Manufacturing defects in fibre-reinforced plastics composites

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Non-destructive evaluation has three major functions for research, development and applications testing of fibre-reinforced composite materials and structures:

☐ *initial inspection of test specimens and confirmation of the structural integrity of new components*
☐ *monitoring laboratory tests in progress and evaluation of components subjected to service loads*
☐ *analysing test results after failure and proof loading of components during their service life.*

The paper reviews the defects which are likely to occur during the manufacture of such materials, and considers the criticality of the defect which may need to be detected. The implications of material inhomogeneity and anisotropy are examined in the context of non-destructive testing techniques.

Introduction

Fibre-reinforced plastics may range from relatively flexible (glass fibre) to extremely stiff (carbon fibre) and from brittle (carbon fibre) to extremely tough (aramid fibre). The performance of such materials is limited by the ability to withstand high stress (mechanical, electrical or environmental). Two forms of problem are critical to the material response, defects (built in at manufacture) and damage (changes due to use).

The effects of damage have been extensively covered in the open literature [eg. [1-4]]. Heslehurst and Scott [3] considered that the "level of structural degradation in engineering properties varied" with:

☐ defect severity
☐ defect location and orientation
☐ frequency of defect occurrence
☐ component load path criticality and stress state
☐ defect idealisation
☐ design load levels and nature
☐ defect detectability and detection capabilities
☐ local repair capabilities
☐ component configuration
☐ environmental conditions
☐ loading history
☐ material property variations, and
☐ acoustic vibration response.

Their paper contains comprehensive tables of the defects and damage pertaining to composite aircraft components.

However, the coverage of the effects of defects in the published literature is limited, but concern for their effects is becoming more widespread, particularly as the aerospace industry begins to adopt them for primary structures. It is the purpose of this paper to review the defects which are likely to result from poor manufacture, and to assess their potential to seriously degrade the performance of the composite. The implications for the development of new non-destructive testing techniques are highlighted.

Fibre orientation

In traditional engineering materials, the mechanical properties do not vary greatly with the direction considered. However, fibre-reinforced composites will have high strength and stiffness along the principal axis of the reinforcement and low strength or stiffness in directions in which there is no fibre. This is a particularly important consideration in fibre-reinforced plastics, where the ratio of stiffness and/or strength between the fibre and the (resin) matrix is high. It is, perhaps, less critical in fibre-reinforced metals or fibre-reinforced ceramics where the matrix has a stiffness/strength closer to that of the fibre and the fibre is used to impart toughness to the matrix.

The effective use of the fibre is a function of the alignment of the fibre with the stress/strain direction. Misalignment of up to ten degrees will cause a small degradation of the mechanical properties. Between ten and twenty degrees, the loading of the fibre changes from direct tension/compression to shear loading of the weaker interface. There is, therefore, a considerable drop in mechanical properties at this level of misalignment. The degree of misalignment which will need to be detected is obviously a function of the safety factors used in the design of the component. Beyond twenty degrees the fibre is loaded by direct tension/compression of the fibre/matrix interface, and the utilisation of the fibre properties is therefore low.

Typical values of the elastic modulus of different glass fibre/polyester composites are shown in Figure 1, together with their dependence on the chosen direction relative to the reference axis. Clearly, non-destructive testing techniques must take account of this anisotropy.

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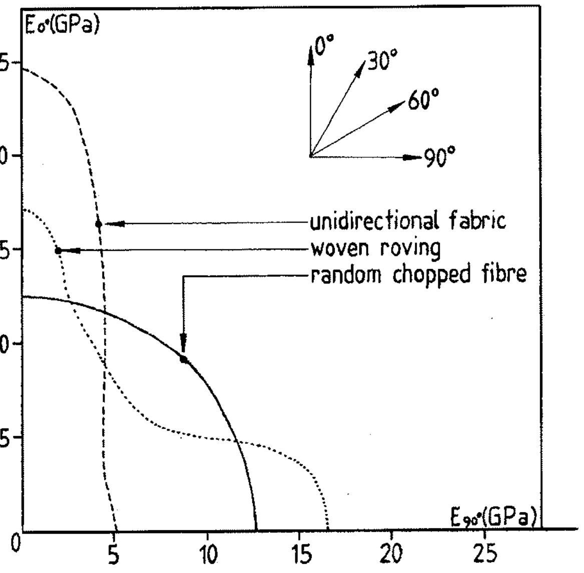

Figure 1. The in-plane orientation dependence of the elastic modulus for different glass fibre/polyester resin composites

Stacking sequence

The stress/strain field in most engineering structures is not usually confined to just one direction. In order to optimise the design, the fibres are arranged in a series of layers with defined orientations. For example, in a structure exposed to biaxial tension, fibres would most appropriately be aligned on the two orthogonal axes. However, if stress is also introduced at the 45 degree angle between the two fibre directions, then neither set of fibres will be well placed to carry the load. At this third (bias) angle, it will be necessary to introduce a further set of 45 degree fibres to avoid all the stress/strain being introduced into the two orthogonal fibre sets by direct tension on the interface.

For tension and compression loading, the relative positions of the fibres in each direction is not highly critical, provided the layup is balanced by symmetry. However, in flexural load situations, this consideration comes to prominence. Fibres at the laminate surface may be considered as equivalent to the flanges of an I-beam, whilst fibres close to the neutral axis are equivalent to the web of the I-beam. It is therefore important that sufficient fibres in the surface layers of the laminate are aligned with the anticipated bending direction. Figure 2 illustrates the effect of changing layer positions in a carbon-fibre-reinforced plastic, by comparison of the different layer sequences with the equivalent aluminium constant cross-section beam in flexure.

A typical aircraft structure may be made up of one- or two-hundred layers of reinforcement fabric. If two adjacent layers have been laid down with their orientation transposed, then the stiffness of the laminate in bending may be compromised. The most critical layers will inevitably be those close to the surfaces of the laminate, but it may be that the transposed layers are positioned on the inner layer of a complex structure, and hence they are difficult to detect by non-destructive techniques.

Fibre waviness

Mansfield and Purslow [5] studied the effect of fibre waviness on the longitudinal shear modulus (LSM) and tensile modulus (LTM) of uniaxial fibre reinforced composites. The paper includes both a general theoretical analysis and an experimental determination of the degree of waviness.

The theoretical analysis shows that an increase in the amplitude of the fibre waviness causes an increase in the LSM, but the increase is smaller at longer wavelengths. For very short wavelengths, increases of 30% are predicted. In unidirectional CFRP, increases of up to 8% in LSM are theoretically possible at half-wavelengths of about ten-times the diameter of the fibre. Analysis for a matrix-embedded wavy fibre does not exhibit any noticeable nonlinear behaviour under end load and the influence of fibre waviness on the LTM is negligible.

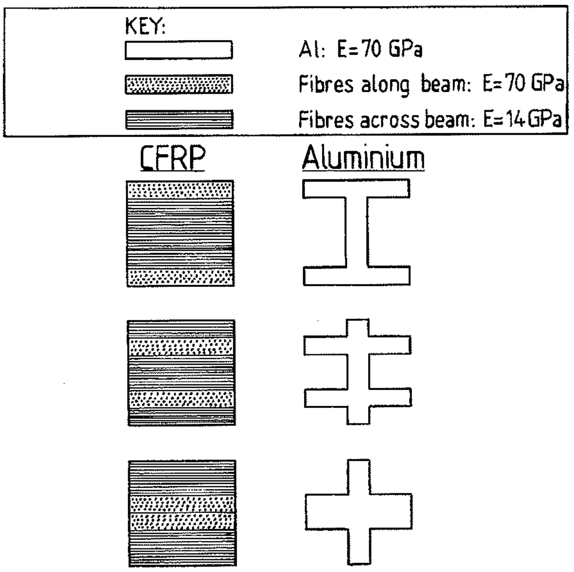

Figure 2. The effect of changing layer positions in a carbon-fibre-reinforced plastic composite. The equivalent aluminium section in flexure is shown on the right

Abraded and polished cross-sections were examined in the scanning electron microscope with six samples taken within a depth of 180 μm. The minimum interfibre spacing for 17 pairs of fibres was monitored. In the sample examined (high-strength type-II carbon fibres in ERCA 4617 resin), the interfibre gaps varied approximately sinusoidally, but the half-wavelength was approximately twice the critical value. The effect of fibre waviness on the LSM was thus negligible.

Jortner [6] has described a theoretical approach (implemented numerically) to analyse the stress equilibrium and strain compatibility for 'wrinkles' formed by co-operative (more-or-less in-phase) distortions in adjacent reinforcement layers.

Garala [7] tested 15 mm-thick carbon/epoxy cylinders under hydrostatic pressure. Failures occurred well below the design pressure and were ascribed to layer waviness (an out-of-plane undulation of one or more layers in a unidirectional laminate). Analysis of the loading condition indicated regions of high interlaminar shear stress and the possibility of interlaminar normal tensile shear [8, 9]. These stresses have been shown to be of sufficient magnitude to produce premature failure below the design pressure of the perfect cylinder [10]. The local changes in volume fractions were implicated (see fibre distribution below).

Adams and Hyer [11] fabricated flat specimens of 0°/90° carbon fibre/polysulphone thermoplastic containing isolated layer waves. Two parameters were used to define the defect severity (see Figure 3), the ratio of wave amplitude (δ) to wavelength (λ) and the maximum angle of fibre rotation, θ_{max}. For harmonic wavelengths, $\theta_{max} = \tan^{-1}(\pi\delta/\lambda)$. A central zero degree wavy layer in a 22-layer bidirectional laminate produced reductions of between 1 to 36% in quasi-static IITRI compression fixture tests. For moderate layer wave geometries ($\delta/\lambda < 6\%$) failure occurred at 88% of the strength of control specimens either in the tabs or within the wavy layer with

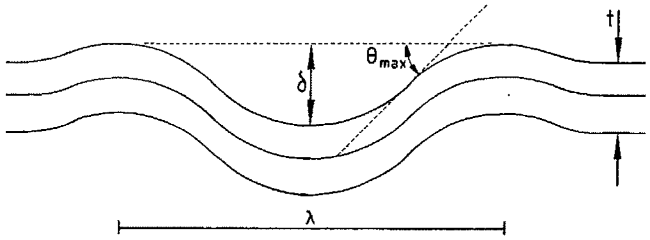

Figure 3. The definition of layer wave geometry parameters (from Adams and Hyer in the April 1993 Journal of Reinforced Plastics and Composites, with the permission of the authors and of Technomic Publishing Co Inc)

brooming failure (creating an extended fracture surface with many individual reinforcement filaments exposed), through-thickness splaying of the layers and numerous delaminations. For $\delta/\lambda > 6\%$ (severe waviness) failure occurred at an average 63% of control strength in a sudden catastrophic manner at the location of the wave.

Frankle *et al* [12] used finite element analysis to predict the response of carbon-carbon involute rings that were intentionally fabricated with wrinkles. The finite element predictions compared favourably with hoop tension data. A computer code was developed to use digital computer tomography and ultrasound transmission data to define spatially varying material properties for use in the finite element analysis.

Highsmith *et al* [13] developed a micromechanics model based on the kinematics of the fibres to predict the behaviour of unidirectional composites with initially (idealised sinusoidal) wavy fibres under compressive loads, including nonlinear shear of the matrix. The model appears to be capable of predicting the compressive behaviour of the T650-42/Radel C thermoplastic laminates tested. The development of fibre waviness during forming operations was highlighted as meriting further study.

Rai *et al* [14] have developed a theoretical elastic model to predict the composite stiffness of a unidirectional lamina as a function of in-plane sinusoidal fibre waviness. The analysis revealed a more critical relationship between fibre waviness and laminae stiffness than anticipated, causing concern for aerospace primary structural applications. For full strength and life to be achieved, manufacturing processes should be re-examined to minimise the possibility of induced fibre waviness.

Mrse and Piggott [15] observed that fibre waviness decreased the compressive modulus of carbon-fibre-reinforced plastics approximately as the square of the mean fibre angular deviation. Compressive strength also decreased. The decrease in modulus with increasing strain appeared to be associated with the fibres themselves, rather than being caused by the fibre waviness.

Wisnom [16] used finite element analysis to investigate the effects of assumed waviness on the compressive strength on unidirectional carbon fibre/epoxy composites under uniform loading. The maximum compressive stress was shown to be largely controlled by the misalignment angle. As the load increased, the shear stress resulting from misalignment caused the fibres to rotate, increasing the misalignment angle. The reduction in shear tangent modulus with increasing shear strain leads to instability with further rotation occurring without any increase in applied load. This mechanism is believed to be responsible for the kink-bands which are seen in compression failures. In a further non-linear finite element analysis, Wisnom [17] showed that the same mechanism of shear instability can occur under pure bending.

This effect on composite performance has risen to prominence in recent years. The full implication of the waviness of the reinforcement fibres is not yet resolved. However, all bidirectional woven cloths suffer from waviness due to the inherent crimp in the weave (Figure 4) and hence the problem has great significance for the industry.

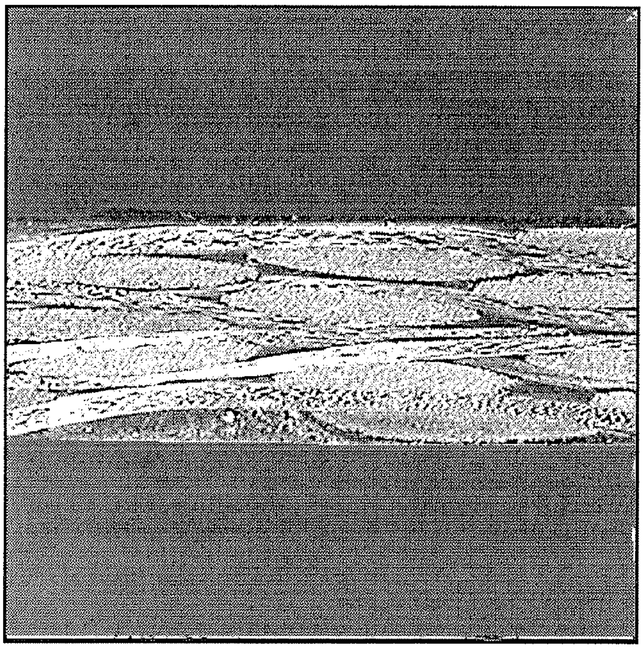

Figure 4. The inherent fibre waviness in a twill-weave fabric is shown by the crimp of the fibres running across the optical micrograph

Fibre distribution

The clustering of particles or fibres has been predicted by Guild *et al* [18-33] to increase the likelihood of early failure in highly-stressed situations. Failure in longitudinal compression has been related to regions containing no fibres [31]. The application of longitudinal compression generates transverse tension which tends to pull the interface apart. The magnitude of this transverse tension increases with increasing inter-fibre distance; that is it is significant around fibres bordering resin-rich regions. The importance of this result is highlighted by the existence of sufficient resin-rich areas to promote premature compressive failure even if the fibres are assumed to be distributed randomly [32].

Failure in transverse loading has also been predicted to be dependent on fibre arrangement, especially in materials reinforced with highly orthotropic fibres where the transverse modulus is lower than that of the matrix material [33]. In contrast to the result described above, stress concentrations in transverse loading were expected to be significant within closely packed regions of fibres, containing a high fibre volume fraction. Although these two results for the different loading conditions show dependence on two different microstructural parameters, namely the occurrence of resin-rich areas or the occurrence of closely packed regions of fibres, it should be noted that the occurrence of either type of region will inevitably produce the other type. The definition of fibre arrangement is a vital step in understanding the failure processes in unidirectional fibre composites.

Basford *et al* [34] have presented experimental compression and apparent interlaminar shear strength values for a series of fabrics with systematic variation of the reinforcement architecture. Both of the measured properties decreased with the increase in the large resin-rich areas due to increasing perturbation of the fibre distribution.

Matrix cure

The vast majority of fibre-reinforced plastics are manufactured using thermosetting resins (for example unsaturated polyester, phenolic or epoxides), rather than thermoplastics (for example polypropylene, nylon or saturated polyester). The thermosets are introduced to the fibres in a liquid state, and caused to solidify by the action of reactive chemicals and/or heat. This curing reaction transforms the short linear/branched polymer molecules into a single extended three-dimensional network, by cross-linking the polymer chains.

There are a finite number of positions at which cross-links may be formed. If the stoichiometric mix of components is incorrect, or if insufficient curing agent or insufficient thermal energy are supplied to the polymer, then the reaction may not proceed to completion. The presence of unreacted sites in the network will lower the glass transition temperature (the temperature at which there is a reversible change in an amorphous polymer from a viscous or rubbery state to a hard and relatively brittle state) of the network, and hence compromise the upper limit of temperature at which creep/fatigue loading will become important to the endurance life of the composite. An incomplete network structure will also permit easier attack by moisture or chemicals.

Marand *et al* [35] compared thermal and microwave curing of epoxy resin and found that, in the latter, rapid crosslinking created a molecular network which was rigid enough to trap unreacted functional groups, thus actually causing a lower degree of cure.

Chekanov *et al* [36] studied the influence of vitrification on defect formation during the isothermal cure of epoxy resins. The cure temperature was found to drastically affect shrinkage defect formation both in the rubbery and glassy states. Vitrification supresses defect formation when gelation occurs first or simultaneously, but promotes crack formation when it precedes gelation.

The curing reaction tends to proceed from liquid to gel to solid with an exponential realisation of mechanical properties. It is therefore difficult to determine the true end-point of the reaction. An effective method is the use of differential scanning calorimetry, but this is an expensive laboratory instrument, and is not truly non-destructive. More commonly, the material is tested by the Barcol hardness test [37], but this is notoriously difficult to undertake with repeatability, and the equipment is only suitable for testing flat plates. The Barcol impressor may also inflict surface damage on the component.

A secondary problem may be the input of excess energy in the curing process. The crosslinking reaction is usually exothermic and, particularly in thick sections or in situations where the heat generated is not easily dissipated, the thermal energy may heat the laminate beyond the degradation temperature of the polymer. This is often easily detected as it results in a darker colour in the cured part. It may additionally result in matrix cracking, fibre-matrix interfacial debonding and/or inter-layer delamination, which may be detected by the non-destructive test methods already applied to damage in these materials. Debonding [38] and delamination [39] damage have been reviewed elsewhere.

Voidage

Void nuclei may be formed during manufacture either by mechanical means (entrapped gas bubbles, broken fibres) or by homogeneous or heterogeneous nucleation. Once established, a void may change size through any of three effects:

a) Changes in vapour mass (solvents, condensation products) and the vapour transfer across the void/material interface.
b) Pressure changes inside the void due to temperature and pressure changes in the material.
c) Thermal expansion due to temperature gradients in the resin.

Models which take into account the first two of these effects, vapour transport and changes in temperature/pressure have been developed by Loos and Springer [40,41] and by Kardos *et al* [42-44].

Judd and Wright [45] reviewed 47 papers to make an appraisal of the effects of voids on the mechanical properties of composites. Regardless of resin type, fibre type or fibre surface treatment, "the interlaminar shear strength of a composite decreases by about 7% for each 1% of voids up to a total void content of about 4%". The decrease in other properties for the first one percent of voids is reported as high as 30% (flexural strength), 9% (torsional shear), 8% (impact strength) and 3% (tensile properties).

Ghiorse [46] found that in the range zero to 5%, each 1% increase in void content decreased the interlaminar shear strength and flexural strength of carbon fibre/epoxy composites by 10% and decreased the flexural modulus by 5%. The void contents were determined by multiple destructive methods: water buoyancy, density gradient, nitric acid matrix digestion and image analysis on two different commercial systems.

Stone and Clarke [47] correlated ultrasonic attenuation measurements on constant thickness panels of unidirectional CFRP with the void content. A simple bilinear relationship was postulated. At low void content (less than 1.5%) the shape of the void tends to be spherical with a diameter of 5-20 μm. At higher contents, the voids are cylindrical and the length can be an order of magnitude more than the diameter quoted above. Generally, the long dimension of a cylindrical void is oriented parallel to the fibre axis. Poor wetting of the filaments during manufacture will aggravate the problem of resin impregnation, increasing the difficulty of resin penetration between tightly packed bundles, and thus lead to failure to displace all the air.

An advisory document has been produced by the Royal Aerospace Establishment [48] to describe a procedure for the assessment of void content of a laminate by an ultrasonic scanning technique. The procedure is valid for single through-transmission using two probes, double through-transmission using a plate glass reflector and double-through-transmisssion using the back surface of the laminate when it is smooth.

Guo and Cawley [49] have recently shown that ultrasonic attenuation from the amplitude of the echo returning from the back wall of a carbon fibre/epoxy resin composite structure is a satisfactory technique for the measurement of porosity in circumstances where immersion of the structure is not possible. The results obtained were very similar to those obtained using the laboratory standard double through-thickness method.

Stringer [50] has shown that the way to minimise the proportion of voids in the vacuum bagging of polymer composites is to apply pressure during a specific window of resin viscosity. He has demonstrated that it is possible to produce aerospace quality composites by this method, without resorting to the use of an autoclave to apply additional consolidation pressure.

Wood and Bader [51, 52] have reported that increasing the pressure in the vacuum bag, whilst maintaining autoclave pressure outside the bag causes the air bubbles to dissolve into the resin matrix.

Summerscales and Fry [53] measured the Poisson's ratio of a high void-content satin-weave glass-fibre epoxy composite (Figure 5). The in-plane Poisson's ratio was found to decrease with increasing stress, until the incremental Poisson's ratio was around zero immediately prior to failure. The use of strain gauges as a non-destructive means of identifying the imminence of failure was proposed.

Inclusions

The contents of a laminate are not always only those items which are supposed to be included. For example, in the vacuum bagging/autoclave processes the fibres are usually pre-impregnated (prepregs) with partially cured resin and supplied on a backing film. The carrier for the prepreg should be removed from the reinforcement before lay-up, but the occasional piece may not be separated and then ends up within the laminate. It is normal practice to count the number of pieces of carrier remaining after lay-up as part of the quality system for manufacture. This problem is removed by the automation of the process, for example computer-controlled tape-laying.

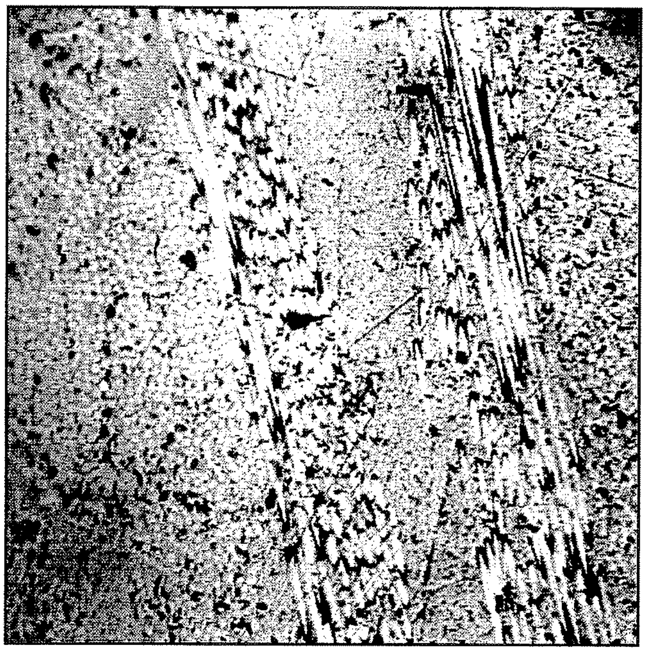

Figure 5. The dark areas in the optical micrograph are unwetted regions in a high void-content fabric

Other inclusions which have been reported include general debris from the factory, the operator's protective glove, the operator's lunch (sandwich) and an apocryphal tale exists of one filament-wound structure containing the operator who tried to make an adjustment without stopping the machine. In the latter case, the gentleman was rescued and lived to tell the tale.

Moisture

Considerable attention has been given in recent years to the effects of water absorption in fibre-reinforced plastic composites. Epoxy resins can take up several percent by weight of water. The equilibrium water content is controlled by the relative humidity and the rate of water absorption increases with increasing temperature. These effects are almost completely reversible, the water being removed by heating in a vacuum or in a dry gaseous atmosphere.

The absorbed water produces a volume increase in the resin and reduces the elastic moduli and glass transition temperature. Matrix cracks can be produced by non-uniform volume changes. The fibres constrain longitudinal expansion and hence volume changes are realised in the unreinforced directions (transverse to unidirectional fibres). These effects can offset the stresses produced by differential thermal contraction between the fibres and matrix during curing.

Browning [54] reported that the resin strength was reduced by an increase in moisture content, although the failure strain may be enhanced. The presence of moisture would thus be expected to have greatest effect on the matrix-dominated properties. For example, in unidirectional carbon/epoxy composites moisture does not impair the axial properties, but transverse and shear strengths and moduli are reduced.

Moisture intrusion caused by environmental exposure for extended periods is known to have an adverse effect on the structural integrity of polymer matrix composite materials [55]. Diffusion through the organic matrix leads to degradation of the polymer structure, and moisture migration along the fibre-matrix interface weakens the interfacial bond.

There is a requirement for non-destructive techniques to monitor the extent of moisture absorption and the severity of associated mechanical degradation. Summerscales [56] has presented a review of five techniques (electric, dielectric, infrared, nuclear magnetic resonance and positron annihilation) which have been used for the non-destructive measurement of moisture content in fibre-reinforced plastics. Portable electric/dielectric and infra-red instruments are commercially available. The other two techniques are expensive and are confined to the laboratory.

Conclusions

As fibre-reinforced plastic composites are increasingly adopted for primary structural applications, there is a greater need for non-destructive methods to confirm the structural integrity of the components. This paper has described those defects which are most likely to cause concern, together with the implications for non-destructive testing techniques. Non-destructive testing (NDT) for defects should be minimised by achieving manufacturing processes which are under control. After all, if the component is found to be inadequate then the process of rework/repair is unlikely to be economic. The use of factorial experiment design (FED) in process development, statistical process control (SPC) in production and quality systems (for example ISO 9000) in management should assist in realising this aim.

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