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In-Plane Testing of Thick Composites: A Review

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In-Plane Testing of Thick Composites: A Review

W R Broughton
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ABSTRACT

Recent developments have seen an expansion in the use of composite materials in structural applications involving thickness sections in excess of 20 mm, often complex in shape. The general perception has been to associate through-thickness properties with thick composites, whereas in fact an equally important issue relates to the measurement of in-plane properties and the effect of physical size of test specimens on measured data. Although extensive developmental work has been undertaken worldwide into test methods and design procedures for in-plane properties of thin laminates, there are no standard test methods available that provide guidance on testing of thick composite sections. The approach generally adopted has been to use existing standards, developed for testing small laboratory-scale specimens, with non-standard (i.e. larger) specimen geometries. A major concern relates to whether data generated from standards for relatively thin specimens are equivalent to or representative of thick section mechanical behaviour.

This report reviews key issues relating to mechanical testing of thick composite sections, highlighting issues that need to be resolved or better understood in order for these materials to fulfil their full market potential. The report covers tension, compression, shear, flexure and multi-axial loading conditions, environmental, impact and fatigue resistance, and issues relating to manufacture and inspection of thick laminates. Recommendations are provided as to future work required for test method development for thick composite laminates.

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1 INTRODUCTION

The complexity and physical magnitude of many engineering structures for use in transportation (road, sea and air), civil infrastructure (building and bridges), nuclear and chemical plant, and off-shore applications requires versatile materials that can be tailored to meet demanding service conditions [1]. Polymer matrix composites (PMCs) offer the user/designer the flexibility and range of mechanical and physical properties to meet these requirements. These materials have tended to be used for their weight reduction and corrosion resistance qualities. The use of PMCs has primarily involved thin membrane structures, but in recent years their use in large, thick structures (i.e. thickness in excess of 20 mm), often complex in shape, has expanded. The wide range of processing routes, fibre types and formats (two- and three-dimensional architecture), and resin matrices provides the means of producing thick complex engineering structures suitable for use in safety critical applications and hostile environments, such as bridges, wind turbines, commercial aircraft and off-shore drilling platforms (see Figure 1).

Figure 1: Off-shore drilling platform.

The general approach has been to concentrate on through-thickness properties of thick composites, whereas in fact equally important issues relate to the measurement of in-plane (intralaminar) properties and the effect of physical size of test specimens on measured data. Although extensive developmental work has been undertaken worldwide into test methods [2] and design procedures for in-plane properties of thin laminates, there are no standard test methods available that provide guidance on testing of thick composite sections. The approach generally adopted has been to use existing standards, developed for testing small laboratory-scale specimens, with non-standard (i.e. larger) specimen geometries.

A major concern relates to whether data generated from standards for relatively thin specimens are equivalent to or representative of thick section mechanical behaviour. Measurement of in-plane properties of thick sections can pose considerable problems from a testing perspective unless scaling effects are understood. Thick laminated sections also pose particular problems in relation to mechanical testing, as the large size of the test pieces often requires high load capacity testing facilities, which are not readily accessible for most organisations. The lack of suitable test methods has resulted in a shortage of reliable engineering data for large structural applications.

This report reviews issues relating to testing of thick composites that need to be resolved, or better understood in order for these materials to fulfil their full market potential. It forms part of the DTI funded part of the Measurements for Materials Characterisation project SM07 "**Validated Measurement Techniques for Improved Design of Large Advanced Composite Structures Subjected to Multi-Axial Loading**". The report evaluates mechanical test methods for characterising in-plane elastic and strength properties in relation to their suitability for testing thick-sections. The report consists of nine sections including the Introduction (Section 1). Tension, compression, shear and flexure testing are covered in Sections 2 to 5. Section 6 considers multi-axial testing methods. Durability (i.e. environmental and fatigue) and impact resistance are considered in Section 7, and manufacturing and inspection issues are covered in Section 8. Conclusions with recommendations for future development work are presented in Section 9.

2 TENSION

Conventional tensile test procedures tend to be used for measuring the stiffness and strength properties of thin composite sections. ISO 527 (Parts 4 and 5) [3-4] specifies methods and specimen geometries for determining the tensile properties for continuous aligned, random mat, woven fabric and multidirectional laminates. ISO 527-4 allows for 10 mm thick isotropic and orthotropic laminates. There is no general consensus within the composite industry as to the preferred specimen width or length.

An apparent scale effect has been observed by a number of researchers for unidirectional, angle-ply, cross-ply and quasi-isotropic lay-ups [5-8]. In these studies, failure loads and strains are shown to decrease with increasing laminate thickness with laminate stiffness appearing insensitive to changes in laminate thickness. These studies however, tend to overlook the effects of manufacturing technique and conditions, loading arrangement and the ply stacking sequence of the laminate. A study, conducted by Sutherland et al [9] concluded that changes in specimen thickness had no substantial effect on tensile strength or stiffness of unidirectional carbon and glass fibre-reinforced epoxy systems. The authors observed [10] that the tensile strength of woven fabric laminates tended to decrease with increasing laminate thickness. This reduction was attributed to the effects of scale of production rather than a genuine size effect (i.e. lower quality specimens are obtained when a thicker laminate is produced). It was concluded that manufacturing variations were the main reason for the observed thickness effect and that manufacturing processes for coupon specimens should closely mirror those used to produce the composite structure.

The presence of interlaminar tensile (peel) and shear stresses at the free edges of a laminate may be sufficient to cause local matrix and interfacial cracking, and edge delamination. Frequently, these stresses and strains induce failure in the laminate. The magnitudes of the stress gradients present at the free edges of a laminate are dependent on the laminate stacking sequence and the level of sub-laminate scaling. Sub-laminates consist of one or more layers, **n**, of the same fibre orientation stacked together. The tensile strength of $[0°_n/90°_n]_s$ cross-ply laminates (**s** denotes symmetry) can be expected to decrease as a result of increasing n the number of plies within the 0° and 90° sub-laminates (i.e. thicker sub-laminates) [11-12]. This is because the tensile stresses at the free edges of the laminate increase with increasing sub-laminate ply thickness.

Tensile tests conducted by Niklewicz and Sims [12] on a quasi-isotropic carbon/epoxy laminate containing "distributed" plies $[+45/-45/0/90]_{ns}$ with **n** = 1, 2, 3 and 4 showed no evidence of a size (scaling) effect. The tensile strength remained constant as **n** was increased - see Table 1. Tests conducted by the authors on a quasi-isotropic laminate with "blocked" plies $[+45_n/-45_n/0_n/90_n]_s$ showed a reduction in tensile strength with an increase in **n** (Table 1). All the tests were performed at the same strain rate. In all cases, failure initiation was observed to occur in the surface plies near the free edge of the laminate followed by ply failure within the laminate. Failures tended to occur near the grip for the **n** ≥ 2 "blocked" specimens, with the predominant failure initiating at the grip. This indicates that blocking the plies together reduces the damage tolerance of the laminate making it more susceptible to stress concentrations initiated by the end tabs.

Table 1: Tensile Strengths for Quasi-Isotropic Carbon/Epoxy Laminates [12]

n	Width x Thickness (mm)	Gauge Length (mm)	Tensile Strength (MPa)	
			$[+45/-45/0/90]_{ns}$	$[+45_n/-45_n/0_n/90_n]_s$
1	10 x 1	75	552	552
2	20 x 2	150	571	499
3	30 x 3	225	564	496
4	40 x 4	300	548	474

Niklewicz and Sims [12], also conducted tensile tests on two generic angle-ply ±45° carbon/epoxy laminates with "blocked" plies $[+45_n/-45_n/45_n/-45_n]_s$ and "distributed" plies $[+45/-45/+45/-45]_{ns}$ and **n** = 1, 2, 3, 4 (see Table 2). For "distributed" plies $[+45/-45/+45/-45]_{ns}$, there was an increase in tensile strength with increasing specimen size, whereas for the "blocked" specimens $[+45_n/-45_n/45_n/-45_n]_s$ the tensile strength decreases. The distributed specimens exhibited ductile stress-strain behaviour, while the blocked exhibited brittle, low strain-to-failure behaviour. For the **n** = 4 specimens, the strain to failure of the blocked plies was around 1%, while for the distributed plies this was approximately 12% with the strength differing by a factor of 2.5.

Table 2: Tensile Strengths for ±45° Carbon/Epoxy Laminates [12]

n	Width x Thickness (mm)	Gauge Length (mm)	Tensile Strength (MPa)	
			$[+45/-45/+45/-45]_{ns}$	$[+45_n/-45_n/45_n/-45_n]_s$
1	10 x 1	75	194	194
2	20 x 2	150	222	146
3	30 x 3	225	253	122
4	40 x 4	300	271	108

In all cases, failure initiation was observed to occur in the surface plies near the free edge of the laminate followed by cracking in the mid-plane where the plies are blocked due to symmetry. Classic laminate theory assumes that all the plies are at the same strain level, however the top (free) surface of the outermost ply in an angle-ply laminate is not as constrained as the inner surface, which is bonded to an adjacent 45° ply. Pipes and Daniel [13] showed, using a simple strain transformation approach, that shear strains can be transformed into significant normal strains, which act transverse to the fibre direction. Since the free surface material is unconstrained in the direction transverse to the fibres, cracking occurs, and therefore the first ply failure that occurs on the surface is a normal tensile fracture rather than a shear mode fracture. The mid-plane of the laminate where -45° plies are blocked is subjected to a reduced constraint when compared to the inter-ply region, where the constraint of the neighbouring +45° ply is at a maximum.

Large specimens are manifestly more difficult to test than small laboratory-scale coupon specimens since larger lateral loads are required to grip the larger specimens to prevent slippage. Stress concentrations in the vicinity of the end tabs, resulting from the gripping fixture, can be expected to increase with laminate thickness, leading to an apparent reduction in tensile strength. Failure commonly occurs in the vicinity of the end tabs where the end tabs terminate and gauge-section begins. For the above reasons, tensile strength dependence on laminate thickness cannot be accurately modelled using simple statistical models based on Weibull distributions of flaw densities and the weakest link approach or by fracture mechanics models.

Niklewicz and Sims [12], using finite element analysis (FEA) showed that the reduction in tensile strength with increasing thickness observed for unidirectional laminates could be attributed to tab induced stress concentrations. The thicker the laminate the higher the stress concentration, and hence the lower the "apparent" tensile strength. Re-design of the end tab as specified in ISO 527 would need to be considered in order to transfer the larger load through the specimen/tab/grip interfaces (i.e. stress transfer is related to surface area and not volume of specimen in the clamps). One suggested solution based on FEA results is to use a reverse tapered tab with a 45° fillet [12] as shown in Figure 2. Subsequent research conducted at NPL [14] has shown that the reverse tapered end tab has an adverse effect in tension.

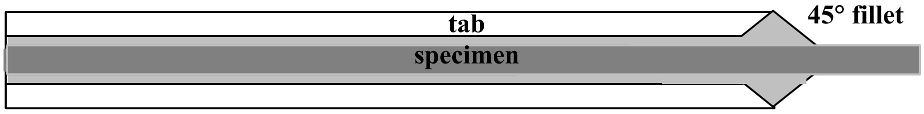


Figure 2: Reverse tapered end tab with 45° fillet.

O'Brien and Salpekar [15] conducted a series of transverse tensile tests on unidirectional carbon/epoxy laminate specimens of different widths and thickness. Widths varied from 12.7 mm to 50.8 mm, and thickness from 4 to 64 ply. Specimen length was kept constant (178 mm). No end tabs were used. Instead thin sheets of cellulose acetate were used to protect the specimen surfaces. Gripping pressure was kept low, but sufficient to prevent slippage. The results for different widths and thicknesses, but the same volume, were pooled together. Test data only included those specimens where failure occurred within the gauge-section. The average strengths were shown to linearly decrease with increasing volume when plotted on a log-log scale. It was suggested that local matrix microcracks and/or fibre-matrix interfacial disbonds were most probably responsible for the decrease in strength with volume. Marginal variations in strength with width were attributed to the criticality of flaws near the specimen edge.

Tensile properties are also strain rate dependent. The tensile strength of glass/epoxy laminates is particularly sensitive to variations in strain rate. It is not always possible to scale specimens to ensure constant strain rate with specimen size. Another factor is the ability to achieve the same consolidation, removal of voids or cure uniformity for thick material compared with thin material. The need to avoid over-heating due to the exothermic cure is well recognised. The detrimental effect of residual stresses on material performance can be expected to be exacerbated thick composite sections (see Section 8).

3 COMPRESSION

There is no in-plane compressive test method specifically developed for loading sections in excess of 10 mm in thickness. BS EN ISO 14126 [16] specifies methods and specimen geometries suitable for determining the compressive properties of continuous aligned, random mat, woven fabric and multidirectional laminates. The standard allows for 10 mm thick material. Particle-filled and short fibre (less than 1 mm in length) reinforced plastics are covered by ISO 604 [17]. Figure 3 shows an end-loading compression fixture that has been modified at NPL to accommodate specimens as wide as 36 mm.

Figure 3: End-loading compression fixture with specimen.

There are three main methods for loading a standard test specimen [14]:

- **Pure shear loading** in which the compressive load is applied to the specimen by shear through the end-tabs,
- **Pure end loading** where the compressive load is applied directly and solely to the ends of the specimen, and
- **Combination shear and end loading** in which both shear and end loads are applied to the specimen.

There are four commonly used loading fixtures for compression testing: IITRI, Celanese, end-loading and hydraulic wedge action grips [14]. However, there is no consensus on the best loading method for generating compression within a specimen due to the lack of supporting experimental evidence. The compression fixtures are generally used to load thin sections (typically 2 mm thick). Research into the applicability of using compression fixtures for testing thick samples has been limited.

The longitudinal modulus and in-plane Poisson's ratios for composite materials are insensitive to specimen thickness, however experimental data has shown that compressive strength decreases with increasing thickness for unidirectional glass and carbon fibre-reinforced laminates [18-20]. Failure commonly occurs in the vicinity of the end tabs. Camponeschi attributes this reduction in strength to compression fixture restraint effects, which according to the author can be explained in terms of through-thickness Poisson's expansion that occurs in thick laminates (i.e. difference between the expansion of the gauge-section and the through-thickness expansion within the fixture grips, known as the effective gauge-section expansion).

This expansion results in fibre curvature where the specimen exits the clamping blocks or gripping fixture. The effective gauge-section expansion increases with increasing thickness, and hence fibre curvature also increases. The larger fibre curvature results in a reduction in buckling forces, and hence the lower compressive strengths observed for thick composite sections. If the end effects are minimised then variations in specimen thickness should have minimal effect on the measured compressive properties.

Compression tests conducted at NPL using an end-loading fixture modified to accommodate a broad range of specimen thicknesses showed that clamping loads are critical in determining the failure mode of the samples (see Figure 3) [14]. Different failure modes were obtained for smooth and serrated loading block faces with higher compressive strength values obtained using serrated clamping surfaces. Serrating the clamping faces in the end-loading rig seems to limit damage occurring within the gripped region, effectively minimising the number of premature failures in this zone.

Applying specific torque levels does not guarantee reliable or repeatable clamping loads, due to factors such as the material used for the bolt and whether the thread is clean or has been greased, etc. It is important that all bolts are kept in the same condition for each test conducted, either fully clean or fully greased to minimise frictional contributions to the measured torque, with each bolt being tightened progressively. The lateral forces applied to the test specimen need to be constant for different specimen thicknesses.

It is generally recognised that fibre misalignment (i.e. misalignment of fibres from the loading axis) plays a key role in failure initiation under compression loading. Fibre misalignment can cause shear stresses resulting in shear strains, and subsequent further misalignment of fibres. At a critical axial strain level, the fibres bend and then fracture as a result of combined compression and bending at the points of maximum fibre curvature. The micro-buckle then spreads across the specimen width by a process of successive buckling and fracture along the fibres and by the transfer of load to adjacent fibres, which have already buckled by the axial strain. A misalignment angle of a few degrees is sufficient to cause a large reduction in compressive strength. Most laminates can be expected to contain regions of fibre misalignment (or waviness) with varying degrees of severity. These regions can be considered as defects (or flaws). In principle, failure should initiate in the worst regions of fibre misalignment with thicker laminates more likely to contain large flaws. Although localised fibre misalignment contributes to a reduction in compressive strength, the effect is less critical than large-scale misalignments [6]. Hence, it is important that when evaluating size effects on compression strength that the material used for producing specimens is identical.

The question arises as to whether it is possible to obtain compression data comparable with thick sections using standard 2 mm thick specimens machined from thick sections. Experimental results from a study at NPL [14] showed that the compression data was comparable provided care was taken to keep the correct fibre alignment and ensuring laminate damage was minimised. As with tensile tests, end tab geometry plays a significant role. The 45° reverse taper has been shown to produce repeatable higher compression strengths when compared to the ISO standard recommended square shoulder [16]. The strength of a unidirectional glass/epoxy laminate increased by 35% when using 45° reverse taper end tabs. A similar increase in strength has been observed in previous unpublished work conducted at NPL.

4 SHEAR

Considerable experimental and analytical effort has been expended on the development of in-plane shear test methods. One of the principal difficulties has been to produce a uniform state of pure shear in specimens. The difficulty of inducing pure shear increases with increasing anisotropy and inhomogeneity of the material. As these characteristics increase, the complex stress states arising at or near the loading zones become more dominant, particularly for continuous unidirectional laminates containing high modulus and high strength fibres. In these materials, it is difficult to obtain adequate regions of uniform shear stress free of extraneous stress components within the specimen, even if the production of the specimen and test alignment are perfect. In addition, extraneous tensile and compressive stress components have a marked effect on the shear strength of these materials. Tensile stresses induce premature failure, whereas compressive stresses delay the onset of failure. The difficulties in producing a state of pure shear in composite specimens are compounded when the material thickness is increased.

The general approach has been to extrapolate data from tests conducted on thin laminates (i.e. 2 to 5 mm in thickness) to thick structures. The most commonly used test methods for the determination of shear properties are as follows [21]:

- Uniaxial tension of a ±45° laminate (BS EN ISO 14129 [22])
- 10° off-axis laminate;
- Two-rail and three-rail shear tests (ASTM D 4255 [23])
- V-notched beam (or Iosipescu) test (ASTM D 5379 [24])
- Plate twist test (BS EN ISO 15310 [25])
- Torsion of a thin-walled circular tube (ASTM D 5448 [26])

The effects of laminate thickness associated with tensile testing of composite laminates apply to both the ±45° and 10° off-axis shear methods, and to the two- and three-rail shear tests (see Section 2). It has been reported that strength measured using the ±45° tension test is dependent on the number of layers, or shearing interfaces, in the specimen [27].

Figure 4: Plate twist test.

The plate twist test (see Figure 4), which was initially developed to measure the shear modulus of plywood (ASTM D 3044 [28]), has proved satisfactory for measuring shear moduli ranging from 0.29 GPa (chopped glass-fibre reinforced polyurethane) to 88.2 GPa (steel). The test method is unsuitable for determining in-plane shear strength. BS EN ISO 15310 recommends a standard plate specimen 150 mm x 150 mm and a length to thickness ratio ≥ 35. There is insufficient data to assess the suitability of the test method for use with thick-sections, however from a testing perspective there is a practical limit as to the maximum size of specimen that can be tested (i.e. approximately 10 mm thick panels).

In the case of the V-notched beam geometry (Figure 5), the stress state induced in the specimen can be expected to become less uniform with increasing laminate thickness with compressive stresses increasing as the laminate thickness is increased. Localised crushing may occur at the loading points due to high compressive stresses. The measured in-plane shear modulus and shear strength tend to be lower for thick sections (> 5 mm thick). The ASTM test fixture is not designed for testing thick material (> 10 mm thick) and has been observed to deform at high loads.

Figure 5: V-notched beam test.

The asymmetrical four-point bend (AFPB) test [29-31], which utilises a slightly modified loading scheme to the V-notched beam shear test, may be more suitable for testing thick sections. The modification allows for easy specimen loading and can be adapted to accommodate thick sections. The fixture recommended by its author (see [29]) includes side restraints.

From an applied mechanics viewpoint, torsion of thin-walled circular tubes is the most desirable method for shear characterisation. Thick-walled cylindrical specimens however, can be tested provided the through-thickness shear gradient is negligible. To ensure a uniform shear stress field, the specimen should have a gauge-length to diameter (**L/D**) ratio > 1 and a wall thickness to diameter (**h/D**) ratio of 0.02, or less (i.e. h = 20 mm and **D** = 1 m) [21]. Testing thick structures requires considerably large specimens and suitable test equipment to accommodate these structures. Increasing the wall thickness, whilst maintaining all other dimensions constant, results in a significant stress gradient across the tube wall, and a reduction in shear strength. Cylindrical wound tubes are suitable for characterising static, creep and fatigue behaviour of composite materials in tension, compression and shear. The test geometry is also suitable for assessing mixed-mode behaviour (i.e. shear + tension and shear + compression).

5 FLEXURE

BS EN ISO 14125 [32] specifies methods for determining the flexural properties of fibre-reinforced plastic composites under three-point and four-point bend loading. The method is suitable for use with all fibre types and fibre formats, and allows for non-standard test geometries. It is possible to use these methods for testing of 25 to 40 mm thick laminated beams using the scaling rules given in the standard. The limitation being the load capacity and physical size of the test machine required for testing large structures. The recommended spans for 25 mm and 40 mm thick GRP beams loaded in three-point bend are 500 mm and 800 mm, respectively.

Thick composite beams frequently fail as a result of a combination of high shear and transfibrile compression gradients, present in the upper portion of the orthotropic beam near concentrated loads. Wisnom [6, 33] observed a change in failure mode between thin and thick unidirectional carbon-epoxy sections in four-point bending. Tension tended to be the dominant failure mode for thin sections, whilst thicker sections failed in compression with failure generally occurring in the vicinity of the inner loading points. The possibility of compressive failure occurring at the loading points can be minimised (not necessarily avoided) by using steel or composite shims placed directly under the central loading rollers to distribute the loads more uniformly. The roller diameter should be commensurate with the specimen thickness (i.e. roller diameter to increase with thickness). Strain rate should also increase with volume to ensure equivalent strain rates are applied to both thin and thick sections.

6 MULTI-AXIAL TESTING

This section will examine multi-axial testing methods that can be used for characterising thick composite materials. Two- and three-dimensional methods will be considered. These tests are important in the development of reliable three-dimensional numerical (finite element analysis) modelling, design and analysis, and failure theories for thick composite structures. Multi-axial testing (see Figure 6), which can be used to identify actual material strengths and failure mechanisms under representative service conditions, is often used simply on the grounds that there is insufficient confidence in three-dimensional failure analysis. There are two distinctively different methods of inducing multi-axial loads/displacements in composite materials (see below) [34].

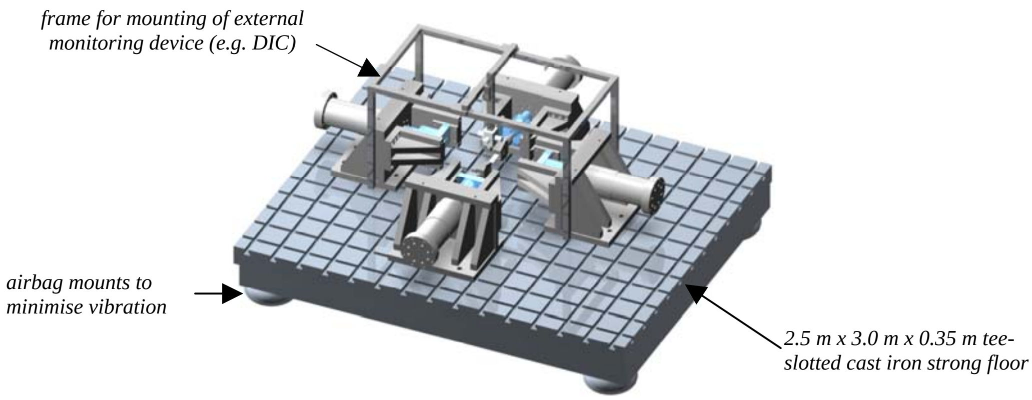
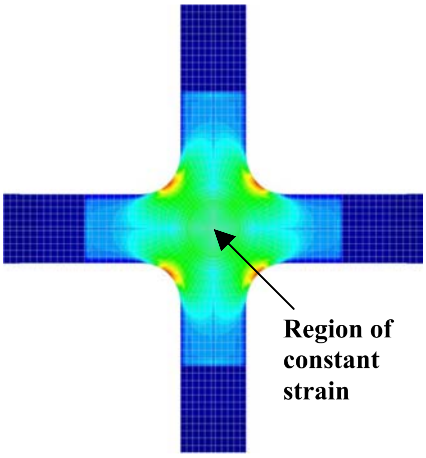


Figure 6: Schematic of NPL's multi-axial strong floor test facility.

6.1 PLANAR COUPON TEST METHODS

Testing consists of applying loads/displacements along the primary, mutually orthogonal coordinate axes of lineal test specimens (i.e. rectangular plates, cubes or two- and three-dimensional cruciforms (see Figure 7)). Specimens are loaded using either planar biaxial or triaxial loading frames (see Figure 6). Servo-hydraulic actuators or screw-driven test frames are used to apply load(s) to the test specimen. Special end tabs and support fixtures are required for loading and constraining the test specimens. The shape and dimensions of the test specimen need to be carefully designed to ensure that a uniform biaxial strain field is induced within the specimen gauge-section. Both biaxial and triaxial test machines require sophisticated control systems for controlling and monitoring loads and displacements of the test specimen throughout the duration of a test. These controls are used to avoid unwanted eccentric loading conditions, thus ensuring inappropriate failure mechanisms or failures outside the instrumented gauge-section do not occur. Extreme care is required in order to prevent undesirable end and edge effects and stress concentrators. Failures are assumed to be valid if failure initiates in the gauge section and is in keeping with the loading ratio. That is, for specimens tested under tension/tension, failures should be tensile dominated in nature.



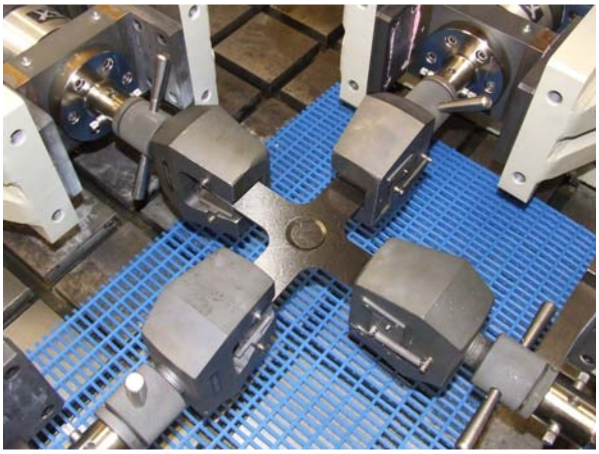


Figure 7: Biaxial testing of cruciform specimen.

6.2 CYLINDRICAL TEST SPECIMENS

This method consists of loading cylindrical specimens either in tension, compression and/or shear. There are numerous biaxial test facilities capable of applying combined axial (tension or compression) and torsional twisting loads (about the longitudinal axis) to the cylindrical test specimens. Triaxial machines, which are similar to biaxial test machines, have the additional facility of being able to induce a pressure gradient across the cylindrical wall of the test specimen by applying either external, internal or both external and internal pressure. Triaxial test machines are well suited for testing filament wound structures, such as pressure vessels and pipes. Cylindrical specimens are free of edge effects due to the hoop continuity of the test geometry. However, end effects and the potential problem of structural instability (or buckling) can have a significant effect on test results and failure mechanisms. Specimen design and loading fixtures need special consideration in order to eliminate the adverse effects of these two factors.

A significant amount of work has been directed towards developing test methods and standards for assessing the performance of pressure vessels and pipe sections (see [35-37]). ASTM D 2992 [38] specifies procedures for determining the long-term cyclic and static hydrostatic strength of a composite pipe. According to Ellyin and Marten [38], ASTM D 2922 specifies a worst-case scenario rather than typical conditions experienced by composite pipes for example in oil fields.

7 ENVIRONMENTAL, IMPACT AND FATIGUE RESISTANCE

Thick sections take a longer time to degrade under hostile environments and exhibit greater impact resistance in comparison to thin sections. The large timescales involved for thick composites exposed to moisture to reach equilibrium, even under accelerated testing conditions, can be impractical, and hence the use of relatively thin specimens to determine the "through-the-thickness" moisture diffusion coefficient. The question arises as to the reliability or relevance of using thin coupon data for predicting long-term behaviour of large thick structures. This section examines the issues of environmental durability, fatigue and impact resistance for thick composite sections.

7.1 ENVIRONMENTAL DURABILITY

7.1.1 Coupon Tests

Prolonged, or even short term, exposure to aqueous environments can cause irreversible changes in the chemical and physical properties of fibre-reinforced polymer composites, particularly at elevated temperatures [39-40]. These changes often compromise the load carrying properties of the material with the level of degradation increasing as moisture content increases. Reductions in stiffness and strength, and changes in thermo-mechanical behaviour can often be linked directly to the amount of moisture absorbed. The combined effect of heat and humidity can induce thermal blistering in thick laminate sections. Resistance to thermal blistering is a function of the moisture content; heat up rate and the thickness of the part.

A review of test methodologies and standards [41] revealed that the general approach to accelerated ageing of fibre-reinforced polymer composites is to expose laminated structures to severe test conditions, often well in excess of actual service conditions. In hot/humid environments (e.g. 70°C and 85% relative humidity (RH)) it may take 10-15 years for a thick composite structure to reach a state of moisture equilibrium or saturation. This condition, however, is even more difficult to achieve under milder and more realistic conditions than those employed to accelerate moisture conditioning. The issue arises as to the applicability of thin coupon durability data for predicting environmental degradation of thick composite sections.

The two main types of basic moisture conditioning are:

- Fixed conditioning, where a test specimen is exposed to a conditioning environment for a specified time; and
- Equilibrium conditioning, where a specimen is exposed until the material reaches equilibrium with the conditioning environment.

The first technique, which is routinely employed for screening purposes, results in non-uniform moisture distribution through the thickness of the test specimen (particularly thick-sections). In principle, test data obtained from specimens conditioned in this manner are only considered suitable for comparing different batches of the same material or for quality control tests. This approach, however, is widely used for generating engineering data. Ideally, comparative studies of water absorption properties of materials should be carried out only using the equilibrium moisture content of polymeric materials exposed to identical conditions. Comparisons between composite systems with different moisture absorption characteristics are possible if the materials are conditioned to equilibrium. The thicker the material the longer the time required to reach equilibrium, hence the use of relatively thin specimens to determine the "through-the-thickness" moisture diffusion coefficient. The large timescales involved, even under accelerated testing conditions, can make this approach impractical.

The International standard BS EN ISO 62 [42] describes a procedure for determining the moisture absorption properties and/or diffusion coefficients in the "through-the-thickness" direction of flat and curved solid plastics. BS EN ISO 62 is suitable for use with polymer and composite specimens. The method can be applied to vapour exposure and liquid immersion. A major disadvantage with many tests is that the test piece is conditioned unstressed in the required environment and then tested. Failure to account for the combined effect of stress and environment will frequently result in an overestimation. This is particularly pertinent to flexural loading conditions where highly stressed surfaces are vulnerable to attack. Standard methods, therefore, need to have the widest possible applicability and represent actual service conditions.

The CEN (Comité Européen de Normalisation) working group on engineering use of pultruded composites has initiated a study item to identify the minimum testing necessary to account for the effect of both vapours (e.g. moisture) and liquid (e.g. chemicals and water) environments. Methods for producing equilibrium saturation are now currently being discussed, but there is a strong industrial demand for fixed exposure times that are more applicable to industrial timescales and costs. Difficulties arise in selecting suitable exposure times for all materials, due to the time dependence on thickness and material. A recent development has been the CEN standard for underground glass fibre-reinforced plastic (GRP) tanks for the non-pressurized storage of liquid petroleum based fuels. The standard accounts for exposure to both the liquid fuel and the vapour.

7.1.2 Product Testing

Combined stress and environmental conditions are used in some product test methods. ASTM D 3681 [43] and BS 5480 (Part 2) [44] are used by industry to determine the chemical resistance of glass fibre-reinforced pipes. Testing consists of exposing the interior of a pipe section to a corrosive solution while the pipe is subjected to a compressive deflection until the structure fails (see Figure 8). The test is carried out at several deflections and the time-to-failure is measured for each test. The long-term chemical resistance of the pipe is obtained by extrapolating to 50 years the log-log linear regression line for the initial strain level. This test is appropriate to structures buried underground, such as sewer pipes or gasoline tanks, which will experience a constant strain throughout the life of the structure.

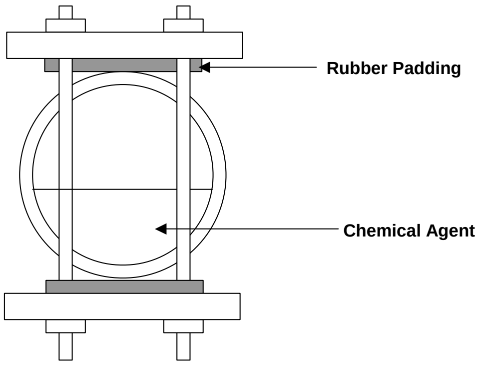


Figure 8: Strain-corrosion test apparatus.

A self-stressing fixture can be used to apply a constant displacement. Alternatively, a constant load, often in the form of a dead weight, is applied to the pipe section and the creep behaviour is monitored. Time-to-failure is plotted either as a function of stress or as a function of initial strain. This test is appropriate to above ground pipes. It is quite common for creep tests under adverse environmental conditions to be carried out using standard test coupons (i.e. flexure and tension coupons). Figure 9 shows a self-stressing four-point flexure test fixture with GRP specimens.

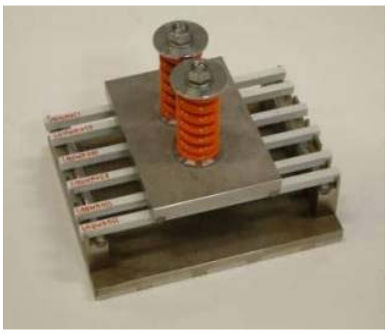


Figure 9: Self-stressing four-point flexure test fixture with GRP specimens.

7.1.3 Design/Analysis

It should be possible to characterise environmental (i.e. moisture) ageing effects, although roughly, on the mechanical properties of thick composite structures. The approach described below could be implemented in FEA and non-FEA software packages (e.g. CoDA).

Step 1 Determine tensile, compressive and shear elastic and strength properties for different levels of moisture content/glass-transition temperatures.

Step 2 Determine the moisture absorption kinetics and equilibrium (i.e. diffusion coefficients, $\mathbf{D_{xx}}$, $\mathbf{D_{yy}}$ and $\mathbf{D_{zz}}$, and saturation weight gain $\mathbf{M_{\infty}}$) by absorption tests on coupon specimens in a controlled environment.

Step 3 Determine the time-sequential moisture distribution in the composite structure.

Step 4 Determine the time-sequential distribution of mechanical properties with moisture content of the composite structure from Steps 1 to 3.

Step 5 Determine time-sequential stress and strain distributions with applied load within the composite structure.

Step 6 Apply suitable failure criteria to each point within the structural element to determine failure onset and progressive failure (i.e. ply by ply).

Semi-empirical formulations could possibly be used to relate mechanical property degradation with moisture content [40, 45]. This would reduce the amount of testing required for generating design data. Future work should examine, through the use of case studies, the feasibility of implementing the approach as mentioned above. If successful, this approach could be used for a wide range of chemical environments.

7.2 IMPACT RESISTANCE

Increasing laminate thickness tends to improve damage resistance. Similarly, impact resistance of sandwich structures is generally improved by using thicker or denser core materials. The efficacy of using thick composite in providing impact protection and for producing primary load-bearing structures has led the Ministry of Defence to announce that the next generation of armoured personnel vehicles and tanks will consist primarily of composite materials. Previous work has shown that residual compressive strength and delamination propagation of impact damaged panels was insensitive to panel thickness over the range of 5 mm to 25 mm. The reason being that the flexural deformation is generally negligible compared with through-thickness shear deformation for thick laminates. A thick laminate behaves as a semi-infinite body [46]. Localised damage in the form of fibre breakage and crushing can be expected to occur within the vicinity of impact. For carbon fibre-reinforced laminates, the threshold force required to initiate impact damage needs to be increased by a factor of approximately 3 each time the laminate thickness is doubled. Increasing the laminate thickness from 2 mm to 60 mm (typical armour plating thickness) would result in a 164-fold increase in the threshold force.

It is recommended that future work focuses on impact resistance of non-aerospace materials (e.g. pressure vessels), curved and joined structures, and defect criticality under uniaxial and multi-axial static, fatigue and creep loading conditions. Defect criticality should take into account the presence of stress concentrations (e.g. cut-outs, ply drops and bond ends) and environmental effects. Damage characterisation/modelling is still far from satisfactory, particularly for non-aerospace materials. From a design perspective, there is a need to establish mathematical relationships between impact force/energy and residual mechanical properties and resultant damage. It may be possible to use a fracture mechanics approach to predict impact resistance.

7.3 FATIGUE RESISTANCE

Most mechanical test methods can potentially be used in cyclic loading, although there are a number of inherent problems associated with load transfer and measurement of property changes (i.e. stiffness) throughout the duration of the test. Software packages exist that allow for dynamic measurement of stiffness and damping factor at selected intervals without the need to stop the test. The main requirement for fatigue testing using these methods is that the damage mechanisms and failure modes are essentially identical to those observed for static tests.

There is a concerted effort to develop ISO standards for fatigue, compatible with static test methods. ISO 13003 [46] defines the general principles applicable to fatigue tests for constant force, strain and displacement amplitude loading conditions at constant frequency.

Test mode, specimen dimensions and applicable calibrations are the same as those obtained from the equivalent test mode under static or monotonic loading conditions. Fatigue tests are normally undertaken at the highest frequency possible in order to minimise the duration of the tests (i.e. accelerated testing). Restrictions on test frequency can arise from test equipment limitations (response time), time-dependent processes and autogenous (self-generated) heating. Autogenous heating, which increases with increasing load and frequency, can adversely affect the fatigue performance of the composite. Test frequencies of the order of 10–30 Hz can result in a substantial increase in the mean surface temperature (up to 150 °C, or greater). The upper frequency limit will be dependent upon the thermal conduction of the fibre/matrix system, mode of loading and specimen size, and the number of interfaces of dissimilar fibre orientation within the laminate. The ability to dissipate heat can be expected to diminish with laminate thickness, and hence the test frequency will need to be reduced in accordance with the volume of material. The effect of autogenous heating is more marked in resin-dominated laminates as the associated strains are larger in comparison with fibre-dominated laminates. Friction between grips or supports and the test specimen can lead to elevated temperatures and fretting in these regions, which can result in premature failure.

8 MANUFACTURE AND INSPECTION OF THICK LAMINATES

Fabrication of thick composite laminates (20 mm or greater) is often difficult, with process induced (i.e. residual) stresses becoming increasingly important as the thickness is increased. Residual stresses can have a significant effect on the engineering properties of laminated structures by inducing warpage, fibre buckling, matrix microcracking and delaminations. These stresses arise from resin chemical shrinkage, as a result of curing and differences in thermal contraction between adjacent plies on cooling the laminate from the cure temperature. The net effect is that the strength properties of the laminate are diminished. Heat generation caused by exothermic chemical reaction of the matrix can result in material degradation. If dissipation of liberated heat through thermal conduction is slow, then the internal temperature may be elevated to levels that induce irreversible thermal damage. The risk of heat damage to the matrix increases with laminate thickness. A second concern relates to the complex temperature and degree of cure gradients that develop in thick-sections during the curing process. These gradients may induce a non-uniform state of cure through the laminate thickness, which can result in poor laminate consolidation, leading to undesirable fibre volume fractions and entrapped volatiles or voids. Good wet out and consolidation are more difficult to achieve with thermoplastic composites.

There is a major drive to develop on-line (real-time) cure monitoring techniques and predictive models that are capable of providing reliable information on the physical state of the composite material in the process environment. Controlling the state of cure is critical to ensuring optimum end-use performance, thereby reducing production costs and scrap rates. The relationship between the degree of cure through the laminate thickness and the physical and mechanical properties in thick composite sections is of particular commercial interest. Establishing validated models that relate process variables (i.e. applied pressure and heat input) to cure-induced residual stresses and strains, non-uniform cure distributions, glass transition temperature and part thickness is seen as a high priority. Work is proceeding in the use of sensors in composites for measuring degree of cure.

The issue of quality control, and hence inspection of thick composite sections is critical to the acceptance of these materials for usage in primary safety critical (and secondary) loading applications. Information on the structural integrity of the finished product is needed in order to verify material conformance to user specifications and acceptance standards, and safety regulations. NDE techniques regularly used for production include visual, ultrasonic C-scan and X-radiography inspection. Techniques, such as thermography, shearography and acoustic emission, are being developed for future use in production and in-service inspection.

The most widely used technique for assessing the structural integrity of a composite is ultrasonic C-scan inspection. The technique can be used to detect a variety of production type defects (e.g. delaminations, voids and porosity, resin starved and resin rich areas) in both thin and thick (40 to 50 mm) laminates manufactured from continuous aligned and woven fabric reinforcement. Detection of defects with ill-defined boundaries (or interfaces with surrounding substrate material) is difficult, particularly as the laminate thickness increases. Detection of defects is further exacerbated in non-crimped fabrics (NCFs), where the material is highly heterogeneous, and hence is highly dispersive/attenuative to ultrasonic signals. X-radiography is frequently used to evaluate bonding of inserts in laminate panels and honeycomb core to skin bonds in sandwich panels. Detection of disbonds becomes more difficult with increasing laminate thickness.

9 DISCUSSION AND CONCLUSIONS

There is strong evidence to suggest that variations in material (particularly strength) properties due to laminate thickness can partially be attributed to differences in processing conditions. Thicker laminates require longer curing cycles in order to achieve complete cure. Increasing the rate of cure may reduce the cure cycle time, but at the expense of material performance. Internal stresses developed during processing can result in defect formation (e.g. voids, delaminations and microcracks). Excessive porosity adversely affects strength properties (particularly matrix dominated properties), and environmental and fatigue resistance. Process-induced thickness effects can be minimised by optimising the cure cycle and by controlling processing variables, such as temperature, pressure, resin viscosity, flow rates, etc. Cure simulation/modelling could play an important part in understanding and controlling the cure kinetics and degree of cure at any point in the composite at any time during the cure cycle. The development of cure processing models and monitoring procedures are needed to optimize material properties, prevent process-induced failures and most importantly reduce costs.

The second primary cause for the apparent reduction in strength with laminate thickness (scaling effect) relates to the efficiency of load transfer into the gauge-section of specimens that have different composite lay-ups and specimen test volumes. Tensile and compression strength were found to be limited by the stress concentration developed at the end tabs. In the case of compression, this problem is compounded by the mechanism in which load is transferred (i.e. shear and end loading) and is dependent on the test fixture and clamping mechanism employed. It is clear that there exist many artifacts exist that suggest size effects, but when carefully evaluated show little evidence of a Weibull weakest-link type size effect.

There are procedures that can be adopted for reducing (if not eliminating) the effect of testing artifacts, such as [12]:

- Ensuring the same strain rate is used for all tests;
- Using "distributed" ply stacking sequences;
- Optimising end tab design and clamping mechanisms; and
- Using 90° and 45º plies on the outside of laminates to reduce end tab stress concentration.

It is advisable to ensure that materials used in comparative studies to assess scaling effects have identical consolidation and thermal histories. This can be achieved by either machining specimens of different thicknesses from the same thick section or by co-forming several laminates in a single processing run (i.e. sandwich structure consists of several laminates separated by release film).

Considerable development work is still required to provide test methods/standards and to verify predictive models for determining the behaviour of thick composite structures (with and without damage) under uniaxial loading conditions. Validated design procedures and safety margins need to be established for curved (i.e. pressure vessels and pipes), hybrid (metallic and composite) and joined structures (i.e. bonded and bolted). The review has highlighted the need for reliable data pertaining to thick composite materials, particularly for non-aerospace applications. Future work needs to rectify this weakness, especially as there is a large market potential for these materials.

Recommendations for Future Developmental Work are summarised below.

Processing and Quality Assurance

- Real-time cure monitoring techniques and standards for on-line processing of both composite laminates and bonded composite structures. Future work will need to account for different material systems and processing routes, and the use of sensors located within or on the surface of the structure.
- Validated models, which relate processing parameters to cure kinetics, resin shrinkage, laminate thickness, glass-transition temperature, stress development and material performance.
- NDE techniques for measuring process induced through-thickness laminate/residual stresses and strain distributions in thick laminate sections and joined structures.
- Extension of NDE techniques to the in-service inspection of thick composite sections (i.e. laminates, sandwich structures and bonded and bolted systems).
- Method(s) of evaluating residual stresses and through-thickness cure gradients.

Thick Composite Properties

- In-plane tension and compression test methods/standards suitable for characterising composite behaviour under static, creep and fatigue loading conditions. This work would need to be extended to high rates, elevated temperatures and hostile environments, and include defect criticality for various production type defects.

- Test methods/standards and failure criteria for multi-axial (i.e. biaxial and triaxial) loading of lineal and thick-walled cylindrical specimens. This work would need to include static, creep, fatigue and hydrostatic loading modes and environmental conditions (including acids, solvents, fuel, salt water, elevated temperature, etc.), and be extended to include composite component testing.
- Test methods/standards for assessing the behaviour of bolted and bonded composite sections with and without defects (i.e. criticality) for various loading and environmental conditions (see above).
- Impact and high rate test methods/standards and predictive models for damage initiation and propagation.
- Development of fire test methods/standards and models for composite materials and structures (to include bonded and bolted systems). The assessment would need to consider the effect of additives (e.g. fire retardants) on short- and long-term performance, and cure processing of composite materials. An assessment of structural integrity of fire damaged composite structures would include loaded structures.
- Reliable design procedures for monolithic and joined composite structures to account for degradation due to mechanical loading, environmental factors, fire and impact.

Development of predictive models/design procedures for monolithic and joined composites would need to consider non-linear material behaviour, effect of fibre format/architecture (e.g. discontinuous and continuous, random and aligned, and 2-D and 3-D woven fabrics), hygrothermal effects, edge effects and “blocking” of lamina of identical fibre orientation. The models will need to account for fibre, matrix and interface failure modes, and be applicable to both in-plane and out-of-plane multi-axial loading conditions.

Recommendations specific to the SM07 research programme are:

- Design and assessment of coupon geometries, end tabs (inc. adhesive fillets) and loading/gripping mechanisms for thick tension and compression tests. Digital image correlation (DIC) strain mapping to be carried out on test coupons to validate FEA predictions.
- Undertake a series of tension and compression tests on unidirectional, and “distributed” ($[+45/-45/0/90]_{ns}$) and “blocked” ($[+45_n/-45_n/0_n/90_n]_s$) quasi-isotropic carbon/epoxy laminates of different thicknesses (2 to 20 mm) with identical consolidation and processing history.
- Evaluate the effect of laminate thickness, end tabbing and loading/gripping mechanism on elastic and strength properties, and failure modes. Tests to confirm whether data generated from standards for relatively thin specimens are equivalent to or representative of thick section mechanical behaviour.
- Development of measurement protocols for characterising the tensile and compressive mechanical behaviour of thick composite material sections.

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