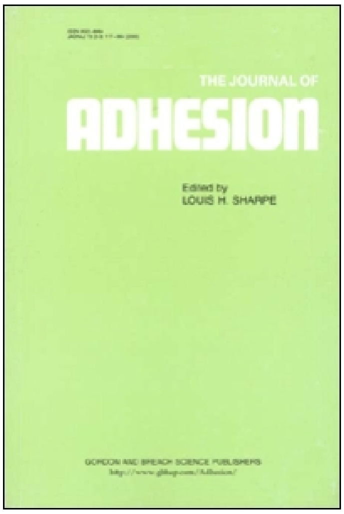

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Ultrasonic Determination of the Cohesive Properties of Bonded Joints by Measurement of Reflection Coefficient and Bondline Transit Time

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An ultrasonic nondestructive technique for the quantitative determination of the cohesive properties of adhesive joints based on the measurement of the reflection coefficient from the top adhesive/adherend interface and the bondline transit time has been developed. The method requires access to only one side of the joint and, for joints with typical aerospace geometries, it can be implemented using a single transducer with a centre frequency below 50 MHz. The technique has been used to determine the longitudinal bulk wave velocity in aluminium-epoxy-aluminium joints to within ±6% of the nominal values determined from bulk samples. The bondline thickness of the samples tested was evaluated to within micrometer accuracy, and thickness variations within the scan area were detected to much better than micrometer accuracy. The method has been tested successfully on joints made with two-part epoxies and with film adhesives containing a "scrim" carrier, and it has also been shown that the different standard adherend preparation procedures have a negligible effect on the results. The method, therefore, promises to provide a reliable, nondestructive means of measuring the cohesive properties of a bonded joint and represents a significant advance on the currently available technology.

KEY WORDS nondestructive evaluation; nondestructive testing; NDE; NDT; epoxy-aluminium adhesive joints; effect of surface preparation; two-part epoxy adhesives; film adhesives.

1. INTRODUCTION

Defects in adhesively bonded joints may be classified into three distinct types:[1] voids, disbonds and porosity which may, in general, be detected by conventional ultrasonic and other nondestructive evaluation (NDE) techniques; poor cohesive strength (*i.e.* a weak adhesive layer); and poor adhesive strength (*i.e.* a weak adhesive/adherend interface). The lack of reliable, robust, quantitative NDE techniques for these latter two types of defect has limited the application of adhesive

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bonding in safety-critical areas. In this paper, an NDE technique is described which can be used to detect poor cohesion in bonded joints.

The curing process radically changes the nature of the adhesive, and the cohesive properties that we choose to monitor should also be affected by the state of cure. Several authors[2,3] have reported the use of ultrasonic bulk wave velocities to measure the curing process in structural adhesives. It has also been shown[2] that the longitudinal and shear bulk wave velocities change by approximately the same proportion during the curing of an adhesive sample after gelling has occurred (*i.e.* after the shear velocity is no longer negligible). Generally, it is easier to excite and detect longitudinal waves so they have been used in this study. In addition, the thickness of the adhesive layer has an important effect on joint strength and should be measured as part of any NDE technique. Hence, the cohesive properties that are chosen for our NDE technique to evaluate are the thickness and longitudinal bulk wave velocity of the adhesive layer.

Guyott and Cawley[4] proposed an ultrasonic technique for the nondestructive determination of the cohesive properties of bonded joints based on the measurement of the frequencies at which the through-thickness vibrational modes of the joint occur. This technique was subsequently improved by Cawley and Hodson.[5] However, further investigation has shown that this method only works for a limited set of adhesive types and joint geometries.[6] The main problem with this technique is that because it measures the dynamic response of the entire joint, the results are dominated by the influence of the adherends which are typically an order of magnitude or more thicker than the adhesive layer, as shown in Fig. 1. This is particularly true of the higher order modes, in which the adherends are strained significantly.

At normal incidence, the reflection and transmission of a bulk wave of infinite extent from a boundary between two semi-infinite elastic half-spaces is determined by the acoustic impedances of the two media.[7] This, in turn, means that the reflection amplitude is determined by the product of the density and bulk wave velocity in each layer. Hence, by measuring the reflection coefficient at normal incidence between the adherend and adhesive in a bonded joint in which the bulk wave velocity and density of the adherend are known, the product of bulk wave velocity and density of the adhesive may be deduced. The assumption of a value for the density of the adhesive would then allow the bulk wave velocity in the adhesive layer (one of the required cohesive properties) to be calculated.

The reflection coefficient from the adhesive/adherend interface can only be measured easily if the reflection from the interface can be separated from the other reflections coming from the joint so that the adhesive layer and the top adherend can be considered to be "semi-infinite." Since the adherends in aerospace applica-

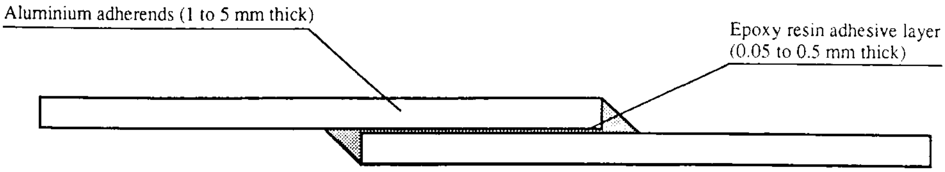

FIGURE 1 Typical aerospace geometry joint.

tions are usually an order of magnitude thicker than the adhesive layer, this criterion will be met if it is possible to resolve the reflections from the front and back faces of the adhesive layer. If this is the case, it should also be possible to measure the transit time of an ultrasonic pulse through the adhesive layer in the time domain. Knowing the transit time and the bulk wave velocity (from the reflection coefficient measurement), it should then be possible to calculate the thickness of the bondline.

The validity of the assumption that the density of the adhesive is little affected by cure has been checked by manufacturing a set of joints which were cured for different times, the adherends being coated with release agent prior to bonding. After being removed from the curing oven, the adhesive layer was separated from the adherends and its density was determined by mass and dimension measurements. The results are shown in Table I, the density measurements being accurate to about $\pm 50\ \mathrm{kg/m^3}$. The results indicate that after gelling has occurred, the density of the adhesive remained constant to within the measurement accuracy so the assumption of a value of density will not cause significant errors in the determination of the adhesive modulus and thickness. This is as expected since adhesives are formulated to avoid the problems associated with shrinkage after gelling.

2. NORMAL INCIDENCE REFLECTION COEFFICIENT TECHNIQUE–THEORY

The reflection coefficient between two ideally-elastic media (medium 1 and medium 2) where displacement and stress are continuous across the boundary is given by,[8]

$$R_{12}=\frac{Z_2-Z_1}{Z_2+Z_1}=-R_{21} \qquad (1)$$

where Z_i is the acoustic impedance of medium i and is given by the product of the density, ρ_i, and the speed of sound, c_i,

$$Z_i=\rho_i c_i \qquad (2)$$

The transmission coefficient across the boundary is given by,

$$T_{12}=1+R_{12} \qquad (3)$$

Consider the model shown in Figure 2, in which a layer (medium "a") is bounded by two semi-infinite media. An ultrasonic pulse is incident from the upper semi-infinite medium (medium "c"). The pulse length is short, and thus the layer may

TABLE I
Density measurements on adhesive samples cured for different times
(adhesive system: MY 750 (100.0 parts by weight); HY 905 (100.5); DY 063 (2.0)).
All are products of Ciba-Geigy

Cure time [hrs]	Measured density [kg/m^3]	Experimental error [kg/m^3]
2.5	1190	±62
3.0	1227	±44
3.5	1182	±32

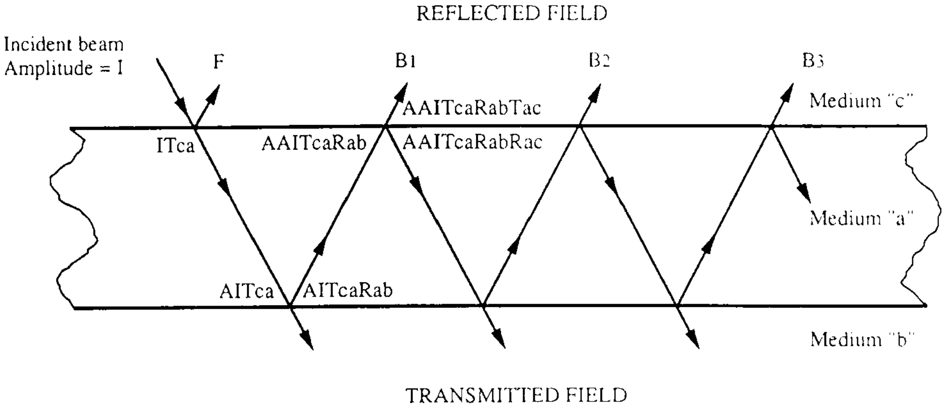

FIGURE 2 Schematic diagram of ultrasonic rays. Normal incidence testing is being considered but the rays are drawn non-normal for clarity.

be considered as a "semi-infinite" medium for the purposes of the reflections at each boundary. Using the standard notation for reflection and transmission coefficients,[8] the amplitude of each reflected and transmitted pulse is expressed in terms of the magnitude of the incident pulse, I, and the attenuation factor, A, which accounts for all losses within medium "a"; the amplitudes of the different rays at selected points along their propagation paths are shown in Figure 2. Let F represent the magnitude of the front face reflection and B_n the magnitude of the nth back face reflection. Then, substituting for the transmission coefficients in terms of the relevant reflection coefficients, X, the ratio of the first back face reflection to the front face reflection is given by,

$$X = B_1/F = A^2 \frac{R_{ab}}{R_{ac}} (R_{ac}^2 - 1) \tag{4}$$

Similarly, Y, the ratio of the second back face reflection to the first is given by,

$$Y = B_2/B_1 = A^2 R_{ab} R_{ac} \tag{5}$$

Consider now the special case where the layer (medium "a") is an aluminium plate (subscript Al) immersed in water and media "b" and "c" are water (subscript W). The parameters X and Y now become

$$X_1 = A^2 (R_{AlW}^2 - 1) \tag{6}$$

$$Y_1 = A^2 R_{AlW}^2 \tag{7}$$

Consider also the case in which the aluminium plate is backed by a semi-infinite layer of epoxy resin (*i.e.* medium "b" is epoxy, given the subscript E). The parameters X and Y now become

$$X_2 = A^2 \frac{R_{AlE}}{R_{AlW}} (R_{AlW}^2 - 1) \tag{8}$$

$$Y_2 = A^2 R_{AlW} R_{AlE} \tag{9}$$

These special cases represent the practical situations of a fully-immersed single adherend and a bonded adherend with water coupling, respectively. These are the cases that are likely to be encountered when attempting to characterise adhesive bonds using ultrasound. These results suggest a possible testing procedure:

(a) Over the water-backed adherend, measure the amplitudes of F, B_1 and B_2. Hence obtain X_1 and Y_1 and calculate R_{AlW} and A from eqns (6) and (7).
(b) Over the bonded zone, measure F and B_1, calculate X_2 and hence deduce R_{AlE} from eqn (8).
(c) Assume, or measure, values for the material properties of aluminium (bulk wave velocity, c_{Al}, and density, ρ_{Al}) and evaluate the acoustic impedance of the adhesive from eqn (1).
(d) Assume a value for the density of the adhesive (ρ_E) and calculate c_E (velocity in epoxy layer) from eqn (2).

For typical aerospace geometries, the measurement over the bonded zone will involve the use of a high-frequency probe in order to resolve the reflections from the bondline. Since high-frequency probes do not need large diameters to minimise beam spreading,[6] this will result in the measurement being taken over a relatively small area. This implies that the robustness of the technique with regard to changes in the cohesive properties over the bonded area is better than that of techniques in which low-frequency transducers are required. Since pulse separation is necessary to evaluate the reflection coefficient, the concurrent measurement of the transit time of an ultrasonic pulse through the bondline should also be possible. The thickness of the bondline, d, may be calculated from the bondline transit time, T, and bulk wave velocity determined from the reflection coefficient measurement, *i.e.*

$$d = T\,c_E \tag{10}$$

The reflection coefficient measurement is attractive because it can be made using the same transducer (and hence at the same time) as the transit time measurement, and because it is related very simply to the cohesive properties. The elastic properties of the adherends do not dominate the reflection coefficient measurement, nor do they affect the determination of pulse transit time. However, the disadvantage of such a technique is that quantitative measurements of amplitudes must be made, which are generally more difficult to achieve than are measurements of frequency or time.

3. EXPERIMENTAL EQUIPMENT AND PROCEDURE

The equipment used to evaluate the technique proposed above is shown schematically in Figure 3. Using this equipment, standard C-scanning of the bonded joint is possible to check for gross defects (voids, disbonds and porosity) in addition to the cohesive property scanning.

The bonded sample under test is placed in a standard ultrasonic immersion tank with the capacity to scan in the plane of the joint (the x-y plane). The microcomputer controls the scan protocol through an external stepper motor driver. An ultrasonic

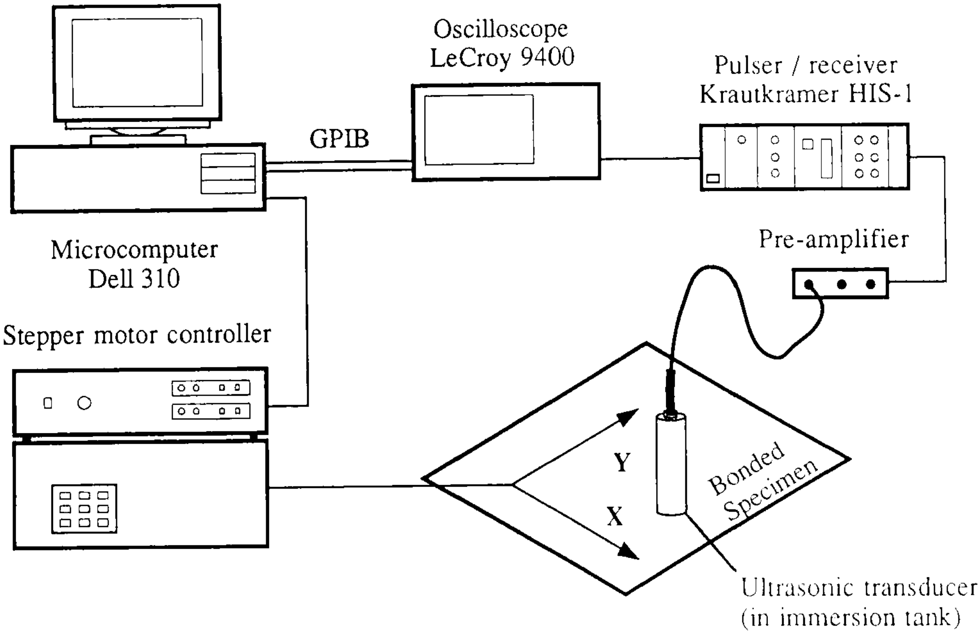

FIGURE 3 Equipment used for scans.

transducer is aligned normal to the joint over the area to be scanned. Resolution of the reflections from the bondline is essential for the determination of the reflection coefficient between the adherend and adhesive, so a highly-damped, high-frequency transducer is used to give as short a pulse as possible. For the joint geometries considered, which were based on a typical aerospace joint with 1.6 mm aluminium adherends and a 0.1 mm thick bondline, a broadband probe with a centre frequency of 34 MHz was found to be satisfactory. The transducer operates in pulse-echo mode and is excited by a low-energy, fast-rise-time voltage pulse from a pulser/ receiver unit. The pre-amplifier is housed in a single unit close to the transducer to minimise the loss of high frequency signals in the electrical cabling. The received signal is further amplified in the main pulser/receiver unit before being passed to the oscilloscope (Le Croy type 9400 which has 8-bit A/D converters) for display and digitisation. The oscilloscope is controlled remotely by the microcomputer over the general purpose interface bus (GPIB), over which the digitised signal is passed for processing. The oscilloscope is also used to average the ultrasonic signal to enhance the signal-to-noise ratio.

Figure 4a shows the time-domain signal from an unbonded aluminium plate using the equipment shown in Figure 3. The signal consists of a reflection from the front face of the plate (F) and a family of back face reflections (B_1, B_2, etc.) as the pulse reverberates within the plate. The reflection coefficient between the plate and the surrounding couplant (water) can be calculated from the magnitudes of F, B_1 and B_2 as outlined above. The time domain signal from a bonded aluminium plate is shown in Figure 4b. The general form is similar to that of the unbonded plate, with the addition of a family of reverberant reflections from the bondline. The magni-

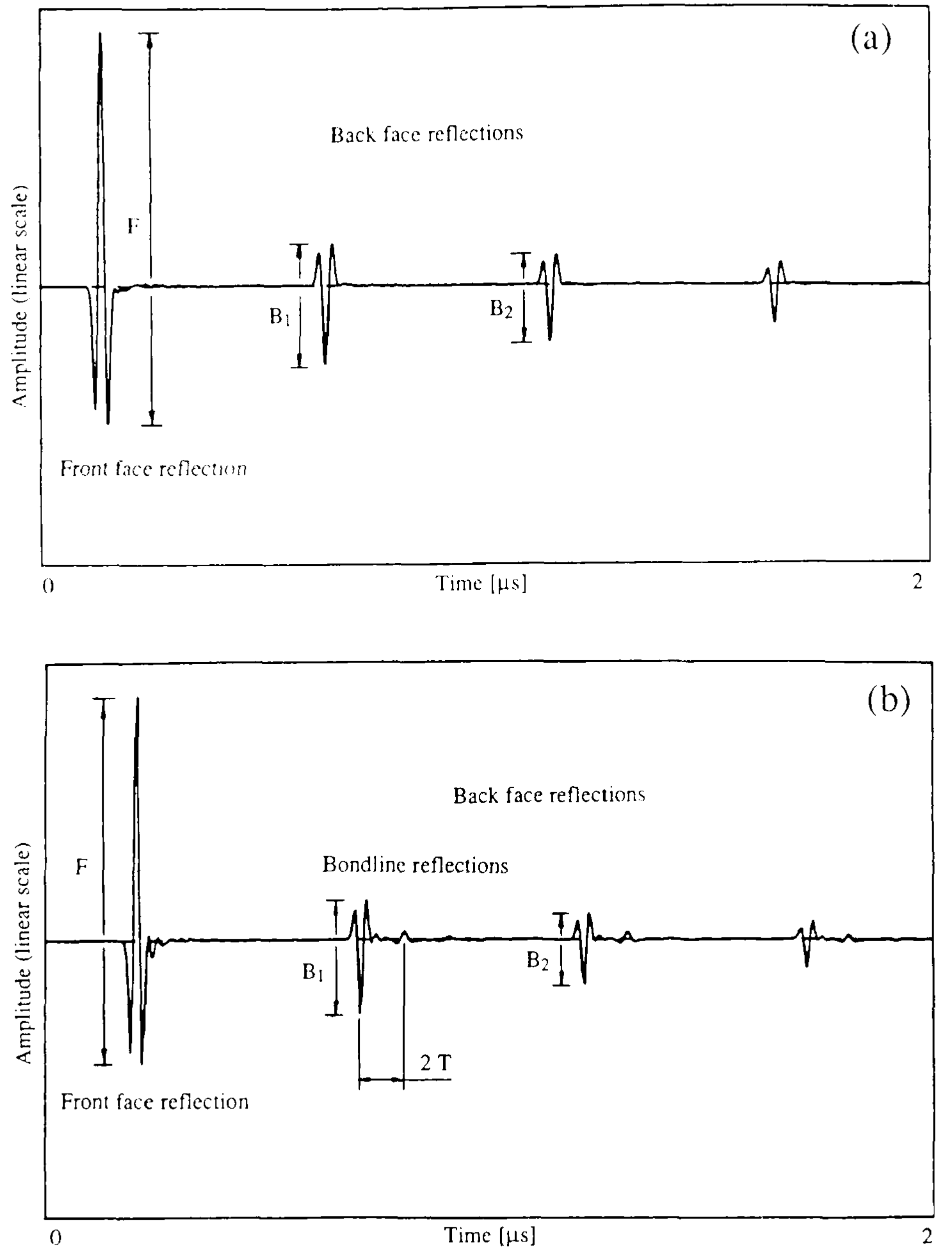

FIGURE 4 Recorded time histories. (a) Unbonded plate; (b) bonded plate.

tudes of the reflections F and B_1 are needed to calculate the reflection coefficient from the aluminium/adhesive interface, and the bondline transit time, T, is needed to calculate the bondline thickness. As the bondline thickness is reduced, the reflections from the top and bottom of the adhesive layer will overlap and it will not be possible to obtain accurate measurements of the amplitude of B_1. (Transit time measurements will still be possible using the frequency-domain processing discussed below.) The lower limit of bondline thickness at which satisfactory measurements could be made using the 34 MHz probe employed in this investigation was between 0.05 and 0.1 mm.

4. SIGNAL PROCESSING CONSIDERATIONS

The time history from a bonded specimen shown in Figure 4b is typical of those obtained from the aerospace-type geometries used in this investigation. There are two sets of measurements to be performed on this data to implement the testing procedure discussed above: reflection magnitude determination and transit time determination. Both of these measurements are discussed below.

4.1. Reflection Magnitude Measurement

The calculation of the reflection coefficients, and hence the cohesive properties in this testing method, is dependent upon the measurement of the magnitudes of the echoes reflected from the bonded sample. Hence, to ensure accuracy in the calculated cohesive properties, it is necessary to ensure good accuracy in the measurement of these reflections.

In the initial tests, the magnitudes of the reflected pulses were determined in the time domain using digitised data. The signal was averaged at least twenty times at each measurement location to give an adequate signal-to-noise level to allow any dc component to be identified and removed from the signal. The magnitude of each reflection was then taken to be the maximum (for the front face reflection) or the minimum (for the back face reflections) in a time window positioned over the relevant reflection, rather than the peak-to-peak value shown in Figure 4. This measurement procedure was chosen to simplify the processing. The phase change between the front and back face reflections (see Fig. 4a and 4b) is due to the fact that the front adherend (a high-acoustic-impedance medium) is sandwiched between two lower-impedance media (water and epoxy resin). Measuring the reflection magnitudes in this way was found to introduce scatter into the measurements because of the digitisation of the signal. If this measurement were to be performed using analogue equipment this problem would not arise, but no such equipment with the necessary bandwidth was available.

There are two different errors introduced into the reflected pulse amplitude measurement in the time domain caused by the digitisation of the signal. These are caused by the "sampling error" (*i.e.* the amplitude difference between the true maximum in a signal and that of the digitised point measured closest to it), and the "digitisation error" (determined by the analogue-to-digital converter used). Analysis of these errors for the experimental equipment shown in Figure 3 suggests that a scatter of around ±5% in the measured reflection coefficient values is to be expected if the measurements are carried out in the time domain. Therefore, in this application, a spatial average was carried out over the scan area to give a single value for the calculated velocity in the adhesive layer, which averaged out the effects of these errors. A further error in time domain measurements is introduced if the adherend material introduces dispersion, causing the wavelets F and B_1 in Figure 4b to have different shapes. However, this is not a significant problem with aluminium adherends.

In order to improve the measurement accuracy of the bondline reflection coefficient, in later experiments the necessary reflection ratios were calculated in the frequency domain, as illustrated in Figure 5. Each interfacial reflection was edited

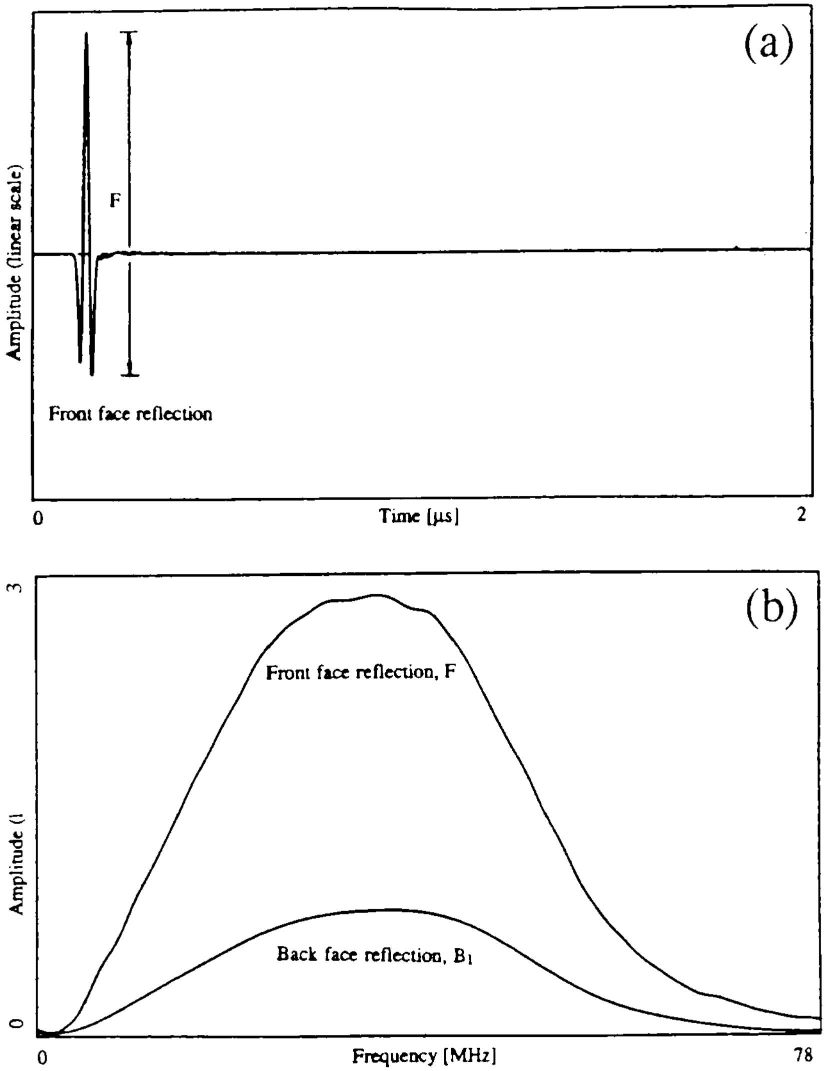

FIGURE 5 Frequency domain measurement of reflection coefficient. (a) Front face reflection, F, edited from signal shown in Fig. 4a; (b) frequency spectra of reflections F and B_1 in Fig. 4a; (c) division of spectrum of reflection B_1 by that of reflection F.

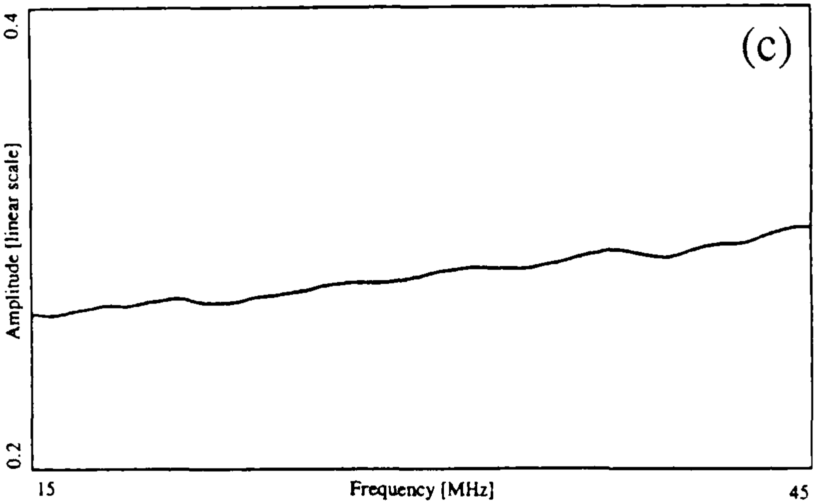

FIGURE 5 (Continued).

from the time domain signals of Figure 4 by setting the values of the points sampled before and after that reflection to zero (for example, Fig. 5a shows the front face echo, F, from Fig. 4a), and processed to the frequency domain using a fast Fourier transform (FFT). The amplitude spectrum obtained in each case represents the magnitude of each frequency component in the reflection. Figure 5b shows the result of processing the echoes labelled F and B_1 in Figure 4a in this way. The spectrum is built using all the data in the edited time domain signal, and hence all the data points contribute to defining the maximum absolute level. This is in contrast to the time domain measurement of maxima and minima, where the single point with the largest absolute amplitude defines the maximum (or minimum) value. Once the frequency spectra have been obtained, the required reflection ratios can be determined by dividing the necessary spectra in the frequency domain (Fig. 5c).

The result of the spectral division shown in Figure 5c appears to show dispersion (*i.e.* frequency dependence of the elastic constants of the materials), the ratio B_1/F appearing to increase as a function of frequency. However, neither water nor aluminium (the two materials forming the interface) are dispersive, so a frequency-dependent reflection coefficient would not be expected for these materials. The reason for the apparent increase in the ratio B_1/F is that the frequency content of the pulse reflected from the back face of the aluminium plate is higher than in that from the front face, due to "focussing" of the transducer. (Although a nominally unfocussed transducer was used, it was necessary to use it with a "stand-off" from the top of the adherend of only 3–4 mm in order to reduce the attenuation of the high-frequency components of the signal in the water coupling. The transducer was, therefore, operating in its near field where significant amplitude variations with the distance between a reflector and the transducer are observed.)[8] It was, therefore, necessary to maintain a constant distance between the transducer and the specimen, and to calculate the reflection coefficient using measured data from the same frequency range over the unbonded and bonded zones of the specimen. The frequency range chosen was that in which the signal-to-noise ratio was known to be maximum (*i.e.* between 30 MHz and 40 MHz).

Transforming to the frequency domain incurs a penalty in terms of the computation time required, but it brings benefits in the form of decreasing the scatter in the data for a given sampling rate. The effects of these signal processing improvements are discussed below with the relevant results.

4.2. Bondline Transit Time Measurement

Since the pulses reflected from the bondline have to be resolvable to enable the reflection coefficient from the adhesive/adherend interface to be evaluated, the transit time of the pulse through the bondline could easily be measured in the time domain using suitable analogue equipment. However, as mentioned above, no such equipment was available, so the transit time was measured after digitisation. One possibility was to measure the time between the minimum in the reflection from the front of the bondline (B_1), and the maximum in the first reflection from the back of the bondline. It was envisaged that this could have been achieved to the accuracy defined by the sampling rate (*i.e.* the time between digitised points).

Rather than searching through the time domain data to find where these maxima and minima occurred, it was decided to edit the bondline reflections from the rest of the time domain signal and process this to the frequency domain by FFT. This process is shown in Figure 6. The time domain signal shown in Figure 6a shows the bondline reflections edited from the time history shown in Figure 4b by setting the values of the points sampled before and after the bondline reflections to zero. Figure 6b shows the amplitude spectrum of the time domain signal shown in Figure 6a. The minima in the spectrum correspond to the through-thickness resonances of the adhesive layer.[9] By doing this, the resonance frequencies of the bondline (the frequencies at which integer numbers of half-wavelengths could fit into the bondline thickness) could be measured, and hence the transit time could be evaluated. The values of transit time, T, corresponding to each minimum decrease slightly with increasing frequency. This is in line with expectations since the adhesive is slightly dispersive and its longitudinal wave velocity increases with frequency. Processing in the frequency domain made measuring the transit time more accurate and easier to perform (the well-defined minima in the spectrum were easy to identify) at the cost of slightly increasing the computational effort. The advantages significantly outweighed the disadvantages in this case, and measurement of the transit time was always carried out in the frequency domain.

5. EXPERIMENTAL INVESTIGATION

5.1. Testing Strategy and Procedure

The technique was applied to a series of bonded samples which were designed to assess the accuracy and robustness of the technique on joints with similar properties to those in use in the aerospace industry. The adhesive system used in each of these joints is shown in Table II, hot curing being used in each case.

This technique differs significantly from those in which the dynamic properties of

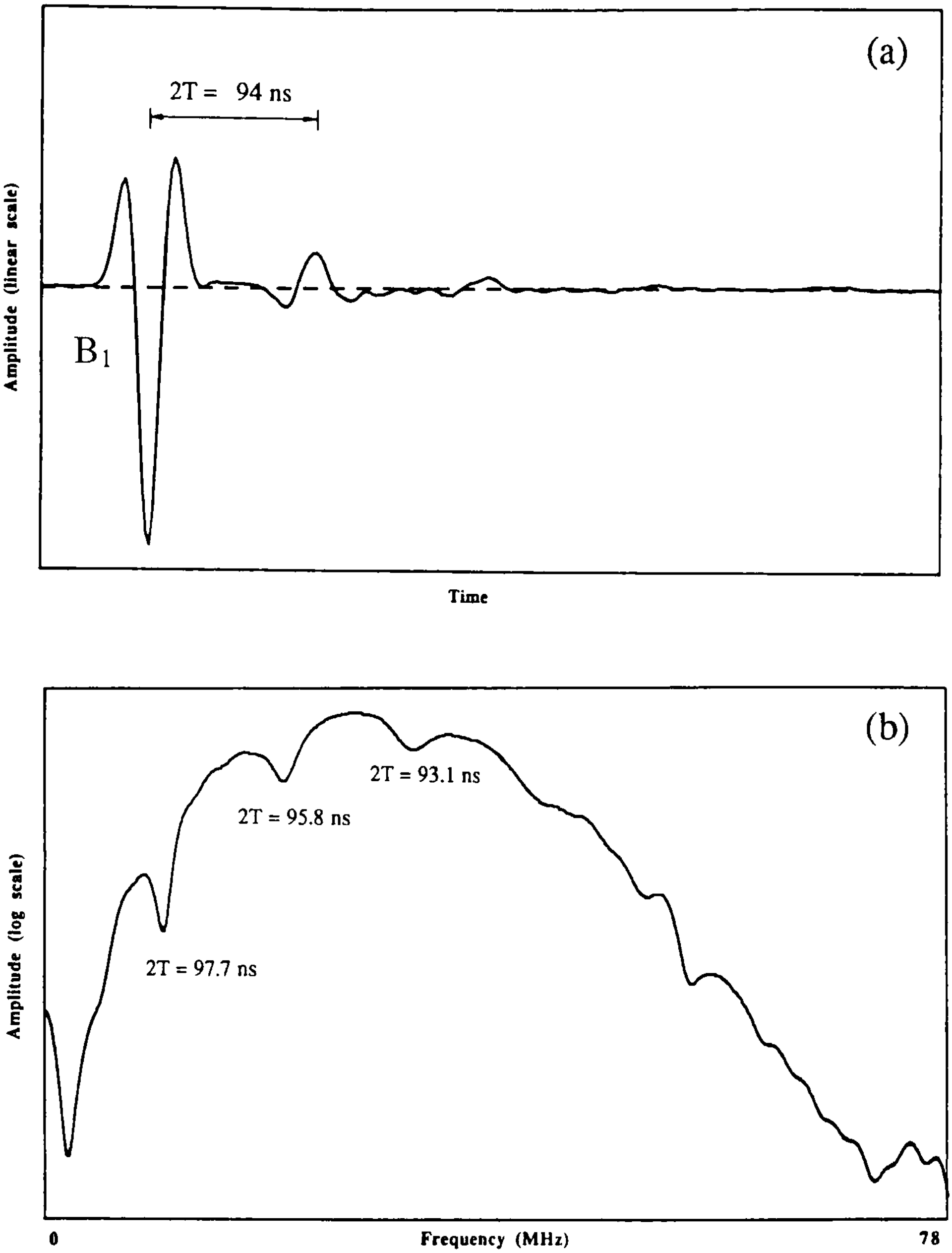

FIGURE 6 Frequency domain measurement of bondline transit time. (a) Bondline reflections edited from time history shown in Fig. 4b; (b) frequency spectrum of (a).

TABLE II
Adhesive composition for the joints used to test the reflection coefficient/transit time scanning technique for cohesive property determination. All materials are products of Ciba-Geigy

Joint	Resin [parts by mass]	Hardener [parts by mass]	Accelerator [parts by mass]	Flexibiliser [parts by mass]
DA1	MY 750 [100]	HY 905 [104.6]	DY 063 [1.4]	—
DA2				
EA1	MY 750 [100]	HY 905 [100.55]	DY 063 [2.0]	—
EA2				
EA3				
EA4				
HA1	MY 750 [100]	HY 905 [99.8]	DY 063 [3.1]	—
HA2				DY 040 [13]
HA3				DY 040 [32]
HA4				DY 040 [49]
JA1	MY 750 [100]	HY 905 [99.8]	DY 063 [4.4]	—
JA2				DY 040 [53]
JA3				DY 040 [60]
JA4				DY 040 [71]

the joint are measured[4-6] because the reflection coefficient is not dominated by the properties of the adherends. Therefore, the main objective of the experimental investigation into this technique was to determine the accuracy to which the bulk wave velocity of the adhesive layer in a bonded joint could be determined by the reflection coefficient method, since for any given joint geometry and adhesive system the bulk wave velocity will give an indication of the cohesive quality. Alers *et al.*[10] determined the failure load of a series of bonded joints as a function of the longitudinal bulk wave velocity in the adhesive. They found that for their specimens, there was a linear correlation between the failure load and the acoustic velocity, an increase of 200 m/s from its initial value of around 2500 m/s in this latter quantity corresponding to an increase in the ultimate strength of the joint of about 30%. Although such results are highly material and geometry dependent, it is likely that an increase in the longitudinal bulk wave velocity in the adhesive of 200 m/s would be significant in any bonded system. The objective of this technique must, therefore, be to determine the bulk wave velocity to this accuracy or better. The effect of miscuring joints is to reduce the bulk wave velocity relative to the fully cured joint,[2,3] so miscuring of joints was simulated by varying the bulk wave velocity with chemical additives.

The testing procedure applied to each bonded joint was as follows. Firstly, the transducer was aligned so that the ultrasonic beam was normal to the front face of an unbonded area of the sample; this was achieved by adjusting the angle of the transducer until the amplitude of the reflection from the front face of the sample was a maximum. A scan was performed over a small area of the unbonded zone, in which the magnitudes of the reflections F, B_1 and B_2 (see Fig. 4a) were recorded. At the end of this scan, the reflection coefficient between the adherend and the surrounding water and the attenuation factor in the adherend could be calculated, the results being averaged over the points tested. Having characterised the adherend, the transducer was moved over the bonded zone and normalised to the front face of the upper adherend. Again, a scan area was defined, and at each point the magnitudes of the reflections F and B_1 (see Fig. 4b) and the bondline transit time were evaluated and written to file. At the end of this scan, the aluminium data were used together with the measured values to produce maps of the longitudinal bulk wave velocity in the adhesive and of the bondline thickness.

5.2. Sample Description and Objectives

The basic joint geometry employed is shown in Figure 7. The adhesive system used was varied to alter the elastic properties of the bondline, as was the thickness of the steel spacer wire to vary the bondline thickness.

The accuracy and usefulness of this technique for the determination of the cohesive properties was evaluated on a range of aluminium-epoxy-aluminium samples with differing adhesive properties (see Table II). The results of this investigation are shown in Table III. The adherends used were of aerospace grade aluminium alloy, with either 1.6 mm or 3.2 mm nominal thickness. The surfaces of the aluminium adherends were lightly buffed to remove surface scratches and hence provide as uniform a testing condition as possible for each specimen. The effects of other surface preparation techniques are discussed later. The results from the reflection coefficient and transit time measurements were compared with measurements on bulk samples prepared at the same time. To ensure as much compatibility between bonded and bulk samples as possible, the bulk samples were prepared as bonded joints in which the adherends had been coated with a release agent. After a common cure cycle, the bulk samples could be prepared by simply peeling away the adherends.

The measurement error in the longitudinal bulk wave velocity from bulk samples was governed by the accuracy to which the thickness of the bulk sample could be measured. Since the bulk samples were thin (to maximise the similarity in properties between the bulk samples and the adhesive layer in the bonded joints), this measurement error was comparatively large. Those joints for which the measurement error in the bulk specimen measurement is particularly large (HA4 and JA1, for example), had corresponding bulk specimens in which there was a thickness variation in the measurement zone. The nominal density for each specimen was also determined from the bulk samples, which were measured using a micrometer to calculate the volume and then weighed to enable the density to be calculated. The

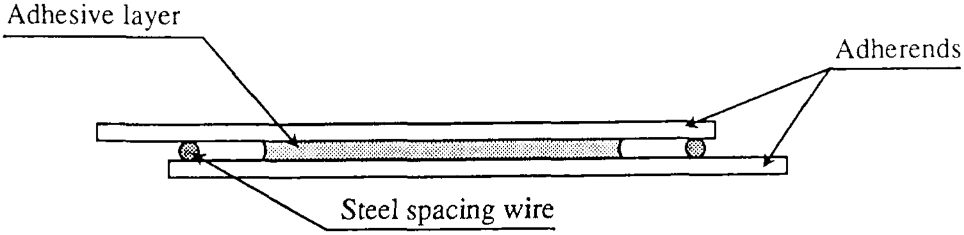

FIGURE 7 Adhesive joint specimen used in this investigation.

TABLE III
Results of longitudinal velocity scans on bonded aluminium-epoxy-aluminium joints, using reflection coefficient and transit time measurements

Joint	d_{adr} [mm]	Scan size [mm]	Scan resol^n [points]	d_{nom} [mm]	d from scan [mm]	$c_{l_{nom}}$ from bulk sample (error [%]) [m/s]	c_l from scan [m/s]	σ [m/s]	$\frac{c_l - c_{l_{nom}}}{c_{l_{nom}}}$ [%]
DA1	3.12	20×20	30×30	0.21	0.20	2607 (±2.0)	2595	114	−0.5
DA2	3.12	20×20	30×30	0.21	0.20	2607 (±2.0)	2406	143	−7.7
EA1	3.12	20×20	30×30	0.26	0.27	2566 (±2.1)	2726	103	+6.2
EA2	3.12	20×20	30×30	0.35	0.35	2537 (±1.1)	2407	132	−5.1
EA3	3.12	20×20	30×30	0.48	0.49	2538 (±1.3)	2700	88	+6.4
EA4	3.12	20×20	30×30	0.60	0.59	2529 (±0.9)	2445	206	−3.3
HA1	1.60	10×10	30×30	0.23	0.23	2528 (±1.0)	2526	107	−0.1
HA2	1.60	5×5	30×30	0.22	0.21	2577 (±0.9)	2529	161	−1.8
HA3	1.60	10×10	30×30	0.23	0.22	2669 (±1.3)	2491	105	−6.7
HA4	1.60	10×10	30×30	0.23	0.23	2609 (±2.6)	2585	163	−0.9
JA1	1.60	10×10	30×30	0.20	0.19	2423 (±2.6)	2443	79	+0.8
JA2	1.60	10×10	30×30	0.30	0.29	2538 (±1.6)	2507	70	−1.2
JA3	1.60	10×10	30×30	0.24	0.23	2488 (±2.0)	2379	99	−4.4
JA4	1.60	10×10	30×30	0.20	0.20	2350 (±2.4)	2270	68	−3.4

measurement of mass was accurate to 5 significant figures (an electronic balance was used) and, therefore, had a negligible effect on the experimental accuracy of the density measurement. However, the volume estimate contains a possible experimental error of ±5%, due again to the low thicknesses of the bulk samples used. In attempting to measure the nominal properties of the adhesive in a bonded joint from bulk samples, there is clearly a problem to be addressed in that using thin samples (to mirror as closely as possible the properties of the adhesive in the joint) introduces measurement errors.

The purpose of the joints in Series "D" was to check whether the procedure for manufacturing bulk samples in parallel with joints described above, which had previously been developed with cold-curing adhesives,[11] could be employed with hot-curing systems. It was found that the procedure worked well and it was employed in all the subsequent tests.

The joints prepared in Series "E" were designed to establish whether the thickness of the bondline in hot-cured adhesive systems has an effect on the stiffness (bulk wave velocity) in the adhesive layer. The results of measuring the thickness and longitudinal wave velocity of each of the bulk coupons are shown in Table III. Clearly, there is no dependence of bulk wave velocity on bondline thickness for these joints, which were all cured to the manufacturers recommended cycle. Measuring the bulk wave velocity using the reflection coefficient technique confirms that there is no thickness dependence for this hot-cured system.

The joints in Series "D" and Series "E" were manufactured with 3.12 mm thick adherends to provide easy separation of the bondline echoes from the reverberations in the adherends. However, having established the success of this technique for the determination of the cohesive properties of the joints, attention was switched to the more challenging case of adherends with 1.6 mm thick aluminium adherends where the reflections from the bondline would be less well separated from the reverberations in the top adherend; in practice, it was found that the reverberations in the adherend posed no difficulties with either adherend thickness.

In joint Series "H" and "J," the effects of adding a known quantity of a flexibilising agent to the adhesive resin prior to curing was investigated. The addition of the flexibiliser was designed to reduce the stiffness of the adhesive (and therefore the bulk wave velocity), and hence simulate miscuring of the adhesive system. The proportions of flexibiliser added to the base systems of Series "H" and "J" are shown in Table II in terms of the mass added per 100 parts by mass of resin. The apparent effect of adding the flexibiliser is to increase the longitudinal bulk wave velocity of the adhesive up to an addition of approximately 50 parts by mass. Thereafter, the stiffness of the adhesive decreases markedly. The trend shown by measurements on the bulk specimens is also followed by the bonded specimens (Table III). The reasons for this behaviour have not been investigated.

5.3. Results of Scans

Table III shows the results obtained by scanning fourteen aluminium-epoxy-aluminium bonded joints using the reflection coefficient/transit time technique. The thickness of the aluminium adherends used for each joint, d_{adr}, is shown in the second column. The scan zone used for each sample is specified in terms of the physical size and the number of measurement points used. The nominal bondline thickness, d_{nom}, obtained by micrometer measurements, and the adhesive bulk wave velocity, $c_{l_{nom}}$, obtained from the corresponding bulk specimen, are shown for each joint. Also shown are the corresponding results obtained from the scanning technique, d and c_l, respectively, the values given being spatial averages over the scan area. In addition, the standard deviation in the measurements of the bulk wave velocity, σ, is shown as an indication of the amount of scatter in the data. In all cases, the average bulk wave velocity of the adhesive layer was determined to within $\pm 8\%$ of the nominal value and the thickness was evaluated to within micrometer accuracy.

All the bulk wave velocity results from the scans shown in Table III, with the exception of those of the final four joints (Series "J"), were obtained by time domain measurement of the received reflection amplitudes. The Series "J" results were obtained by transforming the reflected pulses to the frequency domain and performing a spectral division. Processing the results in the frequency domain considerably reduces the standard deviation of the measurements obtained during the scan of a given sample, confirming that the frequency-domain processing procedure could produce better results than the previously implemented time-domain technique. Consequently, this frequency domain processing technique was applied to all subsequent measurements.

5.4. Joints Bonded with Film Adhesive

Four joints were fabricated from 3.2 mm thick aluminium alloy adherends and a one-part, hot-curing "film" adhesive (American Cyanamid type FM73), in which the adhesive is carried in a polymer mesh. These joints (Series "FLM") were tested using the reflection coefficient and transit time scanning technique discussed above (with frequency domain processing) to evaluate the thickness and bulk wave velocity of the adhesive layer. The results of these tests are shown in Table IV, together with the nominal values of thickness and velocity, determined by micrometer measurement and bulk sample velocity determination, respectively. The nominal thickness variations between the samples were caused by inaccurately aligning the planes of the adherends during the manufacturing process, allowing the adhesive to flow during the initial stages of cure. This was done inadvertently, but permitted the testing of different thicknesses of this adhesive, which is not usually possible using a single layer of adhesive film.

The results again show that the thickness of the adhesive layer was determined to within micrometer accuracy, and the bulk wave velocity to within ±5.5% of the nominal. These are important results, because film adhesives are common in aerospace applications of bonding and the use of the technique would have been severely limited if it had been unable to determine the cohesive properties of joints bonded with such an adhesive. The fibres in the mesh carrier are thin relative to the wavelength of the ultrasound and, therefore, do not have a significant effect on its propagation.

5.5. Adherends with Different Surface Preparations

Kinloch[12] describes some of the surface treatments applied to aluminium adherends prior to bonding. The purpose of these preparations is to reduce the effects of environmental attack at the adhesive/adherend interface. The main effect of the most common surface treatments for aerospace applications of bonding involving aluminium is to modify the structure of the oxide layer that separates the metal from the polymer. It is essential that any technique used to monitor the cohesive properties of the adhesive layer can be used on joints whose adherends have been treated with the different standard surface preparations. A major concern with the reflection coefficient measurement technique is that different surface roughnesses may change the reflection coefficient or make it difficult to measure. Aluminium adherends were, therefore, prepared using a variety of procedures and the reflection coefficient between the adherend surface and water was measured in each case. This measurement is part of the procedure developed for cohesive property determination described above and if this could be performed satisfactorily, there

TABLE IV
Application of reflection coefficient/transit time scanning procedure to joints bonded with film adhesive American Cyanamid type FM73

	Bondline thickness		Longitudinal wave velocity		
Specimen	Nominal [mm]	Measured [mm]	Nominal [m/s]	Measured [m/s]	Error [%]
FLM1	.22	.22	2500	2588	+3.5
FLM2	.15	.16	2500	2375	−5.0
FLM3	.24	.22	2500	2364	−5.4
FLM4	.21	.20	2500	2538	+1.5

was unlikely to be any difficulty with the measurement of the reflection from the aluminium/epoxy interface.

Four surface preparations were considered in this investigation: the "as supplied" condition; polishing the adherend surfaces to produce a "buffed" finish; chromic acid anodisation (CAA); and grit blasting the adherend surface. The specimens were immersed and the reflection coefficient between the adherend and the surrounding water (R_{AlW}) was measured using the procedure described earlier. The size of the area scanned and the resolution (number of measurement points in the scan area) are shown in Table V for each of the specimens. For each sample, the average reflection coefficient over the scan area, and the standard deviation were determined, the results being shown in Table V.

Very little difference was found between the 1.60 mm thick aluminium samples in the "as supplied" and "buffed" conditions. This is because the magnitude of the surface defects removed by buffing were significantly smaller than the wavelength of the ultrasound used (a 34 MHz centre-frequency probe was used, with an approximate wavelength in aluminium of 200 μm). The specimen selected to represent the "as supplied" condition was chosen on the basis that it was not marked by any deep scratches. The grit-blasted specimen had micro pits of the order of 1 μm deep uniformly distributed over its surface. The pits were, therefore, much smaller than the ultrasonic wavelength though larger than the surface features on the "as supplied" and "buffed" specimens. The results in Table V show that this micro pitting slightly reduced the measured reflection coefficient from the adherend/water interface. It is likely that this reduction in reflection coefficient was caused by the surface irregularities scattering the ultrasound.

The small change in the reflection coefficient caused by grit blasting will not significantly affect the operation of the technique for cohesive property determination because the reflection from the aluminium/epoxy interface is compared with that from an aluminium/water interface. Hence, provided the unbonded area tested has had the same surface preparation as the bonded area, the effects will tend to cancel.

The thick (5.00 mm) aluminium plate used in the lower half of Table V has a measured reflection coefficient 2% higher than that of the thin plate used in the upper half. This cannot be explained in terms of the elastic properties of the ma-

TABLE V
Results of reflection coefficient measurement on aluminium adherends with various surface preparations

Specimen thickness [mm]	Surface preparation	Scan size [mm]	Scan resolution [points]	Reflection coefficient [%]	Standard dev^n in reflection coefficient [%]
1.60	As supplied	10×10	30×30	83.96	0.32
	Buffed	10×10	30×30	83.95	0.25
	Grit blasted	5×5	30×30	82.56	0.13
5.00	Buffed	5×5	15×15	86.07	0.34
	CAA	5×5	15×15	86.28	0.38
	CAA/bonded	5×5	15×15	70.82	1.04

terials, the longitudinal bulk wave velocity in the thicker sample (6323 m/s) being virtually the same as that in the thinner sample (6315 m/s), and the densities (2790 kg/m^3 and 2760 kg/m^3, respectively) differing by only 1%. The reason for this discrepancy is that the transducer is operating in its near field and, consequently, the plate is in a very complicated pressure field. However, this does not render the proposed technique inoperable, because the reflection coefficient from the bonded zone is measured relative to that from the water-backed zone, and these results will be consistent provided the transducer is maintained at a constant distance from the top surface during the tests.

The water-backed, chromic acid anodised (CAA) specimen has a similar reflection coefficient to the buffed specimen. The CAA process removes the very thin layer of oxide from the aluminium surface and replaces it with a uniform layer approximately 3.5 μm thick,[12] but the thickness of this layer is so low that it does not significantly influence the reflection coefficient at the frequencies used here. This is an important result, because the CAA process is a standard aerospace surface preparation for bonded aluminium joints. To confirm that the CAA process does not hinder the application of the proposed technique, a layer of epoxy resin (0.45 mm thick) was cast onto the sample used to measure the aluminium/water reflection coefficient with CAA treatment. The reflection coefficient/transit time scanning procedure was then performed to evaluate the cohesive properties of the adhesive material. The reflection coefficient (shown in Table V) was reduced (as would be expected, because epoxy has a higher acoustic impedance than water) and the standard deviation in the data increased slightly. A bulk sample of the epoxy resin used was also produced and the results from the scan were found to agree with the bulk wave velocity to -0.3% of the nominal value measured on this bulk sample. The thickness of the layer was found to within micrometer accuracy (± 0.01 mm). The application of the reflection coefficient and transit time technique to joints in which the adherends have been CAA treated is, therefore, straightforward.

6. DISCUSSION

From the most reliable results shown in Table III (those obtained by frequency domain processing) and those in Table IV, it would appear that the longitudinal bulk wave velocity could be determined by the reflection coefficient technique to within ±150 m/s (approximately ±6%) of the values determined from bulk samples. The thickness of the bondline was determined to micrometer accuracy in all cases, with thickness variations in the bondline measured to much better than micrometer accuracy. This represents a significant improvement over the method of Guyott and Cawley.[4]

Previous work by Lindrose[2] has shown that the longitudinal bulk wave velocity changes by approximately 800 m/s during the curing cycle, so the indications are that the present technique should be sufficiently sensitive to be able to measure the state of cure of an aluminium-epoxy-aluminium joint. A major advantage of this technique over the previously evaluated dynamic property-based techniques[4-6] is that it has reduced the dominance of the adherends in the cohesive property determination. In addition, the necessary measurements can be made with a single transducer, the diameter of which can be small without introducing the problems associated with beam spreading.

The technique uses the measurement of the reflection coefficient from the top adhesive/adherend interface together with prior measurements on an unbonded area of adherend to obtain the acoustic impedance of the adhesive. The longitudinal wave velocity in the adhesive is then obtained from eqn (2) by assuming a value of the adhesive density. This velocity is then used in conjunction with the measured transit time across the adhesive layer to obtain the adhesive layer thickness. The effects of any inaccuracy in the assumption of a value for the density of the adhesive are easy to predict because, from eqn (2), the calculated velocity is inversely proportional to the assumed density. For the systems studied, the density of the adhesive varied by less than the experimental error in its determination (±5%), even when significant amounts of flexibiliser (up to 75 parts per 100 parts of resin) were added. A more accurate study of the density variations during the curing process might be of interest but, since shrinking of the adhesive after gelling is undesirable because of the residual stresses produced, it is believed that adhesive manufacturers would attempt to minimise density variations during cure by careful formulation.

The reflection coefficient from the adhesive/adherend interface could be affected by changes in the interfacial conditions as well as by changes in the cohesive properties of the adhesive. Indeed many workers (see, for example, References 13–15) have attempted to use reflection coefficient measurements to detect the presence of an unsatisfactory adhesive/adherend interface. However, significant changes are only seen with very severe interfacial degradation[15] and the greatest sensitivity to interfacial properties is obtained at oblique incidence. The measurement of the cohesive properties by normal incidence reflection measurements is, therefore, unlikely to be affected by variations in the adhesive/adherend interface.

7. CONCLUSIONS

An ultrasonic nondestructive technique for the quantitative determination of the cohesive properties of adhesive joints based on the measurement of the reflection coefficient from the top adhesive/adherend interface and the bondline transit time has been developed. The technique has been used to determine the longitudinal bulk wave velocity in aluminium-epoxy-aluminium joints to within ±6% of the nominal values determined from bulk samples. The bondline thickness of the samples tested was evaluated to within micrometer accuracy and thickness variations within the scan area were detected to much better than micrometer accuracy. The effect of a standard aerospace surface treatment for aluminium adherends (chromic acid anodisation, or CAA, to UK defence industry specification) has been investigated and found not to present a significant obstacle to the application of this technique to bonds between such adherends. The standard phosphoric acid anodisation (PAA) surface preparation used in the USA produces a thinner oxide structure than the CAA method[16] and so is very unlikely to cause problems. The technique has been developed on a variety of adhesive systems (hot and cold curing two-part epoxies and one-part hot curing film adhesive containing a "scrim" carrier) and has been found to work satisfactorily on each type.

The reflection coefficient/transit time technique has a major advantage over those methods of cohesive property determination involving measurement of the dynamic properties of the joint; it is far less dominated by the properties of the adherends and is, therefore, applicable to a much wider range of bond geometries. The technique requires access to only one side of the joint and, for joints with typical aerospace geometries, it can be implemented using a single transducer with a centre frequency below 50 MHz. A probe with such a centre frequency would typically contain an active element of approximately 5 mm diameter, and the area under inspection would be approximately this size. Significant variations in the cohesive properties over this distance are unlikely, which contributes to the attractiveness of the technique.

The method, therefore, promises to provide a reliable, non-destructive means of measuring the cohesive properties of a bonded joint and represents a significant advance on the currently available technology.

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