

Report

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Thick Composites:
Part I: Mechanical Test Review
Part II : Cure Optimisation

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W R Broughton**

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Part 2 : Cure Optimisation**

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Industry and Innovation Division (I2D)**

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 in Part I tension, compression, shear, flexure and multi-axial loading conditions, environmental, impact and fatigue resistance, and issues relating to manufacture and inspection of thick laminates. Work undertaken to assess the effects of cure on thick laminate properties and to formulate an optimised cure cycle for a 20 mm thick carbon fibre-reinforced plastic (CFRP) material is detailed in Part II. Recommendations are provided as to future work required for test method development for thick composite laminates.

This report was prepared as part of the research undertaken at NPL for the Department of Innovation, Universities and Skills funded project SM07 "Validated Measurement Techniques for Improved Design of Large Advanced Composite Structures Subjected to Multi-Axial Loading" within the NMS Materials Programme 2007.

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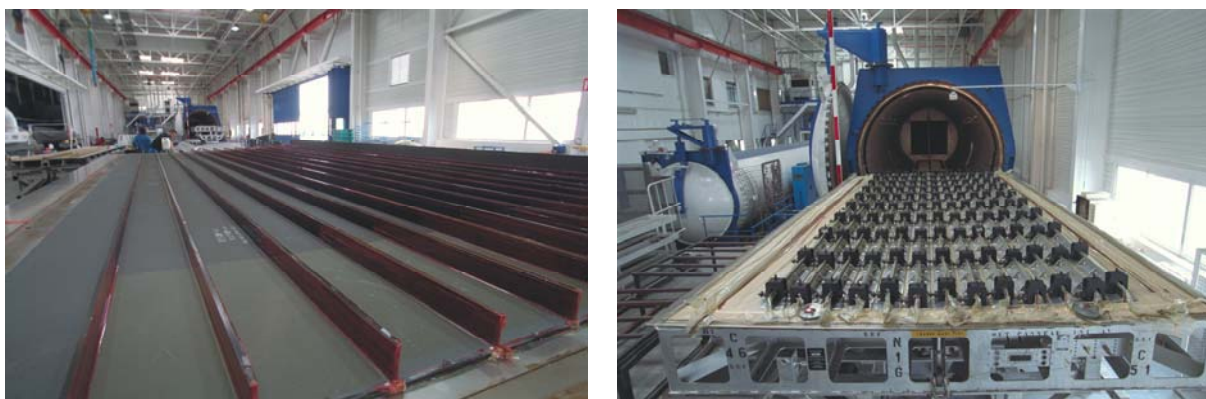
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1 INTRODUCTION

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 10 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 (Figure 1) and off-shore drilling platforms.



Courtesy of Airbus

Figure 1: Manufacture of composite wing box component for the Airbus A380 aircraft

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 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, large test fixtures and increased difficulties associated with load introduction into the specimen. The lack of suitable test methods has resulted in a shortage of reliable engineering data for large structural applications.

This report consists of 2 main parts; Part I 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, and Part II details cure optimisation work undertaken for 20 mm thick laminates. It forms part of the Department for Industry, Universities and Skills (DIUS) funded project SM07 “**Validated Measurement Techniques for Improved Design of Large Advanced Composite Structures Subjected to Multi-Axial Loading**”, within the NMS Materials 2007+ Programme. In Part I of the report mechanical test methods for characterising in-plane elastic and strength properties in relation to their suitability for testing thick-sections are detailed. Tension, compression, shear and flexure testing are covered along with multi-axial testing, durability methods and impact resistance. Part II details initial experimental work undertaken within project SM07 covering manufacturing and cure optimisation issues. Conclusions and recommendations for future development work are presented in Section 5.

PART I

2 MECHANICAL TEST METHODS FOR THICK SECTIONS

2.1 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, while Part 5 is limited to 1 and 2 mm thicknesses for UD laminates.

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 same 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 $[0^\circ_n/90^\circ_n]_s$ 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 $[+45^\circ/-45^\circ/0^\circ/90^\circ]_{ns}$ 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 $[+45^\circ_n/-45^\circ_n/0^\circ_n/90^\circ_n]_s$ 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 $n \geq 2$ “blocked” specimens, with the predominant failure initiating at the grip. This indicates that blocking the

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

n	Width x Thickness (mm)	Gauge Length (mm)	Volume Ratio	Tensile Strength (MPa)	
				$[+45^\circ/-45^\circ/0^\circ/90^\circ]_{ns}$	$[+45^\circ_n/-45^\circ_n/0^\circ_n/90^\circ_n]_s$
1	10 x 1	75	1	552	552
2	20 x 2	150	8	571	499
3	30 x 3	225	27	564	496
4	40 x 4	300	64	548	474

plies together reduces the damage tolerance of the laminate making it more susceptible to stress concentrations initiated by the end tabs.

Niklewicz and Sims [12], also conducted tensile tests on two generic angle-ply $\pm 45^\circ$ carbon/epoxy laminates with “blocked” plies $[+45^\circ_n/-45^\circ_n/45^\circ_n/-45^\circ_n]_s$ and “distributed” plies $[+45^\circ/-45^\circ/+45^\circ/-45^\circ]_{ns}$ and $n = 1, 2, 3, 4$ (see Table 2). For “distributed” plies $[+45^\circ/-45^\circ/+45^\circ/-45^\circ]_{ns}$, there was an increase in tensile strength with increasing specimen size, whereas for the “blocked” specimens $[+45^\circ_n/-45^\circ_n/45^\circ_n/-45^\circ_n]_s$ the tensile strength decreases. The distributed specimens exhibited ductile stress-strain behaviour, while the blocked exhibited brittle, low strain-to-

Table 2: Tensile Strengths for $\pm 45^\circ$ Carbon/Epoxy Laminates [12]

n	Width x Thickness (mm)	Gauge Length (mm)	Volume Ratio	Tensile Strength (MPa)	
				$[+45^\circ/-45^\circ/+45^\circ/-45^\circ]_{ns}$	$[+45^\circ_n/-45^\circ_n/45^\circ_n/-45^\circ_n]_s$
1	10 x 1	75	1	194	194
2	20 x 2	150	8	222	146
3	30 x 3	225	27	253	122
4	40 x 4	300	64	271	108

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.

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 $+45^\circ$ ply is at a maximum.

In addition, Niklewicz and Sims [12] carried out tests on cross ply $[90^\circ/0^\circ/90^\circ/0^\circ]_{ns}$ specimens using the same material as for the quasi-isotropic and $\pm 45^\circ$ lay-ups. All specimens failed within the gauge length with no volume related strength scaling in evidence as shown in Table 3.

Table 3: Tensile Strengths for $[90^\circ/0^\circ/90^\circ/0^\circ]_{ns}$ Carbon/Epoxy Laminates [12]

n	Width x Thickness (mm)	Gauge Length (mm)	Volume Ratio	Laminate Strength (MPa)	Failure strain (%)	0° Failure Stress (MPa)
1	10 x 1	75	1	835	1.3	1767
2	20 x 2	150	8	847	1.3	1795
3	30 x 3	225	27	824	1.3	1740
4	40 x 4	300	64	829	1.3	1754

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 when using tensile grips. 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 transition between where the end tabs terminate and the 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 currently 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, but a beneficial effect in compression (see next section).

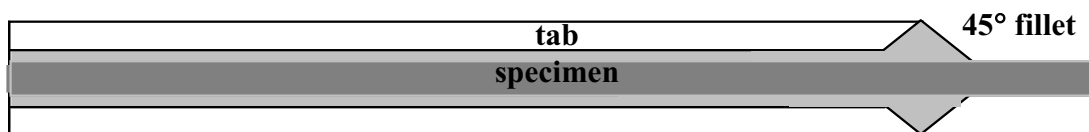


Figure 2: Reverse tapered end tab with 45° fillet.

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 (i.e. 1 mm to 16 mm). 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 disbands 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 by thick composite sections (see Part II).

2.2 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.

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.



Figure 3: End-loading compression fixture with specimen.

There are five commonly used loading fixtures for compression testing: IITRI, Celanese, end-loading, combined end and shear-loading (ASTM D 6641) 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 expected to be 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 were 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.

2.3 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 $\pm 45^\circ$ 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 $\pm 45^\circ$ 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 $\pm 45^\circ$ 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 (e.g. a 10 mm thick specimen requires a 350 mm x 350 mm panel).



Figure 5: V-notched beam test.

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.

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).

2.4 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 transverse 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. However, it is difficult to scale these combined stress specimens effectively.

2.5 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 [34]. 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.

2.6 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 [35] 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.

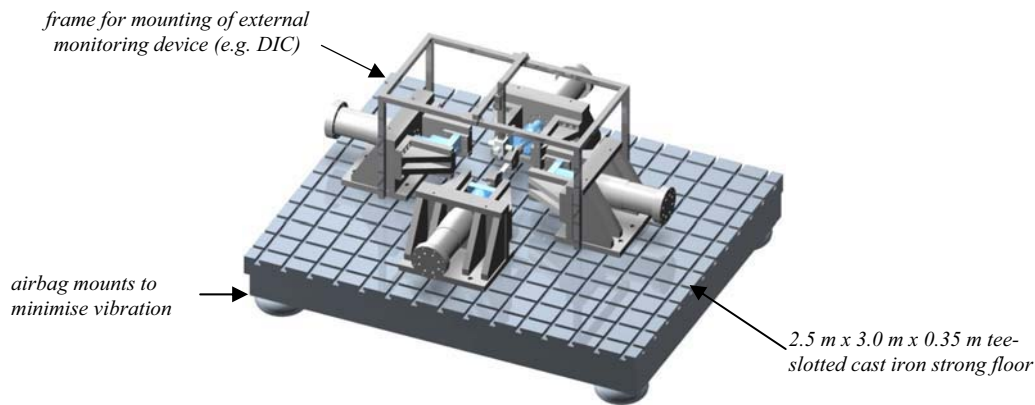


Figure 6: Schematic of NPL's multi-axial strong floor test facility.

2.7 MULTI-AXIAL TESTING

This section examines multi-axial testing methods that have been used for testing relatively thin material sections and that may have potential 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) [36].

2.7.1 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.

Planar coupons have tended to be the favoured specimen geometry for characterising applications in which the material is used in flat laminate form (cf. cylindrical specimens - Section 2.7.2). The issues associated with multi-axial testing of thick material sections using planar coupons are similar to those issues identified for uni-axial tensile and compressive in-

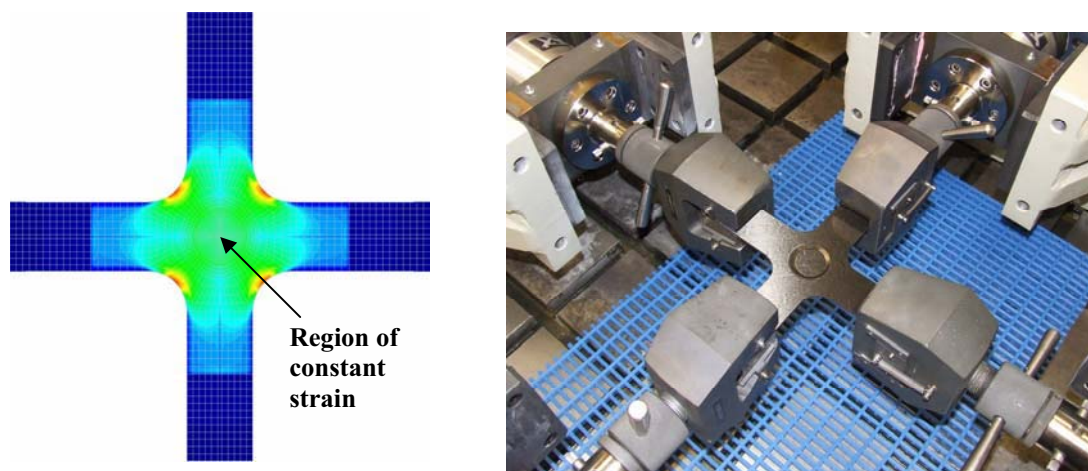


Figure 7: Biaxial testing of cruciform specimen.

plane testing (Sections 2.1-2.2) and include test machine size, loading fixture and coupon design. Thick section multi-axial specimens will require large loads to be introduced into the specimen, necessitating not only the use of extremely specialised and expensive test machines, but appropriate design of gripping fixtures to transfer the load into specimens, and specimen and end-tab design.

2.7.2 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. As for the planar coupons, testing cylindrical specimens with wall thicknesses ≥ 10 mm will necessitate the use of large test machines and specialised fixtures for load introduction.

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 [37-39]). ASTM D 2992 [40] specifies procedures for determining the long-term cyclic and static hydrostatic strength of a composite pipe. According to Ellyin and Marten [39], ASTM D 2922 specifies a worst-case scenario rather than typical conditions experienced by composite pipes for example in oil fields.

3 ENVIRONMENTAL DURABILITY

Thick sections will generally take a longer time to degrade under hostile environments 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 for thick composite sections.

3.1 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 [41-42]. 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 [43] 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.

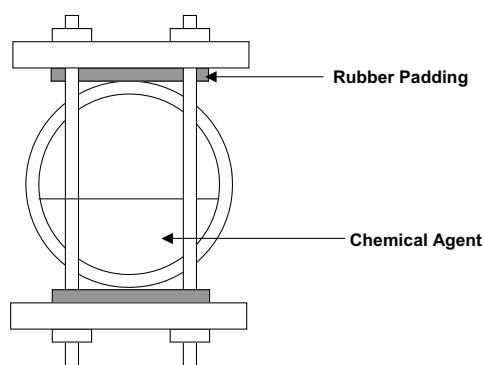


Figure 8: Strain-corrosion test apparatus.

The International standard BS EN ISO 62 [44] describes a procedure for determining the moisture absorption properties of thin material 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. It is important to note that if equilibrium conditions are not met and the material sees varying degrees of moisture content (for example) throughout its thickness, then this may result in apparent but potentially erroneous reporting of scale or size effects. 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.



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

3.2 PRODUCT TESTING

Combined stress and environmental conditions are used in some product test methods. ASTM D 3681 [45] and BS 5480 (Part 2) [46] 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.

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. As mentioned previously, thick material sections will give rise to rig and loading issues.

PART II

4 MANUFACTURE OF THICK LAMINATES

It is suggested 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. Fabrication of thick composite laminates (10 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 may also result in material degradation [47]. 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.

4.1 EXPERIMENTAL ASSESSMENT OF CURE CYCLE EFFECTS

The following section details work that has been undertaken to assess the effect of two different cure cycles on the physical, mechanical and thermal properties of sections of 20

mm thick unidirectional carbon-fibre reinforced plastic (CFRP) laminates. The material system used was SE84LV (~0.3 mm cured ply thickness) and was supplied by Gurit Holdings AG. The cure cycles used were; (a) a standard cure cycle for ‘thin’ (≤ 5 mm thick) laminates as recommended by the material supplier and; (b) an ‘optimised’ cure cycle for ‘thick’ sections (~20 mm thick). Details of how the ‘optimised’ cure cycle was formulated are given in Section 4.1.1.

4.1.1 Standard and optimised cure cycles

The autoclave cure cycle for thin (< 5 mm thick) SE84LV laminates, as per the material supplier’s recommendation, is shown schematically in Figure 10. The temperature ramp rate is $3\text{ }^{\circ}\text{C}/\text{min}$ and there is a temperature dwell at $75\text{ }^{\circ}\text{C}$ at a viscosity of 150 poise where pressure is applied. This is followed by a further dwell for 60 minutes at the $120\text{ }^{\circ}\text{C}$ cure temp. The vacuum is released at a pressure of 1.7 bar.

Previous work by Gurit [48] looked at curing different panel thicknesses up to 28 mm thick and used lower cure temperatures (up to $85\text{ }^{\circ}\text{C}$) and longer cure dwell times (up to 10 hours). In formulating an optimised cure cycle for thick sections, a similar approach was followed at NPL to look at the degree of exotherm in thick sections and how potentially damaging levels of exotherm could be minimised to the levels seen in the curing of thin sections.

Cure optimisation trials were performed in a Climatic Systems programmable fan oven. All panels were rectangular in shape (203 mm x 152 mm) and were vacuum bag cured. No pressure was applied during cure. Initially, a thin, 4 ply laminate was layed-up and vacuum bag consolidated for 10 minutes prior to cure. A ‘K’ type thermocouple was centrally positioned in the panel at mid-thickness and a multi-channel Picolog was used to record the panel temperature every minute. Figure 11 shows the oven and panel temperature throughout the cure cycle. It can be seen that a peak temperature of $123.5\text{ }^{\circ}\text{C}$ was recorded

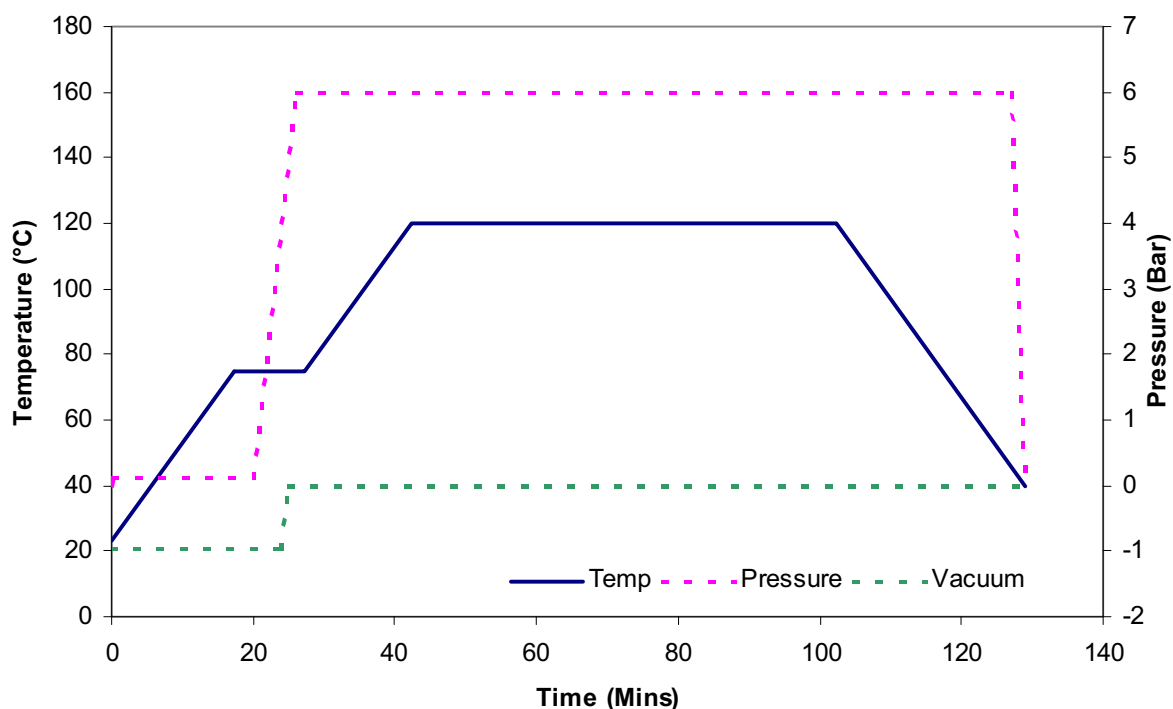


Figure 10: Standard autoclave cure cycle for thin (≤ 5 mm) laminates

approximately 20 minutes after the oven had achieved the cure temperature of 120 °C. Thus the exotherm that occurred within the panel was seen to cause a 3.5 °C temperature overshoot.

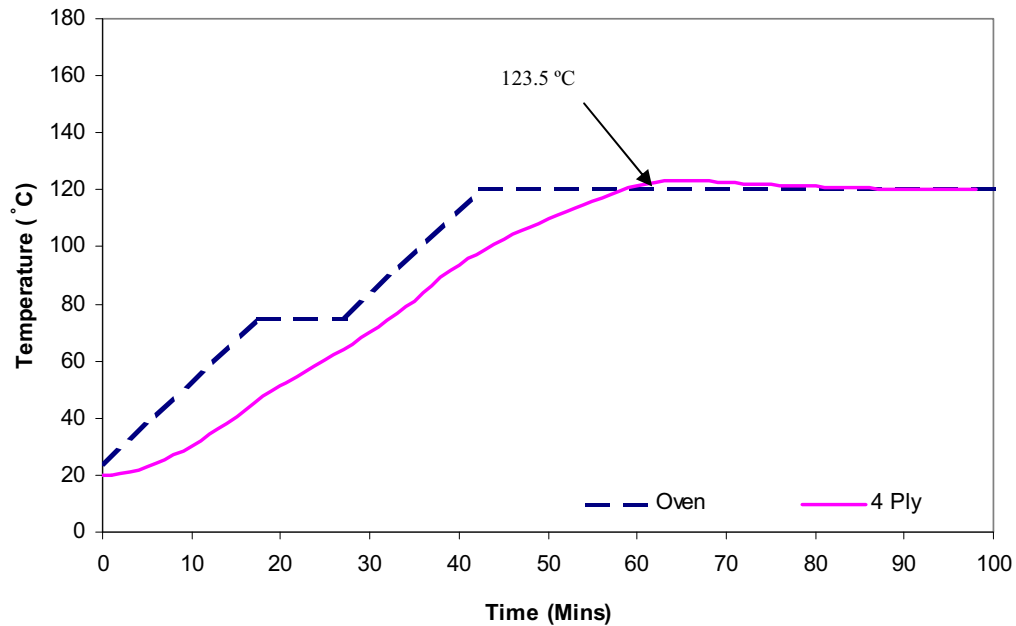


Figure 11: Oven and panel temperature for a standard cure 4 ply laminate

Following the thin laminate trial, a 64 ply, ~20 mm thick panel was layed-up. In order to obtain equivalent consolidation as for the thin 4 ply laminate, the thick laminate was prepared by laying-up 4 plies and then vacuum bag consolidating for 10 minutes, followed by adding a further 4 plies and again consolidating for 10 minutes. This process was followed until 64 plies had been layed-up to form the complete panel. In order to obtain as much information as possible with regard to the temperature distribution and gradients throughout the thick panel during cure, seven 'K' type thermocouples were incorporated at various positions and depths (Figure 12). The 64 ply laminate was then cured using the standard, thin cure cycle in order to investigate the degree of exotherm within the panel.

Figure 13 shows the oven and panel temperatures recorded throughout the cure cycle. It can be seen that there is a substantial increase in temperature (maximum ~158 °C) above the recommended cure temperature of 120 °C in the laminate due to a large degree of exotherm.

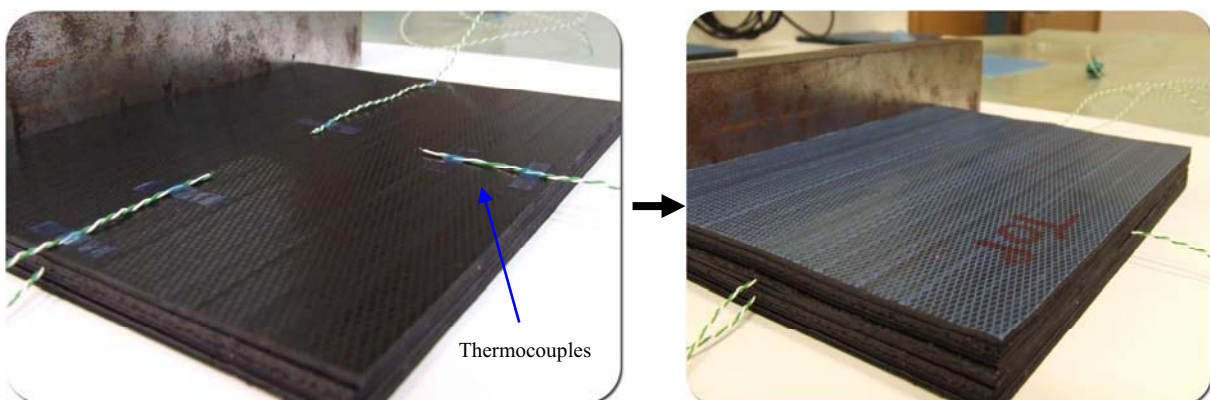


Figure 12: Laying-up of a 64 ply laminate and thermocouple placement

It was observed that the temperature difference between the top and bottom caul plate positions in the panel was $\sim 10^{\circ}\text{C}$ and the mid-thickness panel temperature was 154.5°C . A comparison of the thin and thick mid-panel maximum panel temperatures is shown in Figure 14.

Using the standard 'thin' cure cycle to cure a 64 ply thick panel resulted in a temperature overshoot (above the cure temperature of 120°C at a mid-thickness location) of 34.5°C

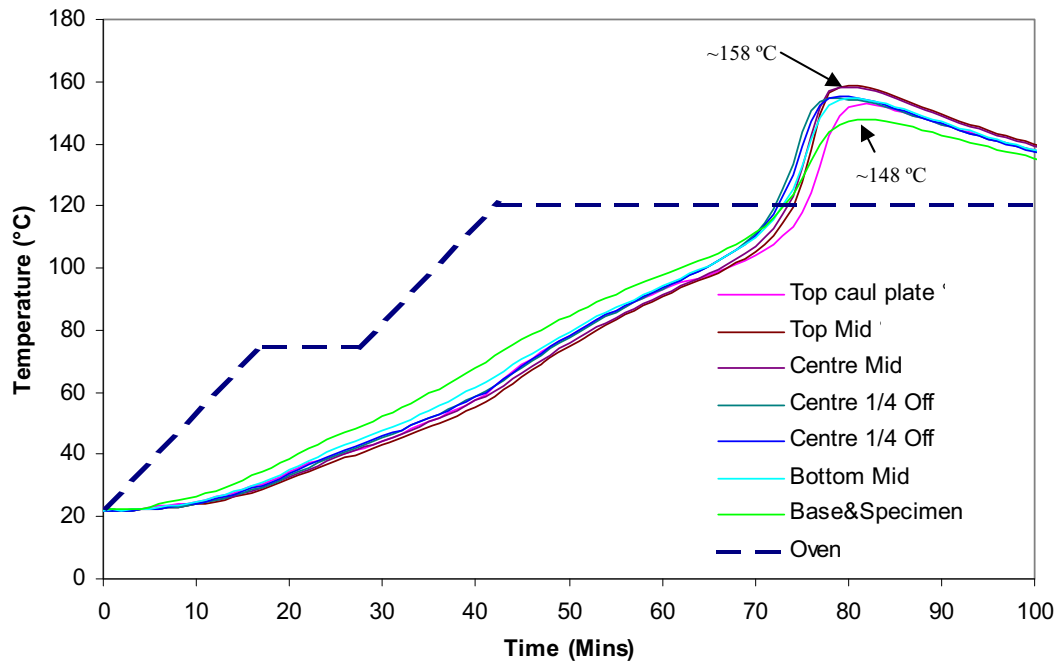


Figure 13: Oven and panel temperature distribution for a 64 ply laminate cured using the standard cure cycle

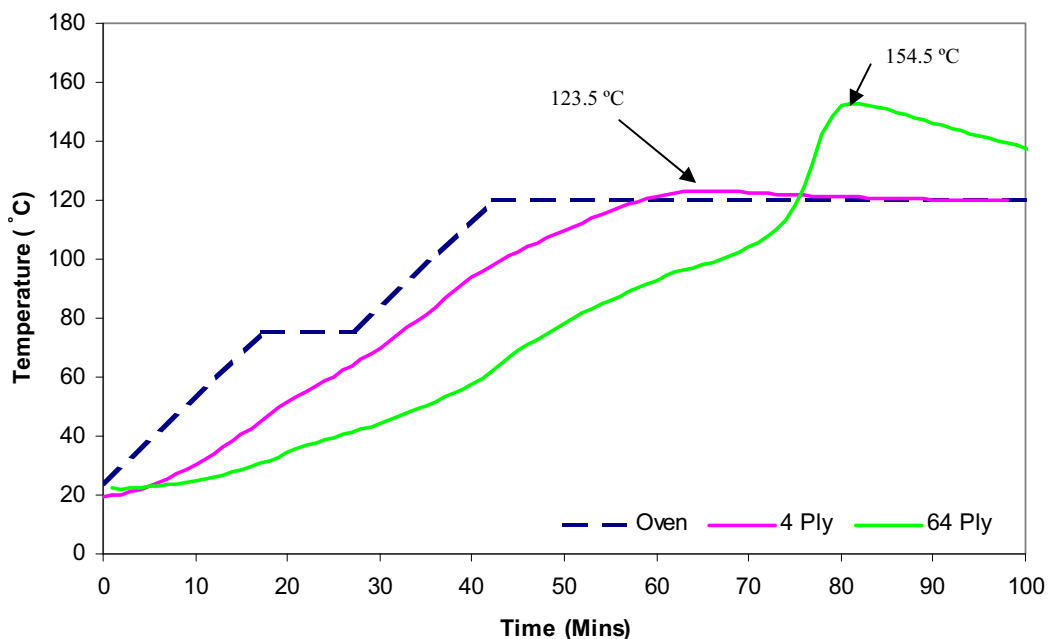


Figure 14: Mid-thickness temperature for 4 and 64 ply thick panels (standard cure)

which was deemed to be unacceptable due to the risk of thermal damage to the material. Therefore, the standard cure cycle was modified by undertaking a series of further cure cycle trials where the number of temperature ramps and dwell periods were altered to minimise the degree of exotherm throughout the cure cycle. The general approach was to use a series of additional temperature ramps, at a slower ramp rate of 1 °C/min (cf. 3 °C/min ramp rate for standard cure cycle), in conjunction with longer dwell periods in order to stagger the exothermic reaction associated with cure so that the maximum panel temperature was similar to that seen for the 4 ply thick panel. The optimised cure cycle formulated for thick panels, after a number of experimental iterations is shown in Figure 15, the standard cure cycle is also shown for comparison. It is noted that the total cycle time has increased by a factor of ~4.

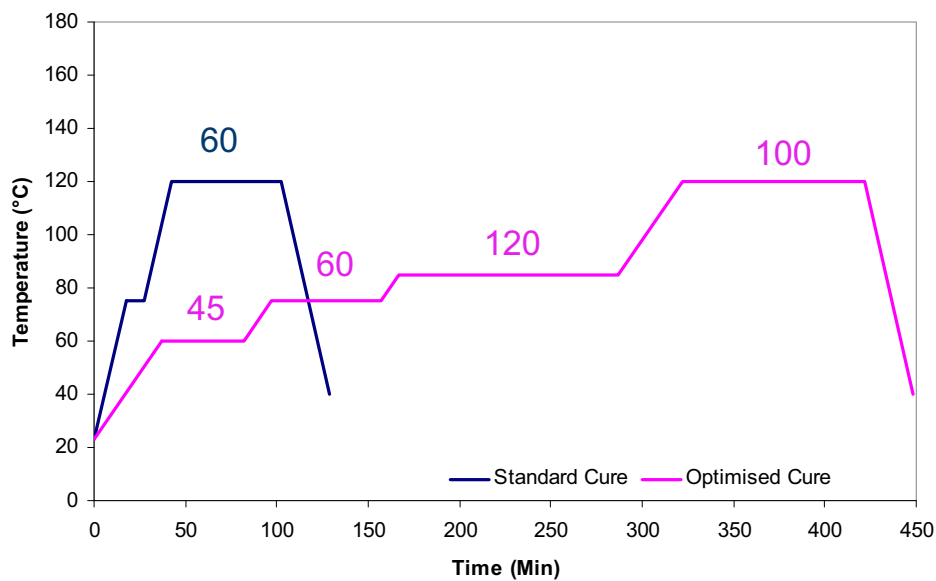


Figure 15: Standard (thin) and optimised (thick) cure cycles

The optimised cure cycle is split into 4 stages. The 1st and 2nd dwells allow the panel to reach the oven temperature, the 3rd dwell reduces the rate of cure limiting the exotherm and the 4th dwell is at the required cure temperature. Figure 16 shows the recorded temperatures (oven and thermocouples) for a 64 ply laminate cured using the optimised cure cycle. It can be seen that the maximum panel temperature no longer shows the large increase observed when using the standard cure cycle and is now close to the recommended cure temperature of 120 °C. Also, less variation in temperature was observed throughout the panel thickness.

Having optimised the cure cycle for thick laminates via a series of oven trials, thick panels were then produced in the NPL autoclave facility. The optimised cure cycle and details of vacuum and pressure application in the autoclave are shown schematically in Figure 17. The thick panels (64 ply) were made using the standard and optimised cure cycles so that the effects of exotherm on the laminate physical, mechanical and thermal properties could be determined (see Section 4.1.2). For both the standard and optimised autoclave cure processes, thermocouples were used to monitor the panel temperature during cure. The recorded temperature profiles are shown in Figures 18-19. As for the oven trials, a large degree of exotherm and variation of temperature throughout the panel thickness was seen for the standard cure. For the optimised cure, the temperature distribution throughout the panel was uniform and there was no excessive exotherm.

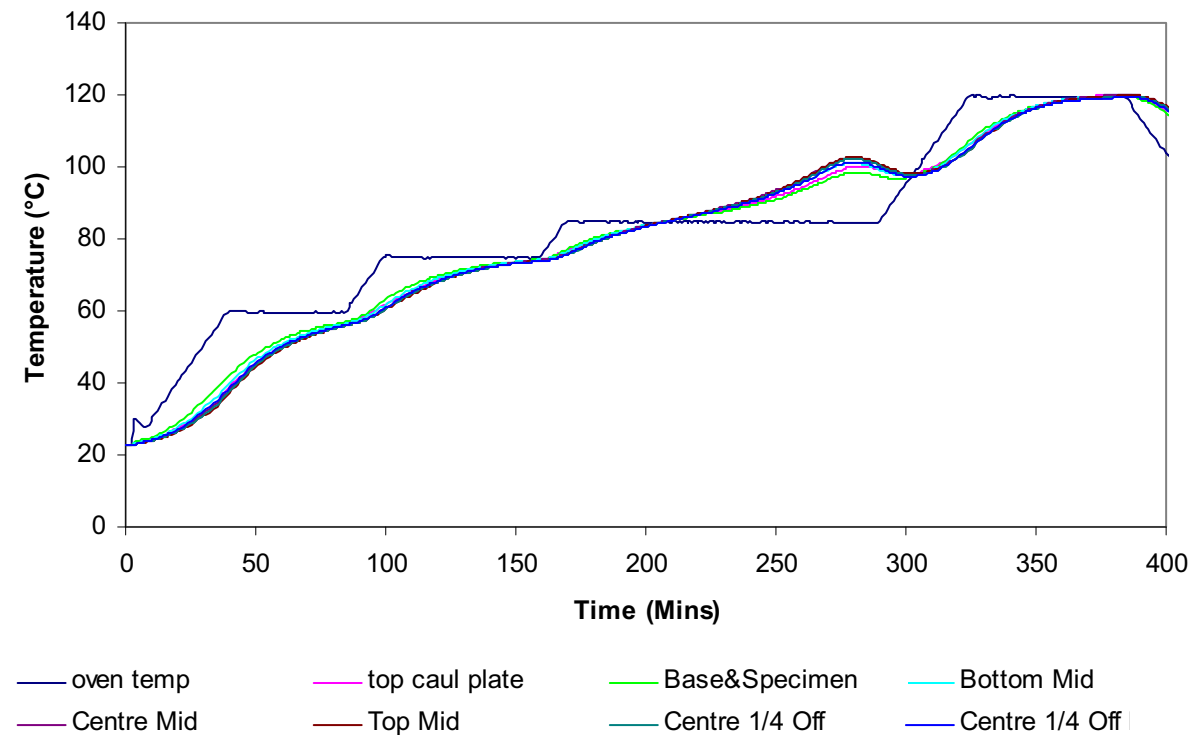


Figure 16: Oven and panel temperature distribution for a 64 ply laminate cured using the optimised cure cycle

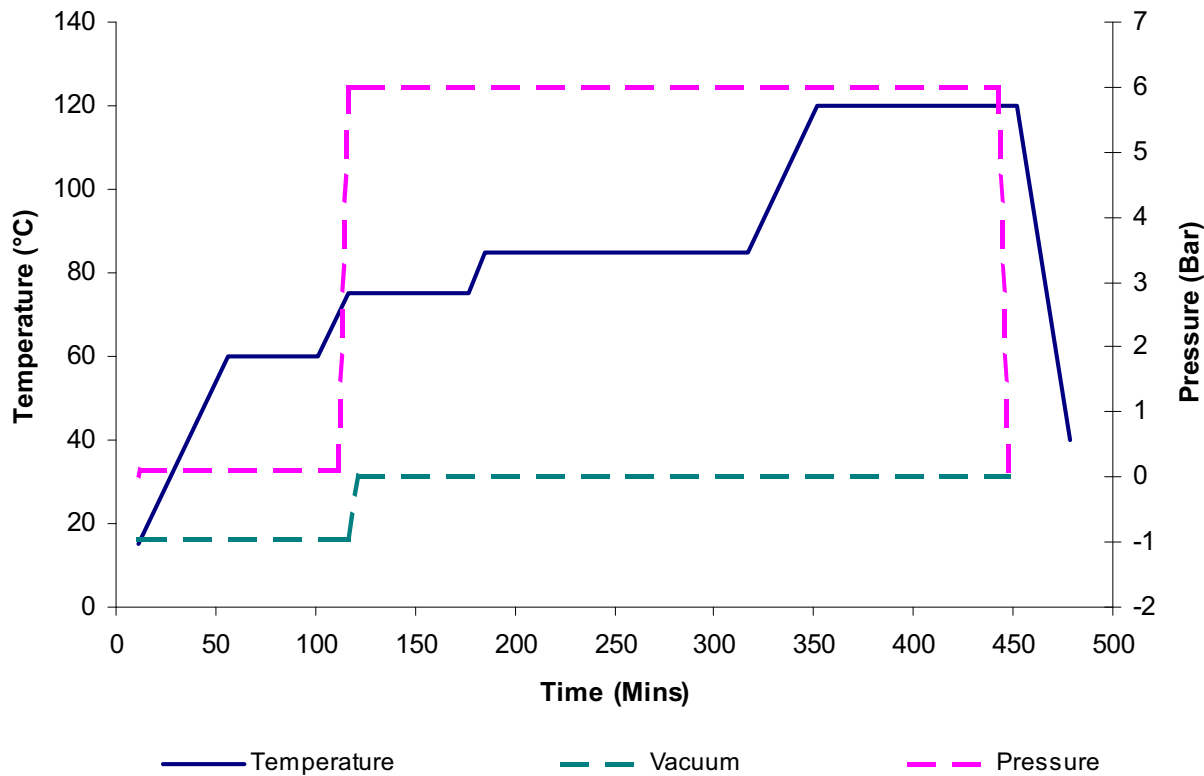


Figure 17: Optimised cure cycle - autoclave

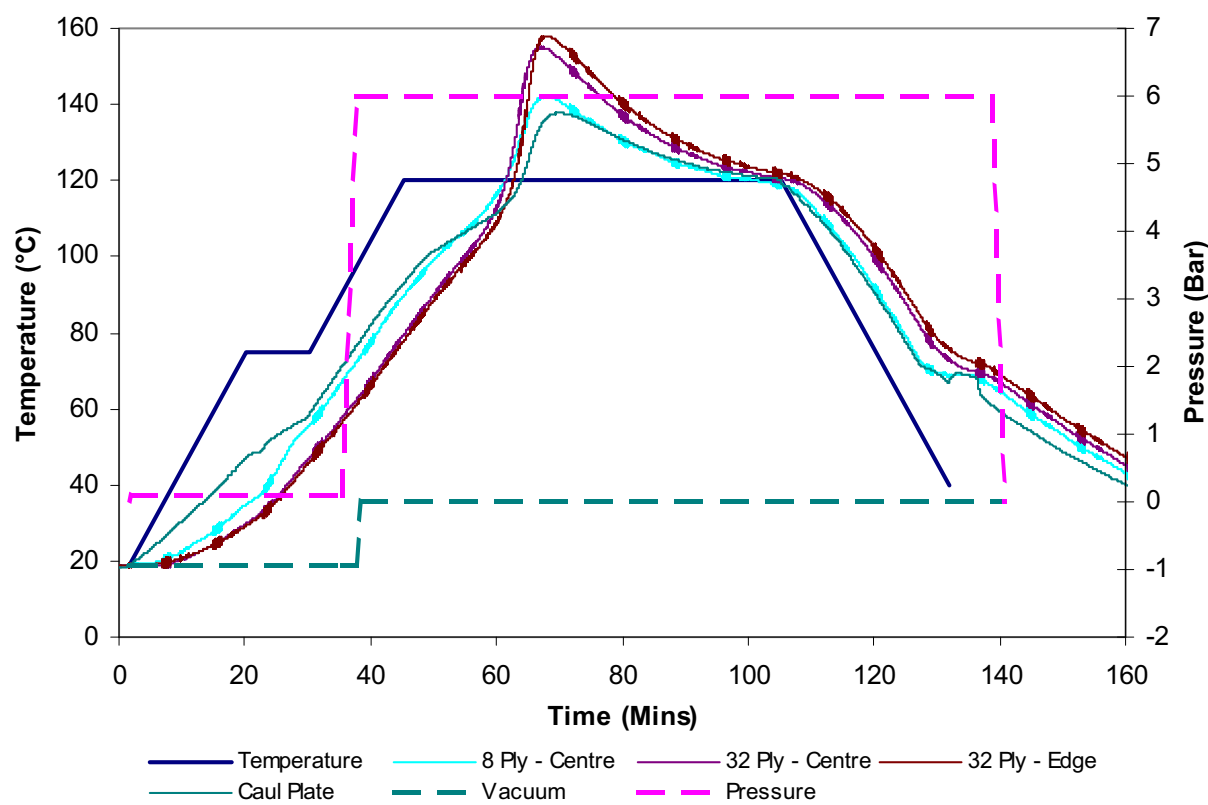


Figure 18: Autoclave and panel temperature distribution for a 64 ply laminate cured using the standard cure cycle

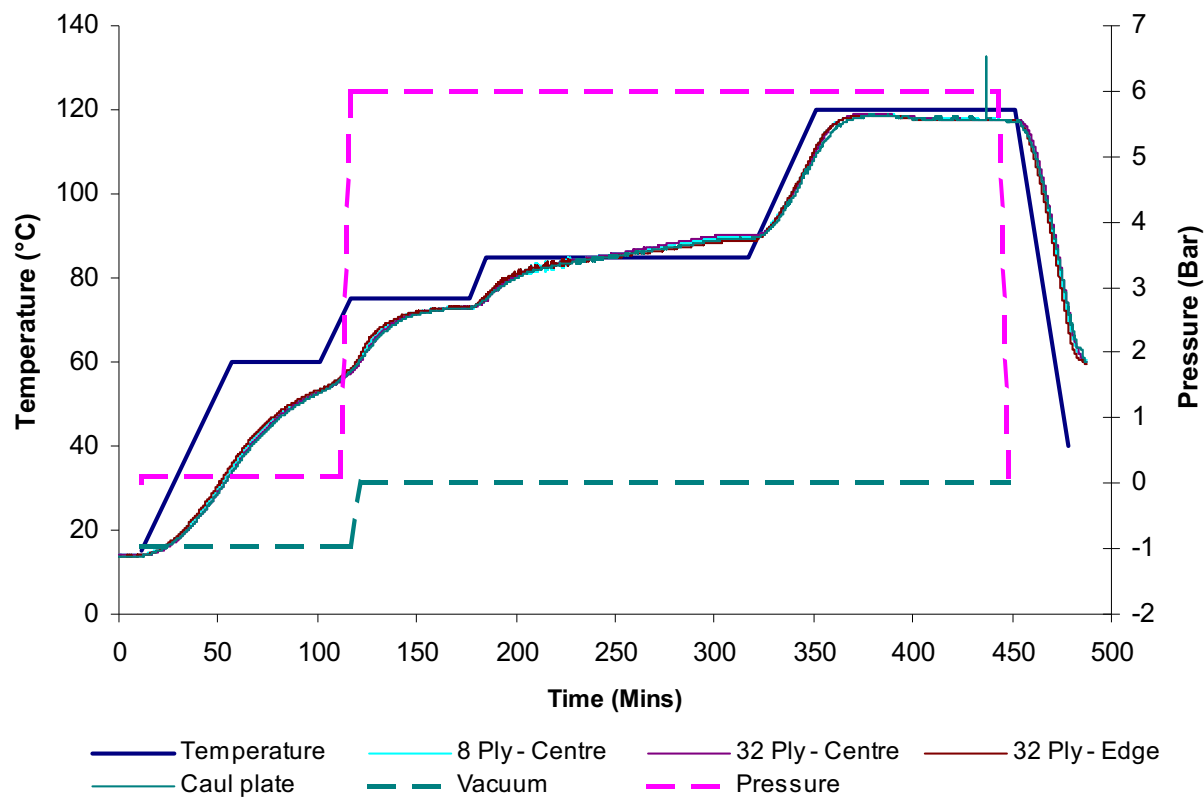


Figure 19: Autoclave and panel temperature distribution for a 64 ply laminate cured using the optimised cure cycle

4.1.2 Assessment of cure cycle effects on physical, mechanical and thermal properties

In order to assess the effects of the standard and optimised cure cycles (detailed in Section 4.1.1) on the properties of cured laminates, a number of physical, mechanical and thermal tests were undertaken. The tests and the reasons why they were undertaken are as follows:

- Fibre and void volume fractions - physical characterisation of laminate microstructure,
- Through-thickness shear strength by double-notch shear - sensitivity to matrix properties i.e. degree of cure,
- In-plane compression - sensitivity to matrix and fibre/matrix interfacial properties,
- In-plane shear properties by the V-notched beam (Iosipescu) method - sensitivity to matrix properties,
- Glass transition temperature (T_g) via differential scanning calorimetry (DSC) - degree of cure of matrix,
- Residual strain measurement by the compliance method (incremental slotting) - indication of presence of residual strains and stresses within cured panels.

The specimens required for these tests (with the exception of the residual strain measurements) were milled to size from the ~20 mm thick cured panels. Where appropriate and possible, specimens for these tests were extracted from top, middle and bottom through-thickness panel locations to assess whether there were any property variations through the panel thickness (see Figure 20). Care was taken to ensure that specimen mid-thickness plane coincided with the mid-thickness plane of the top, middle or bottom sections of the panel. Due to the panel sizes made in these cure trials and the presence of embedded thermocouples within the panels, for some of the tests it was possible to obtain specimens from both oven and autoclave manufactured panels, but for other tests it was only possible to obtain specimens from the autoclave panels. It is noted here that the results of most importance and significance are those obtained for the autoclave panels; as subsequent work in this project towards the development of thick section test methods will be based on autoclave manufactured material. Oven cured material data was produced for comparison to the autoclave data and to assess how sensitive the SE84LV material system was to processing route.

Details of the tests undertaken and respective results are provided in the following sections.

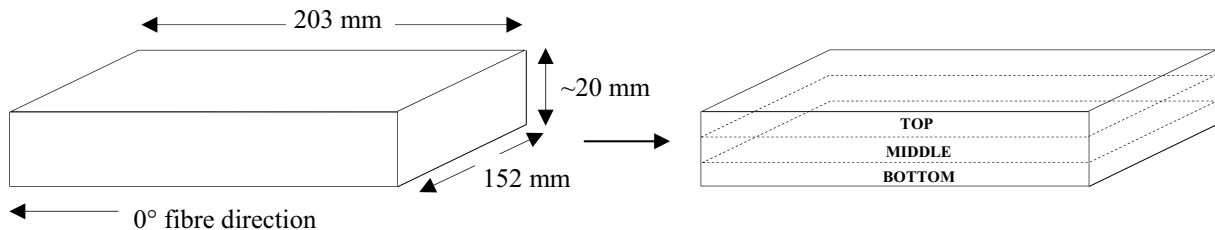


Figure 20: Specimen extraction from ~20 mm thick oven and autoclave, standard and optimised cure panels

4.1.2.1 Fibre and void volume fractions

Fibre volume fraction (V_f) was measured according to the acid digestion (nitric acid) method described (Procedure A) in ASTM D 3171 - Standard Test Method for Constituent Content of Composite Materials [49] and also using optical microscopy on polished sections of laminate. As void volume fractions (V_v) were expected to be quite low, especially for the autoclave cured panels, and the inherent lack of sensitivity of the acid digestion technique for measuring low void volume fractions (<2%), optical microscopy in conjunction with image analysis was used for the void volume fraction measurements. For comparison, V_f was also measured using optical microscopy and image analysis. For V_f and V_v by optical microscopy a series of 20 images per laminate section were analysed using a Nikon MM-60 measuring microscope and a x10 objective. Image-Pro Plus image analysis software was used to analyse the images.

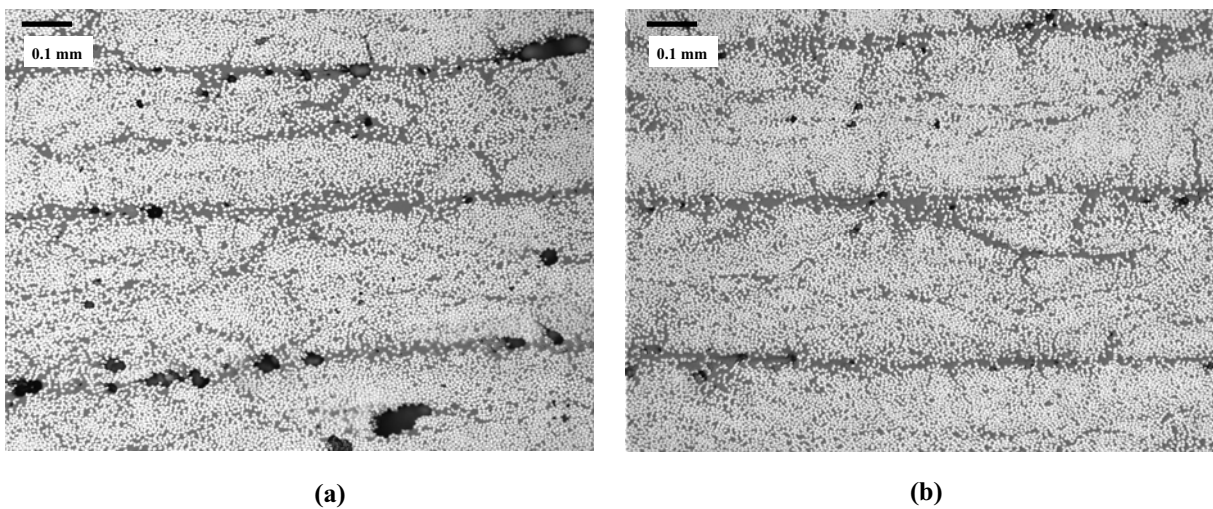


Figure 21: Micrographs for laminates produced using the standard cure cycle; (a) oven and (b) autoclave

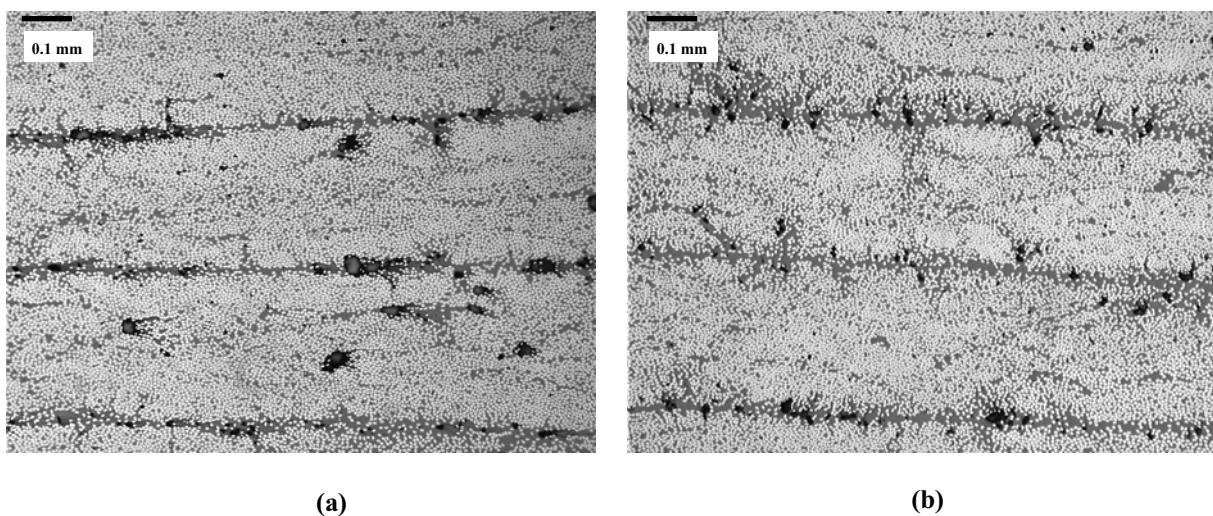


Figure 22: Micrographs for laminates produced using the optimised cure cycle; (a) oven and (b) autoclave

The results are detailed in Tables 4-5. Figure 21 shows typical micrographs for standard oven and autoclave cured laminates. Figure 22 shows typical micrographs for optimised oven and autoclave cure laminates. In general the V_f results for the standard and optimised oven cured laminates were in good agreement and tended to be $\sim 3\%$ higher than for the autoclaved panel values which were also in close agreement. It was expected that the fibre volume fraction results for the autoclave panels would be higher than the oven cured panels as pressure was applied during cure. A possible reason why the oven V_f results were higher than those for the autoclave panels was therefore thought to be due to slight differences in pre-preg roll fibre volume fractions. The V_v results for the oven cured laminates were 1.2-1.6% higher than for the autoclaved panels which is as expected as the oven cured panels were not cured under pressure. For both standard and optimised cure cycle panels (oven and autoclave processes) it was observed that there were no variations in V_f and V_v with regard to panel thickness location.

4.1.2.2 Through-thickness shear strength - double-notch shear method

Through-thickness shear strength (τ_{13}) was measured according to the NPL Draft V.01 [49] method - Determination of through-thickness shear strength using the double-notch shear test. (N.B. This draft is now being balloted as a new ISO standard).

The results for the standard and optimised cure panels (oven and autoclave processes) are shown in Tables 6 and 7. In general there were no significant differences in mean values observed between the two cure cycles and process methods (oven and autoclave). In addition, there were no significant trends seen in the strength values measured from specimens cut from different locations through the panel thickness.

4.1.2.3 In-plane compression

In-plane compression properties (strength and modulus) were measured in the fibre direction (i.e. σ_{11C} and E_{11C}) according to the end-loading method detailed in BS EN ISO 14126 - Fibre-reinforced plastic composites - Determination of compressive properties in the in-plane direction [16].

The strength and modulus results are detailed in Tables 8 and 9. The modulus results for the standard and optimised cure cycles (oven and autoclave processes) are all of similar magnitude and show no significant trends with regard to position through the thickness of the panel. The strength results were generally in good agreement, although it was observed that for the oven, optimised cure specimens the strength results were slightly higher than for the other panels. This was a somewhat surprising result as the void volume fraction, V_v for this panel was the highest value measured of all the panels at 2.7%. Strength values were also normalised with respect to the fibre volume fraction, V_f of the optimised, autoclave panel, however, the strength results for the optimised, oven panel were still the highest. It is worth noting that the coefficient of variation for this panel was twice that of the optimised, autoclaved panel.

4.1.2.4 In-plane V-notch shear

In-plane shear properties (strength, τ_{12} , and modulus, G_{12}) were measured according to ASTM D 5379 M [24]. Only a limited number of specimens were extracted and tested for the standard and optimised, autoclaved cure panels. Specimens were primarily extracted from the top section of the panels as per Figure 20.

Table 4: Fibre and Void Volume Fraction Results - Standard Cure

Panel position	Oven				Autoclave			
	Specimen	ρ (kg/m³)	V _r (%)		Specimen	ρ (kg/m³)	V _r (%)	
Top	ABLN001	1.555	58.2	58.2 (0.3, 0.5%)	ABPE030	1.515	54.7	55.0 (0.4, 0.7%)
	ABLN002	1.546	58.5		ABPE031	1.514	54.9	
	ABLN003	1.530	57.9		ABPE032	1.513	55.4	
Middle	ABLN004	1.544	57.7	57.4 (0.3, 0.5%)	ABPE010	1.528	55.0	55.1 (0.3, 0.5%)
	ABLN005	1.545	57.5		ABPE011	1.527	54.9	
	ABLN006	1.544	57.1		ABPE012	1.532	55.4	
Bottom	ABLN007	1.533	56.4	57.1 (0.6, 1.1%)	ABPE033	1.520	55.2	54.3 (0.8, 1.5%)
	ABLN008	1.538	57.4		ABPE034	1.519	54.2	
	ABLN009	1.538	57.5		ABPE035	1.512	53.6	
Panel mean, SD, CoV		1.541 (0.008, 0.5%)	57.6 (0.6, 1.1%)			1.520 (0.007, 0.5%)	54.8 (0.6, 1.1%)	

Image analysis results - standard	V_f (%)	V_v (%)
Oven	57.4 (1.7, 3.0%)	2.3 (0.6, 26%)
Autoclave	55.6 (2.5, 4.5%)	0.7 (0.3, 43%)

Table 5: Fibre and Void Volume Fraction Results - Optimised Cure

Panel position	Oven				Autoclave			
	Specimen	ρ (kg/m³)	V _r (%)		Specimen	ρ (kg/m³)	V _r (%)	
Top	ABLP001	1.554	59.2	59.2 (0.3, 0.4%)	ABPF030	1.509	53.8	54.3 (0.5, 0.9%)
	ABLP002	1.550	59.0		ABPF031	1.509	54.2	
	ABLP003	1.555	59.5		ABPF032	1.516	54.8	
Middle	ABLP004	1.542	58.0	57.7 (0.3, 0.5%)	ABPF010	1.508	54.9	54.4 (0.4, 0.8%)
	ABLP005	1.546	57.5		ABPF011	1.506	54.1	
	ABLP006	1.542	57.5		ABPF012	1.509	54.3	
Bottom	ABLP007	1.537	56.3	57.2 (0.8, 1.4%)	ABPF033	1.507	54.6	54.6 (0, 0%)
	ABLP008	1.548	57.8		ABPF034	1.516	54.6	
	ABLP009	1.543	57.6		ABPF035	1.515	54.6	
Panel mean, SD, CoV		1.546 (0.006, 0.4%)	58.0 (1.0, 1.8%)			1.511 (0.004, 0.3%)	54.4 (0.4, 0.7%)	

Image analysis results - optimised	V_f (%)	V_v (%)
Oven	53.4 (1.5, 2.8%)	2.7 (0.6, 22%)
Autoclave	55.3 (2.1, 3.8%)	1.5 (0.4, 27%)

Table 6: Double-Notch Shear Results - Standard Cure

Panel position	Oven			Autoclave		
	Specimen	Shear strength (MPa)		Specimen	Shear strength (MPa)	
Top	ABLN030	67.24	59.7 (5.43, 9.1 %)	ABPE001	64.23	61.6 (2.43, 3.9 %)
	ABLN031	51.92		ABPE002	62.69	
	ABLN032	60.06		ABPE003	60.84	
	ABLN033	60.09		ABPE004	58.60	
	ABLN034	59.06		-		
Middle	ABLN036	59.47	58.1 (2.35, 4.1 %)	ABPE005	57.87	56.6 (3.78, 6.7 %)
	ABLN037	59.75		ABPE006	55.22	
	ABLN038	56.16		ABPE007	52.12	
	ABLN039	60.68		ABPE008	61.00	
	ABLN040	57.90		-		
	ABLN041	54.55		-		
Bottom	ABLN042	58.79	59.2 (1.01, 1.7 %)	-		-
	ABLN043	58.86				
	ABLN044	60.94				
	ABLN045	58.48				
	ABLN046	58.66				
Panel mean, SD and CoV			58.9 (3.24, 5.5 %)			59.1 (3.99, 6.8 %)

Table 7: Double-Notch Shear Results - Optimised Cure

Panel position	Oven			Autoclave		
	Specimen	Shear strength (MPa)		Specimen	Shear strength (MPa)	
Top	ABLP030	58.08	56.0 (2.72, 4.9%)	ABPF001	52.56	56.4 (2.72, 4.8 %)
	ABLP031	57.16		ABPF002	56.74	
	ABLP032	57.55		ABPF003	58.96	
	ABLP033	55.64		ABPF004	57.30	
	ABLP034	51.37		-	-	
Middle	ABLP036	53.96	51.2 (4.25, 8.3%)	ABPF005	51.37	56.2 (5.38, 9.6 %)
	ABLP037	55.04		ABPF006	52.62	
	ABLP038	46.93		ABPF007	63.12	
	ABLP039	46.22		ABPF008	57.85	
	ABLP040	53.84		-	-	
Bottom	ABLP042	53.26	52.6 (0.95, 1.8%)	-		-
	ABLP043	52.15				
	ABLP044	53.49				
	ABLP045	52.97				
	ABLP046	51.17				
Panel mean, SD and CoV			53.3 (3.44, 6.5 %)			56.3 (3.95, 7.0 %)

Table 8: Compression Results - Standard Cure

Panel position	Oven					Autoclave				
	Specimen	Strength (MPa)		Modulus (GPa)		Specimen	Strength (MPa)		Modulus (GPa)	
Top	ABLN022	1073	1053 (75, 7.1 %)	108.0	109.6 (2.4, 2.2 %)	ABPE020	1304	1009 (260, 25%)	112.6	113.3 (0.7, 0.6%)
	ABLN023	1107		108.5		ABPE021	908		114.0	
	ABLN024	1054		112.4		ABPE022	814		113.4	
	ABLN025	1106		-		-	-			
	ABLN026	925		-		-	-			
Middle	ABLN018	1019	1038 (36, 3.5 %)	112.7	112.6 (1.5, 1.3 %)	ABPE023	1092	1120 (94.6, 8.4%)	112.6	112.2 (0.5, 0.5%)
	ABLN019	1026		111.0		ABPE024	1225		112.3	
	ABLN021	1045		114.0		ABPE025	1042		111.6	
	ABLN017	1096		-		-	-			
	ABLN020	1002		-		-	-			
Bottom	ABLN011	693	906 (144, 15.9 %)	107.9	109.6 (2.0, 1.8 %)	-				
	ABLN012	823		111.8						
	ABLN013	982		109.0						
	ABLN014	1002		-						
	ABLN015	1031		-						
Panel mean, SD and CoV			999 (112, 11.2%)		110.6 (2.3, 2.1%)		1064 (185, 17%)		112.8 (0.8, 0.7%)	

Table 9: Compression Results - Optimised Cure

Panel position	Oven					Autoclave				
	Specimen	Strength (MPa)		Modulus (GPa)		Specimen	Strength (MPa)		Modulus (GPa)	
Top	ABLP023	1112	1295 (107, 8.2 %)	111.7	110.5 (1.9, 1.7 %)	ABPF020	1076	1086 (8.7, 0.8%)	112.4	-
	ABLP024	1295		111.5		ABPF021	1090		-	
	ABLP025	1334		108.3		ABPF022	1092		115.1	
	ABLP026	1371		-		-	-			
	ABLP027	1364		-		-	-			
Middle	ABLP017	1141	1218 (56.9, 4.7 %)	113.5	114.6 (1.5, 1.3 %)	ABPF023	1015	1013 (2.6, 0.3%)	111.6	110.7 (1.9, 1.7%)
	ABLP018	1280		116.3		ABPF024	1010		108.6	
	ABLP019	1204		114.1		ABPF025	1014		112.0	
	ABLP020	1269		-		-	-			
	ABLP021	1198		-		-	-			
Bottom	ABLP011	1144	1314 (111, 8.4 %)	117.1	116.1 (1.9, 1.6 %)	-				
	ABLP012	1414		113.9						
	ABLP013	1266		-						
	ABLP014	1355		117.3						
	ABLP015	1393		-						
Panel mean, SD and CoV		1276 (97.6, 7.7%)			113.7 (2.9, 2.6%)		1050 (40, 3.8%)		111.9 (2.3, 2.1%)	

Table 10: In-Plane V-Notch Shear Results - Standard Cure

Panel position	Autoclave				
	Specimen	Strength (MPa)		Modulus (GPa)	
Top	ABPE016	100.5	101.5 (2.73, 2.7%)	4.65	4.68 (0.06, 1.2%)
	ABPE017	105.0		4.62	
	ABPE018	98.5		4.70	
Middle	ABPE019	101.9		4.75	
	-	-	-	-	-
	-	-		-	
Bottom	-	-	-	-	-
	-	-		-	
	-	-		-	
Panel mean, SD and CoV			101.5 (2.73, 2.7%)		4.68 (0.06, 1.2%)

Table 11: In-Plane V-Notch Shear Results - Optimised Cure

Panel position	Autoclave				
	Specimen	Strength (MPa)		Modulus (GPa)	
Top	ABPF016	97.4	96.7 (1.51, 1.6%)	4.68	4.76 (0.07, 1.5%)
	ABPF017	96.7		4.74	
	ABPF018	94.6		4.77	
Middle	ABPF019	98.1		4.85	
	-	-	-	-	
	-	-	-	-	
Bottom	-	-	-	-	-
	-	-		-	
	-	-		-	
Panel mean, SD and CoV			96.7 (1.51, 1.6%)		4.76 (0.07, 1.5%)

The strength and modulus results are detailed in Tables 10 and 11. It was observed that there were no salient differences between the standard and optimised panel results for either in-plane modulus or strength.

4.1.2.5 Glass transition temperature (T_g) by DSC

The glass transition temperature (T_g) of specimens extracted from standard and optimised cure cycle panels was measured according to ISO 11357. Part 2: 1999. (BS ISO 11357- 2: 1999.) - Determination of glass transition temperature [51]. Specimens were heated at 20°C/minute to 190°C followed by a repeat scan on the same sample. All DSC results were normalised with respect to the sample weight.

For the standard cure panel, little or no additional cure was observed from the DSC results (Figure 23) indicating that the standard cure panel was almost fully cured. However this degree of cure was only obtained in conjunction with the potentially damaging effects of significant exotherm, temperature variation throughout the panel thickness (which could result in cure gradients) and over-shoot in temperature above 120°C (Figure 18).

For the optimised cure cycle panel, a degree of additional cure was observed in the DSC results (Figure 24) indicating that full cure had not been achieved. However, the optimised cure cycle has advantages over the standard cure cycle in that the panel temperature does not exceed the recommended cure temperature and there are minimal differences between thermocouple readings throughout the panel thickness (Figure 19).

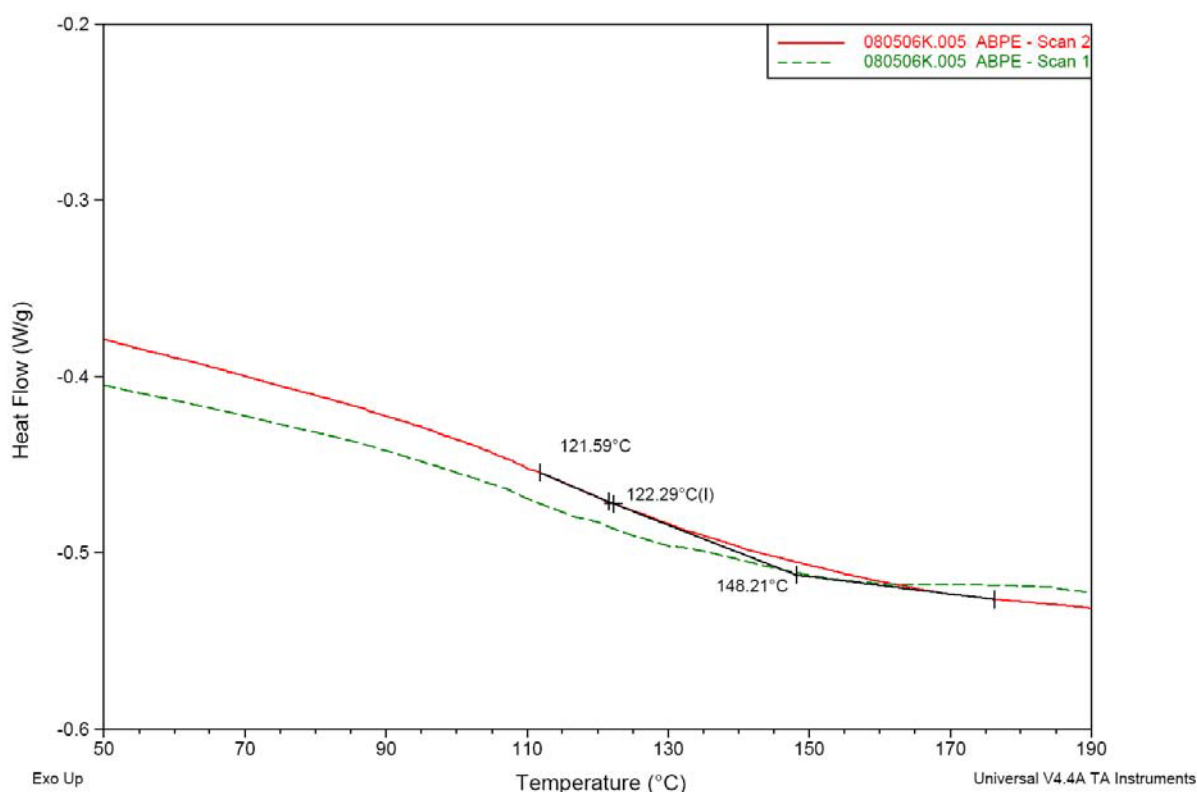


Figure 23: DSC results for standard cure, autoclaved material (N.B. weight normalised)

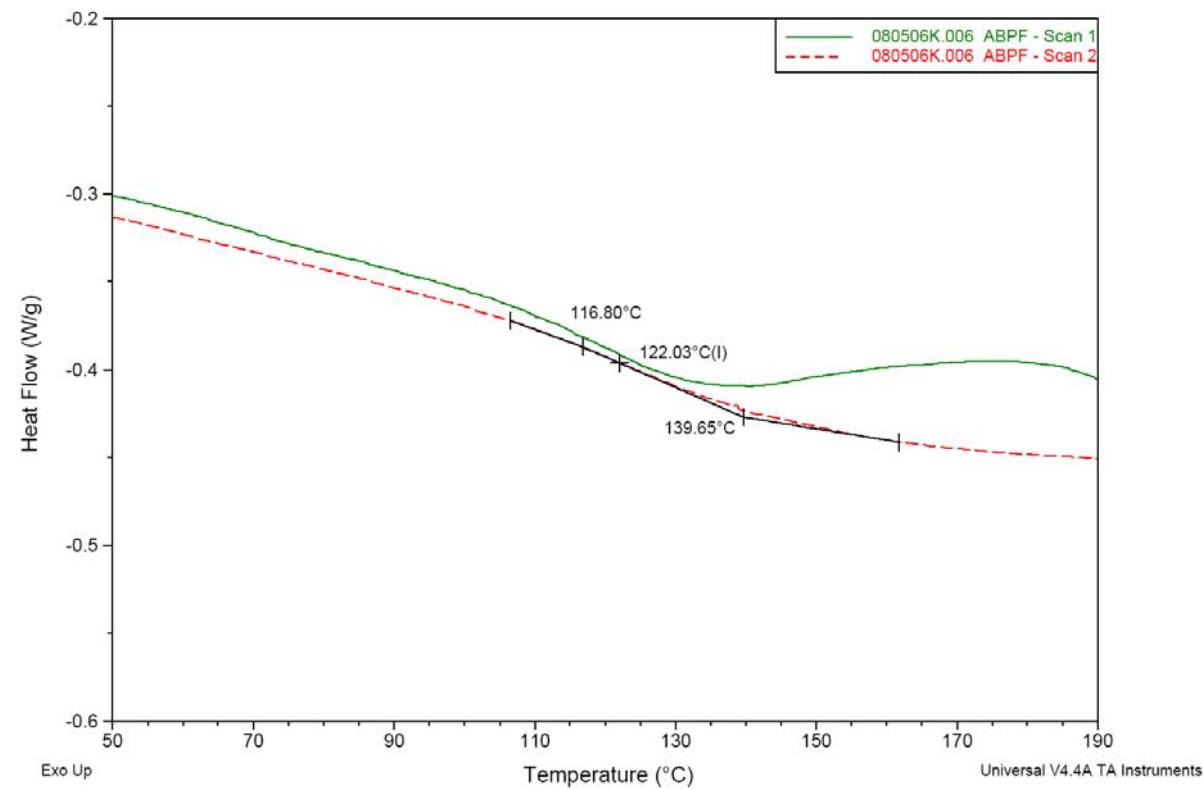


Figure 24: DSC results for optimised cure, autoclaved material (N.B. weight normalised)

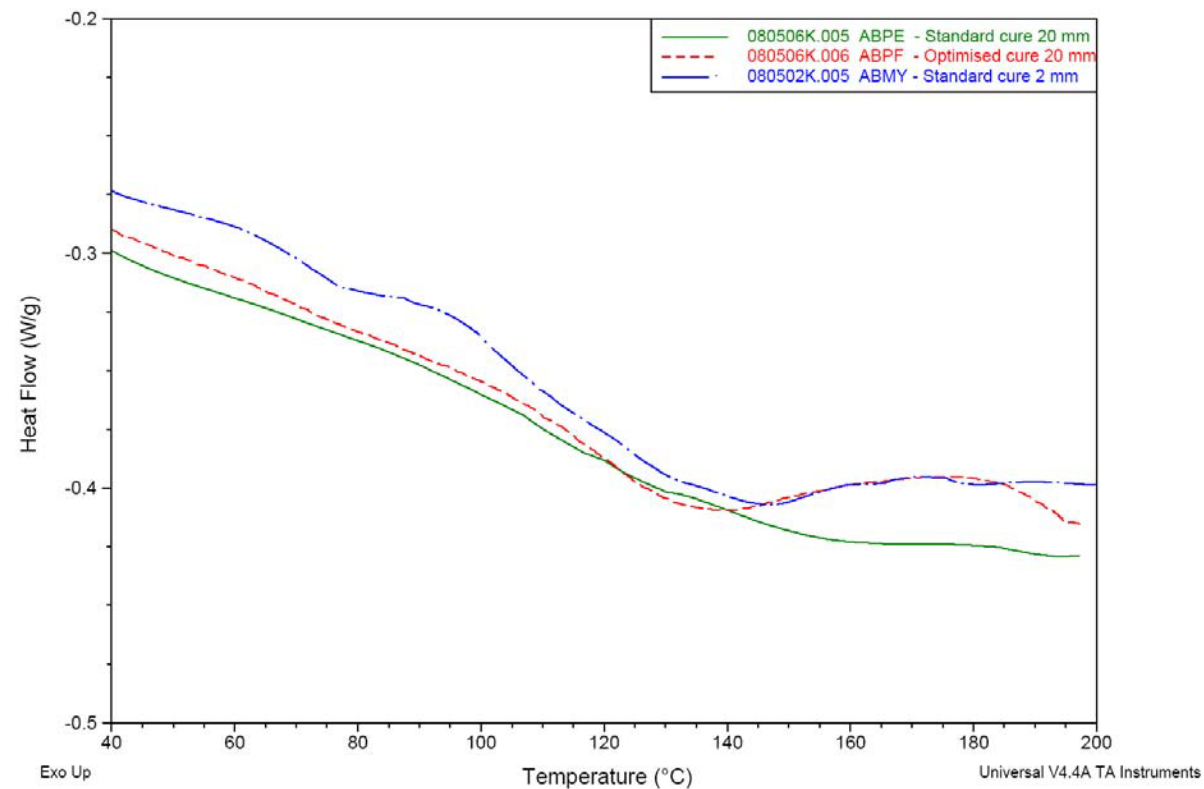


Figure 25: Comparison of weight normalised DSC results for standard cure thick (20 mm) and thin (2 mm) panels, and optimised cure thick (20 mm) panel.

The glass transition temperature, T_g values from the repeat scans for the standard and optimised cure cycles were in close agreement at 122.29°C and 122.03°C respectively.

In addition to the two 20 mm thick materials tested (standard and optimised cure cycles), DSC tests on a sample of previously manufactured 2 mm thick material, cured in the autoclave using the material supplier's recommended standard cure cycle, were also undertaken. This was carried out to compare the degree of cure in previously manufactured thin laminates to that seen in the two thick laminates via the standard and optimised cure processes. From Figure 25 it can be seen that the degree of cure in the 2 mm thick panel is similar to that seen in the optimised cure thick panel. This finding means that although the material may not be fully cured using the optimised cure cycle, the similarity in degree of cure will allow valid comparisons of thick and thin mechanical properties to be undertaken later in the project i.e. any differences in mechanical properties will be as a result of either scaling effects, specimen geometry and/or mechanical test design rather than as a result of cure effects. It is worth reiterating that although the DSC results indicate differences in cure between the standard and optimised cure cycles, there is little evidence to suggest that this has an affect on the mechanical performance of the material.

4.1.2.6 Residual strain measurement by incremental slotting

Residual stresses/strains can occur in thick panels as a result of non-uniform heating and cooling of the laminate due to temperature gradients during processing. In this study, a compliance technique (detailed in [52]) has been used to evaluate the degree of residual strain in standard and optimised cure panels. The method is based on the principle that if a slot is incrementally machined into a thick section then any residual stresses will be progressively released in the material along the plane of the slot. Release of residual stresses will result in deformation of the material. By bonding a strain gauge onto the back face of the specimen this deformation can be measured and can provide an indication of the degree and polarity of the residual strains within the material. For both standard and optimised, autoclave cured panels, incremental slotting tests were performed on two specimens (60 mm long x 20 mm wide by 20 mm thick); one specimen slotted transverse to the fibre direction and the other slotted parallel to the fibre direction (Figure 26(a) and (b)). A 5 mm gauge length strain gauge was bonded to the back face of the specimen coincident with the specimen longitudinal axis (Figure 27). Specimens were clamped along their length as shown in Figure 27 and slotted using a 2 mm diameter 3 Flute slotdrill (i.e. the slot width was 2 mm). Each slot increment was 0.25 mm in depth. After each slot increment had been

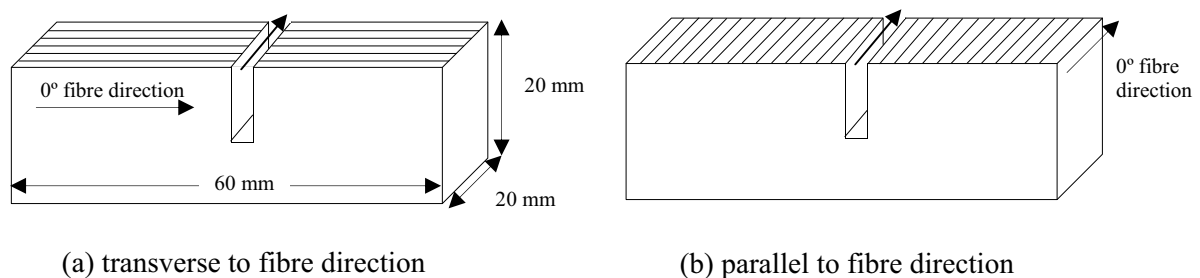


Figure 26: (a) transverse and (b) parallel incremental slotting directions

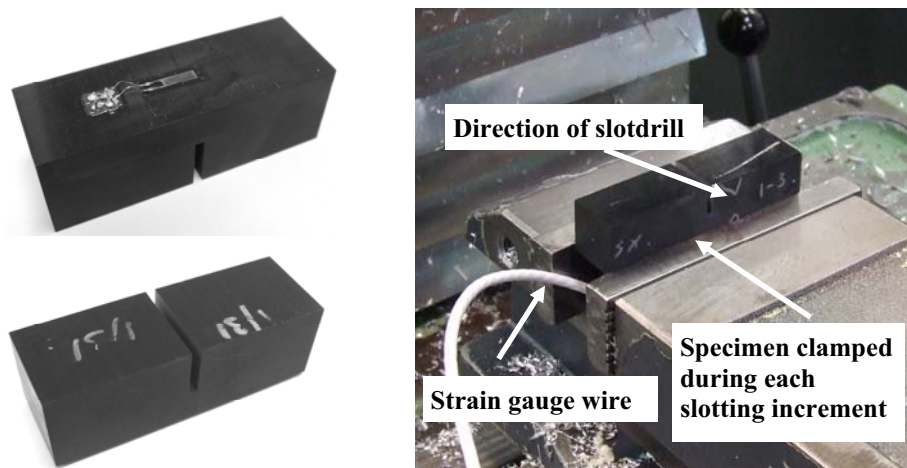


Figure 27: Slotted specimens and method of clamping

machined the specimen was unclamped and left to relax for 10 minutes before a strain gauge reading was recorded. The specimen was then re-clamped in identical fashion, before the next increment was machined. This process was repeated until a slot depth of ~12 mm (maximum possible with slotdrill used) had been machined.

Figure 28 shows the results of the incremental slotting tests for measurement of residual strain. The first observation to be made is that the residual strains in the transverse slot direction i.e. across the fibres, were much lower than for the parallel direction. This is as expected as residual strains/stresses are predominantly present in the resin, hence cutting parallel to the fibres will result in a larger degree of residual strain/stress being released than if cut across the fibres. The second observation is that a larger degree of residual strain release was seen in the standard cure panel than for the optimised cured panel. As the measured strain is positive this would indicate the presence of compressive residual stresses within the material. The compliance method detailed in [52] provides a method by which the residual stresses can be derived from the measured strains, however this has not been undertaken here due to the complexity of the analysis and the time available within the project.

4.1.3 Effect of cure cycle - conclusions

From the cure optimisation work, and physical, mechanical and thermal tests detailed, a number of conclusions have been drawn as to the effects of different autoclave cure cycles on the SE84LV material:

- Large temperature overshoots (due to excessive exotherm) and intra-panel temperature variations were observed when using the standard, 'thin' cure cycle for curing of 20 mm thick laminates,
- The use of an 'optimised' cure cycle resulted in no temperature overshoot (above recommended cure temperature) and little or no intra-panel temperature variation,
- There were no significant differences observed in the physical and mechanical properties between the standard and optimised cure cycle panels. This was reasoned to be due to the 'forgiving' nature of the material system used and it's lack of susceptibility to cure temperatures in excess of 30 °C above the recommended cure temperature,

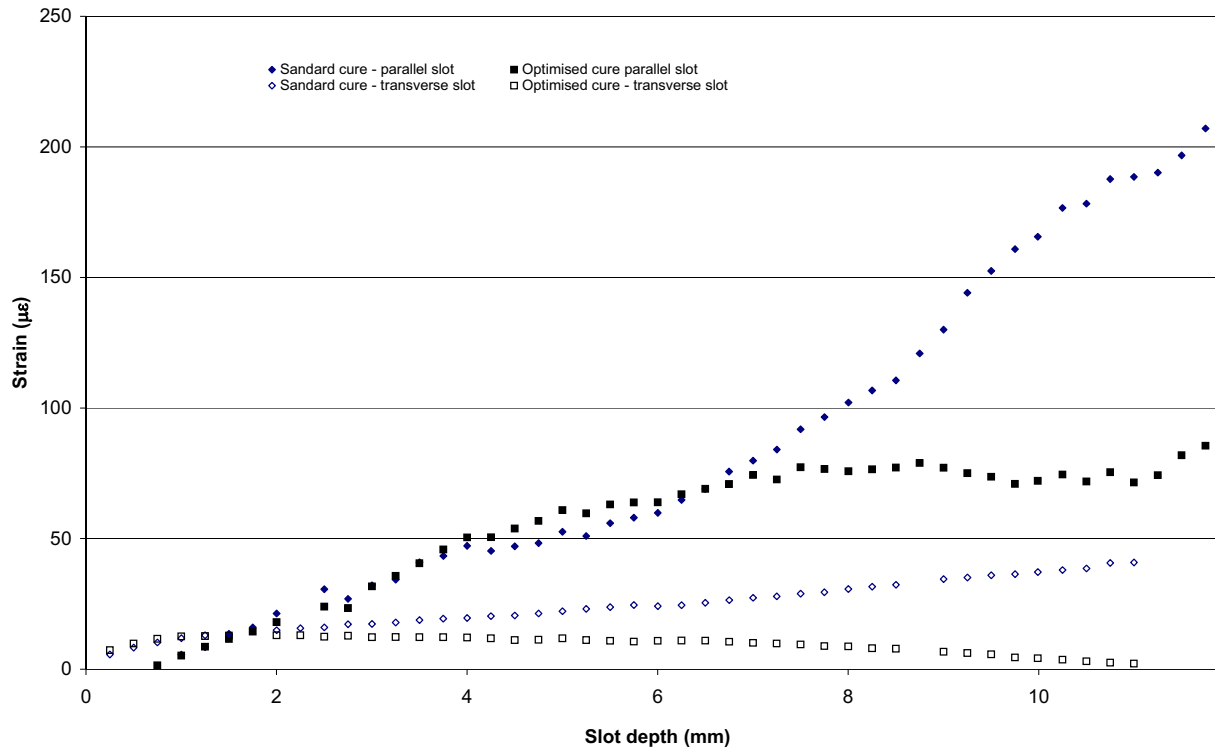


Figure 28: Transverse and parallel (to fibres) incremental slotting results for measurement of residual strain

- The DSC results indicated that there was little additional cure occurring in the standard cure panel, but a degree of additional cure was observed in the optimised cure laminate. Although the optimised cure panel was not completely cured, the cure cycle has the advantages that large degrees of exotherm and panel temperature variations (inherent with the standard cure cycle) are avoided,
- The degree of cure in a sample of 2 mm thick material cured using the standard cure cycle is similar to that seen in the optimised cure thick panel. Therefore, although the material may not be fully cured using the optimised cure cycle, the similarity in degree of cure (to that seen in thin material) will allow valid comparisons of thick and thin mechanical properties to be undertaken later in the project,
- Residual strain measurements showed that the degree of residual strain in the standard cure panels was higher than for the optimised cure panels. This could be due to the optimised cure panel not being fully cured, but is more likely to be as a result of the larger difference between peak cure and room temperature (ΔT) seen by the standard cure panel as a result of excessive and uncontrolled exotherm.

5 DISCUSSION AND CONCLUSIONS

As has been seen in Section 4, when characterising the mechanical performance of thick laminates it is important to ensure that differences in the measured mechanical response between thin and thick sections are purely as a result of scaling/sizing effects (if indeed they do exist) and not as a result of the cure or processing history of the material. 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. 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.

A major 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 compressive 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 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 45° 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. a 'sandwich' structure consisting of several laminates separated by release films).

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 specific to the SM07 research programme are:

Development of measurement protocols for characterising the static tensile and compressive mechanical behaviour of thick composite material sections, including

- 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” ($[+45^\circ/0^\circ/-45^\circ/90^\circ]_{ns}$) and “blocked” ($[+45^\circ_n/0^\circ_n/-45^\circ_n/90^\circ_n]_s$) quasi-isotropic carbon/epoxy laminates of different thicknesses (2 to 20 mm), guidance on panel consolidation and processing to be taken from Section 4 of this report.
- 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.

Related future work needed on thick composites is summarised below.

Extended property assessment

- Extension of static in-plane tension and compression test methods/standards to characterising composite behaviour under creep and fatigue loading conditions. This work would need to be extended to high rates, elevated temperatures and hostile environments, and include assessment of defect criticality.
- 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.
- 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.

Processing and quality assurance

- Better understanding of cure and associated effects through real-time cure monitoring techniques and standards for on-line processing of both thin and thick 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.
- Non-destructive evaluation (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 of evaluating residual stresses and through-thickness cure gradients.

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