Mechanical Characterization of the Damage Process in a Structural Adhesive Joint by Acoustic Emission

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Abstract
The aim of this work is to apply the Acoustic Emission (AE) technique in order to detect in real-time the internal damages of a structural adhesive joint. Metals and organic materials are able to produce large amount of acoustic emission when they are stressed but it is hard to interpret the generated signals. In this work it is tried to identify different parts of the damage process of the adhesive joint when it is subjected to tensile stress, as well as to establish an adequate relation with the emitted acoustic signals. Tensile tests of bonded joints were carried out in a universal testing machine. Four transducers were placed in parallel on the specimen to collect the acoustic emission signals. The adherent material was aluminum anodized with phosphoric acid at low voltage. The adhesive was a DGEBA epoxy resin.

Keywords: adhesive joint, damage process, mechanical characterization, acoustic emission

1. Introduction

The AE technology has been gradually developed for metallic materials in order to detect and to locate sound sources. The acoustic signals of these materials can provide information about the emission source and the internal structure of the material. However, the study of organic materials and composite materials is still under development, so this work tries to analyze the influence of some variables when this technique is applied to characterize the mechanical behavior of an adhesive joint formed by metallic and organic materials.

Adhesive joints can be examined by non-destructive evaluation techniques. The purpose of these techniques is to detect defects in the adhesion and ensure the efficiency of the adhesive joint under stress conditions. These are some of the non-destructive methods that could be used in the study of adhesive joints: ultrasonic inspection, X-ray radiography, neutron radiography, acoustic emission and thermal or infrared inspection. The last two methods are not sufficiently developed so that a systematic study applied to structural joints is proposed in this paper.

2. Experimental

The adhesive used in this work was a DGEBA epoxy resin (Hysol 9466) provided by Loctite. The adherent material was aluminum anodized with phosphoric acid at low voltage. Two sizes of the adherents were:

- Standardized size according to ASTM D 1002: 101.6x25.4x1.6 mm³. Adhesion area of 12.7x25.4 mm². (Figure 1)
- Non-standardized size: 500.0x25.4x1.6 mm³. Adhesion area of 150.0x25.4 mm². (Figure 2)
Tensile tests of bonded joints were carried out in a universal testing machine IBERTEST ELIB-50/W. The test speed for the standardized adhesive joints was 0.1 mm/min and 0.3 mm/min for the non-standardized joints. For these joints the speed was increased at 3.0 mm/min tests after crossing the elasticity limit of the adherent.

Four transducers (VS150L) were placed in parallel on the specimen to collect the acoustic emission signals (Figures 1 and 2). It was expected that two guard sensors would allow to discriminate the events that occur within the adhesive bond. The other sensors were configured as signal sensors. The acoustic emission equipment was an AMSY-5 model of Vallen System equipped with different types of filters. Pencil lead, 2H of 0.3 mm in diameter was broken in the calibration tests. The speed of the sound through the materials was calculated from those calibration tests. Some parameters were analyzed to obtain results according to the characteristics of AE. The optimized AE parameters are shown in the Tables 1 and 2.

### Table 1. Acquisition parameters for the Acoustic Emission

<table>
<thead>
<tr>
<th>Sample rate</th>
<th>Sample per TR-Set</th>
<th>Duration Discrimination Time (DDT)</th>
<th>Reset Time</th>
</tr>
</thead>
<tbody>
<tr>
<td>5000 MHz</td>
<td>2048</td>
<td>50 µs</td>
<td>0.4 ms</td>
</tr>
</tbody>
</table>

### Table 2. Technical parameters of Acoustic Emission

<table>
<thead>
<tr>
<th>Threshold (THR)</th>
<th>Filter (low and high)</th>
<th>preamplifier</th>
</tr>
</thead>
<tbody>
<tr>
<td>VS150-L</td>
<td>Signal channel</td>
<td>Gain</td>
</tr>
<tr>
<td>40 dB</td>
<td>95 and 1450 kHz</td>
<td>95 and 850 kHz</td>
</tr>
</tbody>
</table>

### 3. Results and Discussion

#### 3.1 Standardized adhesive joints
Several calibration tests were carried out for certifying the proper response of different sensors. The calibration tests were performed to determine the speed of the sound through the materials of the adhesive joint, but the speed value depended where the calibration was made. Therefore, the speed of the sound was determined by approximation in order to obtain the most accurate value. The value proposed for the calibrate sound into the material was 7.03 mm/µs and, from this value, the FHCDT parameter was determined (FHCDT=0.026).

Basic analysis of acoustic emission are presented in the graphs of Figure 3. The graph on the left (events vs. time) shows that the emission begins when the test duration is at ~120 s. The graph on the right shows the location of events (green dots) and the stress (purple line) versus time. In this graph the deterioration process can be seen following the location of the events at different times of the test. The position of the signal sensor can be identified in the left axis with the numbers 1 and 2.

Figure 4 represents the force vs. elongation of the adhesive joint during the test. The curve shows a first linear portion, which maintains the proportionality until the middle of the test (force: 1.60 kN and time: 310 s) that corresponds to an elongation of 0.6 mm and at 60% of break stress. From that point the deformation is non-linear and it could be considered partially irreversible.
The number of events is very small before the proportional limit. However, the number of events increases from the proportional limit until the catastrophic failure.

All the information collected during the test allows dividing the deterioration process of the adhesive joint in five parts:

1. Base zone or blue zone: it is defined by the absence of events
2. Initial zone or green zone: from the first events to the proportionally limit (60% of break stress)
3. Intermediate zone or yellow zone: between the proportionally limit and the high emission area (99% of break stress)
4. End zone or red zone: the area between high emission and failure. In this area, very significant emissions take place indicating that the failure of the adhesive joint is near. Moreover, the events are located throughout all the range of values of X-Loc.
5. Breaking or purple zone: corresponding to the break moment (100% of break stress)

The different zones discussed above can be observed in the Figure 5:

Figure 5: Graph of location and stress vs. time. Several zones of the deterioration process are highlighted with different colors.

Figure 6: Graph of cumulative events versus time. Several zones of the deterioration process are showed.
The graph of cumulative events versus time can also be used to identify the different zones defined above. The second area defined in this graph includes the second and the third zones defined by the previous criteria.

![Graph](image1.png)

**Figure 7:** (left) AE event rates versus time. (right) graph of events localization (●) and force (□) versus time

### 3.2 Non-standardized adhesive joints

In this part of the study the adherents had a length of 50.00 cm and the adhesion zone had a length of 15.00 cm. The optimum speed was 3.50 mm/µs with a FHCDT of 0.18 ms.

The proportional limit was detected at time of 550 s (3 mm of elongation) when the force was 5.4 kN (49% of break stress). In addition, a high increase of acoustic emission was detected at a time of 610 s (see the high emission peak in Figure 7), which corresponds to a 93% of break stress.

![Graph](image2.png)

**Figure 8:** Graph of location and stress vs. time. Several zones of the deterioration process are highlighted with different colors.

The information collected during the test lets define different parts of the deterioration process as has been done with the standard samples (Figure 8).
1. Base or blue zone: in which no emission occurs
2. Initial or green zone: where little emission is observed
3. Intermediate or yellow zone: there is a greater emission due to the appearance of peaks with the highest number of events. The situation of events is preferentially located on either side of the adhesive joint. The beginning of this zone can be associated with the yield limit corresponding to the 49% of break stress
4. End or red zone: it lasts from the highest peak of events (93% of break stress) to the catastrophic failure of the adhesive joint
5. Breaking or purple zone: it corresponds to the break moment

Figure 9 shows the cumulative events versus time. As was proposed for the case of standard adhesive joints four zones can be defined according to the turning points of the graph. The first area defined by this criterion would include the two areas defined with the first criteria.

![Figure 9: Graph of cumulative events versus time. Several zones of the deterioration process are shown.](image)

4. Conclusions

The deterioration process of the adhesive joints studied in this work was followed by acoustic emission. Some specific zones were observed:

- Safety zone: that is defined by a first part without events and a second part with low location of events. This zone extends to the proportional limit (50-60% of the break stress). In this zone the acoustic emission occurs preferentially in areas far from the center of the adhesive bond.
- Alarm zone: that is extended from the proportional limit to the high emission area prior to the final break (93-99% of the break stress). In this zone the acoustic emission is high and it preferentially occurs on one side of the adhesive area.

The break of the adhesive joint becomes imminent at higher stress and it is characterized by high levels of acoustic emission, which appears also in the center of the adhesion area.

The zones defined in this research let establish the criteria to predict the failure of systems with polymeric adhesive and metallic adherent when the failure processes is followed by the acoustic emission technique.
References


