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NON-DESTRUCTIVE EXAMINATION OF FIBRE REINFORCED POLYMERS, WITH SPECIAL REFERENCE TO CONTINUOUS CARBON FIBRE REINFORCEMENT

by
C. N. Owston, Senior Research Officer
Cranfield Institute of Technology, U. K.

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"NON-DESTRUCTIVE EXAMINATION OF FIBRE REINFORCED POLYMERS WITH SPECIAL REFERENCE TO CONTINUOUS FIBRE REINFORCEMENT"

C. N. Owston
Cranfield Institute of Technology
Cranfield, Bedford, England.

Abstract

The existence of flaws in any material or component will generally reduce the strength and fatigue performance. The features which constitute defects in metallic components are well known and there are established techniques for their detection, non-destructively.

Fibre reinforced polymer components often fail to reach their design performance, presumably due to the existence of defects. This paper reviews the work at Cranfield concerned with:-

i) ascertaining those features which cause flexural fatigue performance to be lower than expected,

ii) the establishment of non-destructive techniques of examination to locate these defects.

1. Introduction

Structural failures in metallic components, other than design faults, are usually the result of macroscopic imperfections within the metal. Shrinkage cracks in a casting may reduce its static load bearing capacity, surface notches in a component subject to cyclic stressing may reduce its fatigue performance, porosity within a weld may cause the weld to crack. That these features of the metal components are 'defects' has been learnt by experience, sometimes over centuries, and existing non-destructive testing techniques have been devised to detect these defects. When dealing with entirely different materials e.g. fibre reinforced composites it is first necessary to realise that cracks are not necessarily defects, it is only in certain circumstances in metals that we have found that cracks are defects. In other materials and different circumstances cracks may not be important, they might even be advantageous.

Many conventional non-destructive testing techniques, e.g. radiography, ultrasonic pulse-echo flaw detection, can be applied successfully to carbon fibre composites. Successfully must be qualified, successfully in that inhomogeneities within the material can be detected. In the early enthusiasm for carbon fibre composites many people tried conventional techniques, attempted to relate the features found with failure in the components, found the non-destructive tests gave spurious indications and abandoned all testing with the conclusion "It is impossible to non-destructively examine carbon fibre composites".

This conclusion was undoubtedly premature and resulted from failure to appreciate the background to conventional non-destructive testing techniques. Before any conclusion can be reached it is necessary to identify the causes of failure, see if non-destructive testing techniques appropriate to the causes and the material can be devised and finally prove those techniques by correlation studies on test components.

At Cranfield we have been principally concerned with flexural fatigue in carbon fibre composites. Studies of material properties, failure modes and non-destructive testing have run concurrently.

2. Specimens

Most of the work reported in this paper was concerned with flexural fatigue. The specimens (figure 1) were mounted as shown on a powerful electromagnetic vibrator and driven at their lowest natural resonant frequency (150 Hz to 200 Hz depending on specimen). The amplitude was controlled electronically (figure 2). The stress pattern in the specimen was that of a three-point bend test with the stress fully reversed each half cycle. The stress in the specimen was calculated from the amplitude of vibration, the mass of the steel end pieces and the resonant frequency.

The fatigue properties of CFRP in terms of cycles to failure at any given stress are not very reproducible but tests showed the existence of an effective fatigue limit which could be found using a step test. In this type of test the specimens follow a set pattern of gradually increasing steps of applied stress, spending a set period at each level, until a stress is reached where the specimen fails.

FIG.1 . FATIGUE SPECIMEN USED WITH ELECTRO-MECHANICAL VIBRATOR

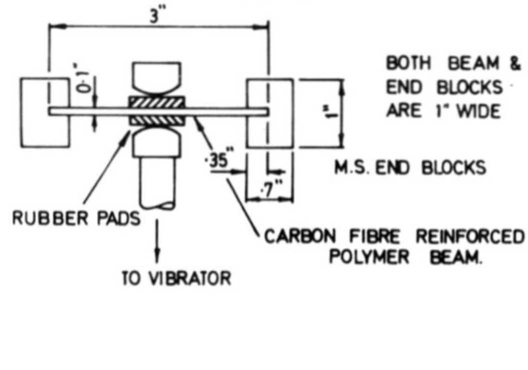
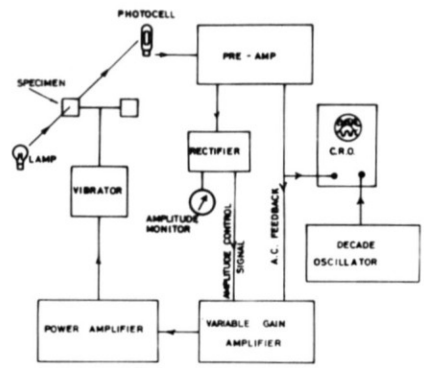


Figure 2. Electrical System



All the specimens concerned in this paper were made from pre-preg sheet containing continuous fibre. The unidirectional specimens had the fibres running lengthwise; for cross-ply specimens orientations are relative to the long axis of the specimen.

3. Material Parameters Relating to Fatigue Performance

The properties of a fibre/resin composite depend on the raw materials, fibre and composition of the resin, on the design of the system, desired fibre array, on processing, the achieved placement of fibres and cure of the resin, and on incidental factors, such as voidage, which are often the result of processing technique. Our work has not been concerned with the raw materials.

One fatigue specimen was made with a $\pm 45^{\circ}$ layup in order to produce large shear stresses with the matrix and to make the matrix properties dominant. The stiffness and internal damping of the specimen were studied as a function of temperature. The mechanical 'Q' of the specimen, showed a minimum at a temperature corresponding to the glass transition temperature, Tg, for the matrix (figure 3). As specimen

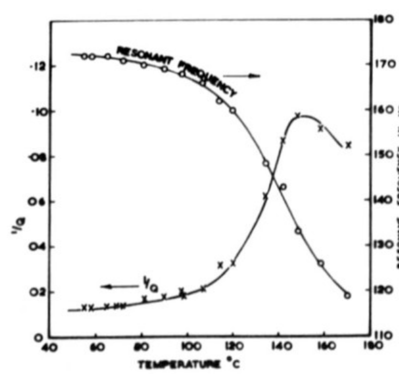


FIGURE 3. VARIATION IN RESONANT FREQUENCY AND

temperature approached Tg the internal damping rose (lower 'Q') and the shear modulus of the matrix (indicated by the resonant frequency of the specimen) fell.

In unidirectional fatigue specimens the fatigue performance (effective fatigue limit) depended on cure time and cure temperature (figure 4). A detailed study of the properties of two resin systems (reference 1) has suggested that optimum fatigue performance invariably coincides with a particular value of Tg; this value being a characteristic of the resin system. The movement of the peak in the fatigue properties to longer cure times when lower temperatures are used merely reflects lower reaction rates leading to longer times to achieve the required Tg. The optimum value of Tg is invariably high, apparently so as to ensure that at normal running temperatures the moduli of the matrix are substantially temperature independent (avoiding the steep fall as Tg is approached, figure 3). This high value of Tg also corresponds with minimum internal damping in the specimen at normal running temperature.

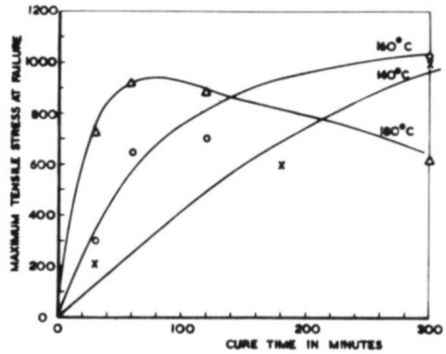


FIGURE 4. EFFECT OF CURE ON THE FATIGUE PERFORMANCE OF CFRP

The effect of fibre layup was studied using a series of fatigue specimens (figures 5 and 6). Specimens 1 to 11 were unidirectional; 25 and 26, $\pm 15^{\circ}$ layup; 27 and 28, $\pm 22^{\circ}$ layup; 29 and 30, $\pm 30^{\circ}$ layup; 31 and 32, $\pm 35^{\circ}$ layup. The results from the unidirectional specimens established the normal fatigue performance. The presence of any cross-ply fibres appeared to reduce the strain at failure (amplitude of vibration of specimen) to a fixed value, lower than for the unidirectional material, and to reduce the failure stress by an amount depending on the severity of the cross-ply. Specimens 13 to 24 contained increasing amounts of fibre 'wash'. This was achieved by crimping the pre-preg sheet before cutting and placing in the mould. As for cross-plying, any wash immediately lowers the failure strain to a new fixed value while increasing wash progressively lowers the failure stress.

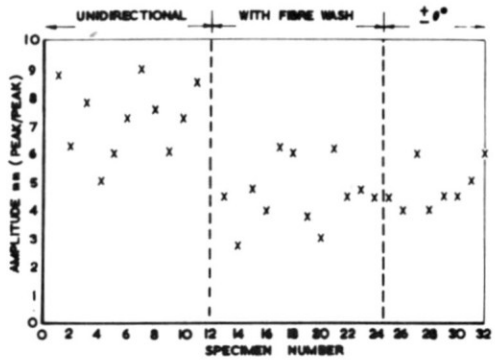


FIGURE 5. FAILURE AMPLITUDE VERSUS SPECIMEN NUMBER

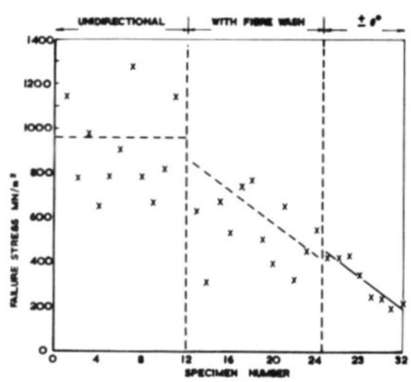


FIGURE 6. FATIGUE FAILURE STRESS VERSUS SPECIMEN NUMBER

4. Fatigue Properties and Modes of Failure

The stiffness of any fatigue specimen fell throughout to fatigue test (figure 7). After an initial rapid fall, the drop was usually linear with time until close to final fracture. The rate of fall in the linear region (expressed in Hz per period in the step fatigue test) varied markedly from specimen to specimen (7.5 Hz/period to 0.1 Hz/period). Many specimens have been sectioned, polished and examined optically. All specimens showed voids, even unfatigued material, but it was found that the voids had grown to a much greater size during fatigue in material which showed a fall in specimen frequency (see also section 5.3, stress wave emission).

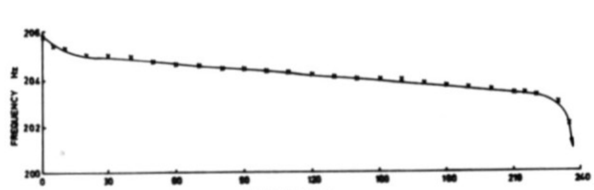


FIGURE 7 VARIATION IN RESONANT FREQUENCY DURING A FATIGUE TEST, A UNIDIRECTIONAL SPECIMEN

Two different types of CFRP have been studied; that made from untreated fibre and having a low interlamina shear strength (i.l.s.) and that made from treated fibre exhibiting a much higher i.l.s. The most obvious feature of fatigue failures in the low i.l.s. material was the extensive interlamina cracking but we believe that this was secondary to the fatigue failures and not the primary cause. In high i.l.s. material interlamina cracking was usually less marked and occasionally absent.

In the vast majority of the specimens sectioned the primary failure was a compressive buckling. Sometimes this initiated near the surface of the specimen but often the failure initiated internally. In the internal failures the fibres were buckled but the resin matrix did not appear to be cracked. This can only be explained if the matrix has softened locally. These soft spots could be due to local compositional variations in the resin but our feeling is that they are the result of local hot spots. Even at 200 Hz, fatigue specimens of CFRP ran cool, bulk temperature rises were less than 5°C and commonly of the order of 1°. Fatigue failures were not due to bulk temperature but this does not exclude the formation of local hot spots. Local distortions in fibre layup and voids could markedly reduce local thermal conductivity. Fatigue performance was impaired when matrix internal damping was high, when fibre layup increased the matrix stresses and when Tg was low. A low Tg meant that a smaller temperature rise was required to lower the stiffness of matrix by an appreciable amount. Rubbing between the surfaces of cracks and among debris within cracks or voids appeared to be a contributory cause in some instances.

In a few cases compressive failure may have resulted from instability consequent on void growth. In a few instances, in high i.l.s. material, failure was the result of fibre and matrix cracking, details of the cause of this mode are not understood.

5. Non-Destructive Examination - Techniques and Individual Results

Experience so far has failed to reveal a technique which will give a comprehensive picture of the material. Assessment of the quality of a component can be made, but it depends on combining the results of several different types of non-destructive examination. The methods which have been tried are first discussed individually and then their combination is discussed in section 6.

5.1 Radiography

CFRP presents no special problems for radiography except that thin sections (~ 2mm) require low voltage (~ 10 KVp) radiation from berylium window tubes. Resolution and sensitivity are usually very good but the information revealed is usually restricted. Except for very thin sections, fibres are not resolved from resin and the radiograph will only reveal porosity and cracks whose plane is parallel to the x-ray beam. Flat interlamina cracks, the commonest fault in CFRP, are difficult to detect.

5.2 Ultrasonics

Standard pulse echo and through transmission non-destructive testing techniques work very satisfactorily and readily detect porosity and interlamina cracking. In spite of the fibrous nature of the material, high frequencies can be used (attenuation ~ 2db/mm at 10 MHz) and resolution is good. The principal problem which is encountered is the vast amount of porosity in most specimens which can result in a very confused screen indication. For this reason we have prefered a scanning, immersion, through transmission technique using fine focus probes and detecting porosity by increased attenuation.

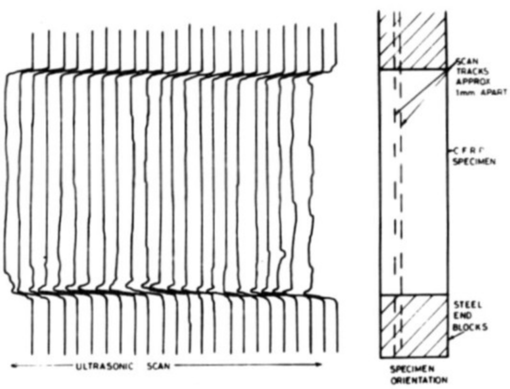


FIG 8 ULTRASONIC ATTENUATION THROUGH THICKNESS OF SPECIMEN 12

The specimens showed considerable variations, even before fatigue testing. Some were apparently free from large porosity (figure 8), others showed some regions of porosity while others could only be described as terrible, (figures 9 and 10). The mean attenuation also varied markedly from specimen to specimen, often not in accordance with the amount of gross porosity. This could be due to the existence of micro-porosity which varied in amount from specimen to specimen.

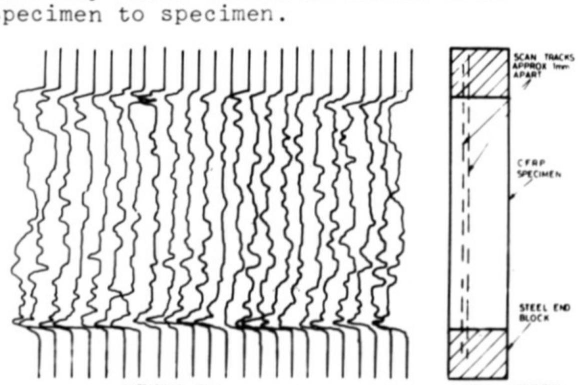


FIG 9 ULTRASONIC ATTENUATION THROUGH THICKNESS OF SPECIMEN 6

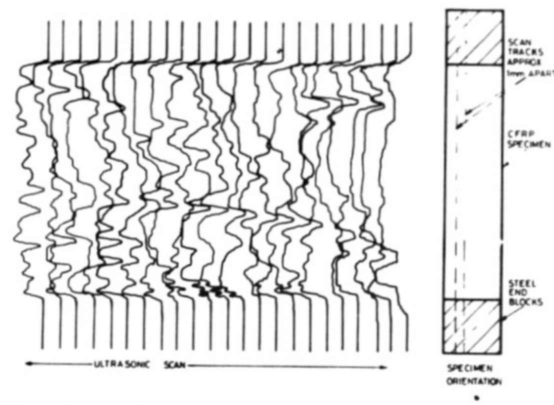


FIG 10 ULTRASONIC ATTENUATION THROUGH THICKNESS OF SPECIMEN 3

The specimens were re-scanned after fatigue failure. The failure zone was sometimes clearly defined (figures 11 and 12). Usually there was a considerable area of easily detected delamination (figure 11) but some specimens showed a crack (figure 12) rather than extensive delamination. A few specimens were eventually found to have failed as a result of extensive microcracking of matrix and fibres.

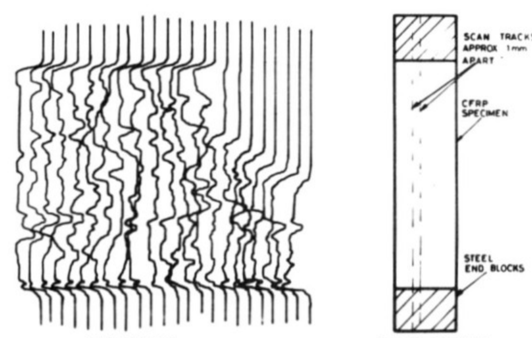


FIG 11 ULTRASONIC ATTENUATION THROUGH THICKNESS OF SPECIMEN 17 AFTER FATIGUE FAILURE

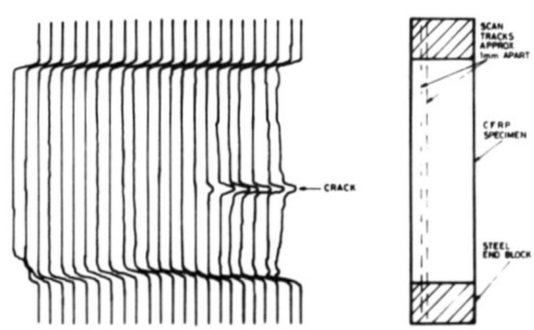


FIG 12 ULTRASONIC ATTENUATION THROUGH THICKNESS OF SPECIMEN 12 AFTER FATIGUE FAILURE

5.3 Stress Wave Emission

Our equipment is very simple. It consists of a ½ **MHz** piezoelectric crystal followed by a high gain, low noise amplifier and a series of pulse height discriminators. The discriminators pass all pulses whose height is greater than a pre-set amount. The relative heights of the thresholds used in the tests described below were:-

Channel number	1	4	7
Relative height	1	4	16

The fatigue specimens, complete with their end masses, were mounted in a simple three point bend frame. The fixed points supported the end masses while the specimen was deflected by a screw jack acting through a metal pad clamped to the centre of the specimen. This produced a load configuration similar to that used in the fatigue tests. The jack was calibrated and specimens were bent by measured amounts. The transducer was clamped to the centre of the specimen on the tension side (opposite face to that pressed on by the jack).

For some specimens a cyclic test was adopted. On the first cycle the specimen deflection was increased in 0.005" steps to 0.040 and the emission during each step increase was determined using various channels in the recording equipment. The specimen was unloaded and then re-deformed to the same total deflection. The emission during the second deformation was determined. The specimen was then repeatedly unloaded and re-deformed and emission during deformation determined.

In much of the reported work on acoustic emission, the test pieces are loaded up to failure and the results show a characteristic large increase in emission close to ultimate failure. The strains (and consequent stresses) used in this series of tests were much less than the ultimate strain for the samples, the tests were by no means "proof tests". The work was an attempt to correlate low strain (stress) emission with eventual performance.

Figures 13 and 14 illustrate the emission characteristics. There were wide variations, both in form and total counts, among the specimens.

During the first deflection of a specimen, emission either did not occur until a moderate deflection had been reached or increased only slowly with deflection. Eventually a threshold was reached beyond which emission increased rapidly with increasing deflection. The position of the threshold in terms of deflection, was much the same for all recorded channels for any specimen but varied from specimen to specimen. Birchon (reference 2) has suggested that this rapid increase in emission after an initial delay is associated with debonding. Because of this threshold effect, the total emission for a given deflection was not especially meaningful and in later work correlations were sought with the rate of rise of emission (counts per than additional deflection) after the threshold. The relative proportion of counts recorded in the channels used varied from specimen to specimen, typically channel 4 recorded 75% of the channel 1 count and channel 7, 35% of channel 1. The variations from the normal values may indicate changes in the type of defect.

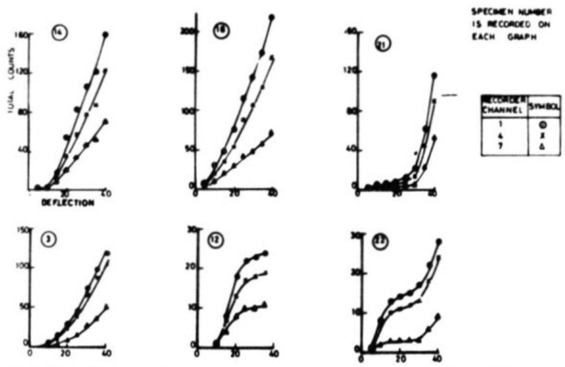


FIG 13 FIRST LOADING ACOUSTIC EMISSION COUNTS AS A FUNCTION OF DEFLECTION FOR A SELECTION OF SPECIMENS.

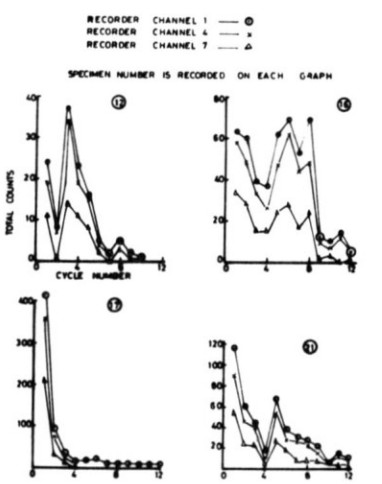


FIG. 14 ACOUSTIC EMISSION AS A FUNCTION OF CYCLIC LOADING

When the test pieces were cycled a variety of effects were noticed. Some specimens (no. 17, figure 14) showed a clear Kaiser effect, several (nos. 12 and 21, figure 14) showed a new maximum emission after a few cycles and others (no. 16, figure 14) apparently show a Kaiser effect a few cycles.

The specimens were re-tested after fatigue failure.

The "after failure" results showed the same characteristics as the initial tests and there are no obvious differences between failed and unfatigued specimens. It has been found that a crack generated at high stresses may not propagate at lower stresses and also that a failed specimen can show "healing". Either of these effects would account for the insensitivity of low stress acoustic emission testing to the presence of serious fatigue cracks. It was mentioned above (section 4) that loss of stiffness appeared to be associated with crack growth. As stress wave emission is also associated with crack growth some correlation might be expected. Figure 15 shows this correlation for material with a low i.l.s.

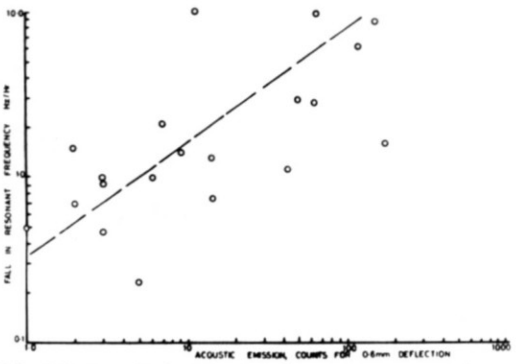


FIG 15 ACOUSTIC EMISSION (COUNTS FOR 0·6mm DEFLECTION) VERSUS Hz/hr DROP IN RESONANT FREQUENCY OF FLEXURE BEAM (1st YEAR SPECIMENS)

The rate of fall in the resonant frequency (in Hz change per hour) is compared with the acoustic emission from the specimen. The correlation is not exact but it is at least hopeful and its existence reinforces the idea of void propagation/generation being responsible for the fall in resonant frequency. A high acoustic emission implies easy propagation/generation of voids or cracks.

Figure 16 shows a similar but unsuccessful, attempted correlation for material with a high i.l.s. It may be that the indicated correlation in figure 15 was accidental or the loss in correlation may be due to the change in material. A greater proportion of the stress wave emission from the low i.l.s. material may have been the result of the propagation of interlamina cracks than was the case for the high i.l.s. material. A change in the principal source for the emission could account for the failure of the correlation for high i.l.s. material.

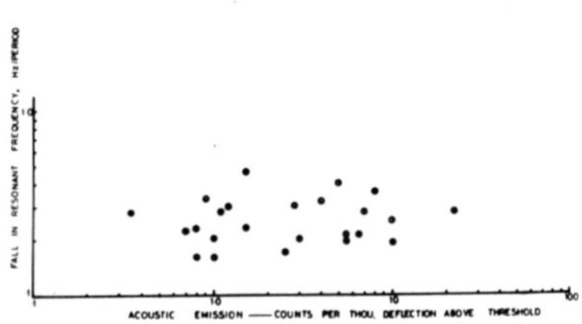


FIG 16 ACOUSTIC EMISSION VERSUS Hz/PERIOD DROP IN RESONANT FREQUENCY OF SPECIMENS FOR 2ND YEAR SPECIMENS

5.4 Eddy Current Testing

At first sight unidirectional CFRP should only be an electrical conductor in the fibre direction. In practice, normal processing techniques result in a great deal of fibre/fibre contact and the material is a conductor in all directions, if somewhat anisotropic. At very high frequencies current can flow perpendicular to the fibre direction by means of inter-fibre capacitance and the material behaves as an exceedingly lossy dielectric.

The theory of eddy current testing in CFRP differs markedly from that applicable to metals and is dealt with in detail elsewhere (reference 3). Sufficient to remark here that by using appropriate working frequencies (~ 50 MHz), suitable probes and measuring equipment it is possible to measure fibre volume fractions, fibre orientation and to detect cracks.

Figure 17 shows a typical volume fraction calibration. Specimens of various volume fractions were measured on the eddy current equipment and then the mean fibre volume fraction found using a burn-off technique. The scatter in the points in figure 17 is partly due to the eddy current reading being a point value while the burn off value is an average over a wider area and partly to the effect of voids etc. on the burn off value.

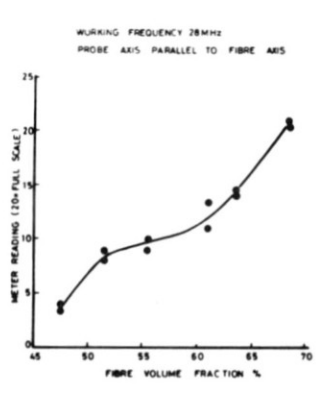


FIG 17 EDDY-CURRENT (FREQUENCY DEVIATION METER) READING VERSUS FIBRE VOLUME FRACTION FOR HORSESHOE PROBE

It is possible to make probes that are orientation sensitive. Figure 18 shows the responses from specimens of various lay-ups. The appropriate specimen was placed on a turntable and rotated beneath the probe; the output of the eddy current equipment was plotted using a pen recorder. As the lay-up of the specimen is changed a change in the form of the plot is obvious. The 0/180/360° peaks become smaller as more of the specimen is oriented at 90° to the surface fibres and peaks appear at 90/270/450° indicating the presence of the 90° layers in the lay-up.

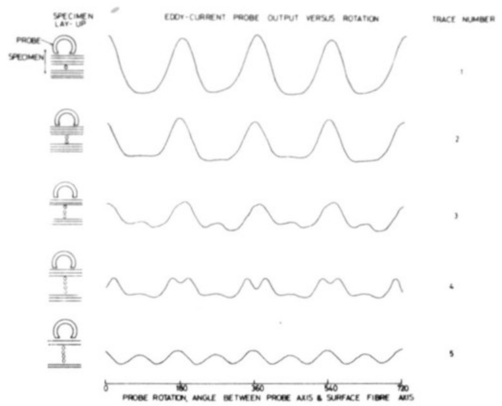


FIG 18 EDDY-CURRENT PROBE SENSING CHANGES IN SPECIMEN LAY-UP

Trace 4 of figure 18 is not particularly convincing in the series and it must be admitted that the traces were specially selected. Figure 19 shows three traces all from the same specimen but with the probe over different points. This is an extreme example but shows the differences which can be found. The differences from point to point on the same board undoubtedly reflect wash in at least some of the pre-preg layers. Some boards even showed wash in the surface layers and these areas certainly gave rise to distorted eddy-current traces.

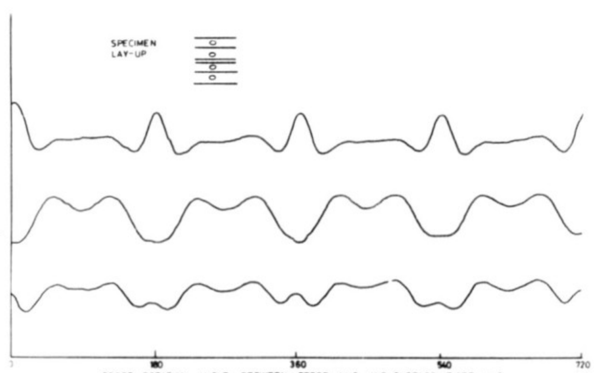


FIG 19 EDDY-CURRENT PROBE SENSING VARIATIONS IN LAY-UP FROM POINT TO POINT ON A SPECIMEN

6. Correlation

One possible correlation has already been mentioned in section 5.2 in dealing with stress wave emission. Other correlations are more complex.

6.1 Initial Resonant Frequency of Specimens

If all other factors remained constant, the stiffness of the specimens, i.e. the initial resonant frequency, would depend solely on fibre volume fraction and hence on mean eddy-current reading. High eddy-current readings were equivalent to high fibre volume fractions and predict high resonant frequencies. Figure 20 shows many deviations from such a correlation.

Volume fraction is not the only material parameter which affects stiffness. Foye (reference 4) has shown that voids lower the stiffness of a composite, i.e. they lower the effective fibre volume fraction. As voidage increases the through transmission ultrasonic attenuation, specimens with a high attenuation should be less stiff. Taking 6 db attenuation as 'normal' the effect of greater and smaller attenuation has been indicated in figure 20 by a series of arrows. The length and direction of the arrows indicates the relative magnitude and the direction of displacement of the plotted points which could be expected if voidage were taken account. The exact displacement cannot be calculated as a correlation between attenuation, voidage and stiffness has not been established. Three points are moved further from the suggested line, six points which were well away are brought nearer and the effect on others is generally advantageous, if small. The general trend of introducing ultrasonic attenuation into the correlation is to effect an improvement.

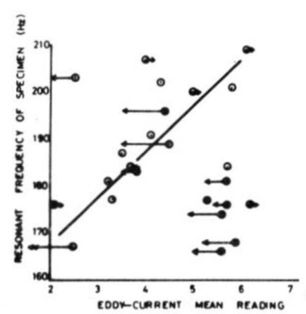


FIG. 20 INITIAL RESONANT FREQUENCY OF SPECIMENS VERSUS MEAN EDDY-CURRENT READING

6.2 Prediction of Fatigue Failure

In CFRP the propagation of fatigue failures often requires only a few cycles after a life of up to 10^7 cycles. In metals, where propagation takes a reasonable fraction of overall life, it is sufficient to detect early fatigue damage, in CFRP it is more essential to ascertain which parts of the material are susceptible to fatigue damage.

Fatigue failure may be due to local hot spots (section 4). Do the measured quantities, stress wave emission, ultrasonic attenuation and eddy-current/volume fraction reading represent important factors relating to failure?

i) high temperature requires low volume fraction, i.e.,

$$\text{temperature rise} \propto \frac{1}{\text{eddy-current reading}}$$

ii) high temperature requires voidage i.e. temperature rise $\propto$ ultrasonic attenuation.

iii) high temperature requires crack rubbing, i.e. temperature rise α acoustic emission (This could be emission on first loading or that which occurs on each cycle of cyclic loading).

The crudest possible combination of these crude indications of performance is:-

$$\text{temperature rise } \alpha \ \frac{\text{Emission x Attenuation}}{\text{Eddy-current reading}}$$

$$= \text{Failure Criterion}$$

A large value for the "Failure Criterion" should indicate poor fatigue performance. Specimens were examined before being put on fatigue test, after failure the location of the failure was identified, the local stress at this point estimated and the "Failure Criterion" for this point calculated. Figure 21 shows the correlation achieved. Considering the crudness of the analysis, the correlation is very good and this would indicate that if:-

i) more appropriate proportions of the three NDT characteristics were taken,

ii) allowance was made for other factors such fibre wash,

iii) a more reliable estimate of stress at the point of initiation of the fatigue failure was obtained,

iv) the acoustic emission from the failure region was used instead of that from the whole specimen,

a better and perhaps more useful correlation would be achieved.

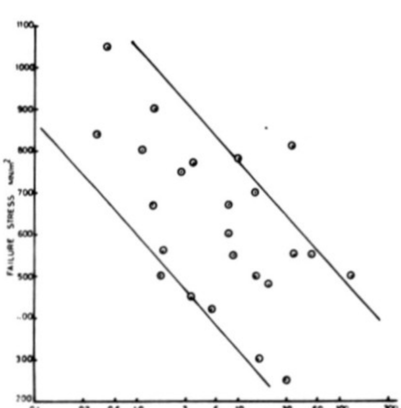


FIG 21 FAILURE CRITERION VERSUS MAXIMUM STRESS AT POINT OF FAILURE

7. Conclusions

The initiation points for mechanical failures in carbon fibre reinforced polymers are often different from the initiation points in metallic structures. The immediate consequence is that non-destructive testing techniques which are valid for metals may give erroneous indications on CFRP. This does not mean that CFRP cannot be examined. The material must be studied to determine causes of failure and then non-destructive techniques devised to locate these causes.

In flexural fatigue, fibre volume fraction, fibre orientation and void content have been identified as the principal factors affecting stiffness. An eddy-current technique has been devised which will indicate the fibre parameters and it appears that ultrasonic testing will give the required information on voidage. Failure, in many samples, appears to be due to the existence of local hot spots and some success has been achieved in correlating fatigue performance with a combination of the results of three non-destructive tests, an eddy-current test, through transmission ultrasonic attenuation and acoustic emission.

8. References

1. JOHNSON, J. B., OWSTON, C. N., "The effect of cure cycle on the mechanical properties of continuous fibre reinforced epoxy resins" to be published.

2. BIRCHON, D., "The Philosophy and Practice of Structural Validation", ASTM Conference on Predictive Testing, Anaheim, California, April, 1971.

3. OWSTON, C. N., "Eddy-Current Methods for the Examination of Carbon Fibre Reinforced Epoxy Resins", to be published.

4. FOYE, R. L., "Compression Strength of Unidirectional Composites", paper presented at AIAA, January, 1966.