

# Methods for the Quality Assessment of Adhesive Bonded CFRP Structures -A Resumé

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**Abstract**. Although the aerospace industry already has experience with adhesive bonding on carbon fibre reinforced polymers (CFRP) technology, a great potential for the manufacturing of high-loaded structures exists. A prerequisite for such an application is that the quality of the adhesive bond can be controlled. Various non-destructive testing (NDT) techniques are adequate for the characterisation of defects like pores, delamination or debonding within adhesive bond. There is however so far no NDT technique able to ensure the detection of a weak bond ('kissing bond') and, by extension, ensure the quality of an adhesive bond. This lack remains the major issue set against a wider application of the adhesive bonding technology.

This paper covers the recent activities of several communities and point out the capabilities and limits of most promising techniques being developed on this topic. Many communities have been working on innovative methods, or on the extension of capabilities of existing NDT techniques to provide a solution. Among these communities for instance, the University of Bristol already demonstrate feasibility of the detection of kissing bonds and fatigue in metallic structures and is involved in research projects for CFRP materials. Another approach relies in the use of proof test methods including laser adhesion test, based on high intensity laser short pulse. These laser adhesion tests generate an elastic shock wave that disbonds the weak adhesive bonds, making the defect visible for other NDT techniques (thermography, US scan, etc...). This approach has already been studied extensively and with success on bonded metallic structures, sandwich composite structures, and tend to extend to adhesive bond in CFRP structures.

## 1. INTRODUCTION

Although the aerospace industry already has experience with adhesive bonding on carbon fibre reinforced polymers (CFRP) technology, a great potential for manufacturing of high-loaded structures exists. A prerequisite for such an application is that the quality of the adhesive bond can be controlled. Various non-destructive testing (NDT) techniques are adequate for the characterisation of defects like pores, delamination or debonding within adhesive bond. There is however so far no NDT technique able to ensure the detection of a weak bond ('kissing bond') and, by extension, ensure the quality of an adhesive bond. This lack remains the major issue set against a wider application of the adhesive bonding technology. In the past years, few methods of conventional NDT (mainly ultrasonic) and other techniques are being developed to improve their detection capabilities and so



characterise the adhesive bond quality. The objective of this paper is to introduce a non-exhaustive overview of the different activities and technologies pushed forward for the inspection of adhesive bonding up to day.

#### 2. ADHESIVE BONDING & ITS LIMITATIONS

# 2.1 History and advantages of the adhesive bonding assembly technique

Adhesive Bonding is one of the oldest assembly process used in the history of the mankind [1,2]. However, over time and especially during the past century with the development of the industry, the demand for adhesives increased: new adhesive products were developed such as synthetic resins and polymers [2]. The aeronautic industry, whose history is closely linked to the World Wars, is one of the most pushing industries [3]. Fokker started using adhesive bonding in 1915, showing the way to other manufacturers. In constant technological progress, adhesive bonding also reached the civil aviation where high performances epoxy based resins are nowadays mostly used. As an example, Airbus started bonding in 1972. In the meantime, Airbus has now more than 345 bonding features in the whole aircraft families [4].

Numerous advantages over others processes apply to adhesive bonding [2-7]: homogenous stress distribution, a full automation capability, good enabler for light, strong and even complex structures design, capability of joining two distinct materials, interesting properties in electrolytic and corrosion protection, and also high fatigue resistance and thus longer service lifetime than structures that have to be machined.

#### 2.2 Limitations

Although bonding is a wide spread assembly method in the industry, many limitations still act against a wider application field the whole scientific world is still not capable of explaining how an adhesive adhere [3]. A real **lack of knowledge** exists regarding the mechanisms of adhesion. For this main reason, industrials and mechanicals do not trust this method enough to use it for high loaded, so-called "primary", structures; instead, adhesive bonding is used for "secondary" structure (e.g. spars, stringers, etc.) were a possible failure would not be directly harmful for the integrity of the product.

Many parameters are decisive in the quality of the adhesive bond: it all starts therefore with the bonding processes, the realisation of a proper bonding requires absolute clean surface that have been prepared and that present a good affinity with the adhesive. A good wetability of the adherend surface is also needed for an optimal bonding process. The bonding process including the surface preparation along with the materials handling and the adhesive cure **require a demanding control** [2-3,6]. Those process parameters are essential for the quality of the adhesive bond and their respect is mandatory in order to avoid defects.

Adhesive bonds as any other material part can indeed **encounter defects**. Within defects are here understood delaminations, disbonds, porosity, voids (high volume porosity), incorrect matrix cure, and cracks. After the bonding process, quality inspection is performed and must be able to detect any defects in the final assembled body which is mostly not available for visual inspection [6,7]. As well as for metals products and metal bonded structures, non-destructive evaluation (NDE) is used on adhesive joints for composite materials. The literature reports a large amount of studies about techniques established for the NDE in composites and bonded joints [8]. These methods will not be detailed in this report but will nevertheless be pointed out hereby to illustrate the diversity of techniques available and reported: out of the five major methods used for metal

inspection, only ultrasound testing [8-12] and X-ray radiography [8,10-12] are used for composites due to the difficulties with an inhomogeneous material [8]. Alternative methods such as low-frequency vibration [8] and infrared thermography [8] were therefore developed.

In addition to those previously cited methods, many other techniques are reported [10]: Visual inspection, Leak test, Tap test, Woodpecker, Fokker bond tester, Acoustic emission, and Shearography.

Those methods are successful in the defect detection as underlined by Baumann et al. in the case of delaminations or in general by Valeske et al. and Nottorf et al.

It has however to be highlighted that none of those mentioned techniques, that would be related to as "conventional" NDT methods, is able to assess the performance of an adhesive bond. The literature unanimously concludes that although defects can clearly be detected in adhesive joints of composites, **up to day no statements can be made regarding the quality of the adhesion, its strength or its properties** [8,10-13].

This final limitation of the adhesive bonding technology is from utmost importance. It represents one major stake in the further development of this technology.

To summarize, the quality of the adhesive bond can be defined as the absence of defects, durability in the service environment and mechanical performance. The quality is thus a parameter that goes beyond the defect detection currently available thanks to NDE. In opposition to the quality of other assembly technologies, the quality of an adhesive bond requires that the physical and chemical properties of the adhesion and in the interface with the adherend surfaces could be characterised [13].

## 3. NON-DESTRUCTIVE TESTING METHOD FOR BOND PERFORMANCES

Reviews of the alternative solutions that may also derive from further development of conventional NDE methods (such as ultrasonics) are presented in this chapter.

All presented methods have in common that they rely on mechanical waves for the evaluation of the bond strength, which is itself seen as a mechanical parameter [14]. This mechanical parameter is not affected in the same way depending on its history and damages it undergoes [15]. Hence different parameters such as frequency, signal form, amplitude, etc. of the response signal coming back from the adhesive joint are key criterion for the understanding of the quality of the adhesive joint [16].

Most of the methods presented here are developed so that their detection capability (measuring probe, sensitivity, etc.) is optimised for the characterisation of the adhesive bond.

## 3.1 Ultrasonic (US) methods

#### **Generalities**

Ultrasonics are probably the electromagnetic waves for NDE techniques the most used in the world. Conventional ultrasonic methods such as pulse-echo and through transmission are widely applied for the detection of defects of all kind in all materials type. Despite the enthusiasm in the use of ultrasonics, this NDE technology is known for not being able to detect changes in the adhesive bond strength [17].

The ultrasonic waves can nevertheless yield information regarding the morphological AND elastic features at the interface of adhesive bonds by their behaviour in the material inspected [18].

Their propagation behaviour in the material is directly linked to intermolecular forces and mechanical behaviour: depending on the mechanical properties (decreased

modulus, nonlinear stress-strain behaviour) the most adequate method of US inspection can be selected. [15] Hence, as an example, according to the strain applied the material may have a nonlinear strain-stress curve and transmit the ultrasound nonlinearly, providing higher harmonics and so, information about the bond strength [14,19].

Smith et al. realised a large experiment campaign regarding several different ultrasonic methods. [15] Among all ultrasonic inspection techniques, Smith et al. assume that all methods can be sorted in two categories whether the technique focuses on measurements of out-of-plane (with compression waves) or in-plane (with shear waves) stress-strain properties regarding the bond interface. It is also suggested that the use of shear waves may be wiser since an adhesive bond is designed to support shear stress more than compressive stress. A description of each ultrasonic technique applied to characterisation of adhesive bonding found in the literature is provided here for a better overview on this technology.

## Normal Incidence Narrow-Band Pulsed Spectrometry

The first technique is the basic one, used also for conventional US inspection. It corresponds to the emission of ultrasonic waves with a defined frequency. Two modes can be highlighted: ultrasonic can be performed whether in the pitch-catch (through transmission) mode, by using one transducer to emit the sound wave and one other for receiving it, or in the pulse-echo mode, by using the same transducer for the emission and detection of the sound wave.

These modes have each ones their advantages and are applied in most of the other existing ultrasonic variant.

# Swept-Frequency (US Spectroscopy) Technique

The swept-frequency method, also known as US spectroscopy involves the capture of multiple superimposed reflections through the structure and determining the frequency content which is particularly efficient if the reflection in the material is complex and frequency dependent regarding as in the case of multiple interfaces of bonded composites.

Smith et al. tested this method on an adhesive bond immerged in water at 50 °C over a period of 17 weeks. They observed that peaks can be selected in the whole frequency spectrum to monitor the properties of adhesive joints; measures showed that the amplitude and frequency between the peaks are indeed dependent on the properties of the adhesive layer (thickness, interaction with ultrasonic, etc.) [15,20].

## Harmonic imaging (Nonlinear Ultrasonic) technique

The method of nonlinear harmonics, also called harmonic imaging, relies on the principle that binding forces have a nonlinear mechanical behaviour and as a consequence, generate a nonlinear modulation of the transmitted or reflected sound wave. The amplitude of the ultrasonic wave needs to be high enough to cause a local mechanical deformation in the adhesive bond to generate non-harmonic components. These components are hence expected to yield information regarding to the adhesive bond strength [17, 21].

Bockenheimer et al. studied the case of acoustic resonance in a 4 week hydrothermal aged multi-layered bonded metallic system. All researches lead on this technique agree on the fact that the technique is not mature enough for an application on adhesive bond, especially in the case of weak bonds in epoxy composite materials. Even if it is considered a promising technique, nonlinear ultrasonic still suffers lots of errors originating from all possible sources (bonded material, measuring devices, probes and coupling system, etc.) that need to be solved [17].

Next development steps for this technique involve further investigations with a better control of the disturbance parameters and theoretical calculations and models. They shall help developing a quantitative measurement of the adhesion strength. Also a

combination of this technique with other US technique such as the oblique spectroscopy ultrasonic method is thought to be a possibility for better results [18].

# Oblique Incidence Ultrasonic technique

The oblique incidence technique is the first method involving shear waves. Shear waves are generated due to the angle of incidence in the material (generally optimal with an angle of 14° for the shortest ultrasonic path in a composite bonded structure) and allow a potentially more sensitive examination of interfacial properties. In Smith et al.'s study, the detection of 'kissing bonds' could however not be achieved even though the method was proven sensitive to degradation through water contamination.

## Guided waves Ultrasonic technique

Guided waves are a variant of the oblique incidence technique, easily performed by moving apart the transducer in charge of the emission and reception. The signal is reflected in the material several times before reaching the detection probe and so, has different modes yielding information about various properties. Smith et al. relate that good results are claimed for the detection of weak bonds, yet only in materials with highly different acoustic impedance. As a consequence, one may doubt any detection capability in the case of an epoxy composite bonded with epoxy adhesive.

# Shear Wave Resonance Ultrasonic technique

Shear wave resonance is a method experimented on metallic adhesive joints. Based on the condition that the longitudinal velocity of the metallic material being twice the shear velocity, series of thickness-shear resonance with particle motions relatively parallel to the surface and so the adhesive bond interface can be observed.

In such cases, an adhesive bond causes a damping and a modulation of the shear wave resonance. The importance of the damping and shift in frequency is believed to be determinant for the characterisation of the adhesive bond modulus. Considered promising in metallic bonded structures, no results of experiments with this technique for adhesive bond in composite has been found in the literature yet.

## 3.2 Laser methods for the control of adhesive bonding quality

A major recent approach in the characterisation of the adhesive bond strength is the use of laser methods. Several work groups dedicated to few laser technologies are to be found in the literature and deserves attention for their respective activities.

# Laser Shock Adhesion Test (LASAT) and Laser Bond Inspection (LBI)

Over the years, laser techniques evolved to become a lot more efficient and allowed their use as new inspection tools. The techniques presented here are called LASer Adhesion Test (LASAT), developed by the CNRS, and Laser Bond Inspection (LBI), developed by Boeing and LSP Technologies. They both use a shock wave generated by plasma originating from a high power laser with short pulse on the front surface. This shock wave propagates through the inspected structure in compression and its reflection on the back surface provokes a tensile strength [22-25].

Experiments were mostly conducted on coating adhesion strength, on bulk laminates and on adhesive bonds of composite parts.

In the case of the adhesive bonding inspection, the principle of those techniques relies on the fact that a weak adhesive bond would be disbonded whereas a good adhesive bond would not be damaged by a determined tensile stress induced by the shock wave. These following introduced techniques appear as the most promising for the inspection of adhesive bond and the optimisation of the integrated bonded design.

In the case of the LASAT, Arrigoni et al. managed to observe systematic delamination in the velocity signal peaks for power density higher than 4.7 GW/cm². It was also stated thanks to the changes in the peaks visible with the FFT, that even for lower power density (around 3,7 GW/cm²) the adhesive bond was affected at the interface. Dynamic loads applied for the proof test are however to oppose to quasi-static conditions where the load can be around 10 times lower [25]. This remark was also done by Bossi et al. for the LBI [24].

Boustie et al. experimented the response of epoxy composite laminates to the shock wave. Depending on the number of plies in the laminate, even at intermediate laser intensity, the composite damage occurs due to internal tension generated by the propagation of the laser shock wave [26]. Jagdeesh et al. found a critical value of 0,36 GPa for the delamination appearance. This value was validated by simulation in their attempt to determine the threshold tensile stress for epoxy bonded aluminum thin sheets [27].

In the case of the LBI which focused more on inspection of adhesive bond than others applications, Bossi et al. established through numerous experimentations that the laminate failure prior to bond line failure was not to fear. Limitations regarding thickness of the adhesive bond due to attenuation of the stress were however suspected even if this issue may be irrelevant for weak bond inspection [22]. The same issue was again later on addressed with a maximal thickness of 23 mm for the whole samples and the satisfaction that no edge effects were influencing the measurement [23].

Bossi et al. also recently observed a "fatigue" effect in the results that bond line delamination occurs at lower input/velocity if repeated pulse were applied. As a conclusion of the LBI results, it can be underlined that no mechanical performance reduction was measured (Lap Shear, DCB tests) on the specimens which were attested as damaged by US scans.

#### Laser Ultrasonic

Apart from the LASAT and LBI, another Laser technique such as the Laser ultrasonic is being developed. In this case, the work groups of interest are the Centre National de Recherche du Canada (CNRC), EADS IW France and Bremens Institute for Applied Beam technology (BIAS).

The principle of the laser ultrasonic inspection method is defined as follow: a pulse laser sends single or multiple pulses of controlled magnitude which are absorbed by the material surface and generate a local heating. A thermo elastic excitation is caused and induces an ultrasonic surface- and bulk- wave propagation in the material. It is the detection and capture of this thermo elastic response that lifer information regarding to the state of the specimen [28,29].

An alternative approach is the laser tapping, which consists in focusing the thermal stresses produced by the laser in order to cause a lifting and bending effect, and so induce vibration of a potential disbonded layer [30]. Both techniques can be used concurrently for more information on the inspected specimen. Kopylow et al. also pointed out that ultrasound wave can be is distinguished in two regimes: non-destructive thermo elastic, or ablative, like for the LBI and LASAT techniques, due to the power level of the laser [28].

The results obtained with this method up to day are relatively promising. The inspection of detached skin area in honeycomb sandwich structures which can be excited and brought to vibration like membranes due to short excitations pulses was successful [31], as well as the detection of all types of defects in composite materials [28,29].

Assessment of the quality of the adhesive bonds with Laser ultrasonic is considered relevant and tests are nowadays run on samples with weak bonds to evaluate the potential of this technique. Unfortunately, no literature could be found yet on this particular topic.

#### 4. OTHERS PROMISING NDT METHODS

Besides the ultrasonic techniques and the laser inspection methods introduced in chapter 3, few others NDT techniques are being tested for the inspection of adhesive bonds.

*Shearography* 

Digital shearography consists in the measurement of in-plane and out-of-the-plane deformations thanks to acquisition from coherent electromagnetic waves between an object and the detector on the inspected surface. The sensitivity of this technique is around a few nanometres [11].

Tests conducted showed sensitivity to bond strength under certain conditions. Heat applied at the surface of the bond caused a larger displacement in the case of a good bond [32]. Other tests showed that weak bonds detectable were in fact delaminations in the epoxy [33]. The detection of delaminations was assessed as feasible with use of shearography applied to epoxy composite exposed to piezo electrically and laser generated lamb waves [34].

# Active Thermography

Active thermography is a well established NDT technique already tremendously studied. [35] It corresponds to the simulation of a thermal flow in the specimen to be examined and the detection of the resulting temperature on the surface part with a sensitivity of mK. As an NDT technique, the active thermography was established unable to assess the presence of weak bond in composite specimens due to anisotropy of the composite material and the low response in the heat diffusion [12]. It can however be suggested that with better resolution and improved tools, adhesive bond quality could be distinguished with active thermography.

# 5. CONCLUSION

The numerous literature sources evaluated in this paper clearly demonstrate that the adhesive bond quality is a critical parameter that cannot yet be assessed by any NDT techniques. Several conventional methods are being optimized and developed for a better detection capability but up to day, no method can reliably and with a good reproducibility detect any weak adhesive bond, either on metal or composite substrate.

Ultrasonic alternative methods (US spectroscopy; Nonlinear US; Guided Waves; Oblique incidence; Shear Wave Resonance; etc.) as well as Laser Proof tests (LASAT; LBI; Laser Ultrasonic) and other methods like Shearography and Active Thermography are reported as method with high potential for the measurements of adhesive bond strength. Those methods have already been applied to the characterization of delaminations, defects, or coating strength measurement in complex structures. Materials including bonded aluminium, epoxy composites laminates bulk or bonded, and even honeycomb sandwich panels have been tested thanks to those techniques. Further development of all those methods needs however to be realized in the different work groups. Evolutions in term of detection capability, resolution, etc. shall demonstrate the true potential for their use as an enabler for adhesive bonding as a major assembly technique.

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