CHARACTERIZING THE CURING OF ADHESIVE JOINTS BY A NONLINEAR ULTRASONIC TECHNIQUE

Guoli Liu and Jianmin Qu
The George W. Woodruff School of Mechanical Engineering
Georgia Institute of Technology
Atlanta, GA 30332-0405

Laurence J. Jacobs
School of Civil and Environmental Engineering
Georgia Institute of Technology
Atlanta, GA 30332-0355

Jiayi Li
Changchun Institute of Art and Sciences
Changchun, China

INTRODUCTION

Adhesives and adhesive joints are widely used in various industrial applications to reduce weight and costs, and to increase reliability. For example, advances in aerospace technology have been made possible, in part, through the use of lightweight materials and weight-saving structural designs. Joints, in particular, have been and continue to be areas in which weight can be trimmed from an airframe through the use of novel attachment techniques. In order to save weight over traditional riveted designs, to avoid the introduction of stress concentrations associated with rivet holes, and to take full advantage of advanced composite materials, engineers and designers have been specifying an ever-increasing number of adhesively bonded joints for use on airframes.

Nondestructive characterization for quality control and remaining life prediction has been a key enabling technology for the effective use of adhesive joints. Conventional linear ultrasonic techniques can only detect flaws (delamination, cracks, voids, etc) in the adhesive. However, more important to the bond quality is the adhesive strength.

Although in principle, strength cannot be measured non-destructively, the slight nonlinearity in the material may indicate material degradation or the onset of failure. Furthermore, microstructural variations due to aging or under curing may also cause change in the third order elastic constants, which are related to the nonlinear acoustic

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parameter of the polymer adhesive. It is therefore perceivable that there might be a correlation between the changes in the nonlinear acoustic parameter and the remaining bond strength.

It has been observed that higher harmonics of the fundamental frequency are generated when an ultrasound passes through a nonlinear material. It seems that such non-linearity can be effectively used to characterize the bond strength. Several theories have been developed to model this nonlinear effect [1-5]. Based on a microscopic description of the nonlinear interface binding force, a quantitative method was presented by Pangraz and Arnold [6]. Recently, Tang, Cheng and Achenbach [7] made a comparison between experimental and simulated results based on a similar theoretical model. A through-transmission setup for water immersion mode-converted shear waves was used by Berndt and Green [8] to analyze the nonlinear acoustic behavior of the adhesive bond. In the meanwhile, ultrasonic guided waves have been used to analyze adhesive or diffusion bonded joints [9-11].

In this paper, the nonlinear parameter is used to characterize the curing state of a polymer/aluminum adhesive joint. Ultrasonic through-transmission tests were conducted on samples cured under various conditions. The magnitude of the second order harmonic was measured and the corresponding nonlinear acoustic parameter was evaluated. A fairly good correlation between the curing condition and the nonlinear parameter is observed. The results show that the nonlinear parameter might be used as a good indicator of the state of curing for adhesive joints.

THROUGH TRANSMISSION MEASUREMENTS

In order to measure the higher order harmonics and to correlate the amplitude of the higher order harmonics to the curing state of the adhesive joints, through transmission tests were conducted. This section discusses the sample preparation, test setup and the measurement methods used in the through transmission tests.

Test Samples

The test samples used in this study were provided by the Boeing Commercial Airplane Company. The sample is an overlap joint of two aluminum plates bonded together by an adhesive layer. The adhesive is a thermosetting modified epoxy, AF-163-2K, in sheet form (knit supporting carrier) made by the 3M Company. The aluminum plate is made of AL2024. The relevant material properties are listed in Table 1.

As illustrated in Fig. 1, the bonded area of the specimen is 12.7 cm x 17.8 cm (5.0" x 7.0"). The adhesive (bondline) thickness is approximately 0.32 mm (12.6 mils) and the adherend's thickness is 1.6 mm (63.3 mils), which yields a total joint thickness of approximately 3.54 mm (139.2 mils).

Table 1 The material properties of Al2024 and AF-163-2K

	ρ(kg/cm^3)	E(GPa)	ν
Al2024	2.78	73.0	0.3518
AF-163-2K	1.214	1.1084	0.34

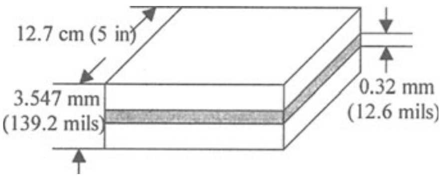

Fig. 1 Bond samples provided by Boeing Co.

Table 2 The curing conditions and the corresponding strength for the four samples

Sample Number	B1	B2	B3	B4
Curing Temperature (°C)	121	82	82	90
Curing Time (min.)	90	60	120	60
Curing Pressure (KPa)	345	345	345	345
Bond Strength (MPa)	35.35	4.01	3.27	4.10

The aluminum plates were anodized and primed prior to application of the adhesive. The joint was then put into a temperature/pressure oven for curing. All 4 samples used in this study were prepared under the same conditions except the curing schedule. The different curing schedules for the four samples are listed in Table 2.

The resulting variations in bond strength due to different curing schedules are also listed in Table 2. The normal (optimal) curing schedule is 121°C (250°F) for 90 minutes under 345KPa (50 psi). Sample A was cured under this condition. The other samples were cured under different curing schedules. It is seen that samples with different states of curing showed drastically different bond strength. It is hoped that such differences in the curing state can be characterized nondestructively and correlated with the higher order harmonics.

Experimental Setup

A block diagram of the experimental set up is shown in Fig 2. A 40 cycle time-harmonic signal of 2MHz was generated by a Waveteck function generator. The signal was amplified by a high voltage amplifier (ENT, DC ~ 10MHz, 50dB) to obtain a high amplitude driving voltage of the generating transducer. Typical output signals of the function generator and the amplifier are shown in Fig. 3. The highest output voltage of the amplifier used in the experiment was 350 volts. A narrow-band contact PZT transducer was used as the generating transducer. Its center frequency is 2MHz (Ultra, KC50-2, 1.25MHz at -6dB). The incident ultrasonic wave from the generating transducer was transmitted perpendicularly through the adhesive layer. The receiver is a narrow-band contact PZT transducer with 4MHz center frequency (Ultra, KC50-4, 3.5MHz at -6dB). The output signal $f(t)$ of the receiver was recorded by an oscilloscope (Techtronix, 150MHz) and analyzed on a personal computer.

The sample and the two contact PZT transducers were fixed by two aluminum plates with a cavity on each side, respectively, to hold the transducers at the same position as shown in Fig. 4. For efficient signal generation, a coupling liquid was used between the transducer and the sample. In addition, the two transducers could be held tightly by adjusting the four bolts.

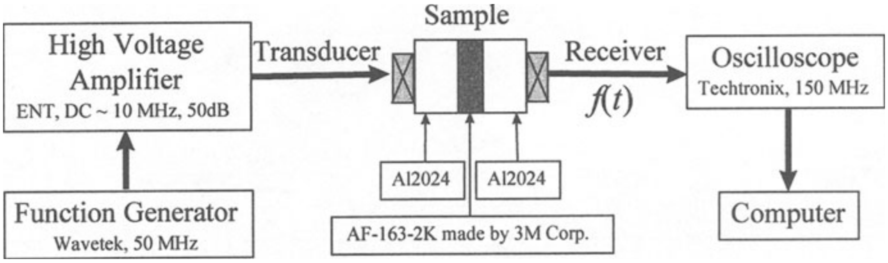

Fig. 2 Through transmission experimental setup.

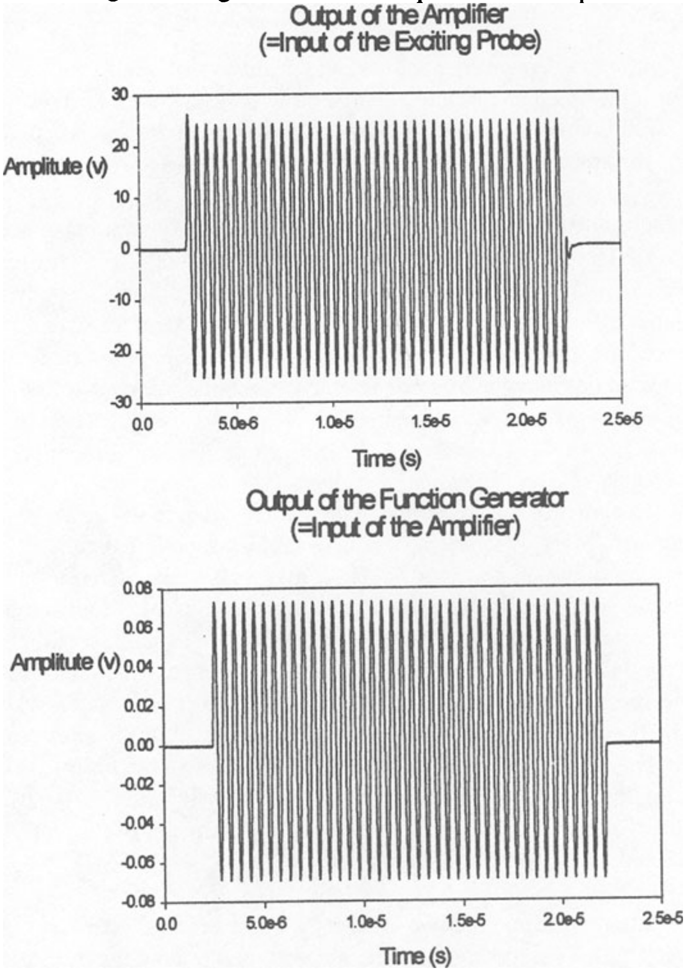

Fig. 3 Typical output signals of the function generator and the amplifier.

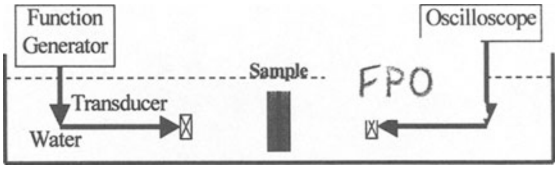

Fig. 5 The immersion test setup.

The amplitude of the higher order harmonic depends on the amplitude of the incident wave. Therefore, in order to compare the results from different samples, the amplitude of the incident wave that passes through the adhesive layer must be the same for different samples. However, even when the same incident voltage was used for all samples, variation may still exist due to sample variations and variations in the transducer/sample coupling. To compensate for such variations between different samples, water immersion tests were also conducted. A schematic of the immersion test is shown in Fig. 5. The transmission coefficient defined by

$$\alpha = \frac{V_t}{V_i} \tag{1}$$

was measured for each sample, where V_i and V_t are the amplitudes of the incident and the transmitted voltages, respectively.

Testing and Signal Processing

During the through transmission test, a 40-cycle time-harmonic signal of 2MHz was generated by a Waveteck function generator. The signal was then amplified by a high voltage amplifier (ENT, DC ~ 10MHz, 50dB) and sent to the generating transducer. The signal received by the receiving transducer, $f(t)$, was recorded by the oscilloscope. Finally, the data was processed through the Fast Fourier Transform (FFT) to obtain the

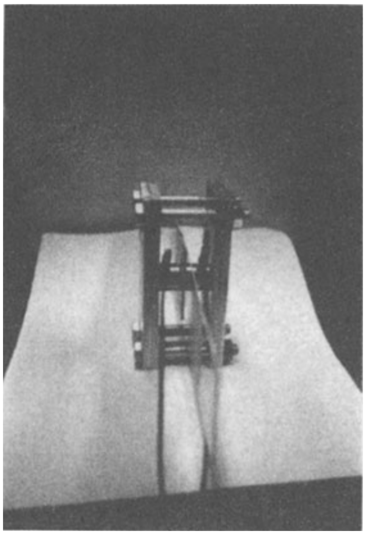

Fig. 4 Adhesive bond sample and the holding of the transducers.

frequency spectra,

$$F(\omega) = \int_0^{\infty} f(t)\exp(i\omega t)dt . \tag{2}$$

The amplitude of the fundamental frequency and the higher order harmonic components are defined as

$$A_n = |F(n\omega_0)|, \qquad n = 1,2,3\ldots \tag{3}$$

where ω_0 is the fundamental frequency of the generating transducer. In this study, $f_0 = \omega_0 / 2\pi = 2\text{MHz}$ was used.

The amplitude of the fundamental frequency component, A_1, in the received signal *f*(*t*) is plotted in Fig. 6 as a function of the incident voltage V_i. The linear relationship between A_1 and V_i confirms that the generation and receiving systems are operating in their linear regime within the voltage range used.

In addition, an aluminum plate with the same thickness as the bond samples was tested first in the same experimental setup. No appreciable higher order harmonics were observed. This indicates that the nonlinear effects from the test system (including the two PZT transducers, the amplifier, the function generator and the coupling liquid) can be neglected.

Following [12], the nonlinear parameter of the adhesive is defined by

$$\beta = \frac{8A_2}{k^2 h A_1^2} . \tag{4}$$

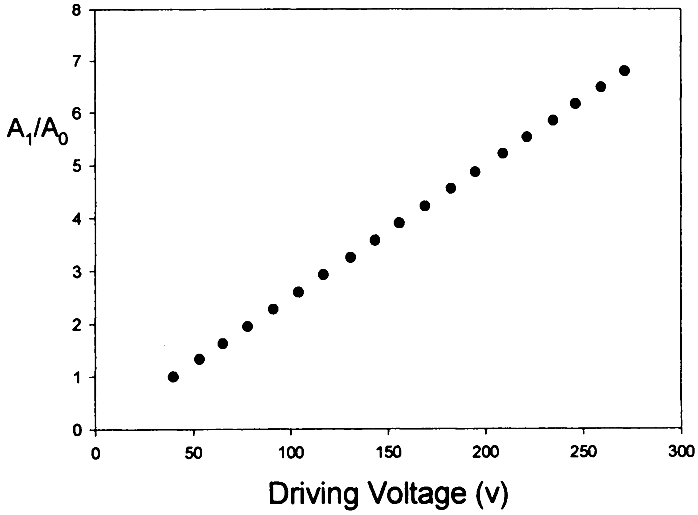

Fig. 6 Amplitude of the fundamental frequency vs. the driving voltage.

where k is the wave number and h is the sample thickness. This nonlinear parameter will be used to characterize the curing state of the adhesive joints.

Since β depends on the amplitude of the incident wave, care must be given to ensure that the same incident wave is used for all samples, if one needs to compare the β values between different samples. However, it is rather difficult to control the coupling when using contract transducers. To avoid this difficulty, the following technique was used.

First, for the same input voltage, the tightness of the transducer/sample assembly in each test is adjusted through the adjustable screws in the test apparatus so that the received signals have the same amplitude for all the samples. Obviously, this guarantees the same transducer/sample coupling if all the samples were identical. However, we have found that this is not the case. In fact, the water immersion tests shown in Fig. 5 indicated that the transmission characteristics are somewhat different between the samples. Therefore, tests with the same generation voltage and the same received signal amplitude effectively have different amplitude of the incident waves upon the adhesive. Thus, to compare different samples, the β defined by (4) must be adjusted by the transmission coefficient for each sample

$$\beta = \frac{8A_2}{\alpha k^2 h A_1^2}, \qquad (5)$$

where α is the transmission coefficient defined by (1). When the tightness of the transducer/sample interface is adjusted so that the transmitted voltage is the same for all samples, the β defined by (5) ensured effectively that the incident waves impinging upon the polymer adhesive are the same for different samples at the same generating voltage. This is because the amplitude of the transmitted waves is linearly dependent on the generating voltage, see Fig. 6.

RESULTS AND OBSERVATION

Values of the nonlinear parameter β were measured following the procedures described in the previous sections. Results for the four samples cured under various

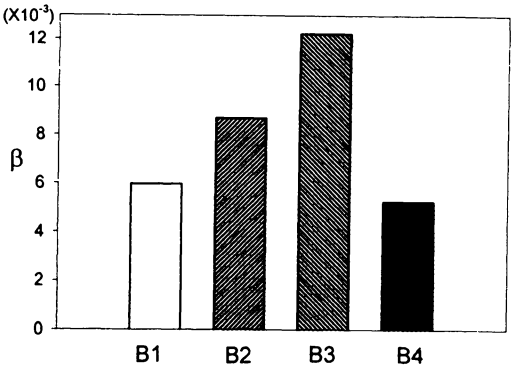

Fig. 7 The nonlinear parameter for the different samples.

curing schedules were obtained and presented in Fig. 7. The different curing conditions are given in Table 2. Note that sample B1 is considered cured under the optimal condition. Samples B2 and B3 are under cured (lower temperature and shorter time), although B3 is better than B2. However, B4 is unclear. It was cured under temperature lower than the optimal, but it had a longer curing time.

It is seen from Fig. 7 that under cured samples have higher non-linearity. The more under cured the sample is, the higher the nonlinear parameter β. The fact that sample B4 has slightly lower β seems to indicate that excessive curing time tends to reduce the nonlinear parameter β.

As a final remark, it must be mentioned that on one hand, the analytical and numerical analyses indeed indicate that higher order harmonics are generated by the material nonlinearity [13]. On the other hand, the test results show that there is a significant increase in the nonlinear parameter for under cured adhesives. However, the fundamental relationship between the curing state and the amount of nonlinearity in the adhesive is an open question. At the moment, all one can conclude is that there seems to be a correlation between the nonlinear parameter and the curing state. The quantitative relationship between the nonlinear parameter and the degree of under curing needs further investigation.

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