

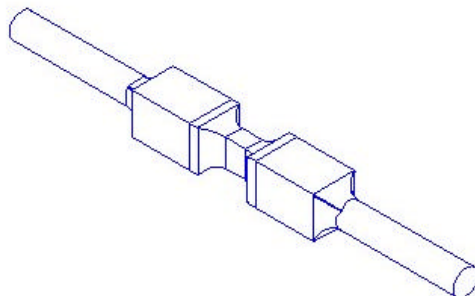
Through-Thickness Tensile Testing of Polymer Matrix Composites

Summary

This Measurement Note details work undertaken to improve through-thickness testing of polymer matrix composites. The objective was to recommend revisions to the current draft test method on the “Determination of Through-Thickness Tensile Properties of Fibre Reinforced Plastic Composites” to support its proposal as an ISO new work item.

The Measurement Note provides details on the design and use of a bonding alignment rig for the manufacture of specimens, with supporting experimental evidence. Investigations into the possibility of reducing the required material thickness below the current 40 mm level are also discussed. These include linearly scaling down the standard specimen geometry and novel flexure and punch-out specimen and test configurations.

The work presented here indicates marked improvements in the consistency of specimen manufacture using the alignment rig and resultant reductions in test scatter. In addition, the scaled down test specimens proved successful alternatives where thicker material was unavailable, however the novel geometries proved to be unsuitable candidates to replace the standard method.



R M Shaw and G D Sims

October 2004

INTRODUCTION

Structural designers and engineers are increasingly required to know the through-thickness properties of laminated composite components. This information is needed to ensure the safe design of primary and secondary structures, performed using finite element analysis. This data is important due to the complex 3D stress states that can occur in laminated composites especially around stress concentrations i.e. flanges, edges, holes or joints. Through-thickness properties are largely dominated by the matrix causing the out-of-plane strength of the material to be many times lower than that of the in-plane. If this factor is not taken into account it can lead to premature failure of a component by means of interlamina or delamination cracks.

It is a common misconception that through-thickness stresses are only an issue for “thick” sections. They can, however, occur in all laminated structures irrespective of thickness or loading. Thus all components are susceptible to problems as a result of through-thickness stresses.

Work on through thickness test methods has been conducted by several organisations ^[1,2], leading to the development of a multitude of different methods for obtaining the through-thickness properties of polymer matrix composites (PMCs). A review of these test methods was conducted previously by NPL ^[3].

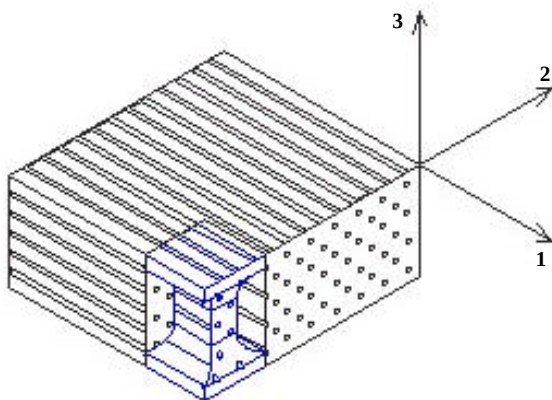


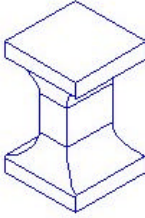
Figure 1 - Symmetry axes for laminate, showing cutting orientation of specimen Type C.

Previous work has shown that identical specimen geometries are suitable for generating through-thickness data in both compressive and tensile modes, an example of which is shown in Figure 1. For the compression test, accurate machining of the specimen and good alignment of the flat loading platens results in low coefficients of variation being obtained, (i.e. $< 2\%$, on good quality materials). This is not the case, however, in tension. The requirement to bond loading arms onto the specimen to enable tensile loading leads to difficulties in terms of both bond strength and also alignment ^[4].

Work conducted by NPL under previous programs, Materials Measurements Program (MMP) and New Materials Work (NMW), highlighted the following issues ^[5,6].

- Difficulties in obtaining materials of sufficient thickness. Although thick material is widely used in industry for specific applications, it is not always readily available, or even appropriate to many applications.
- Load introduction into the specimen. Specimen misalignment, during bonding of the loading bars, causes specimens to fail prematurely during testing. This can also lead to specimens failing during the pre-test clamping stage. The adhesive bond strength was also found to be an issue, with specimens failing incorrectly at the bond line and not within the specimen gauge length.
- Specimen machining and preparation costs. Samples need to be precision ground due to the critical tolerance required on parallelism of the loading/bonding faces. This causes the test to be relatively expensive when compared to other tests/geometries.
- Specimen failure is defect dominated in tension. This can lead to high scatter in the test data.
- Non-uniform stress field, stress concentrations near specimen ends. The specimen needs a region of pure uniform maximum stress to ensure repeatability.

Table 1 - Details of the principal specimen types.

Specimen Type	Details	
A		Description: Plain rectangular block, for elastic properties only. Requires 0°/90° strain gauge rosette aligned on all axial faces, with an active length of 2 mm. Dimensions: 16 x 16 x 40 mm height, suitable heights range from 18 to 40 mm.
B		Description: Plain waisted block, with square cross section and plain radius across the gauge length, for strength measurement only. Dimensions: 25 x 25 x 40 mm height, with a gauge cross-section of 16 x 16 mm. Specimen can be scaled down linearly to a height of 20 mm.
C		Description: Waisted block giving a flat 12 mm gauge length with rectangular cross section. Capable of measuring both elastic and strength properties. Requires 0°/90° strain gauge rosette aligned on all axial faces, with an active length of 2 mm. Dimensions: 25 x 25 x 40 mm height, with a gauge cross section of 16 mm (1-3 direction) and 10 mm (2-3 direction) Specimen can be scaled linearly down to a height of 20 mm.

For this reason further work was conducted to improve the test method for the determination of through-thickness tensile properties of fibre reinforced plastic composites^[7].

CURRENT DRAFT TEST METHOD

The draft test procedure accommodates three main types of specimen geometry, see Table 1, that have been developed for through thickness testing of thermoset resin based PMC's. Due to severe bonding difficulties, alternative methods are required for thermoplastics^[5].

Table 2 - Properties obtained from different specimen types.

Specimen Type	E ₃₃	v ₃₁	v ₃₂	S _{33M}	e _{33M}
A	✓	✓	✓		
B				✓	
C	✓	✓	✓	✓	✓

The measurable properties from each of these types are given in Table 2.

In all cases, the standard specimen height is 40 mm, but there are alternative options available:

- Specimen Type A can use height ranges from 18 to 40 mm,
- Specimens Type B and C can be scaled linearly down to a height of 20 mm, however, data is only available for miniature Type C specimens in compression.

In order to check for alignment throughout the test, a strain gauge is bonded on each face of Type A and C specimens in the mid-plane of the gauge length, aligned axially to the loading direction. For these specimens a biaxial 0°/90° strain gauge rosette is normally used to measure both the axial and transverse strains. Due to the geometry of the Type B specimen, it is not possible to monitor strain accurately using strain gauges.

Acceptable bending can then be calculated using the equations:

$$\left[\frac{\epsilon_{33b} - \epsilon_{33a}}{\epsilon_{33b} + \epsilon_{33a}} \right] \leq 0.10 ; \left[\frac{\epsilon_{33d} - \epsilon_{33c}}{\epsilon_{33d} + \epsilon_{33c}} \right] \leq 0.10$$

Where ϵ_{33a} and ϵ_{33b} represent one set of axial gauges on opposite faces of the specimen and ϵ_{33c} and ϵ_{33d} are the remaining pair of opposing axial strains.

SPECIMEN PREPARATION

Specimen Machining

The nature of the material and the tolerances on parallelism required of the bonding/loading faces require specimens to be machined using a surface grinding machine. All specimens tested were machined using a PA60 standard pink grinding wheel at a spindle speed and feed rate of 2700 rpm and 7500 mm/min, respectively.

Specimen Bonding and Alignment

Due to the apparent misalignment sensitivity of the tensile specimen, it was necessary to develop a method that allowed for the repeatable and accurate manufacture of test specimens. An alignment rig was developed that enables the specimen and loading bars to be aligned axially and held rigidly in place during the bonding process.

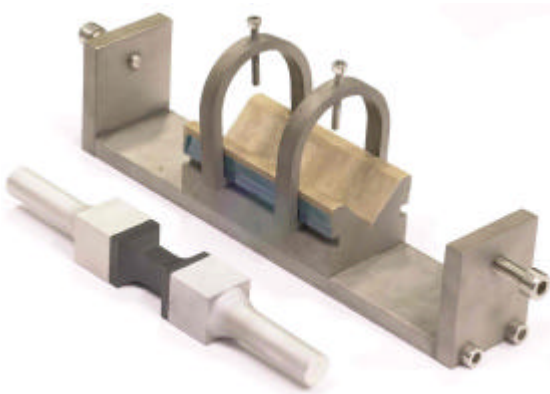


Figure 2 - Bonding alignment rig, shown with assembled specimen.

Figure 2 shows the alignment rig which was used in this work. The loading arms and specimen are aligned by means of a 'v' block and then held in place with two cross clamps. Bolts are used at both ends to apply an axial load, of 3 Nm, to the specimen during curing of the adhesive to produce a stronger bond.

All specimens were bonded using a Redux 312 film adhesive. The typical cure for this adhesive is 30 minutes at 120°C, but due to the additional thermal mass of the bonding alignment rig the cure time was extended to 90 minutes.

EXPERIMENTAL WORK

Test Details

In accordance with the draft test procedure, tests were conducted using a calibrated Instron 4500 series screw driven mechanical test machine. Tensile loading of the specimen was performed using either mechanical wedge action grips or by pin loading the specimen using standard pin fittings. Tests were conducted on batches of between three and five specimens. The specimens were loaded axially in tension at a displacement rate of 1 mm/min.

Data Capture and Analysis

Biaxial 0°/90° strain gauge rosettes with an active gauge of 2 mm were applied to the mid plane on all axial faces of each specimen, using manufacturer recommended adhesive.

Strain gauge conditioning and test data acquisition were conducted using an ELE MM700 Autonomous Data Acquisition Unit (ADU) recording at a rate of 5 Hz. Data were subsequently analyzed by excel spreadsheet.

The through-thickness strength, s_{33TM} (MPa), is calculated using the equation:

$$\sigma_{33TM} = \frac{F}{ab}$$

where:

- F is the maximum load, (N);
 a is the specimen width, (mm);
 b is the specimen thickness, (mm).

The tensile modulus, E_{33T} (MPa), is calculated using the equation:

$$E_{33T} = \frac{\sigma_T'' - \sigma_T'}{\epsilon_T'' - \epsilon_T'}$$

where:

- σ_T' is the tensile stress at $\epsilon' = 0.0005$, (MPa);
 σ_T'' is the tensile stress at $\epsilon'' = 0.0025$, (MPa).

Machine Alignment

When using mechanical wedge action grips it is recommended that the alignment of the test machine is checked, by using the method detailed in Annex B of ISO 527-5 'Determination of Tensile Properties'^[8]. This enables an accurate assessment of the system alignment before embarking on testing.

Results from this check, shown in Figure 3, prove the alignment of the loading train to be within acceptable bending limits.

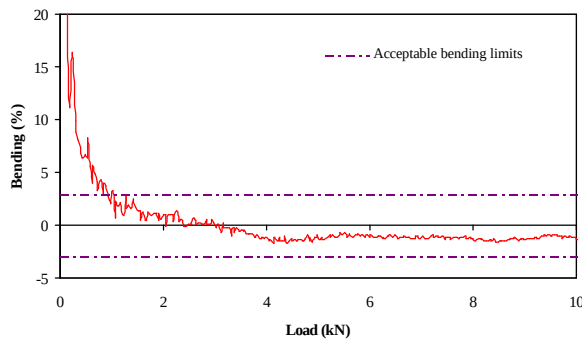


Figure 3 - Test machine alignment as measured using wedge action grips.

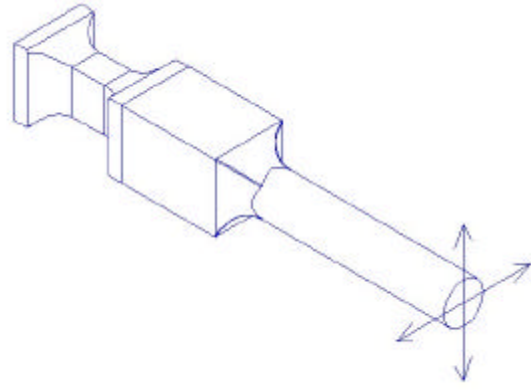


Figure 4 - Cylindrical loading arm geometry.

Specimen Alignment

In order to assess the problems with alignment, firstly the method of specimen manufacture was investigated using the newly designed alignment rig. Specimens were manufactured from two materials, donated by industry:

- Material 1: Woven glass fibre/epoxy, press moulded.
- Material 2: Unidirectional carbon fibre/epoxy, press moulded (CFRP).

The Type C specimen was chosen for this evaluation as it was capable of providing the full range of through-thickness data, including strain to failure. Specimens were bonded to cylindrical loading arms, as shown in Figure 4. Bonding and testing was conducted as described previously.

Alignment - Results

It was noted during clamping of the specimen in the machine wedge grips prior to testing, that it was possible to introduce a load of up to 1500 N in the specimen if sufficient care was not taken.

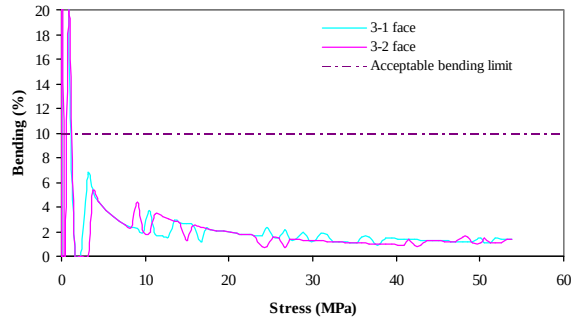


Figure 5 - Plot of stress against average bending on opposing specimen faces; standard Type C specimen.

During analysis of the data it was found that it was not possible to achieve bending below the acceptable level for the entire duration of the test. This was due to the initial settling of the gauge readings, shown in Figure 5, at the beginning of any test. Maximum bending values quoted are therefore taken between 20 and 80% of the ultimate stress. The latter value was chosen to avoid any issues related to gauges failing prior to specimen failure and producing inconsistent strain readings, a not uncommon scenario in materials achieving high failure strains.

Through-thickness strengths and elastic properties of the two materials tested are detailed in Table 3. Typical plots detailing the response of the strain gauges throughout the test for each of the two materials are shown in Figure 6 (a) and (b). It can be seen from these traces that the axial strain gauges on opposing faces have good alignment throughout the test. Alternatively, this same data can be represented as in Figure 7.

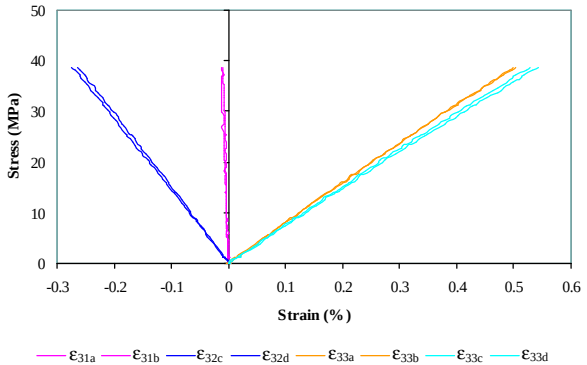
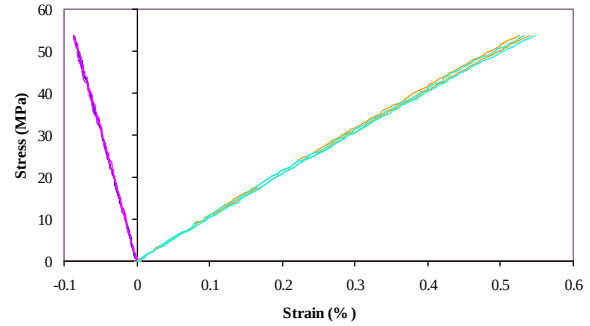


Figure 6 - Typical strain gauge response: (a) material 1 and (b) material 2; standard Type C specimen.

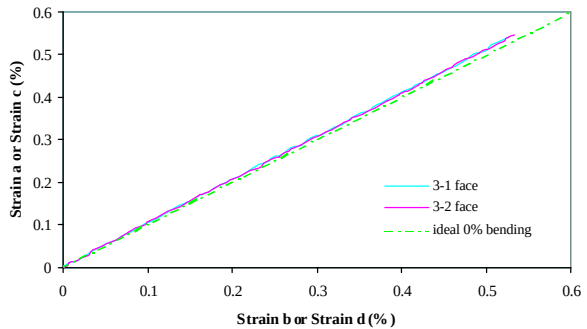


Figure 7 - Plot of axial strain from opposing faces, standard Type C specimen.

Table 3 - Through thickness results for standard Type C specimens.

Property Specimen	E_{33} (GPa)	η_{31}	η_{32}	S_{33TM} (MPa)	ϵ_{33TM} (%)
Material 1	10.78 10.81	0.170 0.165	0.170 0.179	59.85 52.11	0.57 0.49
	10.96 10.73	0.173 0.171	0.178 0.173	55.24 58.78	0.53 0.56
	10.33	0.161	0.167	54.54	0.54
Mean $\pm$ St Dev	10.72 $\pm$ 0.23	0.167 $\pm$ 0.005	0.173 $\pm$ 0.005	56.10 $\pm$ 3.17	0.54 $\pm$ 0.032
Material 2	7.75 7.69	0.002 0.013	0.510 0.497	40.14 24.64	0.39 0.32
	7.88 7.60	0.018 0.022	0.519 0.508	44.08 44.85	0.58 0.61
	7.82	0.013	0.508	40.20	0.52
Mean $\pm$ St Dev	7.75 $\pm$ 0.11	0.017 $\pm$ 0.004	0.508 $\pm$ 0.008	38.78 $\pm$ 8.20	0.48 $\pm$ 0.12

Figure 8 presents a comparison of the maximum bending values for each specimen on the 3-1 and 3-2 faces with respect to the failure stress, lines denote data from individual specimens. All specimens fall comfortably within the 10% bending criterion set out in the test method.

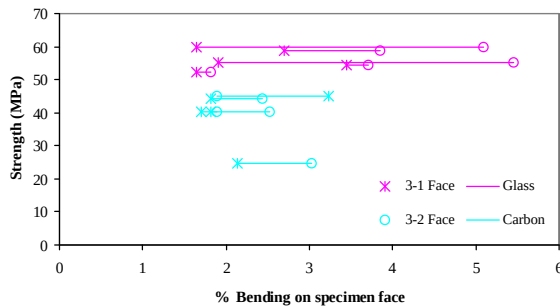


Figure 8 - Plot of % bending against failure stress for opposing pairs of specimen faces; standard Type C specimen.

Alignment - Conclusions

The data obtained using the novel alignment rig suggests:

- The results presented show improvement as compared to earlier test data. Previously, unacceptable failures were not uncommon, caused either by loading arms de-bonding or bonding/loading misalignment causing specimens to fail before testing. No such problem occurred during these tests.
- The scatter within the test data has been greatly improved, giving a coefficient of variation of less than 10% for material 1. The higher scatter in material 2 strength may be attributable to the high void content observed in the material.
- A recommendation suggesting the use of an alignment rig for the manufacture of these test specimens should be added to the current draft standard.
- The re-usable steel loading arms were problematic during the test set-up and added considerable weight to the assembled specimen.

Minimise Material Thickness

Due to problems with sourcing thick materials, ~ 40 mm, alternative methods were investigated to minimise the thickness of material necessary to obtain through-thickness data experimentally. There was little evidence of this issue being addressed in the literature, especially for T-T tension.

Two aspects were investigated:

- Possibility of obtaining suitable test data from miniature Type C specimens.
- Investigating other novel test geometries.

Miniaturised Specimen

Tests were conducted on a miniature version of the Type C specimen. Figure 9 shows a diagram of the scaled down specimen dimensions. The loads which were seen to be applied previously during clamping were found to be close to the calculated failure loads of these miniaturised specimens. It was therefore necessary to adopt a novel approach to load introduction into the specimen.

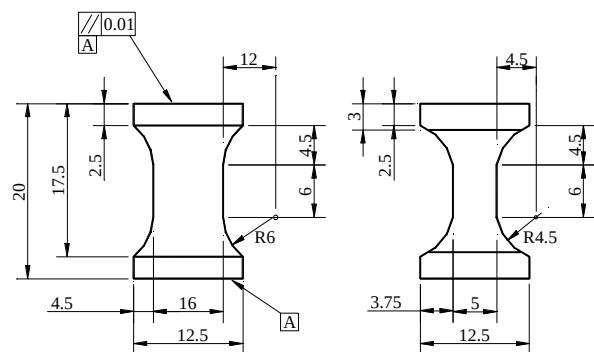


Figure 9 - Schematic of type C miniaturised specimen geometry.



Figure 10 - Assembled miniature specimen with loading arms.

One suggested method employs pin loading, an assembled specimen being shown in Figure 10. In order to achieve this, rectangular blocks were ground with a 6 mm thread tapped in one end, which were then bonded to the specimen using the alignment rig. Once bonded, circular rod ends were screwed into the tapped holes. The idea behind using these loading ends was the self-alignment capability during testing. The same materials as before were used for the investigation. Due to the reduced size of the specimen it was necessary to use smaller biaxial 0°/90° strain gauge rosettes with an active gauge of only 1 mm.

Miniaturised Specimen - Results

The through-thickness strengths and elastic properties of the miniature specimens are detailed in Table 4. Typical through-thickness tensile failures are shown in Figure 11.



Figure 11 - Typical Type C specimen failures.

Figure 12 shows a plot of the maximum bending values for each specimen on the 3-1 and 3-2 faces with respect to the final failure stress, lines denote individual specimens. It can be seen immediately from the data that acceptable levels of bending, as set out in the draft procedure, were not achieved on several specimens. Figure 13 shows a typical strain trace from a specimen with high bending, the axial gauges on opposing faces are plotted against each other with the ideal 0% bending response is denoted by the dashed line. This same data can be represented as seen in Figure 14, where the bending data is plotted against applied stress.

Table 4 - Through thickness results for miniature Type C specimens.

Property Specimen	E_{33} (GPa)	n_{31}	n_{32}	S_{33TM} (MPa)	ϵ_{33TM} (%)
Material 1	11.88 11.53	0.173 0.174	0.170 0.179	70.69 63.24	0.64 0.58
	11.34 11.89	0.177 0.185	0.178 0.173	66.23 71.03	0.61 0.47
	11.45	0.173	0.167	63.44	0.59
Mean $\pm$ St Dev	11.62 $\pm$ 0.26	0.176 $\pm$ 0.005	0.173 $\pm$ 0.008	66.93 $\pm$ 3.73	0.58 $\pm$ 0.07
Material 2	7.25 7.60	0.004 0.016	0.503 0.500	43.46 53.42	0.62 0.75
	7.72 7.00	0.018 0.013	0.392 0.472	43.65 41.65	0.57 0.60
Mean $\pm$ St Dev	7.39 $\pm$ 0.33	0.013 $\pm$ 0.006	0.467 $\pm$ 0.052	45.55 $\pm$ 5.32	0.64 $\pm$ 0.08

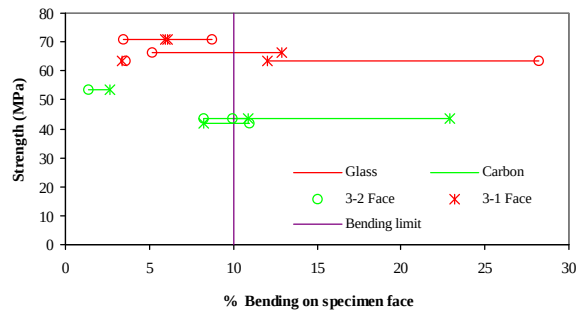


Figure 12 - Plot of % bending against failure stress for opposing pairs of specimen faces; miniature Type C specimen.

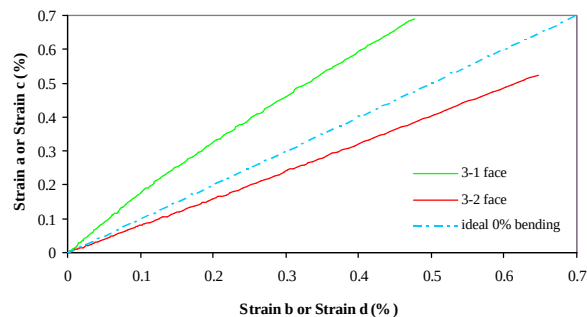


Figure 13 - Plot of axial strain from opposing faces, miniature Type C specimen.

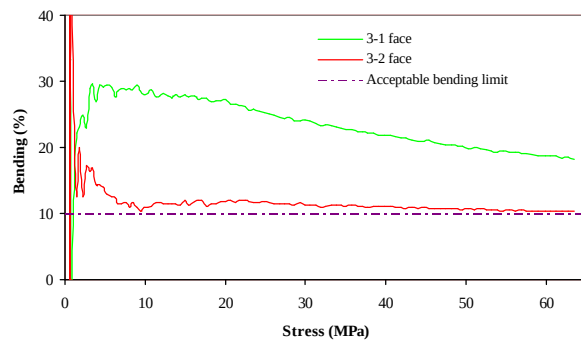


Figure 14 - Plot of strength against average bending on opposing specimen faces; miniature Type C specimen.

Miniaturised Specimen - Conclusions

Data obtained from the miniaturised specimens suggests:

- The method of specimen loading is still an issue. Although the results showed similar or improved coefficients of variation compared to the standard size specimen, the amount of bending induced on the smaller specimen during the test was found to be extremely high in some cases.

Table 5 - Failure data comparison: full size and miniature specimens.

Material	Type	S_{33TM} (MPa)	e_{33TM} (%)
1	Std	56.10 ± 3.17	0.54 ± 0.03
	Mini	66.93 ± 3.73	0.58 ± 0.07
2	Std	38.78 ± 8.2	0.48 ± 0.12
	Mini	45.55 ± 5.32	0.64 ± 0.08

- Failure values are almost 20% higher as compared to the standard geometry, see Table 5. This could be due to one of two causes: 1) tensile failure being defect dominated and 2) end-loading blocks having an unknown detrimental effect on the standard specimen.

Novel Specimens

Three novel specimen geometries were investigated in an attempt to reduce the need for thick materials for through-thickness property determination.

Punch-Out Test

Figure 15 illustrates two related methods requiring either concentric rings or parallel slots to be machined through to the mid-plane of the specimen from both faces. A compressive load is then applied to the top central surface of the material causing the central section to “punch out”.

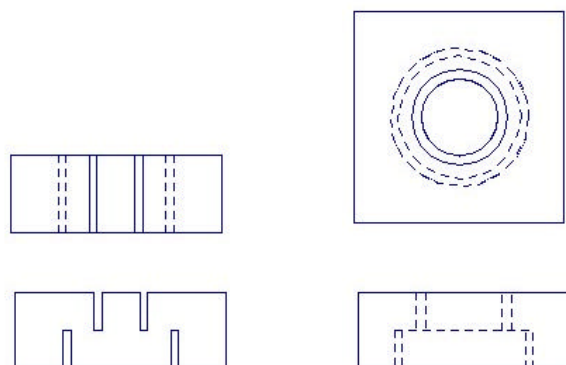


Figure 15 - Punch tests: (a) straight parallel and (b) concentric circular slots.

Table 6 - Through-thickness strength results for straight slotted punch-out specimens.

Width 1 (mm)	Width 2 (mm)	Thickness (mm)	Slot depth	s (MPa)
14	21	5	Mid-plane	30.50
			1 mm past mid-plane	27.34
14	21	21	Mid-plane	36.43
			1 mm past mid-plane	31.92

Failure occurs in the region between the cut faces. Tests were conducted at 0.5 mm/min on material 1, with a range of different separations between the slots. Different slot depths were also investigated for the straight slot test.

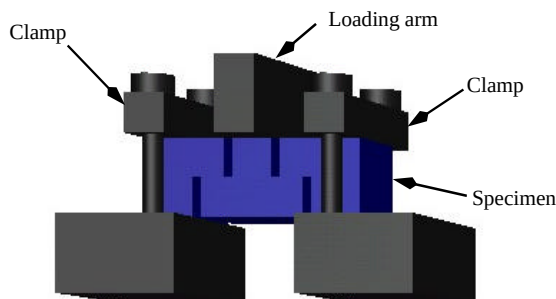
Punch-out test - results

It was seen early on that specimen bending was influencing the test failure and measured results, so a method was developed to restrain the top surface to try to remove any possible bending. This set-up is illustrated in Figure 16. Measured failure strengths are given in Table 6 for circular slots and Table 7 for straight slots.

Punch-out test - conclusions

Data obtained from this method suggests the following:

- Machining the slot depths and holes in the punch-out test accurately is critical, this causes the test to be more complex than desired. Where the depths were not accurate or machined flat, a correct failure was not achieved. i.e. failure was not in tension across a single plane.

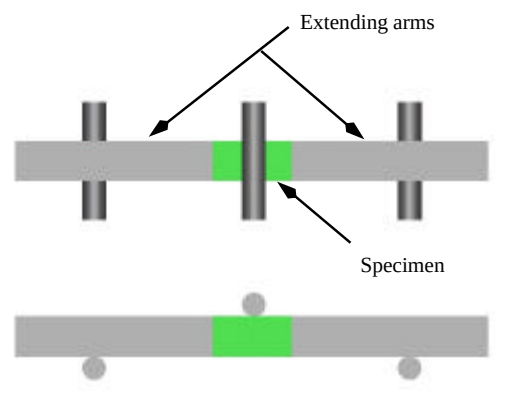
**Figure 16 - Punch-out specimen restrained loading configuration.****Table 7 - Through-thickness strength results for circular slotted punch-out specimens.**

Overlap (mm)	Inner ϕ (mm)	Outer ϕ (mm)	Thickness (mm)	s (MPa)
3	25	31	4	22.02 ± 0.52
6	25	37	4	11.60 ± 0.53

- Only suitable for testing the specific plane of the material that the slots were machine through to, so this test would not be suitable for non-laminated composites. However it can be used to investigate different layers in a non-homogeneous material.
- Failure strength is the only property which can be obtained from this test. However, the strength results did not correspond with previous data obtained from the same material. Possibly due to the large interlamina shear component.

Flexure Test

Figure 17 shows a simple three-point flexure test specimen configuration. Due to the required specimen length, it was necessary to build up the material of interest by bonding steel extension arms on either side. Tests were conducted at 1 mm/min, using 5 mm loading rollers and an initial test span of 50 mm^[9]. Both materials, ranging in thickness from 5 to 40 mm, were investigated.

**Figure 17 - Flexural test specimen and loading configuration.**

Flexure test - results

Strengths are given in Table 8 for those tests which produced acceptable failures. Tests on thinner materials were also conducted at different test spans, but failed to achieve either correct or repeatable failure modes.

Due to the use of bonded steel extension bars, it was essential to derive expressions to determine the maximum flexural stresses within the central composite section.

Flexure test - conclusions

Data obtained from this method suggests:

- It was difficult to obtain acceptable failure modes for materials under 20 mm in thickness due to bond failures at the interface with the extension arms.
- Results were not found to be comparable with any previous data on the same materials. In this case they were much higher than in the direct tension tests, which is likely to be the general effect of flexural testing reducing the sensitivity to defects due to the smaller volume tested.

Table 8 - Through-thickness strength results for flexural specimens.

Material	Width (mm)	Thickness (mm)	Depth (mm)	σ (MPa)
1	20	10	10	104.09 ± 5.20
	40	10	10	96.26 ± 3.52
2	10	10	10	39.35 ± 3.52
	20	10	10	51.46 ± 8.74
	40	10	10	52.56 ± 6.59

CONCLUDING REMARKS

The data presented in this measurement note suggests the following:

- The most reliable through-thickness tensile data was generated by the use of the standard Type C geometry.
- Higher strengths and similar, or improved, coefficients of variation are achievable on the miniaturised version of the Type C specimen but the specimen bending is unacceptably high.
- Novel test methods using thinner materials (i.e. <18 mm) proved unsuccessful in developing data comparable to the standard specimen.
- Improvements have been observed in the test data by the use of an alignment rig to allow accurate bonding of the specimen to the loading arms. Details of this should be included in the draft procedure.

Further work is required to:

- Investigate the high bending on the miniature version of the Type C specimen, as to why it had no effect on the failure strength.
- Assess the impact of different loading bars in order to make the test simpler and also help to improve the test data.

Once this work has been completed an inter-laboratory investigation should be conducted using the draft procedure in order to establish the reproducibility and repeatability of the modified test method.

References

1. S. Mespoulet, J.M. Hodgkinson, *Through-thickness test methods for laminated composite materials – Imperial college report TR99/03*
2. R.F. Ferguson, M.J. Hinton and M.J. Hiley, *Determining the through-thickness properties of FRP materials – Composites Science and Technology, Volume 58, Issue 9, 1998, pp 1411-1420*
3. W.R. Broughton and G.D. Sims, *An overview of through-thickness test methods for polymer matrix composites – NPL Report DMM(A)148, October 1994*
4. W.R. Broughton, R. Ferguson, M.J. Lodeiro and C. Messenger, *Development of through-thickness test methods for polymer matrix composites – NPL Report CMMT(C)10, July 1996*
5. M.J. Lodeiro, W.R. Broughton and G.D. Sims, *Understanding the limitations of through-thickness test methods – 4th European conference on composite testing and standardisation, 1998, pp 80-90*
6. W.R. Broughton, M.R.L. Gower, M.J. Lodeiro and R.M. Shaw, *Through-thickness testing of polymer matrix composites – NPL Measurement note MATC (MN)06, May 2001*
7. *Fibre-reinforced plastic composites – Determination of through-thickness tensile properties of fibre-reinforced plastic composites, NPL working draft, Version 3, March 2002*
8. ISO 527-5:1997, *Determination of tensile properties – Part 5: Test conditions for unidirectional fibre-reinforced plastic composites*
9. ISO 14125:1998, *Fibre-reinforced plastic composites – Determination of flexural properties*

Acknowledgements

The work reported in this Measurement Note was carried out by NPL as part of the “Measurements for Materials Systems” (MMS) programme funded by the United Kingdom Department of Trade and Industry. The authors acknowledge the contribution of their colleagues Dr W R Broughton and Miss M J Lodeiro.

The authors would also like to express their gratitude to SP Systems and Permali Gloucester for supplying the materials.

For further information contact:

Dr Graham D Sims
 NPL Materials Centre
 National Physical Laboratory
 Hampton Road
 Teddington
 Middlesex
 TW11 0LW
 Direct line: 020 8943 6564