Innovating for Structural Adhesive Bonding Evaluation and Analysis with Ultrasounds : A Summary.

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Abstract. In this paper we summarize results from the French research project ISABEAU (Innovating for Structural Adhesive Bonding Evaluation and Analysis with Ultrasounds). The goal of this ANR project (2013-2017) is to overcome the technical obstacles to the verification of the integrity of adhesively bonded joints using ultrasonic NDT methods. For instance, the case of kissing bonds remains a major scientific challenge years after years. The development of an acoustic method quantifying the adhesivion "level" of a bonded joint is also one of the ultimate objectives of this research. Three NDT laboratories and one chemical laboratory gather their experience to achieve a leap in the quantitative analysis of the interfacial adhesion between assembled substrates and the adhesive layer. Surface treatments were chosen to modify the adhesion properties. The project focuses on aluminium/epoxy/aluminium assemblies, which are representative of the difficulties that one may come across in structural bonding.

Three well identified levels of adhesion were obtained and qualified by mechanical tests (lap shear test). Numerous bonded samples were manufactured to make it possible to use various ultrasonic methods, most of them with various levels of adhesion. Several through-transmission ultrasonic techniques were used to evaluate different properties of these assemblies: (1) the material properties of the aluminium and epoxy, (2) the bond homogeneity and thickness, (3) the state of the substrate-adhesive interfaces together with mechanical parameters, which are representative of the interfacial adhesion. Lamb waves and SH waves were used to investigate the variations in the adhesion level, which depends on the treatments carried out on the metal/epoxy interface. As part of this study, the issue of surface roughness was addressed.

Introduction

As bonded structures will be increasingly used in transport industries, there is a need to develop non-destructive testing (NDT) methods to ensure inspection after manufacturing, or after in-service use [1].

Three classical types of defect are investigated [2,3,4]: i. complete voids, disbonds, or porosity, ii. poor adhesion (a weak bond between the adhesive layer and one or both of the adherents), iii. poor cohesive strength (a weak adhesive layer).

In the ISABEAU project (Innovating for Structural Adhesive Bonding Evaluation and Analysis with Ultrasounds), we investigate the second type of defects : poor adhesion. This defect is still extremely difficult to detect, which results in a reduced use of bonding techniques in structural applications. Poor adhesion is a reduction in the strength of the bond between the adhesive and the adherent - a reduction which is not due to the presence of another type of defect, such as porosity, delamination or foreign bodies. When the adhesion is null, this defect is known as the Kissing Bond.

One of the challenges of the project is to create reference defects of this type to qualify NDT measurements. There is clearly a need to produce reduced-strength bonded assemblies to qualify non-destructive methods. A recommendation of a recent report is that the FAA supports the development of weak bond specimen sets to be provided to the NDT community [1].

The poor adhesion defect is extremely difficult to detect using classical ultrasonic techniques because the bond strength is governed by a layer thinner than a conventional ultrasonic wavelength. Surface treatments were chosen to modify the adhesion properties. The project focuses on aluminium/epoxy/aluminium assemblies, which are representative of the difficulties one can come across in structural bonding.

This paper is organized as follows. Section 1 describes the design and fabrication of the weak bond specimens. Specimen design varies according to the objective pursued. Section 2 summarizes the mechanical testing and qualification procedures used to ensure the quality of the reference samples. Section 3 summarizes the quantitative results obtained using transmission measurement and Lamb wave measurement. Section 4 develops theoretical and experimental results from a complementary study about the influence of the roughness. Finally a discussion about these results and a description of what the next steps should be are provided in the conclusion.

Designing reference samples for NDT

Some of the objectives of the ISABEAU project are the development of surface treatments that can be used to create at least three bond strength levels - ideally these levels should be well separated to help the demonstration of NDT qualification - and the design of specific sample geometries to study several ultrasonic characterization methods.

1.1 Geometry design

To quantify bond strength, lap shear test samples were designed in compliance with European norm EN 2243-1. Another objective of the project is to propose samples for material characterisation. These specific samples can be composed of one layer (to characterise the adherent or the epoxy), two layers (adherent-epoxy layer) or three layers (bonded assembly). A third kind of samples, with thicker plates, are designed to test high energy waves (from multiple arrays). Table 1 summarizes the various geometries and the corresponding

objectives. The status of each study is indicated, with references for more details, as well as sample dimensions : height (h), width (w) , adherent thickness (t_a) and bond thickness (t_b).

Design	Dimensions (mm) of bonded area $h*w*(t_a+t_b+t_a)$	Objectives	Status
Lap-shear samples (EN 2243-1)	12.5*25* (1.6+0.2+1.6)	Qualifying surface treatment and quantifying reduced strength	Ended [5]
Sandwich A1 Size : h.w.(t_a-t_b-ta)	200*200* (2+1+2)	Qualifying materials (Cijkl) and studying the bonded interface (k_L, k_T)	Final studies [6,7,8]
Large bonded sample	450*25* (4+0.3+4)	Studying Lamb waves in realistic geometries	Under development
Two and tri-layers assemblies	Mainly 200*200* (5+0.5) Or((5+0.5+5)	Studying the behaviour of bonded joint before assemblies Studying roughness influence	Final studies [9] Ended [10]
Sandwich A3	200*200* (10+1+10)	Studying KB and volumic waves	Under development

Table 1. Summary of sample designs for characterization of reduced-strength samples

1.2 Surface treatments to produce reduced-strength samples

Several solutions were used in the past and are still being used today to create reference samples for NDT qualification. Defects can be artificially created by adding a foreign body at the interface between two adherents [11] or by changing the bond by incorporating a release agent [12] or particles [13,14]. To study the adhesive behaviour of a bonded assembly, the most appropriate approach would be to vary either the surface preparation or the surface treatment [15]. Many parameters can modify the bond strength, and so the challenge is to reproduce the same preparation steps in the same environment [16]. In some studies, chemical pollution is used to reproduce the pollution generated during an industrial process [17]. To obtain variable adhesion, another solution consists in compressing an assembly - which can be considered as representative of a case of dry-contact kissing bonds [18].

In this work, we chose a two-component model system for the epoxy adhesive [5]. The epoxy resin was diglycidyl ether of bisphenol A (DGEBA). The curing agent was a polyetheramine. The epoxy resin and the diamine curing agent were mixed mechanically at room temperature - stoichiometric amino hydrogen / epoxy ratio equal to 1. The mixture was carefully ultrasonically degassed to prevent the formation of air bubbles. A silane adhesion promoter was used to modify the adhesive strength.
Before being joined together, the aluminium adherents underwent a three-step surface treatment: the substrates were degreased (D), by wiping them with a tissue soaked in isopropyl alcohol; then, the degreased substrates were sand-blasted with corundum particles to create surface roughness (D+S); finally, the degreased and sand-blasted substrates were treated with a silane solution (D+S+Si).
The surface treatment was stopped at each step so that three sample sets with different adhesion levels were produced. The nomenclature adopted for these surface treatments was D, DS, and DSSi.

Strength results

Single lap joints were 100 * 25 mm^2 pieces. Substrates were treated according to the three procedures described above, and assembled together with a 12.5 mm overlap (EN 2243-1). A specific mould allowing the preparation of seven specimens was designed. The adhesive thickness was between 0.2 and 0.3 mm. The curing cycle was 48 hours at room temperature, followed by three hours at 160 °C, resulting in a fully cured adhesive.

The most complete description of the bond was sought to ensure the quality of the reference samples. Bond homogeneity was validated using an ultrasonic through transmission setting [19]. We chose high frequency transducers with a diameter of 0.25" so that the beam remained extremely narrow. The focal zone was thus about 0.0625" (1.59 mm). All samples presented a strong homogeneity: no defect and constant thickness.

Mechanical tests were carried out on a standard MTS testing machine with a capacity of 100 kN. The machine was interfaced with a computer dedicated for control and data acquisition. The specimens were tested in static tests, loaded at a constant rate of 0.1 mm/min. An extensometer was used to monitor the real deformation. End tabs were bonded onto the substrates for the alignment in the load fixture to avoid flexure at the bonded interface.

Acoustic emission (AE) was also recorded. The first analyses indicated the presence of two classes of events: a first class of AE signals, corresponding to the interfacial (aluminium/epoxy) debonding, and a second class of higher-amplitude signals appearing after the occurrence of interfacial debonding damage, and probably resulting from resin cracking [5].

All these complementary NDT results ensure the quality of the mechanical results.

Figure 1 shows that three levels of adhesion were obtained as expected, even if there is slight overlapping of the error bars for the two higher classes.
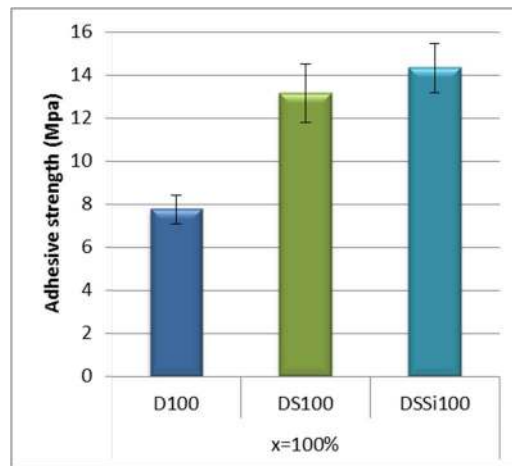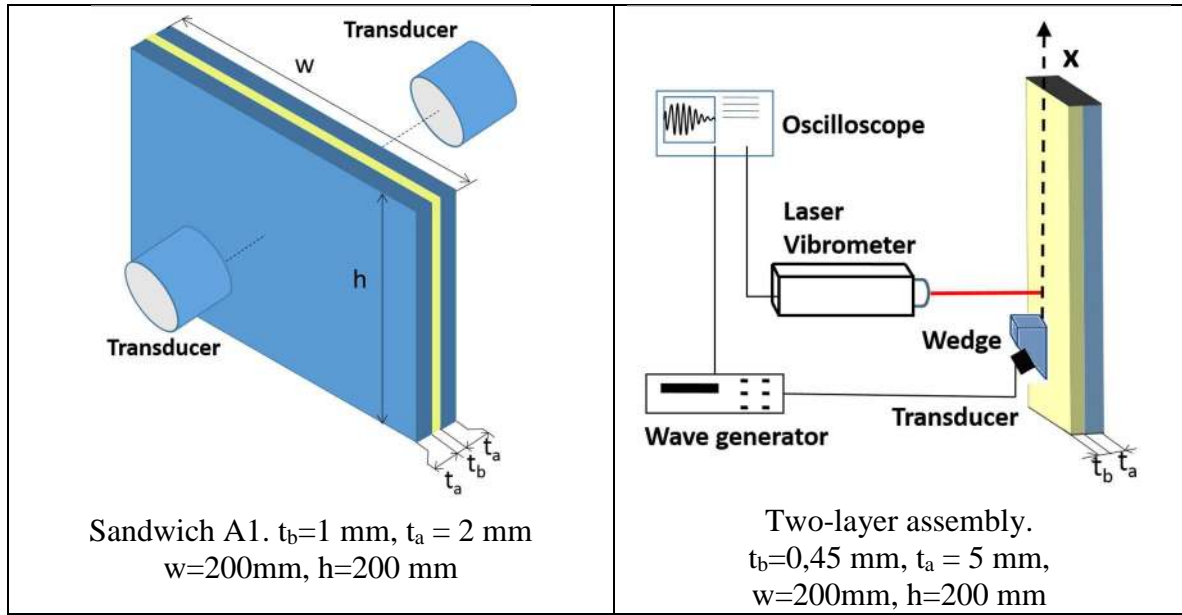

Figure 1. Measured adhesive strengths for the three sets.

Adhesion evaluation using rheological model.

In the ISABEAU project, various ultrasonic methods are used. Figure 2 summarizes two of these methods : through transmission measurement and Lamb wave measurement. To analyse the experimental data we used a spring-like model to describe the mechanical behaviour of the bonded joints. There are several possible models, depending whether cohesive or/and adhesive properties should be represented [20].

| Sandwich A1. t_b=1 mm, t_a = 2 mm w=200mm, h=200 mm | Two-layer assembly. t_b=0,45 mm, t_a = 5 mm, w=200mm, h=200 mm |

2a. Through transmission measurement   2b. Lamb wave measurement

Figure 2. Two ultrasonic measurement setups.

The first method is based on the measurement of the transmission coefficient of an ultrasonic plane [21] wave through a ~~tri~~-layer plate immersed in a water tank. A study of the influence of the interfacial conditions on the evaluation of the elastic moduli of the adhesive layer embedded between the two substrates, was carried out both numerically and experimentally at the I2M laboratory. For this approach, the model included 5 layers: the two substrates, the epoxy bond and two interphase layers between the substrates and the bond.

By inferring four moduli for the adhesive layer from measurements performed for various angles of incidence ($0°\leq\theta\leq50°$), and assuming perfect interfacial conditions, an abnormal apparent anisotropy can be observed, revealing the presence of interfacial weaknesses. Thus, under certain conditions, this method makes it possible to differentiate a cohesion problem from an adhesion problem. Then, if the elastic properties of the adhesive layer are known, an ultrasonic characterization of the mechanical properties of the interphases is possible. Such characterisation was carried out on assemblies with 3 different surface treatments. Two longitudinal and transversal stiffnesses were then inferred, by assuming that the interphase thicknesses, t_{int}, are both equal to 1µm and that $K_L = {C_{11}}/{t_{int}}$ and $K_T = {C_{66}}/{t_{int}}$ [22]. This bonded joint model is thus equivalent to those presented in Figure 3. Stiffness results are presented in Table 2 for the three surface treatments.
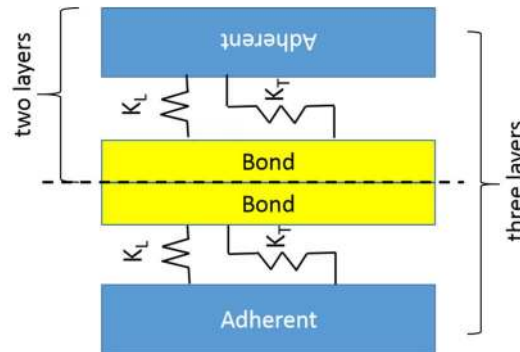

Figure 3. Rheological model for adhesion

As for Lamb-wave measurements, only two bi-layer samples were designed, made of the same materials, and only two surface treatments were applied (DSSi100 and D100, cf. Table 2). An experimental study was conducted using a contact transducer with a centre frequency of 1 MHz placed on a 45° degrees PMMA wedge acting as emitter and a laser vibrometer acting as receiver (Figure 2b). The normal displacement of the propagating wave was measured for several positions at the surface of the sample, in the propagating direction, in steps of 0.1 mm. A double Fast Fourier Transform (FFT) was performed on the space-time data to determine the experimental dispersion curves in the wavenumber-frequency plane. The bi-layer mechanical model also corresponds to a rheological model with two stiffnesses, K_L, K_T (Figure 3). A semi analytic finite element (SAFE) model was developed allowing the prediction of dispersion curves, knowing material densities, ultrasonic velocities and thicknesses [23]. The comparison between experimental and theoretical dispersion curves allows the evaluation of the corresponding stiffnesses [24]. Stiffness values are given in Table 2. Obtaining these results is a first step in the characterization of the adhesion strength. The aim is to extend this procedure to different qualities of adhesion by applying different surface treatments and to determine the corresponding stiffnesses using the inverse problem method.

Through-transmission measurements				Lamb wave measurements		
Surface Treatment	K_L(PPa/m)	K_T (PPa/m)		Surface Treatment	K_L(PPa/m)	K_T (PPa/m)
DSSi100	2.08	1.0		DSSi100	10	10
DS100	0.25	0.1				
D100	0.1	0.02		D100	0.316	0.316

Table 2. Longitudinal and transversal stiffness measurements for two propagation modes

Effects of a rough interface on shear horizontal acoustic wave propagation

Another aspect of wave propagation is considered in this project : the effect of surface roughness at the interface in a bonded bi-layered structure. An analytical contribution has been proposed in the case of shear-horizontal (SH)-guided waves [25]. The thickness of the bond layer is assumed to be non-negligible, and the interfaces between this layer and the plates are considered roughened, with complex shapes and depth profiles. It is also assumed that the average roughness height is a small fraction of the thicknesses of the plates everywhere. The roughness induced changes in the characteristics of the fields created by a harmonic source set at the entrance edge of the structure are expressed using 2D calculation of the displacements and stress perturbations.

The basis of the theoretical approach is the integral formulation which has been developed previously [26] for SH wave propagation along the surface of a rough surface plate. The development that follows involves the roughness-induced modification of the waveguide SH modes occurring when roughness is present. The behaviour of the roughness-pertubed acoustic field is presented mainly in terms of SH modal couplings both inside each plate and between plates through the layer of glue.

The behaviour of the glue is expressed using the classical spring-like model, with a spring stiffness $\mu_g/\ell_g \cdot \mu_g/\ell_g$, where ℓ_g is the thickness of de glue, depending on the position x along the surface.

We considered the perturbed displacement field which is defined as the difference between the displacement field U and the incoming field $U^{(0)}$. In the following equation (1) the right side represents the perturbed displacement field due to the scattering on the roughness in each plate numbered by the index q. This field represents both the coupling of the incoming field ($U_q^{(0)}$) with itself and the cross-coupling between this incident SH-wave and the other modes (maximum number is M) which can exist at the working frequency :

$$U_q(x, z_q) - U_q^{(0)}(x, z_q) = \sum_{m=0}^{M} [H_{qm}(x) + E_{qm}(x)] \times \psi_{qm}(z_q) \tag{1}$$

In this equation, fully explained in [25], in order to express the perturbed field as an expansion involving a finite number of SH-modes, an orthonormal set of functions ψ_{qm} was defined for each mode m and in each layer q using the Gram-Schmidt process [27].

The first term in the right hand side, H_{qm} , represents the boundary modal coupling due to both the slope and the depth of the roughness, and the second one, E_{qm}, represents the perturbation on the coupling between the plates through the layer of glue, which accounts for the effect of the thickness of the layer of glue.

If a propagative mode is incoming in a rough area, the model can predict how the incoming energy is divided in several propagative modes or retro-propagative modes. Another interesting possibility of this modelling is the prediction of phase-matching effects due to roughness spatial periodicity Λ as described by the relation (2). This so-called phonon relationship represents the intersections of the dispersion curves (incident SH-waves, k_I wavenumber) with the phonon curves (k_M wavenumber) :

$$k_I + k_M = 2\pi/\Lambda \tag{2}$$

Spatial periodicities Λ could be simply calculated if the roughness profile is regular or could be deducted from the power spectral density of the roughness profile [28]. Complete studies [25] show that, despite the complexity of the coupling effects through the roughened layer of glue, the structures of the perturbations depend strongly on the nature of the incident SH-wave and on the phase-matching. Several simulations demonstrate that the amplitude of the perturbed waves is five to ten times lower than those of the corresponding incoming waves and that, as expected, the field perturbation does not propagate significantly beyond the roughness.

For the experimental part, the periodic profile has a sawtooth profile (Figure 4a). Parameters were set to Λ=3.7 mm for a total length $\ell = 37$ mm, and the height h of the roughness was 0.1 mm. The tri-layer assembly was composed of two aluminium adherents (5 mm thick) and the bond layer was an epoxy glue with a 0. 5 mm thickness. The working frequency was set to 480 KHz. The SH_0 incident field is shown in the wavenumber-frequency plane in Figure 4b. The SH_0 wavenumber is 958 m^{-1}. The calculated retro-propagative phonon (SH_2 mode) has a wavenumber equal to 738m^{-1} (Figure 4c). Using equation (2) , the term $2\pi/\Lambda$ is equal to 1700 m^{-1} so this relation is largely verified (relative difference is about 0.23%).

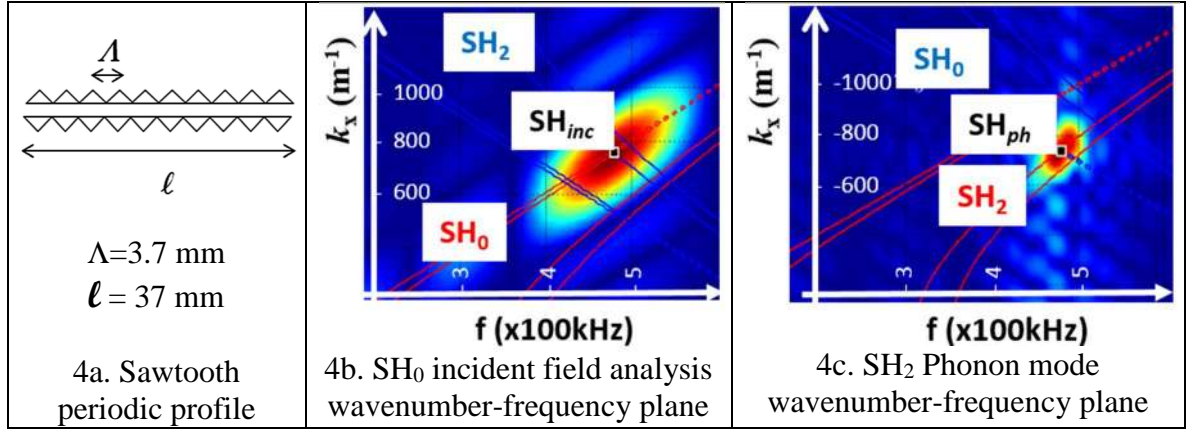|		
Λ=3.7 mm ℓ = 37 mm 4a. Sawtooth periodic profile	4b. SH_0 incident field analysis wavenumber-frequency plane	4c. SH_2 Phonon mode wavenumber-frequency plane

Figure 4. Simulation results for periodic roughness

Considering experimental results on the same sawtooth profile, the wave energy is distributed in a larger domain in the wavenumber-frequency plane but results confirm the phonon relation. Studies undergo to evaluate the transmitted energy through the rough profile depending on the roughness parameters and incident SH wave frequency.

Conclusions and perspectives

The ISABEAU project fulfills its objectives to propose references samples for the development of innovative ultrasonic NDT methods. There were difficulties obtaining qualified reference samples with several adhesion strengths, as bonds should present absolutely no defects in order to certify the quality of the NDT studies. Another difficulty arose when trying to propose numerous sample geometries depending of the ultrasonic method chosen for the investigation as several wave propagation modes could be used : volumic waves, Lamb waves, shear waves.

The first results are promising and will be developed. The spring-like behavior of the bond is the main guide to analysing variations in the ultrasonic measurements. In order to take the discussion further about stiffness results from various wave propagation modes, we will complement this work with the measurement of interphase depth for each surface treatment. This depth will be measured on fractured lap-shear samples (post-mortem study). The depth of the interphases could be evaluated using nanoindentation and atomic force microscopy - these methods have been shown to be effective in studying the mechanical properties of the interphase region at the sub-micrometer scale [29].

The possibility to detect variation in the cohesive strength is also under study by varying the cure process.

In a next step, Kissing-Bond defects could be produced by varying locally the surface treatments. Other ultrasonic linear methods, such as high energy volumic waves (using multiple arrays), could also be evaluated for the detection of this type of defect. Ultrasonic non-linear methods should be also investigated [30].

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