THE NDT OF ADHESIVE JOINTS USING ULTRASONIC SPECTROSCOPY

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ABSTRACT

The use of ultrasonic spectroscopy for the non-destructive determination of the cohesive properties of the adhesive layer in a joint is described. It has been shown that measurements of the through thickness natural frequencies of the joint obtained using ultrasonic spectroscopy can be used to calculate the modulus and thickness of the adhesive layer. The results reported here indicate that the modulus may be determined to an accuracy of ±6%, and the thickness may be found as accurately as it can be measured independently. No other test is available which enables both these parameters to be determined after a joint is made, and it is anticipated that the test will be very valuable in ensuring that the process quality control during the joint manufacture has been satisfactory.

INTRODUCTION

The non-destructive testing of adhesive joints is made difficult by the need to test for three types of defect [1]: the first consists of complete voids, disbonds or porosity in the adhesive layer, the second type of defect is poor adhesion, that is a weak bond between the adhesive layer and adherend, while the third type of defect is poor cohesive strength, or a weak adhesive layer.

Currently, the Fokker Bond Tester Mk II [2], which operates in the frequency range 50-500 kHz, is the only commercially available instrument which claims to measure cohesive strength. The Fokker Bond Tester attempts to measure the specific stiffness of the adhesive, E_S, which is defined as :-

$$E_S = E'_2/l_2 \qquad (1)$$

where E'_2 and l_2 are the apparent modulus [2] and the thickness of the adhesive respectively. The instrument does not measure the cohesive strength of the joint directly, but attempts to correlate the specific stiffness of the joint with the strength. However, it has been shown [2] that the Fokker Bond Tester is not sensitive to changes in the specific stiffness of the adhesive in the range of stiffnesses found in typical high strength joints. Consequently, except when the specific stiffness of the adhesive is very low (less than $1\text{-}2\times10^4$ GN/m^3), it gives a very unreliable measure of cohesive strength. Since most joints commonly used in engineering primary structure have specific stiffnesses of $2\text{-}3\times10^4$ GN/m^3 or greater, there is a need for the development of alternative testing techniques.

The modulus (and therefore ultrasonic velocity in the adhesive) obtained with a particular adhesive, is dependent on the cure cycle and on the correct mixing of hardener and resin, and correlates strongly with cohesive strength [3]. The dependence of joint strength on adhesive thickness is less certain; however, very thin or thick bondlines can cause a reduction in strength [4]. Consequently, joints which have the same specific adhesive stiffness but different adhesive moduli and thicknesses will not necessarily have the same strength. Therefore, a satisfactory non-destructive test for joint strength cannot simply rely on a measurement of the ratio of adhesive modulus to thickness.

Recent work [5,6] has shown that measurements of the through thickness resonant frequencies of a joint obtained using ultrasonic spectroscopy can be used to calculate the thickness and modulus of the adhesive layer in a joint, and hence to provide a check on its cohesive properties. It is not suggested that the technique will give a direct prediction of the cohesive strength of the joint, since this depends on many factors such as the type of loading and the adhesive toughness, in addition to the adhesive modulus and thickness. However, a check on the adhesive modulus and thickness would give a valuable indication of whether the process quality control during the joint manufacture was satisfactory.

The results presented in Reference 6 indicated that the thickness of the adhesive layer could be obtained to an accuracy of ±10% and the modulus to an accuracy of ±20% from the spectroscopy measurements. However, the technique used to calculate the modulus and thickness was relatively crude and the operation required a large volume of data to be stored. It was therefore decided to investigate alternative procedures, and this paper describes the development of an improved method.

CURRENT METHOD

Fig 1 shows the frequencies of the first eleven modes of longitudinal (through thickness) vibration of an adhesive joint with 1.6 mm thick aluminium adherends predicted using the analysis described in Reference 5, as a function of specific adhesive stiffness. The two sets of curves shown in Fig 1 represent typical high modulus (Young's modulus approximately 4.6 GN/m^2 and apparent modulus (E'_2) 7.2 GN/m^2) and low modulus (Young's modulus approximately 0.7 GN/m^2 and apparent modulus (E'_2) 3.6 GN/m^2) adhesives. Fig 1 shows that reducing the modulus at constant adhesive thickness, or increasing the adhesive thickness at constant modulus, both of which reduce the stiffness of the adhesive layer, will cause a decrease in the resonant frequencies of all the modes.

Over the ranges of adhesive modulus and thickness considered here, Fig 1 shows that the first mode is dependent on specific stiffness alone since the two curves representing the different adhesive moduli are coincident. In this instance, increasing adhesive thickness has exactly the same effect as decreasing the modulus by the same percentage. However, in the higher modes the curves for different moduli are not coincident, so provided the resonant frequencies of at least two modes are measured, it should be possible to distinguish a change in adhesive modulus from a change in adhesive thickness.

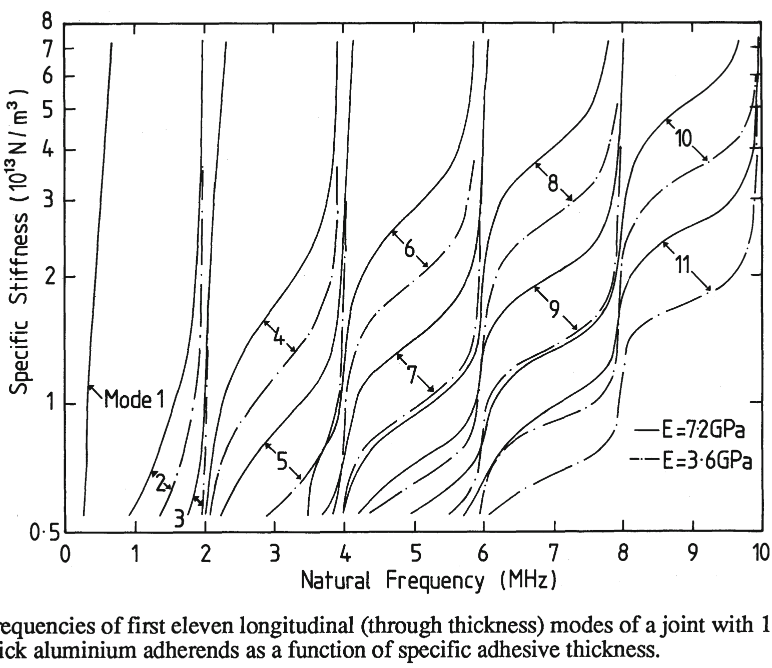

Fig 1. Frequencies of first eleven longitudinal (through thickness) modes of a joint with 1.6 mm thick aluminium adherends as a function of specific adhesive thickness.

Although a unique solution for the adhesive modulus and thickness can theoretically be achieved by measuring the frequency of only two modes of the joint, the equations for adhesive modulus and thickness tend to be ill conditioned, so any scatter in the experimental results would produce large inaccuracies in the calculated values of adhesive modulus and thickness. The accuracy of the calculated adhesive modulus and thickness can be increased if the frequencies of more than two modes are measured. In the work reported in Reference 6, this was achieved by measuring the frequencies of several modes (typically 8) and evaluating a 'best fit' solution for the modulus (E'_2) and thickness (l_2) of the adhesive layer. This was done by computing an error function, $E_r(E'_2,l_2)$, defined by,

$$E_r(E'_2,l_2) = \sum_{n=p}^{n=q} \left[(f_n(E'_2,l_2) - f_n^*)^2 / f_n(E'_2,l_2) \right] \tag{3}$$

where $f_n(E'_2,l_2)$ is the predicted resonant frequency of the n^{th} longitudinal mode of the joint, f_n^* is the measured frequency of the n^{th} mode, and p and q are the start and finish modes for the summation procedure. (The modes used in the summation procedure need not be consecutive.)

A typical spectrum obtained from an adhesive joint using the test procedure described in References 5 and 7 is shown in Fig 2, the dips in the spectrum corresponding to the resonances of the joint. It will be seen that in addition to the longitudinal modes, which are identified by their mode numbers, two shear modes of the joint are excited. This phenomenon is discussed in References 5 and 7. The first mode of the joint was not excited as its frequency lies below the usable bandwidth of the transducer employed. The results of Reference 5 show that excellent agreement was obtained between resonant frequency predictions of the type shown in Fig 1 using independently measured values of the adhesive modulus and thickness, and the experimentally measured frequencies. However, the results of Reference 6 indicated that when the measured frequencies were used to calculate the adhesive modulus and thickness using the procedure described above, the thickness was obtained to an accuracy of ±10% and the modulus to an accuracy of only ±20%. It would be desirable to improve this accuracy.

The method is also unsatisfactory because it requires the computation and storage of a matrix of natural frequencies corresponding to all the different possible combinations of adhesive thickness and modulus. This matrix would have to be computed for every different adherend thickness used, and the computation of the error matrix is also a time-consuming operation; in the work described in Reference 6, the size of the matrix was limited by only considering moduli in steps of 10% of the highest possible value, but this limits the maximum accuracy which can be obtained. The procedure also gives equal weight to all the modes, even though some may be more sensitive to adhesive thickness and modulus than others. An alternative technique was therefore sought.

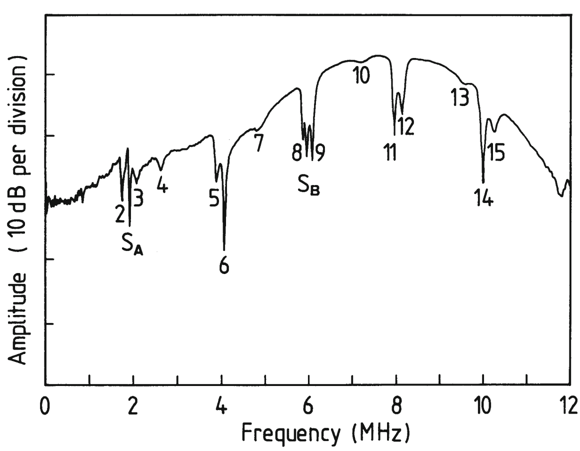

Fig 2. Example of measured spectrum from joint with 1.6 mm thick aluminium adherends, apparent adhesive modulus 7.9 GPa and adhesive thickness 0.53 mm. The numbers on the spectrum are the longitudinal mode numbers corresponding to the resonances. SA and SB are shear modes.

NEW METHOD

Principle

It is possible to compute a curve of possible adhesive thickness and modulus combinations which would give the measured natural frequency of each mode. This is shown in the solid curves of Fig 3a which are plots of adhesive thickness and modulus combinations which give the correct natural frequencies for the first 10 modes of a high specific stiffness, high modulus joint. Clearly, the curves all intersect at the point corresponding to the true values of thickness and modulus (apparent modulus 10 GPa and thickness 0.2 mm in this instance). Hence, in principle, if the natural frequencies of two modes are measured, the adhesive modulus and thickness can be determined.

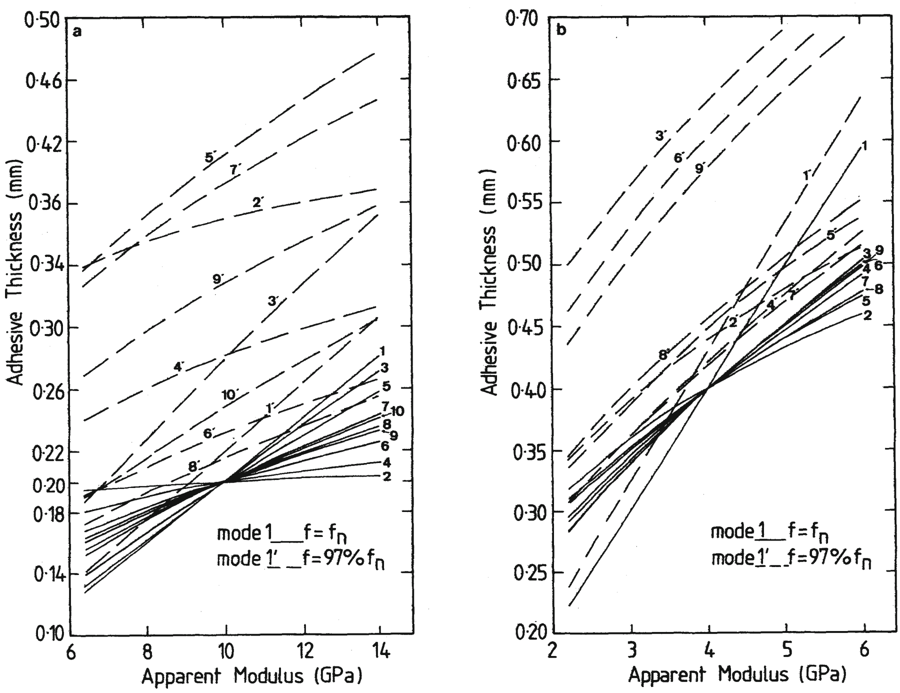

Fig 3. Plots of adhesive thickness and modulus combinations which give the natural frequencies of (a) modes 1 to 10 of high specific stiffness, high modulus joint (b) modes 1 to 9 of a low specific stiffness, low modulus joint. Plots for -3% frequency error also shown.

The form of the curves of Fig 3a may be deduced from the plots of natural frequency versus specific stiffness shown in Fig 1. For example, the curve for mode 2 in Fig 3a is almost horizontal, implying that if the thickness of the joint is changed slightly, a large change in modulus is required to keep the mode 2 natural frequency constant. The natural frequency is therefore almost independent of modulus, and hence of specific stiffness. Inspection of Fig 1 confirms that at high specific stiffnesses, the mode 2 natural frequency shows very little dependence on specific stiffness. This low dependence is due to the low strain in the adhesive layer in mode 2 [5]. The higher even modes have larger slopes in Fig 3a, and Fig 1 shows that they are more dependent on specific stiffness.

Mode 1 has the largest slope in Fig 3a, indicating strong dependence on specific stiffness, which is confirmed in Fig 1. (The slope of the mode 1 curve on Fig 1 is smaller than that of several other modes, but its frequency is much lower, so the percentage change produced by a given increment of specific stiffness is larger.) The higher odd modes are weaker functions of specific stiffness, and so have lower slopes in Fig 3a. The frequencies of the odd modes tend to show more dependence on specific stiffness than the even modes because thay all have a strain maximum at the middle of the joint, while the even modes have zero strain at this position [5]. Fig 3b shows a similar plot to that of Fig 3a for natural frequencies corresponding to a joint with low specific stiffness and low modulus. The form of the curves is similar that of Fig 3a, but the relationship between mode number and slope is changed; again, this can be explained by reference to Fig 1.

The solid curves of Fig 3a seem to suggest that measurements of the frequencies of modes 1 and 2 would give the most satisfactory results since the slopes of the curves are very different, so their point of intersection is easy to determine accurately. However, it is also necessary to investigate the sensitivity of the curves to errors in the measured frequencies. The dotted curves on Figs 3a and 3b show the relationship between modulus and thickness for each mode, but with the natural frequencies 3% below their true values. This shows that at high specific stiffness (Fig 3a), the mode 2 properties are highly sensitive to frequency errors, and so are likely to give unreliable results. Again, this behaviour might be expected from the results of Fig 1 which shows that the mode 2 natural frequency is almost independent of specific stiffness, so a large change in properties is required to account for the 3% frequency change. The sensitivities of the calculated properties to a 3% increase in frequency are slightly larger than those for a 3% decrease, and in this instance, the predicted stiffnesses will increase rather than decrease. Therefore, if the frequencies of several modes are measured, some cancelling of random errors is likely to occur, though the stiffness will tend to be slightly overestimated.

The analysis has shown that some modes are less sensitive to errors than others, and should therefore be selected for use in the prediction of modulus and thickness. The most appropriate modes to use depends on the specific stiffness of the joint, which can be found from measurements of the first mode natural frequency. Fig 1 shows that for any stiffness in the range plotted, there are at least three modes in addition to mode 1 which are strongly dependent on specific stiffness and so will give low error sensitivities.

Mode 1 is particularly useful since it is not only relatively insensitive to frequency errors, but its curve in the property plots (Figs 3a and 3b) crosses the curves of many of the other modes at a significant angle, so the point of intersection is easy to determine accurately. Measurement of the mode 1 frequency also enables the specific stiffness to be determined, and hence the other modes which are relatively insensitive to errors to be selected.

Implementation

For a symmetrical joint (one having equal thickness adherends of the same material) it may readily be shown using receptance analysis [5,8] that the frequencies of the odd numbered modes of the joint are given by the solutions to

$$\frac{\tan(\lambda_2 l_2/2)}{E_2 \lambda_2} = \frac{\cot(\lambda_1 l_1)}{E_1 \lambda_1} \qquad (2)$$

where $\lambda_1 = \omega(\rho_1/E_1)^{1/2}$, $\lambda_2 = \omega(\rho_2/E_2)^{1/2}$, ω is the frequency, ρ_1, ρ_2 are the adherend and adhesive densities respectively, l_1 and l_2 are the adherend and adhesive thicknesses and E_1 and E_2 are the apparent moduli of the adherends and adhesive. The frequencies of the even numbered modes are given from the solutions to,

$$\frac{\cot(\lambda_2 l_2/2)}{E_2 \lambda_2} = \frac{-\cot(\lambda_1 l_1)}{E_1 \lambda_1} \qquad (3)$$

Since the thickness of the adhesive layer only appears in one term in each of the above frequency equations, it may be calculated explicitly from values of natural frequency and modulus. (In the case of an asymmetrical joint, the equations for the thickness are transcendental, and an iterative solution would have to be employed.)

This calculation procedure has been used in the proposed scheme which is shown in Fig 4. The first mode frequency, f_1, is used to calculate the specific stiffness of the joint and hence to select three higher modes, a, b and c which will be insensitive to errors, as described in the previous

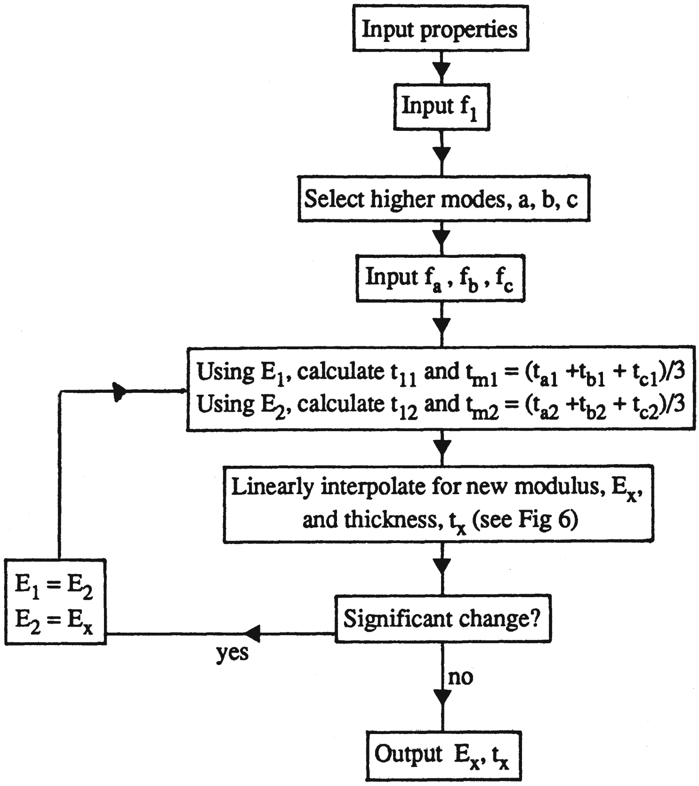

Fig 4. Flow diagram for proposed scheme.

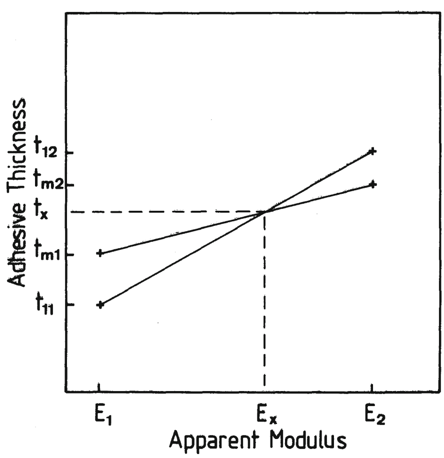

Fig 5. Interpolation to calculate new value of thickness and modulus.

section. The thicknesses, t_{11}, t_{12}, t_{a1}, t_{a2} . . . , required to give the measured natural frequencies in the four modes for each of two values of apparent modulus, E_1 and E_2, are then calculated. These modulus values are normally chosen to be at the extremes of the range likely to be found. The mean thicknesses, t_{m1} and t_{m2}, obtained from the higher modes for the two modulus values are then evaluated. A linear interpolation is then carried out to obtain predicted values of modulus, E_x, and thickness, t_x. This procedure is shown schematically in Fig 5 which shows the thicknesses for mode 1, t_{11} and t_{12}, and the average thicknesses for modes a, b and c, t_{m1} and t_{m2}, at each of the two moduli. This diagram has the same form as the plots shown in Figs 3a and 3b, and an estimate of the modulus and thickness of the joint may be obtained by linear interpolation. The new value of modulus is then used to replace one of the original values and an iteration procedure is followed. Since the curves in Figs 3a and 3b are good approximations to straight lines, the first estimate is usually close to the final value and convergence is rapid.

The thicknesses calculated from the first mode natural frequency at each value of modulus have not been averaged with those obtained from other modes in this procedure because Figs 3a and 3b show that the slope of the first mode curve in the property plots tends to be steeper than that of the other modes and therefore intersects the other curves at a larger angle, leading to lower sensitivity of the intersection point to small errors. Averaging the first mode results with those from other modes would tend to remove this desirable feature. It does, however, mean that it is particularly important to obtain accurate values of the first mode resonant frequency.

RESULTS

The algorithm has been tested on three sets of lap joints made with aluminium adherends and epoxy adhesives; details of the types of adhesive used and the manufacturing procedure can be found in References 5 and 6. An independent value of the apparent modulus of the adhesive used in each joint was obtained from tests on a bulk specimen of the adhesive which was manufactured at the same time as the joints, and the adhesive thickness was obtained by measuring the overall joint thickness with a micrometer and subtracting the known adherend thicknesses.

The natural frequencies of the joint were measured using the same procedure as that described in References 5 and 7, it being necessary to carry out tests with two different transducers (nominal centre frequencies 1 MHz and 10 MHz) in order to measure the frequencies of all the modes of interest.

TABLE 1. RESULTS

Joint Set		1	2	3
Adherend Thickness (mm)		1.60	1.60	1.55
Independently Measured Apparent Modulus (GPa)		3.74	3.63	7.52
Independently Measured Adhesive Thickness (mm)		0.45-0.58	0.07-0.11	0.09-0.16
	Joint No	Calculated Apparent Modulus (% of independently measured value)		
	1	100.8	100.8	101.5
	2	100.8	99.7	93.4*
	3	99.7	94.8	89.6*
	4	95.7	94.5	84.8*
	5	94.1	93.7	74.3*

* porosity found in joint

Calculated thicknesses all within 0.01 mm of independently measured values

The results are given in Table 1 and it can be seen that on joint sets 1 and 2, the bounds of the calculated modulus were +0.8% and -6.3% of its independently measured value. Larger errors were seen on four of the joints in set 3, but when these joints were broken, it was found that they contained large areas of porosity. This indicates that the presence of porosity will tend to be interpreted as a reduction in adhesive modulus; if it is desirable to distinguish between these effects, this could be achieved by time-domain ultrasonics or possibly by checking the apparent damping of the resonances measured by ultrasonic spectroscopy [9].

The results therefore show a marked improvement on those obtained on similar joints using the technique reported in Reference 6 and this must now be confirmed on more specimens. An initial study of the operation of the algorithm suggests that because the changes in the predicted properties obtained when the measured natural frequency is higher than its true value are slightly larger than those obtained when it is underestimated by the same percentage, the effect of random frequency errors will tend to be to reduce both the calculated thickness and modulus. This may explain why the modulus tended to be underestimated in the results presented here, but more tests need to be carried out to confirm this. (The accuracy of the independent thickness measurement was ±10% on the thinner joints, so small inaccuracies in the calculation would not readily be detected.)

DISCUSSION AND CONCLUSIONS

The initial results presented here indicate that the proposed method is a significant improvement over that developed in Reference 6 since the largest error in modulus has been reduced from 20% to 6.5% and the thickness accuracy is as good as that obtained from independent measurements using a micrometer. The technique also removes the need to store a large matrix of data for each possible adherend combination, and the processing time has been reduced to about 2 seconds on an HP9816 micro-computer programmed in Basic. If necessary, this computation time could be significantly reduced by using a more efficient programming language.

Ultrasonic spectroscopy can therefore be used to check the cohesive properties of the adhesive after the joint has been made, a change in adhesive modulus or thickness from the nominal values indicating a fault in the curing process. In their tests, Alers et al [3] found that a 5% reduction in adhesive modulus corresponded to a 7% reduction in joint strength and Rokhlin [10] showed that a 5% reduction in the modulus of an epoxy resin sample would be accompanied by a drop in shear strength of about 10%. While the correlation between adhesive modulus and joint strength will depend on other factors such as adhesive type, joint design and type of loading, these results indicate that a test which can detect adhesive modulus changes of around 5% will be useful in NDT.

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