Methods Development for Non-Destructive Measurement of Bond Strength in Adhesively Bonded Structures

James A. Seydel
University of Michigan–Ann Arbor

Follow this and additional works at: http://lib.dr.iastate.edu/cnde_yellowjackets_1975

Part of the Materials Science and Engineering Commons, and the Structures and Materials Commons

Recommended Citation
http://lib.dr.iastate.edu/cnde_yellowjackets_1975/39

This 8. Adhesives and Composites is brought to you for free and open access by the Interdisciplinary Program for Quantitative Flaw Definition Annual Reports at Iowa State University Digital Repository. It has been accepted for inclusion in Proceedings of the ARPA/AFML Review of Quantitative NDE, June 1974–July 1975 by an authorized administrator of Iowa State University Digital Repository. For more information, please contact digirep@iastate.edu.
Methods Development for Non-Destructive Measurement of Bond Strength in Adhesively Bonded Structures

Abstract
While reasonably satisfactory methods exist for nondestructively determining the presence or absence of an adhesive bond, no method exists for nondestructively determining the strength of what appears to be a good bond. Bonds that do not attain their full strength can be caused by a thin layer of contaminant, improper surface roughness, and a variety of other surface phenomena that are difficult to detect by conventional methods.\textsuperscript{1,2,3} One technique is to use an ultrasonic pulse to investigate the bond interface. Since the amplitude of the pulse reflected by the bond interface is a function of the elastic properties of the bond, it should be possible to correlate the absolute ultrasonic reflectivity with the bond strength.

Disciplines
Materials Science and Engineering | Structures and Materials
METHODS DEVELOPMENT FOR NON-DESTRUCTIVE MEASUREMENT OF BOND STRENGTH IN ADHESIVELY BONDED STRUCTURES

James A. Seydel
University of Michigan
Ann Arbor, Michigan

While reasonably satisfactory methods exist for nondestructively determining the presence or absence of an adhesive bond, no method exists for nondestructively determining the strength of what appears to be a good bond. Bonds that do not attain their full strength can be caused by a thin layer of contaminant, improper surface roughness, and a variety of other surface phenomena that are difficult to detect by conventional methods. One technique is to use an ultrasonic pulse to investigate the bond interface. Since the amplitude of the pulse reflected by the bond interface is a function of the elastic properties of the bond, it should be possible to correlate the absolute ultrasonic reflectivity with the bond strength.

There are several problems with this approach. It has proved quite difficult to construct a mathematical model which will predict the reflectivity as a function of the elasticity, damping, and density of the adhesive. Even if only statistical correlations are sought, the absolute reflectivity is a difficult quantity to measure accurately since small changes in incident angle, surface roughness, and base material attenuation can also affect the absolute reflectivity. And from a materials viewpoint, the bond strength may not be simply related to such linear material constants as the elasticity, damping, and density, and the stress at the interface due to the ultrasonic wave is orders of magnitude lower than the stress used to measure the bond strength.

This project addressed only the problem of making an accurate measurement of the relative reflectivity. Existing mathematical models which treat the adhesive bond as an interface between two systems of springs, dashpots and inertial mass show that the acoustic impedance, and thus the acoustic reflectivity is a function of frequency, and that the form of this function will vary for changes in mass, damping, and elasticity. These mechanical parameters of the adhesive bond would be expected to vary with different states of cure and over areas of improper surface preparation. The reflectivity as a function of frequency is a relative measurement and can be made insensitive to such disturbing influences as base material attenuation and incident angle. Thus, it should be possible to characterize the mechanical parameters of the adhesive bond by a reflectivity measurement that is easier to make and more accurate than an absolute measurement.

This approach has the additional advantage that both the magnitude and phase of the reflectivity are measured. The phase measurement is especially important when dealing with adhesive bonds that are thinner than the acoustic wavelength. Under these conditions, the adhesive contributes an acoustic reactance term that primarily affects the phase portion of the reflectivity function.

*Work supported under AFML Contract F33615-75-C-5134, and reported to AFML as Contract Final Report, January 1, 1975 - June 30, 1975.
+ Present address, Nuclear Engineering Department, University of Missouri, Columbia, Missouri

613
Experimental Method

Figure 1 is a schematic diagram of the automated ultrasonic test system used for this program. Once every ultrasonic pulse period, the computer issues a "trigger" command to the programmable digital delay generator (DDG), which immediately triggers the ultrasonic pulser. When the ultrasonic echo of interest returns to the transducer, the DDG sends a "sample" command to the SHA and a digital sample is taken. The computer controls the time delay between the trigger command and the sample command by loading the DDG with a digital number which represents this time delay in increments of 10 msec. The system is configured so that a conventional RF mode ultrasonic pulse enters the sample-and-hold amplifier (SHA), where a single sample is extracted, converted to a digital format by the analog-to-digital converter (ADC), and stored in the computer memory. The number of samples, the time delay to the first sample, the time interval between samples, and the ultrasonic pulse period are all controlled by the computer and can be set by the operator through the teletype. Typically, a number of data sets are collected and stored on the magnetic disk. Later, they are recalled and processed. The processing is accomplished by a software Data Analysis Routine (DAR). DAR is capable of taking forward and inverse fast Fourier transforms (FFT) of both real and complex data, performing rectangular to polar conversions, calculating power spectra, constructing a variety of filters, differentiating and integrating functions, and other basic data analysis tasks.

As discussed earlier, the ultrasonic system was set up to observe the adhesive interface. The electronic gates (analog) shown in Fig. 1 are adjusted to exclude superfluous reflections as a data set is collected. Since a relative measurement is desired, there is no need for a reflectivity calibration. The collected data set is then Fourier transformed and converted to a polar (magnitude and phase) format. At this stage, the data is a measure of the frequency dependent reflectivity of the adhesive interface as seen through a frequency dependent piezoelectric transducer. To remove the frequency dependence of the transducer, the following procedure is used. First, a transducer reference data set is collected from the front surface of the bond sample (a water-aluminum interface). This reference data set is collected only once but used for all subsequent processing. Second, this reference data set is Fourier transformed and divided into the data containing both the transducer and interface characteristics. Since the transducer acts as a linear filter to the adhesive data, this technique effectively removes the frequency dependent characteristics of the interface remain. After the data has been thus normalized, it is plotted for subsequent correlation analysis.

During the program, each test sample was examined at many different positions, but for the purposes of this report, only the test results from one of these positions are included. In addition to the samples supplied by the Air Force Materials Laboratory, some samples of an aluminum-polyester bond were fabricated with a polyester thickness of about 10 mm. This was done to provide a comparison with the thin (~100 μm) adhesive interfaces supplied by the Air Force.
Fig. 1. Automated Ultrasonic Test System.
Experimental Results

The frequency profile of the 10 MHz transducer used in this program is shown in Fig. 2. The maximum magnitude occurs at 9.7 MHz and within a 10 MHz passband around that frequency, the phase response is quite linear. The automated ultrasonic system has been designed to maintain a high degree of accuracy, and, as a result, accurate measurements can be made at power levels 40 dB below the maximum response. For this particular transducer the 40 dB points are below 1 MHz and above 25 MHz. Thus, the frequency graphs show the reflectivity from 25 kHz to 25 MHz in steps of 25 kHz. Since there are 1024 digital samples on each graph, the curves appear to be continuous.

The accuracy of the magnitude and phase at each frequency is limited by the SHA and ADC. Both are designed for 12 bit accuracy and the SHA has a 200 psec aperture jitter. For a 25 MHz signal, these specifications imply a phase accuracy of 2° and a magnitude accuracy of 0.5%.

The graphs of the ultrasonic reflectivity as a function of frequency are shown in Figs. 4-9. For each test sample, the time signal and the magnitude and phase angle of the reflectivity are shown first. These graphs are followed by the transducer-normalized data. This format is repeated for each test sample. These figures show the reflectivity data from the adhesive bond between two pieces of 3.2 mm thick aluminum. For this thickness of aluminum, it is possible to gate out every reflection except the adhesive-aluminum interface. The time position of the gated signals for Figs. 4-9 are shown inside the box on Fig. 3, which is a complete time scan of the ultrasonic signal received from the 3.2 mm thick aluminum sample.

When comparing the results from the various test pieces, primary attention should be paid to the transducer-normalized data. For the magnitude data, the first horizontal line above the axis represents a magnitude ratio of 1.0. Since all the measurements are relative, it is possible for the magnitude ratio to exceed 1.0. For the phase data, the FFT calculates only a relative phase angle which begins at either 0° or 180°. On the phase angle graphs, a horizontal line is placed every 180°. The absolute value of the phase angle is meaningless, and only relative phase changes as a function of frequency are meaningful.

Since the bond strength of the test samples is not known (it will be established later by destructive tests, see the Appendix) a correlation of ultrasonic data with bond strength data is not possible. Instead, the following interpretation will emphasize the distinctive features of only the ultrasonic data.

The ultrasonic data (from 4 to 22 MHz) taken as a whole indicate that there is very little, if any, correlation with the different types of surface preparation existing on the test samples. This conclusion is most easily seen by comparing the data shown in Figs. 4d-9d and Figs. 4e-9e. There are some minor deviations in the magnitude ratio, but overall, the normalized responses are virtually identical. Outside the range of 4-22 MHz, both the normalized magnitude and phase response show some variation. Figures 6e and 7e have a 360° phase jump relative to Fig. 6e at the low frequencies.
Fig. 2. Test results for a 10 MHz ultrasonic transducer; (a) spectral magnitude, and (b) phase angle as a function of frequency.
Fig. 3. An RF A-scan display of the ultrasonic echoes from the 3.2 mm thick aluminum plate test sample.
Test results from the adhesive interface on the test sample labelled "as received"; (a) A-scan display of the echo; (b) spectral magnitude; (c) spectral phase angle; (d) normalized spectral magnitude; and (e) normalized spectral phase angle.
Fig. 5. Test results from the adhesive interface on the test sample labelled "solvent wiped only"; (a) A-scan display of the echo; (b) spectral magnitude; (c) spectral phase angle; (d) normalized spectral magnitude; and (e) normalized spectral phase angle.
Fig. 6. Test results from the adhesive interface on the test sample labelled "solvent wiped and abraded": (a) A-scan display of the echo; (b) spectral magnitude; (c) spectral phase angle; (d) normalized spectral magnitude; and (e) normalized spectral phase angle.
Fig. 7. Test results from the adhesive interface on the test sample labelled "FPL etch without alkaline cleaning"; (a) A-scan display of the echo; (b) spectral magnitude; (c) spectral phase angle; (d) normalized spectral magnitude; and (e) normalized spectral phase angle.
Fig. 8. Test results from the adhesive interface on the test sample labelled "FPL etch with half alkaline cleaning": (a) A-scan display of the echo; (b) spectral magnitude; (c) spectral phase angle; (d) normalized spectral magnitude; and (e) normalized spectral phase angle.
Fig. 9. Test results from the adhesive interface on the test sample labelled "FPL etch"; (a) A-scan display of the echp; (b) spectral magnitude; (c) spectral phase angle; (d) normalized spectral magnitude; and (e) normalized phase angle.
At around 5 MHz, Fig. 9e has a phase jump of 180° relative to Fig. 6e. Thus, it appears that the data can be separated into three groups: Figures 4 through 5, Fig. 7, and Fig. 8 and 9. The first group contains the samples labelled "As Received", "Solvent Wiped Only", and "Solvent Wiped and Abraded". The second group contains the "FPL Etch without Alkaline Cleaning", and the third group contains the "FPL Etch with Half Alkaline Cleaning" and the "FPL Etch". Generally, it is felt that these group distinctions are rather subtle, and that if the destructive testing shows any correlation, these high and low frequencies should be more thoroughly investigated.

The dip at about 11 MHz in the magnitude response is due to an anti-resonance condition in the adhesive layer. As can be seen, this dip occurs at a slightly different frequency for each test sample, but this merely indicates a slightly different adhesive thickness for each sample. Using this anti-resonance model, the adhesive thickness is an odd multiple of 60 μm. This calculation assumes that the speed of sound in the epoxy materials is 2.7 km/sec, since it was impractical to measure this velocity in such thin samples.

In order to remove the effect of adhesive thickness on the reflectivity data, an aluminum-epoxy interface was constructed with an epoxy layer about 10 mm thick. Two data sets were taken from this sample. The first data set, shown in Fig. 10, was from the aluminum-epoxy interface. The second data set, shown in Fig. 11, was from an aluminum-water interface on the same test sample. Figure 12 displays the results obtained by normalizing the first data set by the second data set. Figure 12 should display the differences between the presence and absence of epoxy on the aluminum surface.

From 5 to 17 MHz, both the normalized amplitude and phase are virtually flat. At about 20 MHz there is a 360° phase jump and a corresponding null in the amplitude response. Below 5 MHz, the normalized amplitude increases. This data suggests that the significant effects occur either above or below the passband of the ultrasonic transducer, and that within the 5 to 17 MHz passband, no distinction can be detected between an aluminum-water interface and an aluminum-epoxy interface.

Summary

This program attempted to characterize adhesive bond strength by a measurement of the ultrasonic reflectivity as a function of frequency. An automated ultrasonic system which provides both magnitude and phase information was used to investigate the echo from the adhesive-substrate interface. This ultrasonic data would then be correlated with the bond strength to determine if this technique can be used as a nondestructive measure of bond strength.

The experimental results demonstrate that phase and magnitude data from a variety of adhesive interfaces are quite similar. A few points of distinction did exist and these were used to group the various test samples for correlation with subsequent destructive tests. The distinctions are quite subtle, however, and if a correlation with bond strength does appear, a more thorough investigation of the technique is recommended.
Fig. 10. Test results for an aluminum-epoxy (10 mm thick epoxy) interface; (a) spectral magnitude; and (b) spectral phase angle.

Fig. 11. Test results for an aluminum-water interface; (a) spectral magnitude; and (b) spectral phase angle.
Fig. 12. Test results for an aluminum-epoxy interface; (a) normalized spectral magnitude; and (b) normalized spectral phase angle.
Appendix

Subsequent to the presentation of this paper, representative adhesive bond specimens were destructively tested for the purpose of obtaining bond strength data. This information has been included in this Appendix since it complements the ultrasonic measurements presented above.

Lap shear specimens were prepared from adhesive bond specimens which were provided by the Air Force. These specimens were then destructively tested. Set 1 consisted of a number of 9 x 4 x 1/4" 2024-T4 aluminum panels bonded in pairs with FM 1000 epoxy materials. Within this set of specimens aluminum surfaces were prepared as:

**Set 1 - Test Specimens**
1. As received (no treatment)
2. Solvent white
3. Solvent white abraded (with steel wool)
4. FPL Etch without alkaline clear
5. FPL Etch with alkaline clear (1/2 normal time)
6. FPL Etch with full alkaline clear

**Lap Shear Strengths of Set 1 Specimens**
1. 3780 ± 10 psi
2. 4340 ± 10 psi
3. 4880 ± 10 psi
4. 7320 ± 10 psi
5. 6460 ± 10 psi
6. 7400 ± 10 psi

Set 2 consisted of 4 current specimens with variations in adhesive cure temperatures. Again these were 9 x 4 x 1/8" panels of 2425-T4 aluminum bonded in pairs with FM 1000 epoxy adhesives.

**Set 2 - Test Specimens**
1. Cured at 300°F
2. Cured at 350°F
3. Cured at 250°F
4. Cured at 350°F

The recommended cure temperature was 350°F. The strength is expected to be degraded with lower cure temperature.

**Lap Shear Strengths of Set II Specimens**
1. 6210 ± 10 psi
2. 6540 ± 10 psi
3. 600 (void detected)
4. 6440 ± 10 psi
References


DISCUSSION

MR. BILL MARTIN (Douglas Aircraft): Isn't the phase angle related to attenuation?

DR. SEYDEL: Yes, it should be. You can't say that it's simply related like that. If you look at just the simple linear model that takes into account the density, the attenuation and the compliance, the phase angle is a function of all those parameters.

MR. MARTIN: You mean a phase angle is a function of--

DR. SEYDEL: The phase angle is a function of all three of those parameters. Now, in certain circumstances it may be a strong function of one or the other. The problem here with this linear model is that you don't have a good feeling for what those parameters are.

MR. MARTIN: Can you use Fresnel's reflectivity equation?

DR. SEYDEL: Yes, except that you don't really need the Fresnel equations, because you're at normal incidence anyway. So, you don't need all the rigor of Fresnel.

DR. YIH PAO (Cornell University): I believe those dips you observed or peaks you observed in the spectrum should disagree with the principal frequency of the specimen. It is like the theory for the cylinder reported by Sachse in a previous paper. It's exactly the same thing. The dips or peaks are related to the standing wave frequencies of your aluminum-epoxy-aluminum layer model.

DR. SEYDEL: Okay. That could be.

DR. HENRY BERTONI (Polytechnical Institute of New York): It seems to me that you would get a much bigger difference between epoxy and water if you were looking at the shear wave propagation rather than the longitudinal wave. I mean, the difference between jello and water is not much different for a longitudinal wave, is it?

DR. SEYDEL: Yes. I wanted to do the shear wave, and I really wanted to have a shear plate transducer which would send out a shear wave normal to the surface, but I did not have one of those available. I think that is an experiment that needs to be done. Yes, definitely.