fabrication the specimens failed in the combined adhesive layer) to interfacial, i.e. at the substrate surface. The phenomenon is related to the progressive oxidation of the steel adherend. It is interesting to notice that after 1 week the type of failure for the contaminated specimens changed from cohesive to interfacial, and the surface of the adherend was covered by large amount of oxide layer. For the group of uncontaminated specimens this fact occurred after 2 weeks and the amount of oxide layer was not as much as for the previous case. A possible explanation is that the contaminant accelerates the formation

of oxide layer in a short time at the adherend surface by increasing the speed of penetration of water through the micro-holes that exist inside the adhesive.

At the end of the exposure, the oxidation was so severe that the failure occurred as a separation within the oxidized layer.

3.2 Moisture absorption

To collect information on the water uptake by the open-faced joints, and relate the exposure time to the amount of water, two sets (contaminated and uncontaminated) of three open-faced specimens each were prepared. To avoid problems regarding the accuracy of the measurements due to the possible oxidation of the substrate or capture of moisture by the protective paint, the steel specimens were replaced by coupons of plastics. *Ad hoc* tests carried out on the coupons alone proved that they did not react or absorbed water (the dimensions of the adhesive layers deposited for these measurements, 25 mm × 12.5 mm × 0.2 mm, were the same as those of the primary bonding of the lap shear joints). The mass of each specimen was weighed as a function of time after wiping the excess moisture from the surface, and the percent moisture uptake $m_{\%}$ was calculated as:

$$m_{\%} = \frac{M_t - M_i}{M_i} \cdot 100 \quad (1)$$

where M_i and M_t are, respectively, the mass of the specimen under initial conditions and at time t . Surprisingly, as shown in Fig. 7, the measurements exhibited an oscillating behaviour after that the “knee” of the absorption curve was reached. Since a reduction in the absorbed moisture while the specimen is still exposed in the climatic chamber is physically implausible (some undetected and unexpected factor, causing inaccuracies, must have come into play), it appears reasonable to extrapolate the results by ignoring the descending values. This gives the dashed curves in the figure. The uncontaminated and contaminated cases do not appear significantly different in terms of absorbed moisture.

In turn, the above mentioned curves can be satisfactorily fitted by the Fickian model [13] for one-dimensional diffusion:

$$\frac{M_t}{M_\infty} = 1 - \frac{8}{\pi^2} \sum_{n=0}^{\infty} \frac{1}{(2n+1)^2} \exp\left[-\frac{D(2n+1)^2 \pi^2 t}{4l^2}\right] \quad (2)$$

where M_∞ is the water mass (g) absorbed at saturation, t is the time (h), D is the diffusion coefficient (mm^2/h), l (mm) is the half-height of the adhesive layer. The diffusion coefficient can be determined from the initial slope of the absorption curve [13]:

$$D = \pi \left(\frac{2l}{4M_\infty} \right)^2 \left(\frac{M_2 - M_1}{\sqrt{t_2} - \sqrt{t_1}} \right)^2 \quad (3)$$

where subscripts 2 and 1 refer to two instants (points on the curve) in the linear part of the diagram (in practice point 1 can be the origin). The Fickian model, based on $D = 2.69 \cdot 10^{-4} \text{ mm}^2/\text{h}$ given by equation (3) and $l = 0.1 \text{ mm}$ is plotted as a solid line in Fig. 7.

3.3 SEM and EDX analyses

To characterize the effects of contamination process on aging under moist environment, scanning electron microscopy (SEM) and energy-dispersive X-ray analysis (EDX) were used. After mechanical testing of the SLJ specimens, one specimen per batch (12 in total), representative of the degradation due to the corresponding time of exposure, was selected for surface analysis using SEM. From each specimen a coupon 50 mm long (because of the limited area inside the SEM device) was cut out using a band saw (during cutting the adhesive was protected with a paper tape). Both halves of each specimen were examined. Both uncontaminated and contaminated specimens exhibited varying degree of interfacial failure after exposure, the features of the failure surfaces were of considerable interest, because the validity of the contamination method requires that, after exposure to warm moisture, the failure surfaces of the contaminated joints be similar to those of the uncontaminated joints, as documented in other studies [8,14].

The SEM images of the fracture surfaces and the EDX spectra of the unexposed joint (as noticed previously, in this case fracture occurs in the combined adhesive layer) are shown in Fig. 8. The aspects of the surfaces are similar, as well as the element content related to the adhesive composition (note that Ag is added by the metallization process required for SEM observation).

After one week of exposure, the situation starts changing, as visible in Fig. 9 where the SEM pictures show the fracture surfaces of the adhesive side: in the uncontaminated case of Fig. 9(a) the rupture is still cohesive, whilst in the contaminated case of Fig. 9(b) it occurs at the interface with the adherend. The smoother aspect of the latter case reproduces the flat surface of the adherend. The compositions are not significantly different.

As stated previously, after five weeks of exposure the rupture occurs in the oxide layer. Fig. 10 shows the fracture surfaces (adhesive side) which are again similar for both uncontaminated and contaminated specimens. The presence of the oxidized layer on the adhesive is confirmed by EDX analysis in which O and Fe are the most dominant elements.

Eventually, Fig. 11 shows CaCl_2 crystals after the exposure period of five weeks.

4 DISCUSSION

The main goal of the work was to evaluate and compare the performance of the open-faced technique in the two cases of joints (with carbon steel adherends bonded by a two-component epoxy), uncontaminated or contaminated with CaCl_2 at the interface to accelerate degradation.

A first remark is that initially, before exposure to moisture, the strength of the open-faced joints was just slightly lower (10%) than that of the joints fabricated in a single step; most of all, such “initial” strength was the same for uncontaminated or contaminated specimens and also the failure occurred in the adhesive in both cases. Exposing the specimens to warm moisture, the ultimate load decreased dramatically and the failure mode changed to interfacial; for the contaminated specimens such a change occurred already during the first

week of exposure. However, at the end of the considered period, both uncontaminated and contaminated specimens exhibited similar tendencies.

A significant role in the failure mechanism was played by the interfacial oxidation of the adherends, since, after long exposure, the failure occurred as a separation of the oxide crust from the rest of the adherend. The phenomenon was so important that the ultimate load decreased monotonically over the observation period (5 weeks) and no residual strength could be found. A confirmation about this role was given by the SEM observation and EDX analyses, which revealed a large presence of O and Fe on the rupture surface on the adhesive side.

Absorption measurements carried out on layers of adhesive of identical size (although affected by anomalous oscillations after the “knee” of the curve) showed that the moisture uptake was not significantly different between uncontaminated and contaminated cases. This leads to conclude that the difference between was not due to the amount of absorbed moisture but to the attraction of the moisture to the adhesive-adherend interface. In this sense it is not surprising that the strength reduction in the joints was not simply proportional to the absorption, as the ultimate load decreased also when the moisture content no longer increased. The latter fact is justified by the surface corrosion process, which kept on developing, reducing the strength to zero.

5 CONCLUSIONS

The results of the experimental investigation, aimed at evaluating the response obtained from open-faced lap joints, contaminated and uncontaminated, exposed to warm moisture, lead to the following conclusions:

- the open-faced bonding technique reduces the strength before exposure by about 10%, but no significant difference is found between contaminated and uncontaminated joints;
- the absorption of the moisture by the open-faced joint can be described by a Fickian model;

- with continuing exposure, failure changes from cohesive to interfacial, due to substrate oxidation;
- the use of a hygroscopic contaminant does not alter the failure mechanism of the joint, thus, the results are still significant; however, the only apparent advantage of the contamination is that it anticipates to the early days of exposure (at the end of the first week the difference between contaminated and uncontaminated specimens is significant) the transition of the failure mode from cohesive to interfacial; over a longer test period contaminated and uncontaminated specimens exhibit similar trends.

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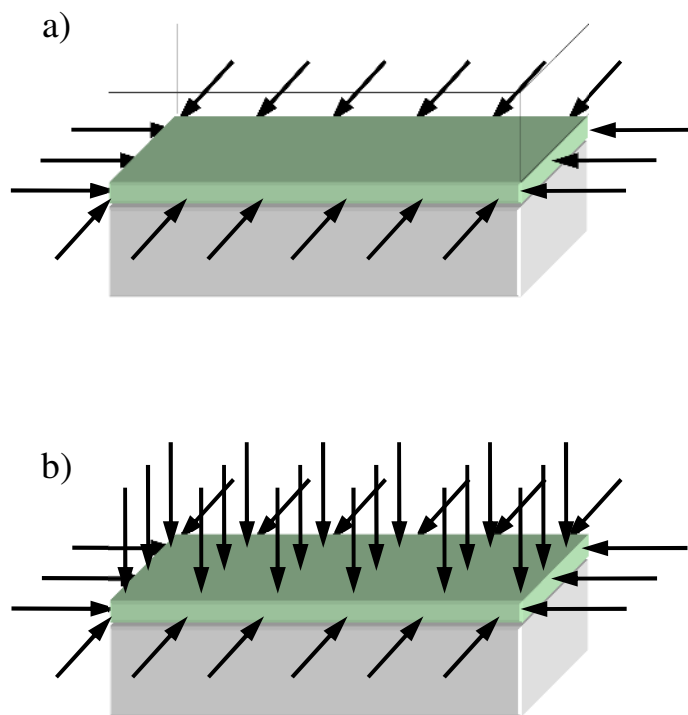


Fig. 1. Path of water ingress in cases of: a) conventional bonded joint; b) open-faced joint.

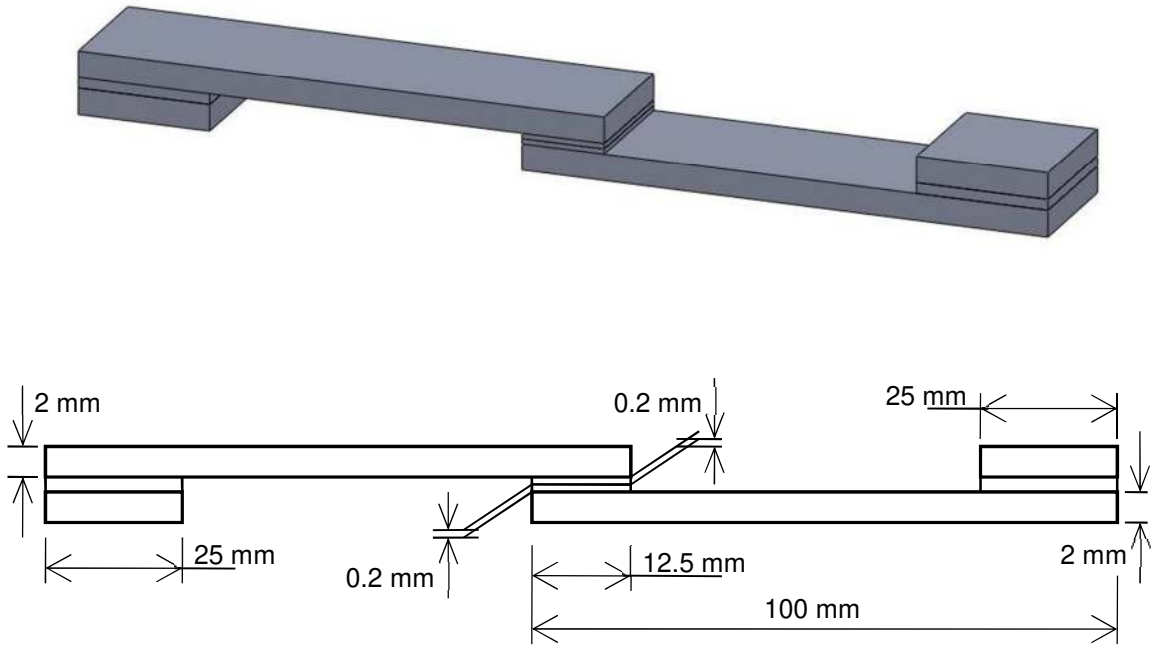
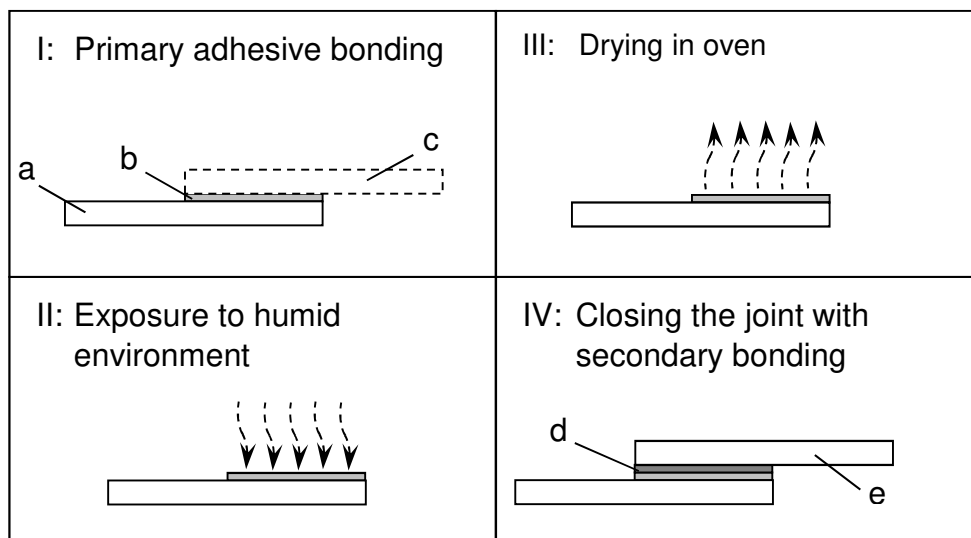


Fig. 2. Single lap joint geometry showing the thickness of primary and secondary adhesive;
(width 25 mm).



a): primary adherend, b): primary adhesive, c): PTFE coated backing plate, d): secondary adhesive, e): second adherend

Fig. 3. Steps of the open-faced SLJ specimen fabrication and exposure to humid and warm environment.



Fig. 4. Open-faced specimens during exposure to humid and warm environment.

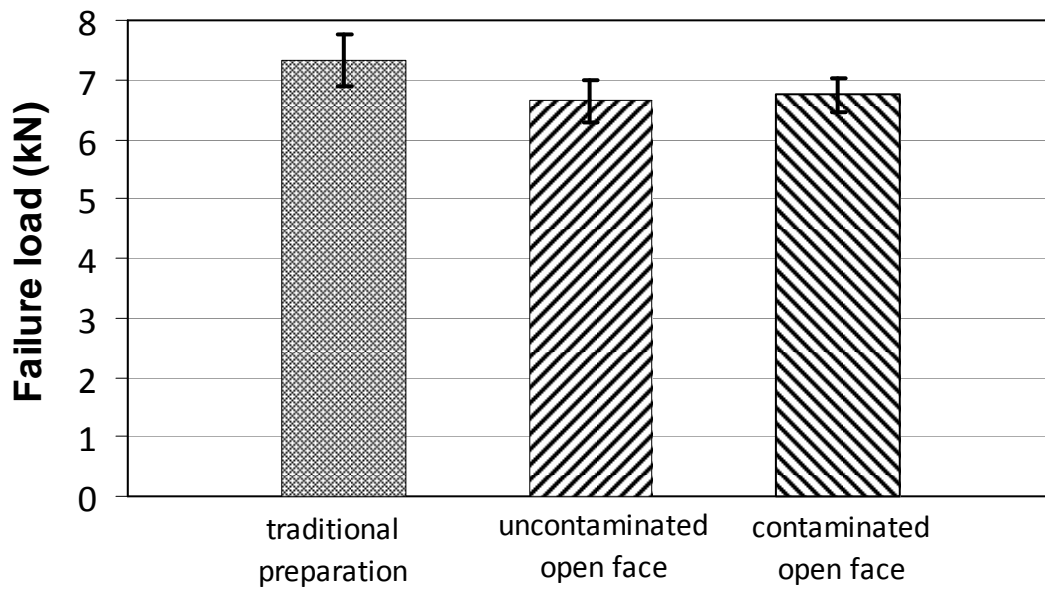


Fig. 5. Comparison of failure loads before exposure to humid and warm environment corresponding to different methods of SLJ preparation (bars show ± 1 standard deviations).

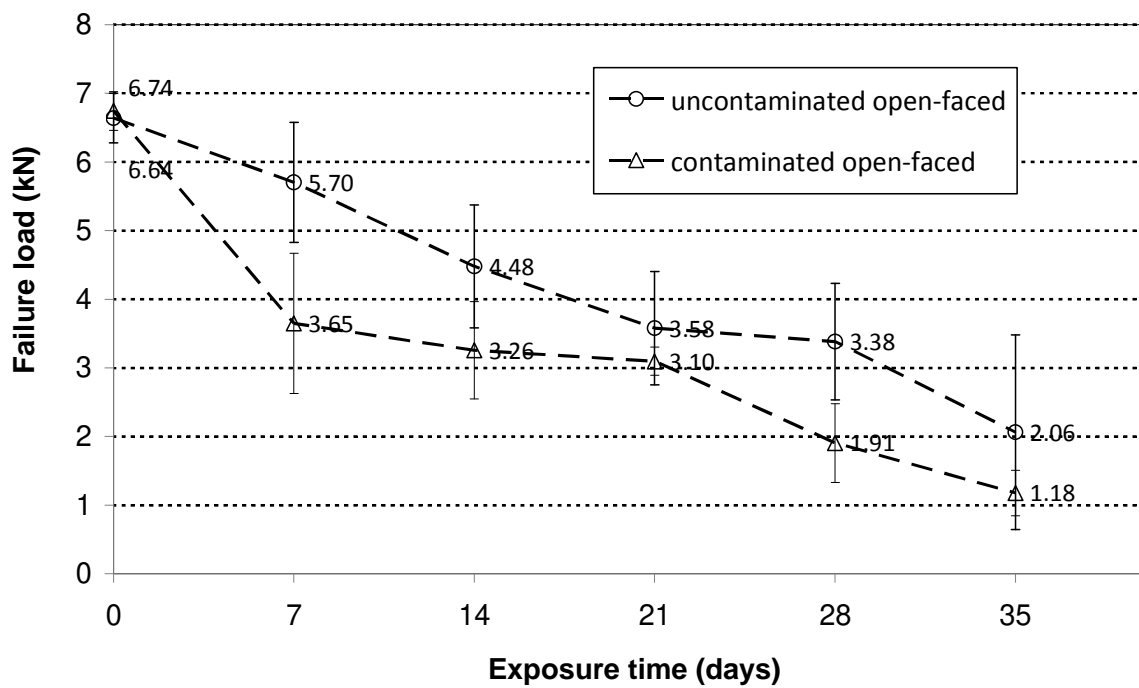


Fig. 6. Failure load reduction of the SLJ due to exposure to humid and warm environment (bars show ± 1 standard deviations).

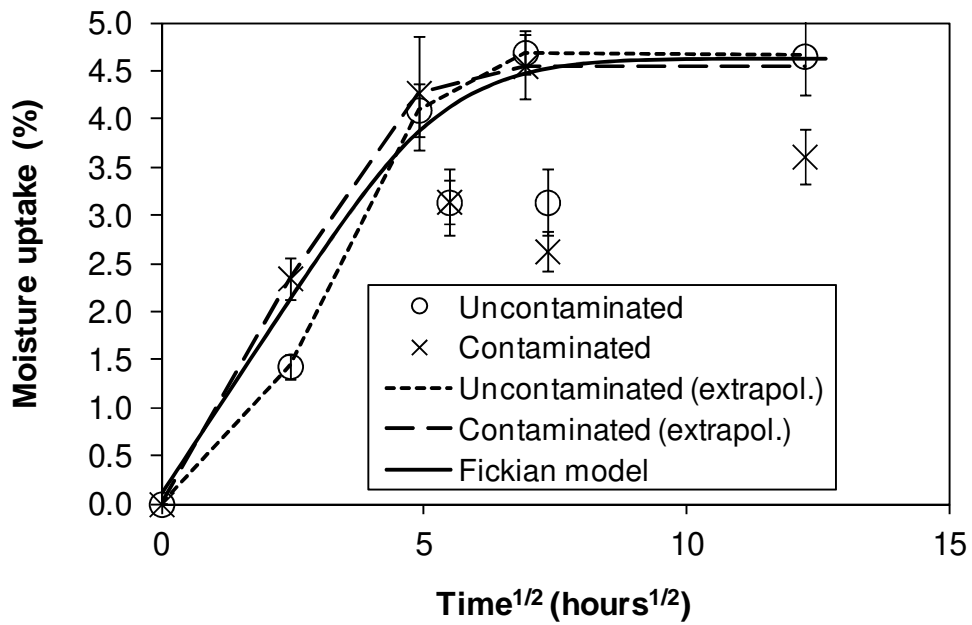
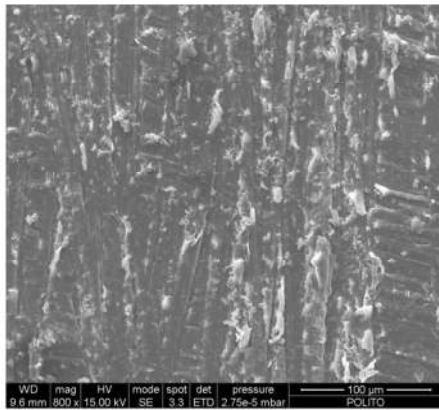
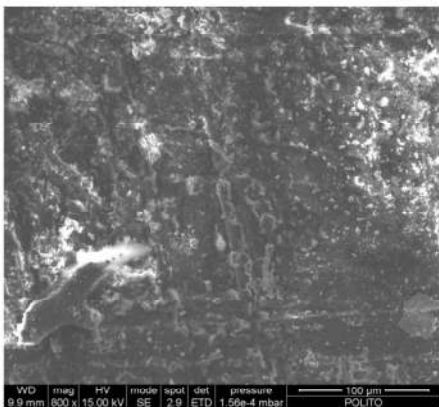
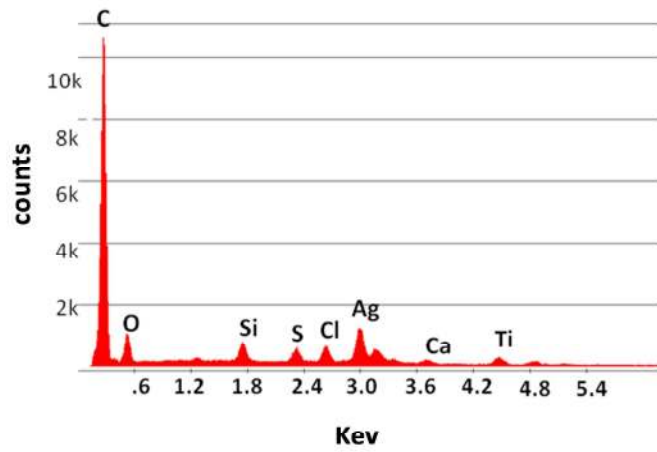


Fig. 7. Experimental moisture absorption curves for the uncontaminated and contaminated adhesive layers (bars show ± 1 standard deviations of the measurements), and related Fickian model.



a



b

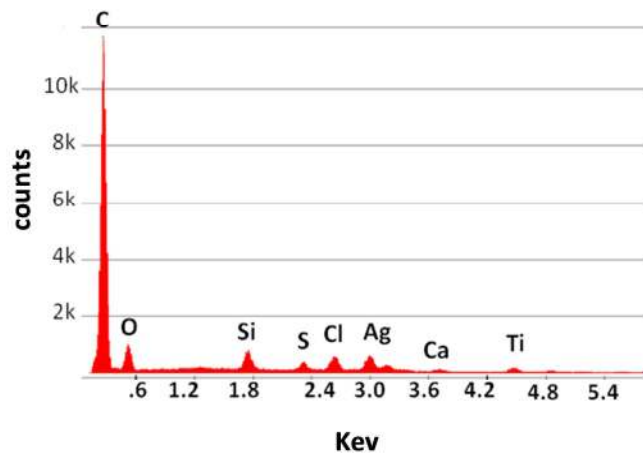
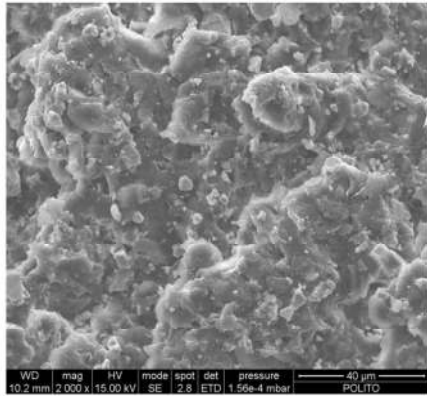
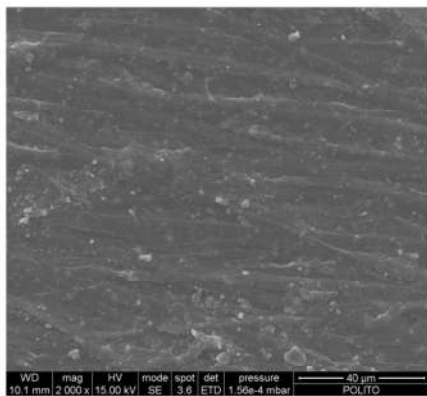
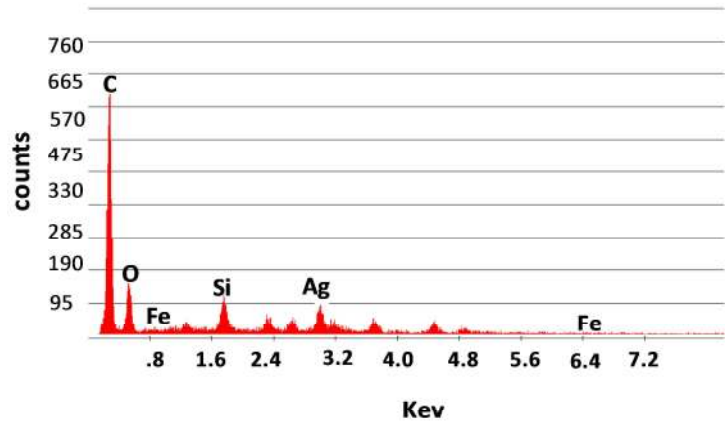


Fig. 8. SEM images (left) and EDX spectra (right) related to the fracture surfaces of unexposed specimens: a) uncontaminated case; b) contaminated case.



a



b

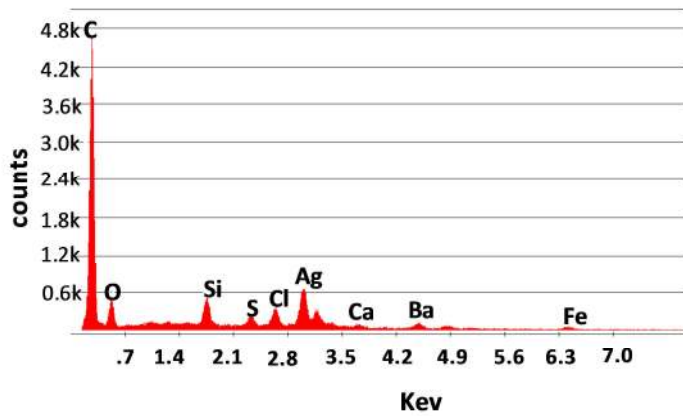


Fig. 9. SEM images (left) and EDX spectra (right) related to the fracture surfaces (adhesive side) after 1 week exposure to humid and warm environment: a) uncontaminated case; b) contaminated case.

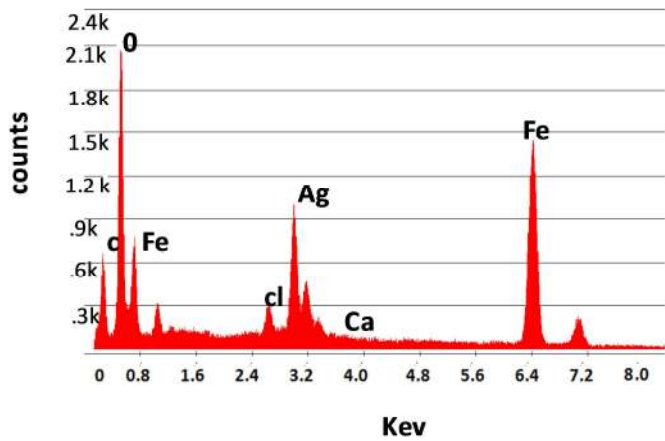
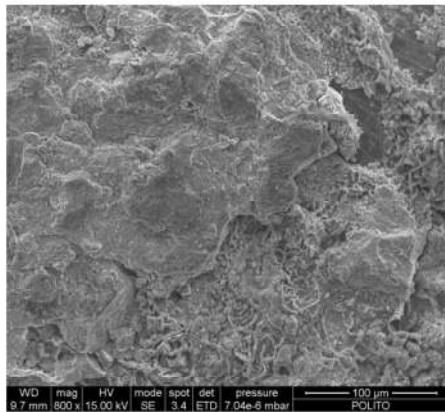
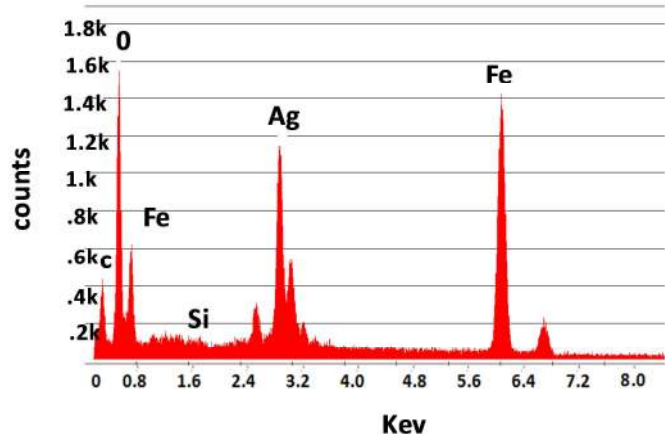
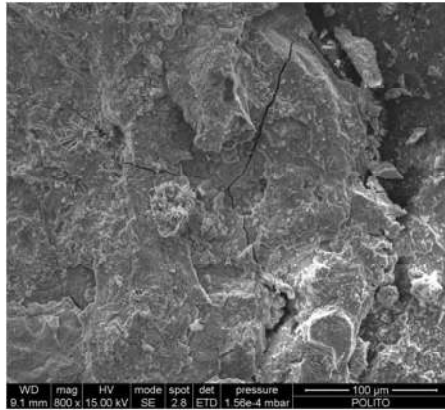


Fig. 10. SEM images (left) and EDX spectra (right) related to the fracture surfaces (adhesive side) after 5 weeks exposure to humid and warm environment: a) uncontaminated case; b) contaminated case.

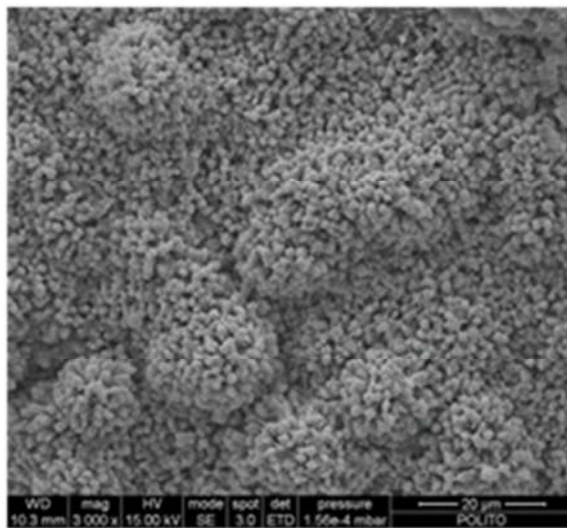


Fig. 11. SEM image of CaCl_2 crystals after 5 weeks exposure.