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► **To cite this version:**

Camille Gauthier, Mounsif Ech-Cherif El-Kettani, Jocelyne Galy, Mihai Predoi, Damien Leduc. Structural adhesive bonding characterization using guided Lamb waves and the vertical modes. International Journal of Adhesion and Adhesives, 2020, 98, pp.102467 -. 10.1016/j.ijadhadh.2019.102467 . hal-03488573

HAL Id: hal-03488573

<https://hal.science/hal-03488573>

Submitted on 28 Jun 2022

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Structural adhesive bonding characterization using guided Lamb waves and the vertical modes

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Abstract

The aim of this paper is to present a method to evaluate and qualify the adhesion level in a structural metal/adhesive/metal bonding using ultrasonic guided Lamb waves. The samples are manufactured with different surface treatments in order to obtain different levels of adhesion. Using Lamb dispersion modes associated to the Jones rheological model, it is shown that it is possible to evaluate different levels of adhesion. Furthermore, the vertical longitudinal mode V_L is particularly focused. Results show that the measurement of its frequency cut-off, which is very simple and fast to achieve, can be a very good indicator on the integrity of the bonding.

1. Introduction

Structural adhesive bonding presents a lot of advantages such as uniform distribution of mechanical stress on the entire surfaces to be bonded, a decrease of weight, a waterproof connection, an assembly of different materials and a reduction of corrosion risks. This is why structural adhesive bonding is more and more used in industrial sectors [1]. Therefore, the development of non-destructive methods is essential for the condition assessment and maintenance of the bonded structure. In the last few years, many of non-destructive methods have been developed : For example, Meola [2] propose a method based on thermography and show that it is possible to evaluate the defect geometry in a structure, Kular [3] use digital image correlation (DIC) in order to evaluate the kissing bond in composite adhesive lap joints or Ecault [4] which study the damage tolerance of composite material with laser chock wave technique. One of the most used methods is the one based on the ultrasonic guided waves propagation. They have been proven to be an effective technique for the inspection of adhesive lap joints [5-8], adhesive bonds between composite [9,10] or the determination of the adhesion level [11]. In particular, Ismaili [12] has shown that Lamb vertical modes have a sensitivity for the characterization of the adhesive layer in a structural adhesive bonding. The aim of this paper is to propose a numerical and an experimental methods to evaluate the quality of a structural adhesive bonding based on the one hand on the propagation of guided Lamb waves in different samples metal/adhesive/metal structures, and on the other hand on the use of the vertical longitudinal mode cut-off frequency measurement. The studied samples are Aluminum/Epoxy/Aluminum where both the cohesive and adhesive aspects are considered, based on the conversion rate of the adhesive film and on the chemical surface treatments at the interface Aluminum/Epoxy.

Both adhesion and bonding problems are treated because of the samples manufacturing. On the one side, adhesion problem refers to a sample with a good bonding and no defect (so no delamination) but with different levels of adhesion obtained by modifying the surface treatment [11]. On the other side, the bonding problem indicates a start of delamination obtained by the introduction of defect during the manufacture of samples like addition of release agent [13] or the use of Teflon [14].

2. Materials and methods

2.1. Materials

The polymer adhesive used in this work is a mixture of a pre-epoxyde DGEBA DER 331 (DOW Chemicals) and a diamine Jeffamine D230 (from Aldrich). After the mix of the two components, the preparation is degased in order to avoid bubbles formation. 3-(glycidoxypropyl)trimethoxysilane also called γ -GPS, used for the chemical treatment is obtained from Aldrich. The chemical structures of the components are given in Table 1. A universal water based release agent (ref : Cirex Si 041 WB) is also used in the aim to create a very low adhesion level. The metallic substrate is an aluminum alloy, A2024, in the form of 5 mm thick sheets.

Components	Chemical structure	Molar mass (g/mol)
Diglycidyl ether of Bisphenol A DGEBA		374
Polyetheramine D230		230
γ -GPS		236

Table 1: Chemical structure of the components

2.1.1 Surface treatments

Before coating, the aluminum substrates were treated as followed:

- Degreasing: This treatment is performed in order to take off all the contaminant particles. Solvent degreasing is performed with a tissue soaked in isopropanol.

- Degreasing and silanisation: The chemical treatments using a coupling agent highly improves the level of the interfacial bond creating strong chemical bonds (covalent bonds) between the metal surface and the adhesive [15]. In our study, the specimens are immersed 10 min in a 1 wt. % solution of γ -GPS in distilled water. Prior to immersion, the pH solution is adjusted to 5 using acetic acid and the solution is stirred at room temperature for 1h to hydrolysis of the methoxy groups. The silane-treated samples are heated for 1h at 93°C in an oven to allow condensation reaction [16,17].

- Degreasing and mould release layer: The last surface treatment is the applying of one layer of commercial mould release, Cirex Si 041 WB, with a tissue soaked then heated at room temperature during 8 hours. This kind of mould release needs to be applied at least 5 times with a heating of 15 minutes between each

layer in order to have a complete action between aluminum and thermosets. The goal of one applying is to always have an adhesion but to decrease critically its level and would reveal the beginning of delamination.

2.1.2. Samples preparation

Set of samples are manufactured in order to obtain three symmetrical different levels of adhesion: the same surface treatment is applied on the both substrates for a given sample. Each tri-layer is then cured at room temperature for one week in order to obtain a partial cross-linking (80% conversion). For two samples, this process is completed by a cure for 1h at 80°C followed by 3 h at 160°C to obtain a full cross-linking (100%) as shown in [18]. It has shown that even with a partial cross-linking, a high level of adhesion is obtained if the chemical treatment is used [11]. All the conditions of samples manufacturing are summarized in Table 2. Three different levels of adhesion are supposed to be obtained by these treatments: a high level one with the sample T-DSi-80 (Tri-layer –Degreases Silane-conversion rate 80%), a low one with T-D100 (Tri-layer-Degrease-conversion rate 100) and a very weak one with T-DSiO-100 (Tri-layer-Degreased mould release SiO-conversion rate 100%).

Sample	Surface Treatment	Thickness of epoxy layer (mm)	Cross-linking (%)
T-D-100	Degreased	1	100
T-DSi-80	Degreased + silanisation	0.5	80
T-DSiO-100	Degreased + mould release layer	0.5	100

Table 2: Set of samples manufactured

2.2 Jones model

In order to study the ultrasonic response to different levels of adhesion, we use an adhesive rheological model [19]. The interfaces between the aluminum substrate and the adhesive layer are modeled by a uniform distribution of springs working in traction /compression and shear (Figure 1). The stiffness coefficients are respectively noted R_L and R_T . The boundary conditions are free surface given by

$$\begin{cases} \sigma_{xz}(z = \pm(2h + d/2)) = 0 \\ \sigma_{zz}(z = \pm(2h + d/2)) = 0 \end{cases} \quad (1)$$

and at the interface, the stress displacement relations are given by Jones and Whittier [20]. We have at $z = d/2$:

$$\begin{cases} \sigma_{xz}^{(1)} = \sigma_{xz}^{(2)} = R_T(u_x^{(1)} - u_x^{(2)}) \\ \sigma_{zz}^{(1)} = \sigma_{zz}^{(2)} = R_L(u_z^{(1)} - u_z^{(2)}) \end{cases} \quad (2)$$

And at $z = -d/2$:

$$\begin{cases} \sigma_{xz}^{(1)} = \sigma_{xz}^{(2)} = R_T(u_x^{(2)} - u_x^{(1)}) \\ \sigma_{zz}^{(1)} = \sigma_{zz}^{(2)} = R_L(u_z^{(2)} - u_z^{(1)}) \end{cases} \quad (3)$$

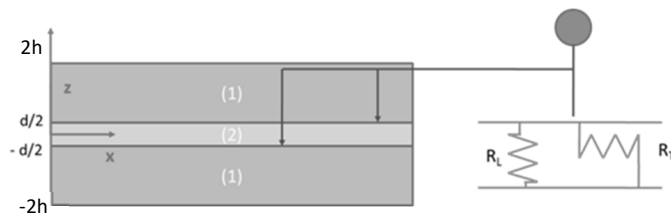


Figure 1: Representation of the rheological adhesive Jones model: (1) aluminum, (2) epoxy

Therefore, we obtain Lamb waves dispersion curves depending on the values of the two springs stiffness. Theoretically, in the case of a perfect adhesion, their values tend to infinite, while in a case of a total delamination, their values tend to zero. Nevertheless, on the numerical point of view, the order of magnitude of these parameters varies between 10^{16} N/m³ to 10^{11} N/m³ respectively [21]. The dispersion curves are obtained by numerical solving with $R_L=R_T$ using commercially FEM software Comsol 5.0 [22]. The thickness (d), density (ρ) and wave velocities (C_L and C_T) for each material are given in Table 3.

Material	d (mm)	c_L (m/s)	c_T (m/s)	ρ (kg/m ³)
Aluminum	5	6380	3100	2800
Epoxy	0.5	2512	1121	1100

Table 3: Elastic parameters of materials

2.3 Experimental characterization

2.3.1 Lamb guided waves

The emitter is a contact transducer with a central frequency of 1 MHz fixed on a PMMA wedge of 30 degrees angle and placed on the aluminum surface of the structure. The time varying normal displacement of the propagating wave is collected by a laser vibrometer, used as a receiver, for several positions at the surface of the sample, in the propagating direction, by 0.1 mm step (Figure 2). The experimental data are recorded for signal processing. A double FFT is performed on the space-time data to determine the experimental dispersion curves in the wavenumber-frequency plane for each sample.

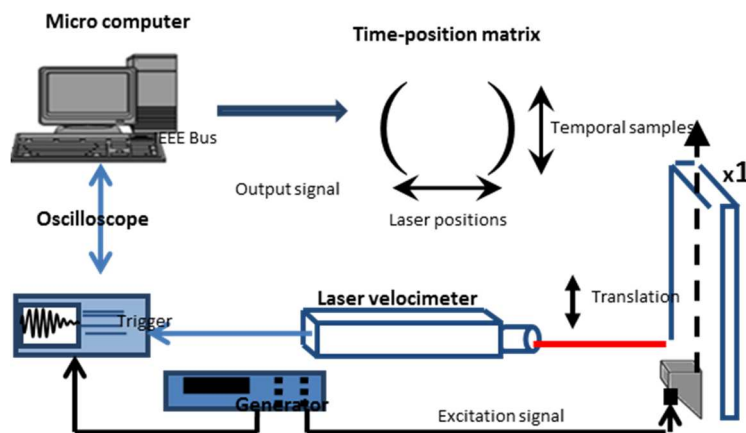


Figure 2: Guided waves experimental set up

2.3.2 Cut off frequencies

The cut-off frequencies measurement is based on the temporal Fast Fourier Transform (FFT) processed on the multi-reflected echos in the section of the structure. A longitudinal transducer with a central frequency of 200 kHz placed in normal incidence on the sample is used. The excitation signal is delivered by an impulse generator. The temporal signal (an example is given on figure 3 for the T-DSi-80 sample), is recorded before the application of the FFT.

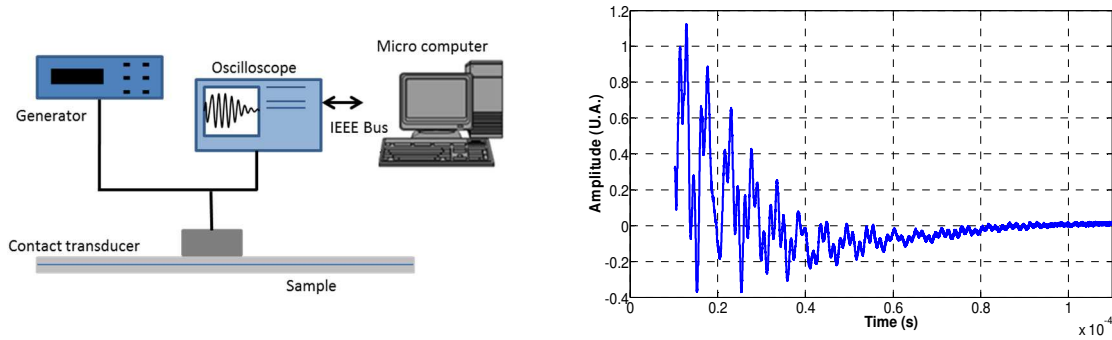


Figure 3: Cut-off frequencies experimental set up (a) and an example of temporal signal obtained from T-DSi-80 (b)

3. Results and discussion

3.1. Characterization using propagating guided Lamb waves

In a previous study [11] on bi-layers Aluminum/Epoxy manufactured with different adhesion levels, it has shown that it was possible to associate a couple of spring stiffness to a level of adhesion by superimposing the numerical dispersion curves to the experimental ones for a given sample. Thus, in the case of a high level of adhesion, the values of the spring stiffness are found to be $R_L=R_T=10^{16}$ N/m³ and for a low level of adhesion like a sample manufactured with a simple degreasing as surface treatment, the values of the spring stiffness are $R_L=R_T=10^{14.5}$ N/m³. The goal of this part is to validate this model in order to evaluate the level of adhesion on a structural adhesive bonding.

The experimental dispersion curves for the sample T-DSi-80 are plotted on figure 4. The numerical dispersion curves obtained for $R_L=R_T=10^{16}$ N.m⁻³ are superimposed. One can see the very good agreement between the numerical and the experimental curves, which confirms the high adhesion level of this sample.

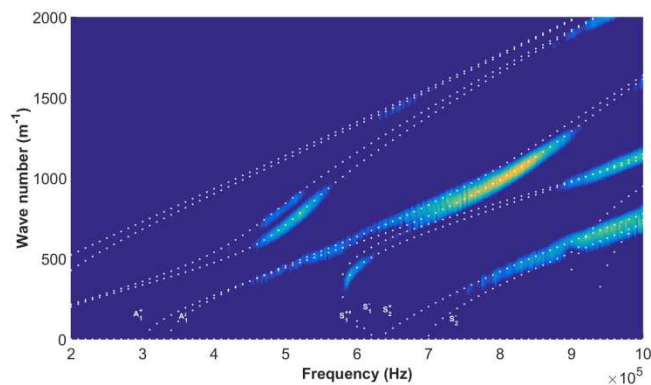


Figure 4: Experimental dispersion curves for the sample T-DSi-80 superimposed with the numerical dispersion curves with $R_L=R_T=10^{16}$ N.m⁻³ in white.

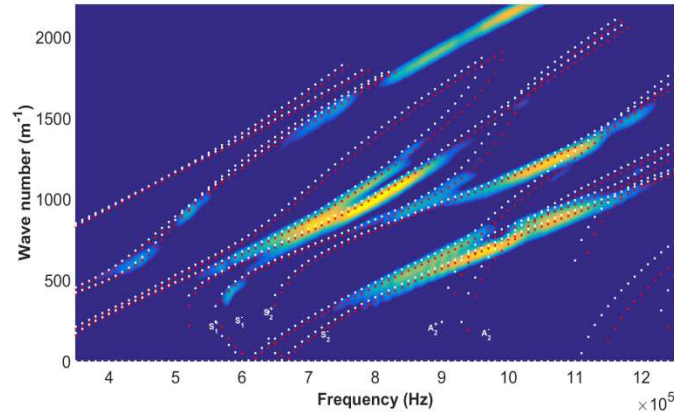


Figure 5: Experimental dispersion curves for the sample T-D-100 superimposed with the numerical dispersion curves with $R_L=R_T=10^{14.5} \text{ N.m}^{-3}$ in red and for $R_L=R_T=10^{16} \text{ N.m}^{-3}$ in white.

On figure 5 are plotted the experimental dispersion curves for the sample T-D-100 which corresponds to a low level of adhesion. In red are plotted the numerical dispersion curves for $R_L=R_T=10^{14.5} \text{ N.m}^{-3}$ and in white for $R_L=R_T=10^{16} \text{ N.m}^{-3}$. One can see the better agreement with $R_L=R_T=10^{14.5} \text{ N.m}^{-3}$, confirming the low adhesion level of this sample.

In the case of the sample T-DSiO-100, the surface treatment has been manufactured using a release agent in order to create a kissing bond i.e. it is not an adhesion level problematic but a bonding problematic. On figure 6 are plotted the experimental dispersion curves for this sample. The numerical dispersion curves obtained with $R_L=R_T=10^{13} \text{ N.m}^{-3}$ (in yellow) and $R_L=R_T=10^{12} \text{ N.m}^{-3}$ (in red) are also plotted. Even if the superimposing is not perfect for $\log(R_L,R_T)=12$, the agreement is better than for $\log(R_L,R_T)=13$.

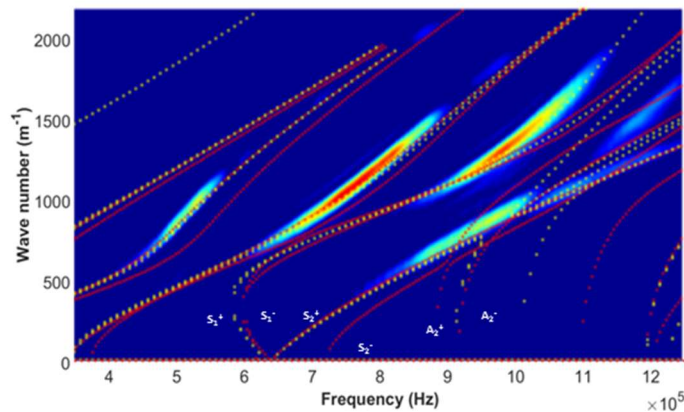


Figure 6: Experimental dispersion curves for the sample T-DSiO-100 superimposed with the numerical dispersion curves with $R_L=R_T=10^{13} \text{ N.m}^{-3}$ in yellow and for $R_L=R_T=10^{12} \text{ N.m}^{-3}$ in red.

In order to verify the creation of a kissing bond by the use of a mould release, a C-Scan has been performed with a transducer of 10 MHz central frequency, working as an emitter and receiver, and excited by an impulse. The transducer is translated by 2mm step on the x and y directions in the central area of the sample, where the acoustic measurements are performed.

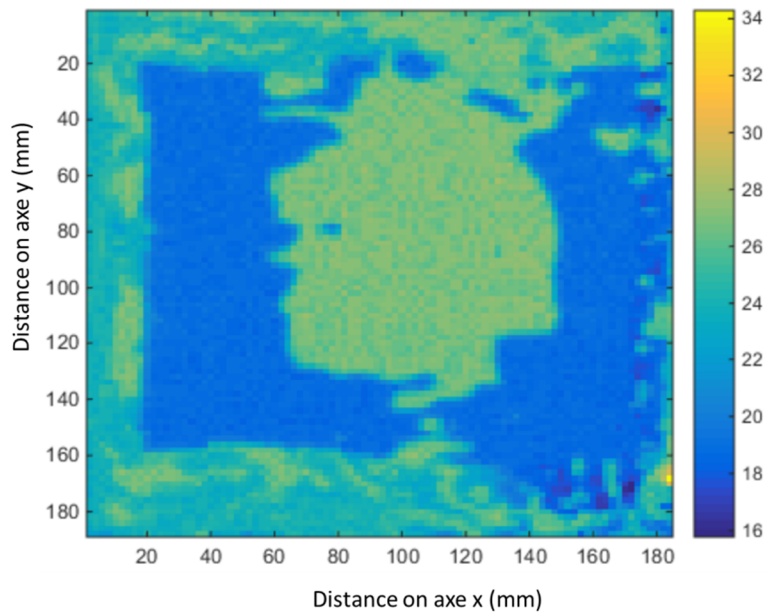


Figure 7 : C-Scan first echo of T-DSiO-100.

Figure 7 presents the obtained result. The principle of the measurement is to compare the amplitude of the reflected echo at the interface Aluminum/Epoxy, for different positions. In the case of good adhesion, a better transmission of the acoustic echo is found, and then a weak reflection should be measured by the transducer (blue color level in fig.7), while for a weak adhesion, the reflected echo at the interface should be more important (yellow color level in fig.7). One can see on Fig.7 that the central area of the sample T-DSiO-100 is in green-yellow color, that could be indicating a weak coupling between the Aluminum and the Epoxy.

3.2 Characterization using the vertical longitudinal mode

Vertical shear and longitudinal modes (noted V_T and V_L respectively) are appearing in three layer structures, and correspond to the characteristic equation solutions found next to the fundamental modes A_0 and S_0 . Several studies have shown their sensitivity to the physical coupling between the layers [12]. The aim here is to use this property for the adhesion level characterization.

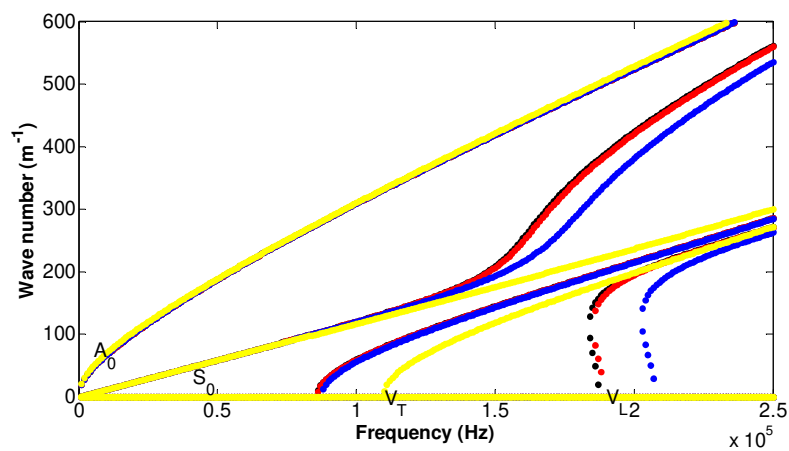


Figure 8: Theoretical dispersion curves obtained by numerical model for stiffness values equal to 10^{16} N/m³ (---), 10^{15} N/m³ (-.-), 10^{14} N/m³ (....) and 10^{13} N/m³ (—)

A numerical study is conducted to predict the variations of these modes with respect to the spring stiffness R_L and R_T . Figure 8 shows the dispersion curves obtained from the numerical model for a range of frequency [0 - 250kHz]. The black curves are obtained for $R_L=R_T=10^{16} \text{ N.m}^{-3}$ and the red ones for $R_L=R_T=10^{15} \text{ N.m}^{-3}$. Even if the most curves are superimposed, there is a small shift of the vertical longitudinal mode V_L around its cut-off frequency, while the shear vertical mode does not show any sensitivity. The blue curves are plotted for $R_L=R_T=10^{14} \text{ N.m}^{-3}$. The dispersion curve of the V_L mode is clearly shifted, and also the S_0 mode around 150 kHz, but not for the V_T mode which stills insensitive to these values of stiffness. Finally, the dispersion curves for $R_L=R_T=10^{13} \text{ N.m}^{-3}$ corresponding to a beginning of delamination are plotted in yellow. The V_T mode is clearly shifted around its cut-off, as the dispersion curve of the S_0 mode, while the dispersion curve of the V_L mode does no more appear in this range of frequency. These results show that the cut-off frequency of the V_L mode is predicted to be a good indicator on the quality of the bonding.

The aim is to check experimentally the cut-off frequency shift of the vertical longitudinal mode V_L in the three studied samples, as the measurement of the frequency cut-off of a given mode is simple to achieve experimentally by collecting the time multi-reflected echoes in the sample section and performing a FFT calculation.

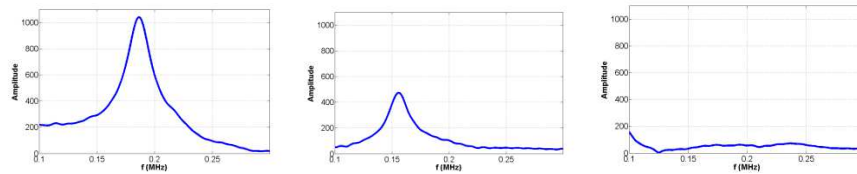


Figure 9: Experimental cut off frequency of the VL mode for T-DSi-80 (a), T-D-100 (b) and T-DSiO-100 (c). The sample T-D-100 is of 1 mm of epoxy thickness and T-DSi-80 and T-DSiO-100 of 0.5mm of epoxy thickness.

The FFT results are finally plotted in a range of frequency between 100 and 300 kHz and are given on Fig. 9 for each sample. The frequency peak is collected and compared to the numerical value. Results are summarized in table 4.

For the sample T-DSi-80 (fig. 8a), the experimental peak is at 187 kHz, which corresponds exactly to the expected frequency cut-off given by the numerical model in the case of perfect adhesion $\log(R_L, R_T)=16$, confirming the high adhesion level of this sample.

For the sample T-D-100 the experimental value measured is 153 kHz (fig. 8b). The predicted cut-off frequency, taking into account the Epoxy thickness film of 1 mm for this sample, and $\log(R_L, R_T)= 14.5$ given by the use of Lamb modes on fig. 5, is 147 kHz. Both values are in good agreement, differing by only 4,5%, which confirm the low adhesion level of this sample.

For the sample T-DSiO-100, manufactured with the applying of one layer of mould release in order to decrease critically the level of adhesion and to reveal a beginning of delamination, no cut off frequency is found in the same frequency range, as shown on fig.8c. This result is in accordance with the predicted theoretical study using $\log(R_L, R_T)=13$ where no cut off frequency of the V_L mode is observed, which could be explained by a beginning of delamination in the structure.

Finally, these results confirm the predicted ones about the sensitivity of the vertical longitudinal mode, and show that a simple and easy measure of its cut-off frequency can give information on the state of the adhesion in the

structure. In particular, the measure of the cut off frequency can assess a bonding problem, i.e. a beginning of delamination.

Sample	T-DSi-80	T-D-100	T-DSiO-100
log(R_L)	16	14.5	<13
Numerical frequency (kHz)	187	147	/
Experimental frequency (kHz)	187	153	/

Table 4: Numerical and experimental results of the cut off of the VL mode for the three level of adhesion. The sample T-D-100 is for 1 mm of epoxy thickness and T-DSi-80 and T-DSiO-100 for 0.5mm of epoxy thickness.

4. Conclusion

After using the rheological Jones model for the characterization of the level of adhesion in a bi-layer, the aim of this paper is to validate this model for a structural bonding assembly. Furthermore, it proposes an easy method to implement in order to qualify the quality of a bonding based on the cut-off frequency measurement of the vertical longitudinal mode. First the sample manufacturing in order to obtain three different levels of adhesion is presented. Then, a modeling of the interfaces by the Jones model allows associating spring stiffness values to the different samples and discriminates each of them by their level of adhesion: a very good one, a low one and a very bad, near to the delamination. A numerical study based on the Jones model shows a sensitivity of the vertical longitudinal mode cut-off frequency. An easy and fast experimental study shows that the measurement of its cut-off frequency in normal incidence allows detecting different adhesion levels, and in particular a beginning of delamination. This method shows its efficiency and, compared to the one using guided Lamb modes at frequencies higher than their cut-off, takes the advantage of being simple and quick to implement.

Acknowledgement

The authors would like to thank the ANR (Agence Nationale de la Recherche), through the ISABEAU project (project ANR 12-BS-09-0022-01), for providing funds for this research work.

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