

# ULTRASONIC SPECTROSCOPY TO EVALUATE ADHESIVES LAYERS

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**ABSTRACT.** Dual beam ultrasonic spectroscopy system was developed to measure quantitatively properties of adhesively bonded structures. The novelty of this approach is that it utilizes both normal and oblique incident ultrasonic beams on the bond line simultaneously and measures the frequency response of the reflected ultrasonic signals. In addition, a low frequency dynamic loading is added to the system, which acts as a parametric enhancement of weak bond characterization. The low frequency excitation parameters are set to make the maximum stress distribution coincide with the bond line. Ultrasonic measurements of adhesively bonded metallic and composite structures are presented.

## INTRODUCTION

The spectroscopic technique [1] is a valuable tool in adhesive bonds characterization. Angle beam ultrasonic spectroscopy at normal and oblique incidence [2] can be used for adhesive bonding layer properties determination. To model imperfect bonds, to select the optimal experimental conditions and to relate ultrasonic signature to joint quality we use the spring model of the interface [3,4] between adhesive and the substrates. When the shear spring stiffness  $K_t$  is infinitely large, the interfacial bond is perfect; when  $K_t$  is infinitely small, the interfacial bond has no resistance to shear stress, i.e. it degenerates into an ideal slip bond

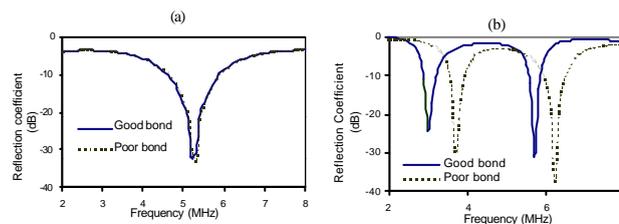


Figure 1: Bondline frequency response at normal (a) and oblique (b) incidence.

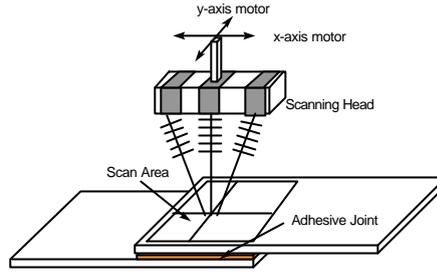


Figure 2: Transducer head

(total disbond). Thus  $K_t$  can be used as a quantitative parameter to describe the extent of interface damage. Using this model we can evaluate critical parameters used in angle-beam spectroscopic inspection techniques, such as angle of incidence, selection of transducer frequency and position of the spectral minima. Figure 1a shows reflection spectra from the bondline at normal incidence for poor and good interface conditions. The results are identical, indicating that normally incident waves are insensitive to the bond quality. Figure 1b shows the same but for obliquely incident waves. In that case, a significant shift occurs for a shear disbond (solid line) from that off a perfect bond (dashed line). This indicates the significance of the oblique incidence measurement.

## THE ANGLE BEAM ULTRASONIC SPECTROSCOPY

### Method description

For adhesive bond characterization we employ the linear and nonlinear methods. In linear approach, we combine obliquely and normally incident ultrasonic beam spectroscopy. The two angle measurements allow decoupling elastic moduli and thickness. To do this we developed a transducer head schematically shown in Figure 2. The head accommodates transducers for the normal and oblique measurements. The ultrasonic wave excited by the transducer is reflected from the interface toward a reflector and returns back to the transducer. The procedure used to scan the quality of the thin adhesive layer embedded between two composite plates is twofold. At each point of the scan, the normal and angle beam time domain signals reflected from the layer are recorded. Then they are analyzed in the Fourier domain and processed using our algorithms to obtain the quality of the bond line. The time signals are recorded using the transducer head. The transducer head is moved over the sample from point to point and each acquisition is recorded. After acquisition of the time signals, the second step is analysis in the Fourier domain of these signals to compute the relevant parameters for the layer properties determination. The final result is displayed as a bond line quality color image in form of the point-by-point bondline quality reconstruction for scanned area.

An inversion algorithm developed allows simultaneous determination of interfacial spring and adhesive bulk properties from normal and oblique reflection spectra. It based on a previously developed algorithm [2] dedicated to the determination of bulk properties (thickness, moduli, attenuation and density of embedded layers). The inversion procedure in this work is extended to include the reconstruction of normal and transverse interfacial stiffness.

The reflection spectrum depends on ten parameters: elastic moduli, thickness, density, longitudinal and shear attenuations and complex normal and shear interfacial spring constants (which represent four parameters: two real and two imaginary):

$$l, m, h, r, a_l, a_t, k_n = k_n' + ik_n'', k_t = k_t' + ik_t''.$$

This is increased by four parameters compared to previous results [2], where interface springs were neglected. One can define two groups of nondimensional parameters. The normal reflection spectra are dependent of the five nondimensional parameters:

$$Z_n = \frac{Z_l}{Z_s}, H_l = \frac{H\mathbf{w}_0}{V_l}, K_n = \mathbf{w}_0 k_n Z_l, \mathbf{a}_l. \quad (1)$$

We choose  $\mathbf{w}_0 = 1\text{MHz}$  for convenience. In addition to the four parameters defined in (1), we define four parameters measured at oblique incidence:

$$H_{lq} = \frac{H\mathbf{w}_0}{V_l} \cos(\mathbf{q}_l), H_{lq} = \frac{H\mathbf{w}_0}{V_l} \cos(\mathbf{q}_l), K_l = \mathbf{w}_0 k_l Z_l, \mathbf{a}_l \quad (2)$$

The unknown variables are fully defined by two sets of nondimensional parameters specified in Eqs. (1 and 2). First the normal and oblique spectra are measured experimentally. Second the parameters defined by Eq. (1) are obtained from the normal spectrum using a least square optimization algorithm. Then these reconstructed parameters and the oblique reflection spectrum are used to obtain other parameters defined by Eq. (2). After all nondimensional parameters have been determined, the dimensional parameters can be found.

To reconstruct the parameters the least square algorithm is used for the minimization of the sum of squared deviations between the calculation  $R_t(\mathbf{X})$  and the experimental reflection  $R_e(\mathbf{X}_0)$ :

$$ER(\mathbf{X}, \mathbf{X}_0) = \int_{f_1}^{f_2} (R_t(\mathbf{X}) - R_e(\mathbf{X}_0))^2 df \quad (3)$$

The least square algorithm searches one of the minimums of the error function in the five-dimensional parameter space. The initial guesses and accuracy of the measurement will affect the convergence of the algorithm. The data scatter also is an important factor for reconstruction precision. To simulate this effect the spectrum with noise is obtained by introducing random noise into the time domain signal calculated by the beam model. The reconstruction error is within 1% even for 2% scatter error in the experimental data.

To enhance interfacial property measurements and to relate the measurement to the local bond strength we use a stress modulation method. We combine excitation of a low frequency resonance vibration of the bonded sample with simultaneous determination of the interfacial stiffness using dual beam ultrasonic spectroscopy. We assume that the interfacial stiffness is independent of stress for a strong bond (it is assumed that the stress is below the adhesion strength between an adhesive and a substrate). In the case of a poor bond, interfacial stiffness decreases under tension due to the failure of molecular bonds in areas of low bond density. This is due to the fact that the load is transferred through fewer molecular bonds until it reaches the failure load.

We utilize Angle Beam Ultrasonic Spectroscopy for weak bond evaluation (as shown in Figure 2) by combining it with a dynamic load acting as a nonlinear (parametric) enhancement. Figure 3 shows schematically our approach. A tension and compression load cycle is applied on the bond using the first thickness resonance of the whole structure. The low frequency excitation parameters are set to make the maximum stress distribution coincide with the bond line. The stress applied on the bondline is alternately in compression and tension depending on the phase of the thickness resonance. The bond layer properties are evaluated using Angle Beam Ultrasonic Spectroscopy for different states of stress of the bondline. This is achieved by controlling the synchronization between the low frequency continuous excitation of the structure and the high frequency pulses used for the spectroscopic measurement. On the right the figure

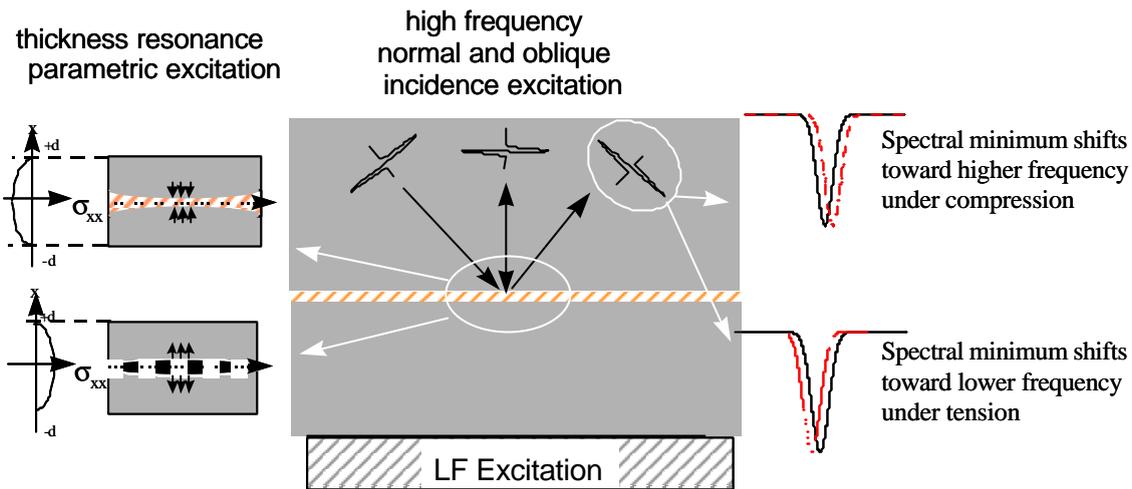


Figure 3: Proposed approach: Angle Beam Ultrasonic Spectroscopy of Adhesive Joints under thickness resonance parametric excitation

schematically illustrates that for an imperfect bond the spectra minimum shifts under the loading.

#### Experimental apparatus for stress modulated Angle Beam Ultrasonic Spectroscopy and method demonstration

A dedicated experimental system for Stress Modulated Angle Beam Ultrasonic Spectroscopy was designed and built. Two methods of excitation of low frequency vibration have been used in the feasibility study: one using a high power ultrasonic transducer in the frequency range 14-20 kHz and a second using tens of Hz cycling using a electromechanical or hydraulic actuation testing machine. This second type of experiment simulates the pressurization cycles often used for tests of aerospace structures. The implementation of the method is shown in Figure 4. The low frequency vibration is excited by a shaker with the frequency controlled by a function generator, the signal of which is applied to the shaker through a power amplifier and impedance matching unit. The bonded sample is excited by the shaker and the vibration amplitude on the sample is measured by an accelerometer. This setup was used for measurements in the mixing mode of the low frequency vibrations and the reflected ultrasonic signal. For perfect bonds no mixing is expected; for weak interfaces the boundary springs change under load leading to mixing higher and low frequency vibrations.

Two measurement approaches were used to record the vibration mixing. One is to directly digitize the high frequency signals with a 12 bit 125 MHz digitizer followed by signal gating and FFT. In a second approach after amplification the signal is fed through the low path filter and the analog amplitude detector, then the low frequency amplifier and is digitized by the 16 bit digitizer.

## EXPERIMENTAL RESULTS

Our approach to characterization of local (micro-structural) interfacial properties is based on the dependence of the reflection spectrum on molecular bond density, which is modeled as an equivalent interfacial spring density. The change of equivalent interface spring density under stress is sign of weak adhesion. The procedure used to determine the spring density at the

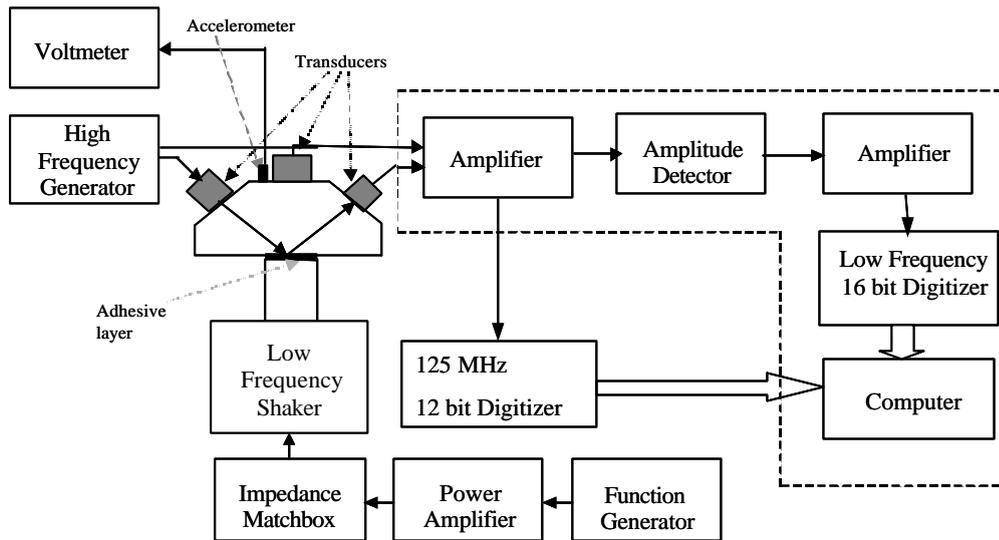


Figure 4: Schematic of the experimental setup for stress modulated ultrasonic spectroscopy in vibration mixing mode

substrate/adhesive layer interface is twofold. First time domain signals, reflected from the bonding layer, are recorded using the experimental configuration shown in Figure 4. Then they are analyzed in the Fourier domain and processed to obtain the frequency reflection spectra; second, from the normal and oblique reflection spectra, the elastic moduli, the thickness of the adhesive layer and interfacial spring stiffness are reconstructed using an inversion algorithm. With this method, the effect of bulk adhesive properties are separated from that of the interface.

Stress Modulated Ultrasonic Spectroscopy measurements have been performed on as-manufactured (reference) samples and environmentally degraded samples. For each sample, adhesive layer properties and interfacial stiffness have been determined at different levels of stress applied to the bond line. When measurements performed at a central point of the sample where degradation does not occur, there is no displacement of spectra minima at different loads. This indicates good bond quality. A different situation occurs for points off the sample center as shown in Figure 5. The ultrasonic reflected signals are collected at different points of the loading cycle. The spectra of the reflected signals are shown in figures (a), (b) for compression and tension parts of the loading cycle. There is no frequency minima shift during the compression part of the loading cycle (c), however the minimum shift occurs during the tensional part. The reflection minimum shifts towards lower frequency with increasing tension load indicating reduction of the interfacial spring. An example of spring reconstruction is shown in Figure 5(d) indicating the interfacial spring stiffness reduction versus tension load.

When environmental degradation is much more severe the situation is different. There is no reflection minimum during the tension part of the loading cycle indicating local disbond near the edge (ultrasonic wave doesn't show the presence of the adhesive layer). However under compression the disbond closes establishing good acoustic contact between adhesive and substrate. The ultrasonic signal is reflected from the bond-line with a characteristic minimum, which is shifted toward higher frequency with increase of compression load.

The provided results indicate how the stress-modulated method enriches ultrasonic results adding an additional dimension to data understanding and interpretation.

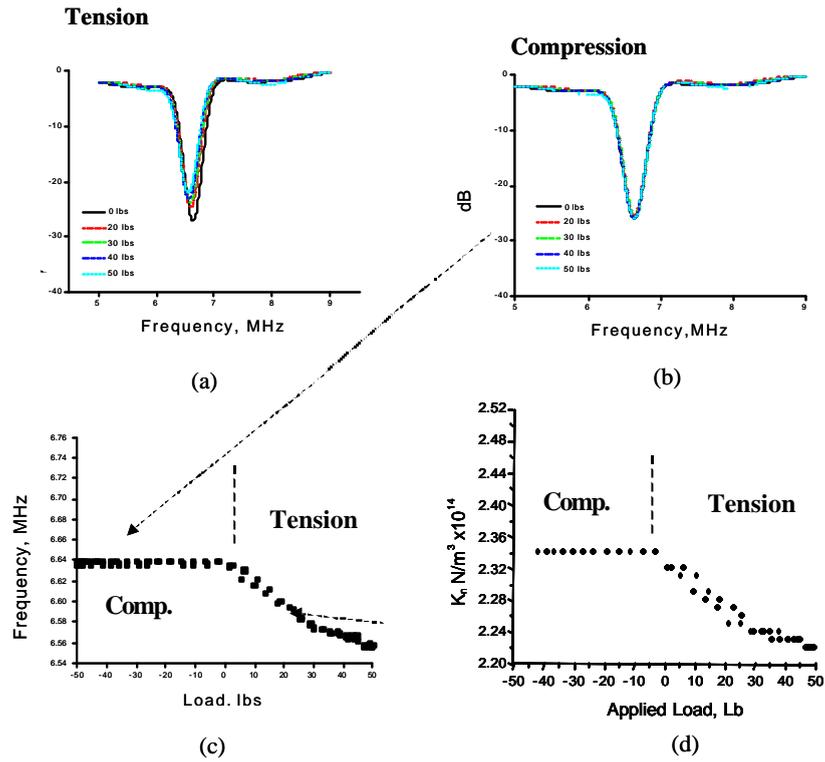


Figure 5: Spectra of the reflected ultrasonic signals near the edge of the environmentally degraded adhesively bonded sample under cyclic load. (a, b) Spectra of the reflected signals at different loads: (a) – Tension (positive), (b)– compression (negative). Shift of the minimum frequency occurs under tension indicating decrease of the number of molecular bonds under tension and creation of micro disbondings. There is no frequency shift under compression. c) Change of reflection minimum frequency as function of load d) decrease of interfacial spring during the tension part of the applied loading cycle.

## REFERENCES

1. D.W. Fitting, and L. Adler, , *Ultrasonic Spectral Analysis for Nondestructive Evaluation*, Plenum Press, New York (1981)
2. A.I. Lavrentyev and S. I. Rokhlin, *J Acoust. Soc. Am.* **102**, 3467-3477 (1997)
3. J. M. Baik, and R.B. Thompson, *J. Nondestr. Eval.* **4**, 177-196 (1984)
4. A.I. Lavrentyev and S.I. Rokhlin, *J. Appl. Phys.* **76**, 4643-4650 (1994)