Quality assessment of CFRP bonded structures with active thermography techniques

Bastien EHRHART *, Udo NETZELMANN, Günter WALLE, Bernd VALESKE

Fraunhofer Institute for Non-Destructive Testing (IZFP), Saarbruecken, Germany
bastien.ehrhart@izfp.fraunhofer.de, udo.Netzelmann@izfp.fraunhofer.de, guenter.walle@izfp.fraunhofer.de, bernd.valeske@izfp.fraunhofer.de

Abstract. Adhesive bonding for lightweight application, especially with carbon fiber reinforced polymers (CFRP) materials regarding aerospace application has a large development potential. Requirements involve a strict control of the bonding process to ensure the quality of the assembly. Several factors may however influence the adherent surface prior to bonding and so induce weak adhesive bond performances. If NDT inspections are involved in the defect detection for the bondline (delaminations, disbonds, porosity, voids, etc.), their potential is limited. They are not capable of assessing the adhesion quality of bonded structures yet.

This paper presents work performed with active thermography for the extensive characterization of CFRP structures before and after bonding operations. Multispectral thermography with optical excitation has been used to characterize CFRP adherent surfaces prior to bonding and determine the presence of common contaminations (water, release agent). At the same time, another set-up involving a standard active thermography with pulsed excitation has been investigated for the characterization of contaminated bonded structures. This second approach presents an additional challenge since the contamination itself is not detected directly anymore but instead, the induced loss of adhesion performance via the influence of thermal resistances. To help appraise this challenge, simulations based on material behavior due to the contamination and their effects on the thermal conductivity of the material are presented.

Keywords: active thermography, composite, moisture, contamination, adhesive bond

1. INTRODUCTION

Adhesive bonding for lightweight application, especially with carbon fiber reinforced polymers (CFRP) materials regarding aerospace application has a large development potential. Requirements involve a strict control of the bonding process to ensure the quality of the assembly. Several factors may however influence the adherent surface prior to bonding and so induced weak adhesive bond performances. The potential of NDT methods is limited to the defect detection in the bondline (delaminations, disbonds, porosity, voids, etc.), and adhesion quality can not be assessed yet [1].

A novel approach as a part of so-called extended-NDT aims at characterizing the chemical-physical properties, which are more directly correlated to the performances of bonded structures. For this purpose, NDT methods and standards characterization techniques out of the laboratory shall be adapted. The characterization is done either prior to bonding on the substrate surface, or after bonding, from the complete adhesive bond [2], [3], [4].
The research presented in this paper focuses on active thermography for the characterization of CFRP structures before and after bonding. Although multi-spectral thermography with optical excitation has been used to characterize CFRP adherent surfaces and determine the presence of common contamination (i.e., water, release agent), this paper will only treat the case of moisture contamination in CFRP. A second set-up involving a standard active thermography with pulse excitation has been investigated for the characterization of contaminated bonded structures. In this case, the focus is set onto the detection the induced loss of adhesion performance via the influence of thermal resistances. The thermal resistance model is pointed out with simulations illustrating the contamination effects on the thermal conductivity of the material.

2. ACTIVE THERMOGRAPHY PRINCIPLE

2.1. Principle of the multispectral active thermography

For the analysis of surface contamination, a multispectral IR camera has been used. To enable the visualization of the accessible spectral content of the measurement, a fusion image is calculated from the two images the camera delivers in the MWIR and LWIR bands. This procedure is applied to the corresponding frames of the recorded image sequences. The algorithm works on the apparent temperatures according to the calibration curves in the spectral bands. A color space triple \((R, G, B)\) is formed for each pixel according to:

\[
\text{color}(R, G, B) = f \left[ \left( \frac{G_{\text{LW}} - G_{\text{MW}}}{2} \right) + \frac{G_{\text{LW}} + G_{\text{MW}}}{2}, \left( \frac{G_{\text{LW}} - G_{\text{MW}}}{2} \right), f \left( \frac{G_{\text{LW}} - G_{\text{MW}}}{2} \right) + \frac{G_{\text{LW}} + G_{\text{MW}}}{2} \right]
\]

where, \(G_{\text{MW}}\) and \(G_{\text{LW}}\) are the transformations of the apparent temperatures in the mid-wave band and in the long-wave band, respectively, in grey levels. The overall minimum of the apparent temperatures is assigned to grey level 0 and the overall maximum is assigned to grey level 1. Here, \(f\) is a variable factor that allows emphasizing even weak differences between the spectral channels. Equal apparent temperatures in MWIR and LWIR result in grey color (from black to white with increasing temperature), higher temperature in the MWIR shifts the hue to blue and higher temperature in the LWIR shifts the hue to red.

2.2. Principle of the standard active thermography

For the investigations of the adhesive bonded specimens, a thermal testing technique with optical heating and an evaluation technique in the time domain were used. This technique evaluates the correlation of material properties (i.e., weak bond) to the thermophysical properties in order to characterize these material properties. Variations in material properties change the thermal conductivity, specific heat capacity and the density and hence the effusivity of the material is changed. The effusivity \(E\) is defined as follows:

\[ E = \sqrt{\lambda \rho c} \quad \text{with } \lambda: \text{thermal conductivity; } \rho: \text{density; } c: \text{specific heat capacity} \]

The effusivity of a material describes the thermal response of a material to transient surface heating as follows:
$$T(t) \sim \frac{1}{E \sqrt{t}}$$ with T: temperature; E: Effusivity; t: the time

By an analysis in the time domain and a normalization procedure it is possible to find measurement values which are inversely proportional to the effusivity of the material. In this way, changes in the material correlated to the effusivity can be measured.

2.3. Experimental set-up

For the surface state inspection, a dual-band QWIP 384 IR camera from Thermosensorik GmbH was used. The camera works simultaneously in the mid-wave infrared (MWIR) around 4.8 µm and in the long-wave infrared (LWIR) around 8 µm. The noise equivalent temperature difference (NETD) is in both bands 25 mK. The detector has a resolution of 384 x 288 pixels with 40 µm pixel pitch (Figure 1). The IR camera used for the inspection of adhesive bonded specimens was a Cedip Silver 420M with a 3.6 - 5.1µm focal plane array and 320 x 256 pixels. The NETD in this case is also 25 mK.

![Figure 1: (a) Setup for pulsed flash thermography. (b) Microscopic view of six detector pixels](source: Fraunhofer IAF)

The excitation is performed using either a flash light lamp with 3.2 kJ pulse energy and about 8 ms pulse duration (Figure 1). Immediately after heating the outer surface of the test object an image sequence of the IR radiation is captured by the IR camera. Alternatively, measurements were performed using periodic heating by an LED array source at 940 nm and subsequent lock-in processing of the image sequences. The LED array then replaces the flash lamp shown in Figure 1. Two arrays, each equipped with 64 single LED lamps, delivered a total optical power output of 40 W.

3. RESULTS

3.1. Characterization of moisture contaminated CFRP surface

Specimens contaminated with different level of moisture were inspected to evaluate the evolution of the thermal properties depending on the moisture uptake. Their contaminated status at the inspection times are recorded in Table 1.

<table>
<thead>
<tr>
<th>Sample conditioning</th>
<th>Weight (in g, after pre-)</th>
<th>Weight (in g, after)</th>
<th>Relative moisture</th>
</tr>
</thead>
</table>
Figure 2 introduces the experimental set-up for the moisture detection tests, as visible with the IR camera (a) and as an output from the infrared camera with the thermal pictures (b).

<table>
<thead>
<tr>
<th>Sample</th>
<th>Condition</th>
<th>Drying)</th>
<th>Conditioning)</th>
<th>Uptake (in wt.%)</th>
</tr>
</thead>
<tbody>
<tr>
<td>1.5.8-UT</td>
<td>Storage in air</td>
<td>23.665</td>
<td>23.664</td>
<td>0.00</td>
</tr>
<tr>
<td>3.13.MO-4</td>
<td>Magnesium-chloride (30 % r.H.)</td>
<td>28.140</td>
<td>28.217</td>
<td>0.27</td>
</tr>
<tr>
<td>3.16.MO-3</td>
<td>Natrium chloride (75 % r.H.)</td>
<td>27.972</td>
<td>28.213</td>
<td>0.86</td>
</tr>
<tr>
<td>3.14.MO-2</td>
<td>Kalium sulphate (95 % r.H.)</td>
<td>28.192</td>
<td>28.554</td>
<td>1.28</td>
</tr>
<tr>
<td>3.15.MO-1</td>
<td>De-ionised water (100 % r.H.)</td>
<td>28.159</td>
<td>28.549</td>
<td>1.38</td>
</tr>
</tbody>
</table>

- Measurements before moisture loading:

Samples MO-n, n=1 to 4, were measured before moisture loading. At a frame rate of 146 Hz, frames were recorded after the beginning of the flash discharge over a total time of 1.8 s.

For evaluation of the cooling curves, the average region of interest (ROI) data from six frames immediately before firing the flash were averaged in time and the result was subtracted from all following ROI averages of the frames. All operations were performed separately for the MWIR and the LWIR spectral range. The resulting thermal contrast curves were plotted in a double-logarithmic plot which confirmed widely the expected thermal contrast decaying proportionally to $1/\sqrt{t}$. Slight differences could be observed between the curves, which may be due to inhomogeneous illumination or variations in the material parameters of the dry samples.

In order to show slight variations more clearly and compensate for shot-by-shot variations of the flash intensity, each decay curve on the sample was normalized to that of the corresponding reference sample in the same measurement. The contrast curves have a characteristic shape as a function of time. There are differences in the relative contrast depending on the spectral range.

- Measurements after moisture loading

The measurements before moisture loading were repeated after the moisture loading process. Again, typical contrast decay curves do show only slight variations. The contrast
curves were plotted normalized to the respective reference sample in order to show smaller effects, as done previously. In contrast to the dried samples, the results of the loaded samples show an increase of the relative contrast with decreasing moisture loading in both spectral ranges. This is shown in Figure 3.a, where the relative contrast at a fixed time of \( t = 0.5 \) s was plotted for the four samples. The relative contrast is not constant before the moisture loading, which indicates some variations in the sample properties even without moisture influence. In order to work out the pure moisture influence, for each sample the relative contrast at \( t=0.5 \) s was further normalized to the corresponding contrast before loading. Figure 3b shows the signal change as a function of the measured weight increase.

With increasing moisture uptake, the relative thermal signal decreases monotonously. This is in accordance with an expected increase of the thermal effusivity of CFRP with moisture uptake, as obtained from a mixing model shown in Figure 4.

As the thermal contrast after pulsed heating is inversely proportional to the effusivity, the contrast should decrease with water uptake. This is also observed experimentally in both spectral regimes. Over the studied range of moisture uptake, the effect is larger in MWIR than in LWIR. Quantitatively, the effect of 1.3% effusivity change for one \( \text{wt.}\% \) water uptake as calculated from the mixing law is smaller than the observed effect of 6% or 7.5% per percent water uptake for MWIR and LWIR, respectively. The extrapolation of these curves does not exceed 1 for a ‘dry-state’ in the LWIR, whereas a higher value than one is already given for 0.27% weight increase in the MWIR. The
discrepancy with the thermal model and the latter observation may point to an additional non-thermal additional effect of the water, e.g., due to water IR emission lines of species at the surface. This would explain the different behavior in the spectral ranges. Heating induced water desorption processes are also known to influence the apparent thermal properties. Finally, the mixing model assumed in the calculation may not be appropriate. The observed dependence of the relative contrast on time after excitation may indicate a depth distribution of the moisture.

3.2. Characterization of weak adhesive bond

In the case of the weak adhesive bond, the samples have been prepared by using similar moisture surface contaminated specimen (ref. to Table 2) for the bonding operations. The moisture exposed laminate and a reference untreated one undergo the bonding process and are then inspected. The goal is to evaluate the effect of the moisture contamination on the resulting mechanical performances of the bondline instead of the amount of moisture present prior to bonding.

Table 2: Weight data of adhesive bonded moisture contaminated specimens

<table>
<thead>
<tr>
<th>Sample</th>
<th>conditioning</th>
<th>Relative moisture uptake (in wt.%)</th>
</tr>
</thead>
<tbody>
<tr>
<td>8.1 UT-3</td>
<td>storage in air</td>
<td>0.00</td>
</tr>
<tr>
<td>2.9 MO 1-8</td>
<td>magnesium-chloride (30 % r.H.)</td>
<td>0.45</td>
</tr>
<tr>
<td>2.7 MO 2-8</td>
<td>natrium chloride (75 % r.H.)</td>
<td>0.81</td>
</tr>
<tr>
<td>2.2 MO 3-8</td>
<td>kalium sulphate (95 % r.H.)</td>
<td>1.13</td>
</tr>
<tr>
<td>1.3 MO 4-8</td>
<td>de-ionised water (100 % r.H.)</td>
<td>1.25</td>
</tr>
</tbody>
</table>

Figure 5: (a) Thermographic image of sample 1.3 MO 4-8, 0.5 s after heating (image field: 48 mm x 48 mm); (b) signal curves (temperature coded in gray values vs. frames number) along the line; (c) measurement results for the five samples

Figure 5.a shows the temperature distribution (gray value coded) at surface of specimen 1.3 MO 4-8, 0.5 s after pulsed heating. The temperature curve (in gray values – arbitrary units) along the red line displayed in

Figure 5.a is presented for three samples. The green curve shows the result for the reference sample 8.1 UT 3, the yellow curve indicates the result of the sample 2.7 MO 2-8 (0.81 wt.% moisture) and the red curve shows the result of the sample 1.3 MO 4-8 (1.25 wt.% moisture).

Figure 5.c finally introduces the results of the five samples inspected along with their relative error determined over 5 measurements.
At 0.5 s after optical excitation, the sample 1.3 MO 4-8, bonded with 1.25 wt.% moisture delivers the lowest measurement value indicating the highest value of effusivity. The lowest value of effusivity is measured for the reference sample 8.1 UT 3. With this measurement at 0.5 s, the surface near region is evaluated.

It can be assumed that the samples at the instant of the inspection are still loaded by moisture because the moisture enhances the effective effusivity of the material due to the fact that specific heat capacity of water is nearly four times higher than that of CFRP material. As also discussed before, it can be estimated that 1 wt.% moisture uptake of a CFRP sample increases the effective effusivity in the amount of about 1 %.

Further evaluations were done at later time, 5 s after heating. At this time, the response of a weak adhesive bond layer is expected due to the heat diffusion coming from a deeper zone as the time increases. The results are presented in Figure 6a, b, c in a similar way to Figure 5. In this case, similar observations as after 0.5 s could be made: only the effusivity of specimens 3-8 and 2-8 seem to have increased. Together, these measurement results would lead to the conclusion that the effect of moisture content in the samples seems to be dominant and go along the discussions in the previous section 3.1.

3.3. Approaches by modeling

In order to simulate the effects of moisture on the measurement results, model calculations were performed based on instationary heat diffusion theory. A plane geometry was assumed with a pulsed heating at specimen surface. It was furthermore assumed that the CFRP plate at the left side has a normal effusivity and the plate at right side has a 1% higher effusivity due to water content.

In Figure 7, the temperature distribution at the sample surface (along a line at the sample surface in x-direction) is presented 0.5 s after heating (Figure 7.a) and 5 s after heating (Figure 7.b). The higher effusivity due to moisture leads to a lower temperature at the sample surface in both cases. This result corresponds to the measurement results where the same behavior of the measurement values could be found (Figures 5.b and 6.b).
In order to study the influence of a weak bond due to moisture contamination, another simulation was performed. It was assumed that a weak adhesive bond decreases the heat conductivity in the adhesive layer. According to this assumption, the left side of the bonded CFRP is considered having normal heat conductivity while the right side only has a value of 75% of that from the left side. The result of the model calculation is shown in Figure 8. It can be observed that the lower value of the thermal conductivity at the right side leads to a significant higher measurement value, indicating a lower effusivity.

Such result was not observed in the measurements of the contaminated bonded samples in comparison to the reference sample. In contrast, lower measurement values were obtained on contaminated samples in comparison to the reference sample. It was hence concluded that the samples are either loaded by moisture content or another effect raises the same conclusion and is dominant in the measurement results.

Additional experiments aiming at verifying that the specimens were not loaded by moisture anymore were conducted recently. All specimens were dried out for 6 weeks in an oven with air circulation at 70 °C until stabilisation of their weight. The mean value observed in weight loss was about 0.25 wt.% for all specimens. This common weight loss indicates that no specimen was loaded more than another one. The results of the thermographic analyse revealed accordingly no other major deviations from the results presented in Figure 6.c. The assumption that an unknown chemical or physical ageing effect occurred in the moisture contaminated laminate is therefore even supported.
4. CONCLUSION

It was shown that the effect of moisture uptake can be detected by thermographic measurements. The detection limits for moisture uptake (about 0.5 wt.% increase) are better than that expected from calculations. A MWIR infrared camera may be a somewhat better choice for detecting small moisture uptake. However, in any case careful referencing of the measurements will be necessary to obtain a reliable result.

In the case of bonded specimens after moisture contamination, the measurement results showed no indication of a weak adhesive bond among the contaminated samples in comparison to the reference sample. The observations made are in contrast suggesting that the samples are still loaded with moisture. Based on assumptions regarding the possible increase of effusivity relatively to a reference sample (without moisture), model calculations have shown that a moisture content of 1 wt.% produces measurement signals which are qualitatively comparable to the thermographic measurement results from the contaminated samples.

Finally, if a contamination in the adhesive layer leads to lower thermal conductivity, then higher measurement values i.e. lower effusivity values would be expected from such a sample in comparison to those from a reference sample with a clean adhesive bond, as illustrated by model calculations. From the latest measurement results and model calculations, we can conclude that the influence of moisture contamination prior to bonding has generated other significant ageing effects in the composite laminate, since they lead to similar observations as the ones that could have been observed if the laminate were still loaded with moisture.

5. ACKNOWLEDGMENTS

The research leading to these results has received funding from the European Union's Seventh Framework Program (FP7/2007-2013) under grant agreement n° ACP0-GA-2010-266226 (ENCOMB, Extended Non-Destructive Testing of Composite Bonds).

6. REFERENCES


