

Laser Adhesion Test for Adhesive Bonded CFRP Structures

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Abstract. The increased use of carbon fibre reinforced plastics (CFRP) and the constant urge for lighter structures make of adhesive bonding an assembly technology with a great potential for high-loaded structures in the aeronautic industry. A prerequisite for such an application is that the bond quality of the adhesive joint can be controlled in a non destructive way. Non-destructive testing (NDT) methods exist for the characterisation of defects like pores, delamination or debonding in adhesive bonds. However, these NDT methods for conventional “defectoscopy” do not allow the evaluation of the adhesion strength of an adhesive bond, what currently prevents a wider use of this assembly technology. Laser Proof Tests in the opposite have been identified as high potential techniques to assess the bond mechanical performance in a non-destructive way (provided the bond has the required level of performance), by simulating a precise tensile effort with shock wave at the bondline-substrate interface.

This paper presents a study of the laser adhesion test from CNRS PPrime institute on the targeted application of adhesive bond test. CFRP bonded samples are prepared in a specific way and characterised by NDT techniques to assess the absence of any detectable defect. Their bond strength is evaluated by mechanical destructive tests. Further on, the bonded samples are tested by Laser adhesion test to evaluate the power and settings of the laser technique required for the bond test. All results are then correlated to estimate what key parameters shall be readjusted.

1. INTRODUCTION

Weak adhesive bonds are from the main showstoppers in the matter of the development of structural adhesive bonding. Several literature sources state that even if defects can clearly be detected in adhesive joints of composites, no statements can be made regarding the quality of the adhesion, its strength or its properties [1–3]. Indeed, no evaluation of the minimum bond performances and by extension no characterisation of weak adhesive bonds is yet by any NDT method available. Based on this observation, the approach ‘Extended NDT’ whose scope is to use and develop characterisation and NDT methods to assess the

performances of adhesive bonds have been introduced over the last years [4–6]. Among the identified Extended NDT method, Laser Proof Tests have been identified as high potential techniques to assess the bond mechanical performance by simulating a precise tensile effort with shock wave at the bondline-substrate interface [7–9].

A feasibility study has been conducted with help of samples manufactured with surface contamination for the generation of weak adhesive bonds. These samples have been tested for the evaluation of the potential of the laser shock adhesion test technique.

2. WEAK ADHESIVE BONDS

2.1. *Generation of weak adhesive bonds*

Generating weak adhesive bond using CFRP substrate is a first challenge in this feasibility study. This task is only poorly present in the literature, in opposition to studies on ageing of adhesive joints, and similar topic but with metallic substrates. Prior to the introduction of the production steps of the weak adhesive bond sample, the criteria for such an adhesive bond state shall be introduced. Under weak bond is called a defective adhesive bond presenting the following characteristics established in the literature [10], [11]:

- the strength measured by mechanical test must be below 20% of the nominal bond strength,
- the mode of failure must be adhesive in type (i.e. purely at the interface between the adherend and the adhesive),
- the weak bond must be undetectable from normal bonds with classical NDT techniques.

To match these criteria and produce relevant sample, the literature and recent experiences mention the key parameters as being surface roughness (mechanical interlocking) and contamination (chemical modifications). In contamination itself, a whole variety of products has been tested, from original organic substances to aerospace linked fluids (kerosene, de-icing and hydraulic fluids). In this study, the method to produce weak bonds has been chosen based on results in literature [10], [12] and in recent research project in order to approach realistic challenges of the composite production. The technique consists in applying a thin layer of a silicon-based mould release agent (Frekote 700 NC) on the adherent surface prior bonding. This type of contamination appears in cases of moulding of CFRP and prevents any efficient further operation on the composite surface, requiring hence a surface activation treatment and cleaning [13].

2.2. *Manufacturing Process*

Composite samples were manufactured with the 180 °C curing temperature system T800/M21 (Hexcel Composite) with 6 plies in orientation (0,0,90)_S in order to reach a thickness of 1,5 mm for a single cured panel. Panels are prepared with a dry peel-ply to master surface cleanliness and roughness as recommended in Airbus processes.

The contamination by mould release agent Frekote 700-NC (Loctite) was brought on the surface later on by dip-coating, a process of controlled immersion for a better homogeneity of contaminant. In this feasibility study, no graduation of adherence is targeted, only extreme cases of reference samples and weak bond. The contamination is therefore applied with a high concentration of 20 % of Frekote diluted in hexane.

After the contamination of one adherent surface, the bonding operation is performed with an epoxy adhesive film FM300 and another per-cured sample in the reference state. The contamination is thus only present on a single adherent surface within the adhesive

bond sample. This ‘secondary bonding’ process -the assembly of two pre-cured composite- is performed in an autoclave at 180 °C.

The final step of manufacturing was to prepare the samples for the characterisation post-bonding to evaluate:

- i. the bonding operation success by NDT inspection,
- ii. the mechanical performance by determination of fracture toughness and,
- iii. the potential of laser shock adhesion test.

The samples are cut out the bonded panels so that a single panel provides samples for the tests i, ii and iii and enable an ideal comparison. Those tests are introduced in the following part 2.3 and 3.2.

2.3. Characterisation of Samples

Prior to the bonding operations, the first panels of M21/T800 were successfully inspected by NDT to ensure the standard quality required. No defect was revealed by the ultrasonic scans.

After contamination with the Frekote solution, the sample surfaces were characterised by X-Ray photon spectroscopy (XPS) to assess the presence of silicon traces. The results of this test have revealed a high Si at.% on the composite surface exposed to the Frekote solution (see Table 1), which is characteristic of a successful silicon contamination.

Table 1 – XPS results for the reference and the contaminated samples

	C (at.%)	O (at.%)	N (at.%)	S (at.%)	Si (at.%)
Reference	72,8	15,9	10,1	1,0	0,3
Frekote	51,2	27,8	2,5	0,2	18,3

The contaminated panels were then bonded and again inspected by ultrasonic techniques. The echo of the bond line and of the backwall for the panel LA (Reference) and the panel LB (contaminated with Frekote) were defect-free according to the US C-scans of both panels. No differences between the samples LA and LB could be observed.

To enable correlations between the different characterisation methods, the bonded panels were tested mechanically to evaluate the bond line strength.

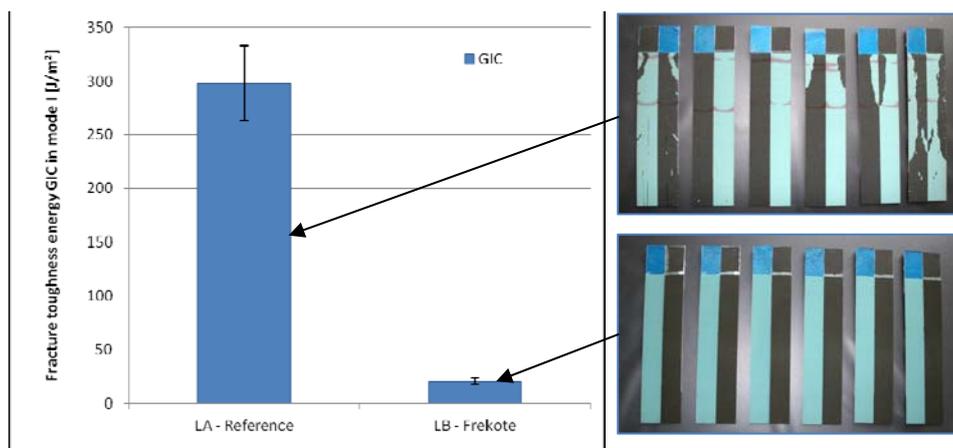


Figure 1 – Fracture toughness energy G_{1C} of LA and LB samples with according sample rupture profiles

A double cantilever beam test G_{1C} was performed with 6 samples of each bonded state (see Figure 1). The fracture toughness results are relatively low in term of performance, which may be attributed to the use of materials near the end of their worklife. The rupture of the adhesive bond is mainly adhesive for both samples, what suit the low performances observed. The performances are in general however still highly influenced by the presence

of contaminant (LB – Frekote series), so that the LB performances reach 7% of the reference adhesive bond strength.

The observations made with NDI and destructive characterisations fulfil the conditions of a weak adhesive bond described in part 2.1.

3. LASER ADHESION TEST (LASAT)

3.1. Laser adhesion test principle

The laser shock wave technique consists in a high power laser irradiation of a target material surface. When focused on a material, it transforms the surface into a dense plasma gas. The expansion of the plasma created on the material surface produces a shock wave (see sketch in Figure 2a). This incident shock wave propagates through the thickness according to properties depending on the multilayer material characteristics and geometry (see in Figure 2b, step 1). When reaching the sample back face, the reflection of this incident shock wave creates a release wave propagating backward. When crossing the incident unloading wave coming from the front face and initiated by the end of the loading (see in Figure 2b, step 2 and 3), it leads to local high tensile stresses which could damage the material if the local damage threshold is over passed. A high level of damage is characterized by the spallation of the material target.

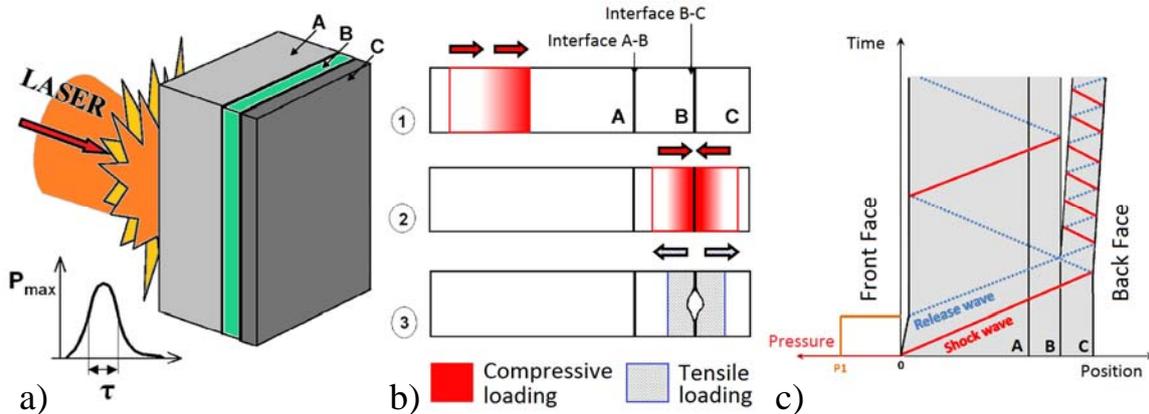


Figure 2 – a) Sketch of the laser shock wave method. b) The wave propagation history c) Time/position diagram showing a 1 dimension shock wave propagation history in case of spallation

Indeed, the resulting tensile stress level is directly linked to the laser shock amplitude whereas its location mainly depends on the material properties and the pulse characteristics. As shown on the Time/position diagram presented in Figure 2c, the laser pulse duration determines the position of the maximum tensile stress. For a given material and a given geometry, a short pulse (10 to 50 ns) would locate the first tensile stresses close to the back face (about 20 to 250 μm), when long pulses (100 to 300 ns) could locate the stresses deeper inside the target (about 500 to 1500 μm). In case of laser adhesion test, the optimized case occurs when the tensile stresses are located around the bonded interface. Then, the laser energy can be tuned to evaluate the damage threshold of this interface by changing the stresses amplitude. Therefore, the bonding quality can be controlled.

3.2. Laser test campaign

Experimental setup & configuration

The bonded composite samples are first conditioned using an aluminum coating on the front face, to enhance the interaction with the laser. Then, they are shocked with various laser energy levels to produce different levels of inside damage. A Nd.YAG laser (1054 nm wavelength) whose energy is tunable in the range [0 – 20 J] and whose pulse duration is about 30 ns was used to produce the shocks (see Figure 3). The laser parameters measured for each sample tested are presented in Table 2. After the shock, the samples are analyzed with two different diagnostic setups (see Figure 3). Optical micrographies of sample cross sections have been performed to observe the inside damage, and Interferometric Confocal Microscopy (ICM) on the sample back face was also used to quantify the surface height variations.

Table 2 – Laser shock parameters used on bonded composite samples

Target reference	Conditioning	Focalized diameter	Pulse duration	Laser energy	Intensity (GW/cm ²)
LB-b-3	Coating & water confinement	4 mm	24,67 ns	7,90 J	2,55
LB-b-4	Coating & water confinement	4 mm	26,66 ns	3,28 J	0,98
LB-b-5	Coating & water confinement	4 mm	25,97 ns	0,70 J	0,21
LA-a-2	Coating & water confinement	4 mm	26,66 ns	7,75 J	2,31

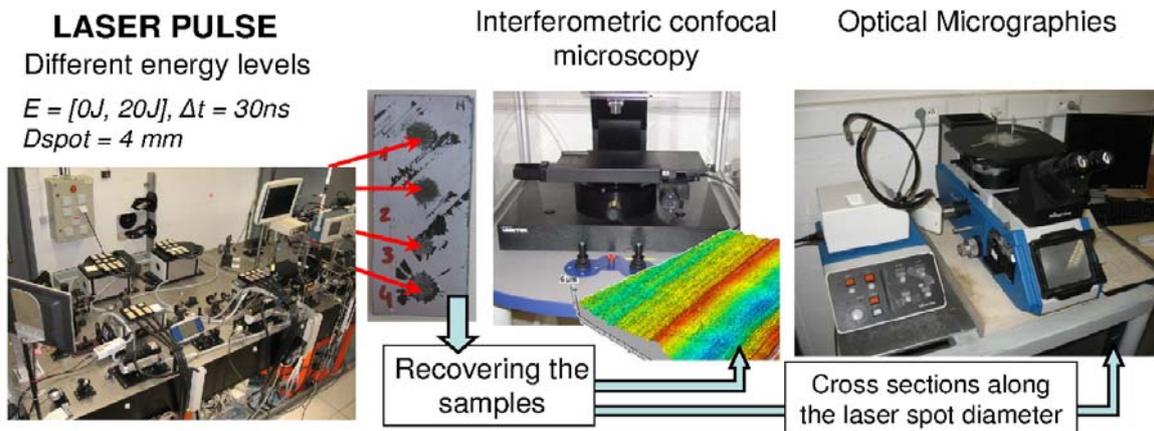


Figure 3 – Sketch of the experimental procedure followed to test and analyze the bonded composites

Laser shock results

A representative overview of damage resulting from laser shock on a bonded composite is given in Figure 4. Three main types of damage can be observed:

- i) *Transverse cracks between the fibers and the matrix, through the ply thicknesses:* these cracks are mainly due to the flexural component of the laser loading.
- ii) *Delamination between the plies:* they are initiated by the high tensile stresses generated by the propagation of the laser induced shock wave inside the composite. Since the delamination occurred between 90° and 0° plies, the back face deformation measured by ICM is oriented in the 0° direction. In these experiments,

the laser irradiation did not yield to spallation of the sample because the stresses were not high enough for that. That is why an elliptical blister can be observed by ICM at the back face. This measurement indicates that the delamination mainly propagated in the 0° direction.

- iii) *Debonding of the bonded interface*: this debonding was possible thanks to the tensile loading propagating from the back face to the front face after the crossing of release waves. Even if some fracture energy was dissipated inside the composite, enough energy remained to initiate the debonding.

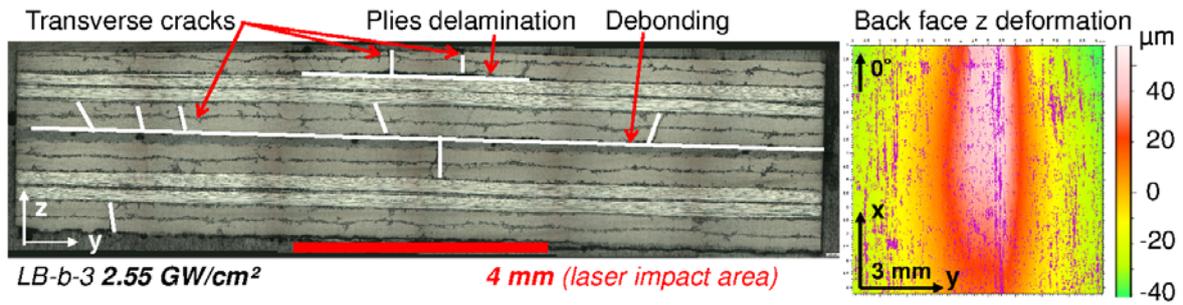


Figure 4 – Representative micrography and ICM height measurements of damage resulting from shock wave propagation inside a bonded CFRP with a very low adhesion rate LB-b-3

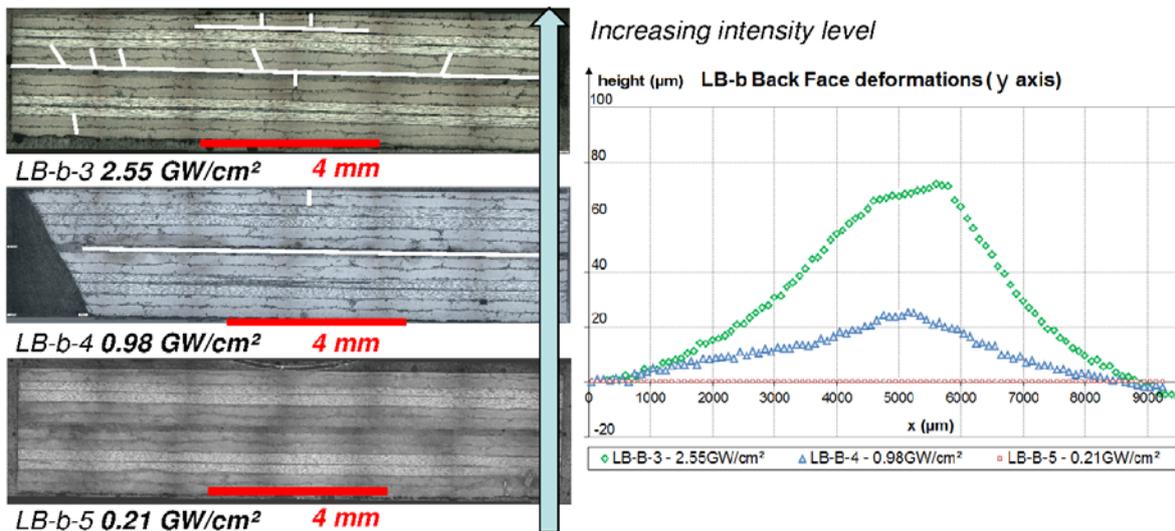


Figure 5 – Correlation between the laser intensity and the damage extent into the bonded composite material observed by micrographies and measured by ICM.

Results obtained with various intensity laser shocks are presented in Figure 5 with the corresponding micrographies and ICM measurements. In the Figure 5 chart, the ICM back face z deformation measurements were reduced to the y -axis profiles. This way, the correlation between the laser intensity and the back face deformation of the bonded sample can be evidenced. The back face z deformation also agrees with the damage extent observed in the micrographies. The LB-b-4 sample presented in Figure 5 is the most interesting one. Indeed, the composite part of the assembly remained unharmed after the laser shock wave propagation. This is due to the fact that the laser intensity used was low enough for the induced tensile stresses to be under the damage threshold of the CFRP composite. Nevertheless, these tensile stresses were high enough to open the bond line of the sample, whose damage threshold was low enough compared to the composite one. With the three shock results presented in Figure 5, it can be concluded that the damage threshold of the bond line tested is between 0.21 GW/cm^2 and 0.98 GW/cm^2 and the composite one is in the range $[0.98 - 2.55 \text{ GW/cm}^2]$.

4. DISCUSSIONS

In Figure 6a, the reference sample LA-a-2 is presented as well as the weak bond sample LB-b-3. The same laser shocks were performed on both samples and the resulting damage has been studied by cross section observations and ICM. In the case of the reference bond, the laser shock propagation did not lead to a debonding whereas the same shock induces the damage in the weak bond assembly. Nevertheless, in both cases, the composite part of the assembly on the back side was delaminated by the high tensile stresses. This observation is confirmed by the ICM measurements. Indeed, the back face z deformations measured of the two samples are really close to each other since the composite damage are also close to each other. This composite damage is due to the fact that the maximum of tensile stress is generated close to the sample back face because of the laser configuration which was not enough adapted to the system tested.

The development of the laser adhesion test requires thus the optimisation of the laser parameters, especially the pulse duration, in order to localize the tensile loading at the bonded interface and so, avoiding the creation of damage in the composite parts. Once the laser parameters shall be optimized, ICM may provide an evidence of debonding by very different back face z deformation profiles.

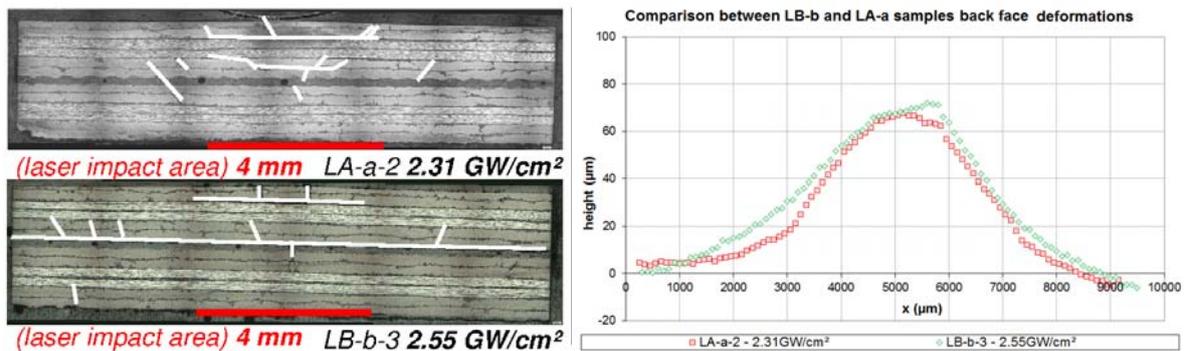


Figure 6 – Comparison between a correctly bonded assembly (LA-a) and a weak bond sample (LB-b) by use of cross section observations and ICM measurements

5. CONCLUSION

The feasibility study performed has shown valuable results regarding the process of generating on purpose weak adhesive bond through surface contamination with release agent. The non-destructive characterisation did not detect differences between the reference and the contaminated specimen, while the mechanical test performed have proven an adhesive bond performance of only 7% of the reference sample, due to the adherent pre-bonding surface contamination. Relevant samples could hence be manufactured for the development of the laser adhesion test method.

The laser shocks applied to the provided samples have shown that it was possible to discriminate two different level of adhesion. The laser configuration used on the bonded specimen was however not exactly matching the needs to manage a bond test without generating composite damages. Indeed, the laser pulse duration induced the maximum tensile stresses to be located inside the composite instead of in the adhesive bond line. This results in a damage inside the composite parts, which has to be avoided for the laser adhesion test to be efficient.

Further work will be performed regarding the preparation of new sample with a wide range of adhesive bond performances, but also regarding the laser source and shoot parameters.

6. REFERENCES

- [1] R. D. Adams et P. Cawley, « A review of defect types and nondestructive testing techniques for composites and bonded joints », *NDT International*, vol. 21, n°. 4, p. 208-222, Août 1988.
- [2] R. D. Adams et B. W. Drinkwater, « Nondestructive testing of adhesively-bonded joints », *NDT & E International*, vol. 30, n°. 2, p. 93-98, avr. 1997.
- [3] D. J. Baumann et D. U. Netzelmann, « Zerstörungsfreie Prüftechniken für Materialverbunde », ISSN 0948-1427, janv. 2003.
- [4] B. Valeske, C. Bockenheimer, et R. Henrich, « New NDT Approach for Adhesive Composite Bonds », *NDT APPLICATIONS 1*, p. 203-209, 05-déc-2008.
- [5] C. Bockenheimer, « Novel Approaches to Non-Destructive CFRP Bond Quality Assessment », SAE 2011 AeroTech Congress & Exhibition, 2011.
- [6] B. Ehrhart, B. Valeske, N. Chobaut, M. Sarambe, A. Gendard, et C. Bockenheimer, « Preliminary tests for the development of new NDT techniques for the quality of adhesive bond assessment », presented at the DGZfP Jahrestagung 2011, Bremen, Germany, 2011.
- [7] R. Bossi, K. Housen, C. Walters, et D. Sokol, « Laser bond testing », *Materials Evaluation*, vol. 67, n°. 7, p. 819-827, 2009.
- [8] R. Ecault, M. Boustie, F. Touchard, L. Berthe, L. Chocinski, B. Ehrhart, et C. Bockenheimer, « Damage of composite materials by use of laser driven shock waves », presented at the Proceedings of the American Society for Composites 26th Annual Technical Conference/2nd Joint US-Canada Conference on Composites, Montreal, Quebec, Canada, 2011, p. 14.
- [9] M. Perton, A. Blouin, et J.-P. Monchalain, « Adhesive bond strength evaluation in composite materials by laser-generated high amplitude ultrasound », *Journal of Physics: Conference Series*, vol. 278, n°. 012044, p. 5, janv. 2011.
- [10] P. Marty, N. Desai, J. Andersson, et C. AB, « NDT of kissing bond in aeronautical structures », presented at the 16th World Conference of NDT Proceedings, 2004.
- [11] N. Decourcelle et E. J. C. Kellar, « Development of a methodology to produce samples and ultrasonic techniques for kissing bonds in adhesive joints », *Research Reports for Industrial Members of TWI*, Granta Park, Cambridge, UK, p. 1-12, sept-2009.
- [12] C. Jeenjitkaew, Z. Luklinska, et F. Guild, « Morphology and surface chemistry of kissing bonds in adhesive joints produced by surface contamination », *International Journal of Adhesion and Adhesives*, vol. 30, n°. 7, p. 643 - 653, oct. 2010.
- [13] B. Ehrhart, B. Valeske, R. Ecault, M. Boustie, L. Berthe, et C. Bockenheimer, « Extended NDT for the Quality Assessment of Adhesive Bonded CFRP Structures », presented at the International Workshop SMART MATERIALS, STRUCTURES & NDT in AEROSPACE Conference NDT in Canada 2011, Montreal, Quebec, Canada, 2011.